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Biological stability and dewaterability of CAS and MBR sludge

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ABSTRACT

This paper investigates and compares the characteristics of sludges produced by membrane bio-reactor (MBR) and conventional activated sludge (CAS) systems. Stability and dewaterability of full-scale MBR and CAS treatment plants are measured and compared. Obtained results show that specific methane production is higher in CAS sludge compared to MBR sludge, although one of the CAS sludges investigated had a sludge retention time (SRT) higher than the two MBR sludges investigated. Nonetheless, MBR sludge results to be characterized by a non-negligible biometanation potential (BMP). Methane production measured during BMP tests is around 200 NmL/gVS for MBR sludge, equaling 2/3 of methane production obtained, in similar condition, for CAS sludge. Dewaterability of the sludge resulted to be linearly correlated with the SRT and the sludge extracellular polymeric substances (EPS) concentration. The higher the SRT and the lower the sludge EPS concentration, the lower the specific resistance to filtration.

Keywords: Dewaterability; Stability; MBR; EPS; Anaerobic digestion; Wastewater sludge

1. Introduction

Among the several advantages of membrane bio-reactor (MBR) compared to traditional conventional activated sludge (CAS) systems is often reported the higher stability of the MBR sludge due to higher sludge retention time (SRT). This advantage is evident in the case of CAS systems that are upgraded to MBR, since the upgrading allows to increase the micro-organisms concentration in the biological reactor, resulting in a higher total biomass and therefore a higher SRT.

This advantage, however, cannot be generalized in the case of new construction MBR facilities. For the latter, the higher concentration of micro-organisms in

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biological tanks corresponds to a decrease in the volume of the tanks compared to the case of CAS systems, which is one of the main advantages of the MBR technology. This decrease in volume, more or less important depending on the sensitivity of the designer, compensates for the increase in the concentration of micro-organisms, resulting in a total biomass in the system, which is not very different from that which would occur in a CAS system aimed at treating the same wastewater influent. Similarly, if the influent wastewater and the mass of micro-organisms in the biological reactors are the same, the amount of sludge produced by CAS and MBR and thus, the SRT will be very similar and the SRT will depend only on the amount of extracted excess sludge. Therefore, the two systems could be operated in such a way to have a similar SRT.

Therefore, the sludge produced by MBR cannot be generally considered as already stabilized and thus as a sludge that does not require further digestion treatment. Even some authors believe it is advisable to conduct the process with a very low MBR SRT compared to what is theoretically possible, in order to maximize the concentration of organic substance in the sludge and thereby increase the energy recovery due to the anaerobic digestion of the sludge itself [1].

With regard to the effect of anaerobic digestion on the MBR sludge dewatering properties, it was not possible to find any data in the scientific literature. This represents a quite surprising lack, since the dewatering of sludge and consequently its disposal is strongly influencing the management costs of the treatment plants [2]. The purpose of this study was, therefore, to compare the effect of the anaerobic digestion process on the MBR and CAS sludge respectively, both in terms of biomethane potential (BMP) and dewatering properties. The dewatering process is found to be influenced by several factors, i.e. properties and composition of the influent to the treatment plant [3,4], particle size distribution [5], amount of suspended solids in the mixed liquor [6], salinity [7], presence of colloidal nanoparticles [8], abundance, and structure of polyanionic substances mostly in the saccharide domain of extracellular polymeric substances (EPS) [3]. More in detail, the EPS of the studied sludge was extracted and characterized in order to relate its composition and concentration to the rheology and dewaterability of the tested sludge.

2. Materials and methods

Tested sludges were collected from two different MBR treatment plants located in Marina del Cantone (Naples—Italy) (MBR sludge 1) and Capri (Naples—Italy) (MBR sludge 2) respectively and from two CAS treatment plant located in Nola (Naples—Italy) (CAS sludge1) and Massa Lubrense (Naples—Italy) (CAS Sludge2).

The operating parameters of the plants where the sludges were collected are summarized in Table 1.

The collected sludges were concentrated by settling for two hours. After this time, the supernatant was discharged and the thickened sludges were therefore characterized by gravimetry in terms of TS–VS according to EPA standard methods (1684). Once thickened aliquots of each sludge were subjected to the EPS extraction as described by Frølund, Palmgren, Keiding, and Nielsen [9]. Dowex marathon C (Sigma-Aldrich) was chosen as the cation exchange resin. Once extracted, the EPS composition was defined in terms of Carbohydrate (CH) [10], Uronic acids (UA) [11,12], Proteins (PR) [13], and humic substances (HA) [9].

2.1. Anaerobic digestion

Biomethanation batch tests (BMTs) [14–21] were conducted, after thickening, on 400 mL of each tested sludge. BMTs were performed in triplicate on a small scale under controlled and reproducible conditions in a 1,000 mL glass bottle GL 45 (Schott Duran, Germany). Each bottle was sealed with a 5 mm silicone disk that was held tightly to the bottle head by a plastic screw cap punched in the middle (Schott Duran, Germany). All digesters were immersed up to half of their height in hot water bath at a constant temperature of 308 K. Methane production was measured periodically by water displacement method after leaving the biogas bubbling in an inverted 1,000 mL glass bottle containing a strongly basic solution (12% NaOH) in order to trap any CO_2 present in the biogas. Once the daily biogas production was lower than 1% of the total BMP, the methane measurement was stopped and the digestate was left in the reactors at room temperature for further 30 d in order to assess the effect of this further period of adjustment on the dewaterability properties. Then the output digestates in each reactor were characterized in terms of VS–TS and again the EPS was extracted for its composition evaluation.

2.2. Dewaterability tests

Dewaterability is evaluated by specific resistance to filtration (SRF) and capillary suction time (CST). SRF is a technological parameter that gives count of the aptitude of sludges to be dehydrated via filtration.

Table 1
Operational parameters of the treatment plants

	HRT (h)	Flow rate (m^3/h)	SRT (d)	COD (mg/L)	N-NH_4^+ (mg/L)	MLSS (g/L)	Membrane
MBR1	20	12	30	450	40	6.33	Hollow fiber
MBR2	24	65	35	350	35	9.05	Plain
CAS1	7	3,300	40	310	22	9.75	–
CAS2	18	100	15	350	35	2.62	–

It is widely used to previse performances of full-scale filters and to compare the behavior of sludges from different plants against filtration processes. It represents the resistance to filtration of a theoretical sludge panel having unitary weight in dry solids per unit of the filtering surface. The SRF was determined by pouring a 200 mL sample into a Buchner funnel lined with N° 541 Whatman filter paper. A negative differential pressure was applied of 49 kN/m^2, and kept constant by means of a pressure regulating system. The first volume of filtrate collected is discarded until the system reached the full vacuum and the volume of filtrate is therefore recorded at regular time intervals by means of a graduated cylinder (precision 0, 25 mL).

The SRF is then calculated according to the following relationship:

$$R = \frac{2PA^2}{\mu \cdot C} b \qquad (1)$$

where R = Specific resistance M/kg), P = Applied differential pressure (N/m^2), A = Area of the filter in the funnel (m^2), μ = Viscosity of the filtrate (kg/m s).

$$C = \frac{C_0\ C_c}{C_c - C_0} \qquad (2)$$

where C_0, C_c are the solid concentrations (kg/m^3) in the sludge itself and in the sludge panel formed in the filter after the filtration.

The parameter b (s/m^6) is experimentally determined by the method heretofore described. It represents the slope of the linear interval of the curve obtained by plotting the values of the filtered volume (V) at time (t) in the graph recording the volume V on the abscissa and the ratio t/V on the ordinate.

The CST test determines the water retention of a certain sludge. A sludge sample is placed in a metal cylindrical funnel on standard chromatographic paper. Water is moving through the paper sheet by capillary suction. The time necessary to reach a specified distance is defined as CST. CST was determined by means of a Triton (UK) standard CST apparatus using a 18 mm diameter funnel on a standard CST paper according to APHA standard method 2710G [22]. Being CST, strongly dependent from the TSS content of the filtering sludge, the absolute values in seconds were normalized according to the referred standard method by dividing into the TSS concentration of the sludge. The CST values are finally expressed as s L/g.

3. Results and discussion

3.1. Anaerobic digestion

BMTs results are summarized in Fig. 1 showing the specific cumulative methane production of the four studied sludges. The methane production per VS mass unit for CAS sludge was higher compared to the MBR sludge (Table 2), in the following order CAS2 > CAS1 > MBR1 > MBR2.

This result was partially unexpected as based on the SRT of the four sludges (Table 1) and previous studies [23,24], the expected order was CAS2 > MBR1 > MBR2 > CAS1. This seems to indicate that the SRT is not the sole parameter influencing the sludge BMP.

However, a quite high BMP of MBR sludges (244 and 186 NmL/gVS for MBR1 and two respectively) was obtained, that is in both cases less than 1/3 lower than the BMP of CAS sludges (304–342 NmL/gVS for CAS1 and CAS2 respectively).

3.2. Dewaterability of sludge

Previous literature data about the effect of anaerobic digestion on sludge dewatering appear to be confusing and some way contradictory. Approximately, the same number of studies show that digestion improves dewaterability or makes it worse [25]. The co-presence of many heterogeneous variables is difficult to take into account and goes beyond the aim of the present work. It is anyway useful to highlight how the different biomass selections due to membrane

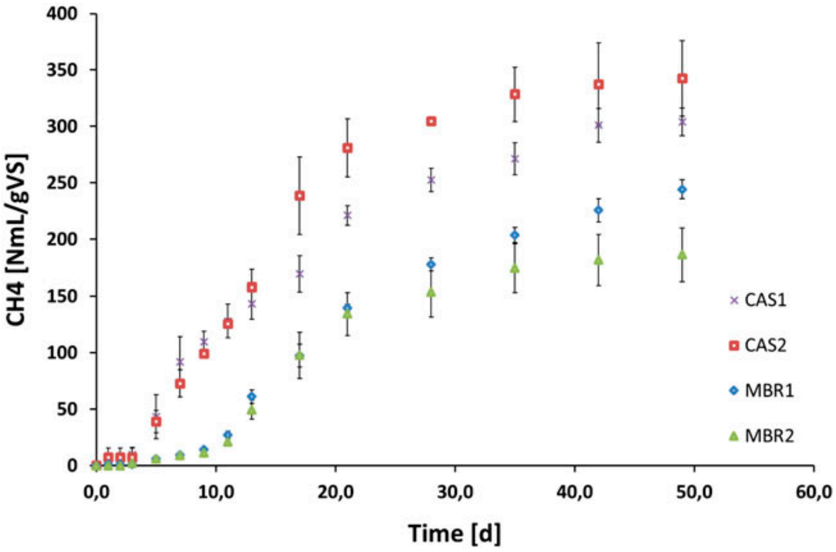


Fig. 1. Cumulative specific methane productions of MBR sludge and CAS.

Table 2
Properties sludge and digestate

	Sludge			Digestate			Δ (%)		BMP (NmL/gSV)
	TS (g/l)	VS (g/l)	VS/TS	TS (g/l)	VS (g/l)	VS/TS	ST	SV	
MBR1	13.2	9.45	0.72	11.2	5.1274	0.60	−18	−45	244
MBR2	12.0	9.71	0.80	9.14	4.80	0.52	−24	−50	186
CAS1	51.2	20.7	0.40	33.0	9.54	0.29	−35	−53	304
CAS2	13.7	11.7	0.85	10.2	6.32	0.62	−30	−46	342

technology application make the sludge completely different from CAS in many of the factors affecting the dewatering process.

Dewaterability parameters are reported before and after anaerobic digestion in Table 3.

According to literature, the MBR sludge is generally presenting a higher resistance to filtration than CAS because of the different properties in terms of EPS concentration and composition, suspended solids and amount of dispersed micro-organisms [18]. This behavior, already reported in a previous study [23] is not confirmed here. The tested CAS2 sludge, not present in the previous study, is in fact presenting the highest SRF value, while the overall SRF values for the other three sludges are in general lower compared to the previous experiments [23]. This reflects how dewaterability is a parameter that presents even in the same plant wide oscillations at different sampling time.

However, a very good correlation of the SRF with the SRT was found in this study (Fig. 2), indicating that a higher sludge retention in the oxidation bioreactors results in better dewaterability properties.

Data in Table 3 show that the sludges have good dewatering behavior, two orders of magnitude lower than the technical limit retained useful for real scale filtration of sludge (i.e. $5 \times 10 \times 10^{12}$ m/kg) [26]. Even the effect of anaerobic digestion is found to be very small, since the filtration properties of the digested sludge are very near to the respective non-digested one. This is not in agreement with the results of our previous study and is probably due to the adjustment time of 30 d that in the present experiments the sludge was subjected to after anaerobic digestion. These extra 30 d in the reactors resulted in a sludge much less difficult to dewater with respect to what happened in the previous study, where the SRF became up to 20-fold higher after anaerobic digestion.

Table 4 reports the characterization of the EPS from the sludge, extracted prior and after the anaerobic digestion phase.

The values are expressed as mg of EPS per g of total solids and the EPS composition in terms of CHs, UA, PR, and HS are reported in relative percentages. While a noticeable variance is observed among the

Table 3
Dewatering properties of sludge and digestate

	Sludge		Digestate		Digestate/sludge	
	SRF (m/kg)	CST (s L/g)	SRF (m/kg)	CST (s L/g)	SRF	CST
MBR1	4.67×10^{10}	0.91	5.55×10^{10}	1.05	1.19	1.15
MBR2	2.96×10^{10}	0.96	2.55×10^{10}	1.29	0.86	1.34
CAS1	1.25×10^{10}	0.27	1.31×10^{10}	0.63	1.04	2.27
CAS2	8.40×10^{10}	1.23	8.11×10^{10}	1.29	0.97	1.04

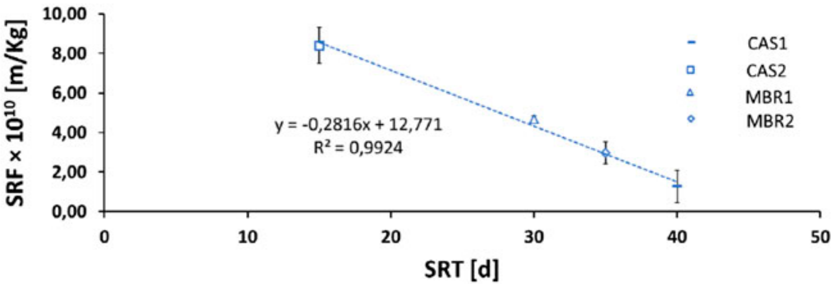


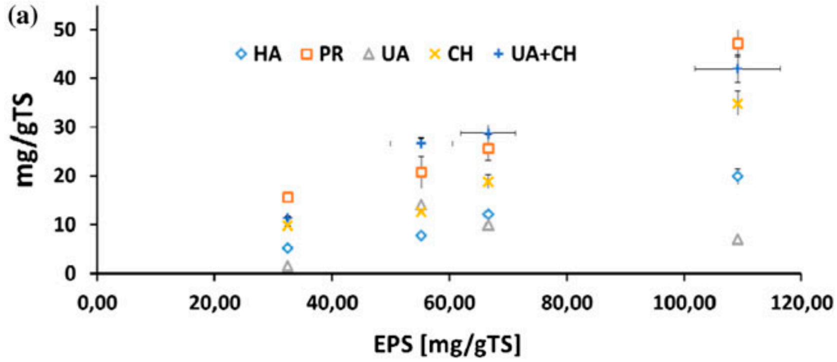
Fig. 2. Effect of the SRT on the SRF.

various sludges in terms of total EPS concentration, the relative composition of EPS results to be quite regular concerning the amounts of PR and HA for sludge and digestate, respectively. The relative abundances of UA are instead much more variable in the sludge EPS and are decidedly higher in the MBR sludge than in CAS. Conversely, the CH content in MBR is lower. This is not surprising, since UA are acid carbohydrates that compose the polysaccharidic part of the EPS. Hence, polysaccharides from the tested MBR sludge present a higher content in UA. Comparing Fig. 3(a) and (b), it is possible to observe the absolute concentrations trends of each of the components of sludge EPS before and after the anaerobic digestion. Values are reported at increasing total EPS concentrations in the sludge.

It is possible to notice how in the raw sludge, from the sludge with the lowest EPS concentration (CAS1—32.50 mg/gTS) to the highest one (CAS2—109.11 mg/gTS), the concentrations of CH, PR, and HA are increasing in linear-like trend. The only exception with regard to the UA is that they are lower in MBR1 with respect to MBR2 either in absolute value (9, 9 and 7.1 mg/gTS respectively) or in percentage of the total EPS (Table 4). Conversely, after AD it is not possible to observe, for any of the EPS components, any trend at increasing the EPS concentration. This means that the EPS structure, amount, and the relative components abundance, are evolving during the anaerobic digestion. Generally, the overall EPS concentration is lower after AD, while its reduction in weight is not comparable with the volatile solids removals reported in Table 2. This means that EPS remains in general refractory to the anaerobic degradation and even the digested sludge after the extra-30 d adjustment time preserves up to

Table 4
EPS composition

	Sludge				Digestate			
	CAS1	CAS2	MBR1	MBR2	CAS1	CAS2	MBR1	MBR2
EPS (mg/g TS)	32.5	109.1	66.6	55.2	24.4	58.8	54.7	44.5
%CH	30.3	32.0	28.3	22.9	34.9	38.2	37.0	25.7
%UA	4.9	6.5	14.8	25.5	5.4	5.2	8.6	9.1
%PR	48.6	43.3	38.7	37.6	49.2	46.6	49.1	58.2
%HA	16.2	18.2	18.2	18.1	10.5	10.0	5.3	7.0



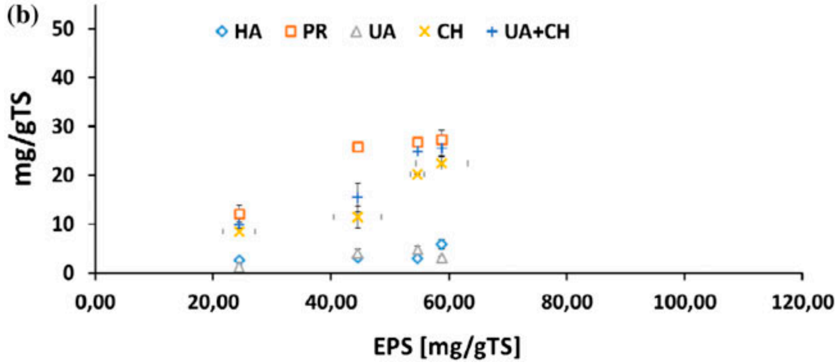
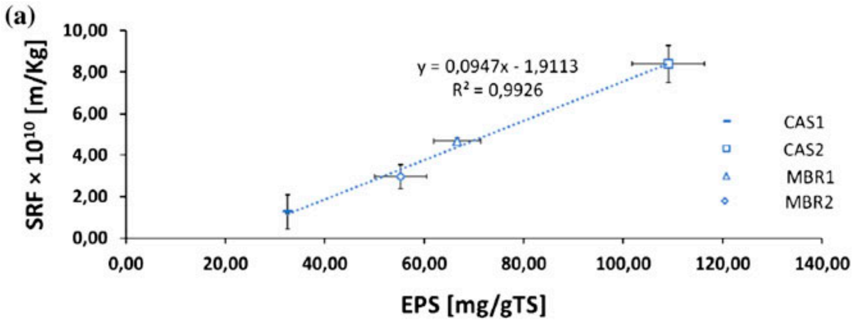


Fig. 3. EPS components concentrations at increasing EPS in the sludges (a) and in the digestates (b).



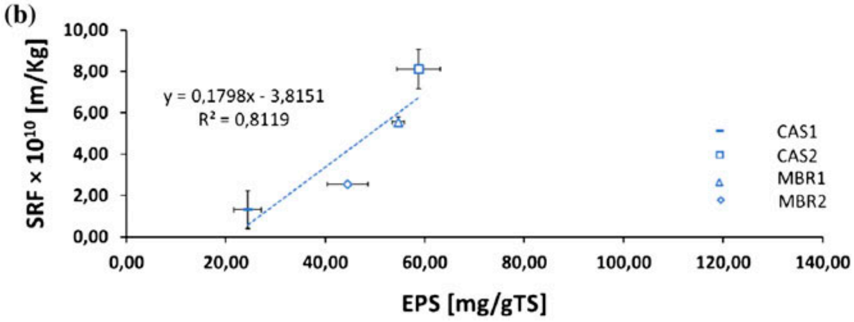


Fig. 4. Correlation between the EPS concentration and SRF in the sludge (a) and in the digestate (b).

the 80% of the total initial EPS. Moreover, anaerobic digestion definitely changes the relative composition of EPS that goes randomly to change sludge by sludge. The random distribution of the EPS components reflects the development, at the end of the test, of different bacterial distributions among the four anaerobic reactors. Such different compositions explain even the effect that the EPS amount has on the dewaterability of the sludge. Fig. 4 shows the relationship between the values of SRF and total EPS concentration obtained from each sludge (Fig. 4(a)) and the respective digestate (Fig. 4(b)).

A very well fitting linear relation is found for EPS concentration and SRF values of sludges, reflecting a dominant effect of the EPS on the rheological properties of the sludge. It is to underline that the tested sludge is from different plants operating with different technologies and operational parameters. Whenever this trend would be confirmed by further experiments with a wider number of sludges, the total EPS concentration could be a parameter to previse the sludge dewatering behavior, or vice versa, the SRF value could give information about the total EPS content in the sludge. Clearly, this EPS effect is less dominant in the digested sludge where the correlation is not so high. It is worth to notice how the SRF remains substantially constant after AD and extra-30 d adjustment time although the EPS concentration results reduced in all the digested sludge. This means that in the digested sludge, although the EPS still strongly contributes to the dewatering properties, it seems that other parameters (e.g. particle size distribution, EPS composition) play a significant role in determining the overall resistance to filtration.

4. Conclusions

The excess sludge from MBR WWTPs is not always to be considered biologically stabilized since the tested sludges showed a relatively high BMP. Such potential makes sustainable and exploitable the anaerobic treatment of MBR sludge. It was also observed that anaerobic digestion followed by a 30 d adjustment time did not cause worse dewatering properties of the tested sludges. Such worsening, found in a previous study, is not present if an adjustment time is waited before sludge dewatering. Moreover, for the four investigated sludges a strong inverse correlation was found between the SRF and the SRT indicating that a higher sludge retention in the oxidation bioreactors results in better dewaterability properties. The EPS structure, amount, and the relative components abundance, were evolving during the anaerobic digestion. A strong direct correlation was also found between the SRF values and the total EPS content, reflecting a dominant effect of the EPS on the rheological properties of the sludge. Such EPS effect is less important in the digested sludge where the correlation is not so high and other parameters (e.g. particle size distribution, EPS composition) play a significant role in determining the overall resistance to filtration.

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References

[1] H.Y. Ng, S.W. Hermanowicz, Membrane bioreactor operation at short solids retention times: Performance and biomass characteristics, Water Res. 39 (2005) 981–992.

[2] C. Solisio, V.G. Dovì, A location-routing approach to optimal sludge management, Chem. Eng. Trans. 35 (2013) 643–648.

[3] J.I. Houghton, T. Stephenson, Effect of influent organic content on digested sludge extracellular polymer content and dewaterability, Water Res. 36 (2002) 3620–3628.

[4] S. Rosenberger, M. Kraume, Filterability of activated sludge in membrane bioreactors, Desalination 146 (2002) 373–379.

[5] P.R. Karr, T.M. Keinath, Influence of particle size on sludge dewaterability, J. Water Pollut. Control Fed. 38 (1978) 1911–1930.

[6] S. Rosenberger, K. Kubin, M. Kraume, Rheology of activated sludge in membrane bioreactors, Eng. Life Sci. 2 (2002) 269–275.

[7] G. Di Bella, D. Di Trapani, G. Freni, M. Torregrossa, G. Viviani, Analysis of biomass characteristics in MBR and MB-MBR systems fed with synthetic wastewater: Influence of a gradual salinity increase, Chem. Eng. Trans. 38 (2014) 445–450.

[8] Y. Qi, K.B. Thapa, A.F. Hoadley, Application of filtration aids for improving sludge dewatering properties—A review, Chem. Eng. J. 171 (2011) 373–384.

[9] B. Frølund, R. Palmgren, K. Keiding, P.H. Nielsen, Extraction of extracellular polymers from activated sludge using a cation exchange resin, Water Res. 30 (1996) 1749–1758.

[10] M. Dubois, K.A. Gilles, J.K. Hamilton, P. Rebers, F. Smith, Colorimetric method for determination of sugars and related substances, Anal. Chem. 28 (1956) 350–356.

[11] N. Blumenkrantz, G. Asboe-Hansen, New method for quantitative determination of uronic acids, Anal. Biochem. 54 (1973) 484–489.

[12] P. Kintner III, J. Van Buren, Carbohydrate interference and its correction in pectin analysis using the m-hydroxydiphenyl method, J. Food Sci. (USA) 47 (1982) 756–759.

[13] O.H. Lowry, N.J. Rosebrough, A.L. Farr, R.J. Randall, Protein measurement with the Folin phenol reagent, J. Biol. Chem. 193 (1951) 265–275.

[14] G. Esposito, L. Frunzo, A. Giordano, F. Liotta, A. Panico, F. Pirozzi, Anaerobic co-digestion of organic wastes, Rev. Environ. Sci. Biotechnol. 11 (2012) 325–341.

[15] G. Esposito, L. Frunzo, A. Panico, G. d’Antonio, Mathematical modelling of disintegration-limited co-digestion of OFMSW and sewage sludge, Water Sci. Technol. 58 (2008) 1513–1519.

[16] G. Esposito, L. Frunzo, A. Panico, F. Pirozzi, Model calibration and validation for OFMSW and sewage sludge co-digestion reactors, Waste Manage. 31 (2011) 2527–2535.

[17] G. Esposito, L. Frunzo, A. Panico, F. Pirozzi, Modelling the effect of the OLR and OFMSW particle size on the performances of an anaerobic co-digestion reactor, Process Biochem. 46 (2011) 557–565.

[18] H.Y. Ng, S.W. Hermanowicz, Specific resistance to filtration of biomass from membrane bioreactor reactor and activated sludge: Effects of exocellular polymeric substances and dispersed microorganisms, Water Environ. Res. 77 (2005) 187–192.

[19] L. Pontoni, G. d’Antonio, G. Esposito, M. Fabbricino, L. Frunzo, F. Pirozzi, Thermal pretreatment of olive mill wastewater for efficient methane production: Control of aromatic substances degradation by monitoring cyclohexane carboxylic acid, Environ. Technol. (UK) 36 (2015) 1785–1794.

[20] W. Owen, D. Stuckey, J. Healy, L. Young, P. McCarty, Bioassay for monitoring biochemical methane potential and anaerobic toxicity, Water Res. 13 (1979) 485–492.

[21] S.M. Oh, M.H. Kim, Y.S. Bae, S.M. Hong, C.H. Park, Estimation of biodegradability and biogas recovery from a two-phase anaerobic process treating piggery wastewater, Desalin. Water Treat. 2 (2009) 30–38.

[22] W. Apha, AWWA (1998) Standard methods for the examination of water and wastewater, Amer. Pub. Health Association, Washington DC, 1998.

[23] L. Pontoni, G. D’Alessandro, G. d’Antonio, G. Esposito, M. Fabbricino, L. Frunzo, F. Pirozzi, Effect of anaerobic digestion on rheological parameters and dewaterability of aerobic sludges from MBR and conventional activated sludge plants, Chem. Eng. Trans. 43 (2015) 2311–2316.

[24] Z. Yu, X. Wen, M. Xu, M. Qi, X. Huang, Anaerobic digestibility of the waste activated sludge discharged from large-scale membrane bioreactors, Bioresour. Technol. 126 (2012) 358–361.

[25] D.F. Lawler, Y.J. Chung, S.-J. Hwang, B.A. Hull, Anaerobic digestion: Effects on particle size and dewaterability, J. Water Pollut. Control Fed. 58 (1986) 1107–1117.

[26] IRSA-CNR, Metodi Analitici Per i Fanghi, vol. 2, Parametri Tecnologici, Quaderni dell’Istituto di Ricerca sulle Acque, Roma, 1984 (in Italian).