

**XXX International Conference on  
Surface Modification Technologies (SMT30)**

29TH JUNE - 1ST JULY, 2016, MILAN, ITALY

**EVALUATION OF ELECTRODE SURFACE TREATMENTS IN  
SLUDGE ELECTRO-OSMOSIS DEWATERING**

Paolo Gronchi<sup>a\*</sup>, Roberto Canziani<sup>b</sup>, Andrea Brenna<sup>a</sup>, Simone Visigalli<sup>a</sup>, Carles Colominas<sup>c</sup>, Francesc Montalà<sup>c</sup>, Victor Cot<sup>c</sup>, Aristide Stradi<sup>d</sup>, Giancarlo Ferrari<sup>d</sup>, Vanni Bellelli<sup>d</sup>, Cristina Diaz<sup>e</sup>, Gonzalo Garcia Fuentes<sup>e</sup>

<sup>a</sup> *Department of Chemistry, Materials and Chemical Engineering, Politecnico di Milano, Milano, Italy*

<sup>b</sup> *Department of Civil and Environmental Engineering, Politecnico di Milano, Milano, Italy*

<sup>c</sup> *Flubetech S.L., Barcelona, Spain*

<sup>d</sup> *AST System Automation S.A.S., Modena, Italy*

<sup>e</sup> *AIN-Asociación de la Industria Navarra, Cordovilla, Spain*

---

**Abstract**

The drying of sludge produced by Wastewater Treatment Plants (WWTPs) is a very hard process due to the presence of the colloid fraction. Electro-osmosis could be a suitable technique to reduce the water content of the final sludge. Electric fields of 10 V/cm, 15 V/cm and 20 V/cm have been studied for electro-osmosis tests in a static or dynamic apparatus, obtaining a dry solids content up to 40-45%, with respect to 25-30% obtained by mechanical methods. In order to optimise the apparatus, the corrosion behaviour of the anodic material appears the main critical aspect, due to the high circulating current density. Moreover, wear may be detrimental for the surface of rotating electrodes. We then investigated the behaviour of materials used as electrodes mainly by evaluating the efficiency of the process and their surface aspect after treatment. The full understandings of the electrochemical reactions developed at the anode are a key factor for the material choice. We compared the efficiency and the corrosion resistance of anodes made of titanium MMO with respect to bare stainless steel (AISI 304) and stainless steel coated by PVD technique with TiN, AlTiN and DLC. Characterization of the anodes samples by roughness tests and by SEM, AFM and potentiodynamic tests, show that corrosion resistance of the DSA was the most suitable for our application. However, efficiencies of the electro-osmosis process for all the materials used have been found comparable, in terms of developed current densities and total energy consumptions, for short-test duration.

**Keywords:** electro-osmosis; dewatering; sludge.

---

**1. Introduction**

The so-called “activated sludge process” is adopted by almost the totality of urban and industrial wastewater treatment plants to achieve the concentration limits of biodegradable pollutants stated by the law to allow the discharge of treated effluents to natural water bodies (rivers, lakes and the sea). About half of the organic pollution load treated by the activated sludge process is oxidised and converted into water and carbon dioxide, while the remaining is converted into biomass, called “excess biological sludge” or “waste sludge”. After reducing the content of biodegradable matter and after reducing the water content through mechanical dewatering or thermal drying, the sludge becomes a product suitable for its final disposal.

When compared with thermal (evaporative) processes for water reduction, mechanical dewatering is often selected due to its low energy requirement. The processes of mechanical dewatering are largely developed on the

---

\* Corresponding author. Tel.: +39-02-23993274.

E-mail address: paolo.gronchi@polimi.it

industrial scale and, thanks to the pressure application, can produce sludge with 20-25% of dry solid (DS) content, and, in some cases, up to 30%.

However, the high DS values demanded for thermal valorisation of sludge cannot be achieved by mechanical dewatering techniques.

Seeking new and efficient methods for dewatering, Yoshida (1993), Barton et al. (1999), Gingerich et al., (1999) exploited electro-osmosis in order to remove water from sludge: the application of an electric field, sometimes in combination with pressure, seems capable to increase the DS content well beyond the values that can be achieved by mechanical means. Among electrokinetic phenomena, electro-osmosis rules this process and leads to a transport of water molecules to the negative electrode (cathode), increasing the dry matter significantly and lowering the energy consumption with respect to conventional techniques.

The application of an electric field, combined with a pressure, tends to increase sludge dewaterability: electro-osmosis reduces the interstitial water and some extent of the vicinal water, thus resulting in a dryer sludge cake.

Many experimental factors can influence the reduction of water content and, consequently, the process yield. The critical processing factors are voltage (or current), pressure, time, particles size distribution, zeta potential, conditioning parameters, polyelectrolyte characteristics, temperature etc.

The electrode reactions are affected not only by the materials of the electrode but also by the ions in the electrolyte: they may cause some hindering during electro dewatering

Reaction at the cathode produces hydroxide ions and those at the anode produces protons and this may result in a pH gradient across the filter cake. Yuan (2003), Citeau (2011) and Tuan et al. (2010) reported that the pH drop relates to the reduction of the absolute value of the zeta potential, thus the decrease in pH (specifically at the anode) reduced the electro-osmotic flow during direct current application (Tuan, 2008).

The oxidation of the anode material, due to oxygen evolution, reduces the process efficiency and can cause in some applications the contamination of the filter cake or filtrate, increasing the operating cost. Anodes such as stainless steel will be subject to corrosion, thus the use of dimensionally stable anode (DSA) materials is necessary. The anodes used in electro-osmosis experiments are usually made of conventional metallic plates (or meshes) such as stainless steel or nickel steel (which have sufficient strength but are easily corroded), graphite (which is cheap but fragile and cannot undergo to pressure conditions) and copper.

Raats et al. (2002) and Saveyn et al. (2006) documented that the use of titanium coated with mixed metal oxide (MMO) plates such as  $\text{Ir}_2\text{O}_3$ -coated titanium as anode prevents corrosion. Anodes made of conducting ceramic materials (e.g. iridium oxide or ruthenium oxide) coated on titanium seem to be highly effective due to their excellent strength, flexibility and corrosion resistance.

The choice of the cathode electrode materials presents far less problems in terms of corrosion resistance: stainless steel, copper and nickel plates or meshes can be used.

In this paper a preliminary investigation of materials to be used as anodes in the process of sludge electro-dewatering is carried out. The anode metal is subject to corrosion and wear due to the low pH of the sludge as the current moves through the cake, oxygen evolution, high organic content, and compacting/rotation of the piston. Dimensionally stable anodes are strongly resistive in this kind of environment. Our aim has been the study of stainless steel AISI 304 as anode in the electro-osmosis process. We compared performances, efficiencies and corrosion resistance with respect to titanium MMO. We also evaluated the behaviour of PVD coating on the metal under the same conditions.

Characterization of the anodes samples by roughness tests, SEM, AFM and potentiodynamic tests were used to express the corrosion resistance of the materials.

## **2. Materials and methods**

### *2.1 Sludge samples*

Sludge samples were taken from San Rocco (Milan) WWTP. We used aerobically digested sludge, pre-dewatered by mechanical treatment with two different devices: the first samples are taken after the Bucher (Bucher Unipektin) treatment, the other ones after filter press.

Each sludge sample dewatered by filter press is crumbled and homogenised by a shredder (Moulinex-La Moulinette), which lead to a uniform cake to be treated. In order to control the sludge quality, the electrical conductivity was measured by a conductivity meter (B&C Electronics-C 125.2) and pH by a pH-meter Metrohm 827 pH Lab.

Sludge samples are stored at 4 °C for less than five days prior to the experiments in order to keep constant their properties (pH and conductivity) and hinder the possibility of degradation. All types of sludge used for our electro-osmosis tests are shown in Table 1.

Table 1. Sludge samples characteristics (DS= dry solid; VS= volatile solid).

Sludge	Mech. dewatering	DS [%]	VS/TS[%]	pH	Conductivity [ $\mu\text{S/cm}$ ]
A	Bucher press	20.1	70.1	5.5	N.A.
B	Filter press	27.9	72.7	6.3	N.A.
F	Filter press	23.6	73.2	6.0	1132
G	Filter press	21.9	75.9	5.5	1277

## 2.2 Device assembling

The lab-scale device used for sludge electro-osmosis dewatering is described in Fig. 1. It consists of: a) cylindrical glass vessel ( $h=176$  mm,  $\varnothing=80$  mm); b) cooling water-jacket; c) compressed air system (1-4.5 bar); d) double effect cylinder (200 mm stroke) SMC-CP96 (piston); e) DC power supply (30 V-5 A), f) anode: DSA Ti (MMO), fixed to the piston; g) cathode: stainless steel mesh (AISI 304); cloth: PTT (polytrimethylene terephthalate).

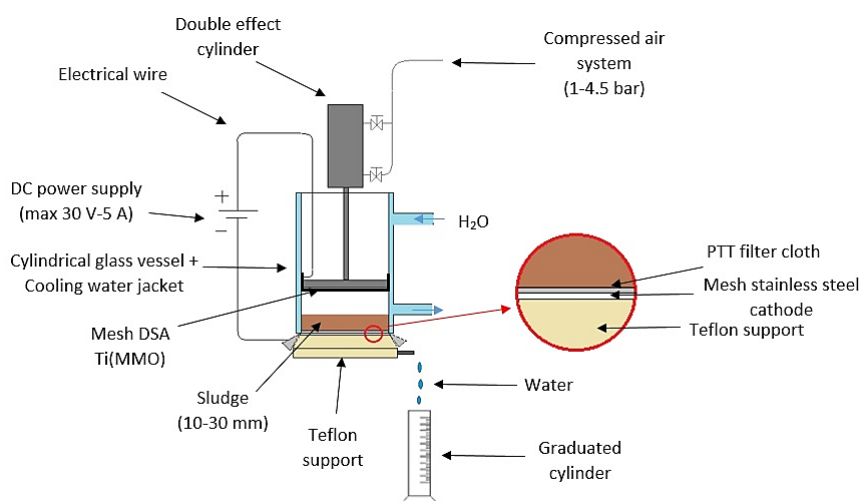


Fig. 1. Lab-scale apparatus for electro-dewatering tests.

The upper electrode (the anode) is attached to the piston, while the lower stainless steel mesh cathode (AISI 304) is covered by the PTT filter medium. The cathode and the anode are connected to the negative and the positive pole of the DC power supply (GBC bench scale generator, maximum 30 V/5 A) respectively. The piston is connected to the laboratory pressurisation system, with pressure regulation.

The water is then collected in a graduated cylinder in order to measure weight of the liquid lost during the experiments with respect to time and calculate the sludge dewatering rate.

In order to control temperature during dewatering tests, a thermocouple (Data logger thermometer OMEGA-HH306A) is inserted into the glass cell.

The rotation of the piston used for static experiments is obtained by bevel gears, which transmit the movement of a mechanical stirrer. In order to hinder the possibility of twisting of wires during the motion of the piston and anode, we put in contact small circular pipes of copper and the lateral parts of the anode and connected crocodile clips with the copper pipe.

## 2.3 Experimental set up and operations.

Following a procedure similar to that used by Mahmoud et al. (2011), the electro-dewatering test procedure follows two stages: a) filtration/compression under pressure: 5 min; b) application of the electric field at the selected operating voltage under pressure too.

The end time of the dewatering test is taken when no more than two drops of filtrate are collected in 5 min.

1<sup>st</sup> step: the glass cell is filled with homogenized sludge: 1 cm of thickness, about 35 g of sludge depending on the initial DS amount, for static piston experiments. Then it is closed by the cover and the piston starts applying pressure. Sludge is pressed between the upper anode (on the PTFE support) and the lower PTT filter cloth (placed on the cathode mesh). Mechanical pressure is applied for 5 minutes. No water was extracted during the application of pressure. This proves that no water could be extracted by purely mechanical means.

2<sup>nd</sup> step: then, electric field is switched on, and values of currents in function of time are recorded. At the same time, every minute the weight of extracted water and cell temperature are registered. When the interval between two drops of water exceeded two minutes, the experiment was stopped.

At the end of the experiment: the dewatered cake is released and weighted. Its DS amount is determined by drying at 105 °C during 12 h (Metodi di analisi del compost - Manuale ANPA. Manuali e linee guida).

Each experiment is repeated three times.

When the dynamic piston is used, rotation is started after 7 minutes of electric field application and a higher sludge thickness (3 cm) is investigated.

#### 2.4 Materials for electrode

DSA (Industrie De Nora SpA, Milan, Italy) materials consist of a titanium matrix coated by iridium dioxide with elements like cobalt (1096 mg/kg), iron (209.09 mg/kg), platinum (117.8 mg/kg), neodymium (25.75 mg/kg), manganese (3.51 mg/kg) and nickel (1.17 mg/kg).

Electrodes made by AISI 304 stainless steel, which has the best quality/price ratio for our applications were then tested for economic reasons.

The coatings applied on steel were TiN, AlTiN, DLC (diamond like carbon), which are usually exploited for their wear and abrasion resistance.

In Table 2 the coatings are reported.

Table 2. Electrodes Coatings (by PVD).

	TiN	AlTiN	DLC
Layer Thickness [ $\mu\text{m}$ ]	1-4	2-4	2-4
Hardness [HV]	2500	3200	2700
Friction coefficient (*)	0.4	0.5	0.1
Oxidation Temp. [ $^{\circ}\text{C}$ ]	450	1100	400
Colour	Golden	Black violet	Black anthracite

(\*) Tested against hardened steel at 25°C and 85% humidity.

#### 2.5 Electrode Materials characterization

Fig. 2 shows the cyclic potentiodynamic tests (scan rate 0.17 mV/s) on X5CrNi18-10 (AISI 304) and MMO-Ti (DSA mesh electrode) before electro-osmosis test.

The curves concerning the AISI 304 electrode show that the free corrosion potential ( $E_{\text{corr}}$ ) is -0.339 V Ag/AgCl<sub>KCl,sat</sub> while, corresponding to -0.170 V Ag/AgCl<sub>KCl,sat</sub>, an increase of the curve slope is observed (potential passive range) in which the current increases slowly for each potential variation due to high anodic resistance of the passive metal. Oxygen evolution reaction and passive film breakdown is close to 1 V Ag/AgCl<sub>KCl,sat</sub>.

The free corrosion potential of DSA is between 0 and -0.2 V Ag/AgCl<sub>KCl,sat</sub>. Similar considerations can be extended with respect to the discussed curves on AISI 304 stainless steel.

However, potentiodynamic tests on AISI 304 and DSA differ strongly in the decreasing potential curve (reverse scan). While in the first case (AISI 304), the curves at increasing polarization and at decreasing polarization (reverse scan) do not coincide, in the latter (MMO-Ti) the curves coincide. The meaning of this behavior is related to the absence of modification of the passive film on the Ti\_MMO electrode during transpassivity. In case of stainless steel, the passivity is destroyed by acidity formed by oxygen evolution reaction and corrosion occurs at strong penetration rate. In case of activated titanium, the mixed metal oxides on the surface are able to resist to the acidity at 10 A/m<sup>2</sup>, without any corrosion of the metal.

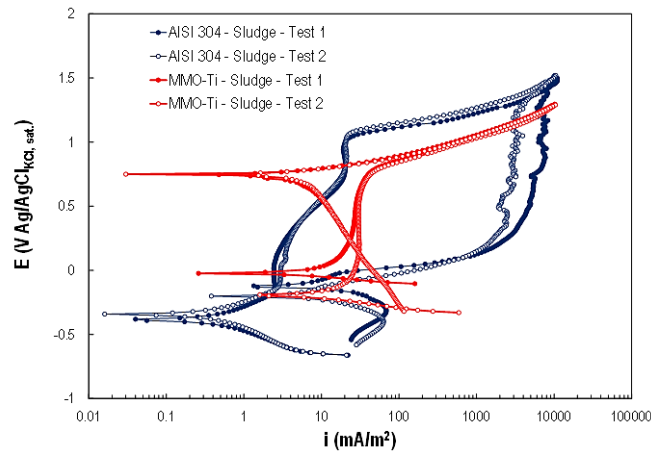


Fig. 2. Cyclic polarization curves on AISI 304 (blue) and DSA (red) electrodes.

### 3. Results and discussion

Our aim in performing our experiments has been the choice of suitable values of pressure, electric field and sludge cake thickness in order to obtain the highest efficiency and the best results in terms of final DS amount.

We set the initial cake thickness at 1 cm, in order to have a thin insulating layer during electro-osmosis: high cake thickness, indeed, leads to low currents due to strong resistance.

The pressure was fixed at 3 bar: this value, according to literature, is sufficient to maintain the contact between sludge and electrodes. The pressure by itself is not sufficient to remove completely the water.

Therefore, the use of an electric field is necessary to reach DS in the range of 40-45%, which is the aimed value, considered the data in literature.

#### 3.1 The dewatering process

Fig. 3 shows the evolution of the most relevant parameters: current density, collected water and rate of the dewatering with time for different applied electric field.

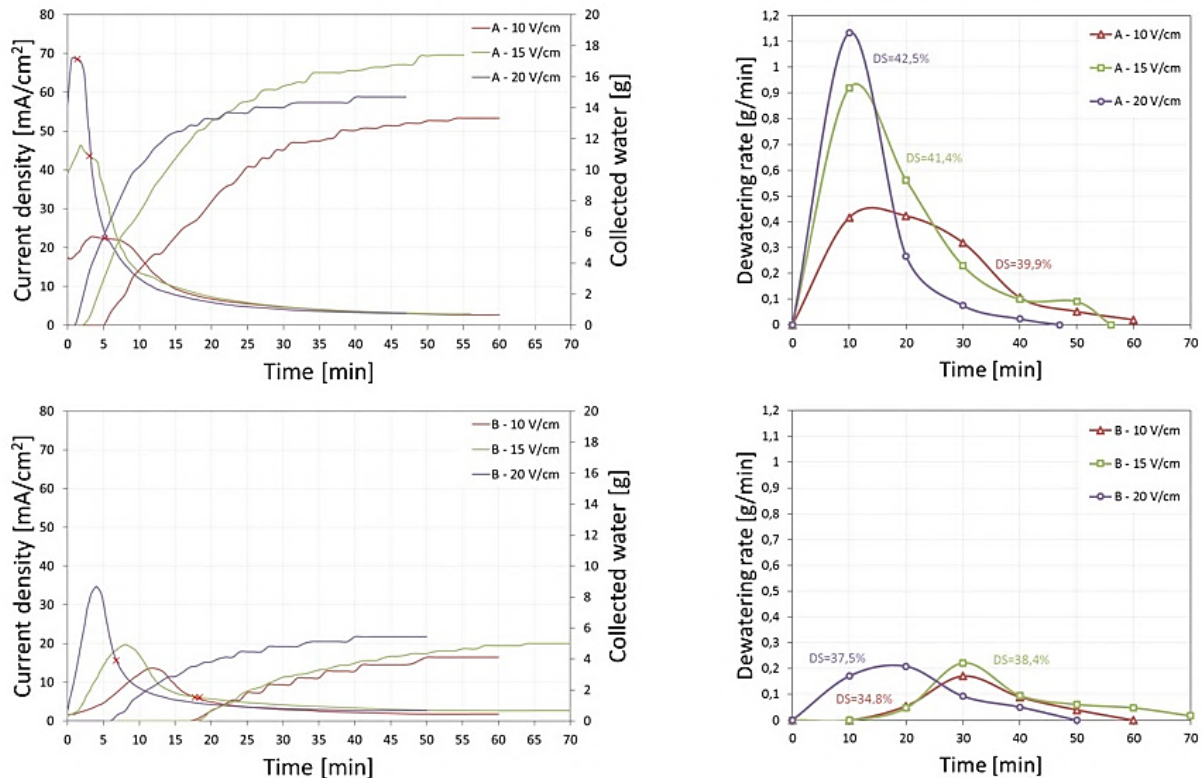


Fig. 3. Process parameters evolution for DSA with A and B sludge at 10, 15 and 20 V/cm.

The curve of the current density shows a maximum in a very short time from the electric field application time ( $t < 5$  min) depending on the DS content of the sludge and then abruptly decreases near 0 in 20-25 minutes. The maximum indeed is at about 70 mA/cm<sup>2</sup> with sludge A and the half with the B sludge, which have respectively 20 and 25% of DS. The dewatering begins soon after the current density maximum, after an induction time, with a rate that is highest at maximum slope decrease point.

The dependence from the DS content of the process efficiencies indicates a strong influence of the wetted percent area of the electrodes. Some coating with hydrophilic surface might increase the process yield.

A specific trend is observed: increasing the electric field (from 10 V/cm to 20 V/cm), the final DS amount does not increase as well. However, a minor time is spent before the dewatering stop (no more than one drop is collected in 5 minutes). This kind of behaviour is confirmed by Tuan (2011): higher the potential, higher is the kinetics of the process, but the DS does not increase too much (and in some cases remains the same) if the potential range is small.

It must be considered that often time at which 90% of water is collected are more comparable and similar if the initial conditions are the same.

The final DS amount is calculated by weighing the cake after 24 h at 105 °C in the oven. This is necessary since the filtrate can contain sludge particles and during the experiments the evaporation of water due to Joule effect must be taken into account.

Potential values used in electro-dewatering applications usually exceed the decomposition voltage of water. This implies that the electrochemical cell exhibits a considerable Ohmic loss, which causes heating in the dewatering device and thus a decreasing liquid viscosity enhancing dewatering kinetics as clearly explained by Mahmoud et al. (2011).

Moreover, higher values of initial DS with greater amount of sludge in the cell, corresponds a stronger evaporation of water: this value could be determined by the difference between the final DS amount computed after 24 h in the oven and the mass of water collected. Our tests confirmed that values of potentials lower than 20 V could not increase the temperature over 30 °C, so the Joule effect and water evaporation are limited.

Moreover, the use of a water jacket guaranteed the thermal stability inside the cell: temperature never exceeded 28 °C at the anode in all the tests performed in this work. The electrode efficiencies may be evaluated on performances base and on corrosion resistance

### 3.2 Electrodes energy efficiencies

The total energy consumption (Wh/kg<sub>(extracted water)</sub>) assessment is computed by dividing the consumed energy by mass of water collected (kg). To do this, the average current (A) is computed by adding together the values of electric charge (Ah), calculated multiplying current and time (h) point wise during the test, and dividing it for the total time; energy (Wh) is computed by multiplying the value of average current (A) with that of imposed potential (V) and with that of total test time (h).

According to literature, after electro-osmosis dewatering, values of energy consumptions found for types of sludge with a DS value lower than 13% are usually under 0.4 kWh/kg<sub>(extracted water)</sub>. These results have been achieved with potentials from 10 V to 50 V and times higher than 3 hours as reported by Tuan (2011) and Mahmoud et al. (2011). However, starting from sludge with an initial DS higher than 22%, the specific energy to remove water is greater. We chose to set our energy consumption threshold at a value of 0.250 kWh/kg<sub>(extracted water)</sub> in order to have a saving with respect thermal treatment at the same initial conditions.

#### 3.2.1 X5CrNi18-10 (AISI 304) anode

Pure stainless steel disc (AISI 304) with five holes ( $\varnothing=8.2$  mm) to let the gas overflow were examined at first.

Starting from sludge F (DS<sub>i</sub>=23.6%), we performed tests with three stainless steel discs at the usual initial conditions: 35 g of sludge (1 cm of thickness) and pressure set at 3 bar. The results are shown in Table 3.

Table 3. Electro-osmosis results of F sludge (DS<sub>i</sub>=23.6%) with stainless steel (AISI 304) anode.

Sludge and value identity	E [V/cm]	t <sub>TOT</sub> [min]	Tot. energy consumption [Wh/kg <sub>H2O</sub> ]	DS <sub>f</sub> [%]
FM1	10	30-35	495.4	27.1
FM2	15	22-25	428.8	29.4
FM3	20	18-23	287.6	37.1

\*Each value is the medium (M) value of 3 tests at least.

The first remark that must be highlighted is that even if the initial DS ( $DS_i$ ) is sufficiently low, the conductivity ( $1132 \mu\text{S}/\text{cm}$ , Table 1) is not so high as expected (the salinity of sludge is lower than the previous ones). This fact strongly affects the efficiency of the tests, bringing to a low final DS ( $DS_f$ ) and to high specific energy consumption, especially for tests FM1 and FM2.

The low value of  $DS_i$  also is coupled with a less evaporation due to Joule effect (especially at  $10 \text{ V}/\text{cm}$ ), which can also be a reason why the final DS is low: in previous experiments, a considerable amount of water was removed by evaporation.

Moreover, the corrosion of the discs is the main problem encountered during the experiments: localized corrosion was evident since the first test and the situation got worse increasing the electric field application time. This fact surely has been one of the causes of low dewatering.

In Fig. 4 we can see that the first drop (usually corresponds to the time of maximum in a Current density-Time diagram) is obtained at relative low times in every tests, as expected for a sludge with a  $DS_i$  under 25%. The current densities developed during the process are comparable with those of DSA anode (with a remarkable transient stage for the tests at  $10 \text{ V}/\text{cm}$  and  $15 \text{ V}/\text{cm}$ ). The electro-osmosis tests last shorter times with respect to the experiments with DSA anode, probably due to the strong corrosion of the surface.

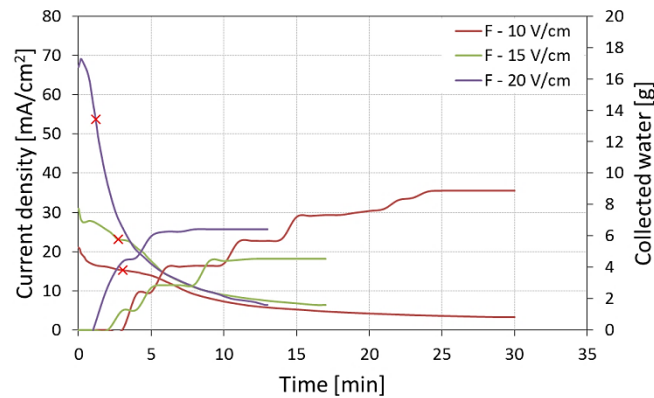


Fig. 4. Current density vs Time diagram of F sludge with stainless steel anodes. Best results. Collected water amount is reported.

### 3.2.2 MMO-Ti (DSA)

DSA<sup>®</sup> electrodes, provided by Industrie De Nora SpA are used as reference material in order to compare our results with those found in literature. Test results are reported in Table 4. The DSA anode does not show evident corrosion after many tests. It is however very expensive and a cheaper material is needed. As a consequence, we tested steel electrodes coated with different ceramic layers.

The DS, after electro-dewatering, increases from 20.1% to 38-40.5%, independently from the potential value set during the tests. An evident change of processing time is observed: it is shorter in tests at  $20 \text{ V}/\text{cm}$  due to a higher kinetics. It is useful to remind that time is not taken as an arbitrary parameter, but it depends on the drop rate (no drops in five minutes).

Table 4. Electro-osmosis of A sludge ( $DS_i=20.1\%$ ) with DSA.

N°	E [V/cm]	$t_{TOT}$ [min]	Tot. energy consumption [Wh/kg <sub>H2O</sub> ]	$DS_f$ [%]
AM	10	65-105	168,7	38,9
AM	15	55-61	266,3	39,9
AM	20	45-52	343,9	40,5

Energy consumption should not exceed 250-300 Wh/kg of water removed, in order to be cost-effective and energy efficient. Therefore the test at  $20 \text{ V}/\text{cm}$  exceeds this threshold. We can deduce that higher is the potential, higher is the total energy consumption (due to an increase in values of current density), if the result is equal in terms of  $DS_f$  amount.

### 3.2.3 TiN

Starting from the results obtained on the stainless steel substrate, we tested two different TiN coatings (1  $\mu\text{m}$  and 3  $\mu\text{m}$  of thickness) at 15 V/cm. The results are shown in Table 5.

As shown, we achieved better results in terms of final DS with respect to those found with stainless steel anode. This fact can be attributed primarily to the conductivity of the sludge that was used after 5 days and in this time it progressively increased until a value of 1211  $\mu\text{S}/\text{cm}$ . Therefore, at 15 V/cm we succeed, also with coated samples, in removing a suitable amount of water, like with DSA experiments, in restricted times.

However, these kinds of results are obtained only with the first tests with the virgin anodes: after 10 minutes of potential application, the TiN discs are corroded and no other experiments can be performed due to their behaviour as a resistor. In fact, when we tried to set a potential of 15 V, we found out that the maximum imposable value was 2 V, with the maximum current born by the power supply (5 A).

Table 5. Electro-osmosis results of F sludge ( $DS_i=23.6\%$ ) with TiN coated anode.

N°	E [V/cm]	$t_{TOT}$ [min]	Tot. energy consumption [Wh/kg <sub>H2O</sub> ]	$DS_f$ [%]
F-10 (1 $\mu\text{m}$ )	15	17	119.2	39.7
F-11 (3 $\mu\text{m}$ )	15	18	136.9	38.8

Diagrams related to the tests with TiN discs are shown in Fig. 5. As expected, the maximum values of current density are achieved for the thinnest coatings, due to a reduced resistance of the ceramic layer. Moreover, the first drop is obtained later in the time with a thick coating.

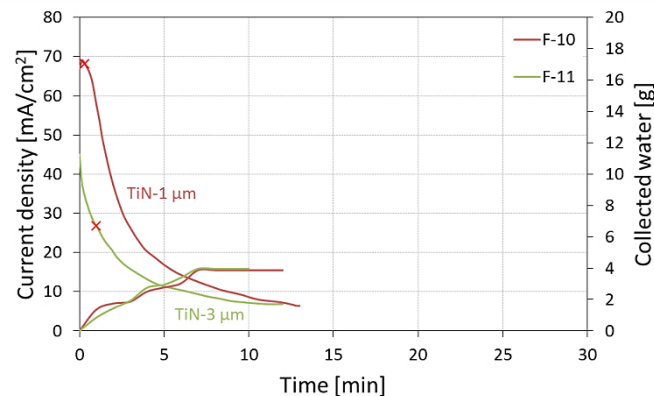


Fig. 5. Current density vs Time diagrams at 15 V/cm of F sludge with TiN discs (1  $\mu\text{m}$  and 3  $\mu\text{m}$ ). Collected water amount is reported.

### 3.2.4 AlTiN

Starting from a cake thickness of 3 cm (equal to 60 g) of sludge G ( $DS_i=21.9\%$ ), we performed two tests by using a AlTiN coated stainless steel anode: one with the static (G-1) and one with the rotating piston (G-2) started after 7 minutes of electric field application. For both the tests, the pressure has been maintained at 3 bar and the potential at 15 V (5 V/cm). The results are shown in Table 6.

Table 6. Electro-osmosis results of G sludge ( $DS_i=21.9\%$ ) with AlTiN coated anode.

N°	E [V/cm]	$t_{TOT}$ [min]	Tot. energy consumption [Wh/kg <sub>H2O</sub> ]	$DS_f$ [%]
G-1 (static)	15	30	385.1	25.7
G-2 (rotating)	15	30	277.3	28.0

As shown above, the efficiency of electro-osmosis is not suitable in the case of high cake thicknesses (3 cm) due to the strong resistance between the electrodes.

However, it seems that the final DS obtained by the movement of the piston is slightly higher and the energy consumption is consequently lower with respect to the static experiment.



Fig. 6 shows the Current density vs Time diagrams of tests G-1 and G-2. The shape of the curve relative to the static experiment is continue and has a maximum in  $26 \text{ mA/cm}^2$ , while for G-2 initially the current density slightly increases and then it raises with a discontinuous step after the starting of rotation (7 minutes).

From this preliminary analysis, it seems that the main characteristic that must be taken into account for the electro-osmosis dewatering is the sludge cake conductivity.

For low times of electric field applications, corrosion is important but not the leading parameter. However, when the coated anodes have a long processing time, it is inevitable that corrosion greatly influences sludge dewaterability. The rotation of the anode seems to be efficient in increasing the conductivity of sludge, but this phenomenon could be more pronounced with a lower initial cake thickness (1-1.5 cm).

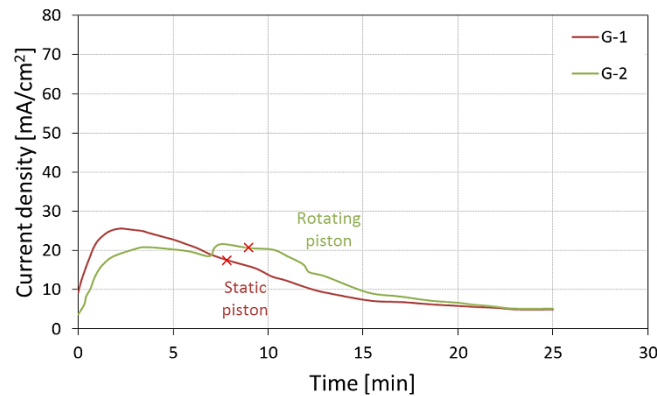


Fig. 6. Current density vs Time diagrams at 15 V/cm of G sludge with AlTiN discs (static and rotating piston).

### 3.2.5 DLC

The same dynamic tests have been performed for DLC coated anodes. By applying 3 bar and 15 V (5 V/cm) on a 3 cm cake, we investigated the behaviour of sludge G after starting the rotation of the piston (minute 7).

Table 7. Electro-osmosis results of G sludge ( $DS_i=21.9\%$ ) with DLC coated anode.

N°	E [V/cm]	$t_{rot}$ [min]	Tot. energy consumption [Wh/kg <sub>H2O</sub> ]	$DS_f$ [%]
G-3 (rotating)	15	30	253.9	28.3
G-4 (rotating)	15	30	296.0	27.5

The DS results are in the range of G-2 test and energy consumption is still quite high. The main issue for low efficiency is not the kind of ceramic coating used, but it mainly depends on the high cake thickness and consequent high resistivity. Current density vs Time diagrams of tests G-3 and G-4 are shown in Fig. 7.

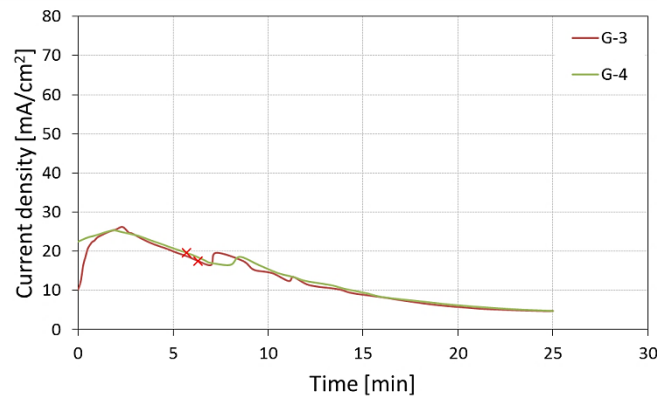


Fig. 7. Current density vs Time diagrams at 15 V/cm of G sludge with DLC discs.

Slopes, after the start of rotation, increase of about  $5 \text{ mA/cm}^2$  thanks to the increase in conductivity got by the movement of the cake surface. However, the efficiency is still too low because of the high sludge thickness.

### 3.3 Electrode surface wear and corrosion.

As above stated, the efficiency of the electro-osmosis process is strictly related to the conductivity of the sludge cake and to the electrode corrosion and damage. In fact, due to the oxygen evolution and the acidic environment on the anode, their performances worsen with time and our purpose is the investigation of their resistance with a preliminary study in the lab-scale device.

#### 3.3.1 X5CrNi18-10 (AISI 304) anode

Localized corrosion is the main phenomenon occurring during electro-osmosis for stainless steel anodes (see 3.2.1). The kind of corrosion is clearly identified observing the photographs reported in Fig. 8. The surface morphology is similar for the three stainless steel discs after the tests at 10 V/cm, 15 V/cm and 20 V/cm with sludge F. It must be highlighted that the disc used for the 20 V/cm test, seems to have a smoother surface. This last observation, together with DS results with tests at 20 V, could be evidence that a high voltage can overcome the strong corrosion of the anode. It must be however taken into account that processing times for the same disc are lower than the other two discs.

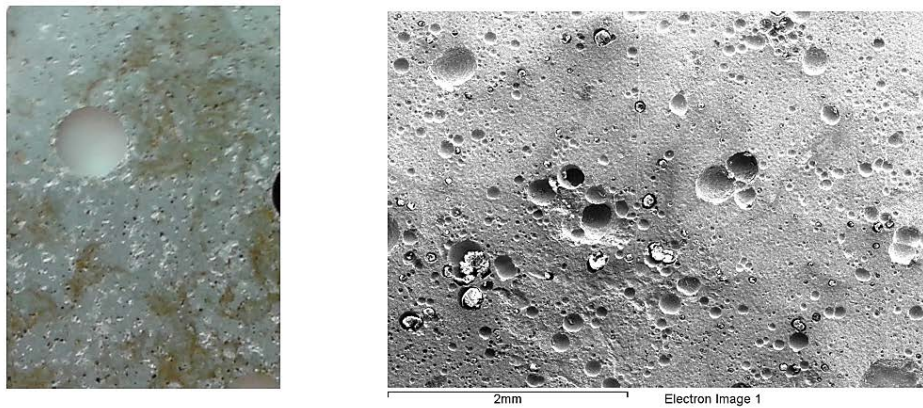


Fig. 8. Stainless steel electrodes surface after an electro-osmotic test. Localized corrosion is clearly visible.

In order to investigate the unusual result of corrosion and efficiency, we wanted to investigate lower times of potential application checking for slighter corrosions.

Starting from sludge G ( $DS_i=21.9\%$ ), we examined the behaviour of the stainless steel anodes in static contact with sludge (50 g of sludge G) situated between the electrodes. A potential of 10 V and 20 V for 30/60 minutes was applied. The weight loss after the test is the index of the corrosion degree. In Table 8 the results of corrosion tests are given in terms of weight loss are shown.

Table 8. Weight loss of stainless steel anodes after electric field application with G sludge.

Disc	Process conditions	Weight loss [%]
a	10 V/cm – 30 min	0.29
b	10 V/cm – 60 min	0.32
c	20 V/cm – 30 min	0.10
d	20 V/cm – 60 min	0.17

The table shows that, also in this case, a stronger corrosion is present for the case at lower potential, where the weight loss value is more than doubled.

### 3.3.2 MMO-Ti (DSA)

The anode has a mesh body, so is very different from the previous ones. After electro-osmotic tests the microphotograph reveal a homogeneous surface without any porosity or coloured oxidised area. Corrosion seems to be inexistent.

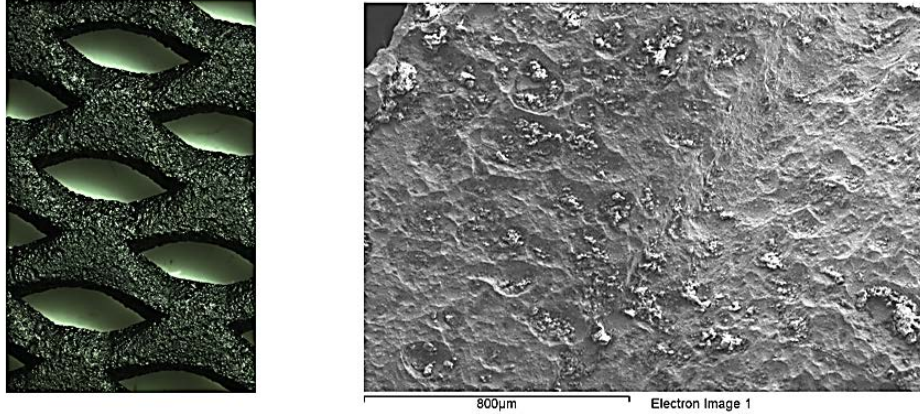


Fig. 9. DSA anode surface after electro-osmosis test at 15 V/cm.

### 3.3.3 TiN

The aspect of TiN anodes after electro-osmosis tests at 15 V/cm is shown in Fig. 10.

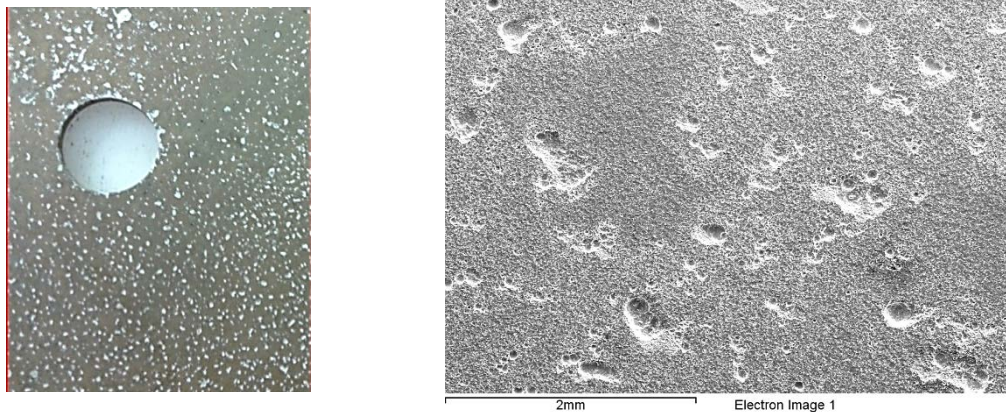


Fig. 10. Corroded TiN disc (3 µm of thickness) after electro-osmosis test at 15 V/cm.

Localized corrosion corrosion is the main mechanism during these tests and it is caused by the great presence of pores in stainless steel bare and in the ceramic-coated stainless steel. The examined ceramic coatings do not obstacle the corrosion oxidation. This fact is deleterious for the efficiency of the process since the contact between substrate and sludge environment causes a rapid dissolution of metal.

We can see that the thin film (1 µm) disc is more corroded than the 3 µm-disc and both of them are less damaged than the stainless steel anodes. However, at the same time, it must be considered that a too much thick coating will condition the resistivity of the anodes and the developed current densities (with a change in total energy consumption). A compromise between these factors must be found.

### 3.3.4 AlTiN

Looking at Fig. 11 we can see the morphology of the AlTiN coated anode after 25 minutes (test G-1) and 50 minutes (test G-2) of potential application (15 V). It is clear that the corrosion is one of the deleterious factors for removing water from sludge, together with the high resistivity of the cake. It is also evident that by using a virgin anode on a static piston the efficiency is lower than the corroded disc on the rotating piston: this fact seems to highlight that, for low experimental times, the higher efficiency is most influenced by an increase in conductivity of the cake than by a higher conductivity of the anode. For low times of electric field applications, corrosion is

important but not the leading parameter. However, when the coated anodes have a long processing time, is inevitable that corrosion greatly influences sludge dewaterability.

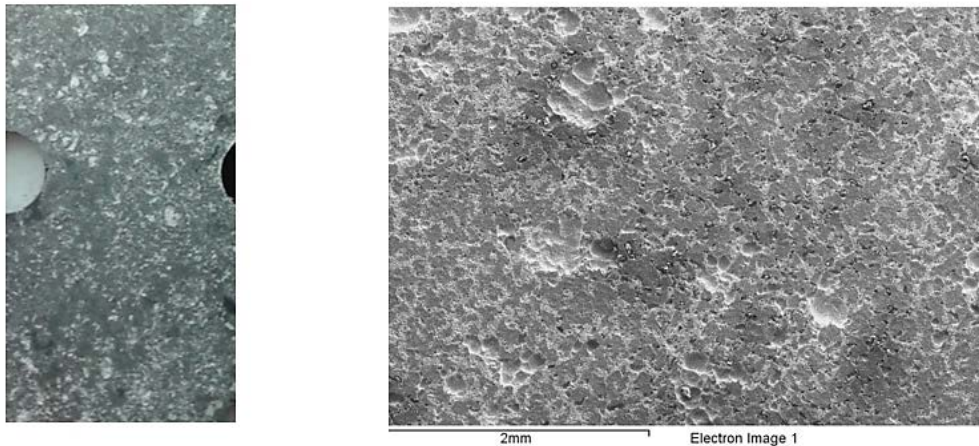


Fig. 11. Corroded AlTiN disc (1  $\mu\text{m}$  of thickness) after electro-osmosis test at 15 V/cm.

### 3.3.5 DLC

In Fig. 12 DLC anode after tests G-3 and G-4 at 15 V is shown. In this case it seems that the main phenomenon occurring during electro-osmosis tests is erosion corrosion: there is a general damage of the anode surface and localized corrosion is not evident like in the previous pictures. This fact could be ascribed also for a greater presence of pores into the DLC layer with respect to other ceramic coatings.

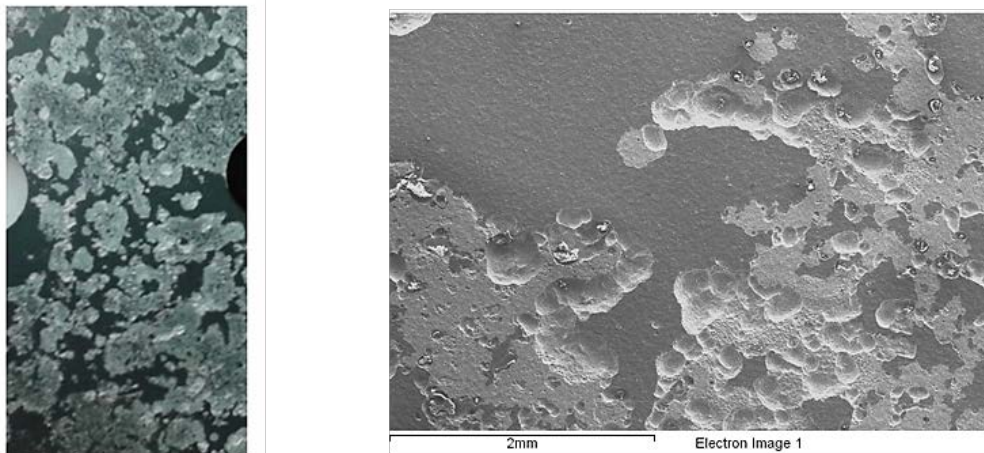


Fig. 12. Wear corroded DLC disc (1  $\mu\text{m}$  of thickness) after electro-osmosis test at 15 V/cm.

## 4. Conclusions

The dewatering of sludge, with the help of an electric field, seems an efficient method to increase the dry solid content of the dewatered sludge. It could offer an alternative process to the more costly thermal drying methods that are usually applied for energy production from sludge through combustion processes. The greatest challenge is to keep an advantageous compromise between the high costs for production and maintenance of corrosion and wear resistant electrodes and the overall cost of the equipment. This article refers to the first steps of the investigation that, taking into account already known chemical physical drawbacks (anodic dissolution of iron based materials), aims to achieve a methodology for determine the issue.

Efficiency and corrosion are useful parameters for the assessment of the process and of the electrode materials. From the efficiency point of view, the limit of 250 Wh/kg<sub>(extracted water)</sub>, that is much lower than values reported in the literature, was respected only by the TiN coated stainless steel electrode. The ranking with an

electric field of 15 V/cm, of the examined materials is: TiN ( $DS_f \approx 39.0\%$ ) > DSA ( $DS_f \approx 39.9\%$ ) > DLC-rot ( $DS_f \approx 27.9\%$ ) > AlTiN-rot ( $DS_f = 28.0\%$ ) > AlTiN ( $DS_f = 25.7\%$ ) > AISI 304 ( $DS_f \approx 29.4\%$ ).

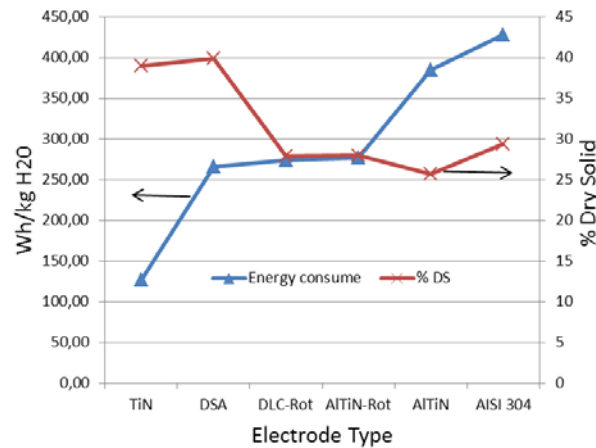


Fig. 13. Specific energy consumption and dry solid content at the end of tests for each electrode ( $E=15$  V/cm).

Energy efficiency data are shown in Fig. 13 together with the DS percentage at the end of tests. It must be observed that the results strongly depend on sludge characteristics and cake thickness. It seems to exist an inversely proportional relation between the two data series: the lower the specific consumed energy, the higher the dry solid percent. This is probably depending on the dry solid content in the wet sludge at the beginning of the experiment.

From the corrosion point of view, stainless steel electrodes are the most corroded ones among those investigated: a large area dense of localized corrosion is evident on the surface. The DSA electrodes are the best performing as for corrosion damage: they do not show any evident surface degradation. The, ceramic coated, stainless steel electrodes exhibit intermediate corrosion degradation.

This preliminary investigation has pointed out the characteristics of some materials for the electro-osmotic tests and it has delineated the methodology of the research. The work indicates that research should focus on a material constituted by a conductive metal coated by a conductive film resistant to wear and corrosion, in order to simulate the performances of commercial DSA electrode. With this aim, organic or ceramic coatings with conductive charges are currently under investigation.

## Acknowledgements

This work has received funding from the Project SLUDGEtreat, co-funded by the European Commission within the FP7 (2007–2013) Marie Curie Actions - Industry-Academia Partnerships and Pathways - IAPP, GA n. 611593) and from the LIFE14 project no. ENV/IT/000039 “ELECTRO-SLUDGE”, partly funded with the contribution of the LIFE Programme of the European Union. The authors wish to thank Metropolitana Milanese Spa for supplying sludge samples and Industrie De Nora Spa for providing DSA electrode.

## References

- Laraia, R., 2001. ANPA-Metodi di analisi del compost - Manuale. Manuali e linee guida, 3/2001.
- Barton, W. A., Miller, S. A., & Veal, C. J., 1999. The electrodewatering of sewage sludges. *Drying Technology*, 17(3), 497-522.
- Citeau, M., Larue, O., & Vorobiev, E., 2011. Influence of salt, pH and polyelectrolyte on the pressure electro-dewatering of sewage sludge. *Water Research*, 45(6), 2167-2180.
- Gingerich, I., Neufeld, R. D., & Thomas, T. A., 1999. Electroosmotically enhanced sludge pressure filtration. *Water Environmental Research*, 71(Issue 3), 267-276.
- Mahmoud, A., Olivier, J., Vaxelaire, J., & Hoadley, A. F. A., 2011. Electro-dewatering of wastewater sludge: Influence of the operating conditions and their interactions effects. *Water Research*, 45(9), 2795-2810.

- Raats, M., 2002. Full scale electrokinetic dewatering of waste sludge. *Colloids and Surfaces. A Physicochemical and Engineering Aspects*, 210(2-3), 231-241.
- Saveyn, H., Van der Meeren, P., Pauwels, G., & Timmerman, R. (2006). Bench- and pilot-scale sludge electro-dewatering in a diaphragm filter press. *Water Science & Technology*, 54(Issue 9), 53-60.
- Tuan, P., 2011. Sewage sludge electro-dewatering., *Acta Universitatis Lappeenrantaensis*. Thesis, Mikkeli University Consortium, Finland.
- Tuan, P., & Sillanpää, M., 2010. Migration of ions and organic matter during electro-dewatering of anaerobic sludge. *Journal of Hazardous Materials*, 173(1-3), 54-61.
- Tuan, P., Jurate, V., & Mika, S., 2008. Electro-dewatering of sludge under pressure and non-pressure conditions. *Environmental Technology*, 29(10), 1075-1084.
- Yoshida, H., 1993. Practical aspects of dewatering enhanced by electro-osmosis. *Drying Technology*, 11(4), 787-814.
- Yuan, C., & Weng, C., 2003. Sludge dewatering by electrokinetic technique: Effect of processing time and potential gradient. *Advances in Environmental Research*, 7(3), 727-732.