

THE SLUDGE DEWATERABILITY IN MEMBRANE BIOREACTORS

M. CAPODICI*, G. MANNINA*

** Dipartimento di Ingegneria Civile, Ambientale, Aerospaziale, dei Materiali (DICAM) Scuola Politecnica, Università' di Palermo, Palermo, Italy*

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Abstract. *The influence of the sludge origin on the dewaterability features has been investigated by comparing the experimental results of six membrane bioreactor pilot plants with different configurations. The capillary suction time (CST) and the specific resistance to filtration (SRF), identified as representative of sludge dewaterability features, were measured. The results were related to operational parameters, such as extracellular polymeric substances (EPS) and soluble microbial product (SMP), influent salinity and hydrocarbon, in order to elucidate the influence exerted on the dewaterability. Furthermore, the effect of biofilm and suspended biomass was also investigated. The results showed that during the experimentation carried out with salt and hydrocarbon the sludge dewaterability features significantly worsened (CST above 120 s and SRF above 20 * 1012 m kg-1). Furthermore, the sludge derived from the anoxic reactor resulted as the most affected by EPS and SMP concentration.*

1. Introduction

The sludge dewatering covers a relevant importance since it is strictly related to the operational costs. In details, sludge dewatering is nowadays recognized as one of the most complex and cost effective operation involved in wastewater treatment cycle (Jin et al., 2004; Bertanza et al., 2014).

As the cost for sludge treatment and disposal ranges around 280 - 470 €/t and since 1t of fresh sludge to be disposed is composed on average by 0.25 - 0.30 t of suspended solids (SS) the correct understanding of dewaterability phenomenon represents a key factor in order to improve the effectiveness of water separation process (Ginestet, 2006). A better understanding of sludge dewatering features, could lead to an improvement in pretreatment approaches focused to enhance the dewaterability efficiency (Mowla et al., 2013). In details, four different types of water contained in sludge have been defined: free water, interstitial water, vicinal water and water of hydratation. More in details, gravitational settling can easily separate free water. Mechanical dewatering devices, such as centrifugation or vacuum filtration, can achieve interstitial water separation. Vicinal water, physically bound to solid particles surface, cannot be separated by any mechanical device. Water of hydratation, chemically bound to solid particles surface, can be separated only by heating at temperature above 105°C (Mowla et al., 2013).

Dewatering process is mainly focused to separate the bound water content, represented by the sum of interstitial, vicinal and hydratation water. Several investigations reported that while in high water content condition (over 80%) the water separation could be achieved with relatively low costs, when the water content decreases under 80%, the energy demand cost, drastically increases (Mowla et al., 2013).

Researcher and designer interest moved in developing and refining treatment technologies such as membrane bioreactors (MBR), capable to reduce the specific sludge production; however despite such efforts mechanical dewatering represents up to nowadays a crucial step in reducing the amount of sludge to be disposed (Marinetti et al., 2009).

Although this is a matter of fact, the effects of the excess sludge dewatering involve the most substantial reduction in the aliquot of the sludge. In this paper the combined effect of reducing the production of sludge and dewatering of the latter with different configurations of MBR pilot plants was investigated. The sludge dewaterability is now of great interest since the scientific literature reports different and contrasting data on the best or lesser sludge dewaterability from MBR compared to those from conventional activated sludge (CAS).

Among the several parameters affecting dewatering, many authors agree in considering the sludge origin as one of the most influencing factors (Novak et al., 2003; Jin et al., 2004; Wang et al., 2014).

Bearing in mind what discussed above, the paper reports the results of an experimental study carried out in order to investigate the influence exerted by the wastewater treatment plant (WWTP) layout on the sludge dewaterability features.

Capillary suction time (CST) and specific resistance to filtration (SRF) (Veselind, 1988; Scholz, 2005; Peng et al., 2011) were selected as representative parameters of sludge dewaterability features. Sludge samples were collected from a MBR pilot plant treating urban wastewater added with synthetic wastewater, salt and diesel fuel in order to elucidate the influence of salt, hydrocarbon and organic loading rate on sludge dewaterability. Furthermore, the pilot plant layout has been changed in order to improve the biological nutrient removal, passing from a pre-denitrification scheme to a University of Cape Town Scheme (UCT) (Ekama et al., 1983). Furthermore, during the experimental period carried out according to the UCT layout, suspended carriers were added in the pilot plant reactors in order to allow the attached biomass growth and to investigate its effect on sludge dewaterability.

It is worth noticing that this study represents an extensive database of MBR sludge dewaterability features. Furthermore, the achieved results can represent a useful contribution to other researcher in order to provide comparable data as well as input data for mathematical modelling.

2. Materials and methods

2.1. Investigated WWTP features

The sludge investigated in the present study were collected from 4 different MBR pilot plant, realized at the Laboratory of Sanitary and Environmental Engineering of Palermo University, fed with wastewater taken from the sewer system and operated in 6 different condition as reported in Table1 and Table 2.

In details, the first configuration investigated was an SBR-DN-MBR (referred in the following as configuration A) composed by an anoxic tank (45 L), an aerobic tank (224 L) and an MBR tank (50L). The pilot plant was operated in accordance to the sequencing batch approach. In details, 8 cycles per day were carried out. During each cycle, 40 L (Q_{IN}) of urban wastewater with salt addition was added to the anoxic reactor. The reaction period and the solid–liquid separation phase were set to 1 h and 2 h, respectively. During the solid–liquid separation phase, a permeate flow of 20 L h⁻¹ (Q_{OUT}) was continuously extracted. For further details the reader is addressed to the literature (Mannina et al., 2016b).

Configuration	MBR scheme	MBR	Duration	Biological	Operative Condition features			
		module	[d]	features				
A	SBR-DN-MBR	ZW ₁₀	90	ANOX - AEROBIC	NaCl (0 to 10 g L^{-1})			
B1	DN-MBR	ZW ₁₀	30	ANOX - AEROBIC	NaCl (10 to 20 g L^{-1})			
B2	DN-MBR	ZW ₁₀	50	ANOX - AEROBIC	20 g NaCl L^{-1} and diesel fuel			
C	UCT MBR	PURON	77	UCT	C:N variation			
D ₁	UCT MB-MBR	PURON	66	UCT	-			
D ₂	UCT MB-MBR	PURON	65	UCT	3 gTSS L^{-1}			

Table 1. *Layout and operative condition of the investigated MBR systems*

Where: SBR-MBR:= sequenting bacth reactor, denitridication-nitrification with MBR; DN-MBR:= denitrification- nitrification MBR scheme; UCT-MBR:= University of Cape Town with MBR; UCT MB-MBR: = University of Cape Town with moving bed biofilm reactor – MBR.

Table 2. Average influent features and operational parameters											
Configuration	COD	NH_A-N	$PO - P$	NaCl	HRT	F/M	TSS	SRT			
	$\mathsf{Im} \mathsf{a} \mathsf{L}^1 \mathsf{L}$	[mg L^{-1}]	$[mg L1]$ $[g L1]$		[h]	[kgBOD $_5$ kg $^{-1}$ SSV d $^{-1}$]	$[a L-1]$	[d]			
A	240	30	4	$0 - 10$	20	0.085	5	∞			
B ₁	350	50	6	$10 - 20$	16	0.076	4.61	∞			
$B2^{\ddot{}}$	350	50	6	20	16	0.045	7.85	∞			

Table 2. *Average influent features and operational parameters*

* *mean concentration of the whole pilot plant; ** TPH inlet concentration equal to 20 mg L-1 ; ***mean concentration of the whole pilot plant imposed; **** mean SRT value resulted from TSS concentration imposed (range 16d -155d);*

D2 595 96 9 0 20 0.20 3

 C 455 76 4.8 0 20 0.113 4.03 ∞ D1 223 62 12.6 0 20 0.065 3.45 ∞

Second and third configuration investigated, realized in accordance with the pre-denitrification scheme, were fed in continuous with urban wastewater with salt addition (configuration B1 in the following) and with diesel fuel (configuration B2 in the following). Diesel fuel were added to the influent in order to achieve a Total Petroleum Hydrocarbon (TPH) concentration equal to 20 mg L^{-1} . Biological reactors kept the same volume of the configuration A, but the permeate flow rate 20 L h^{-1} was continuously extracted yielding an HRT equal to 16 h. For further details the reader is addressed to the literature (Mannina et al., 2016a).

During configuration A, B1 and B2, solid–liquid separation was performed via an ultrafiltration (UF) hollowfiber membrane module (Zenon Zeeweed, ZW 10, with specific area equal to 0.98 m2 and a nominal porosity of 0.04 lm). Conversely, during configurations C, D1 and D2 the solid–liquid separation phase was carried out by means of an ultrafiltration hollow fiber membrane (Koch PURON® 3 bundle). Both membrane modules were periodically backwashed (every 9 min for a period of 1 min) by pumping, from the Clean In Place (CIP) tank a volume of permeate back through the membrane module. Furthermore, in each configuration the mixed liquor from the MBR tank was recycled to an oxygen depletion reactor (ODR), that allowed the oxygen stripping in the

 3^{***} 71 d^{*}

mixed liquor, and thus to the anoxic reactor.

During the C configuration, the pilot plant was operated in accordance with UCT layout. In details, the pilot plant consisted of an anaerobic (volume 62 L), an anoxic (volume 102 L) and an aerobic (volume 211 L). The membrane module was located inside an aerated tank (MBR tank) (36 L). The pilot plant was fed with municipal wastewater mixed with a synthetic wastewater characterized by Sodium Acetate (CH₃COONa), glycerol $(C_3H_8O_3)$, dipotassium hydrogen phosphate (K_2HPO_4) and ammonium chloride (NH₄Cl). The synthetic wastewater was added in order to control the C/N ratio fed to the pilot plant. In details the pilot plant was initially fed with a C/N ratio equal to 10; while further the C/N ratio fed to the pilot plant was set equal to 5. For further details the reader can refer to previous literature (Mannina et al., 2016c).

In order to allow the growth of biofilm within the pilot plant, during D1 and D2 configuration, the aerobic and anoxic reactors were filled with suspended carriers (Amitech s.r.l.) with a 40% and 15% filling ratios respectively, corresponding to a net surface area of almost 205 m² m⁻³ and 75 m² m⁻³ respectively. Furthermore, during the D1 configuration the pilot plant was operated with total sludge retention, corresponding to an infinite SRT. Conversely, during the D2 configuration the sludge withdrawal was constantly operated by a peristaltic pump in order to keep the mean TSS concentration within the pilot plant equal to 3 g TSS L⁻¹.

3. Experimental procedures

3.1. Sludge dewaterability measurements

CST and SRF were measured to investigate the sludge dewaterability features (Veselind, 1988; Scholz, 2005; Peng et al., 2011). The CST and SFR were measured in accordance with international Standards EN 14701-1 and EN 14701-2 by analyzing fresh samples collected from the biological reactors. In detail, the CST measurements were determined by pouring a volume of sample into a sludge reservoir placed on Whatman no. 17 filter paper. An electronic device recorded the time necessary for the filtrate to cover the space between two probes, which detected the advancement of the liquid front on the paper. The CST was assessed as the average value of three replicates. Furthermore, the achieved CST were divided for the TSS concentration of the sludge sample yielding the specific CST (sCST) measured as s L g⁻¹. The SRF was evaluated in the reduced pressure condition (-50 kPa). In detail, the vacuum condition was applied by a vacuum pump connected to a Buchner funnel in which Whatman 41 (20 μm pore size) filter paper was placed. After pouring 100 of sample on the funnel, the filtrate volumes (V) and the corresponding time (t) were recorded. The SRF was calculated in accordance with Equation 1:

$$
r = \frac{2 \cdot \Delta p \cdot A^2 \cdot b}{\mu \cdot C_0} \tag{1}
$$

where Δp is the pressure drop across the filter, A is the filtration area, μ is the viscosity of filtrate at the temperature of the sludge, b is the slope of the linear portion of the curve obtained by plotting t/V versus V, and C_0 is the initial dry residue of the sludge.

The viscosity of filtrate was measured using a Brookfield rotational viscometer. In details, 16 ml of mixed

liquor, derived from the aerobic tank, were put into a metallic cylindrical shaped vessel where the sludge temperature was controlled (20 $^{\circ}$ C ± 0.1 $^{\circ}$ C) by means of a thermostat. The rotor velocity was set equal to 60 rpm and the corresponding viscosity value was expressed in cP. For further details the reader is addressed to literature (Mannina et al 2016a; 2016b; 2016c).

3.2. Chemical parameters and EPS measurements

During each pilot plant operation, the influent wastewater, mixed liquor inside the biological reactors and effluent permeate were sampled and analyzed for total and volatile suspended solids (TSS and VSS), total chemical oxygen demand (COD_{TOT}), supernatant COD (COD_{SUP}) ammonium nitrogen (NH₄-N), nitrite nitrogen $(NO₂-N)$, nitrate nitrogen $(NO₃-N)$, total nitrogen (TN) , phosphate $(PO₄-P)$, total carbon (TC) and inert carbon (IC). All analyses were performed according to the standard methods (APHA, 2005).

Total extracellular polymeric substances (EPS_T) were measured using the thermal extraction method (Cosenza et al., 2013). According to this method, the EPS_T are partitioned into two fractions: soluble microbial products (SMPs) and bound EPS (EPSBound). Both SMPs and EPSbound were fractionated into protein and carbohydrate compounds. The EPST was evaluated as the sum of proteins and carbohydrates compounds of SMPs and EPSBound:

$$
EPS_{T} = \underbrace{EPS_{P} + EPS_{C}}_{EPSBound} + \underbrace{SMP_{P} + SMP_{C}}_{SMP}
$$
 [2]

where the subscripts P and C indicate the content of proteins and carbohydrates in the EPSBound and SMP, respectively.

Carbohydrates in the EPS_T were determined according to the phenol–sulfuric acid method with glucose as the standard (DuBois et al. 1956). Proteins were determined by the Folin method as proposed by Lowry et al., (1951). For further details, reader is referred to the literature (Cosenza et al., 2013; Capodici et al., 2015).

4. Results and discussion

4.1. Investigated WWTP biological performances

For sake of completeness, in table 3 an outlook of the average removal efficiencies of the investigated configurations is provided; while in Table 4 the mean values of specific EPS and SMP are reported.

** total COD removal measured on the permeate flow;* ** *mean removal efficiency achieved as average of 58.1% (during C/N=10) and 31.5 (C/N=5); *** vale achieved during C/N=10;*

Plant	Aenaerobic			Anoxic			Aerobic			MBR						
Conf.	EPSp	EPSc	SMPp	SMPc	EPSp	EPSc	SMPp	SMPc	EPSp	EPSc	SMPp	SMPc	EPSp	EPSc	SMPp	SMPc
Α	$\,$				101.55	39.28	1.39	5.11	92.88	38.41	0.12	6.66				
B1	\blacksquare			\blacksquare	117.12	77.43	0.00	0.15	132.17	74.10	0.00	1.50				
B2	\blacksquare			-	82.99	40.46	4.41	5.19	75.62	38.57	3.47	4.90				
С	150.80	18.69	0.93	0.88	222.88	45.62	0.20	2.38	158.26	29.90	1.03	2.46				
D1	186.10	17.77	14.15	0.00	146.27	21.12	11.44	3.85	149.31	21.45	11.44	3.85	164.42	24.84	8.61	3.16
D ₂	152.73	20.95	59.73	3.99	142.96	30.47	13.68	0.23	154.07	37.36	10.26	3.64	126.24	31.83	2.51	3.07

Table 4. *Average values of specific EPS and SMP (mg EPS, SMP gTSS-1)*

Results reported in table 3 and 4 show that the biological performances of the investigated pilot plant were good in terms of carbon removal. Regarding the nitrogen removal, the salt concentration increasing, as well as the hydrocarbon presence, affected the nitrogen removal during experimentation carried out with B1and B2 configuration. Regarding the UCT configuration (C, D1 and D2), the maximum nitrogen removal efficiency was achieved during B1 period, while during the C configuration carried out with C/N=10, the P removal was on average equal to 70%. Regarding the EPS and SMP contents, data reported in table 4 show a low SMP content during the experimentation. Such result is likely due to the low F/M applied to the pilot plant (Table 2). For further details the reader is addressed to literature (Mannina et al 2016a; 2016b; 2016c).

4.2. Sludge dewatering results

In details, capillary suction time (CST) and specific resistance to filtration (SRF) were measured in sludge derived from MBR systems characterized by layout and operative condition resumed in table 1 and 2. To provide a complete outlook of the achieved results, in Figure 1 and 2 the results achieved for specific capillary suction time and for the specific resistance to filtration are reported for each biological reactor.

With reference to A, B1 and B2 configuration only data derived from aerobic reactor are available. However, data reported in figure 1c and 2c clearly highlight that the co-presence of salt (up to 20 mg NaCl L⁻¹) and hydrocarbon (inlet TPH concentration equal to 20 mg L^{-1}) represented the most influencing parameter that affected the dewaterability of sludge derived from the aerobic reactor. Indeed the CST measured increased by passing from the A configuration (24 s) to the B2 configuration (up to 142 s). In accordance to the CST result, the SRF measured during B2 configuration increased up to 2.19 $*$ 10¹⁴ m kg⁻¹.

With reference to C, D1 and D2 configurations, data from anaerobic, anoxic and MBR reactors were collected.

During experimentation carried out with C configuration, the s CST as well as the SRF increased, on average, in the final part of the experimentation. Such result is likely due to the stress effect exerted on the biomass by imposing the C/N=5. As a matter of fact the carbon scarcity resulted in a worsening of sludge dewaterability features likely due to an incorrect trade off of EPS and SMP; indeed a slight increase in SMP measured during C/N=5 period was noticed.

By comparing results collected during period D1 and D2, it is possible to highlight an increase of the sCST in the D2 period. Such result is likely due to the imposed TSS concentration that resulted in an increase of F/M

Figure 1. *Specific CST results for each experimental lay out in anaerobic (a), anoxic (b), aerobic (c) and MBR (d) reactor.*

ratio and thus in an increase of SMP. However, as the sludge withdrawal affected the TSS concentration as well as the specific EPS and SMP measure, a clear influence of EPS on the dewaterability was not noticed in D2 configuration.

In order to elucidate the influence of biomass features on sludge dewaterability features, the achieved results were compared with sludge viscosity and with EPS and SMP results.

With reference to the A, B1 and B2 configurations, it is possible to highlight how the salt concentration increase exerted a significant effect on CST and thus on sludge dewaterability. Indeed, the CST value measured at 12 and 14 g NaCl L⁻¹ was nearly constant (namely, 24 seconds). Conversely, when the salinity was increased up to 17 g NaCl L⁻¹, the measured CST increased up to 29.33 seconds.

Furthermore, at a salinity of 20 g NaCl L^{-1} , the dewaterability of the aerobic sludge significantly worsened. Indeed, the CST values were almost twice the values measured at 12 and 14 g NaCl L⁻¹. This result is in line with literature and shows how the stress condition exerted by the step-wise salinity increase affected the sludge dewatering (Raynaud et al., 2012). Indeed, the addition of NaCl in activated sludge leads to a longer time for dewatering and also that the addition of NaCl leads to a release of fine particles that are assumed to clog the filter medium, thus limiting the flow throughout the porous system. During B2 configuration, the hydrocarbon dosing led to a considerable increase of the CST (up to 142 s). Such a value is nearly 6 times higher than values

Figure 2. *SRF results for each experimental lay out in anaerobic (a), anoxic (b), aerobic (c) and MBR (d) reactor*

related to a salt rate of 12 to 14 g NaCl L⁻¹. The SRF measurements provided similar results, thus corroborating the CST findings with respect to the progressive decrease of sludge filterability during B2 configuration. Such worsening in sludge filterability was also noticed by filtering the sample collected for the chemical analysis.

Indeed, a longer duration of the vacuum pump operations needed to collect significant volumes of filtered sample was required during B2 configuration. Furthermore, after filtration, the filtration media was covered by a homogeneous jelly layer.

This result was likely ascribable to the progressive increase in sludge viscosity, primarily due to worsening of the bacterial consortium features and reported in Figure 3.

Data reported in figure 3a, confirm the increase of sludge viscosity due to the step-wise salinity increase. Li and Yang (2007) found that the difficulty in sludge dewatering increases with the increase in sludge viscosity and suggested that the increase of the sludge viscosity is primarily related to an increase of loosely bound EPS. Despite the increase of viscosity is recognized to be due to the increase of EPS (Mowla et al., 2013), data reported in figure 3b show an increase of sludge viscosity corresponding to a decrease of EPS. Such result is likely due to the increase of TSS during the experimentation (pilot plant was operated with total sludge retention during A, B1 and B2 configurations. The increase of TSS resulted in an increase of viscosity and in a decrease of specific EPS.

The effect of the bound fraction of EPS on dewatering was also noted in the current study. Figure 3c shows

Figure 3. *Sludge viscosity increase during A, B1 and B2 configuration (a); correspondence between sludge EPS and viscosity (b); and correlation between CST and specific bound proteins EPS (c)*

the influence of specific bound protein fraction of EPS on CST. The highest CST values corresponded with the lowest concentration of specific bound protein. It is worth noting that the role played by EPS in sludge dewaterability still remains controversial (Houghton and Stephenson 2002). Indeed, (Zhou et al., 2015) found that the sludge dewaterability initially increases with EPS content but subsequently decreases once the EPS content exceeds a threshold value. Conversely, other authors reported that the sludge dewaterability improves after the EPS reduction (Chen et al., 2001). Furthermore, according to our findings, the presence of hydrocarbons characterized by high hydrophobicity could also have exerted a significant effect on sludge viscosity and thus on the dewatering phenomena, making comprehension of the results more difficult.

The effect exerted by EPS and SMP on sludge dewaterability was clearly highlighted also during configurations C, D1 and D2. In figure 4 the correlation between protein EPS fraction and SRF during C configuration for the anaerobic reactor is reported.

Results reported in figure 4 are consistent with Chen and co-worker (2001); indeed resistance to filtration was found to decrease in correspondence of an increase of bound protein fraction. However, during C configuration it

was not possible to highlight a clear correspondence between EPS and sludge dewaterability. Such a result was likely affected by the quite total absence of SMP in both experimental phases. Sludge dewaterability during

C configuration resulted mainly influenced by the TSS concentration. Indeed, R^2 values achieved by interpolating the SRF values with the TSS concentration were equal to 0.47, 0.51, 0.57 and 0.68 for anaerobic, anoxic, MBR and aerobic reactor, respectively.

Conversely, during D1 configuration experimentation, the highest correspondence between EPS and sludge dewaterability features were found. The main results are reported in figure 5.

Figure 4. *Correlation between SRF and protein EPS fraction in anaerobic reactor during C configuration.*

Figure 5. *Correlation during D1 configuration: CST with specific bound carbohydrates EPS in anaerobic reactor (a); specific bound proteins and carbohydrates EPS with SRF in anoxic reactor(b); CST and SRF with specific bound carbohydrates EPS in aerobic reactor (c); CST and SRF with specific bound carbohydrates EPS in MBR reactor (d).*

Reported results highlight that the carbohydrate fraction of EPS significantly affected the dewaterability during B1 configuration. In details, sludge derived from aerobic and MBR reactors (Figure 5 c and d) resulted affected from carbohydrate in terms of CST as well as SRF. Conversely, in the anoxic reactor the influence exerted by the protein fraction resulted evident (R^2 =0.81). The anaerobic reactor resulted less affected by the EPS content likely due to the lower TSS concentration. Furthermore, the achieved result was in agreement with literature; indeed the increase in bound EPS resulted corresponding to a slight worsening of sludge dewaterability. It has to be stressed that during D1, as well as C, configuration the sludge was on average easily dewaterable, moreover if compared with sludge investigated during A, B1 and B2 configuration.

With reference to D2 configuration, it was not possible to highlight a dependence of dewaterability with EPS or sludge viscosity. Such result, as early discussed, is due to the sludge withdrawal carried out in order to control the TSS concentration. Such condition significantly affected EPS and SMP measure (specific SMP in the anaerobic reactor were found on average 3 time higher than D1 configuration) and thus the possibility to relate EPS concentration to dewaterability features.

5. Conclusions

The present paper reports the results of an experimentation carried out with the focus to investigate the influence of the sludge origin on dewaterability. Six different experimental campaigns, differing for plant configurations and influent characteristic, were carried out. The CST and SRF measures were identified as key parameters in order to describe the dewaterability features.

The first significant result is represented by the extensive dataset achieved in terms of dewaterability parameters related to MBR systems.

The hardest sludge to be dewatered was found during A, B1 and B2 configuration. However, such result is mainly related to the influent features; indeed the co-presence of salt and hydrocarbon led to sludge conditions close to the industrial wastewater. Nevertheless, these findings allow affirming that further studies are needed in order to investigate the dewaterability of sludge originated from industrial wastewater treatment.

During experimental configuration C, D1 and D2, influent characteristics were consistent with domestic sewage water. Results achieved during the UCT periods, confirmed the key role exerted by EPS concentration in sludge dewaterability. Moreover, during D1 configuration, a dependence of the measured CST with EPS content was clearly highlighted in each biological reactor.

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