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RHEOLOGY OF SEWAGE SLUDGE RHEOLOGICAL PARAMETERS AND SLUDGE CHARACTERISTICS

The rheological properties of sewage sludges were studied. A number of sludges from many wastewater plants and a wide range of solid contents (TS) were examined. Total volatile solids (TVS), surface acidic groups (GAT) and capillary suction time (CST) were correlated with yield stress and rigidity coefficient. A semiempiric model taking advantage of each single property offers a method for calculating the rigidity coefficient which is alternative to rheological measurement.

1. INTRODUCTION

Rheology provides a fluidodynamic characterization and useful information on the physical properties of sewage sludges describing their behaviour in wastewater plants and sludge management processes. The rheological parameters were originally applied to calculation of the head losses in sludge pumping operations [1], [2]; recently, it has been shown that they can affect filtration, thickening [3], [4], dewatering and constitute useful on-line control parameters for sludge conditioning [5],[6]. Measurement of rheological parameters of sewage sludge is complicated due to a number of important factors, including the point of measure (in line, out of line), the choice of instruments, the range of shear rate and the different sizes and nature of particulate matter. Unfortunately, the behaviour of sewage sludge varies with the sludge type, making the comparison between the results obtained by different authors difficult, which hinders the use of rheological properties in sludge treatment. Only the individualization of a general mathematical model or a simple indirect method for estimating the viscous parameters of sludge can be the way of including viscosity in a design and control of the processes.

It is surprising that the literature of the past 30 years on sludge rheology lacks any methodological information. It is restricted to viscosimeter choice and data treatment;

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only for chemically conditioned and anaerobically digested sludge a methodology for measuring rheological parameters was developed [5].

In [7] and [8], we suggested a standardized methodology for rheological measurements. We found that there is no substantial difference between a plastic and pseudoplastic model. A preliminary correlation between rigidity coefficient or yield stress and physico-chemical properties was also presented. This research extends the investigation over a wide range of sludge types with the aim to confirm the results obtained and to study the parameters explaining the rheological behaviour.

2. BACKGROUND

According to the recent works, two classes of viscosimeters appear to be most suitable for measuring rheological parameters. A pipeline viscosimeter is generally used to evaluate in-line head losses to design pipeline-pumping systems [1], [2], [9]. Coaxial cylinder

Table 1

Characteristics of the sludges

Period	Sludge	pH	TVS/TS (%)	SOUR (mg O_2 /g TVS h)	TS range (%)
1988	A-1 mixed	7.1	52.2	24	3.0–14.0
	B-1 aerobically stabilized (20 d)	7.5	52.8	5	4.0–10.0
	C-1 activated	6.9	72.8	32	3.0–09.0
	D-1 primary	6.7	42.8	15	7.0–16.0
	A-2 mixed	7.0	67.3	31	3.0–12.0
	E-1 anaerobically digested	7.4	59.0	8	4.0–09.0
1989	A-3 mixed	7.1	75.1	30	1.5–08.0
	B-2 aerobically stabilized (5 d)	7.2	73.1	15	1.5–06.0
	B-3 aerobically stabilized (9 d)	6.7	70.5	10	1.5–05.0
	B-4 aerobically stabilized (20 d)	7.0	67.7	8	1.3–06.0
	C-2 activated	6.9	88.3	40	1.5–04.7
	D-2 primary	7.1	60.6	30	1.4–07.0
	E-2 anaerobically digested	7.6	62.0	7	2.3–06.2

rotational devices are most common in laboratory tests, because they enable mathematical description of flow and make the execution of tests easier [10], [11]. At a low shear rate, no significant differences were observed, when comparing the data obtained with the aid of a commercially available rotational viscosimeter to the data obtained using a modified version. The latter was adopted to counteract gravity sedimentation of particles and to provide a continuous upward flow [12].

It is generally accepted that sewage sludges are non-Newtonian fluids, but there is a disagreement among research workers about the model formulation. Thus, either the plastic models of Herschel, Bulkley and Bingham or the pseudoplastic models of Ostwald are alternately applied in rheological examination of wastewater sludges. Information available in the literature differs, even for sludges of the same provenance. Raw primary, secondary and digested sludges are normally treated as plastic material [12]–[15], though HATFIELD [16], BEHN [17], ROSE-INNES and NOSSEL [10], and BHATTACHARYA [9] point to a possible pseudoplastic behaviour. Bingham plastic model is sometimes used, as the yield stress and rigidity coefficient can be directly derived to calculate laminar flow head losses [18].

The most important factors affecting rheological parameters are concentration of solids and the type of suspended particles. Total solid concentration (TS % in weight) has been correlated with power equation [4], [10], [13], while exponential relation between yield stress and TS [9] or viscosity and TS was reported [19]. Not many attempts have been made to correlate rheological properties with solid characteristics. FORSTER [20] reported that viscosity of activated and anaerobically digested sludges is exponentially related to surface charge of suspended solids. A partial influence on viscosity is exerted by the ionic strength of individual sludge liquors and by proteic and polysaccharide surface groups. In general, particles of the size below 40 μm, which is required for measurements, are not uniformly distributed in sewage sludge [3]. The effect of temperature on sludge rheology seems to be less obvious and it has been taken into account in a few investigations only. A dependence of the rigidity coefficient on temperature was in direct proportion with the known influence of temperature on water viscosity [2]; yield stress varies almost linearly with temperature in the case of primary sludges [9]. However, none of these studies have offerred any correlation between the rheological data and sludge properties other than the suspended solid concentration.

Now that a new reliable and fast method of measure is proved [23], bound water seems to be a suitable factor in evaluating the sludge properties [22]. Extracellular polimers (PEC) [20] with intercellular bridging effect can also be taken into account, even if the measuring method is questionable. Recently, tentative correlations between technological characteristics such as capillary suction time (CST) and TS [24], or solid surface acidic groups (GAT) and TS [7] and rigidity coefficient or yield stress for sludges of different origins have been reported.

2. EXPERIMENTAL

Characteristics of the sludges used are summarized in table 1. Sludge is firstly sewed with a screen (∅5.7 mm) to eliminate plastic, wood and coarse particle aggregates; then pH, total solids (TS), volatile to total solids ratio (TVS/TS) and specific oxygen demand (SOUR) are determined according to standard methods by APHA-AWWA [25]. Finally, acidic surface groups are titrated (see below). For each sludge, a set of samples, for several solid concentrations (TS from 1.5 to 14%), is obtained by its thickening and/or low speed centrifugation. For each sample, before rheological measurement, the capillary suction time (CST) was calculated according to standard method by CNR-IRSA [26] and specific resistance to filtration (SRF) was determined using a multiprobe CST apparatus [27]. Rheological measurements were carried out by means of a commercial rotational viscosimeter (HAAKE RV 12) using cells with a profiled sensor system (MVIIP and SVIIP) to prevent sludge slippage on inner rotor surface and outer cylinder; temperature was maintained at 20±0.5°C. The procedure adopted is described in more detail in [8]. The conditions of measurement were the following:

sample was stirred directly inside the rheometer for 4 min at low shear rate (100 s^{-1}),
shear rate was increased from 0 to 100 s^{-1} in 120 s,
stirring was kept at 100 s^{-1} for 60 s,
shear rate was decreased from 100 to 0 s^{-1} in 120 s.

A plot of shear stress versus shear rate was obtained on the basis of 100 pairs of data transmitted to interface computer and the best fitting equation calculated.

2.1. TITRATION OF ACIDIC SURFACE GROUPS

A sample of sludge containing 12 g of dry solid is centrifugated at 1000 rpm for 10 min; the solid fraction is next treated with 0.1 N HCl (100 cm^3)and then centrifugated to separate the liquid. The operation is repeated many times (with total consumption of HCl ranging from 250 to 450 cm^3). The remaining solid fraction is washed with distilled water (200 cm^3) many times to reach the pH of the water utilized and then divided in two parts. Deaeration with nitrogen, to eliminate CO_2 interference, was maintained during titration. The acidic groups are determined dosing 0.1 N NaOH to reach the first (pH 7.2–8.5) or the second titration end point (pH 9.5–10.5). The acidic groups linked to extracellular polymers (PEC) are evaluated in the following manner: the acidic fractions are filtered and treated with ethanol (1:3 volume ratio) for 24 h at 4°C; the PEC precipitates are filtered through a 45 μm membrane, washed with ethanol and dried; a wieghed amount of solid is then dissolved in distilled water and titrated with 0.1 N NaOH.

This method is useful for determining acidic groups such as carboxylic, phenolic or groups with equivalent acidity, as reported for other solid matrices [28]. The interferences of inorganic compounds are eliminated, and the possibility of measuring groups with weak charge (such as alcohols, aldehydes or ketones) is precluded, because of the impossibility to use EtONa as titrating agent [29] in an aqueous solution.

3. RESULTS

3.1. SEWAGE SLUDGE TYPES

Five different sludges were examined in two series. A broad distinction between sludges is shown in table 1; initially (1988) volatile to total solid ratio revealed unusual plant management, particularly for mixed (A1), aerobically stabilized (B1) and primary sludges

(D1); nevertheless, selected sludges were used since they represented real operating conditions.

Experimental work was repeated (1989) to dispose of irrelevant data relative to standard operating conditions and to lower TS content. In the 1988 series, the sludges were supplied from different wastewater plants without paying attention to operating conditions and physico-chemical characteristics. In the 1989 series, two basic sludges (primary D2 and mixed A3) were sampled after a control of composition; from these B2-4 and E2 sludges were obtained. Activated sludge (C2) was supplied from a 20,000 m^3/d wastewater treatment plant. The aerobically stabilized sludge (B) was obtained from mixed sludge (A3) in a 50 dm^3 laboratory batch reactor. The hydraulic retention time amounts to 5 (B2), 9 (B3), 10 (B1) and 20 (B4) days; the anaerobically digested sludge (E2) came from mixed sludge (A3) in a 7 dm^3 laboratory batch reactor operating at mesophilic condition 25 during days (retention time).

3.2. RHEOLOGICAL PARAMETERS

Results are expressed for each TS sludge concentration as mean values taken from five measurements. Many rheological models were used to fit flow curve, but only Bingham plastic and Oswald pseudoplastic equations give in all cases a standard error less than 5% ($R^2 \geq 0.900$).

Table 2

Bingham plastic model rheological parameters

Sludge	TS (%)	τ_0 (Pa)	Λ (Pa s)
A1-3	1.5–14	0.50–185	$1–63 \cdot 10^{-2}$
B1-4	1.3–10	0.07–67	$1–41 \cdot 10^{-2}$
C1-2	1.5–9	1.00–214	$1–110 \cdot 10^{-2}$
D1-2	1.4–16	1.00–55	$2–32 \cdot 10^{-2}$
E1-2	2.3–9	1.00–112	$1–39 \cdot 10^{-2}$

$\tau = \tau_0 + \Lambda D$ – Bingham plastic model.

τ_0 – yield stress.

Λ – rigidity coefficient.

D – shear rate.

The results for the best rheological model vary with TS concentration in the same sludge or within the sludges examined. A general trend is observed: model behaviour changes gradually from pseudoplastic to plastic with increasing TS; in effect, an analysis of variance performed on the paired R^2 values, for each TS content, reveals no significant difference at 95% confidence level. This supports the choice of the plastic model to correlate yield stress (YS) and rigidity coefficient (RC) with physico-chemical parameters.

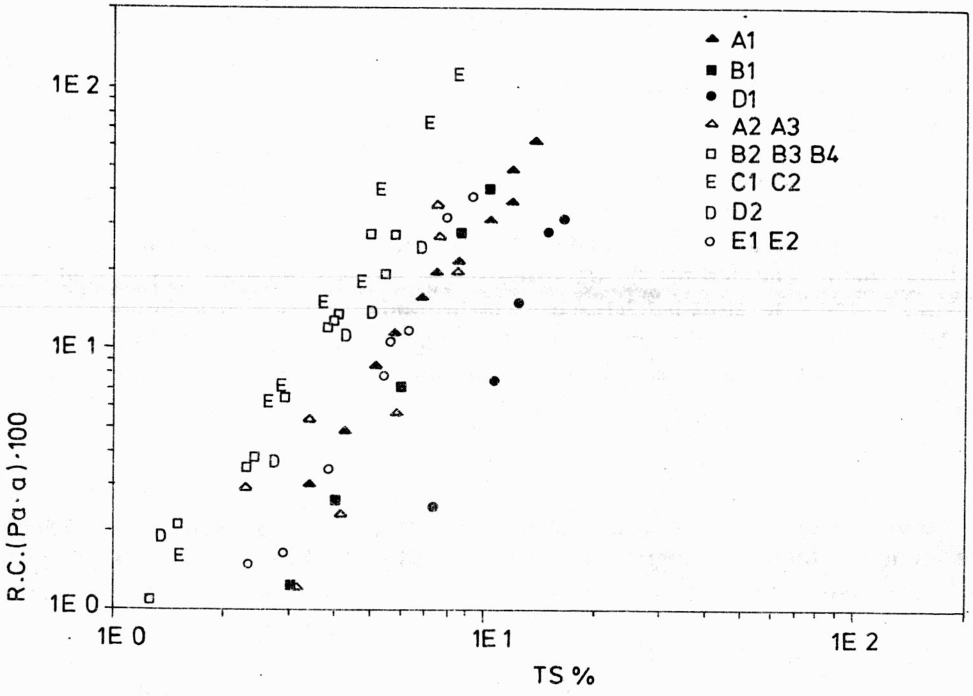

Fig. 1. Rigidity coefficient vs total solids (TS %)

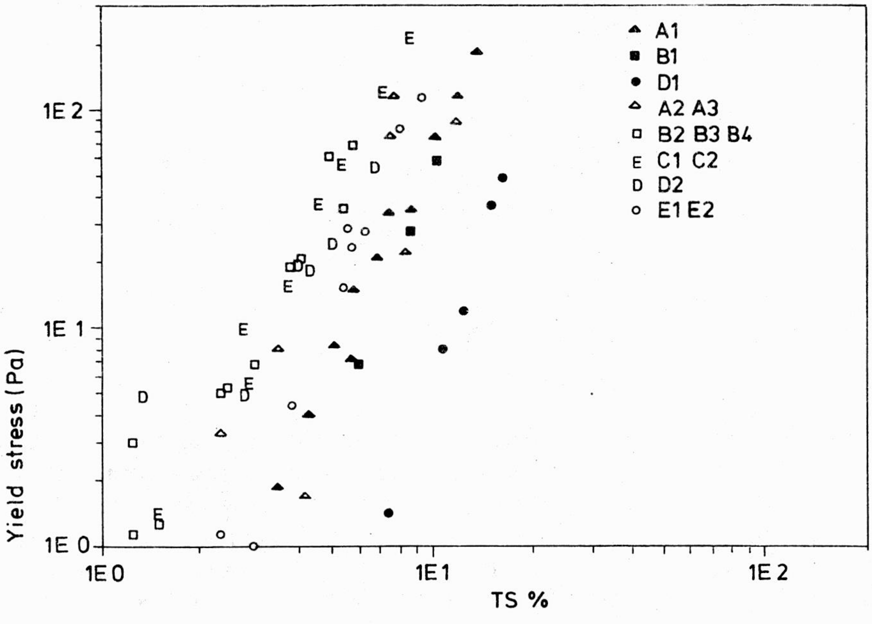

Fig. 2. Yield stress vs total solids (TS %)

The ranges of the values obtained are summarized in table 2, while a more significant representation is shown in figs. 1, 2. The linear plots of rigidity coefficient or yield stress versus TS (on a bilogarithmic scale) show a power type correlation.

The higher viscosity of sludges with a large amount of biological component is also evident. A decreasing order is observed in the sludge series: activated and mixed, aerobically stabilized, primary and anaerobically digested. Furthermore a clear difference exists between sludges with typical and atypical characteristics (TVS/TS) ratio. In effect a correct visualization of RC and YS must be done with respect to TVS, since the fact of viscous behaviour being almost none is attributed to inorganic solids (FS), even if a TS pattern is recurrent in the literature.

Table 3

Correlation between rheological parameters and solid contents (TS) in plastic and pseudoplastic models. Sludge series 1988–1989 (table 1)

Model	Correlation	Experimental coefficients		Values of coefficients given in literature	Ref. no.
$\tau = \tau_0 + \Lambda D$	$\tau_0 = kTS^n$	$k = 2.2 \cdot 10^{-4}$–1.3	$*R^2 = 0.9$	–	[12]**
		$n = 1.5$–5.2		–	
	$\Lambda = PTS^m$	$P = 3.0 \cdot 10^{-5}$–$2.1 \cdot 10^{-2}$	$*R^2 = 0.93$	–	
		$m = 1.2$–3.3		–	
$t = kD^n$	$k = ATS^z$	$A = 3.6 \cdot 10^{-5}$–1.3	$*R^2 = 0.85$	$A = 1.1 \cdot 10^{-2}$	[13]
				$A = 9.0 \cdot 10^{-4}$–$5.4 \cdot 10^{-1}$	[10]
		$z = 1.6$–5.4		$z = 3.4$	[13]
				$z = 2.3$–5.3	[10]

$*R^2$ minimum.

**no absolute values of k and n are published.

Power correlation between rheological parameters and TS contant, for plastic and pseudoplastic model, reveals a limited range for the exponent's values and a wide range (four-five order of magnitude) for the proportionality constants (table 3). This shows the necessity of searching for other parameters to better define a mathematical model with general validity.

Surface group titration (table 4) allows us to measure the groups present on organic particle matter, eliminating the calcium carbonate and the soluble compounds by the adopted methodology. Two principal end points are observed, the first at pH 7.4–8.6 is attributed to carboxylic groups, and the second at pH 9.4–10.5 is attributed to phenolic-like groups.

No substantial hydrolytic effect is observed, as demonstrated by the amount of PEC recovered, maximum 2.2 ‰ in comparison to the 6‰ (in weight of TS) normally present in activated and mixed sludges.

The results obtained reveal acidic group content in the sludges examined decreasing in series: activated, mixed, aerobically stabilized, anaerobically digested, primary. A large variation is observed for sludges sampled at the same treatment plant but at different times.

Table 4

Acidic surface groups titrated on sampled sludges

Period	Sludge	PEC		GAA		GAB		GAT	GATV
		mg/TS	meq/g TS	pH_1	meq/g TS	pH_2	meq/g TS	meq/g TS	meq/gTVS
1988	A-1	22	0.21	7.7	0.21	9.4	0.21	0.42	1.21
	B-1	2	nd	8.5	0.20	nd	nd	0.20	0.38
	C-1	7	nd	8.4	0.23	10.5	0.62	0.85	1.17
	D-1	4	nd	8.5	0.11	nd	nd	0.11	0.26
	A-2	1	nd	8.2	0.22	nd	nd	0.22	0.33
	E-1	nd	nd	nd	nd	10.5	0.32	0.32	0.54
1989	A-3	4	nd	8.8	0.11	9.8	0.04	0.15	0.20
	B-2	nd	nd	8.3	0.18	nd	nd	0.18	0.25
	B-3	nd	nd	7.4	0.08	9.2	0.18	0.26	0.37
	B-4	nd	nd	nd	nd	9.8	0.22	0.22	0.33
	C-2	nd	nd	8.4	0.48	nd	nd	0.48	0.56
	D-2	nd	nd	8.6	0.28	nd	nd	0.28	0.46
	E-2	nd	nd	7.8	0.17	nd	nd	0.17	0.27

PEC – extra cellular polymers.
GAA – acidic surface groups at first titration end point.
GAB – acidic surface groups at second titration end point.
pH_1 – pH at first titration end point.
pH_2 – pH at second titration end point.
nd – not determined.

4. DISCUSSION

The solid–solid and solid–liquid interactions are taken into account in describing the fluidodynamic characteristics of a sewage sludge. Thus, parameters characterizing single properties make way for a theoretical approach, while the studying a global behaviour a semiempiric approach is adopted. Total solids are related to the number of interactions and are always used for estimating the relationship with rheological parameters. However, the attempt to apply TS in the sludge series studied gives poor results (table 5a), with a standard error of 22%, or even bigger in the case of heterogeneous sludges.

Better results are obtained using total volatile solids (TVS) (table 5b) with a consistent reduction in standard error at an acceptable level for rigidity coefficient. The physical meaning of the equation suggests that further parameters must be individualized, since it seems unacceptable that sludges with different organic content can display the same fluidodynamic behaviour. More representative properties like dissolved fatty acids (up to 2000 ppm) and salinity (up to 24, 000 ppm) have been examined [22], but they did not affect the rheological properties determinantly.

Table 5a

Correlation of rheological data with total solids (TS % in weight)

Parameter	$\tau_0 = k\mathrm{TS}^n$		$\Lambda = k'\mathrm{TS}^n$	
	TA	T	TA	T
R^2	0.57	0.82	0.69	0.83
ES (%)	38	24	28	22
d	67	40	67	40
k	0.7	0.59	$0.8 \cdot 10^{-2}$	$0.7 \cdot 10^{-2}$
n	1.9	2.4	1.6	1.9

ES – standard error, d – number of data, TVS – total volatile solids, TF – total fixed solids, TA – all sludges, T – typical sludges.

Table 5b

Correlation of rheological data with total volatile solids (TVS % in weight)

Parameter	$\tau_0 = k\mathrm{TVS}^n$		$\Lambda = k'\mathrm{TVS}^n$	
	TA	T	TA	T
R_2	0.67	0.85	0.79	0.88
ES (%)	33	22	23	20
d	67	40	67	40
k	1.0	1.3	$1.1 \cdot 10^{-2}$	$1.2 \cdot 10^{-2}$
n	2.3	2.5	1.95	2.1

ES – standard error, d – number of data, TVS – total volatile solids, TF – total fixed solids, TA – all sludges, T – typical sludges.

The surface acidic groups (GAT) are considered as valid substitutes of surface charge of sludge particles and can represent intramolecular hydrogen bonds. This is also independent of granulometric distribution, even if measurement methodology ignores the weak oxide groups which have an ability to diffuse in biological organic matter and PEC. An improvement introducing GATV, expressed as meq/g TVS, is obtained; type of the power correlation between rigidity coefficient, TVS and GATV for typical and all sludges is practically the same. Difficulty still persists for yield stress – the results are uncomparable in the two series examined (table 6). This tends to confirm that surface groups contribute in defining the viscosity of sludge to a less extent than volatile solids; for this reason a subdivision of acidic groups in "strong", first end point, and "weak", second point, did not further improve the correlation.

Table 6

Correlation of rheological data with total volatile solids (TV % in weight) and surface acidic groups GATV (meq/g TVS)

Parameter	$\tau_0 = a\,\mathrm{TVS}^b\mathrm{GATV}^c$		$\Lambda = \mathrm{TVS}^b\mathrm{GATV}^c$	
	TA	T	TA	T
R^2	0.70	0.87	0.83	0.90
ES (%)	32	21	21	18
d	67	40	67	40
a	1.3	1.4	$1.5\cdot10^{-2}$	$1.7\cdot10^{-2}$
b	2.3	2.5	1.9	2.0
c	0.29	0.12	0.33	0.32

ES – standard error, d – number of data, TVS – total volatile solids, TF – total fixed solids, TA – all sludges, T – typical sludges.

Table 7

Correlation of rheological data with capillary suction time (CST)

Parameter	$\tau_0 = k\mathrm{TVS}^a\mathrm{TF}^b\mathrm{CST}^f$		$\Lambda = \mathrm{TVS}^a\mathrm{TF}^b\mathrm{CST}^f$ (1)	
	TA	T	TA	T
R^2	0.81	0.88	0.88	0.92
ES (%)	27	20	19	16
d	67	40	67	40
k	$8\cdot10^{-2}$	$2.5\cdot10^{-1}$	$2\cdot10^{-3}$	$3\cdot10^{-3}$
a	1.4	1.2	1.4	1.2
b	–	0.5	–	0.3
f	0.6	0.5	0.4	0.4

ES – standard error, d – number of data, TVS – total volatile solids, TF – total fixed solids, TA – all sludges, T – typical sludges.

Properties connected with activity (SOUR) and particle size (SVI [10]) were used to study rheological behaviour. A general trend, but without any defined mathematical equation, was observed. Recently VESILIND [30] demonstrated how it is possible to define a "filterability constant" (FC) to measure sludge dewaterability from capillary suction time (CST). For the sludges examined, an exponential or power type correlation of CST versus TS was found [30]; at the same time it was impossible to calculate a specific resistance to filtration (SRF) independent of TS content by the multiprobe CST apparatus [27].

For the above reasons the capillary suction time (CST), a quick and easy determinable parameter representing a purely empirical test, has been introduced with the aim to improve the correlation. Splitting TS to TVS and fixed solids (TF) (table 7, eq. (1)) and eventually utilizing the surface acidic group (table 8) satisfactory results were obtained. A mathematical model for interpretation the rigidity coefficient for every type of sludge can now be proposed. The semiempiric equation (2) suggests three fundamental contributions to sludge rheology:

a) solid content, which represents the number of solid–solid and solid–liquid interactions, distinguished as strong (TVS) and weak (TF) viscosity;

b) surface acidic groups (GATV), which differentiates the strength of TVS interactions;

c) CST, which evaluates the degree of sludge dewaterability.

Table 8

General mathematical model for rigidity coefficient in sewage sludges

Parameters	$\Lambda = k\text{TVS}^a\text{TF}^b\text{GATV}^c\text{CST}^f$ (2)	
	TA	T
R^2	0.90	0.93
ES (%)	14	14
d	67	40
k	$3.7\cdot10^{-3}$	$4.6\cdot10^{-3}$
a	1.12	1.10
b	0.22	0.31
c	0.35	0.30
f	0.40	0.35

ES – standard error, d – number of data, TVS – total volatile solids, TF – total fixed solids, TA – all sludges, T – typical sludges.

Furthermore, using eq. (2), a rigidity coefficient can be calculated, which is an alternative method to rheological measurement. The determination of GAT is time consuming and difficult to execute, but using eq. (1) we get satisfactory results (max. error 19%) taking advantage of the parameters that are readily available. The properties examined do not allow us to find a reliable model for yield stress; further work must be done to specify clearly other important factors.

5. CONCLUSIONS

This paper presents the studies on the physico-chemical properties considered in sludge rheological models. The major results can be summarized as follows:

1. Bingham plastic model can be adopted to describe the flow curve of typical and atypical sludges (TVS/TS ratio), with a standard error of data fitting less than 5%.

2. TVS (but not TS) is the best parameter able to correlate yield stress and rigidity coefficient for all the sludges examined; the difference is apparent when sludge is supplied from a treatment plant with operational problems.

3. Sludge properties useful to explain rheological behaviour are GAT and CST. Their correlation with yield stress and rigidity coefficient is demonstrated.

4. The results obtained suggest a method of calculating rigidity coefficient alternative to rheological measurement.

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REOLOGIA OSADÓW ŚCIEKOWYCH. PARAMETRY REOLOGICZNE I CHARAKTERYSTYKA OSADÓW

Zbadano właściwości reologiczne osadów ściekowych pochodzących z różnych oczyszczalni. W badaniach uwzględniono przede wszystkim zawartość ciał stałych w osadzie (TS) wyrażoną w postaci zmodyfikowanych parametrów. Rozpuszczone ciała lotne (TVS), powierzchniowe grupy kwasowe (GAT) oraz czas ssania kapilarnego (CST) powiązano z naprężeniem granicznym i lepkością plastyczną. Semiempiryczny model uwzględniający udział poszczególnych parametrów umożliwia wyznaczanie lepkości plastycznej. Jest to alternatywna metoda pomiarów reologicznych.

РЕОЛОГИЯ СТОЧНЫХ ОСАДКОВ. РЕОЛОГИЧЕСКИЕ ПАРАМЕТРЫ И ХАРАКТЕРИСТИКА ОСАДКОВ

Исследованы реологические свойства сточных осадков, происходящих из разных очистных станций. В исследованиях было учтено прежде всего содержание твердых тел в осадке (TS), выраженное в виде модифицированных параметров. Растворенные летучие тела (TVS), поверхностные кислотные группы (GAT), а также время капилярного отсасывания (CST) связаны с предельным напряжением и пластической вязкостью. Полуэмпирическая модель, учитывающая участие отдельных параметров, дает возможность определения пластической вязкости. Это альтернативный метод реологических измерений.