PRETREATMENT OF WASTEWATER SLUDGE BY
PULSED ELECTRIC FIELD APPLICATION AT DIFFERENT pH CONDITIONS

by
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PRETREATMENT OF WASTEWATER SLUDGE BY
PULSED ELECTRIC FIELD APPLICATION AT DIFFERENT pH CONDITIONS

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ABSTRACT

Wastewater treatment processes produce large amounts of sludge to be stabilized and dewatered before safe utilization or disposal. Biological degradation processes are usually preferred sludge stabilization methods since they are cost-effective and environmentally friendly. However they have some limitations in terms of long treatment times and large working areas requirements. To overcome these limitations and to improve sludge biodegradability, different pretreatment methods are developed.

The objective of this study is to evaluate pulsed electrical field (PEF) application as a sludge pretreatment method. For this purpose, PEF up to 30 kV/cm with different pH conditions was applied on sludge. The results demonstrated that PEF pretreatment deteriorated sludge dewaterability measured in terms of specific resistance to filtration and capillary suction time. However PEF pretreatment improved sludge compactibility measured in terms of cake dry solids content. According to semi-continuous aerobic biodegradability tests, the mean COD removal was increased by 7.8 % and the mean VS removal was increased by 4.6 %, for the reactors fed with PEF pretreated sludge compared to the control reactors. Batch aerobic biodegradation experiments demonstrated that the mean improvements supplied by PEF pretreatment were 3.4 and 2.2 %, for COD and VS removals, respectively.

ÖZET

Atıksu arıtma prosesleri sonucunda oluşan çamurların güvenli bir şekilde kullanılması veya bertaraf edilmesi için öncelikle stabilize edilmesi ve susuzlaştırılması gerekmektedir. Biyolojik çamur çürütme prosesleri ekonomik ve çevre dostu olmaları nedeniyle tercih edilen stabilizasyon yöntemleridir. Ancak bu prosesler uzun hidrolik bekleme süreleri ve geniş arıtma alanları gerektirmektedir. Bu olumsuzlukların giderilmesi ve çamurun parçalanabilirliğinin arttırılması amacıyla çeşitli ön arıtma yöntemleri geliştirilmiştir.

Bu çalışmanın amacı, vurgulu elektrik alan (VEA) uygulamasının bir çamur ön arıtma yöntemi olarak değerlendirilmesidir. Bu amaçla çamur örneklerine çeşitli pH koşullarında 30 kV/cm'e kadar VEA uygulanmıştır. Spesifik filtrasyon direnci ve kapiler emme zamanı analizleri sonucunda VEA uygulamasının çamur susuzlaştırma verimini azalttığı tespit edilmiştir. Ancak VEA uygulaması, çamur kekindeki katı madde içeriği yönünden çamur sıkıştırabilirliğini arttırmıştır. Yarı sürekli aerobik çamur çürütme deneyleri sonucunda, VEA uygulanan çamur ile beslenen reaktörlerdeki KOİ giderim miktarının ham çamur ile beslenen reaktörlerdeki KOİ giderim miktarından ortalama yüzde 7.8 yüksek olduğu gözlemlenmiştir. Bu oranın uçucu katı madde giderim hızı için yüzde 4.6 olduğu belirlenmiştir. Kesikli aerobik çamur çürütme deneyleri sonucunda, VEA ile ön arıtımın KOİ ve uçucu katı madde giderim miktarlarını sırasıyla yüzde 3.4 ve 2.2 arttırdığı tespit edilmiştir.

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LIST OF SYMBOLS/ABBREVIATIONS

Symbol	Explanation	Units used
COD	Chemical Oxygen Demand	mg/L
CST	Capillary Suction Time	s
DD_{COD}	Degree of Disintegration	%
HRT	Hydraulic Retention Time	s
ORP	Oxidation Reduction Potential	mV
SCOD	Soluble Chemical Oxygen Demand	mg/L
SRF	Specific Resistance to Filtration	m/kg
SRT	Sludge Retention Time	s
T	Temperature	^{o}C
TCOD	Total Chemical Oxygen Demand	mg/L
TKN	Total Kjeldahl Nitrogen	mg/L
TS	Total Solids	mg/L
TSS	Total Suspended Solids	mg/L
VS	Volatile Solids	mg/L
VSS	Volatile Suspended Solids	mg/L

BSA	Bovin Serum Albumin
CER	Cation Exchange Resin
EPS	Extracellular Polymeric Substance
HV	High Voltage
PEF	Pulsed Electrical Field
WAS	Waste Activated Sludge

1. INTRODUCTION

The amount of sludge produced from wastewater treatment plants is increasing because of the increase in the population and the amount of wastewater being treated. The produced sludge from wastewater treatment plants has to be stabilized and dewatered before safe utilization or disposal.

Biological sludge stabilization methods including aerobic and anaerobic biodegradation processes are usually preferred since these processes are cost-effective and environmentally friendly. On the other hand, the biological processes have some limitations in terms of long treatment times and large working areas requirements. There are many researches devoted to overcome these limitations and to improve the efficiency of the biological processes. For this purpose, several pretreatment techniques have been developed including mechanical, thermal, chemical, thermo-chemical and biological sludge disintegration methods. These disintegration methods supply the destruction of floc structures and disruption of the cell walls of the microorganisms present in the wastewater sludge. The results are the acceleration of sludge hydrolysis and increased biodegradation rates. There are also several researches investigating the effects of these disintegration methods on the sludge dewaterability.

Pulsed electrical field is an emerging technology that can be used for sludge pretreatment. Pulsed electrical field technology is the application process of a series of short duration (10 ns – 20 μs), high voltage pulses (10–50 kV/cm) to the liquid in a treatment chamber which is placed between two electrodes. There are various studies on the development of pulsed electric field technology for bacterial inactivation in the food industry. Only recently, the applicability of this technology in biomedicine, genetic science and environmental technologies has become the subject of researches. Although pulsed electric field technology has a potential use in several environmental treatment processes such as pretreatment of wastewater sludge, electrodialytic remediation of heavy metals in wastewater and electro-osmotic dehydration, there are only a limited number of researches on these subjects.

The purpose of this study is to evaluate the performance of pulsed electrical field (PEF) as a sludge pretreatment (disintegration) method prior to sludge dewatering and aerobic biodegradation. This study also investigates the efficiency of pulsed electric field at different electric field intensities with different hydraulic retention times and different pH conditions. For this purpose PEF up to 30 kV/cm with hydraulic retention times up to 9.0 seconds were applied to sludge samples at different pH conditions between 3.0 and 11.0. The effects of pulsed electric field application on the sludge samples were evaluated by the degree of disintegration of the sludge samples. Dewaterability properties of the control and pretreated sludge samples were examined by specific resistance to filtration, capillary suction time and compactibility analyses. In order to reveal the reasons of the obtained results, the protein and carbohydrate contents of the extracellular polymeric substances (EPS) in the sludge samples were analyzed.

The effects of PEF pretreatment on the aerobic biodegradability of sludge samples were investigated using four batch and four semi-continuous aerobic bioreactors. These reactors were fed with untreated and PEF pretreated sludge samples. The effects of pulsed electric field on the aerobic biodegradability of the sludge samples were evaluated by comparing chemical oxygen demand and volatile solids removals in these reactors.

2. THEORETICAL BACKGROUND AND LITERATURE REVIEW

Sludge disintegration can be defined as the destruction of sludge by external forces which result in numerous changes in the sludge properties including destruction of floc structures and disruption of the cell walls of the microorganisms present in the wastewater sludge (Müller, 2003a). Thus organic sludge components and fine particles are released into the liquid phase. The released sludge components after disintegration are easily accessible and more susceptible to other chemical and biological processes.

In the following section, the existing sludge disintegration methods are explained and related studies are evaluated.

2.1. Sludge Disintegration Methods

2.1.1. Mechanical Sludge Disintegration

Mechanical sludge disintegration processes include application of pressure, translational or rotational energy on sludge creating mechanical shear on the sludge. This process destroys the floc structure and disrupts the cell walls of the microorganisms. (Müller et al., 1998). Thus, the organic materials in the sludge become more available to other biochemical reactions and new surface areas are created where these reactions can take place. The main mechanical sludge disintegration methods are explained in the following sections.

2.1.1.1. High Pressure Homogenizer. High pressure homogenizer consists of a multi-step high-pressure pump and a homogenizing valve. The pump compresses the sludge to pressures up to several hundred bar. When passing through the homogenizing valve, the pressure drops suddenly. Pressure decrease and rapid velocity increase cause turbulence, cavitation and liquid stress to the cells. The static pressure decreases until the vapor pressure of the liquid is reached resulting in steam or cavitational bubbles which further increases the gas-liquid flow. Collision of these cavitation bubbles induces an energetic shear stress that disrupts the cells (Onyeche and Schafer, 2003). High-pressure homogenizers are found to be very efficient for sludge disintegration but full-scale applications and long-term experiences are quite poor. (Andreottola and Foladori, 2006)

Onyeche and Schafer (2003) used high-pressure homogenizer at relatively low pressures (500 bar) to disrupt sludge cells. The obtained sludge was used to feed anaerobic digesters. The results of Onyeche and Schafer (2003) demonstrated that the biogas from concentrated and homogenized sludge was higher up to two times, when compared to that from untreated samples. Moreover, Onyeche and Schafer (2003) claimed that appreciable reduction in the sludge produced was obtained. Figure 2.1. shows a schematic diagram of the high-pressure homogenizer used by Onyeche and Schafer (2003).

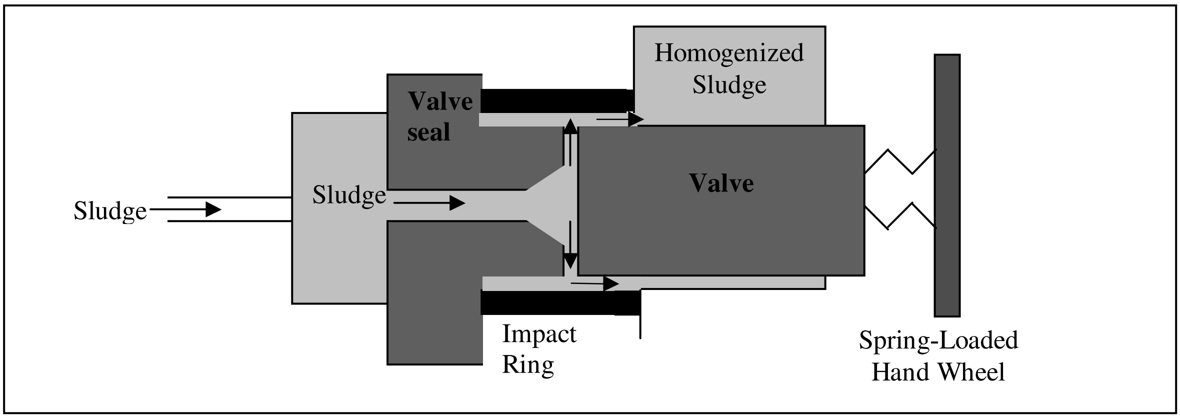

Figure 2.1. High-pressure homogenizer
(redrawn after Onyeche and Schafer, 2003)

Müller (2003b) investigated the effects of mechanical sludge disintegration by high pressure homogenizer on sludge dewatering and filtration process. The results showed that sludge disintegration supplied increase in the dry solids content of the dewatered sludge when applying centrifugal forces, using beaker centrifuge. In order to examine sludge dewaterability results from industrial equipment, Müller (2003b) used conditioned sludge samples. He observed that conditioner demand was increased by sludge disintegration. Using a bench scale thickening decanter, similar results with beaker centrifuge were obtained demonstrating that dry solids content of the thickened sludge was increased by sludge disintegration. On the other hand, the results of filtration using a membrane plate and frame filter showed that sludge dewaterability in terms of filterability was deteriorated because of the disintegration. Müller (2003b) concluded that although mechanical disintegration showed some positive effect on sludge thickening, dewaterability in terms of filterability was deteriorated.

2.1.1.2. Stirred Ball Mill. Stirred ball mill contain a grinding medium (e.g. sand) and rotors that force the grinding medium into a rotational movement. Resulting shear and pressure forces cause to the disintegration of the microorganisms present in the sludge.

Baier and Schmidheiny (1997) used ball milling to disintegrate different sludge samples. They obtained an increase in soluble COD from 1-5 % for original sludge, up to 47% after ball milling. Their anaerobic biodegradation tests showed that ball milling enhanced overall VS reduction of waste activated sludge from 38 % to 57 %. Moreover Baier and Schmidheiny (1997) reported that they obtained an enhancement in overall COD degradation of the sludges by a factor of 1.2-1.5. Net biogas production was enhanced in the same order of magnitude.

2.1.1.3. Lysate Centrifuge. Lysate centrifuge consists of a centrifuge and a ring with a special design (lysate ring) integrated into the centrifugal thickener. The centrifugal forces, created in the lysate thickening centrifuge are applied for cell destruction using lysate ring that dissipates the kinetic energy provided by the centrifuge. Zábranská et al. (2006) used a full scale lysate-thickening centrifuge for the disintegration of excess activated sludge. Their results based on the data obtained from three different wastewater treatment plants showed that sludge disintegration by lysate-thickening centrifuge increased specific biogas production rate in the range of 15-26 % and decreased organic matter in the digested sludge significantly.

2.1.1.4. Mechanical Jet Technique. In the mechanical jet technique, the sludge stream pressurized to 5-50 bar and the pressure is released across a nozzle. Subsequently the sludge stream impinges on a splash plate and disintegrated.

Nah et al. (1999) used jetting and colliding technique at 30 bar for the solubilization of waste activated sludge. The schematic diagram of the system used by Nah et al. (1999) is shown in Figure 2.2. Using this technique they obtained increase in soluble chemical oxygen demand about 5-7 times and decrease in suspended solids concentration about 5 %. Nah et al. (1999) fed pilot scale anaerobic digester with waste activated sludge pretreated by jetting and colliding technique. They observed that this pretreatment allowed a decrease in the anaerobic digester SRT from 13 to 6 days and also enhanced VS reduction and unit gas production. (Related data were not given.)

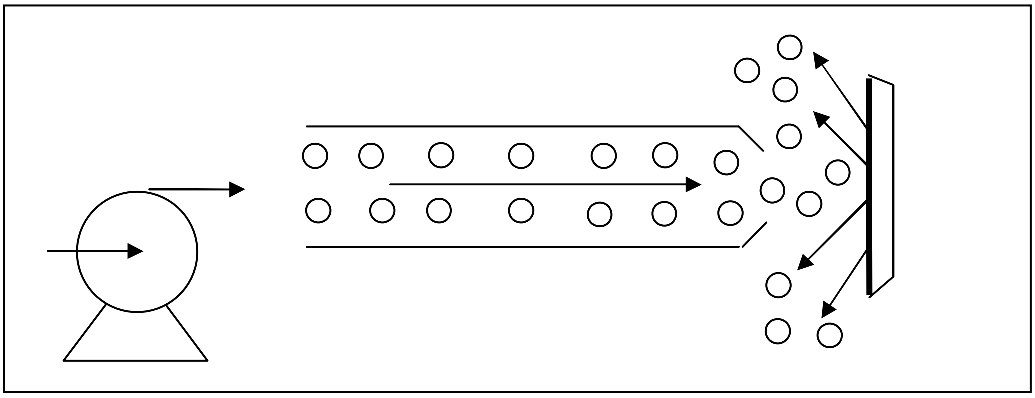

Figure 2.2. Jetting and colliding technique
(redrawn after Nah et al., 1999)

2.1.1.5. Ultrasound. Ultrasonic sludge pretreatment is the acoustic generation of cavitation leading to sludge disintegration. When applied to a liquid, ultrasound propagates forming alternating cycles of rarefactions (negative pressures) and compressions in the liquid. These alternating cycles of compression and rarefaction can produce a phenomenon known as cavitation (Clark and Nujjoo, 2000). Cavitation is the formation, growth and subsequent collapse of microbubbles in milliseconds, thus producing high levels of pressure and temperatures (Gogate, 2002). Mechanisms of ultrasonic sludge pretreatment include the generation of strong hydro-mechanical shear forces during cavitational collapse, which supply the disruption of the adjacent bacterial cells in the sludge. Moreover, sonochemical reactions produce oxidizing radicals contributing to the sludge disintegration (Wang et al., 2005). High temperature regions produced by acoustic cavitation assist to the sludge disintegration by decomposing cell membrane. Schematic diagram of a typical ultrasonic system is shown in Figure 2.3.

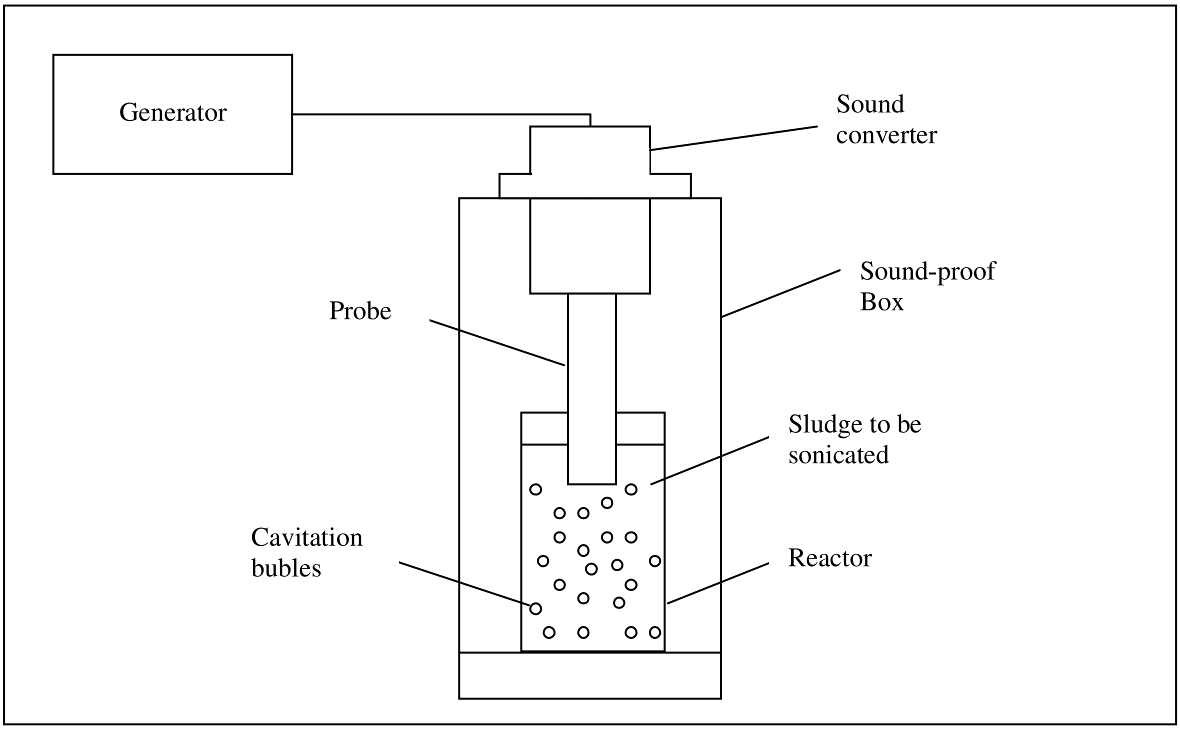

Figure 2.3. A typical ultrasonic system

Tiehm et al. (2001) examined the improvement of anaerobic stabilization of waste activated sludge by ultrasonic disintegration. They fed five semi-continuous anaerobic reactors with control sludge and sludge pretreated by ultrasound with different pretreatment times. The results demonstrated that as the pretreatment time was increased from 0 to 150 minutes, volatile solids degraded by anaerobic biodegradation were increased from 21.5 % up to 33.7 % and the total biogas produced was increased from 2.93 l up to 4.15 l. The increase in digestion efficiency was found to be proportional to the degree of sludge disintegration.

Chu et al. (2001) observed that the dewaterability of sludge has been seriously deteriorated after ultrasonic treatment. According to their results, after 60 minutes sonication at a level of 0.33 W/mL, the CST was increased from 197 s for the untreated sludge up to 490 s for the sonicated sludge. The bound water content for the original sludge was 3.8 kg/kg dry solids and became 11.7 kg/kg dry solids, after ultrasonication. They attributed these results to the great amount of water attached on the large surfaces supplied by the small particles after ultrasonication.

2.1.2. Chemical Sludge Disintegration

2.1.2.1. Mineral Acids or Alkali. As a sludge pretreatment process, addition of mineral acids or alkali to the wastewater sludge are used to disintegrate the organic compounds and solubilize the microbial cells. The use of alkaline addition is more common than the use of acids. (Akerlund, 2008). During the alkaline treatment process, the pH of the sludge is increased up to 12 and kept at this high pH level for a period of time (approximately 24 hours). This process hydrolyses and decomposes lipids, hydrocarbons and proteins into smaller soluble compounds such as aliphatic acids, polysaccharides and amino acids (Chiu and Chang 1997). As a result, alkaline sludge pretreatment disrupts the sludge flocs and cells thus; improves sludge hydrolysis and biodegradability (Kim et al., 2003). Moreover alkaline sludge treatment can release the water held inside cell structure, which can not be released by conventional dewatering processes; as a result, alkaline treatment improves sludge compactibility (Erdincler and Vesilind, 2000).

Lin et al. (1997) pretreated waste activated sludge using different amounts of sodium hydroxide (20-40 meq/l). They observed that the COD removals in anaerobic reactors fed with sludge pretreated with sodium hydroxide were 46-52 % while it was 38 % for the anaerobic reactor fed with untreated sludge. Moreover they noted that sludge pretreatment supplied 30-163 % increase in the biogas production rate during anaerobic biodegradation.

Jin et al. (2009) evaluated combined NaOH and ultrasonic pretreatment on the efficiency of aerobic sludge digestion. They observed that the simultaneous pretreatment supplied higher degree of disintegration when compared to NaOH treatment followed by ultrasonic treatment or ultrasonic treatment followed by NaOH treatment. Jin et al. (2009) conducted aerobic biodegradability tests using the pretreated and untreated sludge samples. They claimed that the degradation efficiency of organic matter was increased from 38.0 % (control sludge) to 42.5 %, 43.5 % and 50.7 % due to ultrasonic pretreatment, NaOH pretreatment and combined pretreatment, respectively.

Neyens et al. (2003) used hot acid hydrolysis as a potential treatment of thickened sewage sludge with dry solids (DS) content of 5–6 %. Neyens et al. (2003) concluded that hot acid hydrolysis is efficient in both reducing the residual sludge amounts and improving sludge dewaterability. According to their results, the amount of hydrolyzed DS was approximately 70 % lower than the initial untreated amount and the DS-solid content of the dewatered cake was increased from 22.5 % (initial untreated) to at least twice this value. On the other hand the rate of mechanical dewatering was not significantly affected. Moreover, Neyens et al. (2003) observed that the organic part of the dry solids (ODS) was preferentially released in the water phase and the BOD/COD ratio was increased in the water phase by hydrolysis indicating a better biodegradability of the water phase. Neyens et al. (2003) also noted that heavy metals and phosphates were released in the water phase which could be subsequently precipitated.

2.1.2.2. Ozone. Ozone (O_3) is an allotrope of oxygen and it has strong oxidative properties. In water, ozone may react directly with dissolved substances, or it forms secondary oxidants which react with solutes (Staehelin and Hoigne, 1985). These properties of ozone can be used for sludge disintegration. Analyzing the mechanism of sludge ozonation, Yan et al. (2009) concluded that after the sludge was exposed to ozone at a dosage less than 0.02 g O_3/g MLSS, the primary effect of ozonation was sludge solubilization. As the dosage was increased, the microorganisms lost most of their activities and ozone transformed the bio-macromolecules into small molecules. Increasing the dosage higher than 0.14 g O_3/g TSS, caused to decrease in the efficiency of sludge oxidation because of the release of radical scavengers such as lactic acid and SO_4^{2-} from the microbial cells in the sludge.

Weemaes et al. (2000) investigated the anaerobic digestion of ozonized biosolids. According to their results, sludge pretreatment by ozone altered up to 67 % of the organic matter via solubilization and oxidation. Weemaes et al. (2000) claimed that in the anaerobic digestion of the ozonized sludge (using a dose of 0.1 g O_3/g COD), the methane production could be enhanced by a factor of 1.8 and the digestion rate was accelerated by a factor of 2.2. They noted that a higher ozone dose had a less pronounced positive effect on the anaerobic digestion.

2.1.2.3. Fenton Process. Fenton's reagent is a solution of hydrogen peroxide and an iron catalyst. It is used to oxidize contaminants and wastewaters. Fenton's reaction is the oxidation of ferrous iron (Fe^{2+}) by hydrogen peroxide to ferric iron (Fe^{3+}), a hydroxyl radical and a hydroxyl anion. This reaction is shown below:

$$Fe^{2+}_{aq} + H_2O_2 \rightarrow Fe^{3+}_{aq} + OH\cdot + OH^{-} \tag{2.1}$$

The ferrous iron (Fe^{2+}) catalyzes the decomposition of H_2O_2 which result in the generation of hydroxyl radicals via a complex reaction sequence in an aqueous solution (Pignatello, 1992). The hydroxyl radicals are very reactive and capable of decomposing a number of organic substances by oxidation.

Erden and Filibeli (2009) investigated the effects of Fenton process on anaerobic sludge bioprocessing. They applied a ratio of 0.067 g Fe(II) per gram H_2O_2 and 60 g H_2O_2/kg dried solids for the pretreatment of biological sludge samples preceding anaerobic digestion. They compared single stage thermophilic anaerobic digestion with two-stage anaerobic digestion (mesophilic digestion prior to thermophilic digestion). Erden and Filibeli (2009) obtained higher solid reduction and higher methane production with the Fenton processed sludge for each experiment. According to their results, thermophilic sludge digestion supplied the highest reduction in the sludge solids. At the end of the 30 day operation of the thermofilic reactors, dried solids and volatile solids contents of the control reactor were 14.4 and 20.5 %, respectively while those of the reactor fed with Fenton processed sludge were 28.2 and 26.8 %, respectively. Although the second stage digestion under mesophilic conditions did not induce an extra improvement in solid reduction, it provided more methane production. Erden and Filibeli (2009) reported that at the end of the 30 day operation period, Fenton pre-treatment provided 1.4 and 1.2 times higher methane productions in single stage of digestion and second stage of digestion, respectively. Moreover, they noted that Fenton process led to the decrease in the resistance of biosolids to dewatering in terms of capillary suction time but it did not improve sludge dewatering on belt-press application.

2.1.3. Thermal Sludge Disintegration

Bougrier et al. (2008) evaluated the effects of thermal treatment on solubilization, physical properties and anaerobic digestion of waste activated sludge samples. They used treatment temperatures between 90 and 210^oC with a treatment time of 30 minutes. According to their results, COD solubilization was increased linearly with treatment temperature for the temperatures lower than 200^oC. On the other hand, solubilization results were found to be more dispersed with treatment temperatures above 200^oC. Bougrier et al. (2008) noted that carbohydrates solubilization was more important than proteins solubilization for temperatures lower than 150^oC. This was explained by the fact that carbohydrates are located in exopolymers whereas proteins are mainly inside the cells. Moreover, carbohydrates concentration decreased at high temperature as they reacted with other carbohydrates or solubilized proteins.

Bougrier et al. (2008) observed that sludge dewaterability in terms of capillary suction time (CST) was deteriorated by heat treatment for the temperatures lower than 150^oC but it was improved for the temperatures higher than 150^oC. They explained this result by the modification of sludge structure and the release of linked water above this temperature. According to the batch anaerobic tests, thermal pretreatments up to 190^oC supplied increase in the biogas produced. Thermal pretreatment above 210^oC caused to decrease in the anaerobic biodegradability. This result was attributed to the formation of inhibitory or toxic compounds above this temperature. Bougrier et al. (2008) related the biogas volume enhancement by thermal pretreatment up to 190 ^oC to the solubilization of COD and to the initial biodegradability of the sludge samples. The impact of thermal pretreatment on the sludge biodegradability was found to be higher for the sludge samples with lower initial biodegradability.

2.1.4. Biological Sludge Hydrolysis

Biological hydrolysis can be considered as a partial anaerobic sludge digestion (Weemaes and Verstraete, 1998). By controlling hydraulic retention time and temperature, anaerobic digestion can be confined to the acidogenic and acetogenic phase, thus supplying the solubilization of organic matter in the sludge. Moreover commercially available enzymes can be added to sludge in order to accelerate the solubilization of the sludge solids for improved anaerobic digestion.

Ayol et al. (2008) studied the effects of the addition of hydrolytic enzymes to aerobic and anaerobic reactors. Their results demonstrated that addition of hydrolytic enzymes improved the VS reduction in the anaerobic reactors from 27.5 % up to 43.4 %. Moreover, Ayol et al. (2008) observed that enzyme-added anaerobic reactors gave almost two times higher gas production than the control one. They also noted that enzyme addition enhanced the degradation of extracellular polymeric substances. This result was found to be consistent with lower CST results for the enzyme added anaerobic reactors than that for the control reactor. Although enzyme additions had no positive effect in aerobic reactors in terms of VS reduction and filterability, they supplied reduction in EPS compared to those in the control reactor.

2.2. Effects of Sludge Disintegration on Dewaterability

In the literature there are many researches demonstrating that sludge disintegration can improve both aerobic and anaerobic biodegradability of sludge (Lin et al., 1997, Weemaes et al., 2000, Tiehm et al., 2001, Bougrier et al., 2008, Jin et al., 2009). However sludge disintegration can improve or deteriorate sludge dewaterability depending on many factors (Feng et al., 2009). Sludge disintegration can alter the water distribution in sludge as well as the amount and composition of extracellular polymeric substances (EPS) in sludge which are important factors in sludge dewaterability. These factors are briefly explained in this section.

2.2.1. Sludge Water Distribution

Sludge dewaterability is highly dependent on the water distribution in sludge. Vesilind (1994) defined four categories of water in sludge as free water, interstitial water, vicinal water and water of hydration. According to this definition, free water is the water that not associated with and not influenced by suspended solid particles. Interstitial water is trapped within the floc structure and microorganisms. Vicinal water is associated with the multiple layers of water molecules held tightly to the particle surface. Water of hydration is chemically bound to the particles and can only be removed by thermal destruction of the particles. (Vesilind, 1994).

Dewil et al. (2006) observed that dewaterability of the waste activated sludge was decreased by ultrasound disintegration in terms of increased CST and decreased dryness of the filter cake after vacuum filtration. Dewil et al. (2006) explained this decrease in the dewaterability by the reduction in the floc size after sonication offering an extended surface area. As a result, more surface water was bound which led to the increase in the CST and the sludge filterability was decreased because of clogging of the cake.

Erdinçler and Vesilind (2000) investigated the effect of sludge cell disruption on the compactibility of biological sludge. In this study, sludge cells were disrupted by alkali treatment, NaCl treatment, heat treatment, and sonication. The results demonstrated that sludge cell disruption improved the solid content of compacted sludge up to 87 %, depending on the cell disruption method used. Erdinçler and Vesilind (2000) explained this improvement by the fact that sludge cell disruption changed the water distribution in the biological sludge by releasing a considerable amount of interstitial water trapped inside sludge microorganisms and within the floc structure. Erdinçler and Vesilind (2000) noted that cell disruption increased unfreezable water content of the sludge by creating additional surfaces for water binding but unfreezable water content did not affect the compactibility of the biological sludge.

2.2.2. Extracellular Polymeric Substances (EPS)

Extracellular polymeric substances (EPS) are organic macromolecules including polysaccharides, proteins, nucleic acids, lipids and other polymeric compounds present at or outside the cell surface (Flemming and Wingender, 2001). EPS can be expected to have an influence on sludge dewaterability through the high level of hydration of the polymer surrounding bacterial cells and its role in flocculation (Houghton et al., 2001).

Feng et al. (2009) studied the effects sludge disintegration by sonication on the dewaterability of waste activated sludge. They concluded that there exist some optimum level of ultrasound energy which enhances the sludge dewaterability, in terms of CST and SRF. The results of Feng et al. (2009) demonstrated that application of low specific energy dosages (<4400 kJ/kg TS) slightly enhanced sludge dewaterability, but larger specific energy dosages (>4400 kJ/kg TS) significantly deteriorated sludge dewaterability. Feng et al. (2009) attributed these results to the fact that sludge sonication resulted in the release of large quantities of EPS into solution. The high concentrations EPS with a high affinity for water, increased the viscosity of the sludge and decreased its dewaterability. Moreover EPS formed a film layer on the surface of filtrating membrane, thus impeded the passage of water during vacuum filtration process. On the other hand, Feng et al. (2009) noted that increasing EPS content up to an optimal dosage by sonication yielded maximum sludge dewaterability. This was explained by the fact that increasing EPS concentration above relatively low levels improved the extent of sludge flocculation (Sanin and Vesilind, 1994). Sludge flocculation reduced the number of small particles present in the sludge leading to improved dewaterability characteristics. Feng et al. (2009) also noted that subjecting sludge to a combination of cationic polymer conditioning and ultrasound pretreatment did not significantly improve sludge dewaterability compared to cationic polymer conditioning alone. They concluded that when both methods were used together, the effects of cationic polymer conditioning predominated over those of ultrasound pretreatment.

2.3. Pulsed Electric Field Processing

Pulsed Electric Field (PEF) technology is the application process of a series of short duration (10 ns – 20 μs), high voltage pulses (10–50 kV/ cm) to the liquid in a treatment chamber which is placed between two electrodes.

Although there are some earlier researches, during 1990's several studies are made to develop this tecnology for bacterial inactivation in food industry. Only recently, the applicability of this technology in biomedicine, genetic science and environmental technologies has become the subject of researches. Although pulsed electric field technology has a potential use in several environmental treatment processes such as pretreatment of wastewater sludge, electrodialytic remediation of heavy metals in wastewater and electro-osmotic dehydration, there are only a limited number of researches on these subjects. Before explaining the use of pulsed electric field (PEF) as a sludge pretreatment technique, the mechanisms of PEF generation, the mode of action of PEF on the microorganisms and the factors influencing the PEF processing are explained in the following sections.

2.3.1. Mechanisms of Pulsed Electric Field Generation

The main components of pulsed electric field processing consist of a power source, a high voltage generator, a capacitor, a high voltage switch and a treatment chamber. These components are shown in Figure 2.4.

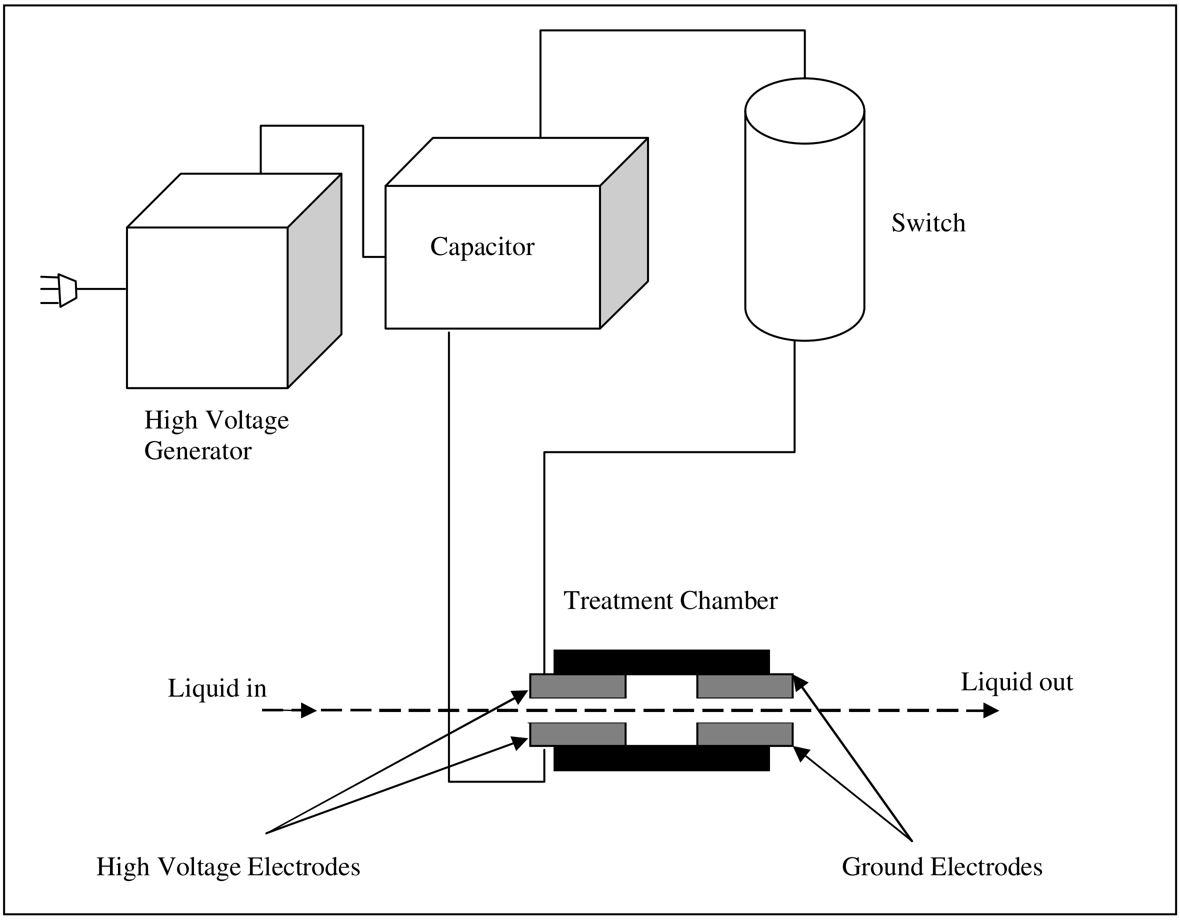

Figure 2.4. The main components of pulsed electric field processing

The direct current high voltage power supply charges the capacitor bank that consists of a series of capacitors. A large amount of energy is temporarily stored in the capacitor bank and discharged when a trigger signal is applied. The high voltage switch is used to discharge this energy in the form of high voltage pulses to the treatment chamber where the liquid to be treated is placed.

The treatment chamber is designed to supply insulation and holding of the liquid to be treated. The system can be either in batch or continuous flow mode. The liquid to be treated is placed or flows between two electrodes on the treatment chamber and receives the high voltage pulses. The electrodes on the treatment chamber are made of inert materials, such as carbon, titanium, gold, platinum etc. The electrode surfaces should be large enough compared to the gap between the electrodes in order to have a uniform and strong electric field during the PEF treatment (Qin et al., 1995a). This limits the capacity of the treatment chamber and many researches are focused to overcome this limitation and scale up the pulsed electric field treatment systems. Different treatment chamber designs include static treatment chambers and continuous treatment chambers such as co-axial and co-field flow PEF treatment chambers which can be used for large-scale operations.

2.3.2. Mechanisms of Pulsed Electric Field Treatment

When applied to a liquid, pulsed electric field cause to the disintegration of cell membrane of the microorganisms present in the liquid and to the formation of highly reactive free radicals depending on the chemical composition of the liquid. The main theories developed to explain the mode of action of pulsed electric field on the cell membrane of microorganisms are dielectric breakdown and electroporation phenomenon. The heat generated during pulsed electric field treatment and the secondary effects of the electrochemical reactions are less relevant when ultra-short duration pulses are applied (Loeffler et al., 2001, Lindgren et al., 2002, Töpfl, 2006). The theories of dielectric breakdown and electroporation are explained briefly in the following sections.

2.2.2.1. Dielectric Breakdown of Cell Membrane. Zimmermann (1986) explained the dielectric breakdown of cell membrane by compression of the cell membrane and formation of the pores on the cell membrane under high voltage. According to this theory, the cell membrane can be considered as a capacitor filled with a dielectric material with small dielectric constant.

As shown in Figure 2.5, when an external electric field is applied, free charges are accumulated on both sides of the membrane but not in high concentration within the membrane hydrocarbon layer (Castro et al., 1993). The opposite charges are accumulated on two sides of the cell membrane and their attraction result in the compression of the membrane and reduction in the membrane thickness. As the membrane potential is increased, the thickness of the cell membrane is further reduced. With further increase in the external field strength, the critical breakdown voltage is reached and the membrane breakdown occurs causing to the formation of transmembrane pores. This process is reversible up to a critical field strength and it becomes irreversible above this field strength. As a result, mechanical destruction of the cell membrane occurs.

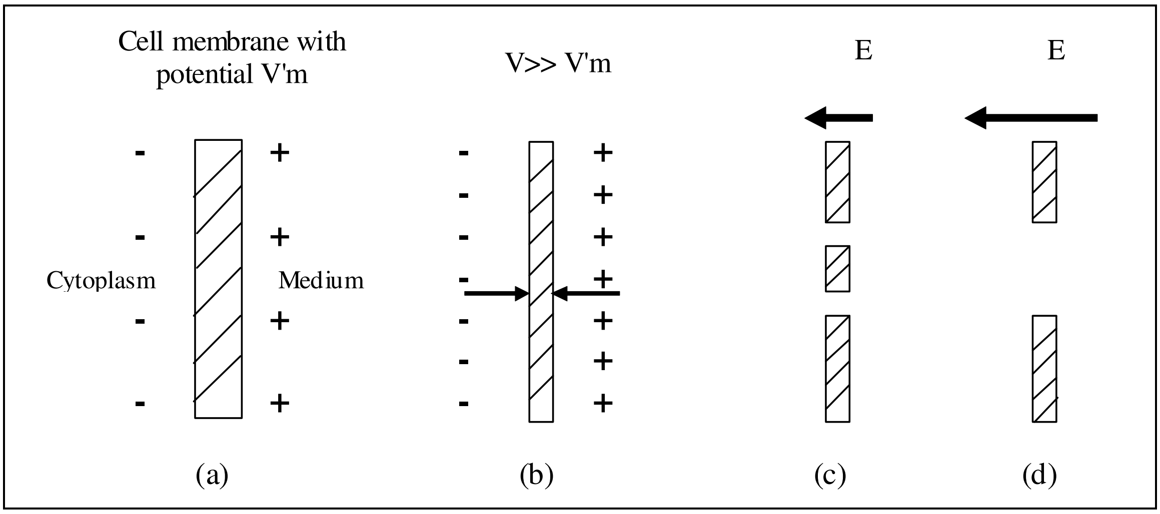

Figure 2.5. Electrical breakdown of cell membrane (a) Cell membrane with potential V'm, (b) membrane compression, (c) pore formation with reversible breakdown, (d) large area of the membrane subjected to irreversible breakdown with large pores (redrawn after Zimmermann, 1986)

<u>2.2.2.2. Electroporation of Cell Membrane</u>**.** Tsong (1991) explained that electroporation in the cell membrane could occur both in lipid bilayers and protein channels. Under high voltage, charges or the electric dipoles of the lipid molecule cause to the reorientation of the lipid bilayers while creating hydrophilic pores. The permeability of the bilayer to ions cause to the current flow, thus, generating local Joule heating and inducing thermal phase transitions of the lipid bilayer. After a critical voltage, the lipid bilayer is rearranged expanding the existing hydrophobic pores, or creating new ones and forming structurally more stable hydrophilic pores. The protein channels present on the cell membranes are also sensitive to high voltage. These channels are opened and closed depending on the transmembrane electric potential. When high voltage is applied, these protein channels are opened and once opened they may experience larger currents than they can conduct. As a

result these channels may be irreversibly denatured by Joule heating or electric modification of their functional groups. After the electroporation, cell membrane becomes permeable to small size molecules. This causes to osmotic imbalance, which lead to cell swelling and finally rupture of the cell membrane. These processes are shown schematically in Figure 2.6.

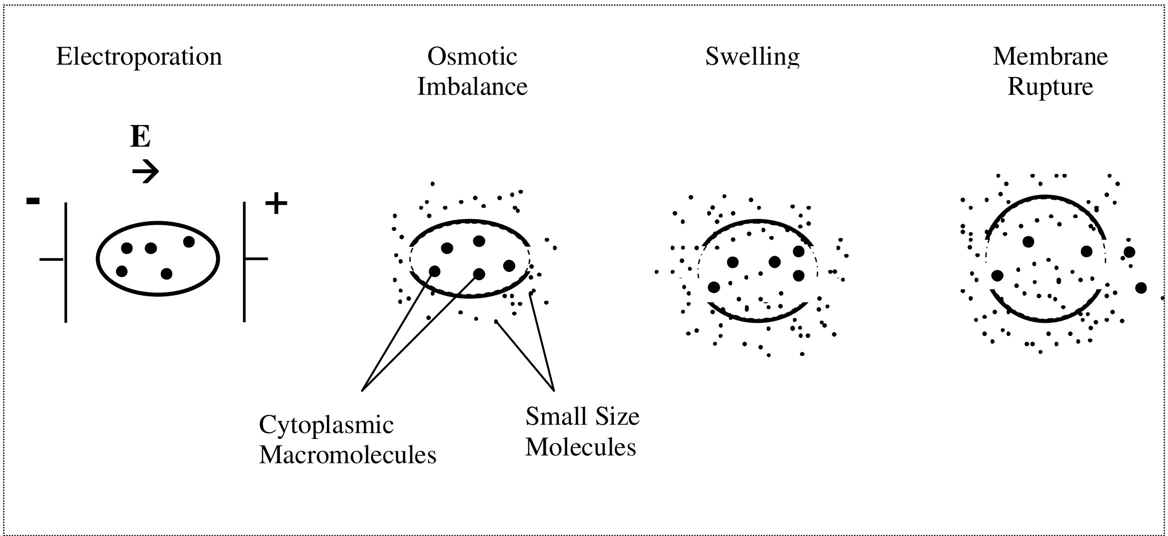

Figure 2.6. Electroporation and cell membrane rupture
(redrawn after Tsong, 1991)

2.3.3. Factors Influencing Pulsed Electric Field Processing

There are several factors influencing pulsed electric field processing. The process factors include electric field intensity, pulse duration, number of pulses, pulse shape and treatment temperature. Other factors can be stated as conductivity, pH, and ionic strength of the treatment medium and microbial factors including the type and concentration of the microorganisms present in the liquid to be treated. The main factors to be considered for pulsed electric field treatment are explained in the following sections.

2.2.3.1. Effects of the Process Factors. The lethal effect of pulsed electric field on the microorganisms increases with the increasing electric field intensity, pulse duration and numbers of pulses. Based on the mathematical approximation expressed by Hülsheger et al. (1981), Qin et al. (1995b) noted that the electric field intensity is more important for the inactivation microorganisms than the pulse number or pulse duration. Aronsson et al. (2001) explained that the interaction of these factors was also significant so that an increase in one parameter can intensify the effect of another one.

Another factor influencing the effectiveness of pulsed electric field processing is the form of the electric field pulses which may be double exponential, square-wave and oscillatory. The square-wave pulse is found to be more energy efficient form of PEF delivery (Zhang et al. 1997).

Beveridge et al. (2005) investigated the influence of the pulse duration on the inactivation of microorganisms by pulsed electric fields. They reported that shorter pulses provided a more energy-efficient inactivation for the conditions of their experiments. Moreover they tried to identify the most effective and energy-efficient pulse profile. They explained two forms of PEF delivery: monopolar and bipolar pulse profiles. They claimed that monopolar pulse profile provided superior inactivation compared to that is supplied by bipolar pulse profile when pulse duration was 1μs. But increase in the pulse duration resulted in a reduction of this superiority. When pulse duration was around 4 μs, bipolar pulse was appeared to be the superior pulse profile. They suggested that these observed differences might be attributed to the reorientation of the cells resulted from the second half of the bipolar pulse.

The temperature of the treatment medium is also an important factor for PEF processing. During pulsed electric field application, the temperature of the liquid to be treated is increased due to the ohmic heating. Although heat application alone supplies the inactivation of some microorganisms, it has a synergetic effect when combined with PEF application. Aronsson et al. (2001) used PEF intensity of 35 kV/cm and 40 pulses with 4 μs duration for the inactivation of microorganisms and they monitored the outlet temperature as 61^{o}C. This increase in the temperature was explained by the joule heating (electric energy dissipated as heat in a resistor). Although microorganisms might be inactivated at this temperature by heat alone, the exposure time was very short. As a result, the effect of heat generated during PEF processing was found to have an indirect effect on the bacterial inactivation. Aronsson and Ronner (2001) explained that the lethal effect of PEF treatment was related to the temperature of the medium in which cells were suspended because of the fact that it influences the membrane fluidity properties. At low temperatures, the phospholipids are closely packed into a rigid gel structure. At high temperatures they become less ordered and the membrane had a liquid-crystalline structure which affects the physical stability of the cell membrane. Aronsson and Ronner (2001) noted that according to Coster and Zimmermann (1975), the critical electrical field strength of the cell membrane decreased as the temperature of the medium was increased. As a result, the increase in the treatment medium temperature enhances the killing effect of pulsed electric field.

Heinz et al. (2003) studied the energy efficiency of inactivation of *Escherichia coli* inoculated in apple juice, by pulsed electric field treatment. They investigated the relation between the reduction in survivor count and electric field strength and treatment temperature. Their results showed that due to the effects of elevated treatment temperature (from 20-30 ^{o}C up to a range of 55-65 ^{o}C) the energy consumption could be reduced from above 100 kJ/ kg to less than 40 kJ/ kg while achieving 6 log cycles reductions in bacterial count. Heinz et al. (2003) suggested that preheating the apple juice provided a possibility to recover the dissipated electrical energy after treatment and this would lead to a reduction in the operational costs of the PEF treatment.

<u>2.2.3.2. Effects of the Microbial Factors.</u> The efficiency of pulsed electric field processing depends on the type and concentration of the microorganisms present in the liquid to be treated. Qin et al. (1998) used a continuous-flow pulsed electric field system for the inactivation of *Escherichia coli*, *Staphylococcus aureus* and *Saccharomyces cerevisiae*. They obtained more than six log cycles reduction in the number of viable cells while the liquid temperature was maintained below 40^{o}C. Qin et al. (1998) reported that among the three microorganisms, *S. cerevisiae* was the most susceptible to PEF treatment. The highest viability after PEF treatment was observed for *S. aureus*, compared to the other two microorganisms. Qin et al. (1998) attributed these results to the fact that the induced voltage across the cell membrane was proportional to the geometric size of the cell under study. *S. cerevisiae* with the largest geometrical size among these three cells exhibited the smallest critical field for breakdown. As a result, it was the most sensitive to PEF treatment. On the other hand, *S. aureus* was the smallest cell among the studied cells and

the induced voltage across the *S. aureus* membrane was smallest under the same PEF treatment conditions leading to lower inactivation rates.

Aronsson et al. (2001) investigated the influence of PEF processing parameters on *Escherichia coli*, *Listeria innocua*, *Leuconostoc mesenteroides* and *Saccharomyces cerevisiae*. In this study, the microorganisms were subjected to electric filed strengths of 25-35 kV/cm with durations of 2-4 μs and number of pulses of 20-40, in a continuous PEF treatment system. Aronsson et al. (2001) observed that that *E. coli* was considerably less sensitive to PEF treatment than *S. cerevisiae*, whereas *L. innocua* and *L. mesenteroides* were the most resistant of the organisms tested. They indicated that their results were in agreement with the general suggestion that gram-negative bacteria were more sensitive to PEF treatment than gram-positive bacteria. They explained this phenomenon by the more rigid and thicker cell wall of gram-positive bacteria which possibly constituted a protection to PEF treatment. Moreover, cell size was also found to be an important factor for PEF sensitivity. Aronsson et al. (2001) explained the effect of cell size on the PEF sensitivity of the microorganisms based on the membrane potential theory and the study of Zimmermann et al. (1976). The critical breakdown potential is lower for larger cell volumes. Therefore, *S. cerevisiae* with the largest cell size among the tested microorganisms, was found to be the most sensitive microorganisms to PEF inactivation (Aronsson et al., 2001).

<u>2.2.3.3. Effects of Conductivity, Ionic Strength and pH of the Medium</u>**.** The electrical conductivity of a medium (σ, Siemens/m) is the ability to conduct electric current. Liquids with large electrical conductivities have lower resistance and therefore generate smaller peak electric fields across the treatment chamber. As a result, the efficiency of pulsed electric field processing is decreased when the liquid to be treated has a large electrical conductivity. Similarly the increase in the ionic strength of the medium causes to the decrease in the inactivation rate supplied by PEF treatment since increase in the ionic strength lead to an increase in the conductivity of the medium.

One of the most important environmental factors influencing the microbial PEF sensitivity is pH of the treatment medium. Several studies demonstrated that the influence of pH on the sensitivity of microorganisms to PEF application depends on the microorganisms investigated (Gomez et al., 2005). Working on the inactivation of *L.*

plantarum by pulsed electric field Gomez et al. (2005) showed that this bacterium was more sensitive to pulsed electric field in a media of low pH. Treatment at an electric field intensity of 22 kV/cm with a treatment time of 400 μs resulted in 5.0, 2.8, 2.6 and 1.1 log reductions in the population of this microorganism at pH 3.5, 5.0, 6.5 and 7.0 respectively. Their results indicated that, as other Gram positive bacteria, *L. plantarum* was more PEF resistant at neutral pH than at acidic pH.

Garcia et al. (2004) examined the effectiveness of the bacterial inactivation by pulsed electric field at different processing conditions. Their results demonstrated that PEF treatment of Gram-positive bacteria at pH 4.0 and that of Gram-negative bacteria at pH 7.0 were more effective, when compared to PEF treatment of these bacteria at other pH conditions. Garcia et al. (2004) explained these results by the difference in the structure and composition between the cell wall of Gram-positive and Gram-negative bacteria. The authors indicated that more research is needed in order to determine the mechanisms of inactivation of Gram-positive and Gram-negative bacteria by PEF.

Devlieghere et al. (2004) reviewed the technologies for microorganism inactivation. They concluded that the effect of PEF could be increased by applying it in combination with other stressing factors such as water activity, pH and mild heat treatments. All of these factors had synergistic effects on the inactivation of microorganisms by PEF treatments. Devlieghere et al. (2004) noted that these synergistic effects were not clear and more researches were necessary to understand the mechanisms behind the effects of these factors. The summary of this section is given in Table 2.1.

Table 2.1. Summary of pulsed electric field processing

Target Bacteria	**Result (log reduction in microbial count)**	**Properties Fluid Medium**	**Process Conditions**	**Reference**
E.coli	6.0	T between 35 and 70 °C	Applied voltage: 8-40 kV/cm Specific energy input: 5-120 kJ/kg Pulse repetition rate: 2-95 Hz	Heinz et al. (2003)
E.coli,	2.4-8.0	T=30 °C	Applied voltage: 25-35 kV/cm Pulse length: 2-4 µs Number of pulses: 20-40	Aronsson et al. (2001)
L. innocua,	0.1-8.1			
L.mesenteroides	0.2-7.0			
S. cerevisiae	1.5-6.4			
E.coli	3.5	Initial T=10°C pH=4.0	Applied voltage: 30kV/cm Pulse length: 4 µs Number of pulses: 20 Flow rate: 41 mL/min Water activity:1.00	Aronsson and Ronner (2001)
	3.4	Initial T =10°C pH=5.0		
	1.4	Initial T =10°C pH=6.0		
	0.9	Initial T =10°C pH=7.0		
L. plantarium	5.0	T=35°C pH=3.5	Applied voltage: 25 kV/cm Treatment time: 400 µs	Gomez et al. (2005)
	2.8	T=35°C pH=5.0		
	2.6	T=35°C pH=6.5		
	1.1	T=35°C pH=7.0		
E.coli	> 6.0	T< 40°C	Applied voltage: 60 kV/cm Number of pulses: 50	Qin et al. (1998)
S. aureus			Applied voltage: 36 kV/cm Number of pulses: 40	
S. cerevisiae			Applied voltage: 30 kV/cm Number of pulses: 10	

2.3.4. Pulsed Electric Field as a Sludge Disintegration Method

The researches focused on the pulsed electric field technology mainly concern the inactivation of microorganisms in food industry. Destructive effect of pulsed electric field on the microorganisms can be used for the pretreatment of wastewater sludge but there are only a limited number of researches on this subject.

Abu-Orf and Dentel (2000) pretreated biosolids with electrical arc technology to enhance dewaterability by belt filter press. The pretreatment process used high-intensity electrical arc, 60 or 120 Hz and operated in short pulses between 10 to 50 kV. Their results revealed that electrical arc pretreatment increased cake solids by approximately 0.5 % to 1.5 %. Abu-Orf and Dentel (2000) noted that the technology's cost-effectiveness would depend on the benefits of this pretreatment versus equipment cost.

Loeffler et al. (2001) investigated treatment of sewage sludge by pulsed electric field. They proposed that pores are formed on the cell membranes under high voltage. As a result, the inner cell materials are poured into the surrounding water. They measured the degree of this destruction by the increase in the soluble chemical oxygen demand and the rate of the biological oxygen dissipation values. They concluded that results from electrical disintegration method are in a comparable range with the results from other mechanical disintegration methods. Loeffler et al. (2001) also claimed that the ohmic heating of the liquid to temperatures up to 45°C had a negligible effect compared to the effect of the pulsed electric field. Nevertheless, a combination of ohmic heating and high voltage pulses led to a higher destruction rate.

Kopplow et al. (2004) studied pretreatment of wastewater sludge by pulsed electric field. Using electric field intensities up to 30 kV/cm with specific energy inputs up to 8000 kJ/kg DS, Kopplow et al. (2004) obtained degree of COD release about 15 %. Kopplow et al. (2004) noted that pulsed electric field supplied foam reduction in the digesters and partial destruction of the filament bacteria. The results of their anaerobic biodegradation tests demonstrated that pulsed electric field pretreatment can improve VSS degradation rate about 9 % and biogas production rate about 20 %.

Choi et al. (2006) used electric pulse-power technology to disrupt sludge floc structure and to enhance digestibility of waste activated sludge (WAS). They used a 19kV pulsed power generator at 150 Hz with a coaxial electrode and 5-ring outer electrode. They observed that the ratio of soluble COD to total COD for pretreated sludge was increased 4.5 times, compared to control sludge. Moreover exocellular polymer content of sludge was increased 6.5 times due to the pre-treatment process. The anaerobic digestion results showed that pretreated sludge supplied 2.5 times higher gas production than that of control sludge. Repeating the anaerobic digestibility tests at different organic loading rates (OLR), Choi et al. (2006) noted that as OLR was increased, the difference between gas production rates (GPR) for the pretreated and control sludge was decreased. Choi et al. (2006) concluded that pulsed power pretreatment enhanced sludge solubilization leading to increased GPR during anaerobic digestion but when high OLR was applied, the methane forming bacteria was affected by accumulation of organic acids and the methane forming stage might became the rate limiting step.

Salerno et al. (2009) also tested pulsed electric field as a pretreatment method for improved biosolids digestion. Salerno et al. (2009) used treatment intensities up to 19.8 kWh/m^3 with applied electric fields up to 24.5 kV/cm and observed that approximately 10 % of the total COD was solubilized. Moreover Salerno et al. (2009) demonstrated that PEF pretreatment increased the biological methane potential of the samples: by 80 % for pig manure and 100 % for waste activated sludge.

2.3.5. Advantages and Disadvantages of Pulsed Electric Field Technology

Pulsed electric field technology is a promising technology as an environmental treatment process. Using pulsed electric field, sludge disintegration and improvements in the sludge biodegradability can be obtained. These would supply enhancement of the sludge destruction rates, reduction of the amount of sludge produced and increase in the biogas production rate during anaerobic digestion. Moreover pulsed electric field technology is a clean technology because it does not require the use of additional chemicals.

On the other hand this technology has some current limitations including high investment and operational costs. Moreover, the availability of commercial units is limited. Another limitation of the PEF processing is that it can be applied only to homogeneous liquids with low electrical conductivity having small sized particles because of the risks of dielectric breakdown, arcing and resulting electrolytic side effects. The presence of air bubbles in the liquid should also be eliminated because of the fact that the air bubbles may lead to operational and safety problems during pulsed electric field processing.

As a result, there exist many research areas to overcome the limitations of PEF processing, to develop cost effective generators and to scale up the system for industrial size continuous process.

3. STATEMENT OF THE PROBLEM

The amount of sludge produced from wastewater treatment plants is increasing because of the increase in the population and the amount of wastewater being treated. The treatment and disposal of sludge is very expensive and it can represent the most complex problem facing the engineer in the field of wastewater treatment (Metcalf and Eddy, 2003). Conventional sludge biodegradation processes are useful but they have some limitations in terms of long treatment times and large working areas requirements. In order to overcome these limitations, different sludge pretreatment methods are developed.

Sludge pretreatment by pulsed electric field (PEF) is a novel research frontier. Although there are some researches on sludge pretreatment by PEF, many related issues need to be studied. The effects of PEF on sludge dewaterability and aerobic biodegradability with the impacts of process parameters including electric field intensity, hydraulic retention time and pH conditions of the treatment medium should be investigated to develop this technology for future applications. The effects of pH conditions of the medium is of special interest because of the fact that the change in pH conditions might positively or negatively affect the efficiency of PEF processing, depending on different factors. The effect of PEF on the protein and carbohydrate components of the extracellular polymeric substances (EPS) in the sludge is another subject of interest since sludge dewaterability is highly dependent on the concentrations of these components.

The aim of this study is to investigate the performance of pulsed electric field application as a sludge pretreatment (disintegration) method prior to sludge dewatering and aerobic biodegradation. The objective of this study is to evaluate the effects of pulsed electrical field on the following sludge properties:

- Disintegration efficiency of PEF at different electric field intensities with different hydraulic retention time and different pH conditions;
- Dewaterability in terms of capillary suction time, specific resistance to filtration and compactibility;
- Protein and carbohydrate components of the EPS in the sludge;
- Aerobic biodegradability of the sludge

4. MATERIALS AND METHODS

4.1. Sludge Source

In this study, sludge samples were obtained from a laboratory scale, semi-continuous activated sludge reactor. The reactor was seeded with waste activated sludge taken from the municipal full-scale treatment plant of İSKİ at Paşaköy/İstanbul. The characteristics of the waste activated sludge samples obtained from laboratory scale reactor and the seed sludge are given in Table 4.1. The capacity of the laboratory scale activated sludge reactor was 6.4 L and the sludge retention time was adjusted to eight days. This reactor was fed by a synthetic feed solution with the composition given in Table 4.2. The reactor maintained at room temperature (20–25^{o}C) and air flux was supplied at a rate of 40 L/h to maintain aerobic conditions and adequate mixing. Distilled and deionized water was used to compensate evaporative losses.

Table 4.1. The characteristics of the seed sludge and waste activated sludge

Property	**Seed Sludge (From İSKİ Paşaköy/İstanbul)**	**Waste Activated Sludge From Laboratory Scale Activated Sludge Reactor**
pH	6.9	6.9
Alkalinity (mg/L as $CaCO_3$)	430	520
ORP (mV)	125	54
COD (g/L)	8.19	8.43
sCOD (g/L)	0.12	0.47
Alkalinity	430	520
TS (g/L)	7.05	11.12
TSS (g/L)	4.37	6.29
VS (g/L)	5.64	8.28
VSS (g/L)	3.09	4.31
TKN (mg/L)	258.1	327.5
Conductivity (µS/cm)	1,068	1,374

Table 4.2. The composition of the synthetic feed solution

Nutrient	**Amount (mg/L)**
Glucose	350
Peptone	78
Dipotassium Hydrogen Phosphate	600
Potassium Hydrogen Phosphate	300
Ammonium Chloride	225
Magnesium Sulfate Heptahydrate	112.5
Iron II Sulfate Heptahydrate	3.75
Zinc Sulfate Heptahydrate	3.75
Manganese Sulfate Heptahydrate	2.29
Calcium Chloride Dihydrate	19.86
Sodium Hydrogen Carbonate	180

4.2. Sludge Pretreatment (Disintegration) Methods

In this study, pulsed electric field (PEF) is applied to the sludge samples at different pH conditions as a sludge pretreatment method. Application of PEF to the sludge samples disintegrated the sludge cells. The pH values of the samples were adjusted to 3.0, 5.0, 7.0, 9.0 and 11.0 by adding 0.5 N H_2SO_4 or 0.5 N NaOH into the samples and mixing them for 30 minutes. Then, pulsed electric field was applied to the samples up to 30 kV/cm. After PEF application pH of the samples was readjusted to 7.0.

The laboratory scale pulsed electric field (PEF) generator system (Model No:TLG-07/1001) was purchased from Samtech Ltd. (UK). The system consists of a high voltage power supply and control unit, pulse forming network, rotary spark gap switch and a treatment chamber. The components of the PEF system and the block diagram showing the wiring layout of the PEF system are given in Figure 4.1 and 4.2 respectively.

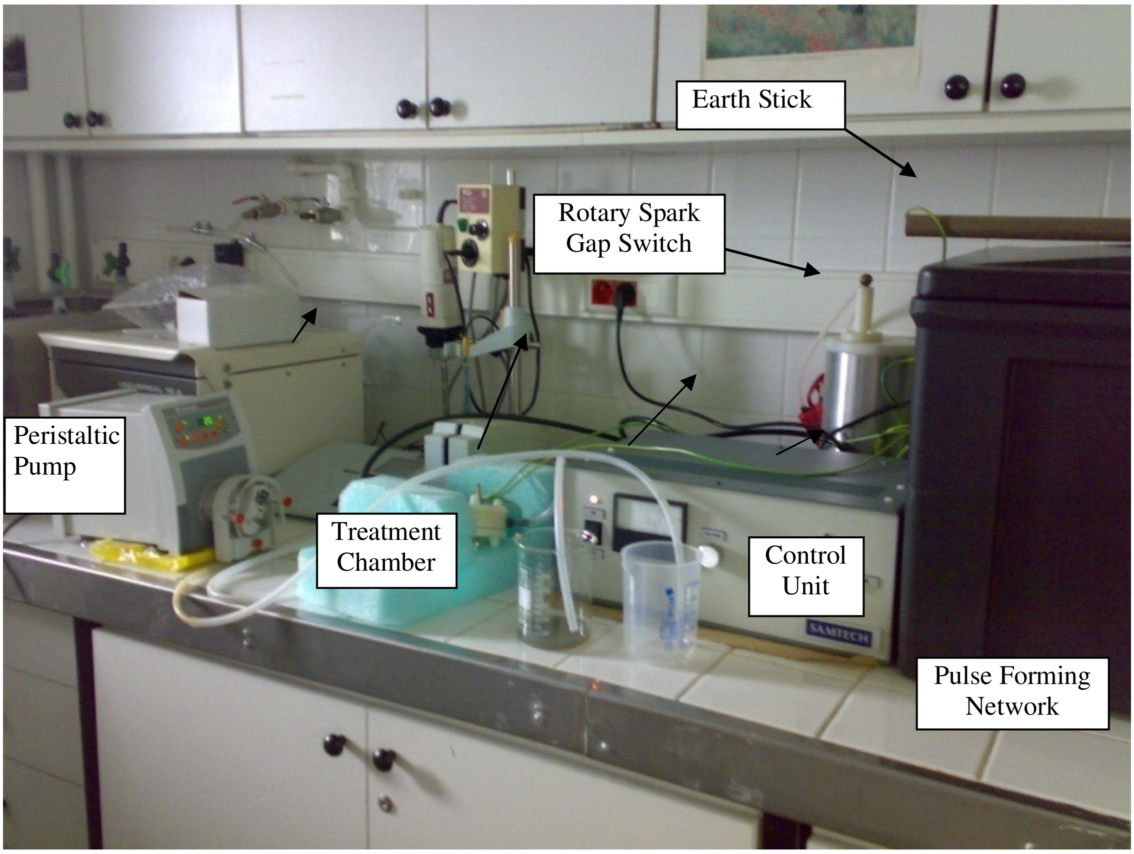

Figure 4.1. Pulsed electric field system

The system generates pulsed electric field up to 30 kV/cm using input voltage at 100-250 Vac, 50-60 Hz. The liquid to be treated is placed in the treatment chamber and the mains power is connected. Using the mains power, high voltage power supply charges the pulse forming network. When the system is fired, the rotary spark gap switch discharges the stored energy through the pulse forming network circuit to the liquid placed in the treatment chamber. The treatment chamber is shown in Figure 4.3.

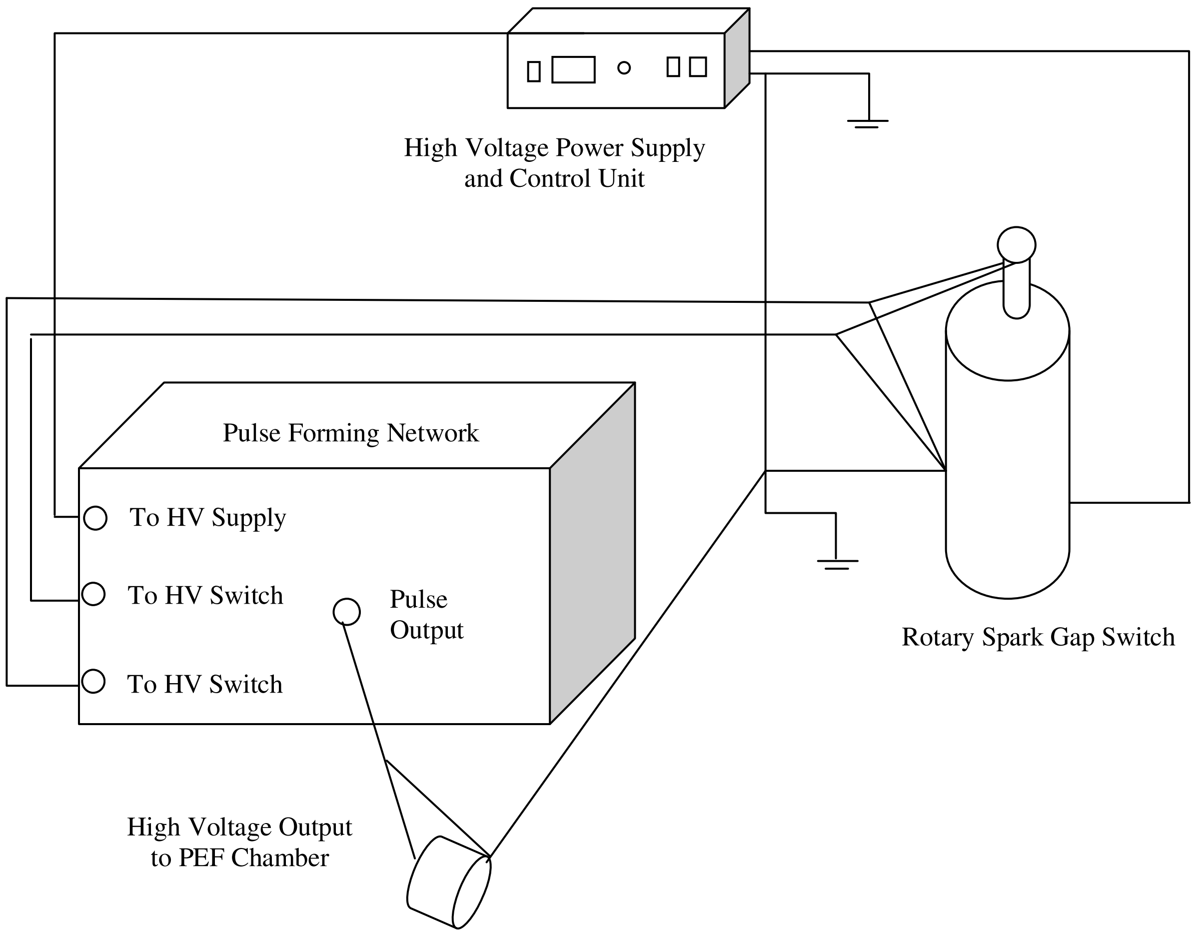

Figure 4.2. Wiring layout of the PEF system

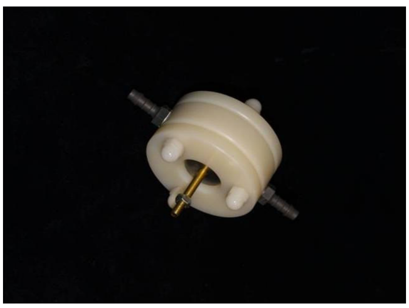

Figure 4.3. Treatment chamber of PEF generator

The generated pulse profile is monopolar rectangular with pulse duration of one microsecond. The pulse repetition rate is 0.5 pulses per seconds. The pulse profile is shown in Figure 4.4.

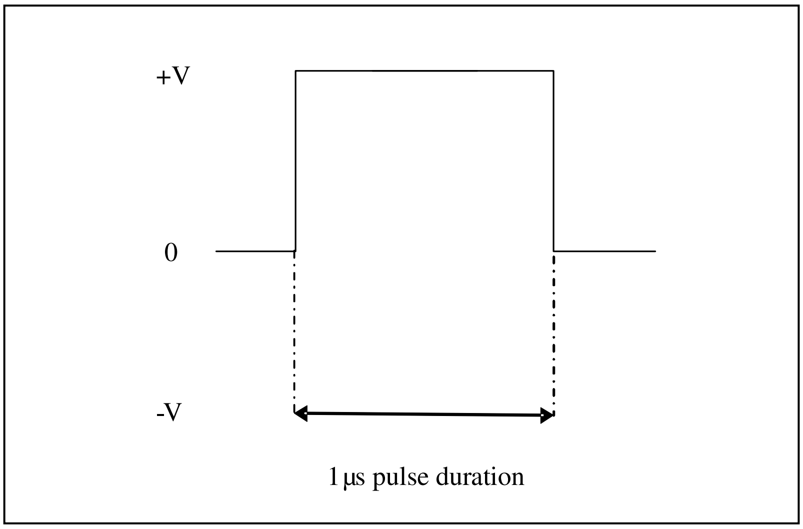

Figure 4.4. Pulse profile

4.2.1. Evaluation of Sludge Disintegration Degree

The results of sludge disintegration experiments were evaluated by using the following formula which describes the degree of sludge disintegration (Müller and Pelletier, 1998 , Bougrier et al., 2005).

$$DD_{COD} = (sCOD - sCOD_0)/(sCOD_{NaOH} - sCOD_0) \times 100 \quad (4.1)$$

where;

DD_{COD} : Degree of disintegration, %

sCOD : COD in the supernatant of the pretreated sludge, mg/L

$sCOD_0$: COD in the supernatant of the untreated sludge, mg/L

$sCOD_{NaOH}$: COD in the supernatant of the chemically disintegrated sludge in one mol/L NaOH for 24 h at 20°C , g/L. (4.24 g/L for the sludge used in this study)

4.3. Analytical Methods

Analytical methods used in this study are shown in Table 4.3. All of the experiments were duplicated and the arithmetic means of the experimental results are given in the study.

Table 4.3. Analytical methods used in the study

Analysis	**Method**	**Instruments**	**Reference**
Alkalinity (mg/L)	Titrimetric Method	WTW Inolab pH meter	APHA, 1998
Carbohydrate (mg/L)	Phenol-Sulfuric Acid Method	HACH DR/2010 Spectrophotometer	Rao and Pattabiraman, 1989
COD (mg/L)	Dichromate Closed Reflux Method	HACH COD Digester HACH DR/2010 Spectrophotometer	APHA, 1998
sCOD (mg/L)	Centrifugation and Dichromate Closed Reflux Method	Hettich Universal 16A Centrifuge HACH COD Digester HACH DR/2010 Spectrophotometer	Bougrier et al., 2008 APHA, 1998
Compactibility as cake solids concentration (%)	Centrifugation and Gravimetric Method	Hettich Universal 16A Centrifuge	Erdinçler and Vesilind, 2000 ; Vesilind, 1978
CST (s)	Instrumental Method	CST Instrument	Vesilind, 1988

Table 4.3. Analytical methods used in the study (Continued)

Analysis	**Method**	**Instruments**	**Reference**
Dissolved Oxygen (mg/L)	Electrometric Method	HACH HQ30d Single-Input Multi-Parameter Digital Meter	APHA, 1998
EPS Extraction	Extraction with CER	Jar Test Apparatus F.6/S , Hettich Universal 16A Centrifuge	Frølound et al., 1996.
pH and ORP	Electrometric Method	WTW Inolab pH meter and WTW SenTix ORP electrode	APHA, 1998
Protein (mg/L)	Modified Micro Lowry Method	HACH DR/2010 Spectrophotometer	Lowry et al., 1951
SRF (m/kg)	Filtration	Millipore Filtration Apparatus	Vesilind, 1978
TS (mg/L)	Gravimetric Method	Julabo Ekotemp TW12 Evaporator, Oven (103^{o}C), Desiccator	APHA, 1998
TSS (mg/L)	Gravimetric Method	Millipore filtration apparatus Oven (103^{o}C), Desiccator	APHA, 1998
VS (mg/L)	Gravimetric Method	Julabo Ekotemp TW12 Evaporator, Oven (103^{o}C), Furnace (600^{o}C), Desiccator	APHA, 1998
VSS (mg/L)	Gravimetric Method	Millipore filtration apparatus, Furnace (600^{o}C), Desiccator	APHA, 1998

4.3.1. Extraction of Extracellular Polymeric Substances

In this study, extracellular polymeric substances (EPS) in the sludge samples were extracted by using a strongly acidic cation exchange resin (Dowex 50*8, 20-5 mesh in the sodium form) obtained from FLUKA. For the EPS extraction, the following procedure was used, which was described by Frølound et al. (1996):

a) In order to remove any EPS from the bulk water of sludge, a washing procedure was performed before the extraction process. 40 mL of sludge samples were taken and centrifuged at 2000g for 20 minutes using centrifuge instrument (Hettich Universal 16A).

b) The supernatants were discarded and sludge pellets were resuspended to their original volume (40 mL) using a phosphate buffer solution consisting of 2 mM Na_3PO_4, 9 mM NaCl and 1 mM KCl at pH 7.0 .

c) The samples were transferred into the extraction beakers and cation exchange resin (Dowex 50*8, 20-5 mesh in the sodium form) was added into the samples at a dosage of 75 g/g VSS.

d) The prepared samples were stirred at 150 rpm for seven hours, using the Jar Test Apparatus F.6/S. In this extraction method, tightly bound EPS were shifted from sludge to liquid phase.

e) The extracted EPS were harvested by centrifugation of the samples for five minutes at 3650g to remove CER from the samples. The supernatants were again centrifuged twice at the same speed for 10 minutes to remove remaining floc components.

4.3.2. Protein Analysis

In this study, protein contents of the samples were determined by using Peterson's Modification Micro Lowry Total Protein Assay Kit, (Product Code: TP 0300 L3549) obtained from Sigma-Aldrich Inc. Bovin Serum Albumin (BSA) was used as standard. The procedure used for the protein analysis:

a) Two milliliters of samples was placed in test tubes.

b) Two milliliters of Lowry Reagent Solution was added to each test tube. The tubes were mixed well and allowed to stand at room temperature for 20 minutes.

c) One milliliter of Folin&Ciocalteau's Phenol Reagent was added into each test tube with rapid and immediate mixing.

d) The tubes were allowed to stand at room temperature for 30 minutes, for the color development.

e) The absorbance values of the prepared samples were measured at 750 nm, using HACH DR/2010 spectrophotometer. These values were compared to the data obtained from calibration curve which is given in Appendix A.

4.3.3. Carbohydrate Analysis

Carbohydrate content of the sludge samples were determined by using a modification of phenol-sulfuric acid method described by Rao and Pattabiraman (1989). Glucose was used as a standard. Carbohydrate analysis was carried out according to the following procedure:

a) Two milliliters of sample was taken in a glass test tube and six milliliters of concentrated sulfuric acid was added. The tubes were left to cool down.

b) Zero point one milliliter (0.1 mL) of 90 % phenol was added into the tubes. The mixture was allowed to stand at room temperature for 30 minutes, for the formation of yellow-orange color.

c) Absorbance values of the prepared samples were measured at 480 nm on the HACH DR/2010 spectrophotometer and compared to the calibration curve which is given in Appendix A.

4.3.4. Determination of Sludge Dewaterability

4.3.4.1. Capillary Suction Time. As described by Vesilind (1988), Capillary Suction Time (CST) analysis is a measure of sludge filterability. High CST values indicate the slow releasing of the liquid part of sludges and low CST values show the easy separation of sludge water. Figure 4.5. represents a schematic diagram of the CST instrument.

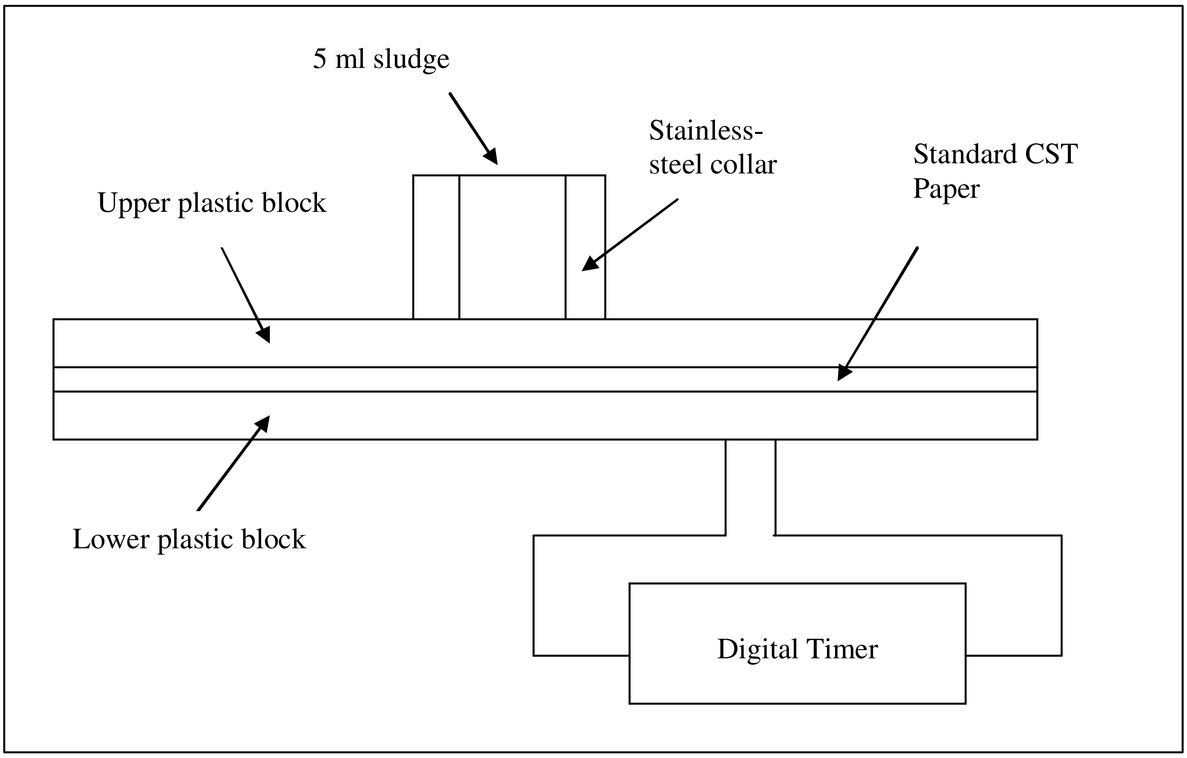

Figure 4.5. CST Apparatus

The CST instrument consists of two plastic blocks, a stainless-steel collar, three electrical sensors fixed on the upper plastic block, a piece of filter paper and an electrical timer (Vesilind, 1988). In this study, CST apparatus supplied by Venture Innovations, Inc. was used for the capillary suction time analysis. The CST paper (Whatman No:17 filter paper) is placed between two plastic blocks and five milliliters of sludge sample is poured into the stainless- steel collar. The poured sample flows through the CST paper forming a circular wet blot on the filter paper between the plastic blocks. The upper plastic block contains two circles with electrical sensors. The inner circle on which two sensors are present has a diameter of 3.2 cm and the outer circle with one sensor has a diameter of 4.6 cm. When the sample reaches the inner circle, the electrical signal starts the timer. After a period, the sensor of outer circle perceives the liquid part of the sludge sample and the timer is stopped. The time required to reach from the inner circle to the outer one was called as “Capillary Suction Time (CST)”, in seconds.

Capillary suction time test depends on the sludge solids concentration and on the instrument used. Temperature also affects the test due to its effect on viscosity (Vesilind, 1988). Temperatures of the samples during the experiments were adjusted to 25^{o}C.

4.3.4.2. Sludge Compaction Analysis. The compactibility of sludge samples were evaluated by using a centrifuge (Hettich Universal 16A). Fourty milliliters of sludge samples were centrifuged at 2,800 g (4,850 rpm) for 30 minutes. The degree of compaction was measured in terms of the cake solids concentrations of the compacted sludge layer (Erdinçler and Vesilind, 2000).

4.3.4.3. Specific Resistance to Filtration Analysis. In order to determine specific resistance to filtration (SRF) of the sludge samples, Millipore Filtration Apparatus and Whatman No:1 filter paper were used in this study. Fifty milliliters of sample was poured on the filter pad in the Büchner Funnel and the sample was allowed to drain by gravity for two minutes. Then, the vacuum pump, supplying a vacuum at 800 mbar was turned on. At every 15 seconds, the volume of filtrate was noted. The test was maintained until the vacuum breaks or the volume of filtrate did not change significantly between readings. Following the filtrations, the weight of the sludge cake deposited on the filter paper was determined by gravimetric analysis.

The following formula is used to calculate specific resistance to filtration of the sludge samples:

$$r = 2PA^2 b / (\mu w) \qquad (4.1)$$

where;

r: specific resistance to filtration , m/kg

P: pressure, N/m^2 (80,000 N/m^2)

A: area of filter, m^2 (1.73×10^{-3} m^2)

μ: dynamic viscosity, Ns/m^2 (1.002×10^{-3} Ns/m^2 at 20° C)

b: slope of the graphic of volume of filtrate versus time/volume (s/m^6)

w: solids deposited per volume of filtrate, kg/m^3 (6.3 kg/m^3)

In order to determine “b” values, volume of filtrate versus time/volume graphs were drawn and the slopes of these graphs were obtained. The slopes of these graphs were multiplied by 10^6 in order to convert them from s/mL^2 to s/m^6.

4.3.5. Other Methods

Chemical oxygen demand (COD) measurements were conducted using Dichromate Closed Reflux Method as described in Standard Method 5220 D (APHA, 1998). The absorbance values were measured using HACH DR/2010 spectrophotometer. Prepared calibration curve is given in Appendix A.

In this study, soluble chemical oxygen demand contents of the sludge samples were estimated according to Bougrier et al. (2008). The sludge samples at 5°C, were first centrifuged at 2,800 g for 15 minutes using centrifuge instrument (Hettich Universal 16A), then chemical oxygen demand of the supernatants were measured. Similarly, soluble protein and carbohydrate contents of the sludge samples were estimated by measuring the protein and carbohydrate contents of the supernatants.

pH and ORP of the samples were measured using WTW Inolab pH meter and WTW SenTix ORP electrode. Alkalinity of the samples was measured according to the standard method 2320 (APHA, 1998).

Total solids (TS), total suspended solids (TSS) and volatile solids (VS) measurements were conducted according to standard methods 2540 B, 2540 D and 2540 E respectively (APHA, 1998)

Total Kjeldahl Nitrogen (TKN) content of the samples was measured according to the Standard Method 4500 Norg-B (APHA, 1998). For these experiments 50 mL of sample was used.

4.4. Aerobic Biodegradability Tests

4.4.1. Semi-Continuous Aerobic Biodegradability Tests

Semi-continuous aerobic biodegradation experiments were carried out in four laboratory-scale reactors with effective volumes of 0.4 L, each. At the beginning of the experiments all of the reactors were filled with untreated sludge obtained from the laboratory scale activated sludge reactor. The sludge retention times in the reactors were adjusted 10 days by replacing 80 mL of digested sludge with control or pretreated sludge, once every two days. Two reactors were fed with untreated sludge and the others were fed with sludge pretreated by pulsed electric field.

The reactors were operated at room temperature (23–25^{o}C) and air flux was supplied in the bioreactors at a rate of 40 L/h to maintain aerobic conditions and to supply adequate mixing. During the operation of the aerobic reactors, evaporative losses were detected and these losses were compensated daily by adding distilled and deionized water. In order to control the operational stability, ORP, pH and temperature of the sludge samples taken from the reactors were measured once every two days. In order to measure the treatment efficiency, chemical oxygen demand (COD), soluble chemical oxygen demand (sCOD) of the sludge samples were measured once every two days. Total solids (TS) and volatile solids (VS) removal rates were measured once a week.

4.4.2. Batch Aerobic Biodegradability Tests

For the aerobic digestion studies, four batch aerobic bioreactors were used with working volumes of 0.4 L, each. Two control reactors were filled with untreated sludge obtained from the laboratory scale activated sludge reactor. The other two reactors were filled with a combination of untreated sludge and PEF pretreated sludge at 1:1 ratio. The reason of using PEF pretreated sludge in combination with untreated sludge is the fact that PEF pretreatment inactivates most of the microorganisms present in the sludge and untreated sludge can supply aerobic microorganisms that are necessary for aerobic digestion.

In order to supply adequate mixing and to maintain aerobic conditions, air flux was supplied in the digesters at a rate of 40 L/h. The reactors were operated at room temperature (23–25^{o}C) and evaporative losses were compensated daily by adding distilled and deionized water. Process parameters including ORP, pH and temperature in the reactors were measured once every two days. Chemical oxygen demand (COD), soluble chemical oxygen demand (sCOD), total solid (TS) and volatile solid (VS) contents of the influent and effluent sludge samples in the reactors were measured in order to evaluate the effects of sludge disintegration on the batch aerobic digestion.

5. RESULTS AND DISCUSSION

In order to investigate the effects of pulsed electric field (PEF) on the sludge disintegration, PEF at different intensities and different pH conditions was applied to the sludge samples obtained from laboratory scale activated sludge reactor. The characteristics of the sludge samples are given in Table 4.1.

The conductivity of sludge is an important factor in PEF pretreatment. The materials with large electrical conductivities generate smaller peak electric fields across the treatment chamber and therefore are not feasible for PEF treatment (Barbosa-Cánovas et al., 1999). Aronsson et al. (2001) obtained effective results by using PEF on the materials with conductivities adjusted to 2,000 µS/cm. The conductivity of the sludge used in this study was measured to be 1,374 µS/cm.

Another important factor in PEF processing is the pulse profile. The pulse profile supplied by the PEF generator used in this study is monopolar rectangular with duration of one microsecond. Beveridge et al. (2005) explained that monopolar pulse profile provided superior inactivation compared to that is supplied by bipolar pulse profile with pulse duration of one microsecond.

5.1. Effects of Pulsed Electric Field on Sludge Disintegration

During the solubilization experiments PEF intensities between 20 to 30 kV were used. The lower electric field intensities were not evaluated because that the COD solubilization results were not statistically significant at these intensities. The experiments were repeated at different hydraulic retention times between 3.0 and 9.0 seconds. Higher hydraulic retention times were not used in order not to damage the electrodes by electrical arching. The obtained results are tabulated in Table 5.1.

Table 5.1. Effects of PEF on sCOD of the sludge samples

PEF intensity (kV/cm)	**HRT (s)**	**sCOD (mg/L)**	**Increase in sCOD (%)**	**sCOD increase /TCOD (%)**	**DD_{COD} (%)**
0	0	471.6	0	0	0
20	3.0	540.8	14.7	0.8	1.8
20	4.5	580.4	23.1	1.3	2.9
20	6.0	596.9	26.6	1.5	3.3
20	7.5	633.2	34.3	1.9	4.3
20	9.0	652.9	38.4	2.2	4.8
25	3.0	577.1	22.4	1.3	2.8
25	4.5	633.1	34.3	1.9	4.3
25	6.0	686.0	45.4	2.5	5.7
25	7.5	715.6	51.7	2.9	6.5
25	9.0	771.0	63.6	3.6	7.9
30	3.0	620.7	31.5	1.8	4.0
30	4.5	685.9	45.4	2.5	5.7
30	6.0	784.8	66.4	3.7	8.3
30	7.5	827.7	75.5	4.2	9.5
30	9.0	880.5	86.7	4.9	10.9

The results demonstrate the ratio of the increase in soluble chemical oxygen demand (sCOD) to total chemical oxygen demand (TCOD) as well as the degree of disintegration (DD_{COD}) which is explained in section 4.2.1. The maximum degree of disintegration is obtained as 10.9 %, using electric field intensity at 30 kV/cm with hydraulic retention time of 9.0 seconds. The degree of disintegration values are higher than the ratio of the increase in sCOD to TCOD since degree of disintegration values are calculated based on the maximum sCOD that can be obtained by chemical sludge disintegration using NaOH (Formula 4.1). The obtained results are shown graphically in Figure 5.1.

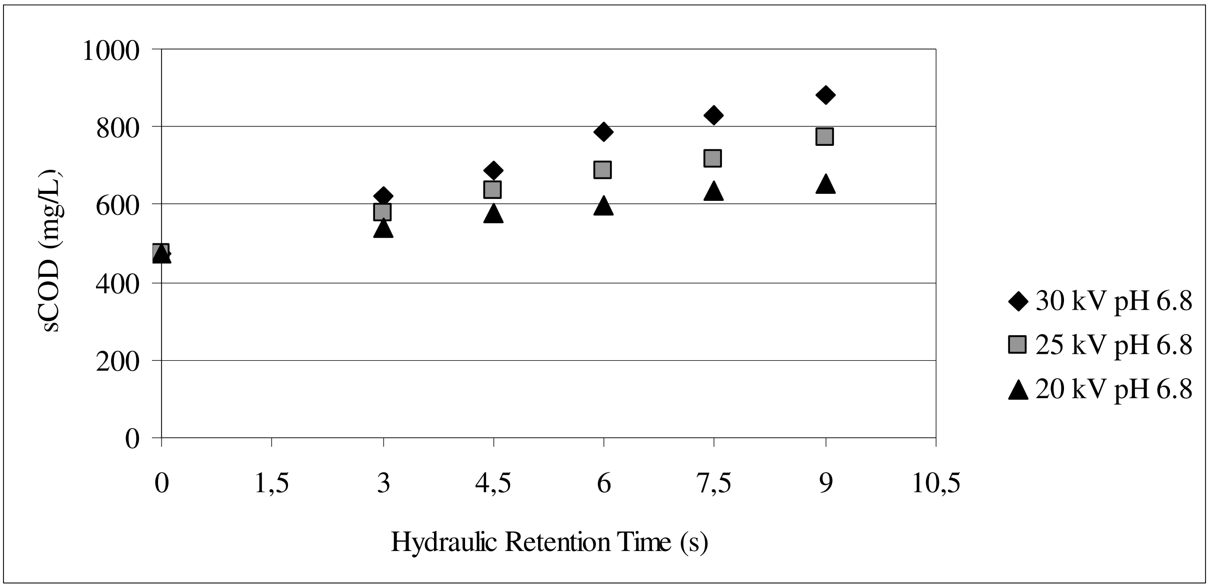

Figure 5.1. sCOD versus hydraulic retention time at different PEF intensities

These results demonstrate that the efficiency of pulsed electric field increased with increasing the electric field intensity and the hydraulic retention time. The increase in the soluble chemical oxygen demand of the sludge samples by the application of pulsed electric field can be explained by the disintegration of the floc structure and the destruction of the cell membrane of the microorganisms present in the sludge. As a result, the organic materials inside the floc structure and the cells of the microorganisms were released into liquid phase, leading to increased soluble chemical oxygen demand contents.

During these experiments, temperatures of the sludge samples were increased up to 1°C after pretreatment by pulsed electric field. This result demonstrates that ohmic heating is not the main mechanism of COD solubilization by pulsed electric field. Similarly, working on the bacterial inactivation by pulsed electric field, Aronsson and Ronner (2001) noted that the effect of heat generated during PEF processing had an indirect effect on the destruction of the cell membrane. When pulsed electric field is applied to a medium, the electric field itself causes to destruction of the cell membrane of the microorganisms present in the medium. The increase in the temperature of the medium during PEF treatment enhances of the killing effect of PEF by decreasing the critical electrical field strength of the cell membrane [Coster and Zimmermann (1975), Aronsson and Ronner (2001)].

The efficiency of pulsed electric field treatment depends on many factors. From the results shown in Figure 5.1, it can be concluded that the efficiency of pulsed electric field processing increased with increasing the electric field intensity and the hydraulic retention time. These results are in accordance with the studies of Qin et al. (1995b). They explained that the lethal effect of pulsed electric field on the microorganisms is increased as the electric field intensity, pulse duration and numbers of pulses are increased.

Many researches concerning the inactivation of microorganisms by pulsed electric field indicate that the stressing factors including heat application and changing the water activity of the treatment medium improve the efficiency of pulsed electric field processing. On the other hand this is not the case for the change in the pH conditions. The results of several researches demonstrated that the effect of pH conditions on the efficiency of PEF processing depends on the types of microorganisms present in the liquid to be treated [Aronsson and Ronner (2001), Gomez et al. (2005), Garcia et al. (2004)].

In this study, to investigate the effects of PEF at different pH values of the treatment medium, pH of the sludge samples was adjusted to 3.0 , 5.0 , 6.8 , 9.0 and 11.0 by adding 0.5 N H_2SO_4 or 0.5 N NaOH into the samples and mixing them for 30 minutes. Then PEF at 30 kV/cm was applied to the sludge samples. The obtained results are tabulated in Table 5.2.

Table 5.2. Effects of PEF on sCOD of the sludge samples at different pH conditions

pH	**HRT (s)**	**sCOD (mg/L)**	**Increase in sCOD (%)**	**sCOD increase /TCOD (%)**	**DD_{COD} (%)**
11.0	0	3235.7	586.1	32.8	73.4
	3.0	3264.7	592.3	33.1	74.2
	4.5	3292.1	598.1	33.5	74.9
	6.0	3318.6	603.7	33.8	75.6
	7.5	3331.5	606.4	33.9	75.9
	9.0	3367.1	614.2	34.3	76.9

Table 5.2. Effects of PEF on sCOD of the sludge samples at different pH conditions (continued)

pH	HRT (s)	sCOD (mg/L)	Increase in sCOD (%)	sCOD increase /TCOD (%)	DD_{COD} (%)
9.0	0	1464.6	210.6	11.8	26.4
	3.0	1509.3	220.7	12.3	27.6
	4.5	1527.8	224.0	12.5	28.0
	6.0	1532.8	225.5	12.6	28.2
	7.5	1571.6	233.2	13.0	29.2
	9.0	1593.1	237.8	13.3	29.8
6.8	0	471.6	0	0	0
	3.0	620.7	31.5	1.8	4.0
	4.5	685.9	45.4	2.5	5.7
	6.0	784.8	66.4	3.7	8.3
	7.5	827.7	75.5	4.2	9.5
	9.0	880.5	86.7	4.9	10.9
5.0	0	1014.5	115.1	6.4	14.4
	3.0	1067.8	126.4	7.1	15.8
	4.5	1093.2	131.8	7.4	16.5
	6.0	1106.8	134.7	7.5	16.9
	7.5	1131.8	140.0	7.8	17.5
	9.0	1183.6	151.0	8.4	18.9
3.0	0	2907.2	516.5	28.9	64.7
	3.0	2918.0	518.7	29.0	65.0
	4.5	2966.7	529.1	29.6	66.2
	6.0	3075.9	552.2	30.9	69.1
	7.5	3114.9	560.5	31.4	70.2
	9.0	3146.6	567.2	31.7	71.0

These results are shown graphically in Figure 5.2.

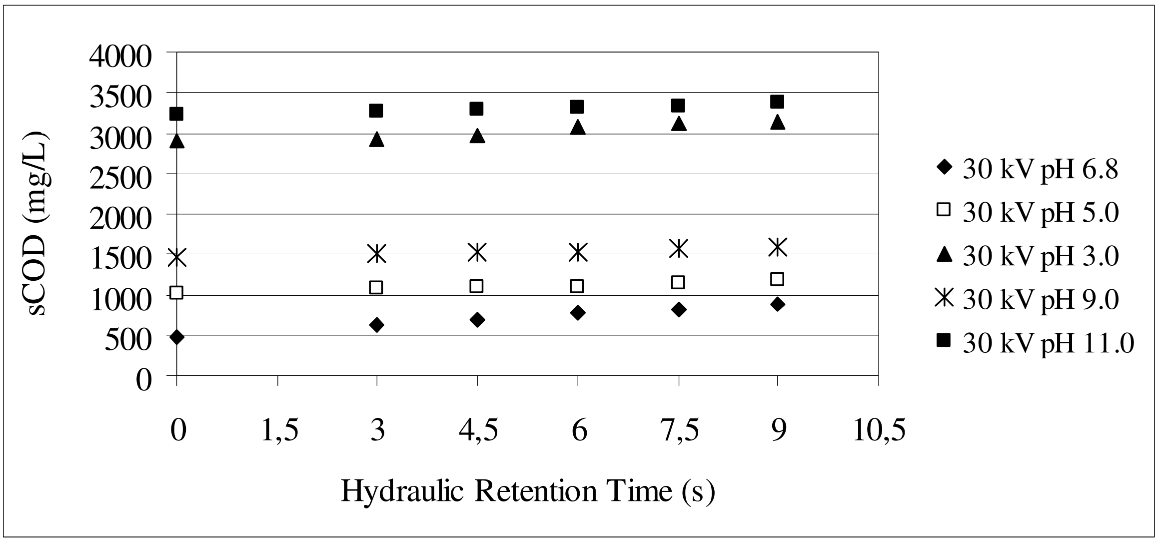

Figure 5.2. Effects of PEF on sCOD of the sludge samples at different pH conditions

The results show that pretreatment of the sludge samples by using acid or base increases the soluble chemical oxygen demand content of the sludge samples. The increases in sCOD contents of the samples were 115.1 % for acid treated sludge at pH 5.0 ; 210.6 % for base treated sludge at pH 9.0 ; 516.5 % for acid treated sludge at pH 3.0 and 586.1 % for base treated sludge at pH 11.0. Moreover pulsed electric field application combined with the pH adjustment of the treatment medium causes to further increase in sCOD values of the sludge samples. The maximum improvements in sCOD contents of the samples were 151.0 , 237.8, 567.2 and 614.2 % for the PEF pretreated sludge samples at 30 kV/cm with pH conditions of 5.0, 9.0, 3.0 and 11.0 respectively.

Observing the results, it can be concluded that although pulsed electric field pretreatment improves the sludge solubilization in terms of increased sCOD content, its effects were less drastic at acidic or basic pH conditions compared to the effects of acid or base pretreatment alone. One of the most important factors in PEF pretreatment is the type of the bacteria in the liquid to be pretreated. The majority of the bacterial genera in activated sludge are gram negative (Shagufta, 2007). Garcia et al. (2004) explained that PEF treatment of Gram-negative bacteria in a medium with neutral pH conditions was more effective than that in an acidic medium. On the other hand, Gram-positive bacteria were more PEF sensitive in an acidic medium than in a medium with pH 7.0. Garcia et al. (2004) explained these results by the difference in the structure and composition between the cell wall of Gram-positive and Gram-negative bacteria but they indicated that more research is needed in order to determine the mechanisms of inactivation of Gram-positive and Gram-negative bacteria by PEF.

5.2. Effects of Pulsed Electric Field on the Sludge Dewaterability

5.2.1. Specific Resistance to Filtration Analyses

In order to observe the effects of PEF on the specific resistance to filtration of the sludge samples, pulsed electric field (PEF) at 30 kV/cm with a hydraulic retention time of 9.0 seconds was applied to the sludge samples. Moreover, the effect of PEF with polymer conditioning was evaluated by repeating the SRF tests with conditioned sludge samples. For this purpose, a high molecular weight, cationic polymer (Zetag 7631) was applied to the sludge samples prior to the SRF tests. For the control sludge samples optimum polymer dose was determined as 3.2 mg/g dry solid and during the experiments, this amount of polymer was used.

The formula used to calculate SRF of the sludge samples is given in Section 4.3.6 and the graphics showing the measurements of filtrate volume over time for the sludge samples are shown in Appendix B. The results of specific resistance to filtration (SRF) analyses is given in Table 5.3.

These results demonstrate that sludge pretreatment by pulsed electric field causes to the deterioration of sludge filterability. Moreover, PEF pretreatment causes to the increase in the polymer amount needed for sludge conditioning. Similar results were obtained for the sludge pretreatment by sonication (Gonze et al., 2003). Using ultrasound at different energy levels up to 156 J/L, Gonze et al. (2003) observed that specific resistance of the sludge cake was increased from 6.4 x 10^{12} up to 2.6 x 10^{15}. They concluded that it was impossible to envisage performing ultrasound treatment prior to filtration step. Such a conclusion can also be drawn for pulsed electric field application on wastewater sludge, since it also deteriorates the sludge filterability.

Table 5.3. Effects of PEF application on SRF of the sludge samples (at pH=6.8)

Sludge sample	r Specific resistance to filtration (SRF) (m/kg)
Untreated sludge sample	418×10^{11}
PEF applied sludge sample	1183×10^{11}
Polymer conditioned sludge sample	56×10^{11}
PEF applied and polymer conditioned sludge sample	127×10^{11}

5.2.2. Capillary Suction Time Analyses

The effects of PEF on the capillary suction time (CST) of the sludge samples were evaluated by applying pulsed electric field (PEF) to the sludge samples at different electric field intensities up to 30 kV/cm with hydraulic retention time of 9.0 seconds. The results of PEF application with lower hydraulic retention times are not given here, since they did not supply significant effects. The results showing capillary suction time of the control and PEF applied sludge samples are given in Table 5.4.

Table 5.4. Effects of PEF application on CST of the sludge samples (at pH=6.8)

PEF intensity (kV/cm)	CST (s)
0	9.3
5	9.7
10	10.9
20	13.4
30	14.3

The obtained results demonstrate that capillary suction time (CST) of the sludge samples has a tendency to increase as the electric field intensity applied to the samples is increased. These results indicate that PEF treatment deteriorates sludge dewaterability, measured in terms of CST. Similar decline is observed for the application of ultrasound to the sludge samples. Chu et al. (2001) noted that after 60 minutes sonication of sludge at an energy level of 0.33 W/mL, the CST of the samples were increased from 197s for the untreated sludge up to 490s for the sonicated sludge. They attributed these results to the fact that a great amount of water could be attached on the large surfaces supplied by the small particles after ultrasonication. Similarly, Dewil et al. (2006) explained the deterioration of sludge dewaterability after sonication, by the reduction in the floc size after sonication, resulting in extended surface areas. As a result, more surface water was bound which led to the increase in the CST. Moreover the filterability of sludge was decreased because of clogging of the cake. Accordingly, the results obtained in this study which indicate the increase in the CST after PEF application can be explained by the disruption of the floc structure, reduction in the floc size and increase in the concentration of extracellular polymeric substances leading to the increase in the water holding capacity of sludge particles.

5.2.3. Compactibility Analyses

Sludge compaction analyses were conducted for both control (untreated) sludge sample and the sludge samples treated with pulsed electric field (PEF) at different electric field intensities up to 30 kV/cm with hydraulic retention time of 9.0 seconds. The compactibility of the sludge samples was measured by centrifuging the samples at 2,800 g for 30 minutes and measuring their cake dry solids. The results showing the compactibilities of the control and PEF treated sludge samples, in terms of cake dry solids contents are given in Table 5.5.

Table 5.5. Effects of PEF application on compactibilities of the sludge samples (at pH=6.8)

PEF intensity (kV/cm)	**Compactibility (cake dry solids content, %)**
0	6.51
5	6.57
10	6.82
20	7.19
30	7.53

As shown in the previous sections, sludge dewaterability, measured in terms of SRF and CST is decreased by the application of pulsed electric field. On the other hand, sludge compactibility, measured in terms of cake dry solids content increased with increasing the PEF intensity which indicates that sludge dewaterability by centrifugation can be improved by PEF treatment. The obtained results show that PEF pretreatment increased the cake dry solids contents of the centrifuged sludge samples from 6.51 % for the control sludge up to 7.53 % for the PEF pretreated sludge, indicating an increment by 1.02 %. Similar results were obtained by Abu-Orf and Dentel (2000), who pretreated biosolids with electrical arc technology. Using electrical field intensities between 10 to 50 kV, Abu-Orf and Dentel (2000) revealed that electrical arc pretreatment prior to belt filter press increased cake solids by approximately 0.5 % to 1.5 %. Abu-Orf and Dentel (2000) explained that the technology's cost-effectiveness would depend on the benefits of this pretreatment versus equipment cost. It was also noted that facilities with hard-to dewater solids might benefit from this technology.

Another related study is by Erdincler and Vesilind (2000), who demonstrated that sludge cell disruption improved the cake solids content up to 87 %, depending on the cell disruption method used. Erdinçler and Vesilind (2000) explained this improvement by that sludge cell disruption changed the water distribution in the biological sludge by releasing a considerable amount of interstitial water trapped inside sludge microorganisms and within the floc structure supplying an improvement in the sludge compactibility.

5.2.4. Impacts of Extracellular Polymeric Substances

Sludge dewaterability is highly dependent on the extracellular polymeric substances (EPS) present in the sludge. Although, high levels of EPS in the sludge result in the deterioration of dewaterability, there exists an optimum level of EPS content when the sludge dewaterability is maximized (Feng et al., 2009). This is explained by the fact that increasing EPS concentration above relatively low levels improves the extent of sludge flocculation (Sanin and Vesilind, 1994). Moreover the ratio of protein and carbohydrate components of extracellular polymeric substances in the sludge has an important role in the sludge dewaterability (Çetin and Erdinler, 2004).

In order to reveal the reasons of dewaterability results obtained in this study, protein and carbohydrate contents in the sludge samples, as well as those in the extracellular polymeric substances (EPS) present in the sludge samples were analyzed. Pulsed electric field at different electric field intensities with a hydraulic retention time of 9.0 seconds was applied to the sludge samples and the results showing the protein and carbohydrate solubilization are given in Table 5.6.

Table 5.6. Effects of PEF application on the protein and carbohydrate contents of the sludge samples

PEF Intensity (kV/cm)	Protein (mg eq BSA/L)	Carbohdrate (mg eq Glucose/L)	Soluble Protein (mg eq BSA/L)	Soluble Carboh. (mg eq Glucose/L)	Percent Disintegration Based on Protein Solubilization (%)
0	1153.8	1377.8	42.5	105.2	0
5	1148.6	1379.1	43.0	106.8	1.2
10	1154.5	1392.4	46.8	113.2	10.1
20	1159.3	1386.2	48.1	120.5	13.2
30	1162.0	1395.7	50.2	122.0	18.1

These results demonstrate that PEF application increases the soluble fraction of protein and carbohydrate contents of the sludge samples while it has a little effect on the measured amounts of total protein and carbohydrate contents. In order to evaluate the impact of sludge disintegration by PEF treatment on the protein and carbohydrate components of the EPS present in the sludge samples, EPS extraction process was performed for control sludge and PEF applied sludge. Then, the protein and carbohydrate contents of the extracted solutions were measured. The obtained results are tabulated in Table 5.7.

Table 5.7. Disintegration percent, protein and carbohydrate components of the EPS of control and PEF applied sludge samples

PEF Intensity (kV/cm)	**Percent Disintegration Based on Protein Solubilization (%)**	**DD_{COD} (%)**	**EPS Protein (mg eq. BSA /L)**	**EPS Carbohydrate (mg eq. Glucose/L)**
0	0	0	152	270.5
5	1.2	0.3	164.6	276.1
10	10.1	1.5	168.4	272.5
20	13.2	4.8	172.1	281.3
30	18.1	10.9	183.5	288.7

Observing the results given in Table 5.7, it is noted that the rate of increase in percent disintegration based on protein solubilization is higher than the rate of increase in DD_{COD} values at PEF intensities below 20 kV/cm. This result indicates that the disintegration kinetics of proteinaceous compounds is higher than that of other COD exerting compounds, at PEF intensities below 20 kV/cm. This result can be explained by cell disruption resulting in the release of high amount of proteinaceous compounds.

Using the data given in Table 5.7, the ratios of protein and carbohydrate components of extracellular polymeric substances (EPS) in the sludge were calculated. Figure 5.3 and Figure 5.4 represent the ratios of protein and carbohydrate components of EPS versus sludge disintegration percent with the impacts on CST and compactibility, respectively.

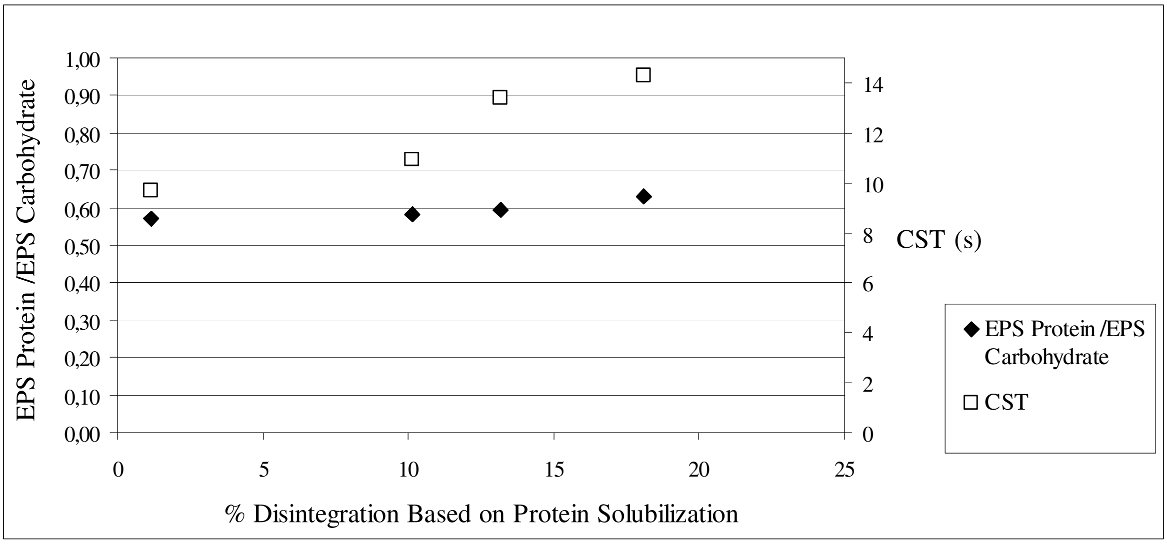

Figure 5.3. EPS protein/EPS carbohydrate and CST vs. disintegration percent

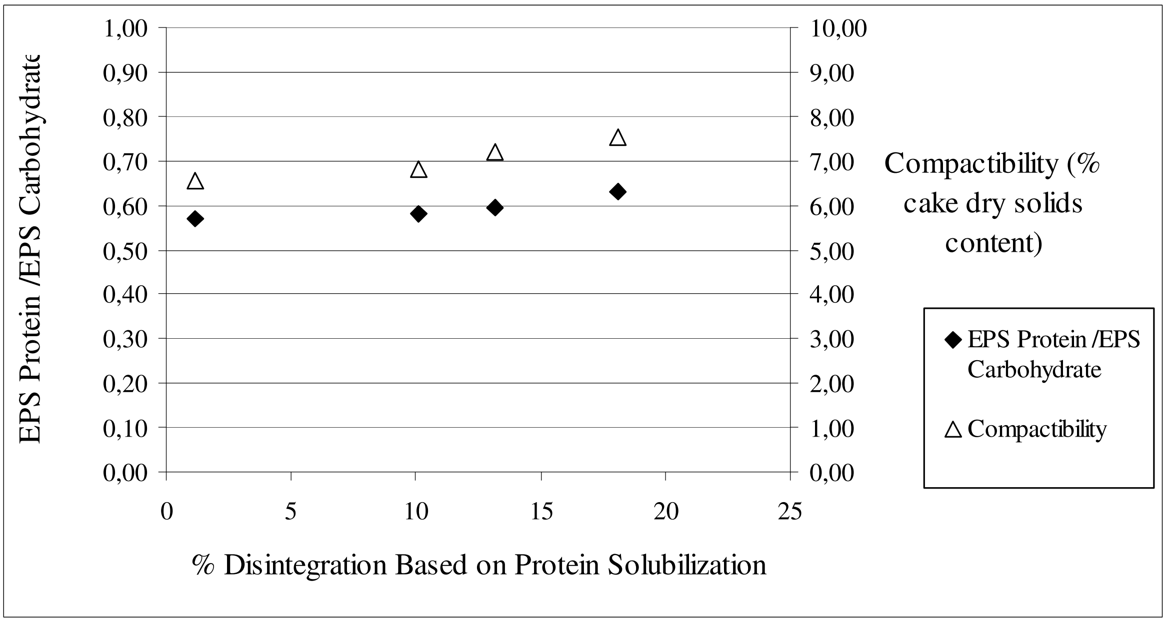

Figure 5.4. EPS protein/EPS carbohydrate and compactibility vs. disintegration percent

Observing the results given in Figures 5.3 and 5.4, it can be concluded that sludge dewaterability is highly dependent on the protein and carbohydrate content of the EPS present in the sludge. The increase in the intensity of the applied pulsed electric field supplies increase in the sludge disintegration percent resulting in the increase in both EPS protein and EPS carbohydrate contents of the sludge samples. The ratio of the increase in the EPS protein content is found to be higher than that in the EPS carbohydrate content as it can be seen from the increased EPS protein / EPS carbohydrate ratios. This result can be used to reveal the reason of the deterioration of sludge filterability after PEF application in terms of increased CST and SRF values. The increased ratio of EPS protein / EPS carbohydrate might detoriate sludge filterability because of water-holding capacity of proteinaceous part of EPS. Çetin and Erdinçler (2004) noted that the inverse relationship between sludge dewaterability and protein part of the EPS can be explained by the water-holding capacity of proteinaceous part of EPS. They also explained that, the increase in the carbohydrate fraction of EPS may have a stabilizing effect on the floc structure leading to larger and stronger flocs, thus supplying improvements in filterability and compactibility of sludge.

5.3. Effects of Pulsed Electric Field on the Aerobic Sludge Biodegradability

5.3.1. Semi-Continuous Aerobic Biodegradability

Semi-continuous aerobic biodegradation experiments were carried out in four laboratory-scale reactors. The operational conditions of these reactors are given in the Section 4.4.1. PEF pretreatment was applied at 30 kV/cm with a HRT of 9.0 seconds on the sludge obtained from laboratory scale activated sludge reactor. The feeding strategies for these reactors are explained below:

- Reactor S1 and S2 : Semi-continuous reactors fed with untreated sludge
- Reactor S3 and S4: Semi-continuous reactors fed with PEF pretreated sludge

During the aerobic biodegradation experiments, the reactors were operated at room temperature (23–25^{o}C) and the pH in the reactors were between 6.8 and 7.2. Dissolved oxygen contents in the aerobic reactors were kept above 2 mg/L by supplying air flux in the reactors at a rate of 40 L/h. The reactor performances between the dates 19.10.2009 and 06.12.2009 are given in Appendix C. In order to evaluate the effects of pretreatment on the reactor performances, COD and VS removals in the reactors were compared. The results demonstrating the COD and VS removals in the reactors are shown in Figure 5.5 and Figure 5.6, respectively.

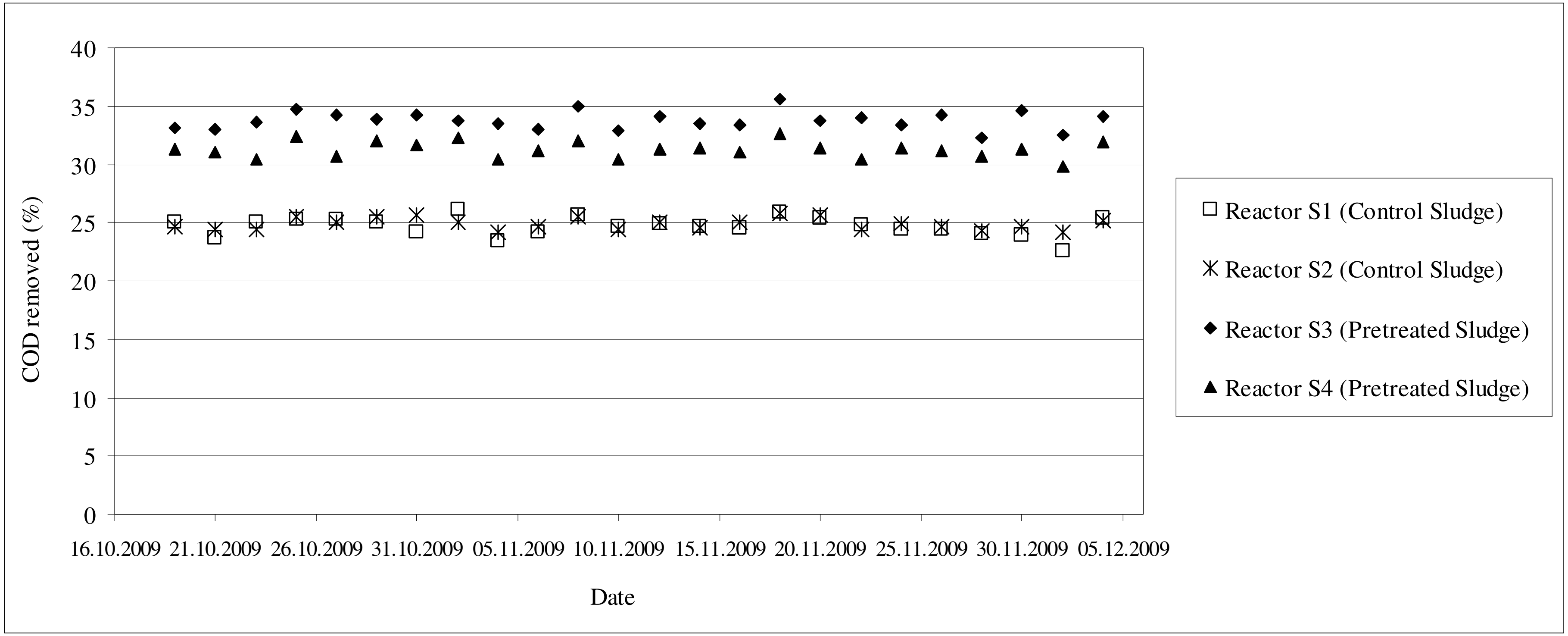

Figure 5.5. Mean COD removals in the semi-continuous aerobic reactors

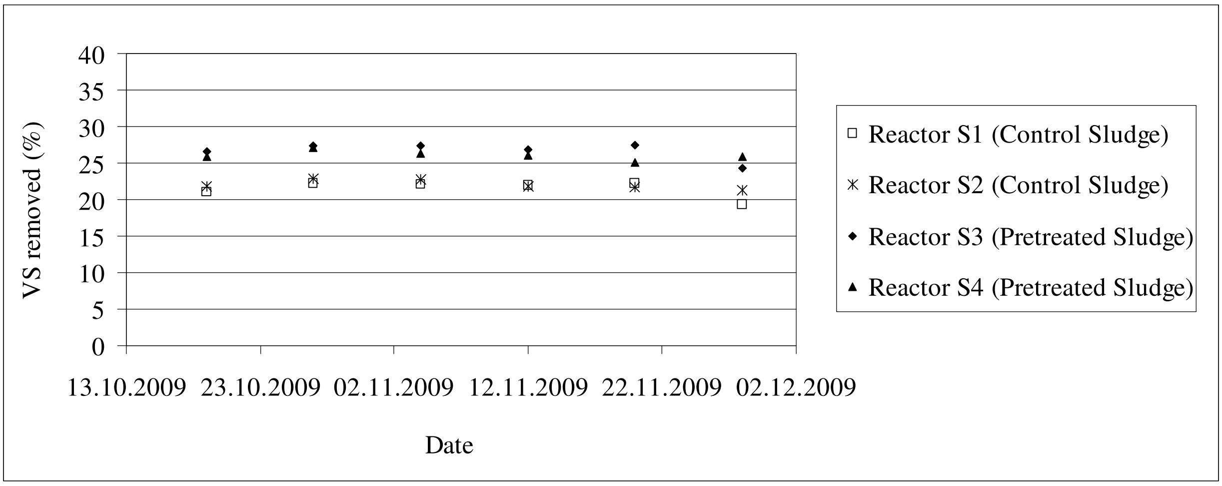

Figure 5.6. Mean VS removals in the semi-continuous aerobic reactors

During semi-continuous aerobic biodegradation tests, the mean COD removals were 24.7 , 24.9 , 33.8 and 31.3 % and the mean VS removals were 21.5 , 22.1 , 26.7 and 26.1 % for the reactors S1, S2, S3 and S4, respectively. Observing these results, it can be concluded that COD and VS removals were higher for the aerobic reactors fed with pretreated sludge by pulsed electric field when compared to the reactors fed with untreated sludge. The mean improvements supplied by PEF pretreatment were 7.8 and 4.6 %, for the COD and VS removals, respectively. Similarly, Yu et al. (2008) showed that pretreatment of excess sludge by ultrasound could improve its aerobic digestibility, leading to enhanced sludge reduction. They attributed these results to the enhancement of enzymatic activities by ultrasonic pretreatment. Yu et al. (2008) also stated that due to the ultrasonic pretreatment, extracellular proteins, polysaccharides and enzymes were shifted from pellet and tightly bound extracellular polymeric substances to slime layers of sludge flocs. The result is increased contact and interactions among extracellular proteins, polysaccharides and enzymes that were originally embedded in the sludge flocs, resulting in improved efficiency in aerobic digestion.

5.3.2. Batch Aerobic Biodegradability

Batch aerobic digestion experiments were carried out in four laboratory-scale aerobic reactors with 15 days operation periods. The operational conditions of these reactors are given in the Section 4.4.2. PEF pretreatment was realized by applying 30 kV/cm electric field intensity with a HRT of 9.0 seconds on the sludge obtained from laboratory scale activated sludge reactor. The feeding strategies for these reactors are explained below:

- Reactor B1 and B2 : Batch reactors fed with untreated sludge
- Reactor B3 and B4: Batch reactors fed with a combination of untreated sludge and PEF pretreated sludge, at 1:1 ratio.

The temperature in the reactors were kept between 23–25°C and the pH in the reactors were between 6.8 and 7.2. Air flux was supplied at a rate of 40 L/h to maintain dissolved oxygen contents in the reactors above 2 mg/L. The performances of these reactors are given in Appendix D. The effects of pretreatment on the reactor performances are evaluated by comparing COD and VS removals in the reactors which are shown in Figure 5.7 and Figure 5.8, respectively.

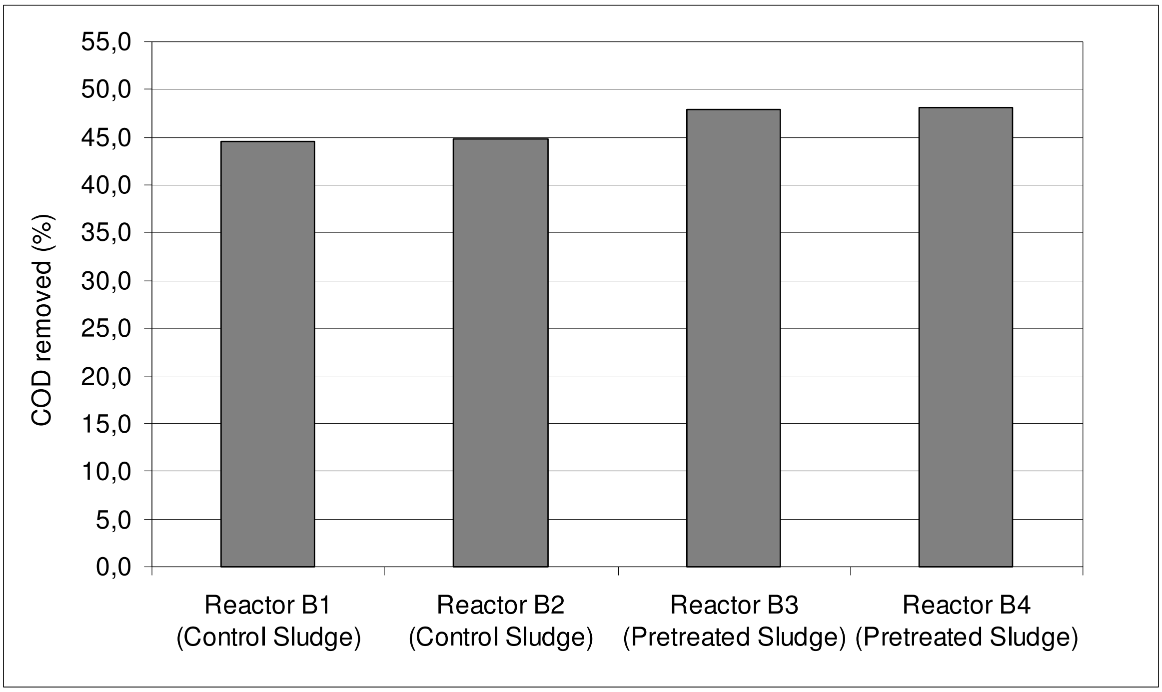

Figure 5.7. COD removals in the batch aerobic reactors

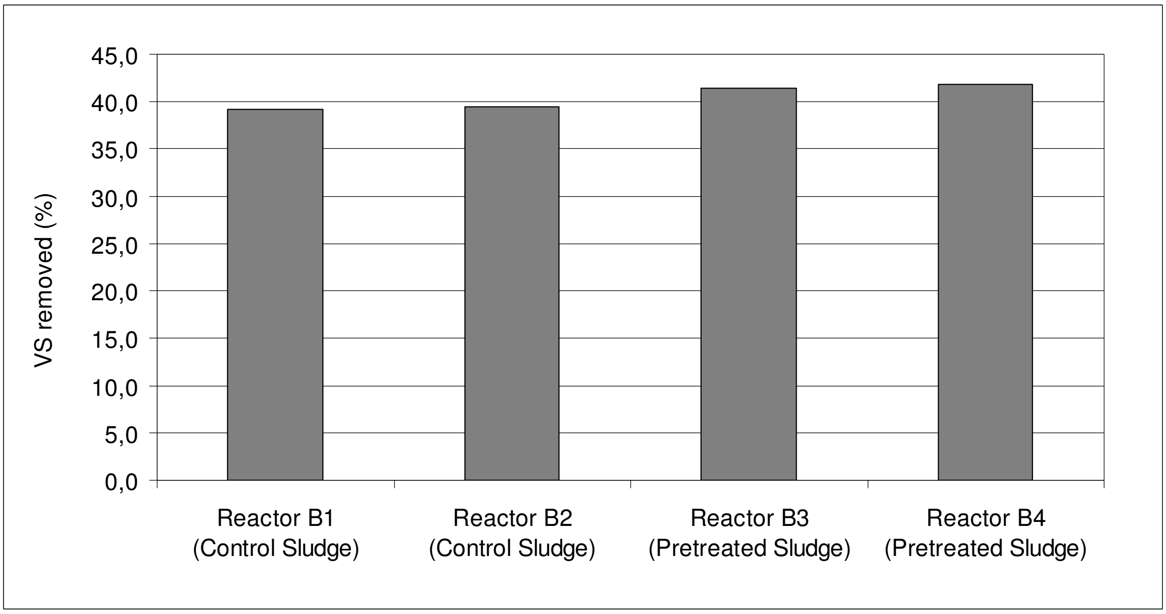

Figure 5.8. VS removals in the batch aerobic reactors

At the end of batch aerobic digestion tests, the mean COD removals were determined as 44.5 , 44.8 , 47.8 and 48.0 % and the mean VS removals were 39.3 , 39.5 , 41.4 and 41.8 % for the reactors B1, B2, B3 and B4, respectively. These results demonstrate that the mean improvements supplied by PEF pretreatment prior to batch aerobic digestion were 3.4 and 2.2 %, for the COD and VS removals, respectively. These values are lower than the mean improvements supplied by PEF pretreatment prior to semi-continuous aerobic biodegradation. These results can be explained by the fact that at the end of batch aerobic digestion experiments most of the biodegradable organic materials were digested. As a result, PEF pretreatment did not supply more improvements in terms of COD and VS removals for the batch aerobic reactors when compared to semi-continuous aerobic reactors.

6. CONCLUSIONS

This study investigates pulsed electrical field (PEF) as a sludge pretreatment method. PEF at different intensities between 5 and 30 kV/cm for hydraulic retention times between 3.0 and 9.0 seconds were applied to the sludge samples at different pH conditions. From the results of the study following conclusions can be drawn.

PEF pretreatment caused to disintegration of the sludge samples and improved their biodegradability by increasing the soluble fraction of chemical oxygen demand of the samples.

The disintegration degree of the sludge sample was 10.9 % when the applied electric field intensity was 30 kV/cm with hydraulic retention time of 9 seconds. COD solubilization experiments demonstrated that the effect of pulsed electric field increased with increasing the electric field intensity and the hydraulic retention time of the pretreatment.

Pulsed electric field pretreatment at pH values different than neutral leads to a further increase in the sludge disintegration degree, as a result of combined effect of acid or alkali addition. The disintegration degree of the sludge sample pretreated at 30 kV/cm with hydraulic retention time of 9 seconds was 71.0 % at pH 3.0 and it was 76.9 % at pH 11.0.

Pulsed electric field pretreatment caused to a decrease in the dewaterability of the sludge sample. The specific resistance to filtration was increased from 418 x 10^{11} m/kg to 1183 x 10^{11} m/kg after the PEF pretreatment. However, conditioning helped to obtain better dewaterability for PEF treated sludge samples compared to the untreated control samples. When the sludge samples were conditioned with a high molecular weight, cationic polymer prior to PEF application, SRF was found to be 56 x 10^{11} m/kg and 127 x 10^{11} m/kg, for control and PEF treated samples respectively.

The results of capillary suction time analyses, based on drainability, also showed that the dewaterability of the sludge samples was deteriorated after PEF pretreatment. The capillary suction time of the control sludge was increased from 9.3 seconds to 9.7, 10.9, 13.4 and 14.3 seconds after PEF application at electric field intensities of 5, 10, 20 and 30 kV/cm, respectively.

On the other hand, compactibility tests, based on centrifugation, showed that pulsed electric field pretreatment improved sludge compactibility. The measured compactibilitiy was increased from 6.51 % for control sludge sample to 6.57, 6.82, 7.19 and 7.53 % for the PEF applied sludge samples at electric field intensities of 5, 10, 20 and 30 kV/cm, respectively.

Based on the results of SRF, CST and compactibility tests, centrifugation seems to be the most appropriate method for dewatering of PEF treated sludge.

PEF pretreatment affected the EPS structure of sludge samples by increasing the ratio of EPS protein to EPS carbohydrate. When the applied field intensity was increased, the ratio of EPS protein to EPS carbohydrate was increased. The deterioration of sludge dewaterability after PEF application can be explained by the increased ratio of EPS protein to EPS carbohydrate. The high water-holding capacity of proteinaceous part of EPS decreases the dewaterability of the sludge.

The results of **semi-continuous** aerobic biodegradation experiments demonstrated that pulsed electric field pretreatment improved the efficiency of aerobic biodegradation of sludge in terms of COD and VS removal percents. The mean COD removal percent was increased by 7.8 % and the mean VS removal percent was increased by 4.6 % for the reactors fed with PEF pretreated sludge compared to the control reactors. The improvement in the aerobic biodegradation rate of sludge is explained by the disruption of the floc structure and sludge cells by PEF pretreatment. Thus, the organic materials in the sludge become more available to aerobic biodegradation leading to increased COD and VS removal percents.

According to the **batch** aerobic digestion experiments, the mean COD removal percent was increased by 3.4 % and the mean VS removal percent was increased by 2.2 % for the reactors fed with PEF pretreated sludge compared to the control reactors. Lower improvements obtained for batch reactors compared to those for semi-continuous reactors after PEF pretreatment can be explained by that the most of the biodegradable organic materials were digested during batch biodegradation process and PEF pretreatment did not supply more improvements in terms of COD and VS removals for the batch reactors when compared to the semi-continuous reactors.

After pretreatment by pulsed electric field, the temperature of the sludge samples was increased up to 1°C. This result demonstrates that the main factor in the pulsed electric field pretreatment is the applied electric field itself rater than ohmic heating.

7. RECOMMENDATIONS FOR FUTURE WORK

This study has demonstrated that pulsed electric field pretreatment can supply sludge disintegration leading to increase in the soluble chemical oxygen content of the wastewater sludge. The result is improved sludge compactibility, as well as improved aerobic biodegradability. It is recommended to evaluate sludge pretreatment by pulsed electric field to improve anaerobic biodegradation rates. The obtained improvements should be considered with cost-benefit analyses before implementation of this technology in full scale treatment plants. Further studies are needed to overcome the current limitations of pulsed electric field technology including high investment and operational costs. Moreover, researches on the development of high rate pulsed electric field equipments and process optimization are recommended.

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APPENDIX A: CALIBRATION CURVES

The calibration curves prepared for protein, carbohydrates and chemical oxygen demand (COD) analyses are given in Figures A.1, A.2 and A.3 respectively.

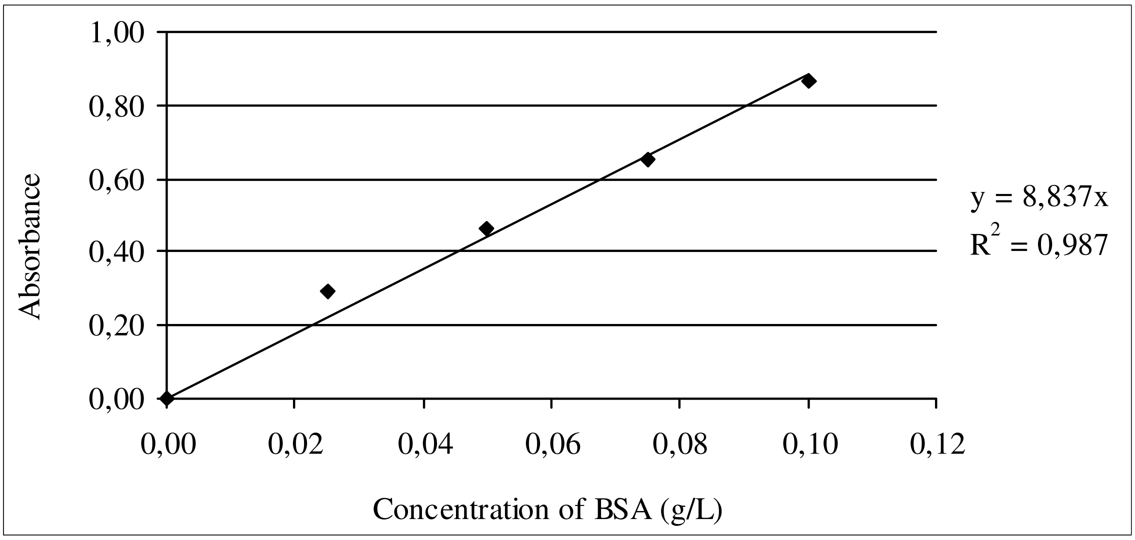

Figure A.1. Calibration curve prepared for the protein analysis

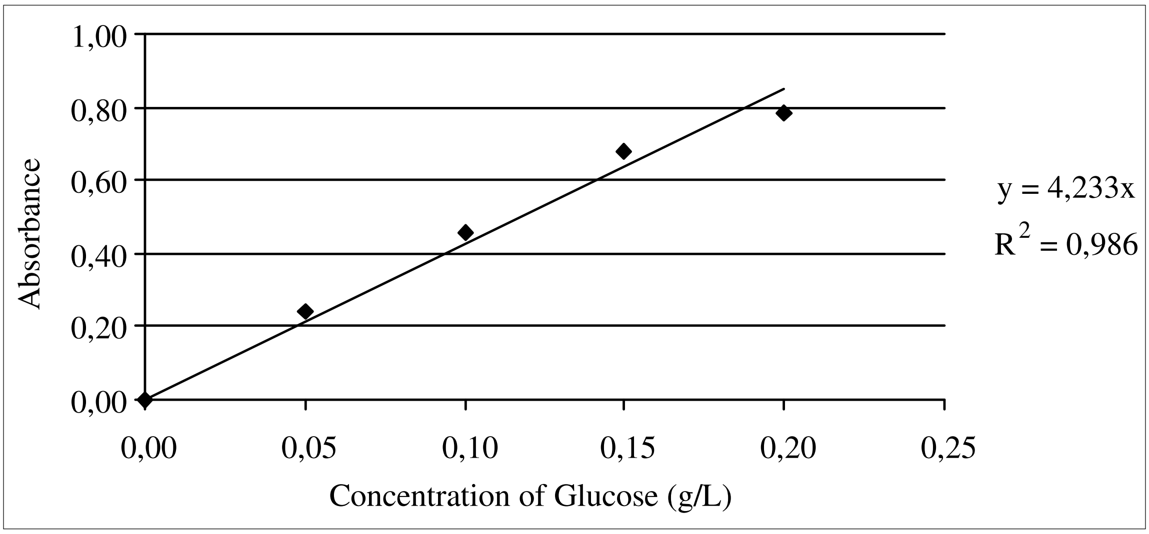

Figure A.2. Calibration curve prepared for the carbohydrate analysis

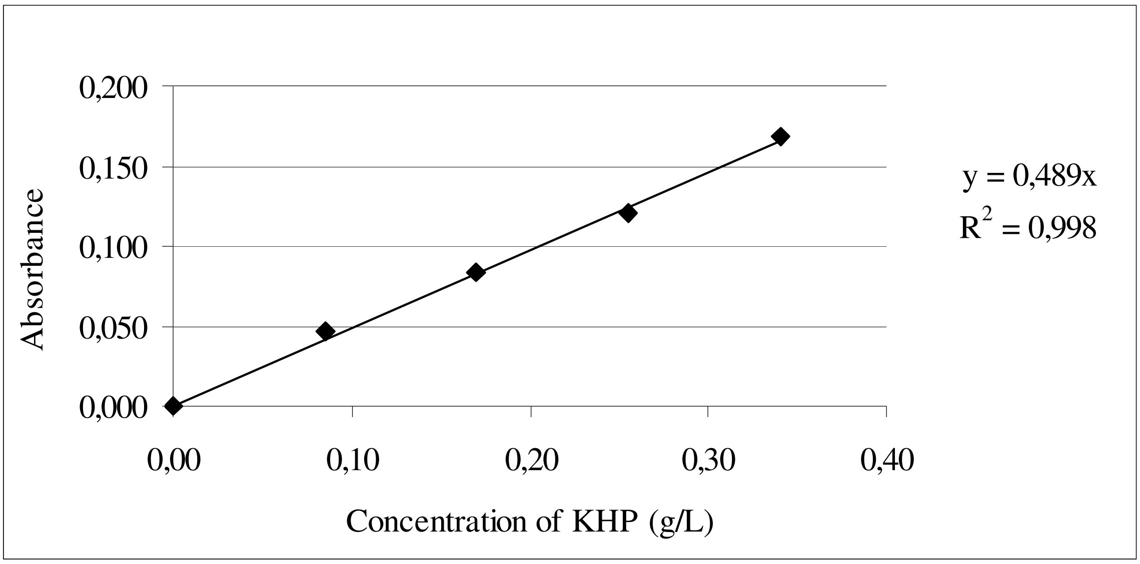

Figure A.3. Calibration curve prepared for the chemical oxygen demand analysis

APPENDIX B: SPECIFIC RESISTANCE TO FILTRATION DATA

The graphics of volume versus time/volume during the filtration of control sludge, PEF applied sludge, polymer conditioned sludge, PEF applied and polymer conditioned sludge are given in Figures B.1, B.2, B.3 and B.4, respectively.

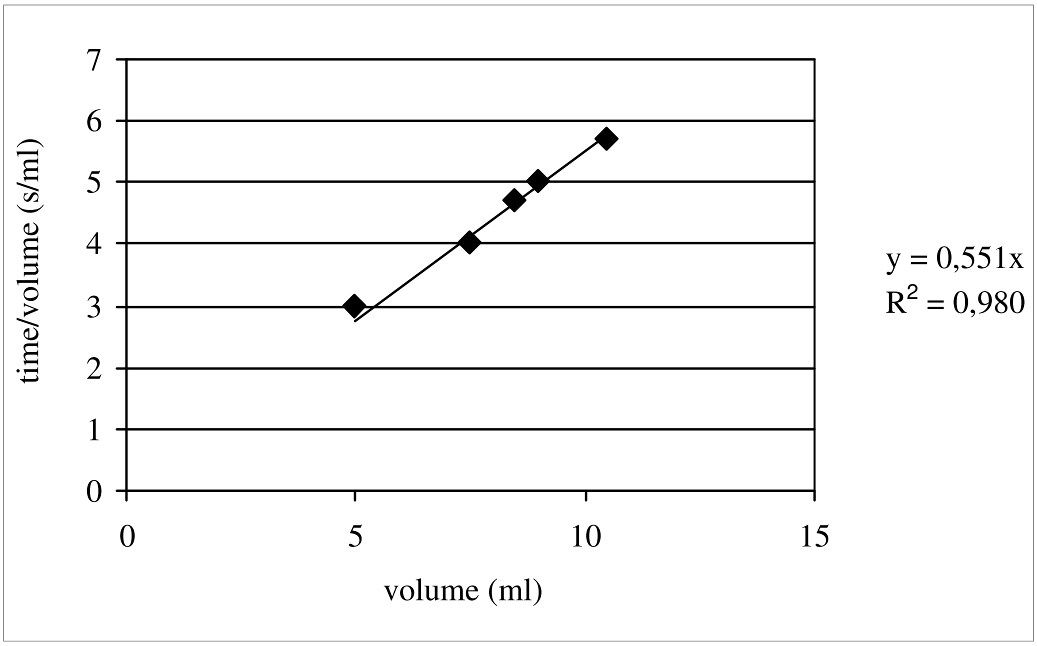

Figure B.1. Volume versus time/volume during the filtration of control sludge

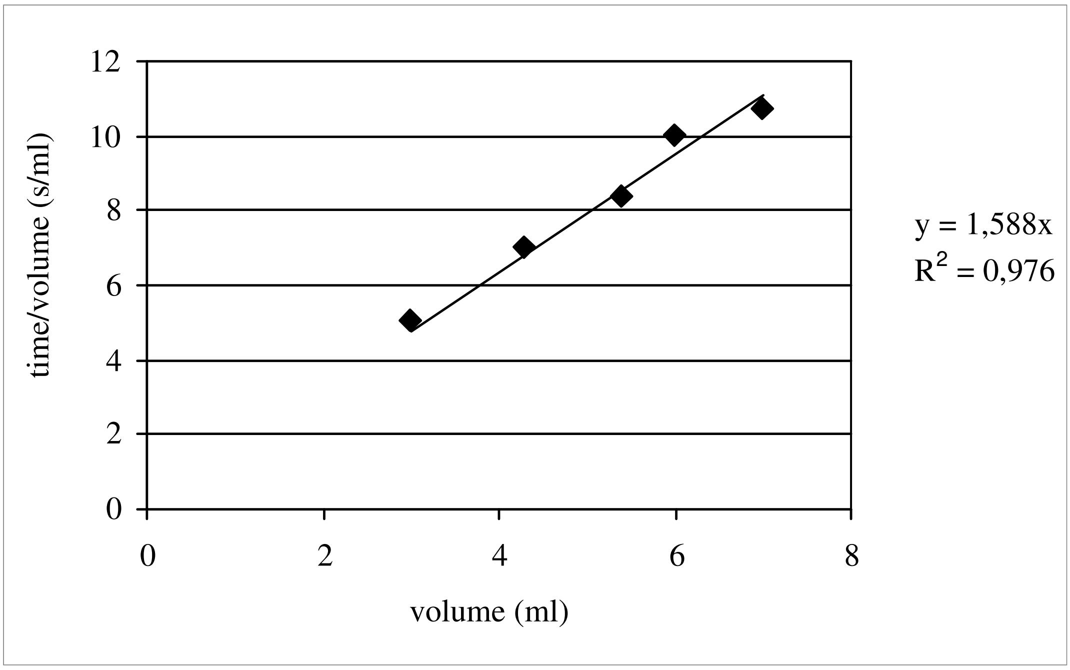

Figure B.2. Volume versus time/volume during the filtration of PEF applied sludge

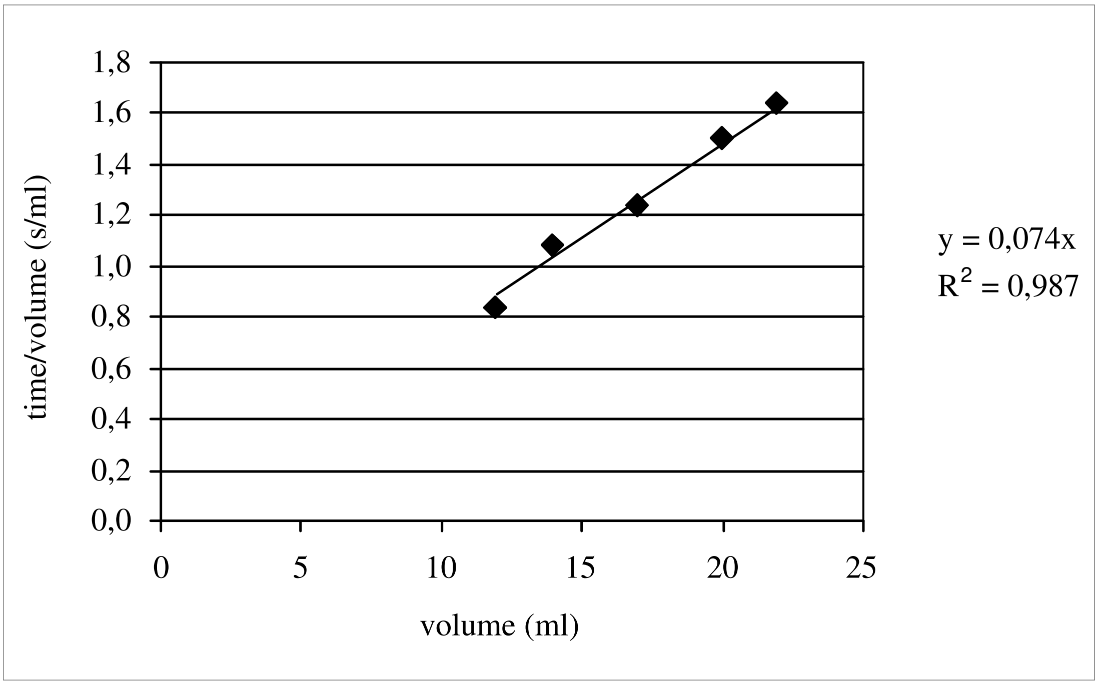

Figure B.3. Volume versus time/volume during the filtration of polymer conditioned sludge

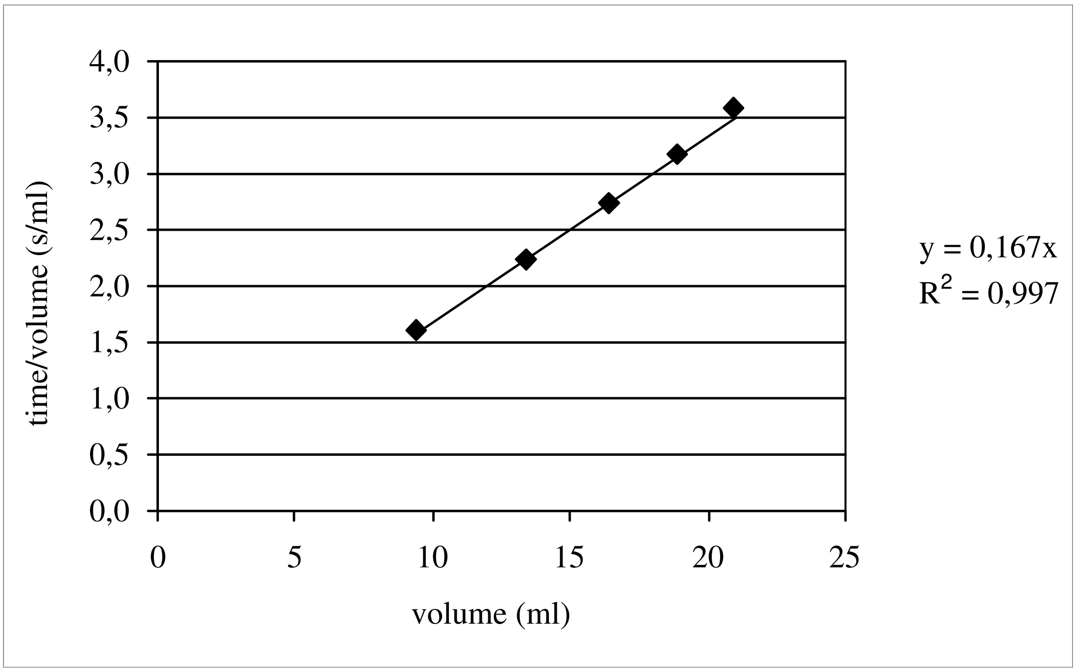

Figure B.4. Volume versus time/volume during the filtration of PEF applied and polymer conditioned sludge

APPENDIX C: PERFORMANCES OF THE SEMI-CONTINUOUS AEROBIC REACTORS

Table C. Mean sludge properties in the semi-continuous aerobic reactors

Property	Reactor 1	Reactor 2	Reactor 3	Reactor 4
Influent pH	7.01	7.01	7.04	7.04
Effluent pH	6.87	6.93	6.96	6.89
Influent ORP (mV)	65	65	64	64
Effluent ORP (mV)	125	117	131	135
Influent COD (g/L)	8.43	8.43	8.43	8.43
Effluent COD (g/L)	3.19	3.17	2.38	2.57
Influent sCOD (g/L)	0.49	0.49	0.91	0.91
Effluent sCOD (g/L)	0.19	0.21	0.29	0.31
Influent TS (g/L)	11.07	11.07	11.07	11.07
Effluent TS (g/L)	6.85	6.71	6.49	6.65
Influent VS (g/L)	8.57	8.57	8.57	8.57
Effluent VS (g/L)	4.57	4.53	4.14	4.21
Dissolved oxygen content in the reactors (mg/L)	2.59	2.46	2.52	2.63
COD * removal (%)	24.7	24.9	33.8	31.3
VS * removal (%)	21.5	22.1	26.7	26.1

* In the calculation of percent COD and VS removals, mixed liquor COD and VS values were accepted as initial COD and VS values.

APPENDIX D: PERFORMANCES OF THE BATCH AEROBIC REACTORS

Table D. Mean sludge properties in the batch aerobic reactors

Property	Reactor 1	Reactor 2	Reactor 3	Reactor 4
Influent pH	6.98	6.98	7.02	7.02
Effluent pH	6.92	6.97	6.86	6.94
Influent ORP (mV)	58	58	53	53
Effluent ORP (mV)	143	138	132	137
Influent COD (g/L)	8.41	8.41	8.41	8.41
Effluent COD (g/L)	4.67	4.64	4.38	4.37
Influent sCOD (g/L)	0.46	0.46	0.69	0.69
Effluent sCOD (g/L)	0.16	0.15	0.15	0.18
Influent TS (g/L)	11.03	11.03	11.03	11.03
Effluent TS (g/L)	7.78	7.76	7.59	7.56
Influent VS (g/L)	8.19	8.19	8.19	8.19
Effluent VS (g/L)	4.98	4.96	4.80	4.76
Dissolved oxygen content in the reactors (mg/L)	2.67	2.58	2.54	2.61
COD removal (%)	44.5	44.8	47.8	48.0
VS removal (%)	39.3	39.5	41.4	41.8