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Karakterizacija blata - Fizikalna konzistenca - Tiksotropija (ļ asovna spremenljivost viskoznosti) in luģļ enje

Characterization of sludges - Physical consistency - Thixotropic behaviour and piling behaviour

Charakterisierung von Schlämmen - Bestimmung der physikalischen Konsistenz, thixotropes und Aufschüttverhalten

Caractérisation des boues - Consistance physique - Comportement thixotrope et comportement au tassement

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Characterization of sludges - Physical consistency - Thixotropic behaviour and piling behaviour

Caractérisation des boues - Consistance physique - Comportement thixotrope et comportement au tassement

Charakterisierung von Schlämmen - Physikalische Beschaffenheit - Thixotropes und Schüttverhalten

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Foreword

This document (CEN/TR 15463:2007) has been prepared by Technical Committee CEN/TC 308 “Characterization of sludges”, the secretariat of which is held by AFNOR.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This CEN-Report “Physical Consistency” derives from the Desk Studies “Physical Properties – Flowability” (HORIZONTAL Report No. 21 [60]) and “Physical Properties – Solidity, Thixotropic Behaviour and Piling Behaviour” (HORIZONTAL Report No. 22 [61]) of the *Horizontal* Project. The “*Horizontal*” project has the objective to develop horizontal and 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*” consisting in the development of Desk Studies on physical consistency, because it is recognized that this property is very important for the characterization of sludge, since it affects almost all treatment, utilization and disposal operations, such as storage, pumping, transportation, handling, land-spreading, dewatering, drying, landfilling. The importance of the physical consistency is also true for the characterization of bio-waste and soil. Also handling and utilization of many other materials, such as cement and asphalt are strictly depending on their physical consistency. The needs for control of operations and also material characteristics are described.

The first action carried out is consisted in searching for existing standards to be possibly used or adapted for utilisation in the specific field of consistency evaluation. The complete list of standards is reported in Annex 1 of the HORIZONTAL Reports No. 21 [60] and No. 22 [61], from which it can be seen that more than 250 standards and non-standardised methods are potentially applicable to consistency evaluation. 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 viscometers, Penetrometers, Rotational viscometers and Flow apparatus.
- Solidity: Shearing apparatus, Vane testing apparatus and Penetrometers.
- Thixotropic behaviour: It should be investigated a combination of methods for determination of the solidity like 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 was evaluated the laboratory or field test feasibility. Apparatuses of the measuring procedures and existing applications to different materials were described. On this basis the applicability of the described methods to the materials of investigation was evaluated and documented in the lists of analysed standards.

The recommended methods are for flowability the coaxial cylinder viscometer as laboratory apparatus, while field apparatus are flow cone, magnesium penetration cone and extrusion tube viscometer. The recommended methods are for solidity the “Laboratory vane shear apparatus” and “Vicat needle” as laboratory reference and the pocket penetrometers for field test. The penetrometers in general could be used for both laboratory reference method and field test. Also for determination of the thixotropic behaviour the penetrometer is together with an energy-input in terms of a vibrating table or a hammer a suitable instrument. For measuring the piling behaviour the Cubic Piling Box (CPB) and the Oedometer are the recommended methods, whereby the CPB could be used in both laboratory and field while the Oedometer could be used only in the laboratory. All methods should be tested and optimized to adapt design and part dimensions to the materials in a future experimental activity.

For the research needs first the basics of methods are explicated and the applicability of methods to the materials is clarified. The questions to be answered (precision, repeatability, reliability, etc.), the route, how to answer them and finally the steps to be taken are important for following procedures.

In the Horizontal Report No. 21 a total of 6 proposals for draft standards are given, whereby one laboratory method and five field tests exist. In the Horizontal-Report No. 22 a total of 11 proposals for draft standards are given, consisting of six laboratory methods and five field tests.

1 Introduction

1.1 The Horizontal project and the Work Package 7

The revision of the Sewage Sludge Directive 86/278/EEC, the upcoming Composting Directive on the biological treatment of biodegradable waste and the Soil Monitoring Directive call for standards on sampling, hygienic and biological parameters, methods for inorganic and organic contaminants, and for mechanical properties of these materials.

In addition, when materials cannot be utilized, landfilling becomes important, in which case leaching becomes an issue as stipulated by the Council Directive 1999/31/EC on the landfill of waste. More recently, a Council Decision establishing criteria and procedures for the acceptance of waste at landfills, pursuant to Article 16 and Annex II of mentioned Directive on the landfill of waste was issued (16/12/02) with physical consistency being one basic parameter of interest.

The "*Horizontal*" project has the objective to develop horizontal and 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.

Part of the work to be carried out will focus on *co-normative* work with an emphasis on horizontal standardization starting from existing standards developed for the same parameter in the fields of sludge, bio-waste and soil. Another part of the work will focus on *pre-normative* research required to develop standards lacking at this point and needed in the next revision of the regulations in these fields.

The work within the HORIZONTAL Project was coordinated in the Work Package 7 "*Mechanical properties*" and done in cooperation of the involved teams. It consists in the development of the Desk Studies on physical consistency mentioned above, because it is recognized that this property is very important for the characterization of sludge e.g., since it affects almost all treatment, utilization and disposal operations, such as storage, pumping, transportation, handling, land-spreading, dewatering, drying, landfilling. In fact, the selection of the most suitable equipment and procedure for land application, storage and transportation of sludge e.g. is strongly connected to its consistency. Similarly, compacting sludge in a landfill or forming a pile in composting is depending on sludge shear strength rather than on its solids concentration. In particular, with reference to the regulations requirements, according to the Sludge Directive 278/86, agricultural reused sludge should have agronomic interest, be healthy and easily usable, i.e. easily stored, transported, handled, and spread.

In Council Directive 1999/31/EC (Landfill Directive), Article 2 (q) gives a definition of "liquid waste", and Article 5 (3.a) does not allow a liquid waste to be landfilled, but a standardized method for this evaluation has to be developed yet. Further, Annex II (2. General principles) requires that "The composition, … and general properties of a waste to be landfilled must be known as precisely as possible", and Annex I (6. Stability) is referring to "... ensure stability of the mass of waste ... particularly in respect of avoidance of slippage", so the shear strength and piling behaviour should be known. Article 2 (h) says, that "treatment means ... processes ... in order to … facilitate its handling". Finally, Article 11 (1.b) asks for: " – visual inspection of the waste at the entrance and at the point of deposit and, as appropriate, verification of conformity with the description provided in the document submitted by the holder", so simple and easy tests to be carried out on the field and followed by the operators should be defined. Further, the Council Directive establishing criteria and procedures for the acceptance of waste at landfills, pursuant to Article 16 and Annex II of mentioned Directive on waste landfilling included "consistency" among the basic parameters to be evaluated for waste characterization before landfilling; for specific cases it is also demanded, that EU Member States must set criteria to ensure a sufficient physical stability and bearing capacity of waste. It is also to be pointed out that in many analytical methods for sludge characterization (e.g. pH, dry matter, leachability, etc.) different procedures are indicated depending on whether the sample to be examined is liquid or not, is solid or not, but no procedures are given for evaluating such properties. The importance of the physical consistency is also true for the characterization of bio-waste and soil.

1.2 Desk study subject

The Task Group 3 (TG3) of CEN/TC308/WG1 defined 3 physical states for sludge (CEN/TC308/WG1/TG3, 2000):

a) ***Liquid***: sludge flowing under the effect of gravity or pressure below a certain threshold.

b) ***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.

c) ***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”) should be evaluated, together with the *piling* behaviour referred both to “compaction and physical stability”. Also the 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 (CEN/TC292/WG2, 2002).

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 above properties, able to give comparable and reliable results, are not available yet. It therefore follows the necessity 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.

In the report “Globally Harmonized system of Classification and Labelling of Chemicals (GHS)” other definitions of liquid and solid are given [59]:

Liquid means a substance or mixture which at 50 °C has a vapour pressure of not more than 300 kPa (3 bar), which is not completely gaseous at 20 °C and at a standard pressure of 101.3 kPa, and which has a melting point or initial melting point of 20 °C or less at a standard pressure of 101.3 kPa. A viscous substance or mixture for which a specific melting point cannot be determined shall be subjected to the ASTM D 4359-90 test [56]; or to the test for determining fluidity (penetrometer test) prescribed in section 2.3.4 of Annex A of the European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR) [55];

Solid means a substance or mixture which does not meet the definitions of liquid or gas.

1.3 Evaluation of needs for control of operations and material characteristics

1.3.1 Evaluation of needs for control of operation

The purpose of using characterisation standards is to control and ascertain the material amenability to handling and different operations. Materials considered are

— Sewage sludge

— Waterworks sludge

— Bio-waste and

— Soil

Materials, which cannot be utilised, are subjected as waste to the Landfill Directive (Council Directive 1999/31/EC), respectively the ordinances of the member states. The member states had to translate this directive into national law. In Germany e.g., there is the landfill ordinance [58], which became operative on 24.07.2002. Furthermore there does exist the waste disposal ordinance, it is a kind of adjustment respectively update of the German “TA Siedlungsabfall”. By this regulation among other things the limit value of ≥ 25 kN/m² for vane shear strength – termed also in the HORIZONTAL Report No.22 [61] - was set. For this regulation it is not important, from where the materials come from. It is valid for different respectively all kinds of wastes.

For handling and operating these materials many parameters should have to be known; they include homogeneity, particles sizes and shape, solids (total, suspended, volatile) that, if available, could define the range of variation of variable considerations (i.e. viscosity, etc.).

The parameters flowability is an overall parameter taking into account all above mentioned material properties or characteristics. In particular, the flowability evaluation for sludges, including wastewater, waterworks and similar sludges, is of fundamental importance in many operations such as pumping, transportation, storage, dewatering, stabilisation, spreading, etc. also considering the possible formation from a gel to a liquid (sol) and vice versa. Similarly, for bio-waste, including the shredded organic fraction of municipal solid waste (OFMSW), in operations such as handling, digestion, reuse, etc. the measure of the parameter flowability have to be considered. Finally, for fine-grained soils, the water content (and therefore consistency and flowability) has always been considered an important indication of their mechanical properties. Moreover in case of soil slurries it is very important to verify flowability as a measurement of their workability and time of setting.

The solidity is also a parameter, which concerns all the material properties or characteristics mentioned above. The determination of this parameter is getting more important for handling of solid materials like dewatered sludge, other bio-waste – e.g. in terms of pieces (compost) – and soil, where the grain size distribution and water content have to be considered, during operations like pumping, transportation, storage, etc.

The measurement of thixotropic behaviour for solid materials is relevant especially for dewatered sludge like sewage, waterworks and related sludge. By dewatering and storage the sludge becomes solid. During operations such as transportation the sludge gets in a liquid state due to the vibration of a truck.

The piling behaviour evaluation is also for dewatered sludge, particular bio-waste and soil of importance. The determination of the piling angle is a useful instrument to characterise the storage properties and calculate the space, which is needed for e.g. storage and transportation. Together with the thixotropic behaviour the piling behaviour refers to the compaction and stability.

However, the development of reliable measurement procedures of all parameters is not a simple matter, because measurements are influenced by below described properties or characteristics. This means that those factors must be considered with great attention and methods defined by avoiding any negative interference with them during measuring procedures. For this reason, it is first essential to select, if any, the most adapted standards or non-standardised methods applicable to sludge, bio-waste and soil or to develop a new one, and then to carry out parallel tests to evaluate how they are affected by the other specific characteristics. In addition, these aspects require to be investigated for both laboratory methods, to be adopted as a reference, and simple tests to be applied in the field.

1.3.2 Material characteristics

1.3.2.1 Sewage sludge

Sewage sludge can be produced from several processes (primary sedimentation, activated sludge process, aerobic or anaerobic digestion etc.). Their solid content cover a wide range from 1 % to 30%, while different total volatile solids percentage on dry matter can vary from 75% to 45%. The presence of coarse particles is strongly related to the sieve adopted in head-works or external material used in some processes (anaerobic co-digestion, etc.). Sewage sludge covers a wide range of physical state from liquid to solid. Bibliography does not offer a characterization of particle size distribution of sewage sludge, a wide range of these characteristics is forecasting in relation to the process adopted (opening of sieves etc.) and different type of sewage sludge treated. Some indications are found for sewage sludge (see Table 1 [1]).

Table 1 — Particle size distribution of sewage sludges

Material	Process	TS basis % cumulative retained w.w.			TVS basis % cumulative retained w.w.		
		5 mm	2 mm	0,84 mm	5 mm	2 mm	0,84 mm
Sewage sludges WTS	Aerobic process	0	3,7	9,4	0	4,7	8,4
Mixed primary sludges ADS	Mesophilic anaerobic digestion	0	10,5	18,5	0	15,5	30,5

Each kind of sludge was analysed for its particle size distribution by wet sieving, using three sieves with openings of 5,2 mm and 0,82 mm. According to these data the samples can be divided into four conventional classes: coarse (>5 mm), medium (from 5 mm to 2 mm), medium-fine (from 2 mm to 0,84 mm) and fine (<0.84 mm). It can be noted that the sewage sludges have no coarse particles but a different percentage of medium and fine particles.

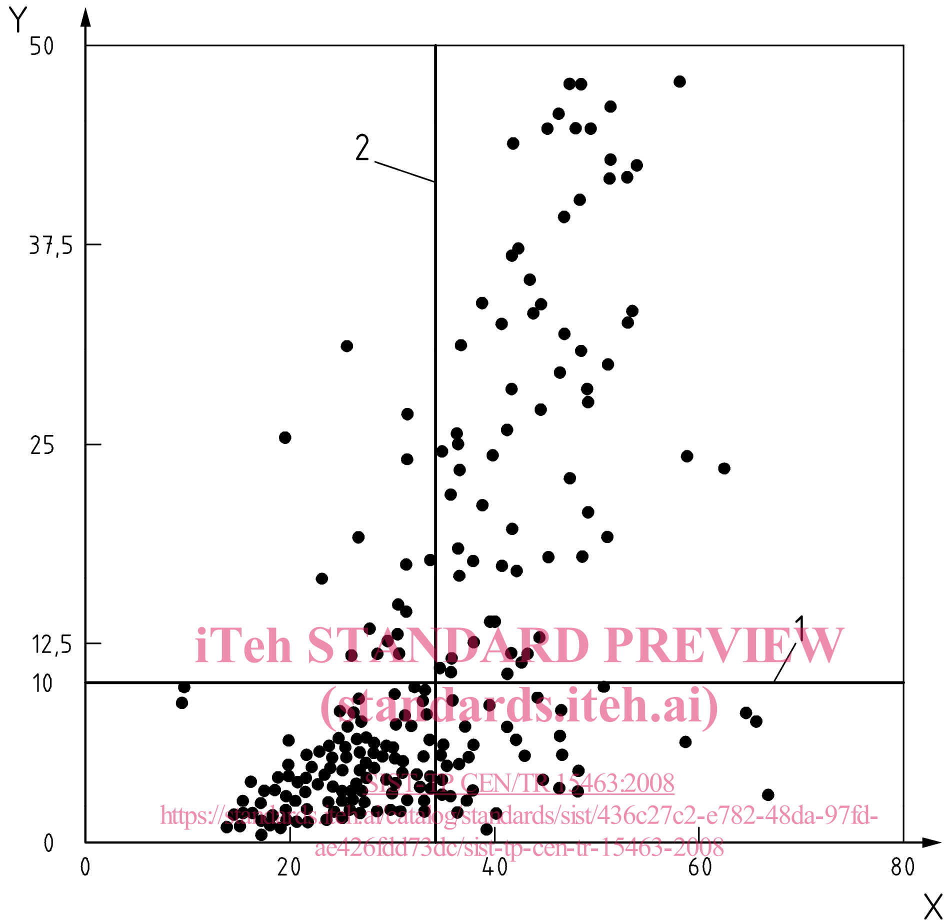
The most diffuse sludge characterization is that rheological, beside CST – capillary suction time, R specific resistance to filtration etc.. Rheological parameters (yield stress, viscosity and thixotropy) were originally applied to calculation of the head losses in sludge pumping operations, recently it has been shown that they can affect filtration, thickening [2], pumping [3, 4] and constitute useful on line control parameters for sludge conditioning and dewatering [5, 6, 7].

Rheological measurements of sewage sludges have been performed using commercial rotational viscometer. The rheological properties normally determined by using the Bingham plastic model are the yield stress (YS) (that is the stress required to start the material flowing) the plastic viscosity (that is the internal resistance to flow under defined shear rate). The Thixotropy, determined by the hysteresis area, is only sometimes observed (Table 2) [8].

Table 2 — Rheological properties of sewage sludges

Sludge	Process	TS range %	TVS/TS	YS Pa	Plastic viscosity mPa · s
Mixed		3,0-14	0,52	1,90-185	30-630
Primary		7,0-16	0,43	1,0-49	20-320
Activated		3,0-9	0,73	5,0-214	70-1110
Mixed Sludges	Aerobically stabilized	4,0-10	0,53	0,07-58	10-410
Mixed Sludges	Anaerobically digested	4,0-9	0,59	1,0-112	20-390
Mixed sludges	Mesophilic anaerobic digestion	3,5-5,0	0,5-0,6	0,4-1,6	8,0-24
Mixed sludges	Thermophilic anaerobic digestion	3,5-5,0	0,5-0,6	0,1-0,5	11,0-17

Mechanical properties of sewage sludge in solid state were studied with the aim to define the feasibility of landfill disposal; a correlation between shear strength and dry solid matter for sewage sludge was done using a Vane apparatus (Figure 1) [9], sludges are suitable for landfilling if their shear strength is of 10 kN/m² at least (limit value in Italy).



Number of values taken into consideration: 292

Key

X dry solid matter (%)

Y shearing strength (kN/m³)

1 minimum strength 10 kN/m³

2 35 %

Figure 1 — Collation between shearing strength and dry solid method [9]

1.3.2.2 Waterworks sludge

In DVGW W221-1 [10] sludges are defined as solid-water suspensions capable of flowing after sedimentation, flotation or thickening. Dewatered sludges are sludges, which were dewatered by natural or mechanical treatments until they are no longer able to flow.

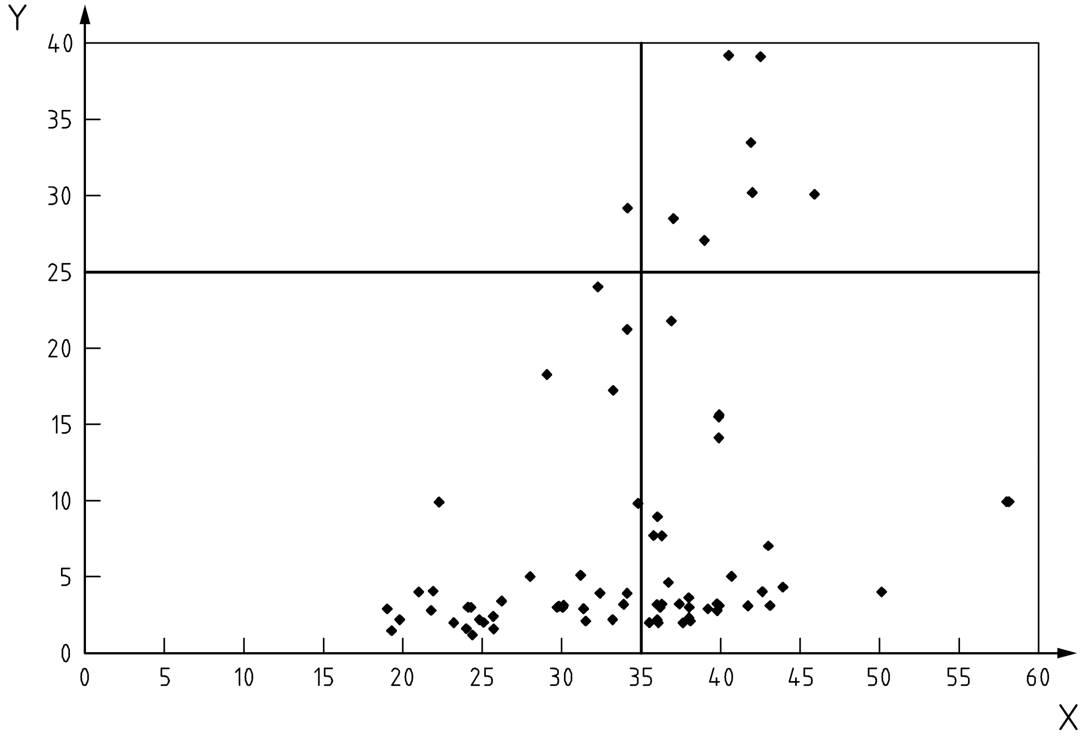
Sludges from water treatment contain several phases differing by their physical state and/or chemical nature. The space distribution of these phases, as well as the physical-chemical interactions between them, gives to sludges their cohesion. A too low cohesion of sludges and/or its high fluctuation in the time commonly generates handling (shovelling- and spreading- ability) and storing difficulties.

An orderly utilisation and disposal of waterworks sludges need the control of the mechanical properties in order to ensure a quality that is demanded for storage, transport and handling. The mechanical measurements should be seen and done in connection with other measurements that have been or will be standardised. There should be mentioned methods for chemical and physical parameters (chemical elements, dry solids, loss on ignition, pH-value) and operational methods (capillary suction time CST, specific filtration resistance). The different composition of waterworks sludges with inorganic (Fe, Ca, Al etc.) and organic (Algae, humid substances, powdered activated carbon etc.) substances depending on the source of raw water and water treatment processes should be considered. An inquiry from *Wichmann et al (2002)* [17] showed, that the waterworks sludges of different types in Germany amounted in 1998 to ca. 181.000 t DS (Lime sludge 40%, Iron sludge 14%, Fe-/Al-Flocculation sludge 13% and other 33%). The composition of the sludge can be determined after [11] (10 parameters) in Table 3.

Table 3 — Determination of the composition of sludge

Parameter	Fraction [g/kg DS] (Fictive value)	Conversion in:	Fraction [g/kg DS]
Acid insoluble (= $HCl_{ins.}$)	40	Insoluble components (e.g. sand, activated carbon)	40
TOC	30	Total organ. content (Factor: 2)	60
Mn	20	MnO_2 (Factor: 1.58)	31,6
Mg	5	$Mn(OH)_2$ (Factor: 2.,4)	12,0
Al	20	Al_2O_3 x H_2O (Factor: 2.22)	44,4
SO_4	5	$CaSO_4$ (Factor: 1.42)	7,1 (Ca: 2,1)
CO_3 (= TIC)	80	$CaSO_3$ (Factor: 1,67)	138,6 (Ca: 57,6)
Ca –total-	65		-
Residual-Ca	5.3	$Ca_3(PO_4)_2$ (Factor: 2,58)	13,7 (PO_4: 8,4)
PO_4 –total-	10		-
Residual-PO_4	1,6	$Fe(PO_4)$ (Factor: 1.59)	2,5 (Fe: 0,94)
Fe –total-	415,7		-
Residual-Fe	414.8	Fe_2O_3 x 1,5 H_2O (Factor: 1.67)	692,6
Total			1042,5

The range of 0,2 % to 80 % dry solids contents of waterworks sludge to be utilized is quiet wide, so that several different mechanical properties have to be measured. Possible measuring methods are coming mainly from the soil mechanical or rheological working fields. There are only few data on mechanical properties of waterworks sludge published. In Figure 2 after *Mc Tigue et al. (1990)* [12] e.g. the result of 72 measurement data of different waterworks sludge types and dewatering processes are shown. A laboratory vane shear apparatus was used. The vertical line marks the dry solids concentration of 35 % that was given from *LAGA (1979)* [13] as a minimum value for disposal of wastes in landfills. Sludge with more than 35 % DS were than be considered to be qualified for landfilling. The horizontal line marks the minimum value of 25 kN/m^2 concerning the vane shear strength that is now demanded in new regulations in the *TA Siedlungsabfall (1993)* [14]. Approximately 90 % of the waterworks sludges tested after mechanical dewatering could not fulfil the required minimum value.



Key

X dry solid [% w/w]
Y vane shear strength [kN/m²]

Figure 2 — Comparison dry solid contents vs. laboratory vane shear strength [12]

1.3.2.3 Bio-waste

Organic fraction of municipal solid waste (OFMSW) is utilized for anaerobic and composting treatment. Anaerobic digestion or co-digestion with sewage sludges is a well-known process where rheological parameters have been studied to control process.

Table 4 — Particle size distribution of OFMSW

Material	Process	TS basis % cumulative retained w.w			TSV basis % cumulative retained w.w		
		5 mm	2 mm	0,84 mm	5 mm	2 mm	0,84 mm
Fresh mechanically sorted (F) OFMSW	Mesophilic anaerobic digestion	8,2	11,1	18,3	6,7	21	23,5
Pre-composted mechanically sorted (P) OFMSW	Mesophilic anaerobic digestion			19,7			26,1
Blend of source sorted and mechanically sorted OFMSW	Mesophilic anaerobic digestion	6,6	11,5	18,3	12,7	16,8	24,2

The OFMSW was analysed for its particle size distribution by wet sieving, using three sieves with openings of 5, 2 mm and 0,82 mm. According to these data the samples can be divided into four conventional classes: coarse (>5 mm), medium (from 5 mm to 2 mm), medium-fine (from 2 mm to 0,84 mm) and fine (<0,84 mm).