

# Characterization of sewage sludges : fundamentals and results

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# **CHARACTERIZATION OF SEWAGE SLUDGES; FUNDAMENTALS AND RESULTS**

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## **SUMMARY**

An overview is given of a set of sludge characteristics. This set, which can be considered as a sludge fingerprint, is estimated to be of some relevance both for thermal and mechanical dewatering processes. Four different types of sludges have been studied, whereby flocculation was carried out under standard conditions in the laboratory. Some typical results are presented and some preliminary conclusions will be given with respect to the mechanical dewatering process and its relation with the preceding flocculation process.

## **1. INTRODUCTION**

In waste water treatment plants the dewatering of sludges by chamber filtration presses, sieve belt presses and centrifuges appears to be a unit operation which is very badly understood [Starkenbug and Rijs, 1991]. The main reason for this is the complexity of the sludge material. By no means sludges can be considered as well defined systems with "beautiful" and constant properties. Numerous chemical components might be encountered, a great variety of shape and size of colloidal and non-colloidal solid particles may be found. Both flocculated and unflocculated particles may be deformable and sensitive to shear stresses and may be disrupted in mixing and stirring processes. Moreover, when flocculated solid particles are collected in a filtration process the formed filter cake appears to be highly deformable as well. This means that by exerting some mechanical forces to the cake material the porous structure collapses and a lot of water remains entrapped within the cake. It is believed that this mechanism is determining to a high extent the attainable final dry solids content in a mechanical dewatering process.

Nowadays there are environmental and economical reasons to strive for a higher dry solids content of sludge cakes. This is explained in more detail elsewhere (La Heij and Kerkhof, 1993). It will be clear that achieving a better dewatering process requires better knowledge of the fundamentals, better design rules for the dewatering equipment and better control strategies.

It is common practice in process technology research to perform studies with well defined model systems having all the relevant properties of the real system. However with respect to sludges this is not an easy approach, because till now the ruling properties are not known

sufficiently well.

In this study it is the aim to establish a set of sludge characteristics. A set that may be considered as a *fingerprint* of the sludge. Subsequently several real sludges (see section 3) are to be characterized; finally the characteristics are to be cross (cor)related and are also to be (cor)related to the dewatering behaviour of the sludges in well defined laboratory tests and in practical plant dewatering equipment. This study is carried out in the Sludge Dewatering Project Team and therefore this paper is closely related to a second contribution from this team to this workshop (La Heij and Kerkhof, 1993).

## 2. SURVEY OF CONSIDERED PROPERTIES

The sludge fingerprint is based on properties that can be divided into the following classes:

- Origin and history
- Composition
- Thermal properties
- Colloidal properties
- Dewatering properties

The properties belonging to each class are summarized below.

### *History*

- origin of waste water (domestic/industrial)
- type of waste water treatment plant
- typical information about plant operation

### *Composition:*

- Dry solids content
- Ash content
- ATP content
- pH
- Electrical conductivity

### *Thermal properties:*

- Sorption isotherms at several temperatures
- Isothermal drying curves (TGA/DTA)
- Freezing curves (DSC)
- Bond enthalpy of water in sludge

Thermal properties are also of immediate importance for thermal dewatering by means of drying processes.

### *Colloidal properties:*

- Sludge Volume Index (SVI) of unflocculated and flocculated sludge
- Particle size distribution and morphology
- Electro Sonic Analysis signal (related to zeta-potential)
- Optimum dosage of flocculant
- Concentration of flocculant in filtrate
- Strength of flocs from rheology

*Dewatering properties:*

- Specific cake resistance and porosity (permeability)
- Cake compressibility (mechanical properties of cake)
- Dry solids content of filtration cake
- Vacuum suction time (VST)
- Capillary Suction Time (CST)

It may be expected that these characteristics are closely related to the real mechanical dewatering processes in water treatment plants by means of chamber filtration press, sieve belt press and centrifuges.

### 3. ORIGIN AND HISTORY OF SLUDGES INVESTIGATED

Different types of sludges may be distinguished depending on the origin of the waste water and on the type of waste water treatment plant. Waste water entering the sewer system originates from households (domestic sewage) and/or from industries (industrial sewage). The following main types of sludges, depending on the waste water treatment, are recognized:

- Primary sludge, which is separated from the incoming waste water in the primary settling tank.
- Secondary sludge, withdrawn from the secondary settling tank. In the preceding step this sludge has been submitted to a biological treatment in an aeration tank, where the organic matter is oxidized.
- Digested sludge or anaerobically stabilized sludge. In a digestion process the sludge is stabilized by anaerobic metabolic processes, in which organic carbon compounds are converted into biogas (methane and carbon dioxide).

In this study four sewage sludges, originating from four different waste water treatment plants, have been characterized. Below some typical characteristics concerning origin and history of the different sewage sludges are listed.

TABLE 1. Survey of sludges investigated

Name of plant	domestic/ industrial	primary, secondary	digested, undigested
Mierlo	60/40	mixture	undigested
de Lage Zwaluwe	100/0	secondary	undigested
Amsterdam-East	100/0	mixture	digested
Veghel	35/65	secondary	undigested

It should be mentioned that all above listed sludges are flocculated in the laboratory, because the industrial conditions of flocculation are not known sufficiently well. Under laboratory conditions the flocculation conditions are better known and at least the same for all sludges. Moreover, the flocculation process appears to be a very critical operation which affects the

dewatering properties of the sludge to a great extent. An ill defined flocculation process would therefore cause a lot of problems in understanding the dewatering properties of sludges.

## **4. CHARACTERIZATION METHODS**

### **4.1 COMPOSITION**

To determine the **dry solids content** of a sewage sludge or a filter cake the sample is dried in a furnace at 110 °C during 24 hours. The addition of inert materials (e.g. during flocculation) causes an artificial augmentation of the dry solids content. By comparing the effect of certain treatments (e.g. flocculation) on dewatering properties in this study the water removal was related to the *initial* dry solid content of the sludge.

The **ash content** is related to the inorganic fraction of the solids in the sludge and is determined by burning the dried sample at 600 °C during 30 minutes.

The **Adenosine TriPhosphate (ATP) content** of a sewage sludge sample is used as a measure for the viable biomass. The determination of the ATP content is based on the luminescent reaction of ATP with luciferase, in which light production is proportional to ATP present [Patterson et al., 1970].

The **pH** and the **electrical conductivity** are ordinary laboratory routines and do not need further introduction here.

### **4.2 THERMAL PROPERTIES**

#### **4.2.1 Isothermal drying curves (TGA/DTA)**

Knowledge about the solid-water bond strength in sludge as a function of the moisture content enables the prediction of the maximum theoretically feasible dry solids content in a certain dewatering process. The solid-water bond strength can be obtained from isothermal drying curves and from water vapour isotherms.

With thermogravimetric analysis (TGA) the mass loss of a sludge filter cake, due to vaporization of water at a constant temperature of 60 °C, is measured continuously. In this way an isothermal drying curve is obtained. From differential thermal analysis (DTA) the heat flow required for the vaporization of water can be calculated. The TGA/DTA equipment provides the possibility to carry out TGA and DTA simultaneously. The ratio between vaporization rate (in kg/s), calculated from the TGA experiments, and heat flow to the sample (in J/s) is equal to the enthalpy of vaporization of water present in the sample (in J/kg). The bond enthalpy is defined as the difference between the actual measured enthalpy of vaporization and the enthalpy of vaporization of pure water. With this technique the bond enthalpy can be calculated as a function of the sample moisture content. Moreover, from the falling rate period of drying curves moisture diffusion coefficients can be derived [Coumans, 1987].

#### **4.2.2 Water vapour (de)sorption-isotherms at different temperatures**

A water vapour (de)sorption-isotherm of a substance is the equilibrium relationship between the amount of water in the substance and its thermodynamic water activity at a constant

temperature. Water vapour desorption isotherms of sludge cake samples were determined with the conventional technique of vacuum exsiccators with (super)saturated aqueous salt solutions to control the water activity. The equilibrium data for the water activity of these solutions were taken from the tables given in literature [Greenspan, 1977]. For keeping a constant temperature during equilibration the exsiccators were positioned in a water bath. In an experiment twelve sludge cake samples were placed in twelve exsiccators, each with their own water activity and covering the whole range of wateractivities between 0 and 1. Equilibrium was assumed if two subsequent sample weighings gave the same results. At reaching equilibrium conditions, the moisture content of the samples was determined and the desorption isotherm was constructed. In this way desorption isotherms were measured at three different temperatures. From the three isotherms, the differential enthalpy of wetting as a function of the moisture content can be calculated by applying the Clausius-Clapeyron equation. The differential enthalpy of wetting corresponds thermodynamically with the bond enthalpy. In Figure 1 the results of both techniques, carried out with Amsterdam sludge flocculated with 27% FeCl<sub>3</sub>, are given.

The discrepancy between the two curves for moisture contents lower than 0.2 kg water/kg dry solids is due to the different experimental conditions: the moisture content of the sample in the exsiccator is determined in an equilibrium situation, whereas in the TGA equipment no equilibrium condition occurs. Moreover desorption isotherms were not measured at very low water activities (< 0.05). So the bond enthalpy calculated in in this region are extrapolated values which might be unreliable.

Moreover, it can be concluded that the bond enthalpy starts to deviate significantly from zero at a moisture content of about 0.4 kg water/kg dry solids.

In sludge filter cakes the moisture content amounts about 4 kg water/kg solids (=20%DS), which means that about 90% of the water in the filter cake is present as *free water* and should, from this point of view, be removable by mechanical dewatering processes. The remaining 10% having a higher bond enthalpy, the so-called *bound water*, can only be removed in e.g. drying processes.

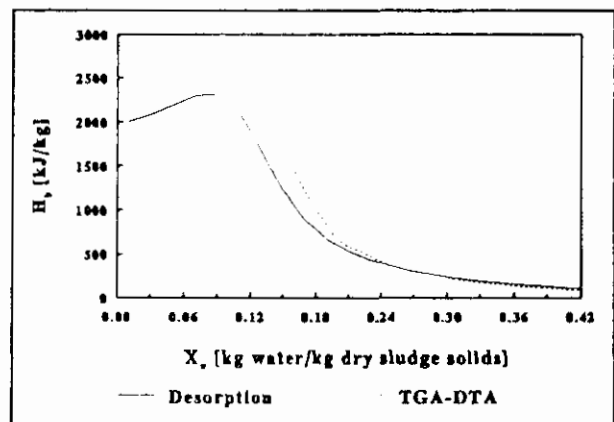


Fig. 1. Bond enthalpy  $H_b$  as a function of moisture content  $X_w$ .

## 4.3 COLLOIDAL PROPERTIES

### 4.3.1 Particle size distribution

The "image analysing technique" has been used to study the particle size distribution of a sewage sludge sample. The experimental equipment consists of an optical microscope, camera and computer. A picture of a sludge sample is registered and digitalized and shown on the screen of the computer system. The surface area of the particles is determined and converted into an effective diameter.

The frequency of the effective diameter is based here on the number of the particles with that

size. The measured cumulative particle size distribution is approximated by the Harris equation  $F(x)$  [Svarovsky, 1990]:

$$F(x) = 1 - \left[ 1 - \left( \frac{x}{x_0} \right)^a \right]^b \quad (1)$$

where  $x_0$  is the maximum diameter,  $a$  and  $b$  are constants. The Harris equation, as represented above, describes an "undersize" cumulative frequency distribution, so  $F(x)$  indicates the fraction of particles with a diameter smaller than  $x$ .

The particle size distribution of sewage sludge samples, flocculated with different types of flocculants and with different dosages of flocculant, are determined and evaluated according to the above method. In Figure 2, typical particle size distributions are given for sewage sludges flocculated with different polyelectrolyte (Rohafloc KF975) dosages. The effect of an increasing amount of polyelectrolyte is the shifting of the distribution curves towards greater diameters.

The median diameter is easily found at  $F(x)=0.5$ . The median diameter appears to be a good characteristic to indicate the particle size. Typical values for the median diameter are:

- 5-10  $\mu\text{m}$  for particles in unflocculated sludges;
- 8-20  $\mu\text{m}$  for particles in sludges flocculated with  $\text{FeCl}_3$ ;
- 500-2000  $\mu\text{m}$  for particles in sludges flocculated with polyelectrolyte (Figure 2).

The particle size also depends on the type of flocculant used and on the stirring and mixing conditions during the flocculation process.

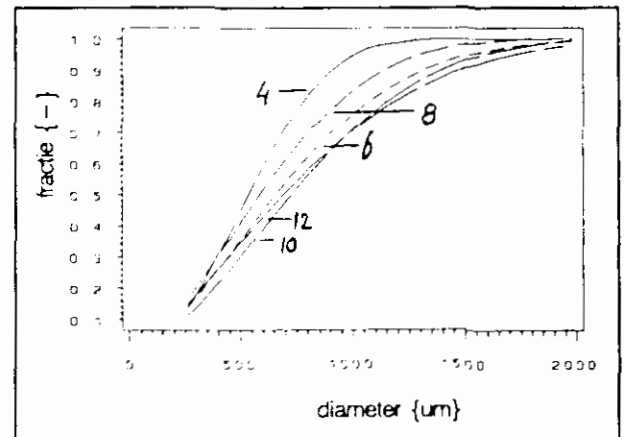


Fig. 2. Cumulative particle size distribution for Veghel sludge flocculated with different dosages polyelectrolyte KF975 (in g/kg ds)

#### 4.3.2 Strength of flocs

During the filtration and expression of sewage sludge shear stresses are exerted on the sludge particles. Sewage sludge is a colloidal dispersion and it is interesting to study its rheological properties. A Searle type coaxial cylinder rheometer is used to determine the rheological behaviour of sewage sludges. A sewage sample is introduced into the small gap between two cylinders. The inner cylinder rotates and the outer cup remains stationary. In an experiment, the angular velocity of the spindle is increased in 15 steps until the maximum speed and is subsequently decreased. The torque needed to maintain the angular velocity is measured and converted to a shear stress. The result of such an experiment is called a rheogram. A typical rheogram of a sludge flocculated with  $\text{FeCl}_3/\text{Ca}(\text{OH})_2$  is illustrated in Figure 3. It is notable that the ascending and descending curves do not coincide. This phenomenon is called thixotropy. It physically means that sludges possess an internal structure which breaks down

as a function of time and shear rate. The surface area between the two curves can be interpreted as the dissipated power per unit volume and is a measure for the strength of particles or flocs.

Unfloculated sludges do not show thixotropic behaviour. Sludges flocculated with  $\text{FeCl}_3/\text{Ca}(\text{OH})_2$  and polyelectrolyte exhibit a maximum value of the thixotropy at a certain dosage.

Rheological behaviour of sewage sludges can be interpreted as pseudoplastic flow:

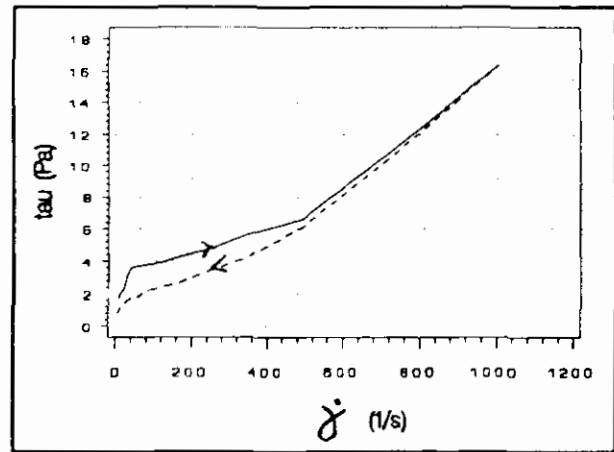


Fig. 3. Rheogram of Mierlo sludge flocculated with 50 g/kg ds  $\text{FeCl}_3$

$$\tau = \tau_0 + k(\dot{\gamma}, t)\dot{\gamma} \quad (2)$$

$\tau_0$  is an initial characteristic shear stress and  $k(\dot{\gamma}, t)$  is called the plastic viscosity, which depends on time and shear velocity.

The viscosity  $k$  as a function of the shear velocity  $\dot{\gamma}$  is calculated from the measured rheogram. The plastic viscosity for flocculated sludges decreases with increasing shear velocity. The plastic viscosity can be determined as a function of time in a stationary shear experiment. It appears that for flocculated sludges the viscosity decreases with time. The decline in viscosity is stronger for higher dosages of flocculant ( $\text{FeCl}_3$  and polyelectrolyte). Unfloculated sludges show no reduction of viscosity as a function of time.

#### 4.3.3 Zeta potential

Unfloculated sludge particles are negatively charged. A measurable parameter which is proportional to the surface charge density of particles is the zeta potential. The zeta potential, often used to characterize the electrostatic stability of colloidal suspensions, can be determined with the so-called electro-acoustic technique (MATEC). In this technique the colloidal suspension is submitted to an alternating electric field. The relative motion between the particles and the liquid generates a sound wave at the frequency of the electric field. This effect has been termed the Electrokinetic Sonic Amplitude (ESA), which is strongly related to the zeta potential of the particles in the colloidal suspension.

In Figure 4 a schematic diagram of the electro-acoustic cell is given. The vessel is stirred and includes sensors for pH, temperature, conductivity and electro-acoustic measurements. The zeta potential is determined from the measured electro-acoustic data. It is possible to perform automatic volumetric titration experiments. This equipment is very suitable for studying the flocculation mechanism because continuous measurement of the ESA-signal is possible during titration of flocculant to a sludge.

A typical example of the ESA signal as a function of the  $\text{FeCl}_3$  concentration in sewage sludge is given in Figure 5. At a certain dosage a sudden change of the ESA signal occurs due to adsorption effects. Iron hydroxide complexes adsorb at the surface of the



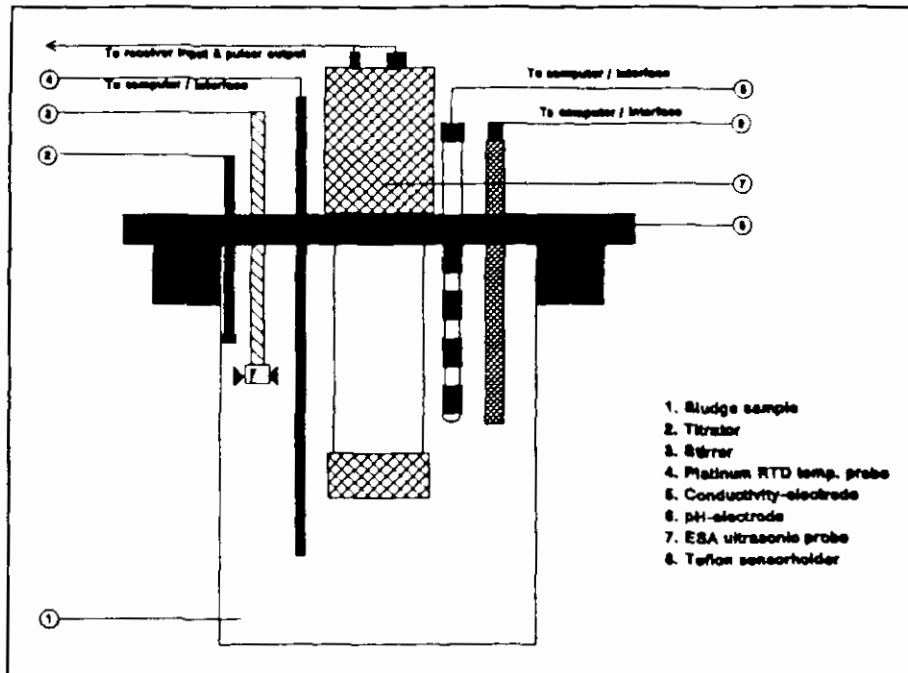


Fig. 4. Schematic diagram of the MATEC ESA sample cell

sludge particles. Sludge particles surrounded by layers of metal hydroxide complexes stick to each other and so flocs are formed. At a certain dosage of flocculant the adsorption of iron hydroxide complexes causes a change of the electric charge of the particles. The ESA-plots and more specific the charge transition point can be considered as sludge characteristics. The charge reversal point (ESA=0) corresponds theoretically to a number of bulk properties of the suspension: maximum dewaterability and minimum electrostatic stability. In the experiments carried out with  $\text{FeCl}_3$ , the dosage where ESA is zero often corresponds with a minimum specific cake resistance.

Unfortunately, so far it seems that the MATEC-ESA system does not provide a reliable and suitable technique for studying sludge floc formation processes induced by organic flocculants.

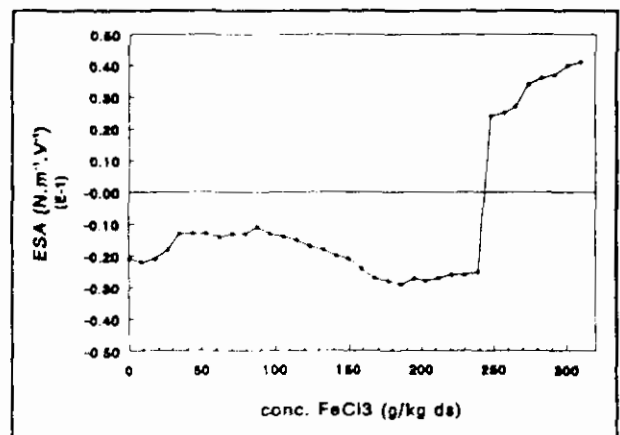


Fig. 5. Measured ESA-signal as a function of concentration  $\text{FeCl}_3$  in Mierlo sewage sludge

## 4.4 DEWATERING PROPERTIES

### 4.4.1 Specific cake resistance and cake permeability

The solid-liquid separation of sewage sludge can be subdivided into a filtration phase and an expression phase [La Heij and Kerkhof, 1993]. In order to study the solid-liquid separation process and to characterize the dewatering behaviour of sludges a filtration-expression cell has been developed. A schematic diagram of this cell is given in Figure 6.

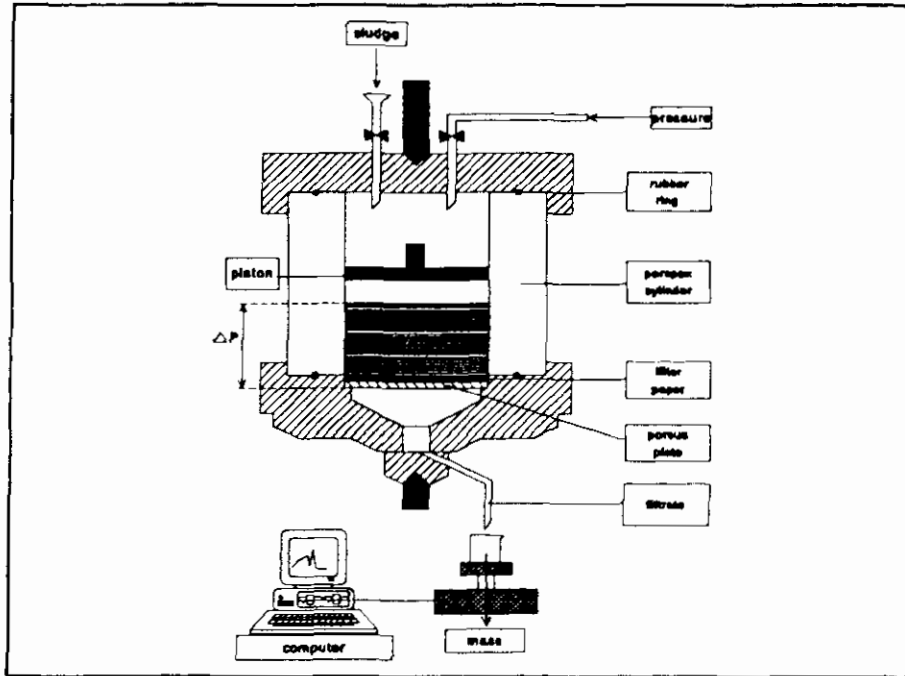


Fig. 6. Schematic diagram of the filtration-expression cell

At the beginning of the experiment a gas pressure is suddenly exerted on the sludge sample. The gas pressure is the driving force for the solid-liquid separation. A filter cake is built up and the filtrate is collected in a beaker glass positioned on a balance. The balance is connected to a computer, which registrates continuously the weight of the filtrate (and thus also the filtrate volume). With this experimental set up it is possible to carry out experiments under different process conditions such as gas pressure, type of flocculant, flocculant dosage and amount of sewage sludge (or final cake thickness). In the Figures 7 and 8 typical results are given for sewage sludges flocculated with respectively  $\text{FeCl}_3$  in combination with  $\text{Ca}(\text{OH})_2$  and polyelectrolyte. The experimental results are fitted with the integrated form of Darcy's law, in which filtration time  $t$  is related to filtration volume  $V$ :

$$t = \frac{\alpha \mu C}{2A^2 \Delta P} V^2 + \frac{\mu R_m}{A \Delta P} V \quad (3)$$

From the fitted equation, the average specific cake resistance  $\alpha$  can be calculated. Knowing the average porosity  $\epsilon$  of the cake, the cake permeability  $K$  can be calculated according to:

$$K = \frac{1}{\alpha \rho_s (1 - \epsilon)} \quad (4)$$

Though the above equations are valid only for incompressible filter cakes they still can be used for characterization of the compressible sludge cakes [La Heij and Kerkhof, 1993]. For modelling purposes and to obtain a better understanding of the dewatering process of a compressible cake also a so-called *compression-permeability cell* has been developed [La Heij and Kerkhof, 1993].

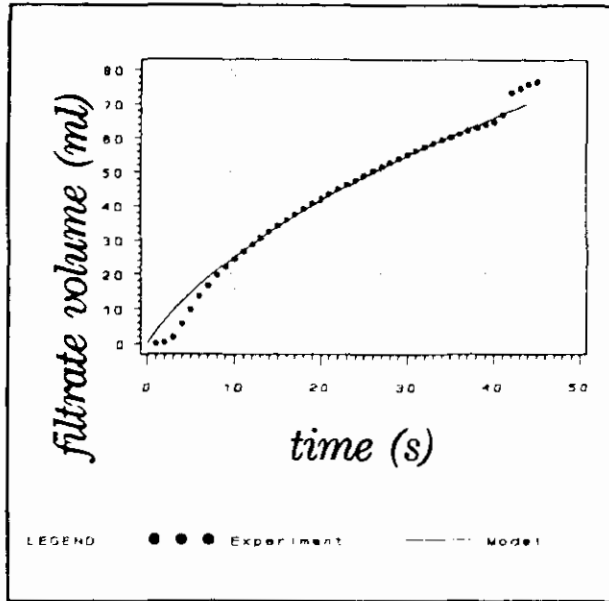


Fig. 7. Result of filtration experiment carried out with Mierlo sludge flocculated with 9 wt%  $\text{FeCl}_3$  and 20%  $\text{Ca(OH)}_2$  on dry solids base

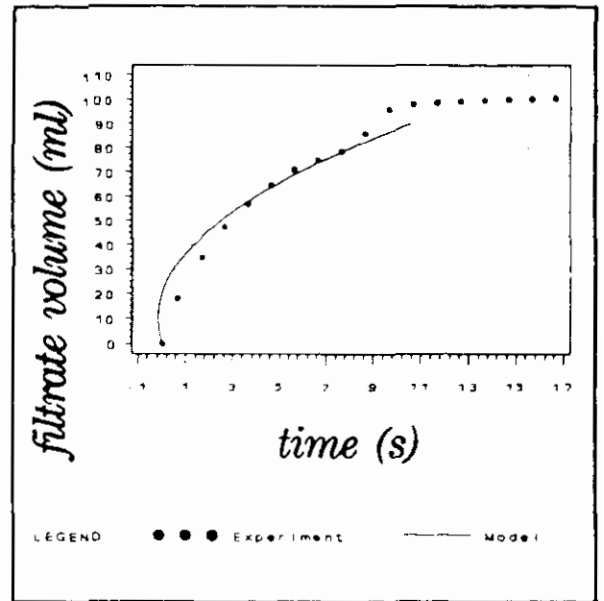


Fig. 8. Result of filtration experiment carried out with Mierlo sludge flocculated with 0.9 wt% polyelectrolyte on dry solids base

In both experiments (Figures 7 and 8) a sudden change in filtrate volume can be observed (40 s respectively 9 s) due to the formation of cracks in the filter cake.

Typical differences between the filtration behaviour of sludges flocculated with respectively  $\text{FeCl}_3/\text{Ca(OH)}_2$  and polyelectrolytes are:

- The dosages of  $\text{FeCl}_3$  and  $\text{Ca(OH)}_2$  needed to obtain a good dewatering result is about ten times higher than the dosage of polyelectrolyte.
- The average specific cake resistance for sludges flocculated with  $\text{FeCl}_3/\text{Ca(OH)}_2$  is about five to ten times smaller than the average specific cake resistance for sludges flocculated with polyelectrolyte.

#### 4.4.2 Modified Filtration Test

With the Modified Filtration Test (MFT) filtration experiments are carried out with a constant under pressure of 0.5 bar [Heide and Kampf, 1978]. In Figure 9 a schematic

drawing of the MFT-equipment, consisting of three parallel filtration tubes, is given. In this set up three experiments can be carried out simultaneously.

A flocculated sludge sample of 100 ml is introduced into the Büchner funnel. By immediately applying the under pressure the filtration process starts and liquid starts flowing into the filter tube. The filtrate volume can be read from a scale on the tube. After the cake has become somewhat dry, a plastic foil is positioned on top of the cake. Next a water layer of about 3 cm is applied on the foil and the expression phase starts. After 10 minutes the experiment is stopped. From this test the following dewatering characteristics can be determined:

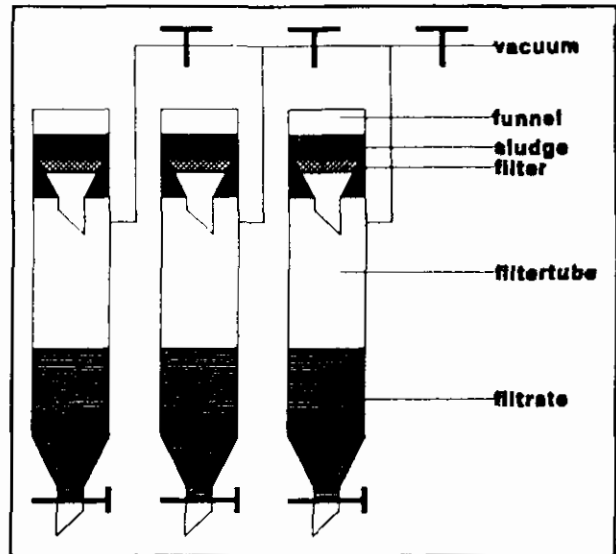


Fig. 9. Schematic diagram of the Modified Filtration Test

1. **"Vacuum" suction time (VST)**, which is defined as the time needed to collect 60 ml of filtrate at an under pressure of 0.5 bar. The VST is a measure for the filtration rate.
2. **Final dry solids content** of the sludge cake based on total solids (sludge solids plus flocculant plus other additives).
3. **Water content of the sludge cake**, expressed in kg water per kg initial dry solids in the sludge sample. This moisture content is corrected for the dry solids from additives and provides a better comparison of the effect of e.g. the nature and amount of flocculant on the dewatering process.
4. **Optimum dosage of flocculant** (which can also be considered as a colloidal property) is defined as the dosage of flocculant which yields the lowest water content of the sludge cake.

The influence of type and dosage of flocculant on the dewatering characteristics can be studied easily with this simple experimental device.

#### 4.4.3 Capillary Suction Time

The Capillary Suction Time (CST) device is a simple instrument to determine the dewaterability of sewage sludges (Figure 10). At the start of an experiment, sludge (2) is poured into the cylindric reservoir (1), resting on the filter paper (6). Under the influence of the capillary suction of the fine capillary-porous paper and the gravity force, filtrate is drawn out of the sludge to saturate progressively a greater area of the filter paper, causing the liquid front to advance outwards from the centre. When the filtrate front reaches the first electrode (4), the timer (5) starts. When the filtrate reaches the second electrode, the timer stops. The capillary suction time is then read directly from the timer.

The lower the CST the better is the filtrability of the sludge.

The CST-test can be considered as a gentle filtration process where sludge cakes are exerted to very low forces.

Results of experiments carried out with unflocculated sludges show a bad reproducibility. The CST method appears to be very suitable and reproducible for sludges flocculated with  $\text{FeCl}_3$ . The instrument appears to be not appropriate for sludge samples consisting of relatively large particles (500-2000  $\mu\text{m}$ ), e.g. sludge samples flocculated with polyelectrolytes.

#### 4.4.4. Sludge Volume Index

The sludge volume index (SVI) is a measure for the sedimentation *rate* of sludge particles. The SVI is defined as the volume taken by sludge particles after 30 minutes of sedimentation in a sewage sludge sample containing 1 g/l dry solids. The SVI is frequently used in waste water treatment plants as a sludge characteristic.

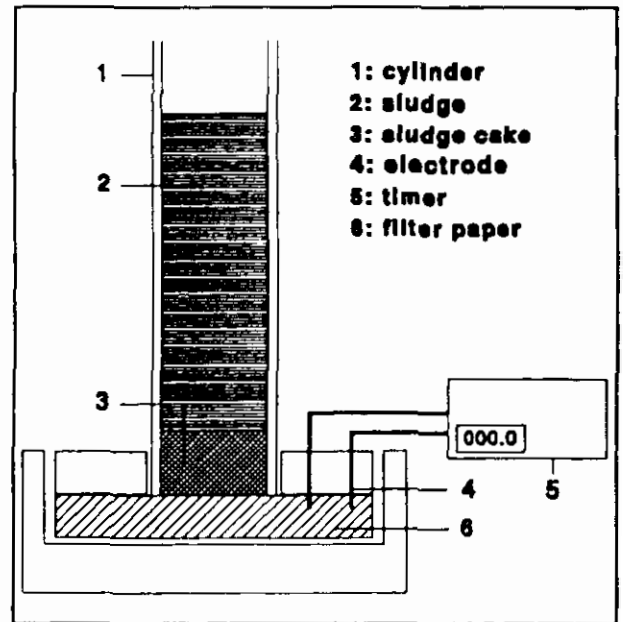


Fig. 10. Diagram of CST device

## 5. CONCLUSIONS

This research is still in progress and unfortunately not all experimental results are evaluated yet, nor are (cor)relations between characteristics fully studied so far. This means that only some preliminary conclusions, valid for all four sludges investigated, can be drawn here:

- Dewatering characteristics appear to be strongly dependent on flocculation conditions (nature and dosage of flocculant, intensity and duration of mixing and stirring). This conclusion, perhaps an open door to many experts, is still emphasized here once more. It is believed that a bad sludge dewatering process, also in real plant practice, may be caused by bad flocculation conditions in the first place. Therefore every fundamental study of dewatering characteristics will soon become a study of flocculation phenomena.

- For obtaining optimum flocculation, and thus the highest dewatering effect in the shortest time, the following characterization tests could be used for controlling the dosage of flocculant:

- Filtration-expression test
- Modified filtration test
- Capillary Suction Time test
- Rheology test
- Image Analyzing test

- By preference the Modified filtration test should be used because of its simplicity and because it gives information both about the final moisture content of the sludge cake and the dewatering rate.

- Sludge volume index, ATP, pH and electric conductivity do not show any correlation with the dewatering characteristics and can *not* be used for optimum flocculant dosage. Due to

analytical problems it is not clear so far, whether the flocculant concentration in the filtrate could also be used as an indicator for the quality of the flocculation process or not.

- By using polyelectrolytes as flocculant both degree and rate of sludge dewatering is higher than by using  $\text{FeCl}_3/\text{Ca}(\text{OH})_2$ .

## ACKNOWLEDGEMENTS

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## LIST OF SYMBOLS

a	constant	-
A	filter area	$\text{m}^2$
b	constant	-
$H_b$	bond enthalpy	$\text{J kg}^{-1}$
k	plastic viscosity	$\text{Pa s}$
K	permeability	$\text{m}^2$
$\Delta P$	pressure difference	$\text{Pa}$
$R_m$	filtermedium resistance	$\text{m}^{-1}$
t	time	s
V	filtrate volume	$\text{m}^3$
x	diameter	m
$x_0$	maximum diameter	m
$X_w$	moisture content	$\text{kg water}(\text{kg initial dry solids})^{-1}$

## Greek symbols

$\alpha$	specific cake resistance	$\text{m kg}^{-1}$
$\epsilon$	porosity	-
$\mu$	viscosity filtrate	$\text{Pa s}$
$\tau$	shear stress	$\text{Pa}$
$\tau_0$	yield stress	$\text{Pa}$
$\dot{\gamma}$	shear velocity	$\text{s}^{-1}$

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