Sewage sludge electro-osmotic dewatering
RINGRAZIAMENTI

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ABSTRACT

Sludge produced by Wastewater Treatment Plants (WWTPs) usually has a poor dewaterability. Industrially, mechanical dewatering is the method employed to increase the dry solids (DS) of sewage sludge. The aim of dewatering is the decrease of transport and disposal costs, but the efficiency of the mechanical dehydration methods is low. Electro-osmosis could be a suitable technique to reduce water content of the final sludge by the application of an electric field. Studies about electro-dewatering reported that the DS could increase to values around 40-45%, with respect to 25-30% obtained by mechanical methods. Here, we investigated the parameters affecting pressure-driven electro-dewatering, with a static and a dynamic device, starting from types of sludge mechanically pre-dehydrated. The applied voltage has been set at 10 V, 15 V and 20 V in order to examine total energy consumption and final DS. The results confirmed values of final DS around 40% with the static piston. Initial DS and sludge conductivity, together with the applied electric field, are the key factors of electro-osmosis tests. The rotating piston highlighted that a mixing of sludge during the potential application could slightly increase the sludge conductivity, but cakes must be kept under 1.5 cm of thickness in order to achieve a good dewatering. In addition, a preliminary analysis of different materials for the anodes to be used in this apparatus has been done, in order to evaluate the influence of corrosion during the process. Stainless steel (AISI 304) has been used as a substrate for ceramic PVD coatings (TiN, AlTiN and DLC), but samples evidenced a low corrosion resistance.
Il fango prodotto dagli impianti di trattamento delle acque reflue di solito ha una scarsa disidratabilità. Industrialmente, la disidratazione meccanica è il metodo impiegato per aumentare la sostanza secca (SS) dei fanghi di depurazione. L'obiettivo della disidratazione è la diminuzione dei costi di trasporto e di smaltimento, ma l'efficienza della disidratazione meccanica è bassa. L'elettroosmosi potrebbe essere una tecnica adatta per ridurre la quantità di acqua del fango finale tramite l'applicazione di un campo elettrico. Studi riguardanti l'elettro-disidratazione hanno riportato che la SS potrebbe aumentare fino a valori intorno al 40-45%, rispetto al 25-30% ottenuti con metodi meccanici. Qui, abbiamo analizzato i parametri che influenzano l'elettro-disidratazione a pressione applicata, con un apparato statico e uno dinamico, partendo da fanghi precedentemente disidratati meccanicamente. Il potenziale applicato è stato di 10 V, 15 V e 20 V con l'obiettivo di esaminare l'energia totale consumata e la finale SS. I risultati hanno confermato valori di SS finale intorno al 40% con il pistone statico. La SS iniziale e la conducibilità del fango, insieme al campo elettrico applicato, sono i fattori chiave delle prove di elettroosmosi. Il pistone rotante ha evidenziato che una miscelazione dei fanghi durante l'applicazione del potenziale può aumentare leggermente la conducibilità dei fanghi, ma lo spessore del fango deve essere mantenuto al di sotto di 1,5 cm per ottenere una buona disidratazione. Inoltre, è stata condotta un'analisi preliminare di differenti materiali per gli anodi da utilizzare in questo apparato, al fine di valutare l'influenza della corrosione durante il processo. L'acciaio inossidabile (AISI 304) è stato usato come substrato per rivestimenti ceramici depositati con tecnica PVD (TiN, AlTiN e DLC), ma i campioni hanno evidenziato una bassa resistenza alla corrosione.
“La parola di un uomo è il più duraturo dei materiali”

(Arthur Schopenhauer)
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”. At present, this technique is the cheapest way to remove colloidal and soluble organic pollutants from sewage, but it produces a huge amount of liquid waste sludge, with a dry solid (DS) content of less than 2%, rich in organic substances, mostly biodegradable. Therefore, it needs further processes to reduce its volume, by reducing its water content, and to reduce its polluting potential, due to its high content of biodegradable organic matter. Regarding the dry matter of sewage sludge, it contains up to 80% volatile solids (at 600°C), made of active biomass, organic debris and residual of cellular decay. The organic fraction (or volatile solids, VS) of the dry solid content of sewage sludge can be reduced down to about 60% after biodegradation through further aerobic or anaerobic biological processes, often called “stabilisation” or “digestion” processes, as they aim at reducing the content of biodegradable matter and “stabilise” the biodegradation processes.

1.1 Aim of the thesis

The aim of this work is to establish the feasibility of electro-osmotic dewatering, under constant voltage, for different sludge types and determine the dewatering efficiency in terms of final DS (dry solid) content, dewatering rate and energy consumption. Therefore, we focused on the optimization of operating conditions to achieve the best results in terms of solids percentage at the minimum energy consumption, comparing the characteristics of the bench lab-scale device used with other electro-osmotic apparatus described in literature.

My Master thesis is part of a project named SLUDGEtreat where Politecnico di Milano (Department of Chemistry, Materials and Chemical Engineering; Department of Civil and Environmental Engineering), AST System Automation S.A.S. (Modena, Italy), Flubetech S.L. (Barcelona, Spain) and AIN-Asociación de la Industria Navarra (Cordovilla, Spain)
1. INTRODUCTION

cooperate in order to design, refine and improve the process of electro-dewatering. All of them, starting from the electro-dewatering device patented by Stradi et al. (N°: US2013/0098765 A1), are trying to improve water removal efficiency, lower capital costs and fulfil Atex rules about oxygen and hydrogen evolution.

1.2 Structure of the thesis

This thesis is organised as follows:

- **Chapter 1** introduces the activated sludge process and states the aim of the thesis and its structure.
- **Chapter 2** describes the problem of sludge derived from Wastewater Treatment Plants in Europe and Italy, laws and regulations that rules sludge production and disposal. It also summarises the state of the art about sludge properties, the types of water, the characteristics that affect sludge dewatering and sludge conditioning.
- **Chapter 3** presents the principal methods of mechanical dewatering. It describes the electro-osmotic dewatering and the electrokinetic phenomena. It also shows a bibliographic research about electro-dewatering in laboratories and on industrial scale.
- **Chapter 4** illustrates the lab-scale device used for the electro-osmotic tests, the methods, the operations and the materials for the experimental part.
- **Chapter 5** shows the results of the tests with different sludge samples, different materials for the anodes and with a set-up modification of the lab scale device, under constant electric field.
- **Chapter 6** presents a summary and the conclusions of the research, with recommendations for further investigations.
2. SLUDGE FROM WWTPs

2.1 The problem of sludge in Europe

The annual production of sewage sludge in Europe in 2010 was about 11.5 million tonnes of DS in 27 member states of the European Union (Table 1): Germany is the largest producer, followed by United Kingdom, Italy, Spain and France.\textsuperscript{1} About 70\% of the annual European sewage sludge production is obtained by these five countries. All over the world, an increase in sludge production is expected.\textsuperscript{2}

Table 1-Sewage sludge production in EU countries in 2010.\textsuperscript{3}

<table>
<thead>
<tr>
<th>Member State</th>
<th>Sewage sludge production [t DS/year]</th>
<th>Proportion of total EU volume [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Austria</td>
<td>273,000</td>
<td>2.4</td>
</tr>
<tr>
<td>Belgium</td>
<td>170,000</td>
<td>1.5</td>
</tr>
<tr>
<td>Bulgaria</td>
<td>47,000</td>
<td>0.4</td>
</tr>
<tr>
<td>Cyprus</td>
<td>10,800</td>
<td>0.1</td>
</tr>
<tr>
<td>Czech Republic</td>
<td>260,000</td>
<td>2.3</td>
</tr>
<tr>
<td>Denmark</td>
<td>140,000</td>
<td>1.2</td>
</tr>
<tr>
<td>Estonia</td>
<td>33,000</td>
<td>0.3</td>
</tr>
<tr>
<td>Finland</td>
<td>155,000</td>
<td>1.3</td>
</tr>
<tr>
<td>France</td>
<td>1,300,000</td>
<td>11.3</td>
</tr>
<tr>
<td>Germany</td>
<td>2,000,000</td>
<td>17.4</td>
</tr>
<tr>
<td>Greece</td>
<td>260,000</td>
<td>2.3</td>
</tr>
<tr>
<td>Hungary</td>
<td>175,000</td>
<td>1.5</td>
</tr>
<tr>
<td>Ireland</td>
<td>135,000</td>
<td>1.2</td>
</tr>
<tr>
<td>Italy</td>
<td>1,500,000</td>
<td>13</td>
</tr>
<tr>
<td>Latvia</td>
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<td>0.3</td>
</tr>
<tr>
<td>Lithuania</td>
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<td>0.7</td>
</tr>
<tr>
<td>Luxembourg</td>
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<td>0.1</td>
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<td>Malta</td>
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<td>0.1</td>
</tr>
<tr>
<td>Netherlands</td>
<td>560,000</td>
<td>4.9</td>
</tr>
<tr>
<td>Poland</td>
<td>520,000</td>
<td>4.5</td>
</tr>
<tr>
<td>Portugal</td>
<td>420,000</td>
<td>3.7</td>
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<tr>
<td>Romania</td>
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<td>1.4</td>
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<tr>
<td>Slovakia</td>
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</tr>
<tr>
<td>Slovenia</td>
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</tr>
<tr>
<td>Spain</td>
<td>1,280,000</td>
<td>11.1</td>
</tr>
<tr>
<td>Sweden</td>
<td>250,000</td>
<td>2.2</td>
</tr>
<tr>
<td>United Kingdom</td>
<td>1,640,000</td>
<td>14.3</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>11,503,800</strong></td>
<td><strong>100</strong></td>
</tr>
</tbody>
</table>

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. The most commonly applied routes are:
2. SLUDGE FROM WWTPs

- direct land application in agriculture or forestry;
- indirect use in agriculture, as a soil-amending material (compost) obtained through further processing with other organic wastes;
- incineration, with or without energy recovery;
- sanitary landfills.

The dry content of stabilised sewage sludge is rich in nutrients such as phosphorous (0.05 to 3%)\textsuperscript{3}, nitrogen (1 to 6%)\textsuperscript{4} and potassium (<1%)\textsuperscript{5} and contains valuable organic matter (humus) that is useful for application to soils that are depleted or subject to erosion. Organic matter and nutrient content in sludge are key elements that make agricultural use of sludge a widespread method of sludge disposal. About 36% of the total amount of sewage sludge in EU is used in agriculture in year 2000 (Figure 1)\textsuperscript{4}, while in 2006 this fraction increased to 39%.\textsuperscript{1}

At European Community level, the use of sewage sludge in agriculture is regulated by the Sewage Sludge Directive 86/278/EEC and the Urban Water Treatment Directive 91/271/EEC. The Sewage Sludge Directive 86/278/EEC encourages the use of sewage sludge in agriculture and regulates its use to prevent harmful effects on both environment and human health. Therefore, environmental issues that may be associated with the use of sewage sludge in agriculture are mainly related to heavy metals, harmful organic compounds, nitrogen if present as nitrate ion (NO\textsubscript{3}\textsuperscript{-})\textsuperscript{6} and pathogenic microorganisms.\textsuperscript{6} However, the presence of high content of harmful substances, organic contaminants, heavy metals such as cadmium, copper, nickel, lead, zinc, mercury and chromium limits its use and leads to a decrease in soil applications (as stated in Waste Framework Directive 2008/98/EC, and Directive 99/31 on landfill of waste).
2. SLUDGE FROM WWTPs

It may be worth noticing that Switzerland has banned sludge landfilling (since 1996) and also its use in agriculture, as stated in the Ordinance on Fertilisers ("Ordonnance sur la mise en circulation des engrais", as modified on November 14th, 2007). Therefore, the only residual option remaining for the disposal of sewage sludge is incineration, according to the Technical Ordinance on Wastes, dated 10th December 1990, updated on July 1st 2011.

Sludge must be dewatered before composting, incinerating, landfilling or reusing as fertilizer, in order to decrease transport and treatment costs (sludge disposal costs are about 280-470 €/tDS in EU and 150-250 €/tDS in Italy). Different techniques, such as thermal, mechanical or electromechanical are investigated in order to reach the highest DS content and the lowest economic impact. The aims of dewatering include: recovery of the product for sale in a dry form, more practicable for further treatments; recovery of water for recycling; transformation into forms that are easily handled and can be safely disposed of.

2.2 The problem of sludge in Italy and Lombardy

Italian legislation on sludge is basically developed as an enforcement of EU Directives, and generally is included in those regarding wastes and wastewaters. In particular, environmental matters are mainly regulated by the Legislative Decree (DL) 152 of April 3rd, 2006 (where wastewater sludge is classified as “special waste”), recently completed and integrated by the DL 4 of January 16th, 2008.

Other important regulations regarding wastes, including sludge, are those relevant to incineration processes, and the DL 36 of January 13th, 2003, as enforcement of European Directive 1999/31/CE on waste landfilling, followed by the Ministry of Environment Decrees (DM) of March 13th, 2003, and August 3rd, 2005, defining criteria for waste admittance in landfill.

In Italy, sludge utilisation in agriculture is regulated by the DL 99 issued in 1992 in application of the European Directive 86/278/EEC, which states general conditions for sludge application and utilisation on land, including limits on heavy metals, Salmonellae, but not on organic contaminants. Each of the 20 Italian Regions may issue and enforce Regional Directives aimed at adapting general requirements to local situations, including introduction of more stringent limits.
2. Sludge from WWTPs

In Lombardy, a production of around one million tonnes of sewage sludge was documented (IRER, 2010). Over half of the sludge, after appropriate treatment, is designed to agricultural reuse; the remaining half is sent, in roughly equal parts, to landfill or for incineration.

Sludge production in Lombardy is going to increase due to the growing consumption and population. Moreover, the tens of millions of Euros allocated in Lombardy for large interventions in response to EC infringement procedure 2034/09, which imposes heavy penalties for purification and sewage systems that will not be in accordance with limits within 2015, will lead to an improvement in the quality of rivers and the environment, but simultaneously to an increase in the quantity of sludge.

Taking into account this trend, management of sewage sludge is becoming a fundamental factor in order to satisfy environmental rules and decrease disposal costs.

Figure 2 shows the sludge final use in Italy according to a research of 2006.
2.3 Introduction to sludge dewatering

Today’s sludge average DS content after mechanical dewatering lies in the range of 20-30% of dry solids, with average values around 24-25%\textsuperscript{11}.

The minimum legislative requirements for thermo-valorisation require that the sewage sludge is dried up to about 30-45% of DS in order to self-sustain combustion at 850 °C. At lower DS content, pre-heating the combustion air by adding additional fossil fuel (such as methane) is necessary to remove the water content from the sludge. Sewage sludge can be also dried up to 90% of DS content in drying ovens, which are heated by fossil fuels and then used in cement kilns or brick furnaces as both additional fuel (for its combustible volatile fraction) and inert filler (for its inert fraction).

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 industrial scale and, thanks to the pressure application, can produce sludge with 20-25% of DS content, and, in some cases, up to 30%. In general, the highest DS content can be achieved for primary sludge, while the lowest DS content is achieved for aerobically digested biological sludge.\textsuperscript{12} However, the high DS values demanded for thermal valorisation of sludge cannot be achieved by mechanical dewatering techniques. In the case of aerobically digested biological sludge, the goal of 25% dry solids can be hardly achieved, even with high-pressure filter-presses.

Seeking new and efficient methods for dewatering, many authors (Yoshida in 1993\textsuperscript{13}, Barton et al. in 1999\textsuperscript{14}, Gingerich et al. in 1999\textsuperscript{15}) 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 electrophoretic 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.

However, the high efficiency of this method is in contrast with its applicability due to the high sludge volumes involved, oxygen and hydrogen evolution at the electrodes (their amount must be lower than a fixed threshold ruled by ATEX), Joule effect caused by the application of electric field and the increase in resistance due to water removal. Electro-osmosis dewatering has been studied mainly at bench-scale, and industrial applications
2. SLUDGE FROM WWTPs

have been limited to very few full-scale plants. Therefore, these critical aspects must be taken into account in order to develop an effective, reliable, and economically feasible sludge electro-dewatering process.

2.4 Aims of sludge dewatering

The important characteristic of having high water amount in sludge (95-99%)\(^{16}\) is the cause of the implementation of dewatering processes in the WWTPs. Summarising, the objectives of dewatering include\(^{17}\):

- recovery of the product for sale in a dry form;
- the transformation of the waste into forms that are easily handled and can be safely disposed of, for environmentally-acceptable landfills and different recreational uses;
- recovery of water for recycling.
2. SLUDGE FROM WWTPS

2.5 Sludge liquid phase

2.5.1 Types of sludge

Sludge types in a WWTP vary according to the processes and methods of operation and they are classified as primary, secondary, tertiary and digested sludge: they are different in physical, chemical and biological characteristics, which have an influence onto sludge dewaterability.

Figure 3 shows the processes of a Wastewater Treatment Plant to achieve sludge suitable for agriculture, incineration and landfill or water for discharge.

Primary sludge
Primary wastewater treatment usually involves gravity sedimentation to remove settled solids and organics, usually by a primary clarifier. Half of the solids suspended in wastewater are removed through primary treatment. The residual material from this process is a concentrated suspension called primary sludge, made up of non-cellular organic components ranging from 70-75% of volatile solid, which will undergo further treatment to become biosolids.

Secondary sludge (also named “excess, biological sludge” or “waste activated sludge”)
In the secondary treatment process, called activated sludge process, microorganisms oxidise organic substrates to get the energy they need to grow. More precisely:
2. SLUDGE FROM WWTPs

- heterotrophic biomass consumes dissolved oxygen to degrade dissolved and suspended organic matter;
- autotrophic biomass consumes oxygen to oxidise ammonium nitrogen to nitrates;
- both kind of microorganisms take up ammonium and phosphate from the wastewater to fulfil their metabolic needs and to grow;
- they produce oxidised inert products (carbon dioxide, water, nitrates) and other microbial by-products.

The organic content of biological sludge varies between 60% and 80%. In general, dewaterability of secondary sludge depends on numerous factors. Excessive growth of filamentous bacteria, due to low dissolved oxygen, nutrients deficiency and high temperature, is a common problem in activated sludge process: the bulking sludge causes poor settling and dewatering. In order to control the bulking sludge, oxidant chemicals such as chlorine and hydrogen peroxide are added into the aeration tank. \(^{19}\) Anaerobic storage significantly reduces dewaterability of secondary sludge due to deflocculation. \(^{19}\)

**Tertiary sludge**

Tertiary treatment is used when high-quality effluent is required, such as direct discharge to a drinking water source, to reclaim water for unrestricted agricultural irrigation or to improve the quality of rivers that receive heavy pollution loads. It is produced by flocculation/precipitation downstream of the biological treatment and final filtration. In the presence of simple filtration, without the addition of inorganic reagents, the nature of the sludge is similar to that of secondary sludge. Otherwise chemical precipitates are present in a variable percentage in function of the process applied, but often sufficient to produce a good degree of biological stability. \(^{6}\)

**Digested sludge**

The purposes of sludge stabilization, performed by biological digestion under aerobic or anaerobic conditions, are reduction of hazardous chemicals and microbes as well as odour. Bio-stabilization reduces the organic content in sludge from 75% to 50%. \(^{19}\) *Anaerobic digestion* is a bacterial process that is carried out in the absence of oxygen. The process can either be thermophilic digestion, in which sludge is fermented in tanks at a temperature of 55 °C, at a retention time of about 15 days, or mesophilic, at a temperature of around 36 °C, at a retention time of about 30 days. The sludge is fed into
2. SLUDGE FROM WWTPs

Large tanks and held for a minimum of 12 days to allow the digestion process to perform the complex sequence of biological reactions that are necessary to convert biodegradable substrates into biogas (a mixture of methane, carbon dioxide, hydrogen sulphide and other minor components) and to produce a stabilized sludge. As a rough approximation, biological reactions can be grouped into four main steps:

- hydrolysis of particulates into colloids, which transforms into soluble compounds;
- acidogenesis, that is the conversion of soluble organic compounds into volatile fatty acids (VFAs);
- acetogenesis, that is the conversion of VFAs into acetic acid;
- heterotrophic methanogenesis, that is the conversion of acetic acid into methane and carbon dioxide; autotrophic methane production from carbon dioxide and hydrogen may contribute to biogas formation, but up to a very limited extent.

Methane is usually 65-70% of the total biogas produced. Part of the biogas is used to feed a boiler to heat the incoming sludge up to the required process temperature and to compensate heat dispersions from the digestion reactor. The excess biogas can feed endothermic engines or gas turbines, but it requires a preliminary treatment to remove humidity (by condensation), hydrogen sulphide (by alkaline scrubbing) and siloxanes (by adsorption on activated carbon granules). However, the large reactor volumes, the sludge heating system, the biogas treatment and the operation of the energy recovery systems entails high capital costs that can be recovered within the useful lifespan of the plant only if the energy obtained from the combined heat and power (CHP) units is subsidised.22

Aerobic digestion is a biological process, where organic matter in the presence of oxygen is converted into carbon dioxide, water and stabilised organic debris (humus). Once there is a lack of organic matter, bacteria die and are used as food by other bacteria (endogenous respiration): solids reduction occurs in this phase. Aerobic digestion takes from 10 to 15 days under ambient temperature and needs no insulation, but only compressed air as oxygen supply. Therefore, the capital costs of aerobic digestion are lower, but the operating costs are greater because of energy used by the blowers.23

After aerobic and anaerobic digestion, the dewaterability of the digested biological sludge is decreased in comparison to raw, undigested excess sludge due to the increase of fine particles.19
2. SLUDGE FROM WWTPs

2.5.2 Water distribution in sludge

Water contained in sludge can be of four types\textsuperscript{24, 25}:

- Free water: water not bound to the particles, which represents the largest part of sewage sludge (70-75\%). Mechanical dewatering technique such as filtration/compression, gravitational settling and centrifugation can easily remove this kind of water.

- Interstitial or capillary water: water bound by capillary forces between the sludge flocs or water trapped in crevices and interstitial spaces. Mechanical dewatering can release this water with a breaking of the floc.

- Surface or vicinal water: water associated with the solid particles. Multiple layers of water molecules hold tightly to the particle surface by hydrogen bonding. This water is held on particle surfaces thanks to the molecular structure of the water molecules and cannot be removed by mechanical methods.

- Intracellular water, water of hydration or chemically bound: water bound within the particle structure that can be released only by thermo-chemical destruction of the particles.

The most popular definition is that the free water freezes at freezing point, while bound water remains unfrozen (it freezes at -20 °C, or at -8 °C).\textsuperscript{19}

The different kinds of water are shown in Figure 4.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{water_distribution_in_sludge.png}
\caption{Distribution of water in sludge.}
\end{figure}
2. **SLUDGE FROM WWTPS**

Large and dense flocs are always preferable for good settling and dewatering: the poorer dewaterability of sludge is related to a larger amount of fine flocs in sludge because they not only clog the pore structure in the sludge cake, but also increase the amount of bound water.\(^{26}\)

The dewatering methods in relation to sludge-liquid phase are shown in Figure 5, where Stern layer and Helmholtz plane are drawn (see Chapter 2.6.3).

\[Figure\ 5:\textit{Dewatering methods in relation to water distribution in sludge.}\]
2. SLUDGE FROM WWTPs

2.6 Parameters affecting dewaterability

Sludge dewaterability is affected by many parameters, as shown in the following list:

- Capillary suction time (CST)
- Time to filter (TTF)
- Specific resistance to filtration (SRF)
- Extracellular polymeric substances (EPSs)
- Zeta potential
- Water content at 105°C
- Total and suspended solids
- Particle size distribution
- Temperature
- Microbial soluble products (MSPs)
- Bacterial species
- Viscosity
- Compressibility

2.6.1 CST, TTF and SRF

Sludge dewatering properties can be described by three parameters: capillary suction time (CST), time to filter (TTF) and specific resistance to filtration (SRF). The CST test (UNI-EN 14701-1)\textsuperscript{28} determines the rate of release of water from the sludge: it is performed placing a sludge sample in a small cylinder on a sheet of chromatography paper (Whatman rectangular filter), which extracts water by capillary suction. The time required to move in a specified distance is monitored measuring the conductivity change between two points appropriately spaced and in contact with the paper. The TTF and SRF (UNI-EN 14701-2) are similar if the viscosity and the content of the sludge do not vary among the compared samples. Both tests are performed by using the same system: a sludge sample is placed in a Buchner funnel with a paper support filter and vacuum is applied, the funnel is connected to a graduated cylinder and the amount of filtrate is measured as a function of time. The time required (seconds) for 50% reduction in sample volume is TTF, while SRF parameter can be calculated as\textsuperscript{19}:
2. SLUDGE FROM WWTPs

\[ SRF = \frac{2 \times A^2 \times \Delta P \times b}{\eta \times DS} \]

Where:
- SRF [m/kg]: specific resistance to filtration
- A [m²]: filtration area
- \( \Delta P \) [N/m²]: pressure drop across the filter cake
- \( b \) [s/cm⁶]: slope of the curve obtained by plotting the time of filtration to volume of filtrate ratio (s/cm³) versus \( V \) (cm³) itself
- \( \eta \) [kg/m/s]: dynamic viscosity
- DS [kg/m³]: solid content of sludge sample

CST is a rapid and simple measurement, but it is unrealistic because of no pressure application. SRF is considered a realizable dewatering measurement but the coefficient \( b \) is difficult to calculate and large errors appear when dealing with low DS of content sludge samples.²⁹

2.6.2 Extracellular polymeric substances

Extracellular Polymeric Substances (EPSs) are high molecular weight compounds accumulating on the surface of bacterial cells. They contain a variety of organic substances (EPS constitutes 50% to 90% of a biofilm total organic matter) such as carbohydrates, exopolysaccharides, exoproteins, DNA, humic acid, uronic acid, etc. EPSs form a protective layer for the cells against the harsh external environments (high toxicity, sudden change of pH) absorbing exogenous nutrients and organic molecules, and aggregate bacterial cells in flocs (Figure 6). Thus, EPSs play an important role in flocculation, settling and dewatering of sludge: an increase of EPSs results in a decrease in sludge dewaterability.¹⁹, ³⁰
2.6.3 Zeta potential

Zeta ($\zeta$) potential is the potential generated by the formation of an electrical double layer and it is responsible of the electrokinetic phenomena of colloid’s stability. Electrical double layer is depicted in Figure 7.

![Figure 7-Structure of electrical double layer, with the corresponding potential distribution with distance from a charged wall.](image)

As shown in the picture, the first layer is at the inner Helmholtz plane and bears the potential $\psi_i$, where co-ions and counterions are not hydrated and are specifically adsorbed to the surface. The second layer is defined by the outer Helmholtz plane with potential $\psi_d$, consisting of a layer of bound, hydrated, and partially hydrated counterions. The outermost and third layer is the diffuse layer, composed of mobile co-ions and counterions, in which resides the slip plane bearing the zeta potential, $\zeta$. In most cases, the outer Helmholtz plane and the slip plane are situated close to each other, allowing the approximation of $\psi_d$ with the $\zeta$ potential for practical purposes. The slip plane, or shear surface, is an imaginary plane separating ions that are immobile at the surface from those that are mobile in solution.\textsuperscript{27}

According to the Helmholtz-Smoluchowski theory, the electro-osmotic velocity, needed to compute zeta potential, can be derived based on the balance of the electrical and frictional forces between water and the wall of the capillary. It is described by the following equation:
2. Sludge from WWTPs

\[ v_{eo} = \frac{\varepsilon \zeta \Delta V}{\eta \Delta L} \]

Where:
- \( v_{eo} \) [m/s]: electro-osmotic velocity
- \( \zeta \) [V]: zeta potential
- \( \varepsilon \) [F/m]: dielectric permittivity of the liquid
- \( \eta \) [kg/ms]: viscosity of the liquid medium
- \( \Delta V \) [V]: electric potential
- \( \Delta L \) [m]: length of the capillary between the electrodes

The magnitude of the zeta potential indicates the degree of electrostatic repulsion between adjacent, similarly charged particles in a dispersion: a high zeta potential will confer stability and resistance to aggregation, while when the potential is small, attractive forces may exceed the repulsion and the dispersion may break and flocculate.\(^{32}\)

Taking into account how the Zeta potential condition the characteristic of sludge flocculation, some authors have studied its influence on performances in solid/liquids separation plants in order to upgrade and improve sludge dewaterability of existing plants.\(^{33, 34}\)

However, because sludge particles size must be lower than 100 \( \mu \)m to be measured by a zeta-meter, samples have to be treated before the analysis. Therefore, the sludge samples are first centrifuged to remove the supernatants at 2000 rpm for 10 min. Next, the retained sludge is re-suspended and washed with distilled water twice at the same centrifugation conditions for the removal of other impurities. Then, they undergo homogenization at 8000 rpm for 1.5 min.\(^{34}\) The samples are placed in the zeta-meter viewing chamber where an electric field is activated. This causes the colloids to move with a velocity that is proportional to their zeta potential, and their direction indicates whether their charge is positive or negative.
2.7 Sludge conditioning

Sludge conditioning is a process where sludge solids are treated with chemicals or various other means to prepare the sludge for dewatering processes. Both chemical conditioning and physical conditioning change the structure of the sludge in order to make the solid and liquid portions become more easily separable. The most widely used methods are chemical conditioning and physical conditioning.

a) Chemical conditioning
The use of appropriate dosage of organic, usually cationic, polymers (such as polyethylene imines epichlorohydrin/dimethylamine, diallyldimethyl ammonium chloride, acrylamine) at appropriate pH eliminate electrostatic repulsion, allowing short-range attractive forces. This fact makes zeta potential (ζ) approaching zero and promote aggregation (charge neutralization model). Otherwise, they can react as cationic sites to bind more than one sludge particle, thus bridging the system of solid particles (bridging model). As a result, flocculation and dewaterability with the use of a high molecular weight polymers increase.

Iron and aluminium salts and lime are commonly used as inorganic flocculants. However, mechanical dewatering methods do not perform well using sludge samples conditioned with inorganic flocculants. To achieve high sludge dewatering, doses of 20-40% of the original dry weight of sludge are usually required, causing a significant increase of sludge volume, which raises transport and disposal costs. Therefore, the use of inorganic flocculants is limited.19

Another chemical technique for sludge pre-treatment is Fenton oxidation by hydrogen peroxide/ferrous ions.

Oxidation by H₂O₂ and transition metal salts (such as Cu(II), Fe(II), Co(II), Ru(III), Ni(II)) or ozone, followed by an activation by UV-light, results in formation of hydrogen radicals and in a significant decrease in the water content of the final sludge cake. Advanced oxidation can help to improve sludge dewatering by degradation of EPSs, which reduces their water retention properties releasing EPSs-bound water, increasing the size of sludge flocs.

In Fenton oxidation, dewaterability of sludge is strongly dependent on the reaction temperature, H₂O₂ concentration and pH (the optimal pH is around 3 where EPSs are
2. Sludge from WWTPs

released from sludge particle surfaces and \( \zeta \) value increases reducing the electrostatic repulsion between particles).\textsuperscript{19, 35, 36}

b) Physical conditioning

Freezing and thawing convert the jellylike consistency of the wastewater to a granular-type that drains readily. During sludge freezing (at least 30 minutes at temperatures ranging from -10 to -200 °C), the free water begins to freeze and pushes the floc particles to the ice front. Once free water is frozen, the interstitial water is extracted by diffusion and added to the growing crystalline structure. All other impurities and solid particles are forced to the boundaries of the ice crystal where they become compressed or dehydrated. Freeze-thaw treatment transforms sludge into a compacted form and reduces the sludge bound water content. Thus, it makes sludge easier for settling and filtration: after thawing and dewatering, dewatered sludge cake can range from 25 to 40% of solids.\textsuperscript{37}

Another physical conditioning method is thermal treatment. Sludge heat treatment in temperature ranging from 40 to 180 °C: experiments performed at temperatures of 120 °C for 60 min showed that thermal treatment reduces the residual sludge amounts and enhances the dewaterability thanks to EPSs degradation and their natural properties are changed. Hydrolysis of the exocellular organic fractions (130-180 °C) leads to the destruction of the colloidal structure resulting in release of the bound water from the sludge particles.\textsuperscript{19}
3. TREATMENT PROCESS AND PLANT ASPECTS

3.1 Conventional dewatering techniques

Mechanical dewatering of sludge is conventionally integrated in WWTPs in order to lower the cost of transportation and disposal. Filtration by vacuum, by plate filter presses, by belt filter presses and centrifugation are the most used processes that exploit mechanical forces to remove water.

3.1.1 Vacuum filtration

Vacuum filters are widely used to dewater low solid slurries, wherein the solid constituent in a sludge is separated by a porous filter cloth rotated through the slurry, with vacuum applied to the inner surface to cause the solids to accumulate on the surface as a cake. The apparatus (Figure 8) consists of a rotary drum filter, a vacuum pump, a filtrate tank and a scraper blade. In the vacuum filtration process, the sludge is drawn to the surface of the drum, which is covered with the filter material, holding the solids and allowing the liquid to enter the drum. The liquid is pulled by the vacuum flow to a filtrate tank. A vacuum is applied to the drum as the drum rotates, drawing air through the solids on the drum face and further reducing their moisture content. Just prior to discharge, a blast of compressed air is used to push the solids away from the drum surface, thereby allowing the scraper blade to operate effectively without tearing the filter material on the drum surface. Nowadays they are very seldom used for dewatering sewage sludge.

![Figure 8 - Vacuum filtration process of sludge](image-url)
3. TREATMENT PROCESS AND PLANT ASPECTS

3.1.2 Plate filter press

A filter press (Figure 9) includes a plurality of plates arranged in a horizontal stack, where a pressure piece is hydraulically pushed towards a headpiece. The plates and headpieces have a central sludge feed port, while filtrate outlet ports are usually located at the corner of the plates. Each plate has a cloth on both sides with appropriate holes for the feed and filtrate ports, thus creating a series of chambers when the plates are held together. The sludge is pumped into the chambers allowing solids to build up in the filter, letting the filtrate to flow through the cloth and along the ribbed plate surface to their filtrate outlets. The press is fed under pressure until the set pressure is reached or until the filtrate flow drops below a minimum value. In the conventional plate filter press, after a press cycle, the feed ports are blown out with air and the plates are separated from each other, allowing cake to drop out of the bottom of the press. After discharge of all cakes, the press is closed again for the next cycle.99

![Figure 9-Plate filter press](image)

3.1.3 Belt filter press

Belt filter presses are commonly used to dewater sewage sludge. They consist of two endless filter cloths belts, which encapsulate the sludge. Drainage is achieved by moving the belts closer together and applying mechanical pressure to the sludge. This is done by passing the belts over a number of closely spaced rollers (Figure 10).

There are three distinct phases in the process:
- Pre-conditioning
- Preliminary drainage
- Secondary dewatering
3. TREATMENT PROCESS AND PLANT ASPECTS

In the pre-conditioning phase, the sludge is pumped into a mixer where it is conditioned by adding flocculants. The pre-conditioned sludge is then discharged into the preliminary drainage zone where the free water is drained by gravity through a porous belt. The sludge then passes to the secondary dewatering phase where it is compressed between the belts with increasing pressure.\(^8\)

\[
\text{Figure 10-Belt filter press.}^{41}
\]

3.1.4 Centrifuge

Centrifuge dewatering processes use centrifugal force to promote solid/liquid separation. The sludge is fed into a rotating basket assembly and accelerated. Solids are pulled out of the slurry by centrifugal force and are collected on the basket wall. The separated liquid flows out of the basket and is collected in the centrifuge housing, before being discharged (Figure 11).\(^8\)

\[
\text{Figure 11-Centrifuge.}^{42}
\]

Centrifugation represents one of the most used dewatering technologies. Current commercial centrifuges can reach up to around 30% of DS content when treating anaerobically digested municipal sewage sludge, but this value generally reduces to 23-24% for aerobically stabilised sludge, especially for sludge deriving from the combined treatment of industrial wastewater with domestic sewage.
3.1.5 Advantages and disadvantages of conventional dewatering techniques

The main advantages and disadvantages of conventional dewatering processes, including natural processes such as drying beds and drying lagoon, are shown in Table 2. Because of a large area requirement and odour potential, natural dewatering processes are applied at small WWTPs only.

<table>
<thead>
<tr>
<th>PROCESS</th>
<th>ADVANTAGES</th>
<th>DISADVANTAGES</th>
</tr>
</thead>
<tbody>
<tr>
<td>PLATE FILTER PRESS</td>
<td>• High cake solid concentration</td>
<td>• Batch operation</td>
</tr>
<tr>
<td></td>
<td>• Low suspended solids in filtrate</td>
<td>• High capital and labour costs</td>
</tr>
<tr>
<td></td>
<td>• Good dewatering process for hard-to-handle sludge</td>
<td>• Requires maintenance skilled personnel</td>
</tr>
<tr>
<td></td>
<td>• Plates can be added to increase capacity without significant increase in floor area</td>
<td>• Requires inorganic chemical conditioning that produces additional solids</td>
</tr>
<tr>
<td>BELT FILTER PRESS</td>
<td>• Relatively low capital, operating and power costs</td>
<td>• Very sensitive to feed sludge characteristics</td>
</tr>
<tr>
<td></td>
<td>• Easy to shut down the system</td>
<td>• Sensitive to polymer type and dosage rate</td>
</tr>
<tr>
<td></td>
<td>• Easy to maintain</td>
<td>• Requires large quantity of belt wash water</td>
</tr>
<tr>
<td>CENTRIFUGE</td>
<td>• Relatively low space required</td>
<td>• Relatively high capital cost</td>
</tr>
<tr>
<td></td>
<td>• Fast start up and shutdown capabilities</td>
<td>• High consuming of direct power per unit of product</td>
</tr>
<tr>
<td></td>
<td>• Does not require continuous operator monitoring</td>
<td>• Requires grit removal from feed sludge</td>
</tr>
<tr>
<td></td>
<td>• Clean appearance and good odour containment</td>
<td>• Requires periodic repair of scroll, resulting in long downtime</td>
</tr>
<tr>
<td>DRYING BEDS AND DRYING LAGOON</td>
<td>• Low capital cost when land is readily available</td>
<td>• Large area requirement</td>
</tr>
<tr>
<td></td>
<td>• Low energy consumption</td>
<td>• Requires sludge stabilization</td>
</tr>
<tr>
<td></td>
<td>• Low chemical consumption</td>
<td>• Design requires consideration of climatic effect</td>
</tr>
<tr>
<td></td>
<td>• Least operator attention and skill required</td>
<td>• Sludge cake removal is labour intensive</td>
</tr>
<tr>
<td></td>
<td></td>
<td>• Potential odour</td>
</tr>
</tbody>
</table>

The main drawback of mechanical dewatering is that the dewatering rate depends on the pore diameter in the sludge, i.e. on its hydraulic permeability: during the process, the layer of sludge near the filter medium is compressed, thus reducing the porosity in that layer and decreasing dewatering rate due to a blocking of the filter medium. As a result,
3. TREATMENT PROCESS AND PLANT ASPECTS

conventional dewatering techniques are not efficient methods to dewater sludge with low hydraulic permeabilities. In the case of centrifugation, dewatering depends more on sludge density and on the contents of colloidal matter. As volatile solids are lighter and colloidal, a lower content of volatile solids generally favours the separation efficiency in centrifuges. Therefore, centrifugation is a suitable dewatering method, but the purpose is to enhance the efficiency, increasing the DS matter of treated sludge. To do this, electro-dewatering is a promising technique that can justify the higher capital cost of the process with a greater dewatering.
3.2 Electrochemical dewatering: process principles

3.2.1 Electrokinetic phenomena

Six different physical processes are usually grouped under the term electrokinetic phenomena:

- **Electrophoresis** is the movement of charged particles relative to a stationary liquid.
- **Electromigration** is the transport of ions in solution with respect to the solution itself. Each species moves according to its own ionic mobility, which is a function of electrical charge, diffusion coefficient and temperature.
- **Electro-osmosis** takes place at the solid/liquid interface of the medium and induces a displacement of the bulk water molecules.
- **Sedimentation** (Dorn effect) occurs when dispersed particles move under the influence of either gravity or centrifugation in an electrolyte solution. This motion disrupts the equilibrium symmetry of the particle's double layer. While the particle moves, the ions in the electric double layer lag behind due to the liquid flow: this causes a slight displacement between the surface charge and the electric charge of the diffuse layer. As a result, the moving particle creates a dipole moment and the sum of all of the dipoles generates an electric field, which is called sedimentation potential. This situation is the opposite of electrophoresis as the particle movement creates the electric field.
- **Streaming** occurs when an electric field is created by an electrolyte solution being forced to flow (by applying a pressure) through a charged surface (e.g. the surface of a capillary tube or through a porous plug). The counter-ions from the diffuse layer are dragged away due to the hydraulic displacement of the liquid over the surface: the ion accumulation in the direction of the liquid flow generates a potential difference. This situation is the opposite of electro-osmosis.
- **Electrochemical reactions** at the electrodes guarantee the continuity of the electrical transportation. When an electric field is applied across electrodes in an aqueous solution, electrolysis of water occurs in order to maintain charge equilibrium. The electrolysis of water produces oxygen gas and protons, $\text{H}^+$, at
the anode, while hydrogen gas and hydroxyl anion, OH\(^{-}\), are formed at the cathode: as a result, the pH near the cathode increases while the pH near the anode decreases.

A brief description of the above-mentioned electrokinetic phenomena is given in Table 3. All electrokinetic phenomena are related to the development of the electrical double layer (EDL), at the particle/electrolyte interface.

<table>
<thead>
<tr>
<th>ELECTROKINETIC PHENOMENA</th>
<th>DRIVING FORCE</th>
<th>RESULTING PHENOMENA</th>
<th>MOVING PHASE</th>
<th>STATIONARY PHASE</th>
<th>QUANTITY MEASURED</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrophoresis</td>
<td>Electric field</td>
<td>Particles movement</td>
<td>Particles</td>
<td>Liquid</td>
<td>Electrophoretic mobility</td>
</tr>
<tr>
<td>Electromigration</td>
<td>Electric field</td>
<td>Ions transport</td>
<td>Ions/particles</td>
<td></td>
<td>Ionic mobility</td>
</tr>
<tr>
<td>Electro-osmosis</td>
<td>Electric field</td>
<td>Pressure gradient</td>
<td>Liquid</td>
<td>Plug or capillary</td>
<td>Electro-osmotic volume flow per unit current</td>
</tr>
<tr>
<td>Sedimentation potential</td>
<td>Particle movement</td>
<td>Electric field</td>
<td>Particles</td>
<td>Liquid</td>
<td>Potential difference per unit of length</td>
</tr>
<tr>
<td>Streaming potential</td>
<td>Pressure gradient</td>
<td>Electric field</td>
<td>Liquid</td>
<td>Plug or capillary</td>
<td>Streaming potential per unit of applied pressure</td>
</tr>
</tbody>
</table>

The most involved events in electro-dewatering are electro-osmosis, electrophoresis and electromigration. A description of these phenomena is given in the following chapters.

### 3.2.2 Electro-osmosis

Electro-osmosis balances the electrical and viscous forces in the double layer. Several theories have been proposed to explain and to predict electro-osmotic behaviour. The Helmholtz–Smoluchowski theory for the electro-osmotic rate in a capillary is one of the most widely followed. Electro-osmotic transport can be induced by an external electric field in capillary or porous plug systems where the electric polarization (charging) of solid surfaces occurs. Due to the fixed surface charge at the solid interface, an oppositely charged region of counterions develops in the liquid to maintain the electroneutrality of the solid/liquid interface: this screening region, as described in Chapter 2.6.3, is denoted as the electrical double layer (EDL).
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The flow rate, caused by electro-osmosis, is described by\(^{19}\):

\[
\frac{d\bar{V}}{dt} = q = \frac{\varepsilon_0 \varepsilon_r \zeta E A}{\eta}
\]

Where:
- \(\bar{V} \,[\text{m}^3]\): water volume
- \(t \,[\text{s}]\): time
- \(q \,[\text{m}^3/\text{s}]\): flow rate
- \(\varepsilon_0 \,[8.854 \times 10^{-12} \, \text{F/m}]\): dielectric permittivity of vacuum
- \(\varepsilon_r\): dielectric permittivity of the liquid
- \(\zeta \,[\text{V}]\): zeta potential of sludge
- \(E \,[\text{V/m}]\): electric field strength across the plug
- \(A \,[\text{m}^2]\): cross-sectional area
- \(\eta \,[\text{kg/ms}]\): viscosity of the liquid medium

During electro-dewatering, electro-osmosis is considered to play the most important role to gain extra water removal from sludge. The zeta potential of sludge usually have a negative value, therefore, the direction of electro-osmotic flow is from the anode to the cathode.

3.2.3 Electrophoresis

Electrophoresis refers to the movement of solid particles suspended in a liquid under the influence of an electric field. The solid particles acquire a surface charge either from their own intrinsic surface ionization such as in the case of clays or sludge, or by the adsorption of polyelectrolytes, surfactants or ions onto their surface. In the case of colloids, the surface area of the particles is large relative to the volume of the particles and therefore electrostatic repulsions dominate over the Van der Waals interaction. The electrostatic repulsion force arises from the electrical double layer, which prevents the particles coagulating or agglomerating. When an electric field is applied across a colloidal suspension, the charge on the particle (negative) interacts with the field and the particles move towards the electrode of opposite charge (anode).\(^{27}\)

\[
\nu_{ep} = \gamma \frac{\varepsilon_0 \varepsilon_r E \zeta}{\eta}
\]
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Where:
- \( v_{ep} \) [m/s]: electrophoresis velocity
- \( \varepsilon_0 \) [8.854 X 10\(^{-12}\) F/m]: dielectric permittivity of vacuum
- \( \varepsilon_r \): dielectric permittivity of the liquid
- \( \gamma \): coefficient for particle shape (for spherical particles \( \gamma = 2/3 \))

Barton et al.\(^{14}\) described the influence of electrophoresis on enhanced water removal. During the initial stages of electro-dewatering, the sludge particles are still free to move in the fluid suspension. Because of negative charge of particles, they tend to migrate towards the anode, thus delaying the onset of cake formation on the lower filter medium and hence leading to enhanced water flow. When DS content in sludge segment becomes higher, sludge particles will be locked in position and are hence unable to move, so only electro-osmosis plays an important role in transporting water from anode towards the cathode.\(^{19}\)

3.2.4 Electromigration

Transport by electromigration is only possible if the electroneutrality balance, which is imposed when a system is at equilibrium, is broken by the exchange of the electrons at the electrodes, as a result of electrochemical reactions. The electron transfer at the electrodes produces electromigration, which acts to restore electroneutrality. A general description of the flux of charged species in an electrochemical system is developed from the Nernst–Planck equation.\(^{27}\)

\[
J_j = u^*_j c_j \nabla (-E)
\]

With:
\[
u^*_j = \frac{D_j z_j F}{RT} \tau n
\]

Where:
- \( J_j \) [mol/m\(^2\)s]: flux of the species j
- \( u^*_j \) [m\(^2\)/Vs]: effective ionic mobility
- \( c_j \) [mol/m\(^3\)]: molar concentration of the species j
- \( E \) [V/m]: electrical potential gradient
- \( D_j \) [m\(^2\)/s]: diffusion coefficient of species j in the dilute solution
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- $z$: charge of chemical species
- $F$ [96,487 C/mol]: Faraday's constant
- $R$ [8.314 J/molK]: universal gas constant
- $T$ [K]: absolute temperature
- $\tau$: porosity of medium
- $n$: tortuous factor

During electro-dewatering, electromigration could occur, but the phenomenon that rules the removing of water from sludge is still electro-osmosis.
3.3 Electro-osmotic dewatering of sludge

Electro-osmotic dewatering is an attractive technique to dewater sludge since it can remove water trapped between fine solid particles when it cannot be removed by the application of pressure or vacuum. This is because electro-osmotic flow is based on the surface and colloidal characteristics of particles in suspension and is independent on pore size. As a result, the low hydraulic conductivity of sludge and the resultant blocking of filter medium do not significantly affect the dewatering rate when applying electro-osmotic dewatering. Therefore, it is an efficient method to dewater low permeability sludge having hydraulic conductivity values less than $10^{-6}$ cm/s. It is usually combined with mechanical methods to improve the rate and efficiency of dewatering.\(^{17}\)

In Figure 12 the processes occurring in sludge electro-dewatering are shown.

*Figure 12-Processes occurring in sludge electro-dewatering.\(^6\)*
3.3.1 Electro-mechanical dewatering process

The application of an electric field, combined with a pressure, tends to increase sludge dewaterability: the phenomenon of electro-osmosis rules the flow in charged particle matrix.

Electro-osmosis reduces the interstitial water and some extent of the vicinal water, thus resulting in a dryer sludge cake: it appears an energy efficient alternative solution to current technologies to bring sewage sludge from 20-25% up to 45-50% of dry-solid content.

Furthermore, electro-dewatering reduces the concentration of heavy metals in the sludge (by separating them in the osmotic process) and this will lead to a high fraction of the sludge that satisfies regulation and can hence be used as not pollutant fertilizer in agriculture.

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, floc size distribution, conditioning parameters, polyelectrolyte characteristics, temperature etc. A study of the influence of certain of these factors should indicate the optimum experimental conditions.

Although electro-mechanical dewatering is still not fully understood, the mechanical and electrical effects, resulting in the reduction of water content, are usually thought to satisfy the following steps\cite{17, 27, 45}:

- The mechanical dewatering reduces the volume of the pores and squeezes the water out of the product.
- The charged particles (usually negative) are still free to move in the fluid suspension. They tend to migrate towards the electrode carrying the opposite charge, where the filter medium is located.
- When the cake has formed, the particles are locked in their position and hence unable to move. Water containing ions is transported by viscous or molecular interactions and as a hydration sheath: it is transported through the porous medium by electro-osmosis.
- Electrochemical reactions at the electrodes are essential to restore charge equilibrium that enables electro-dewatering to proceed beyond the transient stage.
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- Finally, water ceases to be the continuous phase in the cake, and the electrical resistance rise leading to ohmic heating. The resulting reduction in viscosity facilitates the removal of some of the remaining water.

In principle, the achievement of complete water removal within the sludge is not possible since when the liquid stage in the sludge is no longer continuous because of dewatering, the sludge does not conduct electricity and then electro-osmosis ceases.8, 13

In Figure 13 the electrokinetic phenomena that rules pressure-driven electro-dewatering in a lab-scale device are shown.

![Diagram showing electrokinetic phenomena related to sludge electro-mechanical dewatering](image)

Figure 13-Electrokinetic phenomena related to sludge electro-mechanical dewatering.27
3.3.2 Electrochemical reactions effects

In typical electro-dewatering operations, the applied DC voltages are in the range of 5-30 V (some authors applied higher voltages but obtained too high resistive sludge cake). These systems are essentially electrochemical cells, where the application of an electric field produces electrochemical reactions, which affect the performance of the electro-dewatering process. The possible cathode and anode reactions are:

**CATHODE:**

- $2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2\text{(g)} + 2\text{OH}^- \quad \text{E}_0^\circ = -0.83 \text{ V} \quad (a)$
- $2\text{H}_3\text{O}^+ + 2\text{e}^- \rightarrow 2\text{H}_2\text{O} + \text{H}_2\text{(g)} \quad \text{E}_0^\circ = 0 \text{ V} \quad (b)$
- $\text{M}^{n+} + n\text{e}^- \rightarrow \text{M} \quad (c)$

Where $\text{M}^{n+}$ represents cationic species that can be reduced and $\text{E}_0^\circ$ is the standard electrode potential at a temperature of 298 K.

**ANODE:**

- $\text{M} \rightarrow \text{M}^{n+} + n\text{e}^- \quad (d)$
- $2\text{H}_2\text{O} \rightarrow \text{O}_2\text{(g)} + 4\text{H}^+ + 4\text{e}^- \quad \text{E}_0 = 1.23 \text{ V} \quad (e)$

These 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 (a) produces hydroxide ions and reaction (e) produces protons, this may result in a pH gradient across the filter cake. Tuan et al. 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.

Hydrogen and oxygen evolution at cathode and anode respectively leads to an increase in the electrical resistance of the system, with the formation of an electrically insulating layer. On the other hand, it may lead to an explosive mixture, causing a potential safety risk, which in batch devices is usually avoided by venting systems.

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...
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corrosion, thus the use of dimensionally stable anode materials is necessary. As reported in Table 5, the anodes used in experiments are 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.\textsuperscript{50} and Saveyn et al.\textsuperscript{51} documented that the use of titanium coated with mixed metal oxide (MMO) plates such as Ir\textsubscript{2}O\textsubscript{3}-coated titanium as anode prevents corrosion. Anodes made of conducting ceramic materials (e.g. iridium oxide or ruthenium oxide) coated on titanium seems 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.

The electric field used in electro-dewatering applications usually exceed the decomposition voltage of water, leading to a considerable ohmic loss that causes heating in the dewatering device and thus a decreasing liquid viscosity. This heating effect is more pronounced if the electro-dewatering operation takes more time.\textsuperscript{27}
3.3.3 Features of electro-osmotic dewatering

Yoshida\textsuperscript{13} has identified the main features of electro-osmotic dewatering as follows:\textsuperscript{8}:

- The filter medium is not damaged and blocked as in mechanical dewatering, so the effect of blocking on dewatering rate is very small
- The rate of dewatering and its efficiency are easily controlled by regulating the voltage applied to the sludge and electric current passing through the sludge
- Effective dewatering can be achieved in sludge which are not amenable to mechanical dewatering processes
- Electro-osmotic dewatering can be easily combined with mechanical methods, leading to additional improvement in the rate and efficiency of dewatering
- Corrosion of the electrodes as a result of electrolysis can occur, resulting in subsequent contamination of the sludge
- When the electric conductivity of the sludge is very large, electric power efficiency becomes low because of the generation of heat. When conductivity is very low, a very large voltage is required to drive the flow. Therefore, the application of electro-osmotic dewatering is restricted by the electrical properties of the sludge

\begin{table}[h]
\centering
\caption{Advantages and disadvantages of sludge electro-dewatering.}
\begin{tabular}{|l|l|}
\hline
\textbf{ADVANTAGES} & \textbf{DISADVANTAGES} \\
\hline
- High dewatering capacity for hard-to-dewater liquid & - Corrosion of electrodes due to electrochemical reaction and pH change \\
- Significant reduction in polyelectrolyte amount & - Possible Joule effect \\
- Lower energy requirement than filter press & - Possibility of explosions due to gases evolution \\
- Low mechanical support demand & \\
- Long life service & \\
- No damaging and blocking of filter medium & \\
\hline
\end{tabular}
\end{table}
3.3.4 Efficiency of electro-osmotic dewatering

According to many studies, an increase in electric field and pressure application results in an increase of sludge dewatering rate. The former improves electro-osmosis, while the latter, together with the squeezing of the sludge, let a better contact between the electrodes and the sludge itself. However, this kind of behaviour is hindered at voltages higher than a threshold value, in approximation around 40 V. A high electric field implies high currents, so faster electrolysis reactions: oxygen and hydrogen evolution (which are ruled by ATEX) are produced and pH near electrodes changes together with zeta potential. Removal of these gases is a critical aspect of the process, in order to avoid explosions during experiments. At the same time, sludge resistance becomes too high due to the lower water amount and, because of Joule effect, temperature raises too much.19, 25

According to Mahmoud et al.27, current increases with voltage in the early period and, after reaching a maximum, it decreases with time. Indeed, in the early period the electric resistance of the sludge bed decreases with the lowering of distance between anode and cathode with the proceeding of dewatering. Thereafter resistance increases under the influence of the unsaturated sludge layer formed in the dewatered sludge. On the other hand, oxygen and hydrogen evolution leads to the appearance of voids within the bed and increases the electrical resistance of the system: an electrically insulating layer is produced if the process takes much time. Finally, the electrical resistance of the cake formed is too high, the electrical current reduces and eventually ceases causing the interruption of electro-dewatering.27

These behaviours affect considerably dewatering efficiency and must be considered before designing the apparatus.

In general, power consumption is an important parameter in operating an electroosmotic dewatering system. Energy expenditure per unit weight of sludge processed can be formulated as52:

\[ E_u = \frac{P}{W} = \frac{1}{W} \int V I dt \]

Where:
- \( E_u \) [Wh/t]: energy consumption per unit weight of sludge
- \( P \) [Wh]: energy expenditure
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- W [t]: weight of sludge
- V [V]: voltage
- I [A]: current
- t [h]: processing time

As a rule of thumb, it can be considered that much less total energy is required for electro-dewatering of filter cakes that contain less liquid.\textsuperscript{53} Comparing power consumptions of sludge electro-dewatering with those of thermal drying, we should assess that the former involves lower costs, so electro-osmosis as a promising sludge dewatering process. The minimal drying energy requirement refers to the latent heat of water evaporation of about 0.617 kWh/kg, while in industrial devices it can reach 0.9 to 1.2 kWh/kg. However, the data on power consumption reported in the literature are difficult to compare and analyse: several authors employed the additionally electrical energy consumed per unit mass of the dry solids (kWh/kg\textsubscript{DS}) and therefore it is difficult to compare with the data reported for a drying process. Moreover, the electro-dewatering energy expressed as kWh/kg\textsubscript{DS} strongly depends on the initial cake dryness, which is different in each study.\textsuperscript{27}

One of the best ways to compare the power consumption is to calculate the energy per the additional mass of water removed (in comparison to dewatering without electric field). Figure 14 gives an overview of the different specific electric energy consumptions at different pressures and electric fields, in comparison with thermal drying consumptions: it was found that the electro-dewatering technique requires much less energy than thermal drying. These findings confirm that the application of a voltage is an interesting technique to enhance wastewater sludge dewatering.\textsuperscript{54}
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![Figure 14: Energy consumption of conditioned activated sludge as a function of applied voltage for three different pressures.](image)

However, one important consideration is that drying uses heat, electro-dewatering uses electricity. Therefore, the thermodynamic efficiency of the electrical network must be considered when comparing the energy consumption of the two methods. As the thermal efficiency of conventional power stations in Italy is 0.444\textsuperscript{55}, this means that a kWh of energy used in an electro-dewatering device should be compared with $1/0.444 = 2.25$ kWh of thermal energy, unless the electricity is produced from a photovoltaic device. Therefore, reaching industrial devices values from 0.9-1.2 kWh/kg, to be energetically efficient if compared to thermal drying, the electro-dewatering must consume less than $0.9 - 1.2 \times 0.444 = 0.4 - 0.5$ kWh/kg (removed water).

Figure 14 shows that electro-dewatering with applied voltages of up to 50 V and pressure of up to 1200 kPa consume less than 0.4 kWh/kg of water, making electro-dewatering always competitive with thermal drying.
3.4 Literature review of electro-osmotic dewatering of sludge

The earliest studies on electrokinetic dewatering made by Casagrande\textsuperscript{56} focused on the stabilization of soil. In the last decades, the use of electro-dewatering to remove water from different materials such as clay\textsuperscript{57, 58, 59, 60}, tomato paste suspension\textsuperscript{61}, food\textsuperscript{62}, vegetable sludge and sewage sludge started to be developed. The main difference between sludge and other materials is that sewage sludge is composed of organic matters, such as microorganisms and bacteria.

Most of the set-up for electro-dewatering studies have been performed with lab-scale devices: they vary in size, have horizontal or vertical set up, non-pressure, pressure, and vacuum application etc. Electro-dewatering is commonly carried out in cylindrical cell with pressure application: electro-osmosis is combined with conventional mechanical dewatering methods to improve the rate and efficiency of water removal. Electro-osmotic devices can be used in conjunction with conventional dewatering devices such as belt filter press\textsuperscript{16, 24} and pressure filter press.\textsuperscript{63}

Typically, direct electric field has been used in electro-dewatering process, but also interrupted DC electro-dewatering has been studied\textsuperscript{57, 58, 64, 65, 66}: the authors reported that a short interruption of power application enhances electro-osmotic flow. Yoshida tried the use of AC electric field with periodic reversals of electrode polarity.\textsuperscript{67} A constant voltage from 5 V to over 60 V has been used in most studies. Some researchers have reported electric field strength as V/cm in their studies.\textsuperscript{46, 51, 58, 59, 61, 68, 69, 70, 71}

Yoshida proposed also the multi-stage electrode dewatering method\textsuperscript{72} in which three perforated electrodes are vertically arranged at regular intervals within the sludge bed and are sequentially switched on. This method could improve the efficiency of dewatering and reduce power consumption and it has been further investigated in Vijn’s studies\textsuperscript{73} by the use of a “gate” electrode. This configuration can lead to a better control of both current and particles flux, thanks to the intermediate electrode activation, and to an increase of liquid capacity with respect to normal electro-dewatering.

Ho and Chen designed a lab scale device with a flat plate rotated anode\textsuperscript{59}. The anode was able to rotate at different speeds, improving contact between the electrode and sludge and increasing its conductivity to get a dryer cake. A higher rotating speed of the anode has been related to a decrease in water content of the cake, with maximum efficiency at 240 rpm.
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In some non-pressure electro-dewatering tests, electric field was applied directly to the original waste sludge or clay.\textsuperscript{57, 58, 24, 69} In other experiments, sludge was pre-dewatered by mechanical dewatering process, in which some of the water content was removed.\textsuperscript{46, 74} In pressure electro-dewatering, the process last from several minutes to several hours. An increase in the applied pressure resulted in a dryer sludge cake.\textsuperscript{25, 75} During electro-dewatering, sludge at the anode becomes dry: this may deteriorate the electrical contact between the sludge and the electrodes and the increasing in contact resistance causes a drop in the electric field. Thus, higher-pressure application improves the electrical contact leading to more efficient electro-osmotic flow and dryer sludge cake. Sludge electro-dewatering at different voltages has been presented in many studies.\textsuperscript{25, 75} Increase in voltage leads to higher water removal rates and dryer sludge cake. However, higher electric field application consumes more energy. Moreover, the influence of polyelectrolytes and polymers addition, in order to get a higher DS content, has been studied by different authors.\textsuperscript{70, 76, 77}

The use of filter cloth between electrodes and sludge cake to hinder the loss of solids has been investigated by Yu et al.\textsuperscript{71}: they suggested the use of a cathode mesh with small holes size instead of a polypropylene cloth, due to its water removal hindering. Citeau et al. studied the best arrangement of filter cloths and electrodes.\textsuperscript{78}

The energy consumption of electro-dewatering has been calculated in several studies. However, the power consumption of sludge electro-dewatering among the different papers is difficult to compare and analyse: it depends on the initial DS content of the sludge (it is expressed as kWh/kg\textsubscript{DS}). In order to get a comparable value, Gazbar et al.\textsuperscript{79} and Saveyn et al.\textsuperscript{51} calculated the energy per the additional amount of water removal in comparison to normal dewatering without electric field application (kWh/m\textsuperscript{3}): they showed that electro-dewatering consume from 46 to 445 kWh/m\textsuperscript{3} depending on voltage application. This power consumption was lower than conventional thermal evaporation methods, where the energy requirement was 617 kWh/m\textsuperscript{3} based on a theoretical calculation\textsuperscript{80} and up to 1200 kWh/m\textsuperscript{3} in an industrial application.\textsuperscript{79} On the other hand, one must consider that electricity requires heat to be generated, as it has been already shown in Chapter 3.3. According to Tuan et al.\textsuperscript{74}, high voltage applications are not economical from the perspective of energy consumption because the residual water content in the final sludge cake was independent on voltage from 10 to 30 V. However, higher voltages resulted in much shorter dewatering times. In this study, the application of 30 V consumed 150 kWh/m\textsuperscript{3}: energy consumption in this study, depending on the
applied voltage, was four to six times less than the theoretical thermal evaporation of water. Gingerich et al.\textsuperscript{81} showed that the energy consumption is not uniform during electro-dewatering process, but it increases with the increasing of cake DS content because the last water fractions become increasingly difficult to remove because of the high binding strength. Saveyn et al.\textsuperscript{51} highlighted a linear relationship between the energy consumption and the amount of additional water removal.

Gingerich et al.\textsuperscript{81} and Yuan and Weng\textsuperscript{46} made an economic analysis of the electro-dewatering process, taking into account of the additional energy costs and possible savings by transport and disposal, and came to the conclusion that electro-dewatering to intermediate moisture contents is the most cost-efficient.

In Table 5, some examples of studies about dewatering found in literature are shown.
### Table 5: Dewatering studies found in literature.

<table>
<thead>
<tr>
<th>Authors</th>
<th>Material</th>
<th>Initial dry solids</th>
<th>Apparatus</th>
<th>Electrodes</th>
<th>Parameters</th>
<th>Results</th>
<th>Dry solids</th>
</tr>
</thead>
<tbody>
<tr>
<td>Friehmelt et al.</td>
<td>Harbour sludge, sewage sludge, or industrial sludge</td>
<td>15% (h=14.4 mm)</td>
<td>Vertical cylinder</td>
<td>NA</td>
<td>6 V/cm DC</td>
<td>-</td>
<td>40%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>22% (h=7.2 mm)</td>
<td>Piston</td>
<td></td>
<td>IS(30/0.1)</td>
<td>0.25 bar</td>
<td>40%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>IS(30/0.05)</td>
<td></td>
<td>50%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>IS(30/2)</td>
<td></td>
<td>55%</td>
</tr>
<tr>
<td>1996 Gopalakrishan et al</td>
<td>Bentinite clay</td>
<td>10%</td>
<td>Vertical acrylic cylinder (Ø=5 cm)</td>
<td>NA</td>
<td>6 V/cm DC</td>
<td>-</td>
<td>420 s</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>IS(30/0.1)</td>
<td></td>
<td>60%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>IS(30/0.05)</td>
<td></td>
<td>50%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>IS(30/2)</td>
<td></td>
<td>70%</td>
</tr>
<tr>
<td>1997 Shang et al.</td>
<td>Phosphatic clay, sedimented by gravity</td>
<td>13.2%</td>
<td>Vertical plexiglass cylinder (Ø=9 cm, H=20 cm)</td>
<td>Stainless steel mesh electrodes</td>
<td>DC 16-10 V</td>
<td>24 h</td>
<td>25.7%</td>
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<td></td>
<td></td>
<td>19.5%</td>
<td></td>
<td></td>
<td>IS(15/16)</td>
<td></td>
<td>33.5%</td>
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<tr>
<td></td>
<td></td>
<td>15.7%</td>
<td></td>
<td></td>
<td>20-19.5 V</td>
<td></td>
<td>26.3%</td>
</tr>
<tr>
<td></td>
<td></td>
<td>15%</td>
<td></td>
<td></td>
<td>IS(15/20)</td>
<td></td>
<td>26.5%</td>
</tr>
<tr>
<td>2001 Lee et al.</td>
<td>Digested sludge</td>
<td>3%</td>
<td>Vertical teflon cylinder (Ø=7 cm, H=50 cm)</td>
<td>Anode: copper plate</td>
<td>80 V/cm</td>
<td>900 s</td>
<td>26%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Piston</td>
<td>Cathode: copper plate</td>
<td>80 V/cm</td>
<td></td>
<td>30%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>with 3 mm holes</td>
<td>0 V/cm</td>
<td></td>
<td>36%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td>15 V</td>
<td></td>
<td>22%</td>
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<td></td>
<td>30 V</td>
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<td>29%</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3.924 bar</td>
<td></td>
<td>37%</td>
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<td></td>
<td></td>
<td></td>
<td>3.924 bar</td>
<td></td>
<td>38%</td>
</tr>
<tr>
<td>2001 Ho</td>
<td>Bentonite slurry</td>
<td>9.1%</td>
<td>Vertical cylinder (Ø=9 cm, H=25 cm)</td>
<td>Stainless steel plates coated with Sn and Sb oxides</td>
<td>6 V/cm</td>
<td>240 rpm</td>
<td>14.4%</td>
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<tr>
<td></td>
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<td>Piston</td>
<td>Cathode: circular stainless steel plate with 775 holes of 1.5 mm</td>
<td>0 V</td>
<td>ND</td>
<td>23%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>15 V</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>30 V</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.05 bar</td>
<td>Feed: 1.8 m³/h</td>
<td>17%</td>
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<tr>
<td></td>
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<td></td>
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<td>19.5%</td>
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<td></td>
<td></td>
<td></td>
<td>20.5%</td>
</tr>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Feed: 2 m³/h</td>
<td>24%</td>
</tr>
<tr>
<td>2002 Raats</td>
<td>Flocculated WMO-Al sludge</td>
<td>5%</td>
<td>Gravity-driven thickening belt</td>
<td>Stainless steel (or titanium coated with iridium oxide)</td>
<td>30 V</td>
<td>800 s</td>
<td>47%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Conventional belt press (filter belt W=1 m)</td>
<td></td>
<td>0 V</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
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<td></td>
<td>15 V</td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.05 bar</td>
<td></td>
<td></td>
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<tr>
<td>2002 Raats</td>
<td>WMO iron-ion-coagulated sludge, flocculated and pre-dewatered gravimetically</td>
<td>15%</td>
<td>Vertical PVC cylinder (Ø=5.5 cm, H=20 cm)</td>
<td>Stainless steel electrodes</td>
<td>30 V</td>
<td>1000 s</td>
<td>65%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Piston</td>
<td></td>
<td>2.5 bar</td>
<td></td>
<td></td>
</tr>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>2003 Yuan et al.</td>
<td>Belt pressed mixed sludge</td>
<td>15.9%</td>
<td>Horizontal acrylic cylindrical cells (Ø=4 cm): Sludge specimen chamber (L=8 cm)</td>
<td>Graphite rod electrodes</td>
<td>2.5 V/cm</td>
<td>-</td>
<td>16.1%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Cathode reservoir (L=4 cm)</td>
<td></td>
<td>2.5 V/cm</td>
<td></td>
<td>23.1%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5 V/cm</td>
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<td>26.2%</td>
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<tr>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>5 V/cm</td>
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<td>33.3%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5 V/cm</td>
<td></td>
<td>37.4%</td>
</tr>
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</table>
### 3. TREATMENT PROCESS AND PLANT ASPECTS

<table>
<thead>
<tr>
<th>Authors</th>
<th>Material</th>
<th>Initial dry solids</th>
<th>Apparatus</th>
<th>Electrodes</th>
<th>Parameters</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>2003 Weber et al</td>
<td>Quartz sand SF800</td>
<td>NA</td>
<td>Filter press: Filter plates (47 cm x 47 cm) Filter chamber (H=16 cm)</td>
<td>Outer electrodes 0 V 120 V Middle electrode 60 V 120 V</td>
<td>4 bar</td>
<td>1500 s 17 dm³ 20 dm³</td>
</tr>
<tr>
<td></td>
<td>Amberger kaolin</td>
<td>NA</td>
<td>Filter press: Filter plates (47 cm x 47 cm) Filter chamber (H=16 cm)</td>
<td>Outer electrodes 0 V 80 V Middle electrode 60 V 120 V</td>
<td>4 bar</td>
<td>1200 s 390 cm³ 435 cm³</td>
</tr>
<tr>
<td>2004 Al Asheh et al</td>
<td>Tomato paste suspension</td>
<td>5 %</td>
<td>Vertical acrylic cylinder (Ø=9.1 cm, H=25 cm)</td>
<td>Circular stainless steel mesh (AISI 316) discs</td>
<td>100 mA</td>
<td>9000 s 23.5%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>9000 s 20 % 30% 42%</td>
</tr>
</tbody>
</table>
| 2005 Yang et al  | Pet food industry wastewater sludge           | 3-7%               | Horizontal PVC cell (L=14 cm, W=10 cm, H=10 cm) | Anode: graphite sheet 
Cathode: stainless steel mesh | H= 4 cm 2.5 V/cm 5 V/cm 7.5 V/cm H= 6 cm 3.3 V/cm 5 V/cm H= 8 cm 2.5 V/cm 3.8 V/cm | 3 h 42.7% 44.6% 48.4% |
|                  |                                               |                    | Vertical cylinder (Ø=15 cm, H=22 cm) | Anode: graphite sheet 
Cathode: porous stainless steel | IS(60/30) 60 V + IS(10/30) 30 V + 40 V | - 39.3% 36.4% 38.9% |
| 2005 Saveyn et al | Thickened activated, conditioned sludge      | 3.63%              | Vertical plexiglass cylinder (Ø=7 cm) Piston  | Stainless steel electrodes 0 V/cm + 25 V/cm | 4 bar      | 1800 s 1800 s 40%      |
| 2006 Reddy et al | Indiana harbour sediments No polymer         | 21.4%              | Vertical acrylic cylinder | Graphite electrodes 0 V/cm 1 V/cm 1 V/cm 1 V/cm | Gravity | 145 h 145 h 25 % 115 h 290 h 35% 30 h 290 h 22% |
|                  | Polymers: Polyacrylic acid: 0.5% 1% 2%       |                    |                                               |                                           |            | 211 h 32% 210 h 29%    |
|                  | Polymacrylamide: 0.5% 1% 2%                  |                    |                                               |                                           |            | 212 h 42% 212 h 11%    |
| 2006 Shin et al  | Sludge without electrocoagulation             | 2.4%               | Vertical teflon cylinder (Ø=7 cm, H=50 cm) Piston  | Anode: nickel plate Cathode: nickel plate with 3 mm holes | 0 V/cm 3.924 bar 40 V/cm 80 V/cm 120 V/cm | 1500 s 24% 34% 37% 45% |
|                  | Sludge with electrocoagulation                | 4.2%               | Vertical teflon cylinder (Ø=7 cm, H=50 cm) Piston  | Anode: nickel plate Cathode: nickel plate with 3 mm holes | 0 V/cm 3.924 bar 40 V/cm 80 V/cm 120 V/cm | 1500 s 24% 34% 37% 45% |
| 2006 Larue et al | Gelatinous bentonite suspension               | 8.5%               | Vertical polypropylene cylinder (Ø=5.2 cm, Ø=10 cm) Piston (Ø=5.1 cm) Filter base (Ø=13.6 cm) with a cylindrical cavity (1 cm) | Stainless steel electrodes 100 mA 10 bar | 15300 s 35% 35% 37% 45% | |
### 3. Treatment Process and Plant Aspects

<table>
<thead>
<tr>
<th>Authors</th>
<th>Material</th>
<th>Initial dry solids</th>
<th>Apparatus</th>
<th>Electrodes</th>
<th>Parameters</th>
<th>Results</th>
<th>Dry solids</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>2007 Glendinning et al</strong></td>
<td>Digested, conditioned sludge</td>
<td>13.9% 14.8%</td>
<td>Vertical perspex cylinder Piston</td>
<td>Copper discs</td>
<td>1 V/cm 0.5 bar 21 days</td>
<td>27% 23%</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Activated, thickened, conditioned sludge</td>
<td>7.6%</td>
<td>Belt press</td>
<td>Woven polyester electrified with carbon fibre strips</td>
<td>15 V bar 0.25 bar 1200 s</td>
<td>10.28% 15.89% 12.36%</td>
<td></td>
</tr>
</tbody>
</table>
| **2008 Tuan et al** | Primary and secondary mixture, pre-dewatered sludge + alkalinity (NaHCO₃) | 8.6%               | Vertical acrylic cell (H=55 cm, W=17 cm, L=31 cm): 10 cm gravel bed and 10 cm sand bed | Anode: titanium plate  
Cathode: stainless steel mesh | 15 V bar | 16.3%    |
|                  | Anaerobically digested, pre-dewatered sludge                             | 7.3%               | Vertical polyvinyl chloride cylinder (Ø=9.8 cm, L=30 cm) | Anode: titanium plate  
Cathode: stainless steel mesh | 4 V bar 3 V 5 V 10 V 15 V 30 V | 17.7%    |
|                  | Primary and secondary mixture, pre-dewatered sludge + alkalinity (NaHCO₃) | 8.3%               | Vertical acrylic cell (H=55 cm, W=17 cm, L=31 cm): 10 cm gravel bed and 10 cm sand bed | Anode: titanium plate  
Cathode: stainless steel mesh | 15 V | ND       |
|                  | Activated, thickened, conditioned and centrifuged sludge                 | 7.5%               | Vertical acrylic cell (H=55 cm, W=17 cm, L=31 cm): 10 cm gravel bed and 10 cm sand bed | Anode: titanium plate  
Cathode: stainless steel mesh | 15 V | 40.6%    |
|                  | Anaerobically digested, pre-dewatered in a natural sand-drying bed sludge | 6.9%               | Vertical polyvinyl chloride cylinder (Ø=9.8 cm, L=30 cm) | Anode: titanium plate  
Cathode: stainless steel mesh | 4 V bar | 40.2%    |
| **2010 Yu et al** | Activated, thickened, conditioned and centrifuged sludge                 | 21%                | Vertical acrylic cylindrical cell (Ø=7 cm) | Stainless steel electrodes (no filter cloth) | 16 V/cm 20 V/cm 24 V/cm 0.07 bar 300 s | 36% 38% 40% |
| **2010 Tuan et al** | Anaerobically digested, pre-dewatered (in a natural sand-drying bed) sludge | 7.8%               | Vertical acrylic cell (H=55 cm, W=17 cm, L=31 cm): 10 cm gravel bed and 10 cm sand bed | Anode: titanium plate  
Cathode: stainless steel mesh | 0 V 3 V 5 V 10 V 15 V 30 V | 14% 25% 38% 41% 41% 39% |
| **2010 Tuan et al** | Anaerobically digested, pre-dewatered (in a natural sand-drying bed) sludge | 7.8%               | Vertical polyvinyl chloride cylinder (Ø=9.8 cm, L=30 cm) | Anode: titanium plate  
Cathode: stainless steel mesh | 40 V DC IS(45/15) IS(30/30) IS(15/45) | 39% 39% 39% 39% |
| **2011 Citeau et al** | Activated and conditioned sludge                                         | 8.3%               | Horizontal cylinder (V=69.5 cm³) Peristaltic pump | Titanium coated by ruthenium discs (DSA) | 80 A/m² | 5 bar 30000 s | 45-53%    |
## 3. Treatment Process and Plant Aspects

<table>
<thead>
<tr>
<th>Authors</th>
<th>Material</th>
<th>Initial dry solids</th>
<th>Apparatus</th>
<th>Electrodes</th>
<th>Parameters</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>2011 Mahmoud et al.</td>
<td>Activated, conditioned sludge</td>
<td>2.8%</td>
<td>Vertical teflon cylinder (Ø=7 cm, H=14.5 cm)</td>
<td>Anode: titanium coated MMO (DSA) Cathode: Titan</td>
<td>0 V + 50 V 4 bar 2 h+2 h</td>
<td>11-13% + 56%</td>
</tr>
<tr>
<td>2012 Citeau et al.</td>
<td>Activated and conditioned sludge</td>
<td>9.1%</td>
<td>Horizontal polypropylene cylinder (V=69.5 cm³)</td>
<td>Anode: titanium coated MMO (AO2023) disc Cathode: stainless steel disc</td>
<td>0 V + 20 V 20 A/m² 50 V 0 V + 50 V 2 bar 2 h+2 h</td>
<td>16.8% 25.3% 26.8% 28.8%</td>
</tr>
<tr>
<td></td>
<td>Digested and conditioned sludge</td>
<td>10.3%</td>
<td>Peristaltic pump</td>
<td></td>
<td>0 V + 20 V 20 A/m² 50 V 0 V + 50 V 6 bar 2 h+2 h</td>
<td>23.3% 26.8% 25.3% 26%</td>
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<tr>
<td>2013 Weng et al.</td>
<td>Activated sludge, conditioned</td>
<td>31.1%</td>
<td>Horizontal acrylic cylindrical cells (Ø=4 cm): Sludge specimen chamber (L=6 cm) Cathode reservoir (L=4 cm)</td>
<td>Graphite rod electrodes</td>
<td>1.25 V/cm 2.5 V/cm 5 V/cm</td>
<td>- 4 h 36.4% 42.6% 51.2%</td>
</tr>
<tr>
<td>2013 Loginov et al.</td>
<td>Bentonite drilling sludge</td>
<td>23.4%</td>
<td>Horizontal polypropylene cylinder (cross-section=26 cm²)</td>
<td>NA</td>
<td>A) 0 A/m² 40 A/m² 80 A/m² 0.05 bar ND</td>
<td>66.4% 65% 66% 66%</td>
</tr>
<tr>
<td></td>
<td>Conditioning with CaO: A) 0 g/l</td>
<td></td>
<td></td>
<td></td>
<td>B) 0 A/m² 40 A/m² 80 A/m² 20 V 30 V 50 V</td>
<td>67% 66.4% 67.2%</td>
</tr>
<tr>
<td></td>
<td>B) 0.3 g/l</td>
<td></td>
<td></td>
<td></td>
<td>C) 0 A/m² 40 A/m² 80 A/m² 20 V 30 V 50 V</td>
<td>66% 66.2% 66.2%</td>
</tr>
<tr>
<td></td>
<td>C) 0.5 g/l</td>
<td></td>
<td></td>
<td></td>
<td>D) 0 A/m² 40 A/m² 80 A/m² 20 V 30 V 50 V</td>
<td>64.2% 64.2% 64.2%</td>
</tr>
<tr>
<td>2014 Feng et al.</td>
<td>Activated, conditioned sludge</td>
<td>6%</td>
<td>Vertical plexiglass cylinder (Ø=7 cm, V=2 L) Teflon piston</td>
<td>Titanium coated mixed metal oxide (DSA) electrodes</td>
<td>0 V + 10 V 0 V + 30 V 0 V + 50 V 2 bar 2 h+2 h</td>
<td>11% 13% 20%</td>
</tr>
<tr>
<td>2014 Olivier et al.</td>
<td>Activated, conditioned sludge</td>
<td>2.5-3.2%</td>
<td>Vertical teflon cylinder (Ø=7 cm, H=14.5 cm) Teflon piston</td>
<td>Anode: titanium MMO (DSA) Cathode: Titan</td>
<td>0 V + 30 V 4 bar 2 h+2 h</td>
<td>11-13% + 56%</td>
</tr>
<tr>
<td></td>
<td>Anaerobically digested, conditioned sludge</td>
<td>2.5-3.2%</td>
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<td></td>
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<td>15-18% + 63%</td>
</tr>
</tbody>
</table>
3.5 Industrial applications of sludge electro-osmosis dewatering

Among the patents related to electro-osmotic dewatering, like U.S. 5034111 (December 27, 1989), the energy consumption is considered too high compared to the benefits in term of increased DS content, for a commercial use.

However, some commercial full-scale equipment are currently on the market, such as CINETIK™ Linear Electro-Dewatering (Eimco Water Technologies, Canada), ELODE® electro-osmosis dehydrator (ACE Korea Incorporation), EDW (Water Technologies of Australia), and Electrokinetic (Electrokinetic Limited, UK).

Elode-SELO (patent: WO 2008/140175 A1) is a compact second stage dehydrator which uses electro-osmosis to further dehydrate the treated sludge cake from conventional dehydrators, in order to obtain an higher dryness and a significant reduction in sludge weight. Sludge is transported on belts and run through rollers, which press the sludge reducing moisture content. It can be integrated into any of the existing conventional dewatering machines such as belt-press, filter press, or centrifuge.\(^86\)

The sludge shall be treated to or above 5% dry solids by conventional thickening devices before it can be treated by SELO.

Elode-BELO is a dehydrator that combined first stage of mechanical dehydration by belt press and second stage by electro-osmosis method. This equipment brings the sludge cake to much higher dryness, reducing the DS content to 60% (at the dehydration ratio of 40%). It also reduces volume of the sludge to or less than 10% of input liquid sludge or at least 50% compared to conventional dehydrator.

BELO is a compact and robust machine, suitable for plants run by less skilled personnel, where low noise and ease of operation, flexibility and low maintenance are critical.\(^86\) However, this kind of machines are not particularly efficient because sludge dries at the anode and as soon as its dry content increases, its conductivity decreases and it acts as a dielectric, preventing further drying of the sludge layers that is behind it, which is not in contact with the anode surface. Part of the sludge dries much more slowly and remains wet, reducing the overall efficiency of the process. Furthermore, operators must pay attention on the possibility of noxious vapours (ammonia, toxic organic compounds) outflow that can arise from the driest sludge cake that is in contact with the anode.
Elcotech’s Cinetik™ is a batch press, which extracts water from sludge upon the combined action of a controlled mechanical pressure and electro-osmosis, where an electric field generated inside the sludge liberates and carries away the water bonded to hydrophilic particles.

As shown in Figure 16 Elcotech’s CINETIK™ incorporates the electro-dewatering process in a metallic structure (2) housing a linear filter belt (4) arrangement which moves the sludge cake along multiple serviceable power blocks (3) that apply pressure and DC current via modular rectifiers (1) to the sludge, hence dewatering it. When applied to a sludge of about 10-15% dryness, the CINETIK™ process can yield dryness levels of 30 to 50% or greater.

However, this process is long and time consuming because only a limited and predetermined quantity of sludge can be treated in each cycle: there are various interruptions of the dehydrating process, which enables only a small quantity of sludge material to be treated per unit of time. The efficiency of this process is not ideal since it
3. TREATMENT PROCESS AND PLANT ASPECTS

produces dehydrated sludge with a percentage of dry matter which is still low (around 30-33%), because its ineffective mixing decreases sludge conductivity.
Electrokinetic geosynthetic (EKG) belt filter presses have increased the solids contents of sewage cake from 20% to 31% and diamond mine tailings from 62% to 75%.88

In Table 6 cost savings referred to EKG belt press with respect conventional belt press are shown.

![Figure 17-Electrokinetic geosynthetic belt press.](image)

<table>
<thead>
<tr>
<th></th>
<th>Conventional belt press</th>
<th>EKG belt press</th>
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<tbody>
<tr>
<td>Loading dry solids [kg/h]</td>
<td>540</td>
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<tr>
<td>Operating hours</td>
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<td>Cake dry solids [%]</td>
<td>19</td>
<td>31</td>
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<tr>
<td>Disposal cost [€/m³]</td>
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<tr>
<td>Disposal cost [€/year]</td>
<td>483,510</td>
<td>296,070</td>
</tr>
<tr>
<td>EKG saving per machine [€/year]</td>
<td>-</td>
<td>187,440</td>
</tr>
</tbody>
</table>
4. MATERIALS AND METHODS

4.1 Device assembling: static piston

The first part of my work has been devoted to the assembly of a lab-scale device able to produce both a mechanical pressure and an electric field. The use of both these systems could be efficient to increase the amount of dry solids in the final sludge.

The assembling of the apparatus has been a crucial phase of my thesis. We tried to get a low economical effect on the project during this stage (being only the preliminary part) and, at the same time, our aim has been the building of a device similar to those used in literature. In order to get this, we collected different parts and materials and, with appropriate design and manufacturing, we assembled our equipment.

This device, as illustrated in Figure 18, consists of:

- Cylindrical glass vessel (h=176 mm, Ø=80 mm)
- Cooling water-jacket
- Compressed air system (1-4.5 bar)
- Double effect cylinder (200 mm stroke) SMC-CP96 (piston)
- DC power supply (30 V-5 A)
- Anode: DSA Ti (MMO), fixed to the piston
- Cathode: stainless steel mesh (AISI 304)
- Cloth: PTT (polytrimethyleneterephthalate)
Starting from a glass cell provided with a cooling water jacket, with height of 176 mm and internal diameter of 80 mm (Figure 19), we designed and manufactured a Teflon cathode support (Figure 20) with holes (diameter of 5 mm) distributed randomly. Thanks to its shape, the Teflon frame is able to bear pressures of 1-4.5 bar and at the same time let the flow of water that has to be collected. One of the main problems encountered with this cell configuration is the sealing of the apparatus, since in some experiments lack of water influenced the results.

The presence of a cooling water jacket is fundamental in order to keep low the value of temperature of sludge during the process: a too high Joule effect is detrimental for electrodes and for the process itself.
In order to avoid loss of sludge solids due to pressure application, a filter cloth is used: we choose a PTT (polytrimethylene terephthalate optical image in Figure 21), able to let water flow through and hold the solids. The best apparatus configuration would be that with the filter cloth put between the cathode and the Teflon frame, as it would keep a good conductivity between the electrodes in lab-scale experiments. However, the first experiments have shown that electro-osmosis becomes inefficient and temperature raises too much because the electrodes get in direct contact, as a wide-spaced mesh at the cathode would allow pressurised sludge to flow under the cathode itself. In order to prevent this situation, we tried to put PTT filter cloth between sludge and cathode: the obtained results with pressure and electric field application have shown comparable values with those found in literature. However, as shown by Mahmoud et al.\textsuperscript{89} and by Yu et al.\textsuperscript{71}, the presence of filter cloth (especially for polypropylene ones) between the cathode and the sludge cake is detrimental for energy consumption, due to its high resistivity, and dewatering rate. To
4. MATERIALS AND METHODS

To overcome these problems, it could be advisable to select low thickness filter cloth manufactured with electrically conductive material or to use a cathode mesh with small pore size without a cloth.

![Figure 21: Picture obtained by optical microscope of PTT filter cloth.](image1)

We fastened a movable piston (SMC-CP96 double effect cylinder, Ø=32 mm) on the upper part of the cylindrical vessel and then screwed a Teflon disc, able to resist at high temperatures, on the piston itself in order to press the sludge (Figure 22).

![Figure 22: SMC-CP96 double effect cylinder with Teflon disc.](image2)

The upper electrode is a dimensionally stable anode DSA® (manufactured by Industrie De Nora) made of titanium coated with mixed metal oxide (MMO) and 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. Then, the piston is connected to the laboratory pressurisation system, with a manometer able to regulate incoming pressure values (Figure 23).
4. MATERIALS AND METHODS

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

![Glass cell with piston, DC power supply, precision scale and thermocouple.](image1)

In order to control temperature during dewatering tests, a thermocouple (Data logger thermometer OMEGA-HH306A, Figure 24) is inserted into the glass cell.

![Data Logger thermometer OMEGA-HH306A.](image2)
4.2 Device assembling: dynamic piston

In order to improve dewatering of sludge, an important step has been the introduction of a rotating anode. Unlike the static piston, we tried to improve the conductivity of the sludge cake during electro-osmosis by introducing a movement in the double effect cylinder: the dry cake crust close to the anode is scratched and mixed by the piston and dewatering slightly improves.

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 (Figure 25).

Moreover, a modification of the Teflon support has been done fastening a plastic circular frame on it in order to make the rotation of the anode in the middle of the glass cell (Figure 26).

*Figure 25-Rotating piston with copper pipe electrical connection and Teflon cylinders for the mixing of sludge.*
4. MATERIALS AND METHODS

Figure 26 - Cathode frame for the rotating piston apparatus.

In Figure 27 the entire sludge electro-dewatering apparatus is shown.

Figure 27 - Rotating piston electro-dewatering apparatus.
4.3 Sludge samples

Sludge samples were taken from San Rocco (Milan) WWTP with a capacity of 4 m³/s, corresponding to 1.050.000 population equivalent. We used 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 (made of 150 plates).

The main characteristics and concentrations of micro and trace metals of sludge samples from Milano San Rocco WWTP are shown in Table 7.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Sludge after aerobic digestion</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>7.78 - 8.1</td>
</tr>
<tr>
<td>Conductivity [mS/cm]</td>
<td>0.2 - 1.7</td>
</tr>
<tr>
<td>Total dry solids at 105 °C [%]</td>
<td>20.7 - 25.2</td>
</tr>
<tr>
<td>Volatile solids at 600 °C [%]</td>
<td>6.5 - 7.4</td>
</tr>
<tr>
<td>Humidification degree [%]</td>
<td>26.1 - 30.3</td>
</tr>
<tr>
<td>Extractable organic carbon [%DS]</td>
<td>29 - 31.2</td>
</tr>
<tr>
<td>Total nitrogen [%DS]</td>
<td>4.2 - 6.8</td>
</tr>
<tr>
<td>Total phosphorous [%DS]</td>
<td>0.5 - 2.1</td>
</tr>
<tr>
<td>Potassium [g/kgDS]</td>
<td>2.0 - 4.6</td>
</tr>
<tr>
<td>Arsenic [mg/kgDS]</td>
<td>&lt;0.1 - 4.4</td>
</tr>
<tr>
<td>Cadmium [mg/kgDS]</td>
<td>&lt;0.6 - 3.9</td>
</tr>
<tr>
<td>Chromium [mg/kgDS]</td>
<td>10.2 - 124</td>
</tr>
<tr>
<td>Hexavalent chromium [mg/kgDS]</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Mercury [mg/kgDS]</td>
<td>&lt;1 - 2</td>
</tr>
<tr>
<td>Nickel [mg/kgDS]</td>
<td>6.9 - 79.6</td>
</tr>
<tr>
<td>Lead [mg/kgDS]</td>
<td>28.4 - 99.7</td>
</tr>
<tr>
<td>Copper [mg/kgDS]</td>
<td>76.9 – 443</td>
</tr>
<tr>
<td>Sodium [mg/kgDS]</td>
<td>552 – 2183</td>
</tr>
<tr>
<td>Zinc [mg/kgDS]</td>
<td>78.6 – 605</td>
</tr>
<tr>
<td>Calcium as CaO [%DS]</td>
<td>2.4 - 4.5</td>
</tr>
<tr>
<td>Sulphur as SO₃ [%DS]</td>
<td>2.1 - 2.5</td>
</tr>
</tbody>
</table>
4. Materials and Methods

Each sludge sample dewatered by filter press is crumbled and homogenised by a shredder (Moulinex-La Moulinette, Figure 28), which lead to a uniform cake to be treated. In Figure 29 are shown pictures of sludge before (a,b) and after the shredding by Moulinex (c,d).

![Figure 28-Moulinex-La Moulinette.](image)

![Figure 29-Sludge before (a,b) and after (c,d) shredding by Moulinex.](image)

In order to control the sludge quality, pH and electrical conductivity are measured by a pH-meter Metrohm 827 pH Lab) and a conductivity meter (B&C Electronics-C 125.2 conductivity meter).
4. Materials and Methods

![Figure 30: pH-meter Metrohm 827 pH Lab and B&C Electronics-C 125.2 conductivity meter.](image)

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. In Table 8 all types of sludge used for our electro-osmosis tests are shown.

<table>
<thead>
<tr>
<th>Sludge</th>
<th>Mech. dewatering</th>
<th>DS [%]</th>
<th>VS [%]</th>
<th>pH</th>
<th>Conductivity [µS/cm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Bucher</td>
<td>20.1</td>
<td>70.1</td>
<td>5.5</td>
<td>N.A.</td>
</tr>
<tr>
<td>B</td>
<td>Filter press</td>
<td>27.9</td>
<td>72.7</td>
<td>6.3</td>
<td>N.A.</td>
</tr>
<tr>
<td>C</td>
<td>Filter press</td>
<td>22.9</td>
<td>71.3</td>
<td>7.2</td>
<td>1524</td>
</tr>
<tr>
<td>D</td>
<td>Filter press</td>
<td>26.8</td>
<td>69.1</td>
<td>7.3</td>
<td>1154</td>
</tr>
<tr>
<td>E</td>
<td>Filter press</td>
<td>22.2</td>
<td>70.9</td>
<td>5.9</td>
<td>1313</td>
</tr>
<tr>
<td>F</td>
<td>Filter press</td>
<td>23.6</td>
<td>73.2</td>
<td>6.0</td>
<td>1132</td>
</tr>
<tr>
<td>G</td>
<td>Filter press</td>
<td>21.9</td>
<td>75.9</td>
<td>5.5</td>
<td>1277</td>
</tr>
<tr>
<td>C_I</td>
<td>Filter press + hydration</td>
<td>20.0</td>
<td>N.A.</td>
<td>7.2</td>
<td>1455</td>
</tr>
<tr>
<td>D_I</td>
<td>Filter press + hydration</td>
<td>19.4</td>
<td>N.A.</td>
<td>7.1</td>
<td>1025</td>
</tr>
<tr>
<td>D_{II}</td>
<td>Filter press + hydration</td>
<td>21.3</td>
<td>N.A.</td>
<td>7.0</td>
<td>1453</td>
</tr>
</tbody>
</table>

Differently from most of the lab-scale electro-osmosis tests found in literature, we treated types of sludge with an initial DS higher than 20%: this fact would be an important parameter when energy consumption and dewatering rate will be computed. Low initial water amount, indeed, is the main cause of low conductivity of the samples and a decrease in electrical field efficiency. Therefore, our aim is to find the behaviour of dry samples under electro-osmosis and understand if the results can be comparable with those found for more hydrated types of sludge.
4. MATERIALS AND METHODS

4.4 Materials for electrodes

The evolution of oxygen during sludge dewatering leads to high positive potentials combined with an increase in the acidity leading to very aggressive conditions for the anode material. Hence, most materials are in their anodic dissolution or transpassive region. These conditions greatly restricted the selection of suitable materials that could be used for our purposes.

We started by using DSA®, provided by Industrie De Nora, in order to compare our results with those found in literature. These anodes 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). The presence of metals such as titanium, iridium and platinum implies a high cost of production, which is one of the critical aspects for their use on an industrial scale.

Our experiments confirmed that these kind of anodes are the best in terms of corrosion resistance during electro-osmosis tests. In Figure 31, the DSA before and after electro-osmosis tests is shown (after electro-dewatering sludge particles remain attached to the surface, but there is no corrosion).

Figure 31 - DSA anode before (a) and after (b) electro-osmosis tests (10 hours).

However, our task has been also the replacement of DSA by the use of cheaper anodes, good in terms of corrosion resistance, wear resistance and with a suitable efficiency (final DS amount).

From an economical point of view, the stainless steel family was the best candidate to be tested as a substrate of anodes in electro-osmosis. We chose AISI 304 stainless steel, which has the best quality/price ratio for our applications.
4. MATERIALS AND METHODS

However, stainless steel alone is not able to bear the conditions developed during the tests: acidic environment, evolution of oxygen, presence of organic matter are detrimental for the stainless steel, which encounter pitting, and crevice corrosion.

In order to increase the durability of the anodes, we studied appropriate coatings, deposited by PVD technique by Flubetech, with the aim of lowering cost production and keeping the same efficiency of DSA.

The coatings proposed by Flubetech have been TiN, AlTiN, DLC (diamond like carbon), which are usually exploited for their wear and abrasion resistance but thanks to their relative high thickness, could be also considered as a barrier in the electro-osmotic environment. However, the main parameters that must be investigated are the porosity of the coatings, which is detrimental for corrosion resistance, and the conductivity of the anode.

In Table 9 the coatings proposed by Flubetech are reported.

<table>
<thead>
<tr>
<th></th>
<th>TiN</th>
<th>AlTiN</th>
<th>DLC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Layer Thickness [µm]</td>
<td>1-4</td>
<td>2-4</td>
<td>2-4</td>
</tr>
<tr>
<td>Hardness [HV]</td>
<td>2500</td>
<td>3200</td>
<td>2700</td>
</tr>
<tr>
<td>Friction coefficient (*)</td>
<td>0.4</td>
<td>0.5</td>
<td>0.1</td>
</tr>
<tr>
<td>Oxidation Temp. [º C]</td>
<td>450</td>
<td>1100</td>
<td>400</td>
</tr>
<tr>
<td>Colour</td>
<td>Golden</td>
<td>Black violet</td>
<td>Black anthracite</td>
</tr>
</tbody>
</table>

(*) Tested against hardened steel at 25ºC and 85% humidity.
4. MATERIALS AND METHODS

4.5 Experimental set up and operations

In literature, both constant current (C.C.) and constant voltage (C.V.) modes are used for electro-osmosis tests. However, for C.C. mode the electric resistance of cake and voltage increase, causing a temperature rise by ohmic effect, leading to the stopping of the process in order to protect the filter cloth and filter cell. In C.V. mode, there is a constant increase of the cake electric resistance and a decrease of electric current intensity. Therefore, the process can be extended to the end. However, for the C.V. mode at high voltages (V>30 V), heating was not controlled because of current intensity peak in the earlier period. Moreover, the power expenditure of a constant current operation is expected to be greater than the constant voltage process according to Weng et al. Starting from the idea of using low potential values, in order to hinder the possibility of high ohmic heating and obtain low energy consumptions, we choose to work with the usual C.V. mode.

Following a procedure similar to that used by Mahmoud et al., the electro-dewatering procedure consists of two successive stages:

- filtration/compression with a pressure applied: 5 min are sufficient to reach the equilibrium phase because of previous mechanical treatment;
- electric field at the selected operating voltage with a pressure applied: the end of this dewatering stage is automatically detected when no more than two drops of filtrate are collected in 5 min.

As a first step, the glass cell is filled with homogenized sludge: 1 cm of thickness, usually corresponding to 35 g of matter (this value depends on the initial DS amount), for static piston experiments. Then the cell is closed by the cover and the piston starts applying pressure on the sludge. In our device configuration, sludge is pressed between the upper anode (on the PTFE support) and the lower PTT filter cloth (placed on the cathode mesh). After 5 minutes of pressure, where no water has been extracted due to the mechanical pre-treatment, electric field is switched on and values of currents in function of time are recorded. At the same time, every minutes extracted water weight and temperature of the cell are registered.
4. MATERIALS AND METHODS

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 in an oven and weighing (*Metodi di analisi del compost*).

Each experiment is repeated three times (when possible) because of the variability of the initial sludge samples, in order to find a mean value and behaviour.

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.
4.6 Analysis methods: characterization of sludge

In order to obtain the highest DS content in the sludge with our electro-dewatering device, the most important parameters useful to characterize the sample are dry matter, volatile solid content, pH and conductivity. These analyses have been performed both before and after electro-dewatering and they highlighted the influence of these properties on the process and how they changed with water removal.¹

**Dry solid content**
300-400 g of fresh sample are weighed and put in a preheated oven at 105 °C for 12-24 hours, then they are cooled down at room temperature in a drier for 10-15 minutes and weighed. This procedure is repeated until a constant weight is reached. The dry solid content is expressed by the following formula:

\[
DS \left( \frac{g}{kg} \right) = \frac{a - b}{c} \times 1000
\]

\[
DS \, (\%) = \frac{a - b}{c} \times 100
\]

Where:
- DS = dry solid content
- a = weight of container and sample after drying
- b = weight of container
- c = weight of the fresh sample

**Volatile solid content**
A container is filled with 10 g of sludge sample, previously dried at 40 °C and milled. The container is put in a muffle furnace at 550 °C for 2 hours, and then cooled down in a drier since room temperature is reached and then weighed. Later the sample is dried in the oven at 105 °C for 4-6 hours, cooled down at room temperature in the drier and weighed. This procedure is repeated until a constant weight is reached (with approximation of 0.001 g). The container with the dried sample is put in the muffle furnace at 550 °C and incineration is stopped when a constant weight is obtained (6-8 hours). After cooling down in the dryer, the ashes are weighed.
4. MATERIALS AND METHODS

The volatile solid content is expressed by the following formula:

\[
VS \left( \frac{g}{kg} \right) = \frac{a - b}{a - T} \times 1000
\]

\[
VS(\%) = \frac{a - b}{a - T} \times 100
\]

Where:
- \( VS \) = volatile solid content
- \( a \) = weight of container and sample dried at 105 °C before incineration
- \( b \) = weight of container and sample after incineration
- \( T \) = tare of container
- From the VS value, ashes content (AC) can be obtained:
- \( AC(\%DS) = 100 - VS(\%) \)

\[ \text{pH} \]

10 g of sludge sample are put in a beaker (V=250 ml) where 100 ml of distilled water are added. The mixture, after a mixing with a stirrer for 30 minutes, is left at rest for other 30 minutes and then is filtered. The extracted liquid undergoes to pH measurement with pH-meter.

\[ \text{Conductivity and salinity} \]

10 g of sludge sample are put in a beaker (V=250 ml) where 100 ml of distilled water are added. The mixture, after a mixing with a stirrer for 30 minutes, is left at rest for other 30 minutes and then is vacuum filtered. The extracted liquid undergoes to a measurement by conductivity meter.
4. MATERIALS AND METHODS

4.7 Analysis methods: total energy consumption

In order to compute the total energy consumption obtained with the electro-osmosis experiments, we had to keep in mind the values of time spent, the currents developed during the tests, the imposed potential and the mass of water collected.

The average current (A) is computed by adding together all the values of electric charge (Ah), calculated multiplying current and time (h) pointwise during the test, and dividing it for the total time.

Then, 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).

Finally, the total energy consumption (Wh/kg) is computed by dividing energy by mass of water collected (kg).

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(extracted water). These results have been achieved with potentials from 10 V to 50 V and times higher than 3 hours. However, treating small amounts of sludge with an initial DS higher than 22%, liquid-solid bond energy is higher and specific energy to remove water is greater. We chose to set our energy consumption threshold at a value of 0.250 kWh/kg(extracted water) in order to have a saving with respect thermal treatment at the same initial conditions.
5. RESULTS AND DISCUSSION

5.1 Static piston electro-osmosis tests

Our first 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.

As a first step, we set the initial cake thickness at 1 cm, in order to have a not too much insulating layer during electro-osmosis: high cake thickness, indeed, leads to low currents due to strong resistance.

Moreover, we chose to fix the pressure at 3 bar: this value, according to literature, is sufficient to maintain the contact between sludge and electrodes. However, the pressure by itself is not sufficient to remove water from sludge, due to the initial high DS amount (DS_i) reached after pre-dehydration in Milano San Rocco depurator.

Therefore, the use of an electric field is necessary to reach DS in the range of 40-45%, which is the value we predetermined to obtain with our apparatus, also according to results of scientific papers in literature.

Collecting values of current density, time and final DS, we will observe a specific trend: increasing the electric field (from 10 V/cm to 20 V/cm), we will not have a higher final DS amount but we will obtain a reduction in times. This kind of behaviour is confirmed by literature\(^\text{19}\): 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.

Moreover, as all experiment are interrupted when no more than one drop is collected in 5 minutes, it must be considered that often time at which 90% of water is collected are more comparable and similar if the initial condition are the same.

Another important consideration must be done: the final DS amount is computed by the weighing after the 24 h in the oven, because the amount of filtered liquid collected during the process is not reliable. The filtrate can contain sludge particles (pH≈11) and, in addition, during 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
5. RESULTS AND DISCUSSION

decreasing liquid viscosity enhancing dewatering kinetics. This heating effect is usually more pronounced if the electro-dewatering operation takes more time and if the electric field is high.\textsuperscript{54}

\[ H \propto I^2 \times R \times t \]

Where:
- \( H \) = amount of heat [J]
- \( I \) = current [A]
- \( R \) = resistance [\( \Omega \)]
- \( t \) = time [s]

Moreover, at higher values of initial DS and 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.

Looking also at the results found in literature\textsuperscript{54}, 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.
5. RESULTS AND DISCUSSION

5.1.1 Sludge from Bucher press

In Table 10 the values of tests time, total energy consumption and final DS (DSf) amount for a sludge from Bucher press are shown.

All the electro-osmosis tests with sludge A (DSi=20.1%), have been performed starting from 1 cm of sludge thickness (45 g) and by applying 3 bar of pressure.

Table 10: Electro-osmosis results of A sludge (DSi=20.1%).

<table>
<thead>
<tr>
<th>N°</th>
<th>tTOT [min]</th>
<th>Tot energy consumption [Wh/kgH2O]</th>
<th>DSf [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>E=10 V/cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>A-1</td>
<td>105</td>
<td>181.9</td>
<td>38.6</td>
<td>18.5</td>
</tr>
<tr>
<td>A-2</td>
<td>81</td>
<td>172.9</td>
<td>38.1</td>
<td>18.0</td>
</tr>
<tr>
<td>A-3</td>
<td>65</td>
<td>151.2</td>
<td>39.9</td>
<td>19.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E=15 V/cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>A-4</td>
<td>55</td>
<td>265.3</td>
<td>39.6</td>
<td>19.5</td>
</tr>
<tr>
<td>A-5</td>
<td>65</td>
<td>263.8</td>
<td>38.8</td>
<td>18.7</td>
</tr>
<tr>
<td>A-6</td>
<td>61</td>
<td>269.9</td>
<td>41.4</td>
<td>21.3</td>
</tr>
<tr>
<td></td>
<td></td>
<td>E=20 V/cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>A-7</td>
<td>45</td>
<td>360.4</td>
<td>38.5</td>
<td>18.4</td>
</tr>
<tr>
<td>A-8</td>
<td>52</td>
<td>327.4</td>
<td>42.5</td>
<td>22.4</td>
</tr>
</tbody>
</table>

Here we can see that DS, after electro-osmosis, increases from 20.1% to 38-42.5%, independently from the electric field value set during the tests. Instead, an evident change of processing times can be seen: they are shorter in tests at 20 V/cm, with respect to cases at 10-15 V/cm, due to a higher kinetics. It must be reminded that time is not taken as an arbitrary parameter, but depends on the last drop time (if there are no drops in five minutes the test is interrupted). 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 final DS amount. Power consumption should have a maximum value of 250-300 Wh/kg of water removed in order to be considered a powerful application in this field, so the cases at 20 V/cm exceed this threshold.

Looking at plots of current density vs time in Figure 32, the first drop time (marked by the X) and the slope of the curves are similar with equal processing parameters (especially at 15 V/cm and 20 V/cm). This result, together with the values of DS amount, let us to say that the test reproducibility is not perfect but appreciable. It must be considered the
5. RESULTS AND DISCUSSION

fact that characteristics of the sludge change quickly with small variation of temperature and humidity, and the granulometry is not completely the same in the tests. According to Mahmoud et al., the standard deviation on the final dry solids content of the dewatered cake is around ±3%\(^a\), so our results can be considered reliable. This situation will be shown further in the following paragraph, where also the higher or lower influence of Joule effect (and consequently a different water evaporation) changes the result in terms of DS but also in terms of reproducibility.
5. RESULTS AND DISCUSSION

Figure 32 - Current density vs time diagrams at (a) 10 V/cm, (b) 15 V/cm and (c) 20 V/cm of A sludge.
5. RESULTS AND DISCUSSION

Often the current density, by switching the electric field on, increases until it reaches a maximum value, then it lowers until it reaches a plateau. The current peak is the result of two opposing effects. As water is removed, the bed height decreases and this decreases the bed resistance but, at the same time, the percent of solids increases, thus increasing the bed resistance. Moreover, since cracks appeared in the upper part of the bed, electrical contact between the bed and the anode is progressively disrupted with dewatering. Oxygen and hydrogen evolution at both electrodes leads to the appearance of void spaces within the bed and increases the electrical resistance of the system. This may produce an electrically insulating layer, if the electro-dewatering operation takes more time. On the other hand, increasing of the electrical resistance causes a drop in the electric field, and thus in the driving force, inside the cake. Near the end of the tests, the electrical resistance of the cake becomes too high, the electrical current reduces and eventually ceases and electro-dewatering stops and no more water is removed.83

As a first account, higher the electric field higher is the maximum current density and the slope of the curve changes due to a different kinetics. This result is shown in Figure 33 where one test with the best result in terms of final DS for each case is depicted.

The filtrate pH resulted to be around 11 in all the tests, due to electrolysis reaction at the anode and the cathode, with the production of H⁺ and OH⁻ respectively.
5. RESULTS AND DISCUSSION

5.1.2 Sludge from filter press

Starting from the results obtained by electro-osmosis of sludge A, we proceeded by testing sludge from filter press. The different technique of mechanical dehydration leads to a different granulometry of the sludge, so the use of Moulinex in order to homogenize the cake has been necessary.

Except from test B-5, where the mass of sludge put inside the glass cell has been maintained at 45 g (like the previous tests), we decided to lower the weight value to 35 g in order to keep constant the thickness of the initial cake (1 cm) because of the different particles size after filter press and Moulinex treatment.

Table 11 shows the results of tests on sludge B (DS=27.9%).

<table>
<thead>
<tr>
<th>N°</th>
<th>t_{TOT} [min]</th>
<th>Tot energy consumption [Wh/kgH2O]</th>
<th>DS_f [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>B-1</td>
<td>55</td>
<td>329.8</td>
<td>33.7</td>
<td>5.8</td>
</tr>
<tr>
<td>B-2</td>
<td>65</td>
<td>265.5</td>
<td>34.8</td>
<td>6.9</td>
</tr>
<tr>
<td>B-3</td>
<td>55</td>
<td>250.5</td>
<td>35.8</td>
<td>7.9</td>
</tr>
<tr>
<td></td>
<td><strong>E=10 V/cm</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B-4</td>
<td>75</td>
<td>432.8</td>
<td>38.4</td>
<td>10.5</td>
</tr>
<tr>
<td></td>
<td><strong>E=15 V/cm</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>B-5*</td>
<td>35</td>
<td>466.8</td>
<td>35.8</td>
<td>7.9</td>
</tr>
<tr>
<td>B-6</td>
<td>35</td>
<td>599.6</td>
<td>33.8</td>
<td>5.9</td>
</tr>
<tr>
<td>B-7</td>
<td>55</td>
<td>556.2</td>
<td>37.5</td>
<td>9.6</td>
</tr>
<tr>
<td></td>
<td><strong>E=20 V/cm</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*different mass of sludge with respect other cases (45 g)

The most important factor that must be underlined here is that, starting from a high value of DS, conductivity lowers too much and electric field is not efficient to remove water: final DS amount are low and test times are not comparable.

The reproducibility, as shown in current density vs time curves (Figure 34), is not good in this case due to an inefficient dewatering. The influence of granulometry and the different path of water inside the cake in each test could be an explanation: being the sludge too dry, water hardly find its own path and when it succeeds, times are different from experiment to experiment, even at the same conditions. Moreover, the non-uniformity of the current distributions caused by the heterogeneous local changes in bed
5. RESULTS AND DISCUSSION

conductivity is one of the reasons that explains the variation in the dewatering kinetics for the same sample with the same operating conditions. Looking at the total energy consumption, we understand that the process of electro-osmosis with a too much dry sludge is not useful, since costs become too high and final DS are not optimal.

As stated above, the first water drop time and the values of current density are not equal also with the same initial conditions. To confirm this point, we can also look at plots of B-5 and B-7, which are similar but have different initial conditions in terms of initial mass of the sludge (45 g and 35 g respectively). Therefore, the high value of initial DS tends to
5. RESULTS AND DISCUSSION

give unsatisfying results: delay in the first collected drop, high energy consumptions and low dewatering.

Figure 35 shows slopes which at different potentials have different maximum current density value, but the previous considerations about higher kinetics at higher potentials is not still valid here: first drop at 15 V came later than the test at 10 V. These results confirm that high DS, are not suitable for electro-osmosis tests.

Figure 35-Comparison of current density vs time diagrams at 10 V/cm, 15 V/cm and 20 V/cm of B sludge.
5. Results and Discussion

In Table 12 we can see that the final DS is increased with respect to the previous tests, where the starting sludge was drier: in this case, the DS$_i$ is 22.9% and the final result is about 39% on average.

Table 12-Electro-osmosis results of C sludge (DS$_i$=22.9%).

<table>
<thead>
<tr>
<th>N°</th>
<th>t$_{TOT}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H2O}$]</th>
<th>DS$_f$ [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>E=15 V/cm</td>
<td></td>
</tr>
<tr>
<td>C-1</td>
<td>35</td>
<td>274.5</td>
<td>40.2</td>
<td>17.3</td>
</tr>
<tr>
<td>C-2</td>
<td>50</td>
<td>354.7</td>
<td>38.3</td>
<td>15.4</td>
</tr>
<tr>
<td>C-3</td>
<td>37</td>
<td>296.5</td>
<td>37.1</td>
<td>14.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>E=20 V/cm</td>
<td></td>
</tr>
<tr>
<td>C-4</td>
<td>37</td>
<td>426.0</td>
<td>40.0</td>
<td>17.1</td>
</tr>
</tbody>
</table>

Values of energy consumption are lower than for B sludge thanks to a much higher presence of water in the starting cake: conductivity and efficiency of the process increase. A test at 20 V of potential has been performed to compare the current densities developed during the process and the power consumption with respect C-1, C-2 and C-3 tests. As stated previously, final DS are pretty the same for tests at 15 V/cm and 20 V/cm, but the total energy consumption lead to the choice of not considering it as suitable.

As shown in Figure 36, the most important difference from tests on drier sludge is that the first drop is collected in short times (at 15 V/cm: under 5 minutes from the application of electric field with respect to minute 18 for B sludge) so an increase in the rate of the process means a large efficiency.
5. RESULTS AND DISCUSSION

As stated above, a comparison of the slope of the two curves in Figure 37 show the different kinetics in test at 15 V/cm and 20 V/cm.
5. RESULTS AND DISCUSSION

As shown in Table 13, it is clear that the reproducibility of sludge D (DS$_i$=26.8%) is not good, like with B sludge experiments: the final DS concentration ranges from 35.1% to 42.7%. As the cited case, the sludge is too dry to have a suitable electro-osmosis efficiency: energy consumption are too high with respect the threshold of 250 Wh/kg, also for tests at 15 V/cm.

Table 13-Electro-osmosis results of D sludge (DS$_i$=26.8%).

<table>
<thead>
<tr>
<th>N°</th>
<th>$t_{TOT}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H_2O}$]</th>
<th>DS$_f$ [%]</th>
<th>$\Delta$DS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>E=10 V/cm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D-1</td>
<td>52</td>
<td>312.8</td>
<td>35.1</td>
<td>8.3</td>
</tr>
<tr>
<td>D-2</td>
<td>50</td>
<td>267.1</td>
<td>37.8</td>
<td>11.0</td>
</tr>
<tr>
<td>D-3</td>
<td>47</td>
<td>315.7</td>
<td>35.5</td>
<td>8.7</td>
</tr>
<tr>
<td></td>
<td>E=15 V/cm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D-4</td>
<td>30</td>
<td>364.6</td>
<td>37.0</td>
<td>10.2</td>
</tr>
<tr>
<td>D-5</td>
<td>35</td>
<td>378.6</td>
<td>38.0</td>
<td>11.2</td>
</tr>
<tr>
<td>D-6</td>
<td>35</td>
<td>434.9</td>
<td>36.0</td>
<td>9.2</td>
</tr>
<tr>
<td></td>
<td>E=20 V/cm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>D-7</td>
<td>35</td>
<td>597.3</td>
<td>36.9</td>
<td>10.1</td>
</tr>
<tr>
<td>D-8</td>
<td>26</td>
<td>501.3</td>
<td>39.2</td>
<td>12.4</td>
</tr>
<tr>
<td>D-9</td>
<td>35</td>
<td>466.6</td>
<td>42.7</td>
<td>15.9</td>
</tr>
</tbody>
</table>

In Figure 38 we can see that the slopes are similar at the same electric field value, but the first drop time has a five minutes range and this is clearly due to a low reproducibility of sludge with high DS amount.
5. RESULTS AND DISCUSSION

*Figure 38* - Current density vs time diagrams at (a) 10 V/cm, (b) 15 V/cm and (c) 20 V/cm of D sludge.
5. RESULTS AND DISCUSSION

In Figure 39 it is clear that kinetics of electro-osmosis processes at different voltages changes, like in the previous tests.

Figure 39-Comparison of current density vs time diagrams at 10 V/cm, 15 V/cm and 20 V/cm of D sludge.
5. RESULTS AND DISCUSSION

Starting from sludge E (DS$_i$=22.2%), reducing the processing time of the tests with respect to the previous ones (by waiting only 2 minutes from the last drop), we obtained a low final DS amount together with a low energy consumption. This fact highlights the importance of a good compromise between time processing and final DS results (see Table 14).

<table>
<thead>
<tr>
<th>N°</th>
<th>$t_{TOT}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H_2O}$]</th>
<th>DS$_f$ [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>E-1</td>
<td>35</td>
<td>176.4</td>
<td>34.4</td>
<td>12.2</td>
</tr>
<tr>
<td>E-2</td>
<td>30</td>
<td>285.9</td>
<td>35.2</td>
<td>13</td>
</tr>
<tr>
<td>E-3</td>
<td>20</td>
<td>379.3</td>
<td>36.9</td>
<td>14.7</td>
</tr>
</tbody>
</table>

These results highlighted that tests at 20 V/cm have a too strong energy consumption, which make them not suitable for an industrial application. Figure 40 shows the values of current densities during the experiments at the three applied electric fields.

![Figure 40-Comparison of current density vs time diagrams at 10 V/cm, 15 V/cm and 20 V/cm of E sludge.](image)
5. RESULTS AND DISCUSSION

5.1.3 Hydrated sludge

In order to simulate electro-osmosis tests of less dry types of sludge, we decided to add a suitable amount of distilled water to sludge dehydrated by filter press: our aim was the comparison of the results in terms of final DS and energy consumptions.

Starting from sludge C (DSi=22.9%), by simple computation, we added water (14.5 ml) to 100 g of sludge in order to reach a 20% of initial DS. After mixing and stirring, we put the solution at rest for one day and then used it for electro-osmosis experiments. Electro-osmosis test results of 35 g (1 cm) of sludge under 15 V of potential are shown in Table 15.

<table>
<thead>
<tr>
<th>N°</th>
<th>tTOT [min]</th>
<th>Tot energy consumption [Wh/kg,H2O]</th>
<th>DSf[%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>C1-1</td>
<td>37</td>
<td>260.5</td>
<td>39.2</td>
<td>19.2</td>
</tr>
<tr>
<td>C1-2</td>
<td>37</td>
<td>249.7</td>
<td>41.0</td>
<td>21.0</td>
</tr>
</tbody>
</table>

We can see how, starting from a wetter cake, removing water by electro-osmosis is easier. However, this fact could be ascribed to a weak “bond” between water and sludge particles (this is the water that usually is collected by mechanical dewatering in depurator). It must also be highlighted the decrease in conductivity from C to C1, from 1524 µS/cm to 1455 µS/cm, due to a less presence of ions in the solution.

Plotting the results on a current density vs time diagram (Figure 41), we can see that reproducibility is almost perfect: first drop time are similar (≈3-4 minutes) and the slopes are superimposed. Moreover, even though sludge conductivity is lowered by adding distilled water, maximum current densities for sludge C and C1 are similar (>35 mA/cm²)
5. RESULTS AND DISCUSSION

Figure 41: Current density vs time diagrams at 15 V/cm of Cl sludge.
5. RESULTS AND DISCUSSION

Afterwards, we decided to use the same procedure to sludge D (DS$_i$=26.8%): we added 34 ml and 24.7 ml to 100 g of sludge, with the aim of obtaining 20% and 23% of initial DS respectively. However, the real initial DS values measured after the 24 h in the oven, have been 19.4% and 21.3% respectively: humidity and lack of good homogenization are the parameters that influence more the hydration process.

In Table 16 and Table 17, results of dewatering tests at 15 V of the two sludge samples are shown. Final DS is high in comparison to all the previous tests (40-42% for D$_i$ and 42-46% for D$_{II}$) and total energy consumption is good in terms of saving costs (under 250 Wh/kg most of the time).

<table>
<thead>
<tr>
<th>N°</th>
<th>t$_{TOT}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H2O}$]</th>
<th>DS$_f$ [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>E=15 V/cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D$_{I}$-1</td>
<td>33</td>
<td>201.5</td>
<td>42.1</td>
<td>22.7</td>
</tr>
<tr>
<td>D$_{I}$-2</td>
<td>27</td>
<td>185.7</td>
<td>40.4</td>
<td>21.0</td>
</tr>
<tr>
<td>D$_{I}$-3</td>
<td>28</td>
<td>199.1</td>
<td>41.1</td>
<td>21.7</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>N°</th>
<th>t$_{TOT}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H2O}$]</th>
<th>DS$_f$ [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>E=15 V/cm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>D$_{II}$-1</td>
<td>37</td>
<td>259.7</td>
<td>42.2</td>
<td>20.9</td>
</tr>
<tr>
<td>D$_{II}$-2</td>
<td>40</td>
<td>227.6</td>
<td>46.0</td>
<td>24.7</td>
</tr>
<tr>
<td>D$_{II}$-3</td>
<td>34</td>
<td>209.9</td>
<td>43.6</td>
<td>22.3</td>
</tr>
</tbody>
</table>

We can deduce that a high presence of water inside the cake is favourable for a good dewatering, even though a less presence of ions is the cause of a lowering in conductivity (1025 μS/cm for D$_{I}$).

In Figure 42 we can see the current density vs time plots.
5. RESULTS AND DISCUSSION

![Current density vs time diagrams at 15 V/cm of (a) D_I and (b) D_II sludge.](image)

The slopes are nearly superimposed and times of first drop are similar. Like the experiments at 15 V of C_I, reproducibility is high and efficiency is more than acceptable for both D_I and D_II. As stated previously, this behaviour could be ascribed to the different type of water in sludge: adding water, also letting the slurry stand for one day, could not be sufficient to make it as hydrated water and this fact increases dewaterability.
5. RESULTS AND DISCUSSION

5.1.4 Dewatering rate

Summarising the dewatering results of the studied sludge samples, we can assess that a model behaviour is hard to be deduced due to the many parameters that condition the electro-osmosis process. Conductivity of sludge, particles size, water evaporation and initial DS amount (DSi) are the main responsible of a good or bad dewatering at the same values of potential, pressure, cake thickness and temperature.

However, as we have shown in the tests above, we can state that, in a low range of electric field (10 V/cm, 15 V/cm and 20 V/cm), final dry matter amount has often similar values, but dewatering rate increases with the potential. This behaviour is expressed by a higher amount of water in the first minutes for stronger electric fields, together with a shorter duration of tests.

In this paragraph, we show how a change in the sludge types can condition the dewatering rate (expressed in g_extracted water/min). We wish to recall that the values shown in the following diagrams are not perfectly correct due to the influence of higher or lower evaporation from test to test (dependent on the parameters cited above) and also due to the stuck of water in the outflowing path (filter cloth absorption, water retaining in the pipe and on the Teflon cathode support).

In Figure 43 dewatering rate diagrams of tests A-3, A-6 and A-8 are shown. Sludge A (DSi=20.1%) was pre-dehydrated by Bucher and its DS results after electro-osmosis have been in the range 38.1%-42.5%.

![Figure 43-Dewatering rate vs time diagrams of A sludge (DSi=20.1%).](image)
5. RESULTS AND DISCUSSION

As expected, we can see that higher is the potential, greater is the amount of water removed in the first 10 minutes of electro-osmosis process. Moreover, A-8 test is also the shorter one because all the extractable filtrate is collected in the first minutes. Tests at 15 V and 10 V, instead, have a smoother peak, with a lower slope because times widen lowering the potential value: at larger times (30 min), the rate for A-8 test is the highest because small amount of liquid has been removed until that moment.

In order to study dewatering rates of types of sludge with a higher value of DS_i, we can see in Figure 44 and Figure 45 a comparison of sludge C (DS_i=22.9%) and sludge E (DS_i=22.2%).

![Figure 44-Dewatering rate vs time diagrams of C sludge (DS_i=22.9%).](image1)

![Figure 45-Dewatering rate vs time diagrams of E sludge (DS_i=22.2%).](image2)
5. RESULTS AND DISCUSSION

Dewatering rates for sludge C are in agreement with the previous considerations: at higher potential, the dewatering rate is greater in the first 10 minutes. However, even though the final DS amount is the same, C-1 slope does not widen as expected (it has a sharper peak than the test at 20 V). This fact could be an evidence that a higher evaporation or a greater stuck of water have been achieved in this test.

Conversely, the diagrams for sludge E follow the expected behaviour: slopes sharpen increasing the potential and test E-1, performed at 10 V, have no water removed in the first 10 minutes. However, test E-3 should have a greater rate at 10 min and this could be due to a stronger evaporation.

Comparing diagrams for sludge B (DS\textsubscript{i}=27.9%) and D (DS\textsubscript{i}=26.8%), as shown in Figure 46 and Figure 47, we can see that the studied behaviour is observed also for dry types of sludge.

![Figure 46-Dewatering rate vs time diagrams of B sludge (DS\textsubscript{i}=27.9%).](image-url)
5. results and discussion

For sludge B times are broadened and dewatering rates are lower with respect other tests: the filtrate that can be extracted is obviously less starting from a great amount of dry matter in the sludge.

Sludge D, instead, has slopes similar to those previously shown for other tests.

In Figure 48 dewatering rate diagrams of hydrated sludge C and D, C (DS\textsubscript{i}=20%), D\textsubscript{i} (DS\textsubscript{i}=19.4\%) and D\textsubscript{II} (DS\textsubscript{i}=21.3\%), are shown. All these tests have been performed at 15 V and have the same slope. The only difference are the rate values that approximately follow the final DS results: D\textsubscript{II} has a greater value at 10 min but lower in the second part of the test with respect D\textsubscript{i}.
5. RESULTS AND DISCUSSION

In conclusion, dewatering rate vs time diagrams highlight how DS_i, conductivity and particles size of sludge affect electro-osmosis process. They condition duration of tests, first drop time and amount of removed water in a strong way.
5. RESULTS AND DISCUSSION

5.1.5 Energy consumptions

As stated before, total energy consumptions for electro-osmosis tests performed at 10 V, 15 V and 20 V are dependent on the amount of water removed, applied potential, times of the processes and on the developed current values. According to this behaviour, we evidenced that higher the potential, higher is the power consumption if the water amount is the same.

Figure 49 shows average values of energy consumptions at 10 V, 15 V and 20 V for sludge A, B, C, D and E.

![Figure 49: Average energy consumptions for sludge A (DS=20.1%), B (DS=27.9%), C (DS=22.9%), D (DS=26.8%) and E (DS=22.2%) at 10 V, 15 V and 20 V.](image)

The increase of energies is almost linear from 10 V to 20 V and the slope of the curves are almost the same for all the sludge types.

As expected, dry samples, such as B and D, have the greatest energy consumption due to a lower amount of water that can be removed, while A, which has a DSi=20.1%, has the lowest ones. From these considerations, we can deduce that 20 V tests are not suitable for this kind of experiments due to the strong energy consumptions.
5. RESULTS AND DISCUSSION

5.2 Electrode choice

5.2.1 Efficiency of electrodes

In order to evaluate the performances of other kinds of anodes and compare them with the DSA, we started by using a stainless steel disc (AISI 304) with five holes (Ø=8.2 mm) to let the gas overflow. Our first goal has been the comparison in terms of current densities produced during the electro-osmotic tests, together with an investigation of corrosion of the anodes.

Since DSA anode after the previous experiments has not been corroded, we started from one of the simplest electrode with the purpose of coating it with different ceramic layers, which could be less expensive and maybe have the same efficiency.

5.2.1.1 Stainless steel (AISI 304) anode

Starting from sludge F (DS$_i$=23.6%), we performed tests with the usual initial conditions: 35 g of sludge (1 cm of thickness) and pressure set at 3 bar. We used three stainless steel discs: one for tests at 10 V/cm, the second for tests at 15 V/cm and the last one for tests at 20 V/cm.

Results are shown in Table 18.

<table>
<thead>
<tr>
<th>N°</th>
<th>$t_{TOT}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H_2O}$]</th>
<th>DS$_f$ [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>E=10 V/cm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>F-1</td>
<td>35</td>
<td>152.3</td>
<td>33.6</td>
<td>10.0</td>
</tr>
<tr>
<td>F-2</td>
<td>35</td>
<td>495.5</td>
<td>27.2</td>
<td>3.6</td>
</tr>
<tr>
<td>F-3</td>
<td>30</td>
<td>495.3</td>
<td>27.0</td>
<td>3.4</td>
</tr>
<tr>
<td>E=15 V/cm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>F-4</td>
<td>25</td>
<td>488.3</td>
<td>29.4</td>
<td>5.8</td>
</tr>
<tr>
<td>F-5</td>
<td>22</td>
<td>369.3</td>
<td>29.3</td>
<td>5.7</td>
</tr>
<tr>
<td>F-6</td>
<td>20</td>
<td>819.0</td>
<td>25.7</td>
<td>2.1</td>
</tr>
<tr>
<td>E=20 V/cm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>F-7</td>
<td>23</td>
<td>307.1</td>
<td>36.2</td>
<td>12.6</td>
</tr>
<tr>
<td>F-8</td>
<td>18</td>
<td>268.5</td>
<td>37.2</td>
<td>13.6</td>
</tr>
<tr>
<td>F-9</td>
<td>18</td>
<td>287.1</td>
<td>38.0</td>
<td>14.4</td>
</tr>
</tbody>
</table>

Table 18-Electro-osmosis results of F sludge (DS$_i$=23.6%) with stainless steel (AISI 304) anode.
5. Results and Discussion

The first remark that must be highlighted is that even if the initial DS is sufficiently low, the conductivity (1132 µS/cm) is not high as expected (the salinity of sludge is lower from the previous ones). This fact strongly affected the efficiency of the tests, bringing to a low final DS and to a high energy consumption, especially for tests from F-1 to F-6. The low value of DS also brought to a less evaporation due to Joule effect (especially at 10 V/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: pitting 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 Figure 50 we can see that the first drop is obtained at relative low times in every tests, as expected for a sludge with an DSi under 25%. The efficiency (especially in cases at 10 V/cm and 15 V/cm) is not high as expected, due to low conductivity of sludge and corrosion of the anode, but the current densities developed during the process are comparable with those of DSA anode (with a remarkable transient stage for the tests at 10 V/cm and 15 V/cm). The electro-osmosis tests last shorter times with respect to the previous experiments with DSA anode, probably due to the strong corrosion of the surface.
5. RESULTS AND DISCUSSION

Figure 50: Current density vs time diagrams at (a) 10 V/cm, (b) 15 V/cm and (c) 20 V/cm of F sludge.
5. RESULTS AND DISCUSSION

Reproducibility of tests seems to be appreciable for experiments at 20 V, which made a lower damage of disc 3, as shown in Chapter 5.2.2.1. In Figure 51 the best results obtained with the three values of electric field are shown.

![Figure 51: Current density vs time diagram at 10 V/cm, 15 V/cm and 20 V/cm of F sludge with stainless steel anodes.](image)
5. RESULTS AND DISCUSSION

5.2.1.2 TiN anode

Starting from the results obtained on the stainless steel substrate, we tested two different TiN coatings (1 µm and 3 µm of thickness) at 15 V/cm. The results are shown in Table 19.

<table>
<thead>
<tr>
<th>N°</th>
<th>t_{tot} [min]</th>
<th>t_{tot} energy consumption [Wh/kg_{H2O}]</th>
<th>DS_{i} [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>F-10 (1 µm)</td>
<td>17</td>
<td>119.2</td>
<td>39.7</td>
<td>16.1</td>
</tr>
<tr>
<td>F-11 (3 µm)</td>
<td>18</td>
<td>136.9</td>
<td>38.8</td>
<td>15.2</td>
</tr>
</tbody>
</table>

As shown in Table 19, we achieved better results in terms of final DS with respect those found with stainless steel anode. This fact can be attributed primarily to the conductivity of the sludge that after 5 days progressively increased until a value of 1211 µS/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).

In Figure 52 the diagrams related to the tests with TiN discs are shown. As expected, the maximum values of current density are achieved for the thinnest coatings, due to a less resistance of the ceramic layer. Moreover, the first drop is obtained late in time with a thick coating.
5. **RESULTS AND DISCUSSION**

![Figure 52: Current density vs time diagrams at 15 V/cm of F sludge with TiN discs (1 µm and 3 µm).](image)

*Figure 52: Current density vs time diagrams at 15 V/cm of F sludge with TiN discs (1 µm and 3 µm).*
5. RESULTS AND DISCUSSION

5.2.2 Corrosion and wear of electrodes

As stated above, the efficiency of the electro-osmosis process is strictly related to the conductivity of the electrodes and to their 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.

5.2.2.1 Stainless steel (AISI 304) anode

In Figure 53 are shown the three stainless steel discs after the tests at 10 V/cm, 15 V/cm and 20 V/cm with sludge F. Pitting corrosion is the main phenomenon occurring during electro-osmosis for stainless steel anodes. It must be highlighted that disc 3 seems to have a smoother surface. This fact, together with DS results with tests at 20 V, could be an evidence that a high voltage can overcome the strong corrosion of the anode. It must be also taken into account that processing times for disc 3 are lower than the other two discs.
5. RESULTS AND DISCUSSION

Figure 53-Stainless steel discs after electro-osmotic tests: disc 1 (F-1, F-2 and F-3 at 10 V), disc 2 (F-4, F-5 and F-6 at 15 V) and disc 3 (F-7, F-8 and F-9 at 20 V).
5. RESULTS AND DISCUSSION

In order to investigate the unusual result of corrosion and efficiency of disc 3, we performed an additional analysis: we wanted to study if the lower time of potential application was the responsible of weaker corrosion for disc 3. Starting from sludge G (DS\(_i\)=21.9%), we studied the behaviour of the stainless steel anodes by applying a potential in a configuration like that shown in Figure 54. We applied 10 V and 20 V for 30/60 minutes between 50 g of sludge G and then measured the weight loss.

![Figure 54-Configuration for anodes corrosion tests.](image)

In Table 20, the results of corrosion tests in terms of weight loss are shown.

<table>
<thead>
<tr>
<th>Disc</th>
<th>Process conditions</th>
<th>Weight loss [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>a</td>
<td>10 V – 30 min</td>
<td>0.29</td>
</tr>
<tr>
<td>b</td>
<td>10 V – 60 min</td>
<td>0.32</td>
</tr>
<tr>
<td>c</td>
<td>20 V – 30 min</td>
<td>0.10</td>
</tr>
<tr>
<td>d</td>
<td>20 V – 60 min</td>
<td>0.17</td>
</tr>
</tbody>
</table>

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. This fact is shown also in Figure 55, where pitting corrosion is evident.
5. RESULTS AND DISCUSSION

Figure 55-Stainless steel discs a (10 V-30 min), b (10 V-60 min), c (20 V-30 min) and d (20 V-60 min) after electric field application.

Therefore, increasing the potential value, a lower corrosion seems to be observed. This kind of behaviour must be further investigated, with a deep study of the involved phenomena.
5. RESULTS AND DISCUSSION

5.2.2.2 TiN anode

In Figure 56 and Figure 57, TiN anodes after electro-osmosis tests at 15 V/cm are shown.

Pitting corrosion is the main mechanism during this tests and it is caused by the great presence of pores in the ceramic coating on the stainless steel disc. This fact is deleterious for the efficiency of the process because it let the contact between substrate and sludge environment.

We can see that the 1 µm-disc is more corroded than the 3 µm-disc and both of them are less damaged than the stainless steel anodes. This fact is an evidence that a coating on the surface is an improvement from the corrosion point of view. 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 and dewatering efficiency). A compromise between these factors must be found.
5. RESULTS AND DISCUSSION

5.3 Dynamic piston electro-osmosis tests

The aim of designing and building an apparatus with a rotating piston has been the investigation of the behaviour of sludge dewatering with the mixing and homogenisation of the cake during the tests. Sludge, during electro-osmosis, encounters loss of water and increase in resistivity next to the anode. By the use of this kind of apparatus, we tried to hinder this problem, also for greater sludge thicknesses.

5.3.1 Efficiency of electrodes

The efficiency of the process with the rotating piston (around 10 rpm) is strongly decreased due to the higher initial cake thickness. Therefore, the electrodes studied in this chapter, which have the same stainless steel substrate, are assumed to have a similar conductivity of the previous anodes, but the results in terms of DS will be worse.

5.3.1.1 AlTiN anode

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 21.

<table>
<thead>
<tr>
<th>N°</th>
<th>$t_{tot}$ [min]</th>
<th>Tot energy consumption [Wh/kg$_{H2O}$]</th>
<th>DS$_f$ [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>E=5 V/cm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>G-1 (STATIC)</td>
<td>30</td>
<td>385.1</td>
<td>25.7</td>
<td>3.8</td>
</tr>
<tr>
<td>G-2 (ROTATING)</td>
<td>30</td>
<td>277.3</td>
<td>28.0</td>
<td>6.1</td>
</tr>
</tbody>
</table>

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.
5. Results and Discussion

Figure 58 shows the current density vs time diagrams of tests G-1 and G-2. The slope of the static experiment is linear and have a maximum in 26 mA/cm², while for G-2 currents slightly increase and fluctuate after the starting of rotation (7 minutes).

![Current density vs time diagrams at 15 V/cm of G sludge with AlTiN discs (static and rotating piston).](image)

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).
5. RESULTS AND DISCUSSION

5.3.1.2 DLC anode

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).

Results are shown in Table 22.

<table>
<thead>
<tr>
<th>N°</th>
<th>tTOT [min]</th>
<th>Tot energy consumption [Wh/kgH₂O]</th>
<th>DSf [%]</th>
<th>ΔDS [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>E=5 V/cm</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>G-3 (ROTATING)</td>
<td>30</td>
<td>253.9</td>
<td>28.3</td>
<td>6.4</td>
</tr>
<tr>
<td>G-4 (ROTATING)</td>
<td>30</td>
<td>296.0</td>
<td>27.5</td>
<td>5.6</td>
</tr>
</tbody>
</table>

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.

In Figure 59, current density vs time diagrams of tests G-3 and G-4 are shown. Slopes, after the start of rotation, increase of about 5 mA/cm² 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.
5. RESULTS AND DISCUSSION

5.3.2 Corrosion and wear of electrodes

5.3.2.1 AlTiN anode
Looking at Figure 60, 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.

![Figure 60-AlTiN coated stainless steel anode after test G-1 and G-2.](image)

5.3.2.2 DLC anode
In Figure 61, DLC anodes after tests G-3 and G-4 at 15 V are shown. In this case it seems that the main phenomenon occurring during electro-osmosis tests is friction corrosion: there is a general damage of the anode surface and pitting 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.
5. RESULTS AND DISCUSSION

Figure 6.1 - DLC coated stainless steel anode after test G-3 and G-4.
6. CONCLUSIONS

In this work, pressure driven electro-osmotic dewatering has been carried out using a lab-scale device with the aim to establish the feasibility of electro-osmotic dewatering, determine the dewatering efficiency in terms of final DS (dry solid) content, the dewatering rate and the energy consumption. Different types of aerobically stabilised and mechanically dehydrated sludge samples have been used, in order to investigate the amount of extracted water by the application of an electric field. The main parameters which control electro-osmosis process were sludge cake thickness, potential value, initial DS, sludge conductivity, material of the electrodes. The experiments have been performed by measuring the current densities developed during the tests, the temperature at the anode inside the cell, the final DS of the cake and computing the energy consumption. We also investigated the behaviour of a larger amount of sludge under electro-osmosis dewatering with the anode rotation: by means of scratching and remixing of the cake surface, we intended to increase sludge conductivity during the tests.

By using a static device, we set electric fields values at 10 V/cm, 15 V/cm and 20 V/cm with a pressure of 3 bar. Tests with DSA have shown a final DS comparable with the results found in literature: a DS range between 37% and 43% have been obtained. The amount of water decreased of 15% on average applying an electric field. We found also that in this range of electric field, the final value of DS is often similar but the main result is that with stronger potential a higher dewatering rate is obtained. This data are reported with the total energy consumption of the electro-osmosis process that must be under the arbitrary threshold of 250 Wh/kg\(_{\text{extracted water}}\): this goal is usually obtained for types of sludge that have DS lower than 25%. Moreover, with stronger potentials we obtained higher current densities, so the energy consumption increased.

By adding distilled water to sludge in suitable amounts, we also investigated dewatering of more wet cakes, where conductivity changed, and usually decreased, due to a less presence of ions. We evidenced that, lowering the initial DS amount of 3-7%, dry matter increases of 1-7% and energy consumptions decreases up to 200 Wh/kg.

Therefore, pressure-driven electro-osmotic tests have shown that, also for dry sludge samples (21%<DS<27%), a dewatering comparable to results found for more wet cakes has been achieved (40% on average). Moreover, energy consumptions are naturally higher than the cases studied in literature, due to stronger bonding between sludge and
6. CONCLUSIONS

water molecules, but these values are always less than 400-500 Wh/kg\(_{\text{extracted water}}\), the threshold for saving with respect thermal drying.

Despite of the increase in final DS amount obtained with pressure driven electro-osmosis dewatering, some characteristics of this kind of lab-scale device are the limit of a possible application on an industrial scale:

- The apparatus can treat only low cake thicknesses (1-1.5 cm) corresponding to low sludge amount in order to keep the conductivity of the process high enough to remove water.
- With the proceeding of dewatering, sludge inside the cell increases its resistance to filtration because of the lower water content. The upper part of the cake, where the anode is placed, results to be more compact, while towards the cathode water concentration is expected to be higher.
- Continuous feed of sludge is not possible in the lab-scale device: only a limited and predetermined quantity of sludge can be treated each cycle.
- The use of a filter cloth between the electrodes increases the resistance inside the cell during the process.
- The energy consumptions for electric fields higher than 20 V/cm exceeds thermal drying values.
- The electrochemical reactions on the electrodes produces gases, which decrease the efficiency and lead to possible explosions inside the cell. Moreover, the oxygen evolution on the anode is the cause of corrosion.
- Electro-dewatering of dry sludge (DS>15%) has both a lower efficiency and a higher energy consumption than the wetter ones.

With the purpose of overcoming one of these critical aspects, we designed and built a rotating piston apparatus, able to scratch and mix the cake surface. A thickness of 3 cm has been studied, but the efficiency was too low (DS<30%) due to the high insulating sludge layer, even though a slight increase (5 mA/cm\(^2\)) of current densities was found when rotation started. These results proved that rotation could improve sludge dewaterability, by increasing conductivity, only for low cake thicknesses (1-1.5 cm).

In order to lower the economic effect on designing an industrial apparatus, we tried to study different materials to be used as anodes. In fact, DSA is too expensive due to the...
6. CONCLUSIONS

presence of titanium, platinum and iridium. However, its strength in strong acidic environment, such as those that are developed during the electro-osmosis tests, is higher than any other kind of coating. We studied, starting from stainless steel (AISI 304) as substrate, different ceramic coatings deposited by PVD technique (TiN, DLC, AlTiN) in order to find a compromise between production costs and resistance to corrosion. However, electro-osmosis tests proved that efficiency of the anodes was good only for low times of potential application, under 30 min, since the presence of pores in the ceramic layers causes high corrosion.

In order to select a suitable material to be used as anode, lowering costs, future studies could be focused on:

- Increase ceramic coating thickness (>30 µm) keeping the same conductivity.
- Use of duplex stainless steel substrate, which offers higher corrosion resistance.
- Sealing of the ceramic layer pores with epoxy coatings.

In conclusion, in this thesis work we go more deeply into the feasibility exam of the electro-osmosis dewatering. It is a promising technique to remove water from sewage sludge, since it is able to increase DS of 15% on average with respect industrial mechanical dewatering but some critical aspects, highlighted in this work, are today a limit for its industrial use.

Moreover, this thesis proved that the treatment of a high DS cake is more difficult than a non-dewatered sludge due to a lower conductivity during the process.

We wish to recall here that we always started from a sludge obtained by industrial process (filter press and Bucher drying system) that means sludge with the maximum amount of DS now obtainable.

6.1 Future research

According to the results, further research is needed in order to optimise sludge electro-dewatering. More information would be achieved by a deep investigation on the influence of the sludge initial DS on the process. Researchers should focus on the study of a great variety of sludge types and analyse the behaviour of each samples under the same conditions, in order to get data and compare the efficiencies.
6. CONCLUSIONS

The purpose of this work is the study of sludge electro-osmosis process and of materials that could be used in the industrial device patented by Stradi et al. (N°: US2013/0098765 A1).

In their apparatus (Figure 62), a potential difference is established between the anode and the cathode in order to subject the sludge to an electric field capable of creating an electro-osmotic process. The sludge crosses the compacting and dehydrating chamber 4, from the anode A and the cathode C, where sludge is subjected to dehydration by means of a screw. This device allows a continuous sludge feed thanks to the presence of an inlet and outlet zone that continuously renew the sludge material. At the same time, the new inlet sludge is wetter than that dewatered, so the solution turns out to have a higher conductivity. Moreover, the action of the screw has an important effect of remixing the sludge on its advancing, leading to a constantly wet and conductive solution. Dry matter is expected to reach 50-75%.

![Figure 62-Stradi et al. electro-osmotic patent device.](image)

1. Apparatus for the treatment of sludge
2. Outer container casing, internally hollow
2a. First end
6. CONCLUSIONS

2b. Second end
3. Inner body, coaxial with the casing, connected electrically to a positive pole of a control unit to form an anode A
4. Compacting and dehydrating chamber along which the sludge runs
5. Inlet zone, equipped with a hopper, to supply the sludge F
6. Outlet zone for unloading the treated sludge outside of the apparatus
7. Movement means, which cooperates with the inner body 3 and promote the compacting and advancement of the sludge F from 5 to 6
7a. Electrical gear reducer
7c. Motion transmission shaft connected to the impeller 14
8. Collection chamber for liquids and gases expelled from the sludge, where a certain degree of depression is generated inside
9. Empty space
10. Filter means (siliceous) arranged inside the casing, delimiting the compacting and dehydrating chamber
11. Container nets, connected electrically to a negative pole of a control unit to form a cathode C
12. Hydraulic circuit
13. Nozzles to moisten the filter means
14. Impeller, with annular shape, set in rotation by movement means 7 and electrically connected to the positive pole of a control unit; it is characterized by a variable pitch, which decreases from the inlet zone 5 to the outlet zone 6 (the material along the route undergoes a progressive loss of volume and mass and the decrease in pitch keep it compacted)
15. Longitudinal insulating battens, inserted into the body of the filter means
16. Anti-wear skates arranged in discrete positions along the perimeter profile of the impeller, also with the function of electrically insulating the anode (the impeller) from the surfaces of the filter means
17. Static compacting chamber, where sludge stands temporarily while waiting to be pushed onto the impeller (14)

Therefore, this kind of device is an improvement of a mechanical centrifuge, where, thanks to the electric field application, a higher DS amount could be obtained without some of the critical aspects of a lab-scale device.
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