Novel conversion of waste activated sludge to Class A biosolids with support of electrokinetics

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Abstract

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Bianca Jitaru

Waste activated sludge (WAS), a common by-product of WWT requires treatment before reuse or disposal. WAS constituents include heavy metals and pathogens that can pose serious health concerns. Electrokinetics a novel and versatile technology used in sludge treatment can accomplish Class A biosolids. In this thesis, the effects of electrokinetic treatment in conjunction with enhancers on WAS were assessed. The experiment, done at a lab scale used a low voltage gradient under 5V/cm and low concentrations of BioxyS, (organic, non-toxic agent that acts as a biocide) and ammonium salts. The reaction took place in a BioElectro reactor composed of two stainless steel electrodes and a lid comprising of 18 silver probes designed to increase conductivity. Three different sources of WAS were used: WAS 0.6 % total solids (TS), WAS 5% TS and WAS 6% TS belonging to two different WWTPs. The samples of WAS depending on their total solids content reached required temperature at different time but always not more than 2.2 hours retention time. Also, higher voltage gradient (less than 5V/cm) permitted to reach the required temperature faster. For example, (5% TS) reached 65°C in approximately 55 minutes at a higher voltage gradient and in 130 minutes at a lower voltage gradient. Testing for E. Coli and total CFU showed that the biosolids that underwent BioElectro treatment with enhancers can be categorized as Class A and can therefore be dedicated to land application.

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List of abbreviations

AN	ammonium nitrate
[AN]	ammonium nitrate concentration
BS	BioxyS
[BS]	BioxyS concentration
CO_2	carbon dioxide
CFU	coliform forming units
CH_4	methane
DAED	diacetyl ethylene diamine
EK	electrokinetic
FC	fecal coliform
H_2O	water
NH_3	ammonia
NO_3	nitrate
O_2	oxygen
ORP	oxidation-reduction potential
PAA	peracetic acid
TAED	tetraacetyl ethylene diamine
TriAED	triacetylethylenediamine
TS	total solids
TSS	total suspended solids
VSS	volatile suspended solids
WWT	wastewater treatment
WWTP	wastewater treatment plant

CHAPTER 1: INTRODUCTION

1.1 Problem statement

Wastewater treatment plants (WWTPs) treat human waste (sewage), but in the process, produce sewage sludge, a residual material. There are concerns associated with sludge production and its management. Sludge poses a threat on human health due to its pathogen and heavy metal constituents (USEPA, 2006) and therefore, requires appropriate treatment before dispersion into the environment. Treatment of sludge represents on average 30 % of costs associated with energy consumption sustained by a WWTP (Water research foundation (WRF); Electric power research institute (EPRI), 2013), however, improved processes and novel technologies can reduce these costs. After treatment, generally, biosolids disposal follows one of the following paths: landfill, digestion, incineration or land farming. USEPA (2006) estimates that about 50% of the biosolids are used beneficially in land applications or other uses.

First of all, health concerns must be addressed. Heavy metals and pathogens need to be removed from sludge in order to produce top quality biosolids. Examples of heavy metals found in sludge include: cadmium, lead, chromium, arsenic, mercury, nickel, copper, zinc selenium and molybdenum. Examples of pathogens include: viruses, bacteria, protozoa, and worms (helminths). In order to deal with the issues, the government has developed regulations that impose concentrations limits in order to quantify biosolids' quality and decide on their further use. In Canada, the Canadian Council of Ministers of the Environment (CCME) is responsible for biosolids management, while each province has its own restrictions. Regulations in the United States are governed by Part 503 and is categorized as Class A or Class B. Pathogens and Vector Attraction Reduction. Pathogen reduction can be divided into two different levels, Class A and Class B. In Class A, disinfection is almost complete, fecal coliform (FC) levels are below 1000 MPN/g total solids (TS) dry weight and pathogens are reduced below detection levels. In class B, (or process which significantly reduce pathogens) disinfection is incomplete, FC usually have a 2-

log reduction and are below 1 million/g of dry-TS and pathogens are reduced by approximately 10% (USEPA. 2006). In Quebec, Canada there are similar requirements but it has a different classification (C-P-O-E) with a total of 24 different classification possibilities. The fertilizing residuals (biosolids) are classified according to the chemical contaminant content (heavy metals: C1 or C2), pathogen content (P1 or P2), odour (O1, O2 or O3) and foreign matter (E1 or E2).

Second of all, sludge treatment is still a costly process to a WWTP and requires proper management. The quantity of sludge that will require processing is expected to increase. As, for a period of twenty years leading to 2008, fifty million more people connected to the sewage system (WRF, EPRI 2013). For example, in the United States, there has been a 74% increase of electricity use from 1996 to 2013 in processes related to sludge (WRF, EPRI 2013). According to CCME (2012), in Canada, WWTPs produce over 660000 dry tons, or 2.5 million wet tons of biosolids annually with a constant yearly growth. In the United States, there are over 7 million dry tons of biosolids produced every year (NEBRA, 2007).

However, sludge has been proven to be a source of energy and nutrients if it undergoes adequate treatment, but unfortunately, it is still not sufficiently recognized as a source of value-added products. Biosolids are a source of organic matter (soil amendment) and nutrient fertilizer (N, P, Fe) (CCME, 2002) and of course, a source of potential energy. For example, according to USEPA (2011), influent with a flow of one million gallons a day equals to 26kW of electric capacity and 2.4 MBtu/day of thermal energy. With pressure derived from climate change and the increased awareness over the past few decades, there are now higher expectations when it comes to recycling and energy recovery from waste.

Although there are many treatments available today, no method proves to be as versatile as electrokinetics (EK). In the previous studies on biosolids EK treatment (Elektorowicz and Oleszkiewicz 2007; Esmaeily et al. 2006; Safaei et al. 2013a, Elektorowicz et al. 2016a), EK combined with enhancing agents managed to achieve dewatering, heavy metal and pathogen removal through creation of adequate conditions in electrokinetic electro-bioreactors. A combination of electrokinetic phenomena with conditioners permitted the inactivation of *Clostridium sp*. spores in anaerobically digested sludge. However, the feasibility of BioElectro has not been verified on waste activated sludge (WAS) yet. It is a hypothesis that a combination of similar conditions will rise exothermic heat and inactivate the pathogens in WAS. Subsequently, a series of tests is required to prove this hypothesis at lab scale.

1.2 Objectives

The main objective of this study was to assess feasibility of application of electrokinetic process to wasted activated sludge (WAS) in order to convert it to Class A. Detailed objectives were:

a) Perform bench scale tests with variety of conventional WAS solutions in EK reactors
b) Assess the distribution of temperature rise in bench EK reactors
c) Assess impact of an initial sludge temperature on temperature rise rate
d) Investigate interactive impact of EK exposure and enhancers on temperature rate
e) Assess the applicability of BioElectro to sludge after secondary treatment with respect to class A biosolids
f) Assess feasibility of the BioElectro application for subsequent thermophilic and mesophilic process

1.3 Thesis structure

In order to achieve the above objectives, a thesis composed of five chapters will describe a step by step research and experimentation.

In the first chapter, the problems and concerns involving sludge processing are presented. Briefly, these are related to health concerns due to sludge constituents, and energy costs associated with sludge treatment. Then, the goals of this research are presented, with the main one being the assessment of the rate of temperature increase in the EK reactor.

The second chapter, which is the literature review, has eleven subchapters which review the most pertinent research. The literature review is focused on regulations in Canada and the United States, sludge management, sustainability, energy required in the treatment of sludge, contemporary anaerobic treatment options and of course, electrokinetic treatment of sludge and its versatility.

Chapter three explains a step-by-step methodology which includes the experimental setup, describes the characteristics of the sludge used in the experiment, the experimental parameters and the approach used, as well as a detailed methodology for the measurements of the sludge features at the end of the experiment.

Chapter four presents the results followed by the discussion, presented in subchapters.

Finally, chapter five presents the conclusion, the contributions and future work required of this thesis.

CHAPTER 2: LITERATURE REVIEW

2.1 Sewage collection systems

All around the world evidence has been found about ancient sewage systems, encompassing complex networks that lead to treatment “facilities”, with methods out of which some are still used in these days. The following paragraphs will only enumerate some examples; as much more archeological evidence has been found.

In South Asia, during the Harappan period (3000 BC-1500 BC), sewage and drainage was a complex network, composed of latrines, soak-pits, cesspools and pipes. According to Andreas et al. (2014), toilets were made of clay bricks with a seat, or a whole in the ground. The waste was then transported through terracotta pipes to pits made of clay bricks. A decentralized system was also used; the outflows were collected in U-shape channels made of wood and terracotta bricks and then to be poured into “jars” placed in the main street (Andreas et al., 2014).

Cities of Mesopotamia, from the end of IV and beginning of III millennium BC also showed evidence of wastewater networks. Sumerian settlements at Habuba Kebira (3500 BC), also reveal U-shaped gutters made from clay, or stone conduits (Viollet, 2007).

Archeological evidence from the Bronze era shows advanced water management and sanitary techniques were practiced in several settlements on Crete, during the middle and Late Minoan periods (2000-1100 BC) (Andreas et al., 2014). Moreover, in Ancient Greece, in Akrotiri, evidence was found dating back from to 1650 BC and Ancient Rome back from around 800 BC. According to Andreas et al., (2014) several systems were used for water and wastewater at the time, such as aqueducts, cisterns, filtering systems, rainfall harvesting systems, terracotta pipes for water, fountains, baths, sewers and lavatories.

In the 21st century, sewage requires treatment in order to avoid major health problems from exposure to pathogens and toxic substances. Treatment of sewage is done in a few stages, and it is usually designed for the plant's specific needs. After exiting the primary, secondary, and sometimes tertiary treatment, the sludge requires further treatment in order to ensure there will be no negative impacts on humans or the environment. There are many methods of treatment available today, and depending on the desired quality, the treatment may involve few to many more steps.

2.1 Regulations with respect to sludge disposal

2.1.1 Sludge disposal in the United States

The "Standards for use or disposal of sewage sludge" are regulations where, 40 CFR Part 257, 403, 503 are applied to biosolids quality.

Part 503 is responsible for the criteria for the final use or disposal of sewage sludge/ biosolids if they are applied to: i) land for conditioning the soil, ii) or fertilizing the crops; iii) or other vegetation, iv) they are placed on a surface disposal site for final disposal; v) they are to be incinerated (USEPA, 1994). Part 503 also includes five subparts which have an established set of standardized requirements for pollutant limits, management practices, operational standards, monitoring and recording (USEPA, 1994):

- General provisions
- Requirements for land applications
- Surface disposal
- Pathogen and vector attraction reduction
- Incineration

There are two classes of biosolids with respect to pathogen content: Class A and Class B. When sludge is characterized as Class B, certain limits are imposed by USEPA. For example, if the land (parks, playgrounds or golf courses) has a high public exposure, then the access will be restricted for 1 year after sludge application to the land. If the public exposure is low in places such as farmland in rural areas or remote islands, then access is restricted for the first 30 days (USEPA, 1994).

2.1.1.1 Class B requirements

There are three alternatives for meeting Class B pathogen requirements. Fecal coliform can be used as an indicator or the sludge can be treated with a process used to significantly reduce pathogens (PSRP) or in a process similar to a PSRP. Class B biosolids have more restriction concerning their use and disposal (USEPA, 1994).

Alternative 1. As mentioned, FC can be used as an indicator for pathogen presence. In order to comply with Class B, the geometric mean of seven samples must be less than 2 million MPNs per gram TS or less than 2 Million CFU per gram of TS at the time of use or disposal.

Alternative 2 comes with five different processes to significantly reduce pathogens (Table 2.1):

Table 2.1 Alternative 2: Processes to significantly reduce pathogens. Source: USEPA (2006)

	processes to significantly reduce pathogens
Aerobic Digestion	Mean cell residence time between 40 days at 20°C and 60 days at 15°C
Air drying	Drying required for at least 3 months, and for 2/3 months the temperature should be above 0°C.
Anaerobic digestion	Mean cell residence time between 15 days at 35°C to 55°C and 60 days at 20° C
Composting	Temperature must be raised to 40°C or higher for at least 5 days and for 4/5 days, the temperature must exceed 55°C
Lime stabilization	pH=12 after 2 hours from addition

Alternative 3 deals with sewage sludge treated in a process that is equivalent to a PSRP. The equivalent process will be determined by a permitting authority responsible.

2.3.1.1.1 Restrictions for Class B

Class B biosolids will have restrictions when applied to land. The restrictions on use apply to food crops, grazing animals, turf growing and of course public access. Therefore, if food crops were in contact with the biosolids, it must not be harvested within the first 14 months. If the food crop is below the surface, there is a harvest delay of 20-38 months depending on the time biosolids spent on land surface before incorporation. However, if the crop is not in contact with biosolids, it can be harvested after 30 days and the same restriction applies for grazing animals. Public access will be restricted for 30 days in areas of public access with low potential of exposure and 1 year for high potential of exposure (USEPA, 1994).

2.1.1.2 Class A requirement

Fecal coliform can be used as an indicator in order to ensure that the biosolids do indeed achieve Class A. According to USEPA (2016), Class A biosolids must contain a fecal coliform density below 1,000 MPN/g of total solids (dry weight basis). *Salmonella* spp, enteric viruses and viable helminth ova can also be used as indicators for Class A biosolids (Table 2.2).

Table 2.2 Class B and Class A pathogen reduction requirements. Source: USEPA 2016

Pathogen	**Class B**	**Class A**
Fecal coliform	< 2 million CFU or MPN per gram total solids (dry weight)	< 1,000 MPN per gram total solids (dry weight)
***Salmonella* spp**	Reduced by a factor of 10	< 3 MPN per 4 grams total solids (dry weight)
Enteric viruses	Reduced by a factor of 10	< 1 PFU[3] per 4 grams total solids (dry weight)
Viable helminth ova	Not applicable	< 1 viable ova per 4 grams total solids (dry weight)

USEPA's Part 503 offers six alternatives for sewage sludge treatment. The alternatives are presented in Table 2.3, adapted from USEPA (1994), while Table 2.4 focuses on the temperature regimes required by USEPA in order to achieve Class A biosolids. Regime D can be applied to the sludge used in this experiment due to its total solids content requirement.

Table 2.3 Alternatives for Class A Pathogen Reduction. Source: USEPA (1994)

Number	**Alternatives presented by USEPA 1994**
1	**Thermal treatment**: There are 4 different temperature regimes. Refer to Table 3.
2	**High pH-high temperature treatment proces**: must meet pH, temperature, and air drying requirements. ◦ pH≥12 at T=25°C for at least 72h. ◦ T>52°C and pH>12 for t≥12h ◦ Air drying at over 50% solids after the 72h period of high pH and meet all the pathogen requirement for Class A
3	**Other process**: proven reduction of enteric viruses and helminth ova and specif operating conditions after pathogen removal ◦ The density of enteric viruses must be less than 1 PFU/4gTS ◦ The density of viable helminth ova must be less than 1/4grTS
4	**Unknown processess**: testing required for: Salmonella sp, fecal coliform, enteric viruses, and viable helmith ova at disposal.
5	Treatment **process to further reduce pathogens**: ◦ Composting ◦ Heat drying ◦ Heat treatment ◦ Thermophillic aerobic digestion ◦ Beta ray irradiation ◦ Gamma ray irradiation ◦ Pasteurization
6	Process equivalent to alternative 5.

Table 2.4 The four temperature regimes as given by the USEPA for Class A Pathogen Reduction Under Alternative 1. Source: USEPA 1994.

Regime	**Applies to:**	**Requirement**	**Time-temperature relationship**
A	Biosolids with 7% solids or greater (except those covered by Regime B)	Temperature of biosolids must be 50°C or higher for 20 min or longer	$D = \frac{131700000}{10^{0.14t}}$
B	Biosolids with 7% solids or greater in the form of small particles and heated by contact with either warmed gases or an immiscible product	Temperature of biosolids must be 50°C or higher for 15 seconds or longer	$D = \frac{131700000}{10^{0.14t}}$
C	Biosolids with less than 7% solids	Heated for at least 15 seconds but less than 30 minutes	$D = \frac{131700000}{10^{0.14t}}$
D	Biosolids with less than 7% solids	Temperature of sludge is 50°C or higher with at least 30 minutes or longer contact time.	$D = \frac{50,070,000}{10^{0.14t}}$

Vector attraction reduction is the third indicator of sludge quality, and refers to the degree of attractiveness of animals and insects that could potentially further transmit pathogenic organisms to humans. USEPA (1994) offers 10 options (Table 2.5) to reduce vector attraction if sludge is to be applied to land.

Table 2.5 Options for vector attraction reduction for sewage sludge applied to agricultural land, forest, public contact site or reclamation site. Source: USEPA 1994.

Option number	**Option for vector attraction reduction**
1	Reduction of volatile solids (VS) mass of at least 38%
2	Aanaerobic sludge is to be further digested for 40 days at mesophilic temperatures, in order for VS to be reduced by 17%.
3	Aanaerobic sludge with less than 2% of solids is to be further digested for 30 days at 20 °C, in order for VS to be reduced by 15%.
4	Specific oxygen uptake rate (SOUR) for sewage sludge treated in an aerobic process shall be equal to or less than 1.5 milligrams of oxygen per hour per gram of total solids (dry weight basis) at a temperature of 20 °C.
5	Aerobic treatment for 14 days or longer at a temperature higher than 40 °C and the average temperature higher than 45 °C.
6	The pH raised to 12 or higher by alkali addition and, without the addition of more alkali, pH at 12 or higher for two hours and then at 11.5 or higher for an additional 22 hours.
7	The percent solids that does not contain unstabilized solids generated in a primary wastewater treatment process mustl be equal to or greater than 75 percent based on the moisture content and total solids prior to mixing with other materials.
8	The percent solids that contains unstabilized solids generated in a primary wastewater treatment process must be equal to or greater than 90 percent based on the moisture content and total solids prior to mixing with other materials.
9	For below the surface disposal there are two options: • No significant amount of sludge is to be present on the surface within one hour after disposal. • For Class A, sludge is to be inject below the surfce within the first 8 hours following treatment.
10	• Sludge must be incorporated into the soil within 6 hours after application or placement on land. • For Class A, sludge is to be injected below the surfce within the first 8 hours following treatment

2.1.2 Canadian approach to sludge disposal

In Canada, the use and disposal of biosolids is controlled by the Canadian Council of Ministers of the Environment (CCME), however, each province has its own set of regulations and restrictions (Table 2.6).

Table 2.6 Governing bodies for biosolids disposal by province. Source: CCME (2012).

Province / Territory	Governing body
Ontario	Permission is required from Waste Disposal Site- Organic Soil Conditioning from Ministry of Environment (MOE) and there are no provincial restrictions for use or disposal of biosolids.
British Columbia	The Environmental Management Act (1996) is responsible for the management of organic wastes such as biosolids. The regulations are the same as those prescribed by the USPA, Class A and Class B.
Alberta	Permission is required from the Director of the local office of Alberta Environment before use or disposal and there are no specific restrictions or bans throughout the province.
Nova Scotia	The Guidelines for the Land Application and Storage of Biosolids will ensure that the biosolids to be used or disposed of will have achieved Class A or Class B classification and will not pose any health risks.
Manitoba	The Environment Act E125, 1998 is responsible for environmental management along with the Onsite Wastewater System Regulation and the Farm Practices Protection Act.
New Brunswick	The Clean Environment Act, the Water Quality Regulation, the guidelines for Site Selection, Operation and Approval of Composting Facilities offer the guidelines pertinent to use or disposal of biosolids.
Prince Edward Island	The Ministry of Environment and Energy overseeing biosolids. The Environmental Protection Act and the Sewage Disposal Systems Regulations will insure proper use or disposal.
Saskatchewan	Environmental Protection and Management Act, Water Regulations 2002, Guidelines for Sewage Works Design, Saskatchewan Water and Wastewater Works Operator Certification Standards, 2002

2.1.2.1 Sludge disposal in Quebec

In Quebec, biosolids are considered fertilizing residuals, and by definition they are:

> *"...residual materials that can be used to maintain or improve, separately or simultaneously, plant nutrition, as well as the physical and chemical properties and biological activity of soils."*

There are several regulatory bodies in Quebec governing the fertilizing residuals:

Canadian Food Inspection Agency (CFIA) administers the Fertilizers Act and the fertilizer Regulations for products that are sold or imported such as fertilizers and supplements. Ministère du Développement durable, de l'Environnement et de la Lutte contre les changements climatiques du Québec (MDDELCC) is responsible for:

> *"...promoting the achievement of the environmental objectives for reclamation set out in the Québec Residual Materials Management Policy 1998-2008 (Québec, 2000), while ensuring that these activities are carried out in a manner that respects the environment and human health*"

Commission de protection du territoire agricole (CPTAQ) administers the laws and regulations related to the protection of agricultural land and activities as fertilizing residuals reclamation in agricultural zones are considered to be an agricultural activity by the CPTAQ (with some exceptions). Ordre des agronomes du Québec (OAQ) is responsible for protecting the public in keeping with the Professional Code and the Agronomists Act. It is also responsible for ensuring the competency of agronomists and their compliance with the Code of Ethics. The Bureau de normalisation du Québec (BNQ) is a standard development organization certified by the Standards Council of Canada and authorized to draw up commercial standards for fertilizing materials in Canada.

The fertilizing residuals are classified according to chemical contaminant content, C-Category (heavy metals: C1 or C2), pathogen content P-Pathogen (P1 or P2), odour (O1, O2 or O3) and foreign matter (E1 or E2) with a total of 24 different possible classifications.

In Table 2.7, the maximum limits for metals are presented and divided into elements considered essential for plants and animals or elements that are considered strict contaminants.

Table 2.7 Chemical content classification for biosolids in the province of QC. Source: MDDELCC 2015.

Pollutant	**Maximum limits (mg/kg-dry)**	
	C1 category	**C2 category**
Elements considered essential or beneficial to plants or animals		
Arsenic	13	41
Cobalt	34	150
Chromium	210	1060
Copper	400	1000
Molybdenum	10	20
Nickel	62	180
Selenium	2, 0	14
Zinc	700	1850
Elements considered strict contaminants		
Cadmium	3, 0	10
Mercury	0.8	4
Lead	120	300
Dioxins and furans	17	50

In Table 2.8 the pathogen classification is presented as P1 and P2 for the province of Quebec. In order to achieve P1 for pathogen removal, the discussed methods of sludge treatment are thermal drying and alkaline treatment or any other equivalent combination that is to satisfy the USEPA Class A requirements.

Table 2.8 Pathogen classification for biosolids in QC. Source: MDDELCC 2015

P1	For residuals contaminated with human fecal matter 1) Thermal drying: - Salmonella not detected in 10 g wet weight for residuals with dryness greater or equal to 15% (or in 50 g wet weight for other residuals)- in at least 2 of the 3 samples - AND drying temperature of at least 80 °C - AND final dryness greater or equal to 92% 2) Alkaline treatment- where salmonella has no been detected in at least 2 out of 3 samples. 2) Any other equivalent combination according to the USEPA to satisfy the class A requirements for pathogen reduction (including mandatory salmonella analysis) and vector attraction reduction.
P2	a) Lime to pH >= 12 for at least 2 hours and maintain at pH >= 11.5 for at least 22 hours b) *E. coli* < 2 000 000 MPN/g (d.w.) and aerobic biological treatment and O_2 uptake rate of <= 1 500 mg O_2/kg organic matter/hour. c) *E. coli* < 2 000 000 MPN/g (d.w.) and incorporation of residual into soil in less than 6 hours. d) *E. coli* < 2 000 000 MPN/g (d.w.) and biological treatment with sludge age >= 20 days old. e) *E. coli* < 2 000 000 MPN/g (d.w.) and biosolids from a lagoon not emptied since >= 4 years ago. f) Salmonella not detected in 10 g wet weight, for residuals with a dryness >= 15% (or in 50 g wet weight for other residuals) and O1 or O2 odour category g) Any other USEPA-approved combination that meets Class B requirements for the reduction of pathogens and vector attraction. E. coli: geometric mean

Table 2.9 presents the odour levels corresponding to the odour category which is low odour, malodorous, strongly malodorous, or the odour will have no category, such in the case of municipal biosolids from anaerobic digesters that are dehydrated using high-speed centrifuges, except if they have been deodorized by composting, liming or heat drying.

Table 2.9 Odour classification for biosolids in QC. Source: MDDELCC 2015

Odour categories	**Residuals**
O1-low odour	-cement kiln dust -wood ashes -lime mud from paper mills -magnesium residuals -other non-putrescible liming amendments -compost -dead leaves -bark -paper mill biosolids and deinking residuals with C:N≥70
O2-malodorous	-Municipal biosolids - lagoons not emptied since ≥ 4 years -Municipal biosolids - dried (6) -Municipal biosolids - limed -Septic tank biosolids -Limed abattoir biosolids -Paper mill biosolids with C:N ≥ 50 and < 70, not from a kraft process -Paper mill biosolids from lagoons not emptied since ≥ 4 years -Paper mill biosolids - acid treated
O3-strongly malodorous	-Municipal biosolids - biological treatment in a plant -Limed abattoir biosolids -Paper mill biosolids with C:N ratio < 50, not acid treated, not originating from lagoons with prolonged accumulation and not resulting from a kraft process. -Paper mill biosolids from a kraft process, with C:N ≥ 50 and < 70 -Whey -Declassified milk -Potato residuals -Grass clippings
OC-no category	-Municipal biosolids from anaerobic digesters that are dehydrated using high-speed centrifuges, except if they have been deodorized by composting, liming or heat drying. -Paper mill biosolids from a kraft process, with a C:N ratio < 50 that have not been treated for odours.

Foreign matter (corps étrangers) was added to the C-P-O classification in 2012 and refers to matter with a size greater than 2 mm (Table 2.10).

Table 2.10. Foreign matter classification for biosolids in QC. Source MDDELCC: 2015

	E1- no restrictions	**E2-** restricted usage
Sharp foreign matter (CETr: where the size >3 mm)	≤1 per 500ml	-
Sharp foreign matter (length >25 mm and width >3 mm)	≤2 per 500 ml	-
Total foreign matter (>2 mm)	0.5% (dry)	1.0% (dry)

2.2 Sludge management

Depending on the characteristics of sludge, and the desired quality of biosolids, treatment of sludge will generally follow the steps presented in Table 2.11. There are three important factors to consider when treating biosolids: pathogen reduction or disinfection, odour elimination and minimization of the putrification potential (Acquisto et. al, 2006). According to Acquisto et. al (2006), stabilization and disinfection are very important in biosolids treatment, as they are directly related to the quality of the biosolids, and thus decisive in their further use.

There are three main technologies for stabilization and disinfection (physical, biological and chemical) and eight main stressors (temperature, pH, irradiation, desiccation, pressure, ultrasound/cavitation, oxidation and non-charged chemicals/biochemical by-products) used in the treatment process. Such stressors can solubilize organics, destroy cell membranes and break down cellular structures and DNA (Safaei et al 2013b).

Acquisto et al. (2006) presented the main factors involved in disinfection and stabilization of sludge. As shown, the temperature is important in most physical and biological methods, while pH is the key factor in chemical treatment. Therefore, temperature is the most common method used in disinfection. According to Strauch (1998), the following combinations of time and temperature can be used to ensure that pathogens cannot survive: > 62°C at one hour, > 50°C for one day, and > 46°C for one week.

According to Carrere (2010), anaerobic digestion is preferred over biofuel production, microbial cells, incineration, gasification and pyrolysis, and supercritical wet oxidation due to its low capital investment and low operation and maintenance costs. The end product, biogas is mainly composed of methane (CH_4) and carbon dioxide (CO_2). Since the amount of biogas produced can only be as high the amount of readily available biodegradable matter different methods can be employed in order to optimize the reactions and increase the rate of biogas production, but more importantly increase the percentage of methane production in the biogas.

Table 2.11 Potential operations and processes used in treating solids and biosolids. Source: WRF, EPRI 2013.

	Processes for sludge treatment					
	Thickening	**Stabilization**	**Conditioning**	**Dewatering**	**Heat drying**	**Thermal reduction**
Operations for sludge treatment	Gravity	Anaerobic digestion	Chemical conditioning	Belt filter press	Rotary drum	Multiple hearth incinerator
	Dissolved air flotation	Aerobic digestion	Heat treatment	Centrifuge	Rotary disk	Fluid bed incinerator
	Centrifuge	Chemical stabilization		Vacuum filter	Flash	Wet air oxidation
	Gravity belt	Composting		Filter press	Multiple effect	
				Drying beds		

2.2.1 Sludge management in United States

Biosolids are managed by the Code of federal regulations (CFR), Title 40, Part 503. This requires pathogen and vector attraction reduction. Pathogen reduction can be divided into two different levels, Class A and Class B. In class A, disinfection is almost complete, fecal coliform (FC) levels are below 1000MPN/g TS dry weight and pathogens are reduced below detection levels. In class B, (or process which significantly reduce pathogens) disinfection is incomplete, FC usually have a 2-log reduction and are below 1 million/g of dry-TS and pathogens are reduced by approximately 10%. (Acquisto et al. USEPA).

Over the past years, the trend in biosolids treatment and management has shifted towards reducing the produced quantity and/or achieving Class A. According to United Nations Human Settlements Programme (2009), composting in larger cities is less desired mostly due to the higher operating costs and foul smell.

According to NEBRA (2007) the most common stabilization and dewatering operations are aerobic (45%) and anaerobic digestion (21%), followed by chemical treatment with lime/alkaline treatment (19%). In what concerns dewatering operations, the belt filter press (49%) and the drying beds (30%) are most frequent (NEBRA, 2007).

Costs associated with biosolids management have two major components: the actual dollar costs associated with capital and the operated expenses and the value of resource recycling and the cost of the environmental risks (Elliott et al. 2007). A study was done by Elliott et al. (2007) on 168 treatment facilities in Pennsylvania on landfill and land use costs. There were 83 small facilities, 52 medium facilities and 23 large facilities involved. The average costs per dry ton are presented in Table 2.12. This study shows that larger WWTPs will have a lower cost per dry ton than smaller WWTPs and that the cost for land applications is significantly lower than its disposal in the landfill. These results represent only one of the many reasons for which biosolids should always be treated to achieve Class A standards.

Table 2.12 Average cost per dry ton of WWTP solids by facility size. Source: Elliot et al. 2007, Biosolids disposal in Pennsylvania.

	Average costs per dry ton in USD	
	Land application	**Landfill**
Small Facilities (< 1 MGD)	252	280.35
Medium Facilities (1 to 5 MGD)	201.65	256.08
Large Facilities (> 5MGD)	145.16	260.32

2.2.2 Sludge management in Canada

In Canada, biosolids management is operated at a provincial/territorial level but they use the guidelines provided by the Canadian Council of Ministers of Environment (CCME). CCME (2002) classifies the biosolids in two broad categories: beneficial and for disposal.

According to Huang et al. (2008), biosolids are usually used for land applications, or are disposed to sanitary field or treated by thermal oxidation. Quebec and Ontario mostly use thermal oxidation as a management technique (CCME, 2002).

In Ontario, 40% of biosolids are used in land applications, 40% are disposed to in the landfill, while the other 20% are incinerated. In contrast, Quebec only used 27% of its biosolids for land applications, while the rest, 31% goes in the landfill and 42% is incinerated (Ville de Montreal, 2016). The WWTP in Montreal has primary treatment only and incinerates its sludge which later ends up in the landfill (Ville de Montreal, 2016).

2.2.2.1 Thermal oxidation

Water Environment Federation Residuals and Biosolids Committee Bioenergy Technology Subcommittee (2002) define thermal oxidation as the combustion of organic solids in wastewater solids in order to form carbon dioxide, water and inert material (ash). The equation below shows the basic oxidation of methane:

$$CH_{4\,(g)} + 2O_{2(g)} = CO_{2\,(g)} + H_2O + heat \qquad (2.1)$$

(Metcalf and Eddy, 4th edition, page 1667)

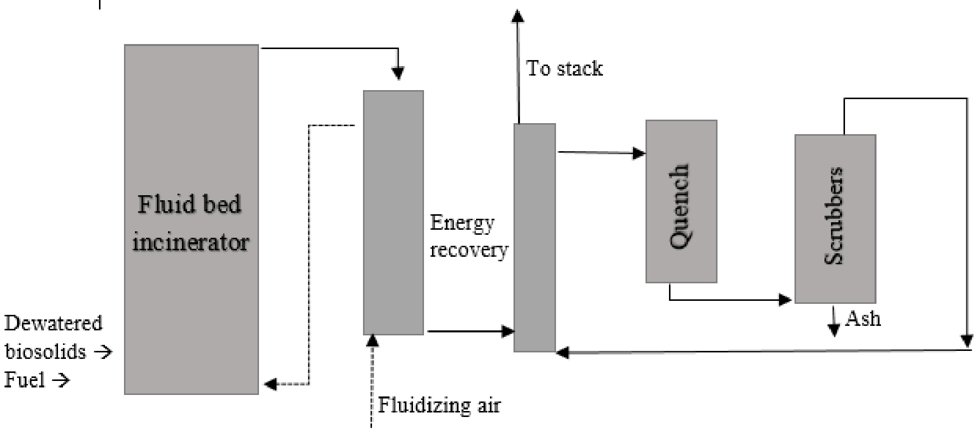


Figure 2.1 Thermal Oxidation Process. Source: Water Environmental Federation WEF, Bioenergy Brochure (2002).

Thermal oxidation is used in order to achieve complete oxidation and is mostly used for wastewater which is more concentrated. The process involves preheating the odorous gases before passing them into the combustion chamber (Metcalf and Eddy. 2003). The combustion occurs at temperatures ranging between 425 to 760°C (800 to 1400°F) (Metcalf and Eddy, 2003). There are two main types of reactors used for thermal oxidation: fluidized bed or multiple hearth. Multiple hearth tends to prevail, however new facilities constructed in the past 15 years use a fluidized bed. The basic thermal oxidation process is showed in the Figure 2.1. Thermal oxidation has advantages and disadvantages. It has a low life cost but a high initial cost. It produces recoverable energy that can be used to produce heated air, gas, water, and oil that can be used for process and building heating or converted to electricity in steam-driven equipment. Is not the most appropriate technology for non-continuous operation (though fluidized-bed systems are commonly operated 24 hours a day, four to five days a week) (WEF, 2003). The processing costs for fluid bed and multiple hearth have a net cost of $125-210 per dry tonne for fluid bed and $142-263 for multiple hearth.

2.2.2.2 Anaerobic Digestion

Anaerobic digestion is a biological process in which specific bacteria (acid formers and methane formers) break down matter in the absence of oxygen in different stages. The first stage is known as hydrolysis; complex waste components (polymers: carbohydrates, fats and proteins) are broken down by heterogeneous facultative and anaerobic bacteria into triglycerides, fatty acids, amino acids and sugars. In the second stage, acidogenesis (or fermentation), acetate, along with hydrogen, CO_2 and ammonia is produced. In acetogenesis, the third stage, volatile fatty acids are broken down to acetate and hydrogen. In the last step, methanogenesis, the acetate, hydrogen and CO_2 are converted to methane and more CO_2 (Davis and Cornwell, 2010). According to Schink and Stams (2005), acetogenesis and methanogenesis are the most important steps because they are to go through a syntrophic interaction. Most precisely, propionate and butyrate are the most important intermediates as their degradation rate can become the rate limiting step due to the fact that they tend to accumulate in the digester if the process becomes unstable (Amani, Nosrati, and Mousavi, 2011).

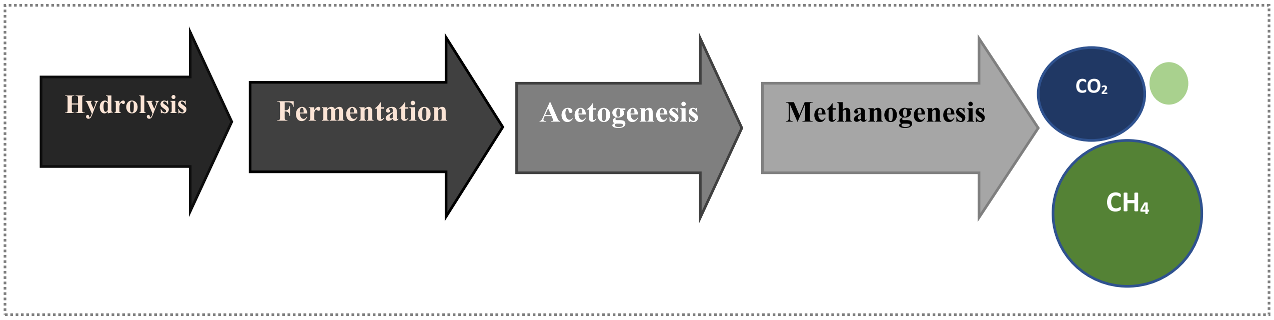


Figure 2.2. Schematic diagram of anaerobic process

During hydrolysis, insoluble particles are solubilized and organic polymers are decomposed into monomers or dimers, which can pass through the cells membrane. such process is accomplished by extracellular enzymes or physical/chemical reactions. The rate of hydrolysis depends on: particle size, production of enzymes and diffusion and adsorption of enzymes to particles.

During fermentation, the dissolved organic matter is degraded by heterogeneous microbes to VFAs. It mainly depends on: interspecies hydrogen transfer, pH, HRT and the acclimation of the anaerobic bacteria.

The next step is acetogenesis. Homoacetogenesis is production of acetate as a sole end product from CO_2 and H_2. Thermodynamically, it is less favorable than methano-genesis and sulfate reduction. Synthrophic acetogenesis is anaerobic oxidation of propionate and butyrate to acetate and H_2. Propionate and butyrate oxiditation are inhibited by even low H_2 partial pressures, therefore can occur only if H_2 is consuming by methanogens, SRB and homoacetogens (Amani, Nosrati and Mousavi, 2011).

The last step of the process is methanogenesis. In this phase, the acetate, hydrogen and CO_2 are converted to methane and more CO_2 (Davis and Cornwell, 2010).

The main conditions and variables for anaerobic digestion are: total solids, retention time, carbon to nitrogen ratio, organic loading rate, temperature, pH, mixing. Overall, anaerobic digestion will accomplish positive results, however it has limited abilities of treatment. Anaerobic digestion can disinfect sludge depending on the temperature in the digester.

$$C_5H_7NO_2 + 5O_2 = 4CO_2 + + H_2O + NH_4^+ + 4\ HCO_3^-$$

Although anaerobic digestion will accomplish disinfection, spores from different pathogens or heavy metals will not be successfully removed to comply with Class A regulations.

2.2.2.3 Aerobic Digestion

Aerobic digestion, is a biological process that occurs under the presence of oxygen. Digestion is accomplished with the help of aerobic heterotrophs. The organic material will be used to synthesize new biomass, but most of it will be oxidized to carbon dioxide, water and soluble inert material (Davis and Cornwell, 2008). When the organic material becomes exhausted, microorganisms enter endogenous respiration in which the cellular material becomes oxidized (Davis & Cornwell, 2008). It is said that about 75-80% of the cell tissue will be oxidized and the rest is not biodegradable (Metcalf and Eddy, 2003). According to Metcalf and Eddy (2003) the following reactions are involved in aerobic digestion:

Biomass destruction:

$$C_5H_7NO_2 + 5O_2 = 4C0_2 + H_2O + NH_4+HCO_3 \qquad (2.2)$$

Nitrification of released ammonia nitrogen:

$$NH_4+ + 2O_2 = NO_3 + 2H + H_2O \tag{2.3}$$

Overall equation with complete nitrification:

$$C_5H_7NO_2 + 7O_2 = 5CO_2 + 3H_2O + HNO_3 \tag{2.4}$$

Using nitrate nitrogen as electron acceptor (denitrification):

$$C_5H_7NO_2 + 4NO_3 + H_2O = NH_4^+ + 5HCO_3^- + 2NO_2 \tag{2.5}$$

With complete nitrification/denitrification :

$$2C_5H_7NO_2 + 11.5O_2 = 10\ CO_2 + 7H_2O + 2N_2 \tag{2.6}$$

Aerobic digestion is used in treatment of WAS, mixtures of WAS and primary sludge or WAS from extended aeration plants (Metcalf and Eddy, 2003) and has the purpose of reducing the volume of solids to be disposed. In general, aerobic digestion is employed by WWTP with flows of up to $2m^3/s$ (Metcalf and Eddy, 2003). There are three common aerobic processes that are usually used: conventional aerobic digestion, high purity oxygen aerobic digestion and autothermal aerobic digestion (ATAD).

2.2.2.3.1 Conventional aerobic digestion

The main factors involved in aerobic digestion and process design are temperature, volatile solids, the concentration of feed solids, oxygen, energy requirements for mixing and process operation (ex. pH) (Metcalf and Eddy, 2003).

2.2.2.3.2 Dual Digestion

This involves using thermophilic aerobic digestion in the first place with temperature ranges of 55°C-65°C and 18-24 hours of residence time, and mesophilic anaerobic digestion in the second stage with temperature ranges of 36°C-42°C and residence time of about 10 days. With dual digestion, there is an increased reduction in pathogen levels, volatile solids, increased production of methane in the anaerobic stage and less odours.

2.2.2.3.3 Autothermal aerobic digestion (ATAD)

When compared to the conventional process, the higher temperatures will inhibit nitrification. In this process, sludge tends to be thickened prior to entering the digester and the reactor will be insulated in order to conserve the heat produced during the exothermic process as no extra heat is put in the system. There are many advantages that come with implementation of an ATAD process, however, there are disadvantages as well. According to Layden et al (2007), ATAD achieves higher rates of biodegradability, less wasted sludge and therefore smaller volume requirements. Moreover, there is a higher reduction of volatile solids with a shorter hydraulic time. The ATAD system also has its disadvantages; the end product of the ATAD is more difficult to settle and dewater, it requires cooling and polymer addition, and the odours require emission control. In conclusion, ATAD can achieve Class A biosolids from different sludge types, with a sustainable end product, but so far, the disadvantages (e.g. foaming) have not made it the most popular choice (Lyden et al. 2007).

2.2.2.3.4 High purity oxygen aerobic digestion

In this process, oxygen is used instead of air and tends to be used in colder climates in order to increase the rate of biological activity. Usually the tanks will be closed in order to conserve the temperature and increase the rate of digestion.

2.3 Sustainable sludge management

With the overuse of natural resources, the balance between human actions and the environment must be continuously sought. Although the United States and Canada are developed nations, their sustainability practices still require improvement. In this century, the trend is aimed towards clean and reusable resources, and sludge had been proven to have enough energy stored in order to belong to this category.

2.3.1 United States approach

In 2006, USEPA estimated a production of about 8 million dry tons of biosolids annually. In order to determine the potential for sustainability of the solids, the following characteristics need to be taken into account: organic content, nutrients, pathogens metals and toxic organics (Metcalf and Eddy, 2003) along with treatment. A comparison of typical fertilizer nutrient characteristics and biosolids characteristics is presented in Table 2.13. This shows that biosolids have the potential to at least partially replace fertilizers.

Table 2.13 Comparison between fertilizer and biosolids characteristics. Source: Metcalf and Eddy (2003).

Product	Nitrogen	Phosphorus	Potassium
Fertilizers for typical agricultural	5	10	10
Typical values for stabilized wastewater biosolids (based on TS)	3.3	2.3	0.3

In 2004, NEBRA completed a study regarding beneficial use of biosolids and estimated that about 55% of biosolids were used towards beneficial practices, meaning, with the purpose of improving soil characteristics, such as structure, nutrient or organic properties. For example, 41% of all biosolids, were used for agricultural farmland. Another 12% was of public use, as Class A (exceptional quality) biosolids designated for landscaping, horticulture and agriculture. Therefore, in USA, the beneficial use of biosolids has increased gradually, showing potential in achieving sustainable levels of practice.

2.3.2 Canadian approach for sludge management

Compared to United States, Canadian regulations take place at a provincial/territorial level, therefore, sustainability practices in Canada differ from province to province. In this context, biosolids are classified as beneficial if they are within the required norms, otherwise, they are to be disposed of in the landfill.

Biosolids are characterized as beneficial if they reach a certain nutrient and organic matter value and energy content that can be then used in: energy production, compost and soil products, different applications as a soil conditioner (agriculture and forestry) and land reclamations (CCME, 2012).

The CCME (2012) considers the biosolids to be a valuable resource of nutrients, organic matter and having an energy potential. In their Canada-wide Approach for the Management of

Wastewater Biosolids report, the CCME presents a four-principle approach in order for biosolids to be considered beneficial and sustainable (Table 2.14):

Table 2.14 Four-principle approach to beneficial biosolids. Source: CCME 2012.

Principle 1	Municipal biosolids, municipal sludge and treated septage contain valuable nutrients and organic matter that can be recycled or recovered as energy.
Principle 2	Adequate source reduction and treatment of municipal sludge and septage should effectively reduce pathogens, trace metals, vector attraction, odours and other substances of concern.
Principle 3	The beneficial use of municipal biosolids, municipal sludge and treated septage should minimize the net GHG emissions.
Principle 4	Beneficial uses and sound management practices of municipal biosolids, municipal sludge and treated septage must adhere to all applicable safety, quality and management standards, requirements and guidelines.

Biosolids from municipal WWTP contain primary nutrients such as nitrogen, phosphorous and organic matter, vital to soil fertility, structure and plant growth (SYLVIS, 2009). They also contain secondary nutrients such as calcium, iron, magnesium and zinc (Epstein, 2003). As a result, this can reduce the dependence on fertilizer production and use, and encourage a shift towards an overall more sustainable way of life.

Biosolids can also be considered beneficial under certain combustion cases. For example, if they produce a positive energy balance, emit low levels of nitrous oxides and a significant quantity of ash or phosphorous is recovered (CCME, 2012).

SYLVIS and CCME (2009) published the Biosolids emissions assessment model (BEAM) in order to estimate and compare emissions from different treatment/management options, estimate the impact of GHG emissions that result from changes made to management options and in order to allow stakeholders to understand the main factors that have an impact on emissions. Table 2.15 below shows sources of negligible GHG.

Table 2.15 Treatment options producing negligible (non-point source) GHG. Source: CCME and SYLVIS (2009)

Unit Process	Negligible GHG	Rationale
Facultative lagoon	CH_4	Aerobic surface layer effectively oxidizes CH_4
Aerated lagoon	CH_4	Maintenance of an aerobic environment limits production
Mesophilic aerobic digestion	CH_4 and N_2O	Maintenance of an aerobic environment limits production
Physical addition of polymer in thickening	CO_2	Minimal energy requirement for process
Biogas recovery and combustion	N_2O	Anecdotal evidence of small releases, but not supported by data
Composting	CH_4 and N_2O	Considered negligible if total solids content of windrow is > 55% as this promotes an aerobic environment
Composting	CH_4	Considered negligible if process air treated in a biofilter
Composting	N_2O	Considered negligible if C:N > 30
Compost curing	CH_4 and N_2O	Compost is stable and microbial processes minimized; supporting data indicates minimal emissions
Gravity thickening	CH_4 and N_2O	Assumed that this process is kept aerobic
Passive dewatering	CO_2	Minimal energy use
Thermal drying	CH_4 and N_2O	Lack of supporting data

There are three main factors that play a role in asserting sustainability: economy, society and the environment. The human waste produced, must have a minimal impact on future ecosystems. In other words, the wastewater must be treated to a maximum in order to reuse the water, recycle the nutrients, use/sell the potential energy, by using the most efficient technologies which will have a positive impact on the local economy and in turn on society, without hurting the environment.

2.4 Energy required for biosolids stabilization and disinfection

The consumption of energy in WWTP is directly related to the quality of treatment that is desired (WRF, EPRI 2013). As the population continues to increase in North America, there will be a higher demand for modern and efficient WWTPs. In 2013, in the US, there were 15000 publicly owned treatment plants which handled a total flow of 32,000 MGD and served 74% of the population (WRF; EPRI, 2013). With the increase in secondary and advanced treatment methods, notably 48% increase from 1998 to 2008 (WRF; EPRI, 2013), it can be said that the total amount of energy required will increase.

Table 2.16 shows the increased demand of electricity by WWTP in the United States. While the secondary and tertiary treatment plants increased in number, about 48% since 1998 (EFRI, WRF, 2013), the electricity demand increased by 74% from 1996. So far, the secondary and tertiary treatment are expensive additions to the WWTP, but this can be ameliorated by introducing energy efficient treatments, customized for the plant’s need.

Table 2.16. Comparison of annual power use in 1996 and 2013. Source: (WRF; EPRI, 2013)

	Annual Electricity use (billion kWh/year)		**Increase (%)**
	1996	**2013**	
Public water supply and treatment	28.3	39.2	39
Municipal wastewater treatment	17.4	30.2	74

Over a period of twenty years, from 1988 to 2008, approximately fifty million more people are connected to the sewage systems, and as a result, the wastewater requiring treatment increased by about 13% (Table 2.17). The focus is now on improving the secondary and advanced treatments. This can lead towards plants becoming zero net energy consumers, and at the same time, offering beneficial by-products (e.g. input of methane into the grid, fertilizer pellets).

Table 2.17. Comparison of wastewater treatment statistics for 1988 and 2008. Source: (WRF; EPRI, 2013).

Year	**Number of facilities**	**Existing flow (MGD)**	**Population served (millions)**
1988	15,591	28,739	180
2008	14,780	32,345	226

Different processes involved in wastewater treatment require different amounts of energy. According to Metcalfe and Eddy (2003), about 30% of the operation cost of a WWTP is in energy requirements. Moreover, most of the energy is used in secondary and tertiary or advanced treatment. According to WRF, EPRI (2013), up to 52% of the total energy consumption of a treatment plant will be for secondary treatment. An example is the aeration required in activated sludge treatment. Another is in the case of trickling filters, where pumping is required for effluent recirculation. Overall, there are many processes involved in WWT and many mechanisms that contribute to operational costs with the most significant ones presented in Table 2.18.

Table 2.18. Processes and mechanisms involved in WWT. Source: WRF, EPRI 2013.

Processes and mechanisms involved in wastewater treatment	
Pre-treatment	-feeders, pumps, screens, comminutors, grit chambers
Primary treatment	-clarifiers, aerated channels
Secondary treatment	-activated sludge (blowers, mechanical aeration), -trickling filter pumps, clarifiers -aerated channels, aerated lagoons -anaerobic (mixers)
Advanced treatment	-nitrification aeration (blowers, mechanical aeration) -trickling filter pumps -granular media filters (filter feed, backwash pumps, backwash blowers), -membrane bioreactors
Disinfection	-chlorine feeders, sulfur dioxide feeders, mixers, UV lamps, process water pumps

According to Metcalf and Eddy (2003), the main energy costs in a commonly used treatment method such as activated sludge are: the clarifiers and sludge pumps, the aeration process, dewatering and heating. In such system, 1100-2400 MJ of electricity are required to process every 1000m^3 (1200 to 2500 kWh per M/gal) of wastewater (Metcalf and Eddy, 2003).

Subsequently there are the different processes in sludge treatment and solids processing: pumps, grinders, sludge digesters (mixers, aerators), chemical feeders, thickeners, centrifuges, belt press, dryers, incinerators (WRF; EPRI, 2013). Some processes require more energy than others, but in total, it will account to about one third of the total energy use of the WWTP (WRF; EPRI, 2013).

A comparison of the different level of energy use is shown in Table 2.19. It is important to be aware of which processes have a significant use of electricity in order to know which operation needs amelioration in energy consumption. For example, dewatering is a process that demands significant amounts of energy, but the BioElectro can accomplish at the same time with secondary and advanced treatment, therefore, the consumption is reduced by each process.

Table 2.19 Power requirements for different sludge treatment methods. Source: EPRI, WRF, 2013

Biosolids processing or disposal function	**Operation or treatment method**	**Impact on electricity use**
Preliminary operations	Pumping	Moderate
	Grinding	Small
	De-gritting	Small
	Solids blending and storage	Small
Thickening	Gravity thickening	Small
	Flotation thickening	Moderate
	Centrifugation	Moderate
	Gravity belt thickening	Small
Stabilization	Lime stabilization	Small/moderate
	Heat treatment	Significant
	Anaerobic digestion	Small/moderate
	Aerobic digestion	Moderate/significant
	Composting:	Small
	Windrow	Moderate
	Aerated static pile	Significant
	In-vessel	
Conditioning	Chemical conditioning	Small
	Heat treatment	Significant
Disinfection	Pasteurization	Moderate
	Long term storage	Small
	Drying beds	Moderate
	Lagoons	Small
Dewatering	Vacuum filter	Significant
	Centrifuge	Significant
	Belt press filter	Small/moderate
	Filter press	Moderate/significant
	Biosolids drying beds	Small
	Lagoons	Small
Heat drying	Dryer variations	Moderate
	Multiple effect evaporator	Significant
Thermal reduction	Incineration	Significant when used
	Wet air oxidation	Significant when used
Disposal	Land application	Small
	Landfill	Small
	Lagooning	Small
	Chemical fixation	Moderate

Table 2.20 is sourced from the WRF; EPRI, (2013) and presents the energy use for solids handling, treatment and disposal for different average flow intervals.

Table 2.20 Daily power requirements in kWh/day Source: EPRI, WRF 2013

Unit process	**Average plant flow in million gallons a day (MGD)**						
	1	**5**	**10**	**20**	**50**	**100**	**250**
Aerobic digestion	1000	5000	1000	N/A	N/A	N/A	N/A
Anaerobic digestion	N/A	550	1100	2100	5000	10000	250000
Gravity belt thickener	30	140	240	480	1200	2400	6015
Dissolved air flotation	N/A	N/A	1805	2920	6260	11820	44740
Centrifuge thickening	80	290	390	775	1950	3890	9715
Belt filter press	N/A	230	460	690	1390	2550	4400
Screw press	20	90	160	340	560	1120	2520
Centrifuge dewatering	260	1300	2610	5215	13040	26070	65175

In conclusion, the right choice of treatment will greatly impact the costs that the WWTP will have to suffer. It is important to choose the treatment depending on sludge characteristics and the output desired, budget and regulations in order to avoid unwanted costs. However, improvement is always necessary when it comes to quality and efficiency of treatment as new technologies become available. Sludge treatment is expensive, especially when Class A is required, thus cost-efficient methods need to be developed.

2.5 Energy required for anaerobic treatment

Anaerobic treatment is a popular choice in sludge. According to the EPA (2011), a WWTP with an influent greater than one (million-gallon day) MGD, will exceed 400 MW of electricity and 38000 MMBtu/day of thermal energy in output. According to the WRF; EPRI, (2013), for an average plant flow of 5 MGD, the electricity consumption will be 550kWh/ day, and for a flow of 250 MGD, the consumption will be 25000 kWh/day. As compared to other treatments, anaerobic digestion has a medium energy requirement, however many WWTP will use the biogas produced and many times will approach zero net energy use (WRF; EPRI, 2013).

2.5.1 Case study: South WWTP, Renton, Seattle, King County

The South WWTP is Located in NE of United States, in Renton, WA. The plant has been designed for an average daily flow of 115 MGD and has a maximum capacity of 325 MGD. On average, it receives 90MGD. During the winter months, the plant treats wastewater with the following sources: 43% residential, 38% storm water inflow and groundwater infiltration, 17% commercial and 2% industrial. The plant has a secondary biological treatment which uses aeration tanks and will produce waste activated sludge. The effluent undergoes disinfection with hypochlorite before it is sent to Puget Sound. Some of the water will undergo tertiary treatment with advanced filtration and disinfection in order to meet Class A Reclaimed Water standards for use in irrigation. The sludge obtained from the primary sedimentation tank and the secondary clarifier is blended, thickened and then it is sent to anaerobic treatment. Before becoming final product, the biosolids will undergo dewatering using polymers to aid in coagulation. The detailed process of the WWTP is shown in Figure 2.3. The plant produces about 100MG of reclaimed water per year and about 60000 wet tons of biosolids per year.

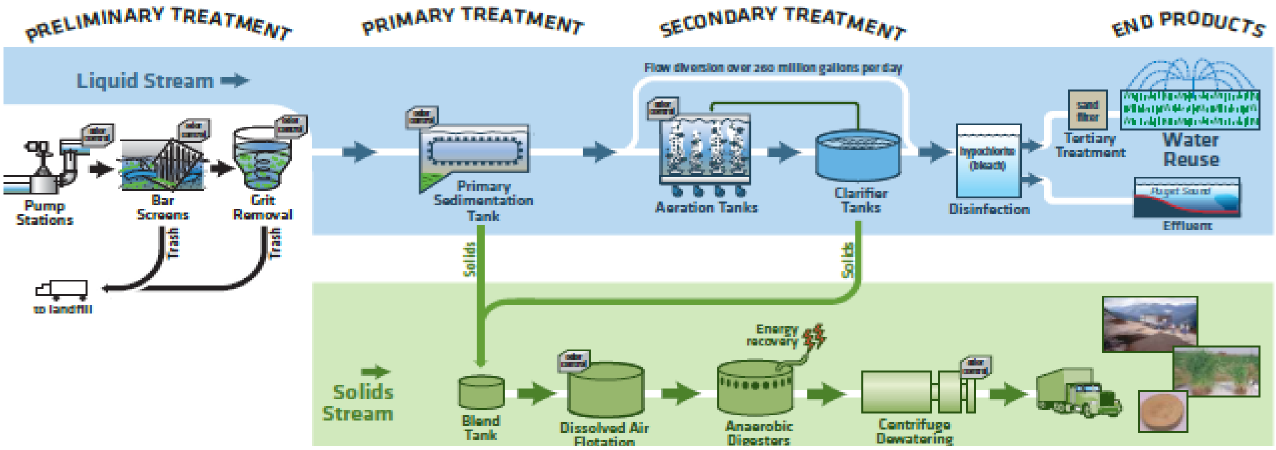


Figure 2.3 Wastewater treatment process use by the South WWTP in in Renton, WA.

In what concerns energy, the South WWTP uses the biogas produced from the anaerobic digesters for the boilers in order to heat up the plant and the digesters. According to Felix Brändli (2015), the energy analyst in the WWT division, it takes about 0.12kWh of electricity to scrub 1kWh of biogas. The South Plant also has a cogeneration facility which can run on biogas or natural gas. The heat from cogeneration is sometimes used to heat the digesters. The plant produces around 180000 MMBtu of methane per year and an electrical production of up to 33 million kWh per year.

From information provided by the South Plant on electricity use at the solids treatment substation, Figure 2.4 was produced in order to give an idea on energy use habits of the plant. The data provided also takes into account the energy used to heat up the plant, therefore, the summer months will be a more appropriate approximation on the energy used to heat the digesters. As a result, the energy required to heat the anaerobic digesters for a WWTP with an inflow of approximately 100MGD in oceanic climate is between 27000 and 37000 kWh/day.

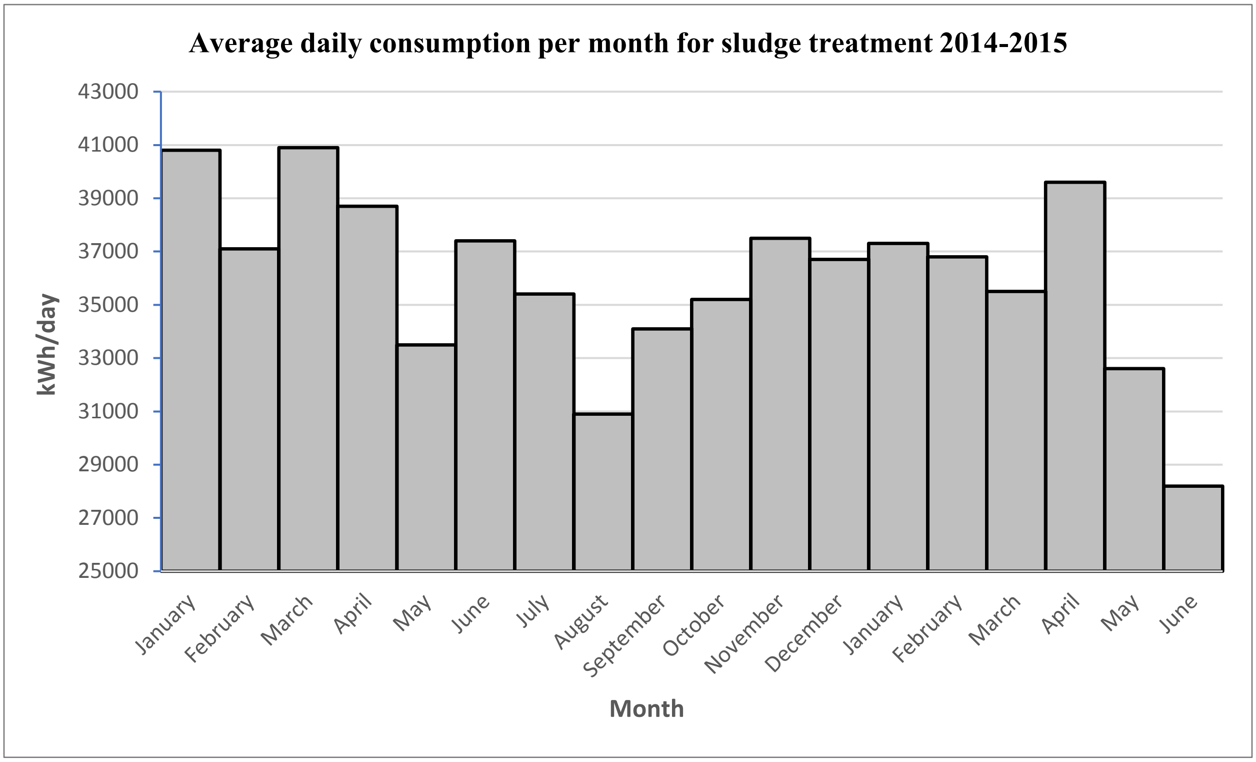


Figure 2.4. Energy used in sludge processing for 2014-2015. Source: Felix Brändli, 2015

2.7 Contemporary anaerobic facilities

The predominating tendency of development is the application of disintegration methods to intensify methane and volatile fatty acids (VFA) production. As it was already stated, a preliminary hydrolysis phase is the limiting step in a digestion process. During this phase, particulate organics become solubilized and they can serve as substrate and energy source for anaerobic bacteria, responsible for the process. Below are presented different contemporary anaerobic pre-treatments and treatments.

2.7.1 Multi-stage Anaerobic treatment

In a multi-stage, the stages of treatment are separated over time by adding different levels of heating at different times in the process by a process called temperature phased anaerobic digestion (TPAD). For example, in 2002, the wastewater treatment facility in Waterloo, Iowa, rehabilitated its existing anaerobic digestion system to operate as a TPAD system, in which the first digesters were operated in the thermophilic range (50- 60 °C) to promote pathogen destruction with the intent of producing Class A biosolids, while subsequent digesters were operated in the mesophilic range (30- 38 °C) to reduce VS (Iranpour and Windau, 2004).

In a pilot-scale experiment, Athanasoulia, Melidis and Aivasidis (2012) determined the potential of thickened secondary sludge for methane production using two CSTRs that are connected in series. The system consists of a first mixed-culture methanogenic reactor and a second methanogenic reactor for further reactions. The purpose of this system is to analyze the effect of hydraulic retention time on performance (biogas yield) and stability (volatile fatty acids). The system operated at HRTs ranging from 12.3-19.7 days. The process continuously receives sludge through the first digester and it overflows to the second digester and constant volume was maintained. The operational temperature was of 37 °C and the pH was kept in the neutral range (6.8-7.2). The pilot-scale experiment shows that the rate of methane production increases with lower HRTs. The preferred results were at HRT=12.3 days. As the HRT increased to 19.7 days, the cascade system showed increased efficiency over the single stage one. At HRT=12.3 days the cascade system was 9.5% more efficient than the single stage, while at 19.7days, the efficiency increased up to 40%. Thus, a cascade system had the potential of improving biogas production from 9.5-40% and the concentration of volatile fatty acids in the effluent can be decreased (Athanasoulia et al., 2012).

The major advantages of multi-stage anaerobic digestion systems versus single-stage anaerobic digestion systems is that multi-stage systems can optimize the various steps in the process by separating them in space or time and optimizing the specific conditions under which the various steps take place. The major disadvantage of multi-stage anaerobic digestion systems is that they have higher operation and maintenance requirements than single stage (EPA, 2006). Although an improved system as compared to a single stage digestion, this process still requires thickening and heating of the reactor. It is not suitable for areas with a cold climate additional energy costs will occur.

2.7.2 Thermal disintegration

In order to reduce the digestion time, thermal disintegration can be used as a pre-treatment method for WAS before entering the anaerobic reactor. According to van Veen et al. (2001), thermal hydrolysis, can aid the separation and decomposition of particles into a less complex form with increased solubility. During so called "subcritical wet oxidation" (van Veen al., 2001) a complete destruction of organic fraction occurs at 3740 °C (22.1 MPa). Another method of thermal disintegration consists of heating of the excess sludge to a temperature above 1500 °C for over 30 minutes. According to Hobson et al. (1994), this method will contribute to the disruption of activated sludge cell membranes and convert the sludge to a more readily digestible form. Although this pre-treatment method can help reduce digestion time, the operational costs involving heating of large amounts of sludge will increase considerably for a WWTP.

2.7.3 Thermal hydrolysis "Steam Explosion" process.

The thermal hydrolysis process (THP) patented by Cambi AS is a pre-treatment of sludge combined with anaerobic digestion. During this process, the sludge will be dissolved and disintegrated using temperature and pressure (temperature of 165° for 20 minutes, at 6 bar, followed by a sudden drop in pressure, resulting in so-called "steam explosion"). The disintegration of the cellular structure of the bacteria is achieved by solubilizing exopolymers, producing an easily digestible product. The sludge is pre-dewatered and introduced into a reactor saturated steam hydrolyzes and changes its internal structure. According to Cambi AS, this process reduces sludge viscosity, increases its biodegradability and shortens hydraulic retention time. Cambi AS states their process increases the production of biogas in digestion, reduces the volume needed for digestion, increases the dryness of the final dewatering of digested sludge, eliminates odors, and provides pasteurized final sludge Class A.

The Cambi plant at Thames Water's Chertsey WWTP (London, UK) has capacities of up to 7 kg VS m3/day and retention times of 10-12 days (Menco, 2012). According to (Menco, 2012), the use of Cambi thermal hydrolysis will generate a higher biogas production, reduce final sludge volume by improving dewatering. The sludge will be pasteurized and have odour decreased by 70%.

Although this method of treatment shows positive results; the capital costs and energy demands are high and the system becomes very costly for smaller sized WWTPs.

2.7.4 Ultrasonic disintegration

Introduction of ultrasonic technology is quite a new approach in sludge processing. This is another pre-treatment method that could be used in order to reduce the digestion time. Low frequencies are used, in the range from 20 to 100 kHz and demonstrated the ability to have a destructive power. The experiments on disintegration of primary sludge (Neis and Tiehm, 1997), 1:1 mixture of primary and excess sludge (Quarmby et al., 1999) and excess sludge only (Zielewicz, 1997), showed that the process generates smaller sludge particles and enhanced production of VFA.

Anglia Water has done pilot testing in a US WWTP by adding a disintegrating unit to the pipe which carries the sludge to the anaerobic reactor and obtained an energy consumption of 0.3 kWh/kg dry solids. Full scale tests were done on WWTPs in Guangzhou, China and Bamberg, Germany with positive results showing destruction of volatile solids with some decrease in sludge volume (Woo and Goldhardt, 2015). However, it has high capital costs and is an energy intensive process (Weemaes and Verstraete, 1998) and mixed results are observed as a result of the frequency and intensity.

2.7.5 Disintegration with ozone

There are some pre-treatment methods of activated sludge involving disintegration with ozone before the anaerobic reactor. Ozone applications are considered a chemical method of sludge disintegration. Due to ozone action, a disruption of microbial cell membranes occurs, accompanied with a release of the intercellular material (Ried. et al., 2002). The application of ozone helps the partial oxidation of slowly degradable material into simple readily biodegradable forms. Therefore, at the wastewater treatment plant where the carbon/nitrogen ratio is not favorable, disintegrated sludge may serve as an additional source of organic carbon for denitrification. So far, the most popular is disintegration of excess sludge and a part of recycled sludge. The ozone doses vary from 30 gO_3/ kg dry solids to 80 gO_3/ kg dry solids; at these doses, the floc structure of sludge is no longer noticeable (Ried et al., 2002). The energy consumption of this process is about 0.6 and 1.7 kWh/kg VSS, respectively. According to Braguglia et al. (2012), ultrasonic disintegration is more efficient on sludge pre-treatment as compared to ozone disintegration. For example, ultrasonic pre-treatment had positive effects on volatile solids and COD and an increased rate of biogas production by 26% as compared to ozone pre-treatment which only showed a removal of volatile solids at higher doses (Braguglia et al. 2012). Ozone can disinfect with the help of nitrous acid at a pH of 2.3 and is not a viable disinfecting process for sludges.

2.7.6 Pre-Pasteurization

Pre-pasteurization involves heating raw solids to 70° C before transferring to a separate holding tank (Sieger and Stone, 2001). The solids in the holding tank are maintained at a minimum of 70° C for 30 minutes. When pasteurization is complete, the solids temperature is decreased to about 40° C in a heat exchanger and transferred to the digester. By using a sludge/sludge heat exchanger, over 60% of the thermal energy can be recovered prior to entering the digester. Pasteurization following mesophilic anaerobic digestion is not acceptable due to pathogen regrowth. Pasteurization may enhance VSS destruction, but further research is required to validate this claim (Sieger and Stone, 2001). Pasteurization may be an effective method for pathogen destruction, but it also requires a large amount of energy to heat sludge to 70°C.

2.7.7 Co-digestion

Co-digestion is the process of adding organic waste to anaerobic digesters. The most common examples of suitable organic wastes include fats, oils, and grease as well as food waste. The co-digestion process can improve digester performance and also increase the amount of methane produced, but the additional organic waste must be added carefully and mixed well.

An experiment was done by Fountoulakis, Petousi, and Manios, (2010) in order to determine the limiting concentration of glycerol in the mixture and the methane production during the anaerobic process. The most successful mixture for co-digestion was the one containing 1% glycerol. The highest rate of methane production was at 1106 ml/d without addition of glycerol, and 2353 ml/d with a concentration of 1% glycerol.

There are many examples of full-scale co-digestion WWTPs all around the world which show a versatile choice of co-substrates (Nghiema et al. 2017). For example, in Roverto, Italy, the

WWTPs will daily add ten tons of food waste to the main substrate which is composed of primary sludge and waste activated sludge. In Moosburg, Germany 22 tons of pre-treated food waste and 18 tons of lacto-rich waste will be added daily to every 100 tons of primary and waste activated sludge mix. In Kurobe, Japan eight tons of ground coffee and up to five tons of septic tank sludge are added to every 80 tons of primary and secondary sludge. In East Bay, United States, the WWTP will add food waste and fats, oils and grease (Nghiem et al. 2017).

2.7.8 Improvement of limitation of biogas production

Cell lysis has the overall purpose of destroying the cell membrane of the microbes, in order to speed up digestion. This can be tried via physical and chemical methods. They include ultrasound, heat and pressure, pasteurization, high shear mixing, ozone and peroxide, sonication, and focused electric pulse. The cell lysis process improves methane yield and reduces the volume of sludge that must be disposed. (WRF; EPRI, 2013).

Al-Mashhadani et al. (2016) tried sparging a digester with CO_2 microbubbles in order to assess methane production. It was found that the digester which had CO_2 microbubbles had a methane production that increased by 109% as compared to the digester that was not sparged with CO_2.

In another study done by Yang et al. (2017), granular activated carbon was added during batch experiments and it was found that by adding 5g of granular activated carbon per 150mL of WAS the methane production increased by 17.4% while the sludge reduction rate increased by 6.1%.

2.7.9 UASB - Upthane™

Upthane™ is an Upflow Anaerobic Sludge Blanket (UASB) treatment system specifically designed for the treatment of low strength wastewaters like municipal sewage. The process was developed by Veolia. Sewage enters Upthane™ through a gravity fed influent distribution system in order to achieve maximum contact between the incoming sewage and the anaerobic biomass in the reactor. Sewage enters at the bottom of the reactor and following an upflow path, it passes through a thick anaerobic sludge blanket. A gas-liquids-solids separator is located in the upper section of Upthane™. The positioning of deflectors prevents biogas from entering into the settler section and instead directs the biogas into the gas hood from where it can be collected. The biogas contains approximately 75 % methane. The solids are entrained into the settling zone where they settle and slide back into the digestion zone.

2.7.10 Exelys™ Continuous thermal hydrolysis

This process was developed by Veolia. Exelys™ combines thermal hydrolysis and anaerobic digestion in order to achieve: 25 to 35% less dry solids, 30 to 50% more biogas, no odours, and a pasteurised digestate. Continuous thermal hydrolysis operates 24 hours a day with feed and removal levels that are adjustable. Exelys™ operates under controlled temperature (165°C), pressure (9 bar) and duration time (~ 30 minutes) conditions. The system is controlled by a PLC that modulates the steam flow rate in line with the amount of sludge injected.

2.7.11 BIOZONE-AD® Sludge disintegration using ozone

WABAG has developed the BIOZONE-AD® process for a higher level of sewage sludge stabilization. This process supplements standard anaerobic sludge stabilization with an additional phase and involves the injection of pre-digested sludge with ozone. This “reactivated” sludge is returned to the digesters and intensifies the degradation process. The process was developed by Veolia.

Components of the BIOZONE-AD® process consists of one or two digestion stages, an ozone generator and a reaction tank. Ozone enters the tank via an external injection. Easily degradable organic compounds result from disintegration and these are either thickened, returned to the digester directly, or in the case of serial operation, transported to the second digestion stage. The substances released through oxidation are metabolized and additional biogas is formed. WABAG states that micro-pollutants (polycyclic aromatic hydrocarbons) are degraded. This can be of relevance for agricultural use when parameters such as that for benzo(a)pyrene exceeds the statutory limits. According to WABAG there will be an increase in the degradation of organic dry substance by up to 40% and an increase in biogas production by up to 40% as well as a 15% improvement in dewatering of biosolids. Although this process shows promising results when it comes to methane production, it is not capable of achieving Class A standards of treatment.

2.7.12 Pyrolysis

Pyrolysis uses high pressure and temperature in the absence of oxygen to decompose the organic material in biosolids into gas, liquid (bio-oil), or char, which is a combustible carbon.

There are two categories of pyrolysis: slow pyrolysis and fast pyrolysis. Slow pyrolysis does not produce bio-oil, whereas fast pyrolysis does. Pyrolysis typically occurs at temperatures lower than incineration or gasification. A single commercial application of the pyrolysis process currently in use is the SlurryCarb™ installation in California. It operates at a temperature of about 449°C. The reaction alters the molecular structure of the solids and releases CO_2, thus reducing the mass of the solids by approximately 40 percent. The resulting "carbonized" solids are made into a slurry that is thermally dried and pelletized to a solid fuel, called E-fuel, which can be combusted directly in pulverized coal boilers, gasifiers, fluidized bed incinerators, or used off-site as an alternative fuel. While pyrolysis has limited application to date, the potential energy recovery is promising. Projected energy balances of the California installation indicate a net energy production of 2,100 kWh/ton dry solids.

2.8 Electrokinetic technology and electrokinetic phenomena (EK)

An electrokinetic process will take place when direct current is applied to a media with the help of electrodes; one becoming positive (anode) and one negative (cathode). There are three main processes involved that will influence the behaviour of the system with time: Faradaic charge reactions, ohmic heating and the electrokinetic phenomena.

The electrokinetic phenomena, is a series of physico-chemical reactions, and although discovered in 1809 by Reuss, still hasn't been applied to its full potential. Reuss observed that clay particles dispersed in aqueous media would migrate in an electric field (Lyklema, 1995) and that water can be transported within a porous material (Hansen et. al 2015). In other words, EK is based on the application of a low intensity direct current (DC) or voltage potential between anode and cathode. Over the years EK treatment has been used in soil remediation, and in the past few decades, the technology is also applied for sewage sludge and sediment remediation. According to Elektorowicz (1995) and Wang (2004), contaminants will migrate towards the electrodes in an electric field.

Starting with the 1930s, EK has been used to remove excess salts from alkali soils in India, and to reverse the seepage flow direction in Germany (Hansen et. al 2015). In the 1990s, EK has been used in remediation of soil contaminated with chlorinated solvents in USA. In 1992 in Netherlands, the largest EK project was completed over an area of 3600 cubic yards to treat soil contaminated with cadmium and other metals. EK has also been used in removal of uranium, lead, chromium, and copper (Hansen et al. 2015).

EK has many applications in wastewater treatment and sludge treatment. Bench and pilot scale experiments have been done in dewatering of sludge, heavy metal removal and pathogen removal with successful results at Concordia University in Montreal, Canada in the past decades.

2.8.1 Electrical double layer (EDL)

When particles are exposed to fluids in an electrokinetic system, they will develop a double layer of charges at the surface. This is determined by the spatial distribution of the ions around it. The first layer of the particle will have a fixed charge which is bound to the particle, while the second layer is more diffuse, comprised of free opposite charges. The fixed layer is also called the Stern layer, and the diffuse layer is called Gouy layer (Delgado et al., 2007).

The formation of the EDL is very important in explaining the EK phenomena because the formation of charges and the forces created are related to the movement of fluid and particles in the system.

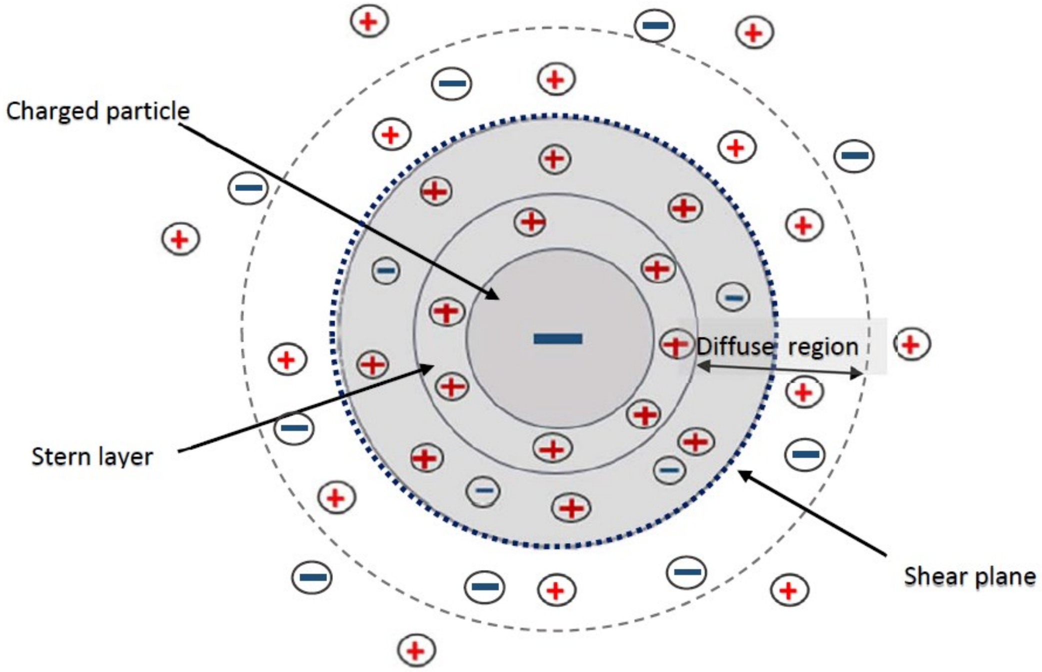


Figure 2.5: The Double Layer

2.8.2 Electrophoresis

Electrophoresis will induce the movement of suspended colloidal particles in a solid-fluid mixture with applied direct current. When applied to biosolids, the negatively charged particles will migrate towards the anode. Therefore, if contaminants are in the colloidal or ionic micelles forms, they will start to aggregate (Masliyah and Bhattacharjee, 2005; Brett and Brett 1993). According to Brett and Brett (1993), the following forces are responsible for the movement of particles:

- forces of the electric field on the particle
- frictional forces
- forces due to the action of the electric field on ions of the opposite charge to the particle within the double layer
- induction forces in the double layer caused by the electric field.

According to Kok (2000), in a solution, the electrostatic force, that is exerted on a particle in solution is proportional to the net charge of the particle and the electric field strength (or voltage gradient):

$$F=qE \tag{2.7}$$

F: electrostatic force
q: net charge of the particle
E: voltage gradient or strength of the electric field.

For a spherical particle, the viscous force is given by Stokes' equation:

$$F = 6\pi\eta\, r\, v_e \tag{2.8}$$

F: viscous force
η: viscosity of the solution
r: particle radius

v_e: electrophoretic velocity

Ideally, the electrophoretic velocity will become constant:

$$v_e = \frac{qE}{6\pi\eta r} \tag{2.9}$$

In conclusion, the ions and particles will migrate in the solution depending on the charge and effective radius.

2.8.3 Faradaic charge-transfer reactions

As particles are set in motion, and the water present undergoes redox, the pH will decrease at anode and will increase at cathode. The low pH might dissolve metals and change sludge characteristics such as zeta potential and dewaterability. The equations that govern the redox reaction are shown below:

Oxidation: $2H_2O = O_2(g) + 4H^+(aq) + 4e^-$ (2.10)

Reduction: $4H_2O + 4e^- = 2H_2(g) + 4OH^-(aq)$ (2.11)

2.8.4 Electroosmosis

While electrophoresis is responsible for the movement of particles, electroosmosis is responsible for the movement of water in the system. In theory, water surrounding the ions is dragged due to the existing friction forces as the cations and anions move in the system (Eykholt and Daniel, 1994). The most commonly used theory which explains electro-osmosis is the Helmholtz- Smoluchowski model.

According to Mitchell, (1991) when an electrical field is applied tangentially to a solid-liquid media, an electrical body force is exerted on the excess counterions in the diffuse layer of the EDL. Thus, the cations and anions will move under the influence of the electrical field, and while dragging some liquid along. Electroosmosis flow rate can be given by following equation (Mitchell, 1991):

$$q_e = K_e \nabla E A \tag{2.12}$$

Where:

q_e = Electroosmosis flux (ms^{-1})

K_e =Electroosmosis conductivity ($m^2V^{-1}s^{-1}$)

∇E =Electrical potential gradient (Vm^{-1})

A = Cross sectional area (cm^2).

2.8.5 Ohmic/ Joule heating

Ohmic heating will take place as the current will pass through the particles and will be dependent on the electrical conductivity. According the Sun (2005), applications using ohmic heating look very promising because heat distribution will become uniformly distributed in viscous substances.

$$\boldsymbol{I=V/R} \tag{2.13}$$

As the current passes through the sludge, it will release heat. According to Joule's Law, the power (P) will be dissipated as heat according to the formula below:

$$\boldsymbol{P = (V_A - V_B)I = IV = I^2R = \frac{V^2}{R}} \tag{2.14}$$

Therefore, the heat output can be controlled by changing the variables involved, such as the current flow, the voltage potential or the resistance. In order to accomplish this, the ohmic heating of fluids should be expressed as equivalent field form (Alwis, 1990). According to

Petersen, et al., 2004), the power dissipated can be defined in terms of voltage gradient and current density.

$$\boldsymbol{W = E^2 \cdot \kappa = \frac{J_{CD}{}^2}{\kappa}} \tag{2.15}$$

W: power dissipated per unit volume (Wm^{-3})

E: votage gradient (Vm^{-1})

J_{CD}: current density (Am^{-2})

κ: specific electrical conductivity ($ohm^{-1}m^{-1}$)

2.8.6 Electrocoagulation

During the electrocoagulation process, cations such as Al^{3+} and Fe^{3+} will form, act as a coagulant and lead to the destabilization of suspended particles and colloidal structures (Hakimipour, 2001).

2.9 Electrokinetic application to biosolids

Sludge is a valuable waste product, but untreated, it poses great danger to human and animal health. Research done in Europe and North America proves the potential of electrokinetic treatment applied to biosolids (Elektorowicz, 1995; Esmaeily et al (2006); Elektorowicz and Oleszkiewicz, 2010; Safaei et al., 2013b Electorowicz et al. 2014b; Electrowicz et al. 2016a; Electorowicz et al. 2016b). To date, the technology has been successfully used in sludge dewatering, metal removal and disinfection.

2.9.1 Sludge dewatering

Dewatering of sludge is a high energy demanding process (Metcalf and Eddy, 2003; EFRI, WRF, 2013) and while technologies improve, it still remains an expensive step of the operation. By analyzing the sludge composition, more efficient dewatering can be achieved.

The water present in sludge is usually of two types. It will also have different chemical and physical properties such as vapour pressure, enthalpy, entropy, viscosity and (Katsiris and Kouzeli-Katsiris, 1987). Free water is the dominant type and has more or less the same properties as pure water. Bound water represents a small fraction of the water found in sludge and is greater in what concerns mass than in solid phase (Colin and Gazbar, 1995).

In the past few years, commercial full-scale equipment of sludge electro-dewatering are available on the market such as CINETIK™ Linear Electro-Dewatering (Eimco Water Technologies), ELODE® electro-osmosis dehydrator (ACE Korea Incorporation), EDW (Water Technologies of Australia), and Electrokinetic (Electrokinetic Limited, United Kingdom).

An experiment done by Esmaeily et al. (2006) using electrokinetic treatment with a low voltage gradient and conditioners, showed positive results. The average solid content at the end of the experiment was of 48% higher than in experiments done without conditioners, with the total solid at 62% in the anode area.

In study done by Tuan (2011), the dry solid content in sludge increased from 15% to 40% in non-pressure applications and from 8% to 41% in pressure applications. The applied voltage was one of the major parameters affecting dewatering time, water removal rate and TS content of the sludge cake. Tuan (2011), also tested intermittent DC supply and concluded with a negative impact on dewatering in this study.

Elektorowicz and Oleszkiewicz (2009) and Habel (2010), also found a positive relation between the voltage applied and the TS % at the end of the experiment. In this bench trial, the highest %TS reached was about 24% at cathode. According to Elektorowicz and Oleszkiewicz (2009); Habel (2010), the application of higher voltage gradient in early stages of the trial will have a positive impact on the %TS at the end of the experiment. In the pilot testing, the highest water removal was in the cathode region with about that 22 % of TS at the end of the experiment, and an average of 14 % TS.

Huang et al. (2008) found during their study that a lower voltage gradient has shown the highest product of total solids at the end of the EK treatment. It was observed that the percentage of TS was higher at the anode area, due to electrophoresis phenomena. Low voltage will create an environment for a slow electro-coagulation process where solids will take a compact structure and thus achieving a TS content of 96% in the final stage (Huang et al., 2008).

In a study done by Mahmoud et al. (2011) on "Electro-dewatering of wastewater sludge: Influence of the operating conditions and their interactions effects", it was also found that there is a relationship between the voltage applied and the dewaterability efficiency. Although this study involved a combination of mechanical pressure and direct current application, the results are still pertinent. Under the influence of conditioners, there was an additional of 10-24% of water removed by the end of the experiment.

2.9.2 Sludge Disinfection

The electrokinetic process will produce oxidation reactions which will aid in neutralizing bacteria and viruses (such as *C.perfringens* and Reovirus) (Elektorowicz et al, 2012). During a study, Safaei et al (2013a), also observed that the metal distribution in the system was responsible for increasing the effectiveness of the oxidizing agents by catalyzing the formation of its hydroxyl radical. This experiment managed to eliminate pathogens and spores (*C.perfringens*) at low voltage gradients (0.5V/cm-1.5V/cm) using conditioners such as glutaraldehyde and Bioxy S during the electrokinetic process (Safaei et al, 2013b). In another study done by Safaei et al. (2017), the BioElectro achieved disinfection temperatures after 40 minutes of treatment (at 45-60°C), pasteurization after 60 minutes (at 65-70°C), and sterilization after 100 minutes (at temperatures over 90°C).

In research thesis done by Esmaeily (2002), fecal coliform removal was achieved in batch experiments that underwent electrokinetic treatment with low voltage ranging from 0.5 V/cm to 1.5 V/cm and conditioners such as di-amonium- phosphate.

Habel (2010), achieved inactivation of *Ascaris suum* Ova by ohmic heating to a viability of less than 0.001% by using different conditioners such as: $NH_4(NO3)$, $(NH_4)_2HPO_4$, $CO(NH_2)_2$ at a voltage gradient of 3V/cm.

Huang et al. (2008) achieved reduction of *Salmonella* spp. in all batches; the reduction ranging from complete to 6-log reduction. The batches that underwent electrokinetic treatment in combination with conditioners achieved the highest reduction. According to Huang et al., (2008), the area around the anode showed the most successful reduction, due to the acidic environment.

2.9.3 Metal removal in sludge

Most biosolids contain trace metals which also include heavy metals such as: Cadmium, lead, copper, zinc, chromium, arsenic and mercury. Such metals can pose a risk to humans and the environment due to their toxicity (Govind and Madhuri, 2014). Heavy metals bioaccumulate in the animal body and with time, it can pose serious health threats (Govind and Madhuri, 2014). Table 2.21 shows the required standards for metals in biosolids for each class, as well as the standard required by the Fertilizer Act of Canada. Generally, metals will end up in the wastewater by industrial or residential ways, but acid rain can also break up soils and runoff could end up the sewers. For example, copper may end up in wastewater from pipes and roofs.

Table 2.21. Standard level of metals in biosolids. Source: (NGSMI, 2003).

	Unit	**Class A**	**Class B**	**Fertilizer Act of Canada**
Arsenic	mg/kg total solids	41	75	75
Cadmium	Mg/kg total solids	39	85	20
Chromium	mg/kg total solids	1200	3000	-
Copper	mg/kg total solids	1500	4000	-
Lead	mg/kg total solids	300	840	500
Mercury	mg/kg total solids	17	57	5
Molybdenium	mg/kg total solids	-	75	20
Nickel	mg/kg total solids	420	420	180
Selenium	mg/kg total solids	36	100	14
Zinc	mg/kg total solids	2800	7500	1850

Research done on metal removal using electrokinetics emphasizes on the pH and speciation requirements of successful metal removal. In order for metals to be successfully removed, they must be in ionic form, as electro-migration is the main transport mechanism. According to Elektorowicz (1995), the pH needs to be low at/around cathode.

In research thesis done by Esmaeily (2002), results on heavy metal removal show that electroosmosis was the main responsible phenomena for Pb removal. The Pb removal was from 31% to 100%. In the same experiment, Cadmium was completely removed while Zn removal varied from 25% to 85% depending on the voltage and enhancers used. In the research thesis done by Habel (2010), 60% of initial Zn was removed.

2.10 BioElectro

The BioElectro is a new process designed by Elektorowicz et al. (2012) to treat biosolids and achieve Class A quality. The reactor uses a low voltage gradient in conjunction with enhancers such as BioxyS and ammonium salts in order to produce an exothermic reaction with increased ionic strength. The system can produce high temperatures in less than an hour and achieve a high level of disinfection (Elektorowicz et al., 2012). The main reactions are presented in Figure 2.6.

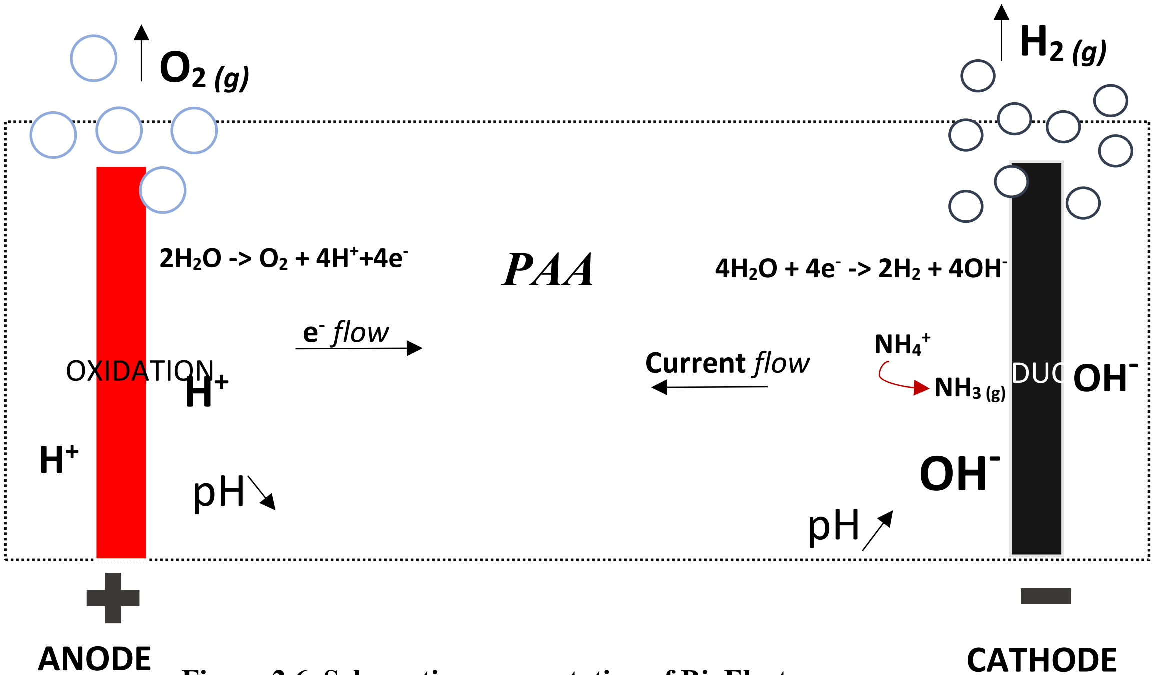


Figure 2.6. Schematic representation of BioElectro process.

In experiments conducted in laboratory setting using a 3-L prototype reactor and *C. Perfringens* as an indicator of pathogen removal, the BioElectro reactor shows great potential (Elektorowicz et al., 2012). Anaerobically digested sludge underwent a voltage gradient of less than 10 V/cm and the addition of enhancers. Log 3 reduction of *C. perfringens* was reached in less than an hour and Log 9 reduction in less than 2 hours (Figure 2.7). According to the kinetic analysis done by Elektorowicz et al. (2012), the addition of BioxyS, a solid form of PAA will initiate fast decay at a rate of 0.293 min-1 leading to entire decomposition within 15 min.

The design can be adjusted to individual demands and used in various applications. For example, there is a great potential in the conversion of existing aerobic digesters to produce Class A biosolids by adding an upstream batch system or a continuous flow which will create the perfect conditions for thermophilic aerobic digestion. This will produce further biosolids stabilization and due to fast reaction time, a short hydraulic retention is needed, and therefore minimal space requirements.

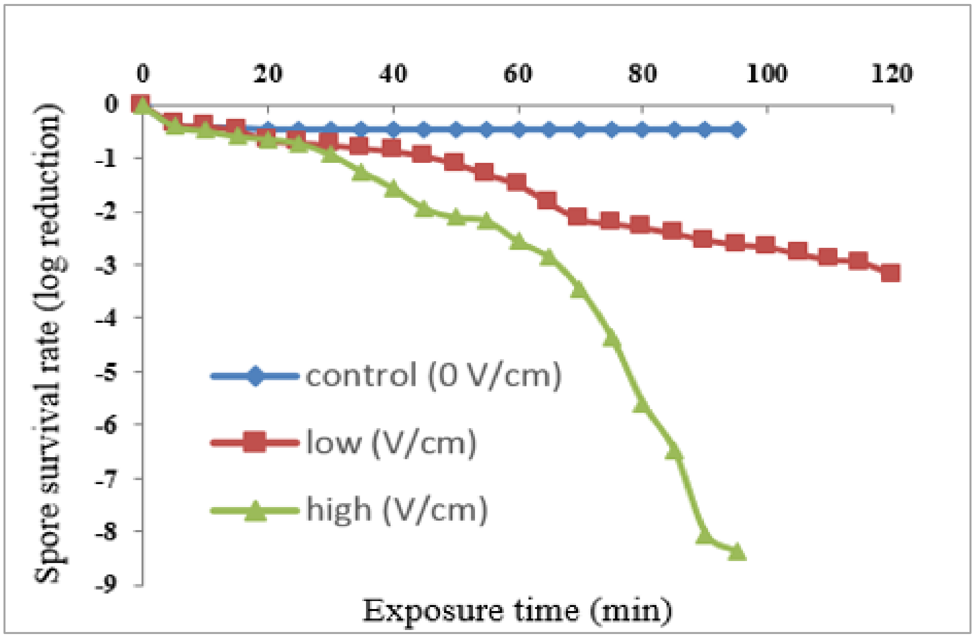


Figure 2.7 Spore survival over time. Source: Elektorowicz el al. (2012)

In a bench scale experiment conducted by Esmaeily et al. (2006) targeting dewatering and fecal coliform inactivation obtained promising results. Using low voltage gradients of 0.5 V/cm, 1.0 V/cm, 1.5 V/cm and conditioners, a solid content of 62% was achieved and no coliforms were observed at the end of the experiment.

Elektorowicz et al. (2009) and Elektorowicz et al. (2016a) achieved inactivation of C. perfringens and Reovirus, and determined that an oxidant mixture of BS and glutaraldehyde in conjunction with EK treatment have positive effects. In this study, it was observed that glutaraldehyde will interact with the *C. perfringens* spore's surface and under alkaline conditions it may have the capacity of penetrating the spore's cortex (Elektorowicz et al, 2014a). Glutaraldehyde also has an impact on the Reovirus as it reacts with proteins making the viral capsid or the viral-specific enzymes vulnerable. Elektorowicz et al. (2014a) and Safaei et al. (2013a) also determined that BS has oxidizing properties which create a mechanism that will pull electrons away from the cell membrane and further destabilize the membrane, leading to its destruction. Metals present in the sludge also have an impact on the electrokinetic system because they will be attracted to the electrodes and the higher metal concentration will increase the effectiveness of the oxidant agent (Safaei et al., 2013a). As a result, EK treatment at a voltage gradient of 1.5V/cm indicated a $\log_{10}$ reduction of 2.00 units for *C. perfringens* and $\log_{10}$ reduction of 11 for the Reovirus.

CHAPTER 3: METHODS

3.1 Methodological approach

The experimental setup is presented in Figure 3.1. This included 2 phases which consisted of 3 stages each. Each phase considered different origin of sludge. Combined sludge (primary and secondary) from Robert O. Pickard (Ottawa, ON) WWTP was used in Stage 1 of Phase 1, however, WAS from the same WWTP was used in Stage 2, Phase 2. Phase 2 considered two concentrations of WAS from St Hyacinthe WWTP (St Hyacinthe, QC), namely low (Stage 1) and high (Stage 2). Stage 3 in both Phases were related to pathogenic assay.

In order to achieve the objective of the experiment, several steps were involved:

1. Setting up a series of bench scale experiments in the environmental laboratory

Reactors with a volume of 500 ml and a pair of electrodes were used in all tests.

2. Determining the concentration of conditioners

As shown in Fig 3.1, during the Stage 1 and 2 of both Phase 1 and Phase 2, the ideal concentration of conditioners was determined. The conditioners were chosen based on previous study on anaerobic digested sludge (Safaei E, 2012) where BioxyS and ammonia salts were applied.

3. Determining the voltage gradient

Two viable voltage gradients were also identified during the Stage 1 and 2 of both Phase 1 and Phase 2. The electrical parameters such as: voltage gradient, distance between electrodes and material were used similarly to previous study on anaerobic digested sludge.

4. Initial temperature

-three different initial temperatures were used for Phase 1, Stage 1: 5°C, 14°C and room temperature (20°C-22°C)

-one initial temperature was used for Phase 1, Stage 2 and Phase 2: 14 °C

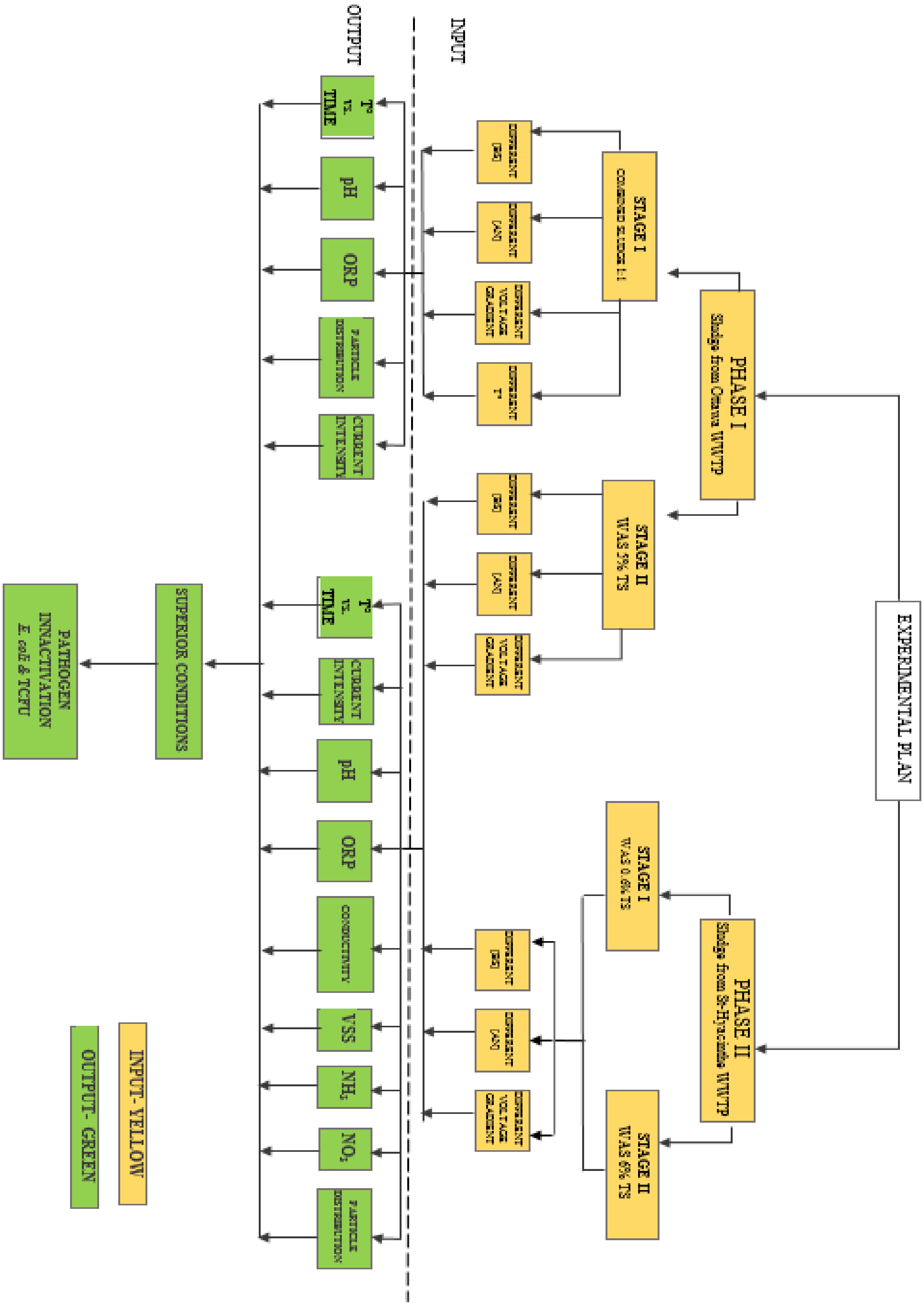


Figure 3.1 Experimental setup

3.2.2 Sludge used in Phase 2: City of Saint-Hyacinthe WWTP, Quebec

The WWTP serves a population of 53,236 (Statistics Canada, 2011). Every day it treats 49500 cubic meters of wastewater. It is mostly of domestic provenance (60%), industrial (25%) and runoff (15%). The BOD_5 is between 6000 kg and 11000 kg per day, in which 50% of it can be attributed to industrial wastewater. The amount of total suspended solids that need to be removed is 8000 kg, as well as 160 kg of phosphorous. Detailed characteristics of the influent are presented in Table 2.19. According to Ville the St Hyacinthe website, over 90% of BOD_5 is removed during treatment.

The WWTP serves a population of 53,236 (Statistics Canada, 2011). Every day it treats 49500 cubic meters of wastewater. It is mostly of domestic provenance (60%), industrial (25%) and runoff (15%). The BOD_5 is between 6000 kg and 11000 kg per day, in which 50% of it can be attributed to industrial wastewater. The amount of total suspended solids that need to be removed is 8000 kg, as well as 160 kg of phosphorous. According to the town of St Hyacinthe website, over 90% of BOD_5 is removed during treatment. Tables 3.1a and 3.1b present the influent characteristics of St Hyacinthe WWTP for the years 2013 to 2015.

The WWTP of St Hyacinthe has a pre-treatment, primary and a secondary treatment, e uses anaerobic treatment for biosolids in order to produce methane.

Table 3.1a. Wastewater characteristics at the St Hyacinthe WWTP. Source: St Hyacinthe WWTP.

Year	**Average Flow**	**Rain**	**pH**	**Temp.**
	m^3/j	mm	Avg	ºC
2015	36761	892.2	7.1	11
2014	44277	72.1	7.0	16
2013	44302	78.2	7.4	16

Table 3.1 b. Wastewater characteristics at the St Hyacinthe WWTP. Source: St Hyacinthe WWTP.

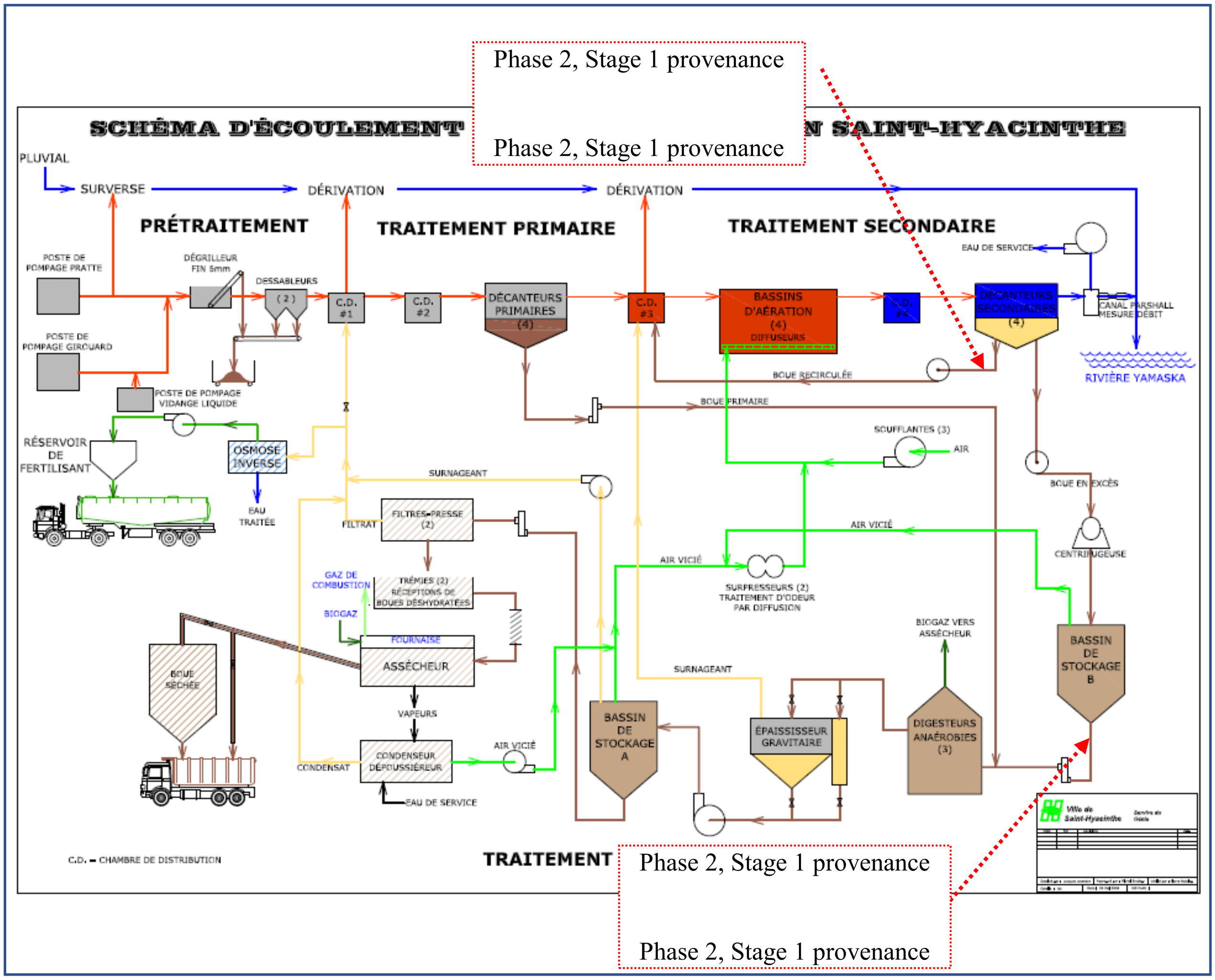
Year	**Characteristics of influent**											
	BOD_5		**COD**		**TSS**		**VSS**		**$N\text{-}NH_4$**		**P_t**	
	mg/l	kg/j	mg/l	kg/j	mg/l	kg/j	mg/l	kg/j	mg/l	kg/j	mg/l	kg/j
2015	101	4479	312	14722	143	6341	105	4656	7.4	304	2.9	129
2014	173	7644	363	16078	171	7565	125	5549	11.7	517	3.6	161
2013	164	7282	372	16479	168	7454	124	5494	11.5	509	4.3	191

Pre-treatment is done in two stages: screening and grit removal. A bar screen removes debris (rags, plastic parts, etc.). Then, two grit chambers remove sand and gravel. The retention time is 15 minutes. Four circular clarifiers are used to remove about 70% of the suspended solids. The solids are collected at the bottom of settling tanks and driven towards a central hopper by a scraper system. The fat and other substances floating on the surface are collected and delivered in a foam pit. Primary sludge and scum is pumped to the anaerobic digesters. The retention time is 4 hours in the clarifiers.

During the secondary treatment, 75 % of the BOD_5 will be treated in the aeration basin. The retention time is 5 hours in the aeration basins. Four more circular clarifiers are used to settle the waste activated sludge (WAS). This is recirculated about four- five times. The excess sludge is sent to treatment. The retention time is 7 hours in the circular clarifiers.

Primary sludge and thickened secondary sludge is pumped to anaerobic digesters where biomethanation transforms organic matter into biogas sludge. Each kilogram of organic matter digested produces about one cubic meter of biogas consisting of 62% methane. This has the effect of reducing by 40% the sludge produced by the station. The biogas is fed to a dryer to be burned and heat the digesters.

Digested sludge (digestate) are then dewatered by centrifuging and then, make a paste called solid cake. The dehydrated cakes are sent to a processing site, or dried and pelletized before being evacuated.


Phase 2, Stage 1 provenance

Phase 2, Stage 1 provenance

Phase 2, Stage 1 provenance

Phase 2, Stage 1 provenance

Figure 3.2 Collection and sampling of sludge for Phase 2, Stage 1 and 2. Source: St Hyacinthe WWTP.

Sampling: the sludge was collected right before entering the anaerobic digester. The sludge was thickened using polymers and centrifuged. Ferric sulfate was also added in order to avoid the formation of hydrogen sulfide. When collected, the sludge had total solids of around 6%.

3.3 Materials and equipment

3.3.1 Materials

- Amino acid reagent (HACH, 1934-32)
- Hydrogen peroxide 50%
- Enhancers (Bioxy S)
- Ammonium nitrate (AN)
- Ethylene glycol (HACH, 2039-53)
- Ethyl alcohol, reagent grade, 95% (A995-4)
- prepared agar plates (BBLTM MI Agar)
- Parafilm (13-374-12)
- pH meter buffer solution (pH= 4, 7, 1 0)
- Water (reagent-grade)
- Distilled Water

3.3.2 Equipment

- Analytical Balance (Accumet AR25)
- Buchner funnel (1000 mL)
- Beakers (100, 200, 500, 1000-mL)
- Binder-Free Glass microfiber filters 37mm¢ (Whatman, 0987412B)
- Centrifuge tube (50 mL)
- Filters, Membrane, 47-mm, 0.45-µm, gridded, sterile;
- Filtration assembly;
- Forceps;
- Incubator;
- Microscope
- Volumetric cylinder (5, 10, 20, 25 mL)

- Desiccator
- Erlenmeyer flask (50, 100, 250, 500, 1000-mL)
- Electric vacuum pump (Neuberger Knf)
- Funnel (7cm)
- Filter paper (Whatman 40, FSSP 97 51 058)
- Fine pipette Thermo 1mL-5mL (1438685)
- Gooch crucible 40mL (08-195E)
- Graduate cylinder (10, 25,400, 500, 1000-mL)
- Kimble Screwed test tubes, 25X150mm (14930E)
- Kimble glass vial, 21 X70mm (60940D-4)
- Micro pipette 2-20 pi (Fisher Brand, 14-900-28)
- Micro pipette 20-200 pl (Fisher Brand, 14-900-26)
- Micro pipette 30-300 pl (Thermo,)
- Muffle furnace (Fisher Scientific)
- Millipore syringe driven filter unit 25mm, 0.2 J.111l (SLFG 025LS)
- Microscope (Micromaster)
- Pipette (5, 10 mL)
- Petri dishes,IOOX15mm (08-757-13)
- Portable spectrophotometer (HACH, DR 2800)
- pH meter (Fisher AR25)
- Shaker (Oanadarride Scientific Digital Orbital)
- Volumetric flask (5, 25, SO, 100-mL)

3.3.3 The electrokinetic reactor (EK)

The EK reactor used in this study was the same that has been used for investigating the electrokinetic (EK) treatment of anaerobically digested sludge (Safaei 2007). The system has the following components:

-the cell, made of Plexiglas, (Figure 3.3).

Figure 3.3 EK Reactor with two electrodes, cover and series of electrode probes

-inside the cell, two perforated stainless-steel electrodes were fitted, a top view is shown in Figure 3.4.

The electrodes had a diameter of 1 cm, a thickness of 0.065 cm and a length of 10 cm. During the experiment, the electrodes were covered with a stainless-steel mesh.

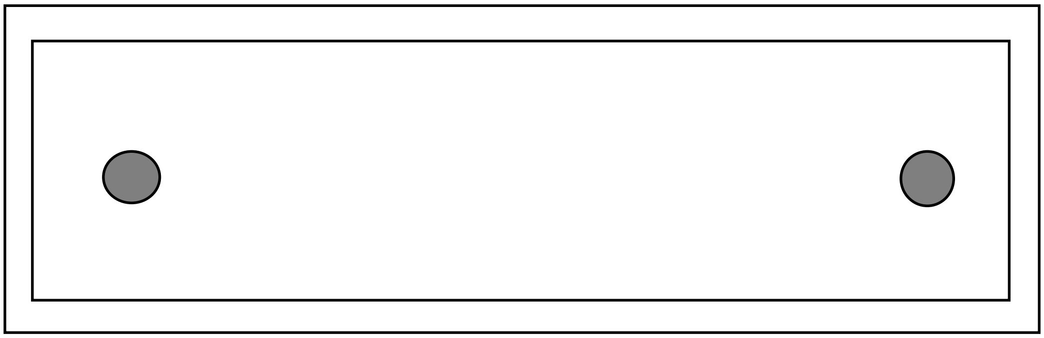


Figure 3.4: Electrode configuration, top view

- A Plexiglas cover fitted with 18 silver probe electrodes, with a diameter of 1 mm (Figure 3.5).

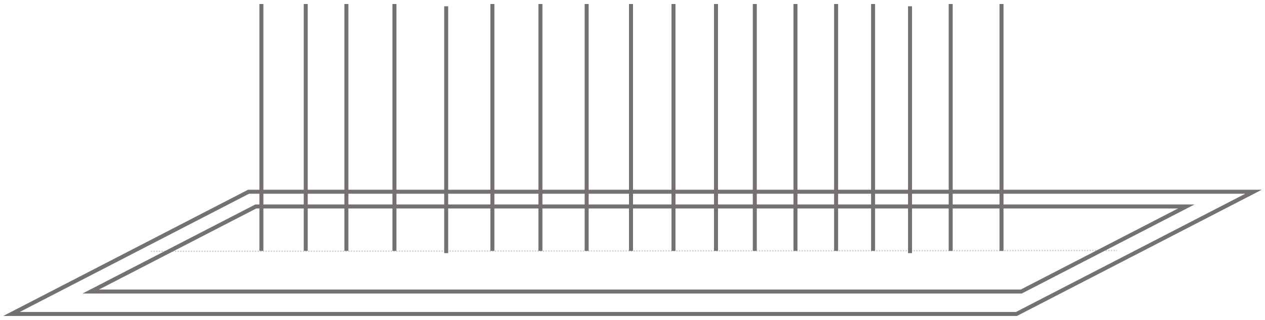


Figure 3.5 EK reactor cover with silver probes

3.4 Experimental parameters

The main experimental parameters of this experiment were: voltage gradient, exposure time and enhancing agents. Two voltage gradients were used in this experiment: 2.0 V/cm and 2.5 V/cm. The exposure time depends on the voltage gradient and enhancing agents' concentration. Taking into consideration Regime D under Alternative 1 for Class A Pathogen Reduction, the higher the temperature of exposure, the lower the contact time required.

3.4.1 Bioxy S^{NEW}

BioxyS (BS) was used in this study as a conditioner. It was used in concentrations varying from 0.5 g/L to 25 g/L. Bioxy S is a powdered compound which is able to produce biocide in situ. In contact with water it acts as a strong oxidant composed of sodium percarbonate (a hydrogen peroxide (HP) precursor), tetraacetyl ethylene diamine (TAED) (peracid precursor) and sequestrants. It was observed that, when introduced to sludge and exposed to EK, it creates exothermic reactions and undergoes a fast decay.

According to Dagher and Dagher (2006), when dissolving the BS in water the following reaction will occur:

$$\text{T.A.E.D.} + 2\text{HOOH} \rightarrow 2\text{CH}_3\text{COOO}^- + \text{D.A.E.D.} \quad (3.1)$$

Afterward, the system generates heat and peracetic acid which act as biocide. According to HERA (2002), this reaction is a stepwise process through the intermediate Triacetylethylenediamine (TriAED). Studies assessed by HERA (2002) show that TriAED only exists for a short time, and almost all TAED is transformed to DAED which is a biodegradable compound.

Table 3.2. Properties of BioxyS. Source: www.atomesbio.com

Properties of BioxyS:	
Appearance	White powder
Odour	Mild
pH (0.2%)	7.00±1.00 / Neutral
pH (2.0 %)	7.00±1.00 / Neutral

BioxyS is defined as an organic, non-foaming, non-toxic agent with a pH that remains neutral. It is also non-flammable and doesn't emit an offensive odour. The solution of BioxyS is also non-toxic and no trihalomethanes (THM) or chlorinated by-products were formed according to the manufacturer. Another positive aspect of BS is that it kills pathogens such as Listeria and Legionella and it prevents formation of biofilms.

The transport of BS is cost effective as it comes in powdered form and it is not regulated as a dangerous good. If the powder is spilled in powder or in water solution, no harmful chemicals are formed.

A comparison was done by the Atomes company between BS and liquid peracetic acid in order to assess their physical and chemical differences (Table 3.3).

Table 3.3. Comparison between BioxyS and liquid peracetic acid. Source: Atomesbio.com website, 2016.

DESCRIPTION	**BIOXYS- atomes**	**LIQUID PERACETIC ACID**
Activity	10% active- 2 times more concentrated	5% active
Concentration used	0.2% or 2g/L	0.4% or 4mL/L
Physical status	Solid	Liquid
Odour	No odour	Offensive- strong acetic acid smell
pH	Reacts with water to generate peracetic acid at neutral pH levels.	The pH is highly acidic
Corrosion to surfaces	No induced corrosion	Extremely corrosive to surfaces
Handling employees	Safe to handle	Extremely dangerous to handle
Storage	Requires limited space	Requires a large and secured space to prevent leaking
Chemical stability	Stable	Decomposes if exposed to heat or organic materials

3.4.2 Ammonium salt (AN)

Ammonium salt was used as another conditioner in this study. An important advantage of adding azote is firstly improve fertilizing properties of the final product, subsequently improving P:N ratio. Furthermore, ammonia state changes with pH and temperature, subsequently, influences properties of spore and cell membranes, permitting on electroporation. In this in study, ammonium nitrate (AN) was used as an ammonia salt.

The concentration of ammonium nitrate used during the experiment varied from 0-8g/L. The salts were first dissolved in 10 ml of distilled water before being added to the sludge. When added, the sludge was mixed for 2-3 minutes in order to insure proper distribution.

3.5 Experimental approach

A series of experiments were conducted in a fume hood, where temperature was kept at 20 °C +/- 1 °C. An effective volume of sludge in the reactor is was 0.6 L, for preventing overflow.

The sludge was well mixed before the required amount is removed from the 20L bucket. Only the volume to be used for the experiment is taken out of the fridge and held at room temperature until the desired initial temperature was reached: 5°C +/- 0.5°C, 14°C +/- 0.5°C or 20°C+/-0.5°C. The ammonium salts are first added to the sludge before it reaches the chosen temperature and well mixed in. When the desired temperature is reached (14°C +/- 0.5°C) the BioxyS™ is added and the sludge is further mixed for 2-3 minutes. As soon as the BioxyS is mixed, the sludge is poured into the cell as exothermic reaction begins and every second lost in the process will decrease its catalytic benefits. According to Safaei (2012), the PAA is consumed in the first 15 - 20 minutes.

All the experiments were ran in triplicate. Tables 3.4a and 3.4b present the reference number and different concentrations used for the experimental parameters. Several different combinations of enhancing agents and voltage gradients were used in batches in order to determine the lowest cost for treatment.

Table 3.4a Batch experiment reference number for different voltage gradient and concentration for Phase 1.

Phase 1, Stage 1 (Primary+WAS 5% 1:1)

Initial temperature (C)	**Concentration (g/L)**		**Voltage** (V/cm)	**Reference number**
	BioxyS	**AN**		
5	0.5	0.5	2.5	OM-1
	1	1		OM-2
	2	2		OM-3
	8	8		OM-4
15	1	6	2.5	OM-5
	2	6		OM-6
20	0.5	0.5	2.5	OM-7
	1	1		OM-8
	7	7		OM-9
	25	26		OM-10

Phase 1, Stage 2 (WAS 5% TS)

Initial temperature (°C)	**Concentration (g/L)**		**Voltage** (V/cm)	**Reference number**
	BioxyS	AN		
14	0	0	2.5	O-1
	8	8		O-2
	1	6		O-3
	0.5	3		O-4
	2	6		O-5
	0.5	3	2.0	O-6
	1	6		O-7
	0	3		O-8
	0	6		O-9
	2	6		O-10

Table 3.4b Batch experiment reference number for different voltage gradient and concentration for Phase 2.

Phase 2, Stage 1 (WAS 0.6% TS)				
Initial temperature (°C)	**Concentration (g/L)**		**Voltage** (V/cm)	**Reference number**
	BioxyS	AN		
14	0	0	2.0	SHR-1
	2	6		SHR-2
	4	8		SHR-3
	8	8		SHR-4
	3	8		SHR-5
	3	6		SHR-6
	2	6	2.5	SHR-7
	2	1		SHR-8
Phase 2, Stage 2 (WAS 6% TS)				
Initial temperature (°C)	**Concentration (g/L)**		**Voltage** (V/cm)	**Reference number**
	BioxyS	AN		
14	1	3	2.5	SH-1
	2	6		SH-2
	0	0	2.0	SH-3
	1	3		SH-4
	2	6		SH-5

3.6 Measurements

Several measurements were performed before, during and at the end of the experiment. Temperature and amperage were recorded during the experiment in order to assess the rate of temperature increase with time and electricity consumption. pH, conductivity, ORP, TSS, VSS, particle size distribution and fecal coliform.

3.6.1 Temperature

The temperature was measured using type K thermocouples. The thermocouples were connected to a data logger (Agilent BenchLink Data Logger 3). Three probes were installed to measure the temperature in the reactors throughout the experiments. The thermocouples were placed in the EK reactor, one at a distance of 1 cm from each electrode and one in the middle (Fig. 3.6).

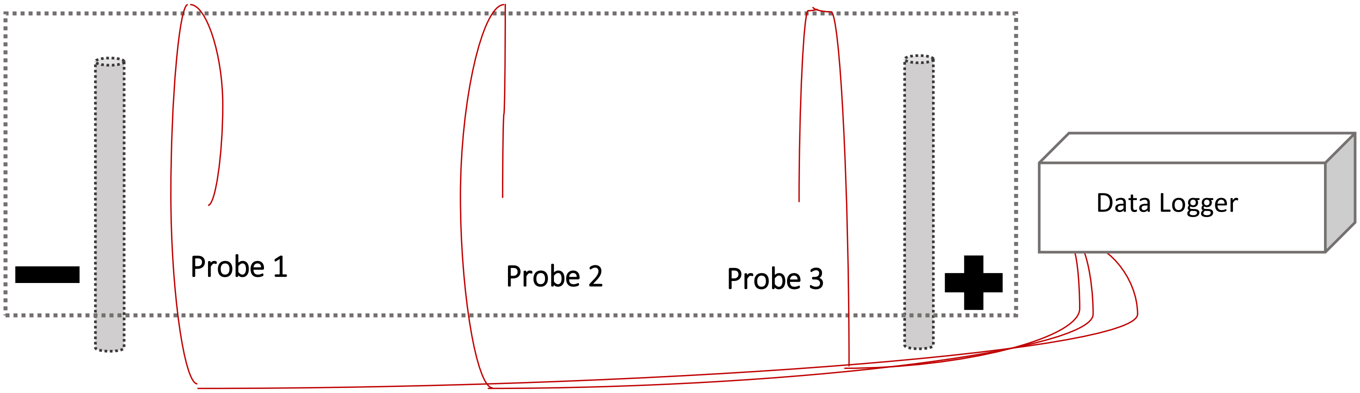


Figure 3.6 Configuration of thermocouples in the EK reactor.

The probes were left unmoved during the experiment and the temperature was registered at specific times (t=0 min, 3 min, 6 min, 10 min, 15 min, 25 min, 35 min, 45 min, 55 min, 65 min, 75 min, 85 min, 95 min, and so on…).

In Chapter 2, Table 2.5, USEPA (1994) presents four different temperature regimes as an alternative option for treatment under thermal treatments. According to the equation provided in Regime D under Alternative 1 for Class A Pathogen Reduction, “the temperature of sludge is 50° C or higher with at least 30 min or longer contact time” with the following Time-Temperature Relationship (Table 2.5):

$$D = \frac{50{,}070{,}000}{10^{0.14t}} \tag{3.2}$$

Where:
D=time in days

t=temperature in °C

This alternative can be applied to this experiment as the sewage sludge used has a solid content ranging from 0.6-6%.

3.6.2 Amperage used

The EK reactor worked under constant voltage conditions, however, a change of amperage with time was monitored with a power source at the same time as the temperature in the reactor was registered. (at time =0 min, 3 min, 6 min, 10 min, 15 min, 25 min, 35 min, 45 min, 55 min, 65 min, 75 min, 85 min, 95 min and so on). The amperage was used for power calculation.

3.6.3 pH measurement

The pH, oxidation reduction potential, conductivity, nitrates and ammonium were measured with HQ40d Digital Multi–Parameter Meter. The pH probe was connected to the meter and the first step involved calibration according to the HACH user manual using the standard pH solutions. The pH was then measured by inserting the probe in the sample collected. The probe was thoroughly rinsed with distilled water after each sample.

The pH was measured from samples of 100 ml collected from the anode and cathode area taken at a distance of 5 mm from the anode and cathode respectively. The pH of the biosolids was also measured from samples taken once the contents of the reactor were thoroughly mixed. Then average was done for each triplicate and the standard deviation was included in the results table.

3.6.4 Electrical conductivity measurements

In order to measure the conductivity, the same multi-parameter meter was used (HQ40d). The conductivity was first measured before any addition of enhancers or electrical phenomena took place. Then the conductivity was measured in samples collected at the end of the experiment.

3.6.5 Oxidation-reduction potential (ORP) measurements

The oxidation reduction potential was measured using the specific probe connected to the same HQ40d multi-parameter meter. The ORP was measured from samples of 100 ml collected from the anode and cathode area. The ORP of the biosolids was also measured from samples taken once the contents of the reactor were thoroughly mixed.

3.6.5 Total suspended solids and volatile suspended solids (TSS and VSS) analysis

3.6.5.1 Total suspended solids

The TSS were determined using the EPA method 160.2. (Appendix 1)

3.6.5.2 Volatile suspended solids

The VSS were determined by using the same sample from the TSS. (Appendix 2)

3.6.6 Ammonia (NH_3-N) and nitrate (NO_3) analysis

The concentration of ammonia was measured at the end of each experiment. Same as for pH and ORP the HQ40d Digital Multi–Parameter Meter was used to measure the NH_3-N and NO_3^-

The probe used for NO_3-N is the ISENO 3181. It was calibrated according to the steps shown in the user manual: (Appendix 3).

The probe used for NH_3-N is the ISENH 3181. It was calibrated according to the steps shown in the user manual: (Appendix 4).

3.6.7 Particle size distribution measurement

The particle size distribution was determined using the Partica, laser scattering, HORIBA.

In order to determine the particle size distribution, the following specifications were followed:

1. *Partica* is turned ON
2. The software LA–950V0 was used
3. The software was used to Set the conditions:
    - Refractive index: 1.52
    - ASTM and Phi scale
4. The sample is diluted for transmittance stays above 70%
5. When the measurements ended, the graph functions provided by the software were used to save the data for further analysis.

When the measurements ended, the graph functions provided by the software were used to save the data for further analysis.

3.6.8 Fecal coliform analysis

In order to determine if the EK treatment using the BioElectro™ and enhancing agents at small concentrations are sufficient to comply with Class A biosolids, fecal coliforms where determined for selected samples. Selected samples underwent the treatment at 2.5V/cm with different BS and AN concentration. The fecal coliform was measured in CFU/100mL and CFU/dry gram and was determined using the membrane filtration test.

Prepared agar plates (BBL^{TM} MI Agar) and sterile Membrane filter were used in this step of the experiment. Dilution of 100-200 times was used with Milli–Q R water (Millipore–Advantage A10) in order to obtain between 20-80 colonies per filter.

Steps:

1. All the testing tools were disinfected using 70% ethanol before and after each sample.
2. Using sterilized forceps, a membrane filter was placed, grid side up, onto the filtration assembly
3. The sample was inverted for 30 seconds, then poured into the funnel.
4. Vacuum was applied in order to filter the sample, and the walls are rinsed with distilled water.
5. Using sterilized forces, the membrane was transferred onto the MI agar plate with a slight rolling motion, side up. The agar plate is checked for air trapped under the filter and proper contact is insured.
6. The petri dish lid was placed and labeled and sealed with parafilm.
7. The petri dish was inverted and incubated at 35 ±0.5 °C for 22-24 hours.
8. After incubation, the colonies are counted and recorded. The number of blue or indigo colonies under normal/ambient light represent the *E. coli* count.
9. Then the plate was exposed to long-wave ultraviolet light (366 nm) to obtain the total coliform count

CHAPTER 4: RESULTS AND DISCUSSION

A series of experimental tests as defined in the Chapter 3.5 permitted to generate information about relationships between the concentrations of conditioners and the temperature rise in EK reactors containing various types of sludge. In Phase 1, Stage 1 combined (primary to secondary = 1:1) sludge was applied. While in the Stage 2 of Phase 1 wasted activated sludge (WAS) with a total solid content (TS) of 5% was used. However, in Phase 2, WAS containing TS of 0.6% and 6% were applied in Stage 1 and 2 respectively.

These stages permitted to assess the period required for an adequate temperature increase in various experimental conditions.

Furthermore, the change of physical, chemical and biological characteristics such as pH, ORP, conductivity, nitrates and ammonia, TSS and VSS, particle size distribution and fecal coliform were evaluated.

4.1 Temperature rise in the EK cell

4.1.1 Phase 1, Stage 1

In this Stage, EK treatment was applied to combined sludge (1:1) denoted as OM. Three different initial temperatures were used (5°C, 15°C, 20°C) when a higher voltage (2.5 DC V/cm) was used.

The conditioner concentration ranged from 0.5g/L to 26g/L. As expected at higher concentrations, the rate of temperature increase will be higher. At the initial temperature of 20°C the temperature in the EK reactor with OM-10 (BS=25g/L and AN=26g/L), reached 53°C in only 14 minutes (Fig 4.1). For reactor OM-8 (BS=1g/L and AN=1g/L), 48°C was reached in 65 minutes. Thus, according to the second-degree equation for OM-8 from Table 4.1, the temperature would have reached 53°C in approximately 75 minutes. For the sample OM-7, which had a half of the concentration of OM-8, the time required to reach 50°C was 80 minutes.

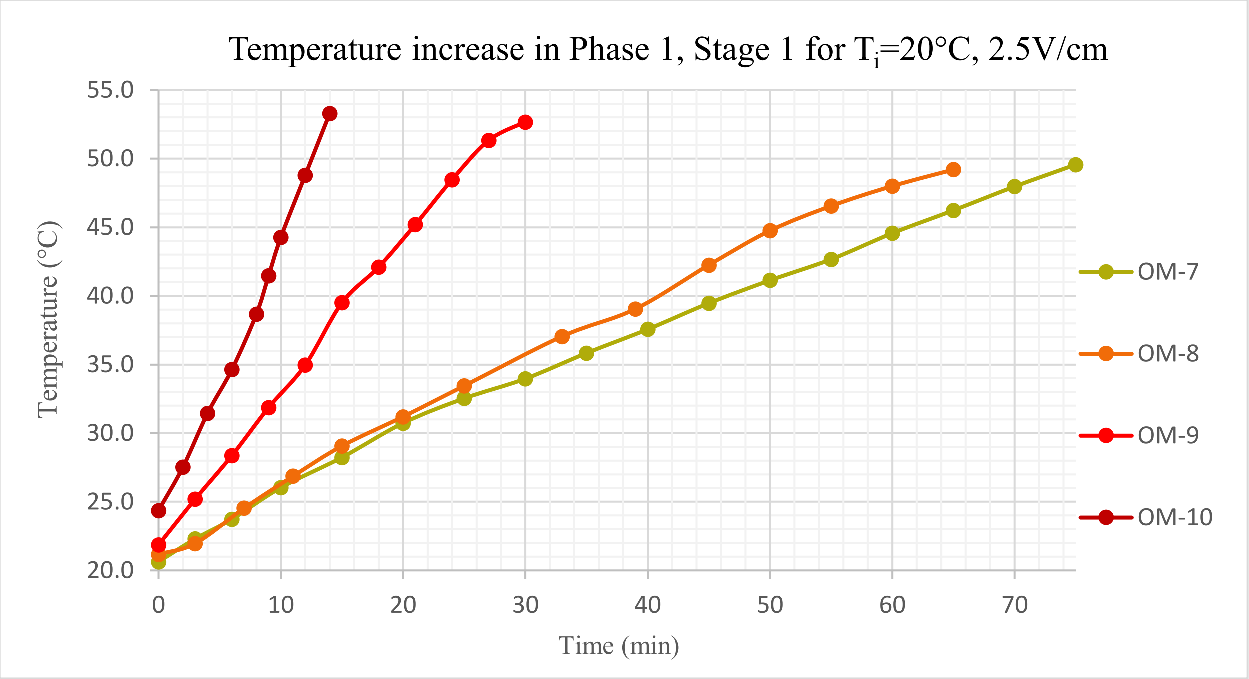


Figure 4.1 Temperature increase in Phase 1, Stage 1 (combined sludge 1:1) for Ti=20°C, 2.5V/cm OM-7=0.5g/L BS/ 0.5g/L AN=[0.5/0.5], OM-8=[1/1], OM-9=[7/7], OM-10=[25/26].

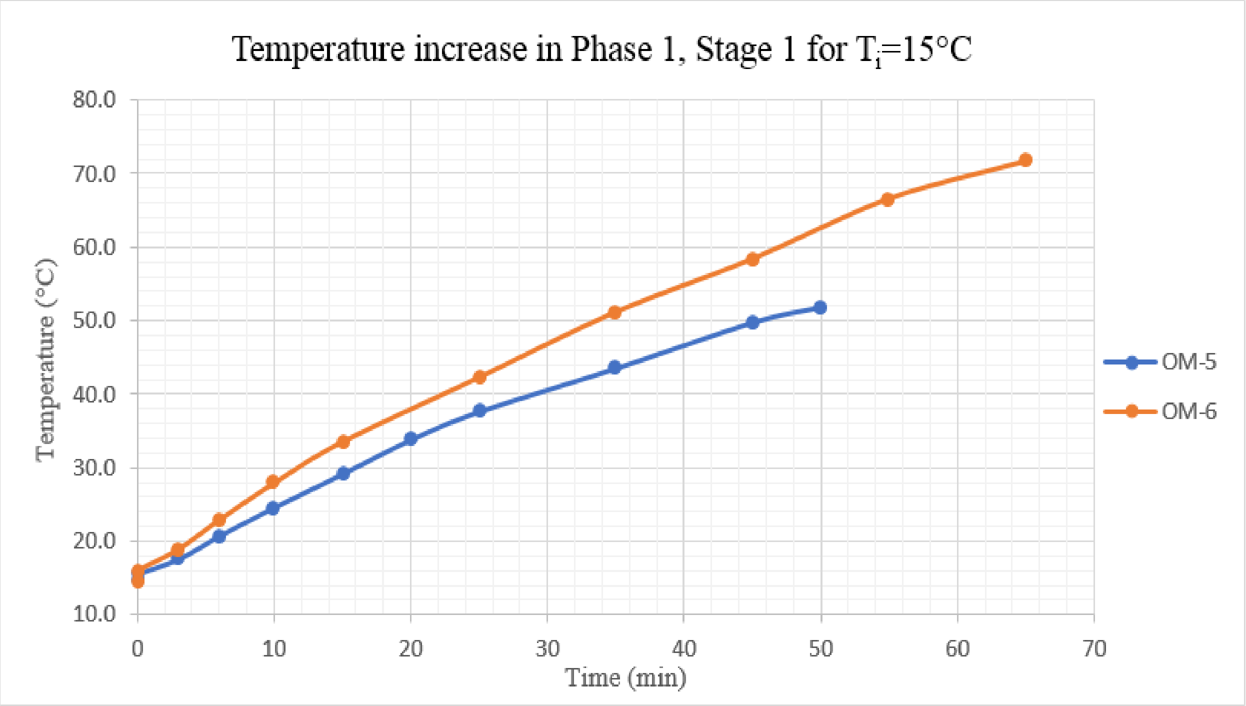


Figure 4.2. Temperature increase in Phase 1, Stage 1 (combined sludge 1:1) for T_i=15°C. OM-5=[1/6], OM-6=[2/6].

Figure 4.2 shows the temperature increase for combined sludge at initial temperature of 15°C. The sample OM-5 (BS=1g/L and AN=6g/L) reached 50°C in 50 minutes while OM-6 (BS=2g/L and AN=6g/L) reached 66°C in 55 minutes. It can be observed that when the BioxyS concentration was doubled, the rate of temperature increase was significantly higher.

For sample OM-6, the temperature increase in the EK cell is relatively uniform. The temperature gradient between anode, cathode and in the middle of the reactor was low and it continued to decrease with time. At the end of the experiment, the middle of the reactor was only 1 degree lower than at anode, which had the highest temperature value.

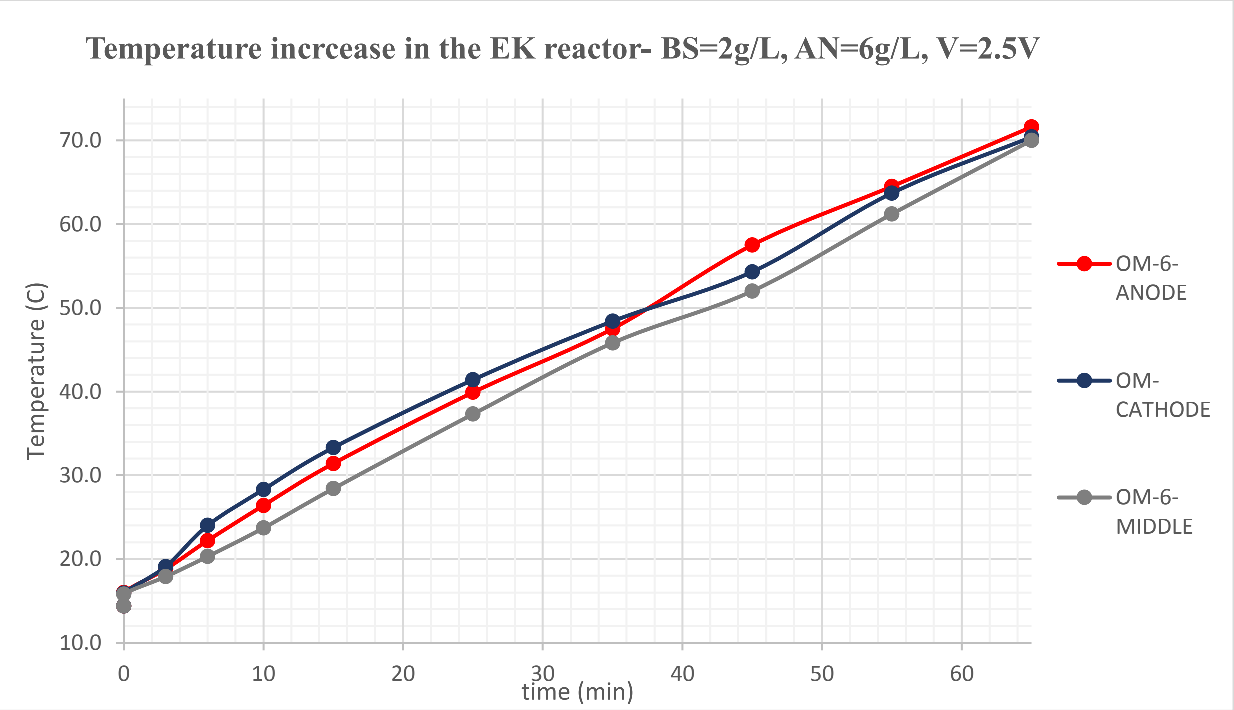


Figure 4.3. Temperature increase in Phase 1, Stage 1 (combined sludge 1:1) throughout the reactor for Ti=15°C. OM-6=[2/6].

At an initial temperature of 5°C, EK treated sludge reached a temperature of 64°C under one hour for sample OM-4 (BS=8g/L and AN=8g/L). For OM-3 (BS=2g/L and AN=2g/L), the rate of temperature increase was slower, with sludge reaching 50°C in 85 minutes (Fig. 4.4).

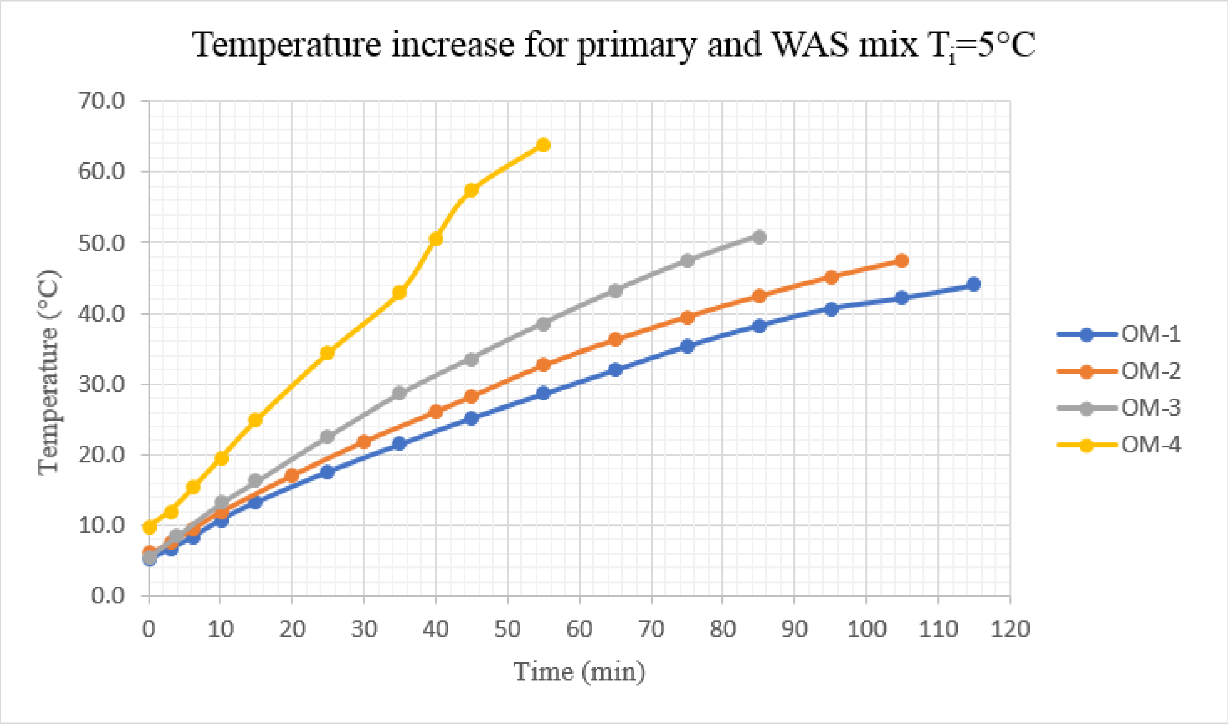


Figure 4.4. Temperature increase in Phase 1, Stage 1 (combined sludge 1:1) for T_i=5°C. OM-1=0.5g/L BS/ 0.5g/L AN=[0.5/0.5], OM-2=[1/1], OM-3=[2/2], OM-4=[8/8], V=2.5V/cm.

Figure 4.5 shows the performance of combined sludge at very high concentration (BS=25g/L, AN=26g/L) and voltage gradient of 2.5V/cm. At anode, the temperature reached 50°C in just 25 minutes while the middle of the reactor reached 50°C in just 27 minutes.

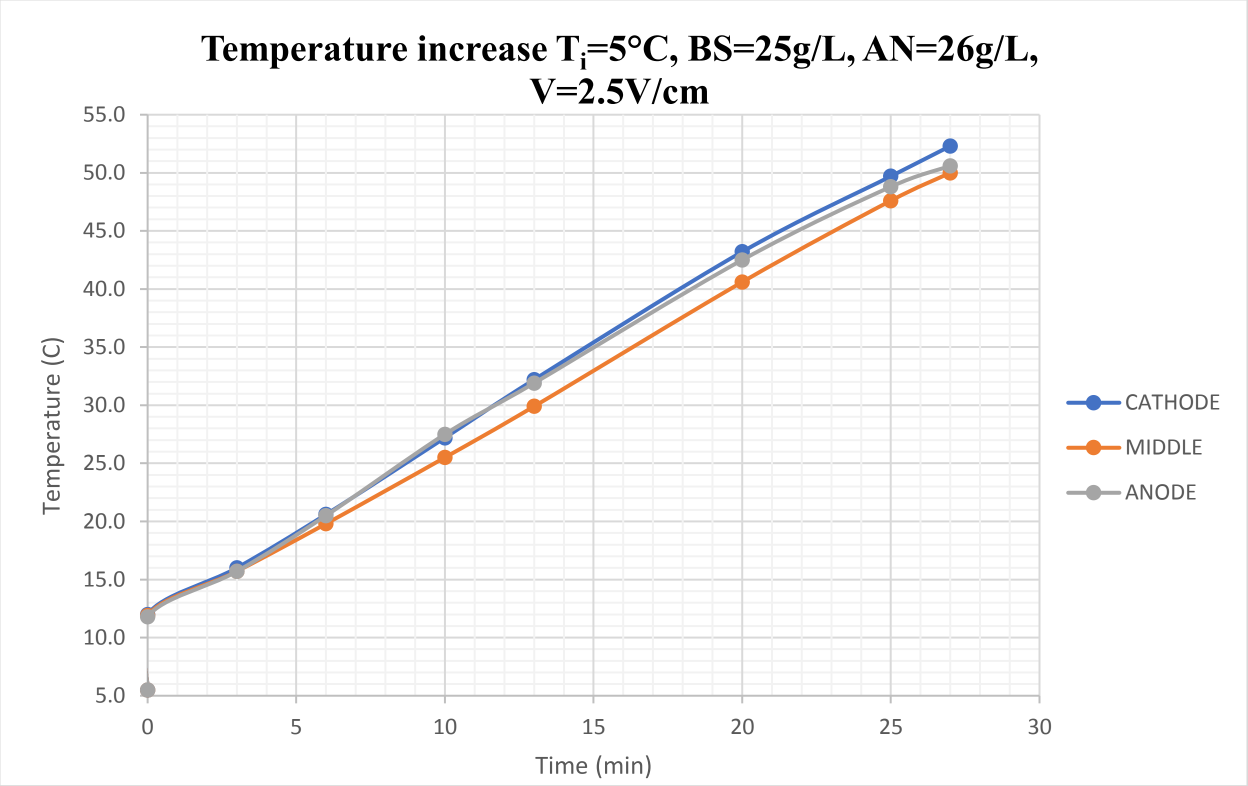


Figure 4.5 Temperature increase at anode and cathode in Phase 1, Stage 1 (combined sludge 1:1) for Ti=5°C. BS=25g/L, AN=26g/L, V=2.5V/cm

Overall, combined sludge that underwent EK treatment with enhancers showed a promising rate of temperature increase. As expected, the rate of increase is directly related to the concentration of conditioners. The initial temperature in the reactor also played an important role in the rate of increase. For example, OM-1 and OM-7 had the same concentrations, but different initial temperatures ($OM\text{-}1_i$=5°C and $OM\text{-}7_i$=20°C), and OM-1 required an extra 55 minutes to reach 45°C. Similarly, the samples OM-2 and OM-8 with the same concentrations ($OM\text{-}1_i$=5°C and $OM\text{-}8_i$=20°C) and OM-2 required an extra 45 minutes to reach 45°C.

4.1.2 Phase 1, Stage 2

In the Stage 2, EK at 2.5 DC V/cm was applied to the thickened WAS (6% TS) at the initial temperature of 15°C (Fig. 4.6). The rate of temperature increased significantly (in 40min) in sample O-2 to reach 65°C when the high concentrations of enhancers were used. Samples containing AN=6g/L and BS=2g/L (O-5), reached the target temperature (65°C) in 57 minutes (Fig 4.6).

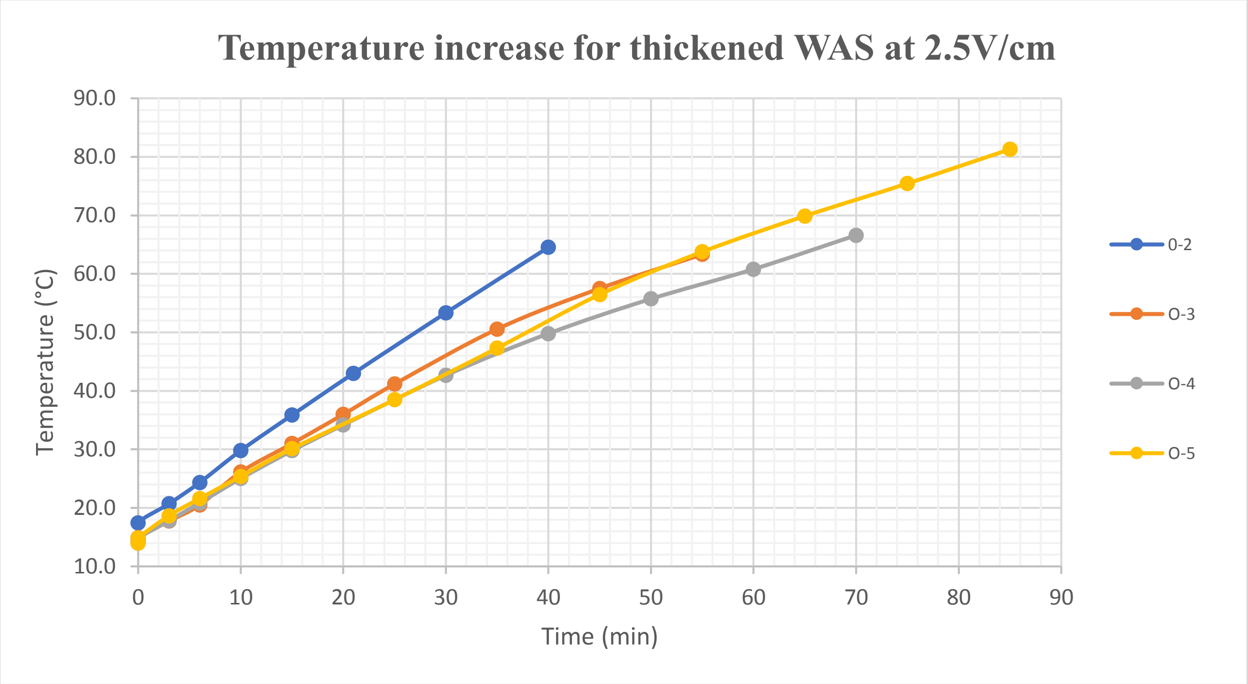


Figure 4.6 Temperature increase in Phase 1, Stage 2 (WAS 5% TS) for T_i=15°C. O-2=8 g/L BS/ 8g/L AN=[8/8], O-3=[1/6], O-4=[0.5/3], O-5=[2/6].

Figure 4.7 shows the temperature increase for WAS (5%TS) at two different concentrations at lower (2.0 V/cm) voltage gradient. Although the temperature increase for the two concentration had very similar values initially, it can be observed from the Figure 4.7, that, as time passes, the adequate concentration of BS will ensure a constant temperature increase, which is required in achieving the target temperature. With a higher BS concentration (2 g/L), the system will reach the required temperature about 15 minutes faster.

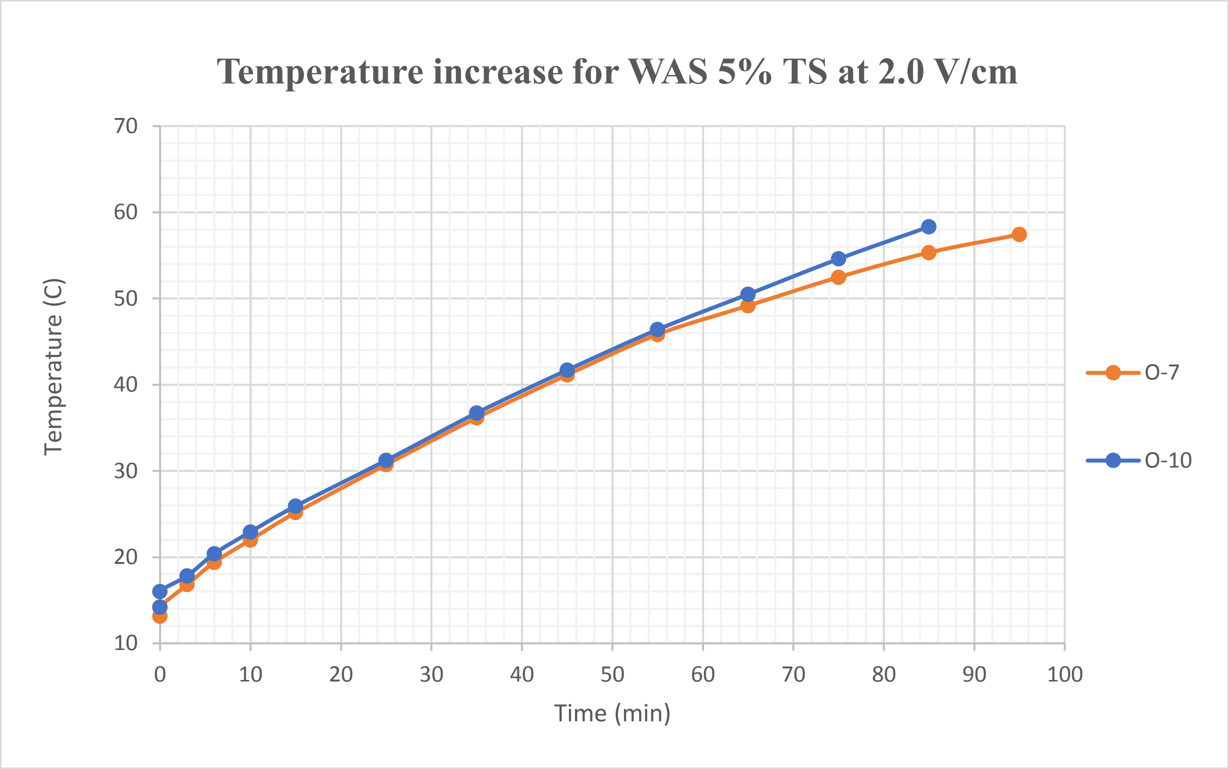


Figure 4.7 Temperature increase in Phase 1, Stage 2 (WAS 5% TS) at low voltage. O-7= [1/6], O-10=[2/6].

As expected, EK treated WAS at a higher voltage gradient will decrease the reclamation time significantly. For example, as shown in Fig.4.8, the time required to achieve successful treatment is almost halved using the same concentration, but at higher voltage gradient. Retention time is very important in order to determine design at full scale and save on required space needs.

Results showed that the following concentrations of conditioners: BS=2g/L and AN=6g/L express a stable reaction with efficient temperature rise. It was also observed that AN tends to increase the temperature more at cathode at its higher concentrations. Mixing of the system might be suggested to keep a uniform temperature in entire EK cell. An experiment was conducted where the sludge would me mixed every 10 minutes. It was noticed that the current intensity would drop by about 0.5-1.0 A due to changes in sludge properties. It might affect the rate of temperature

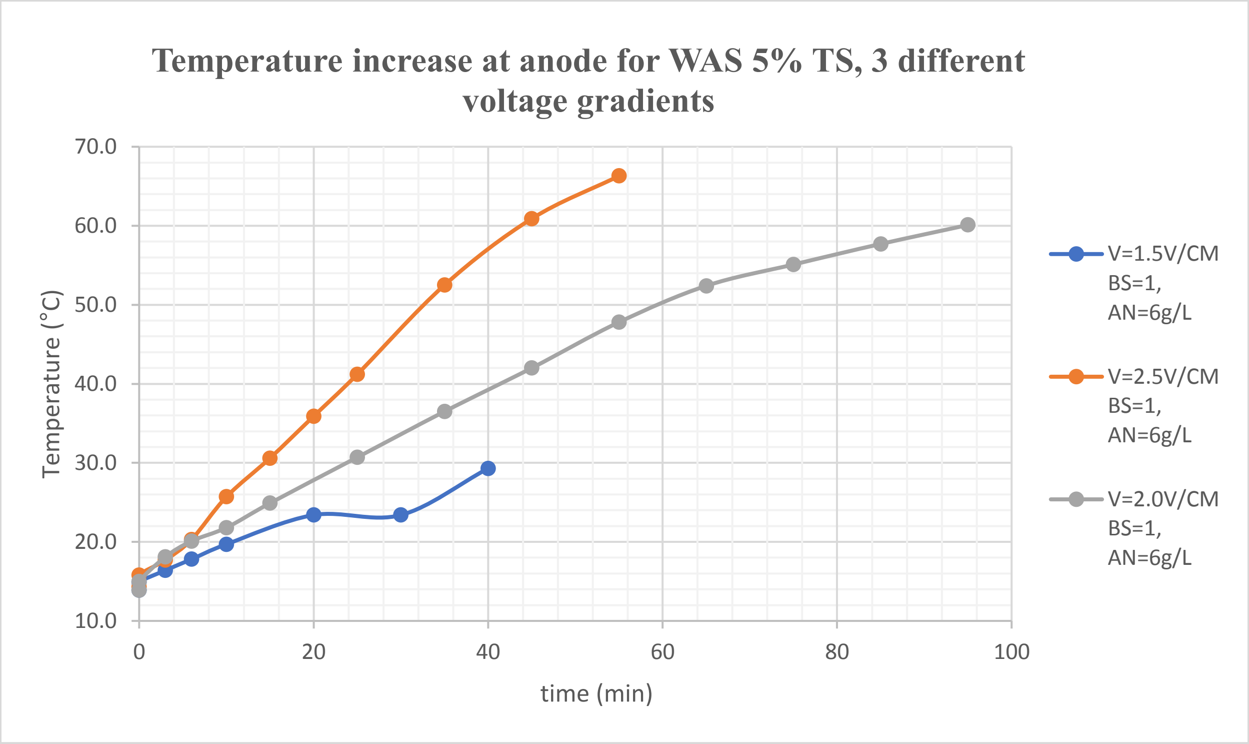


Figure 4.8. Temperature increase Phase 1, Stage 2: comparison of voltage gradient.

increase. Therefore, by adding BioxyS, the temperature at anode will increase at about the same rate as the temperature at cathode- a ratio of 3:1 (AN:BS) would be applied to achieve satisfactory results.

Figure 4.8 shows that a higher voltage gradient will have a significant impact on temperature increase. For example, by deriving the second-degree polynomial to calculate the rate of increase for 2.0 and 2.5V/cm at BS=2g/L and AN=6g/L the importance of higher voltage is proven. As a result, for 2.0 V/cm, $dT/dt= -0.005t+0.7$ which so a decrease in the temperature rate with time; at 10 min, for 2.0V/cm, $dT/dt=0.65$°C/min, and at 50 min, $dT/dt=0.45$°C/min. For 2.5V/cm, $dT/dt=-0.013t+1.33$; at 10 min, $dT/dt= 1.20$°C/min, while at 50 min, $dT/dt=0.68$°C/min (Table 4.1). Therefore, at higher voltage gradient (2.5V/cm), the loss in the rate temperature increase will be lower compared to a lower voltage gradient (2.0V/cm).

4.1.3 Phase 2, Stage 1

In this stage, the sludge collected in return having initial 0.6% TS was used (Chapter 3.5). In order to examine the effects of enhancing agents on the sludge in the BioElectro, several concentrations were applied. The initial temperature in this Stage was 14°C. An amount of 8g/L was the highest concentration for AN and BS. Two voltage gradients such as 2.0V/cm (Figure 4.9) and 2.5V/cm (Figure 4.10) were applied. The linear equation confirmed that higher concentrations increase the rate of temperature in the EK reactor. SHR-3 and SHR-4 have the same AN concentration while the BS concentration for SHR-3 is half that of SHR-4 but the time required to reach 70 °C was the same.

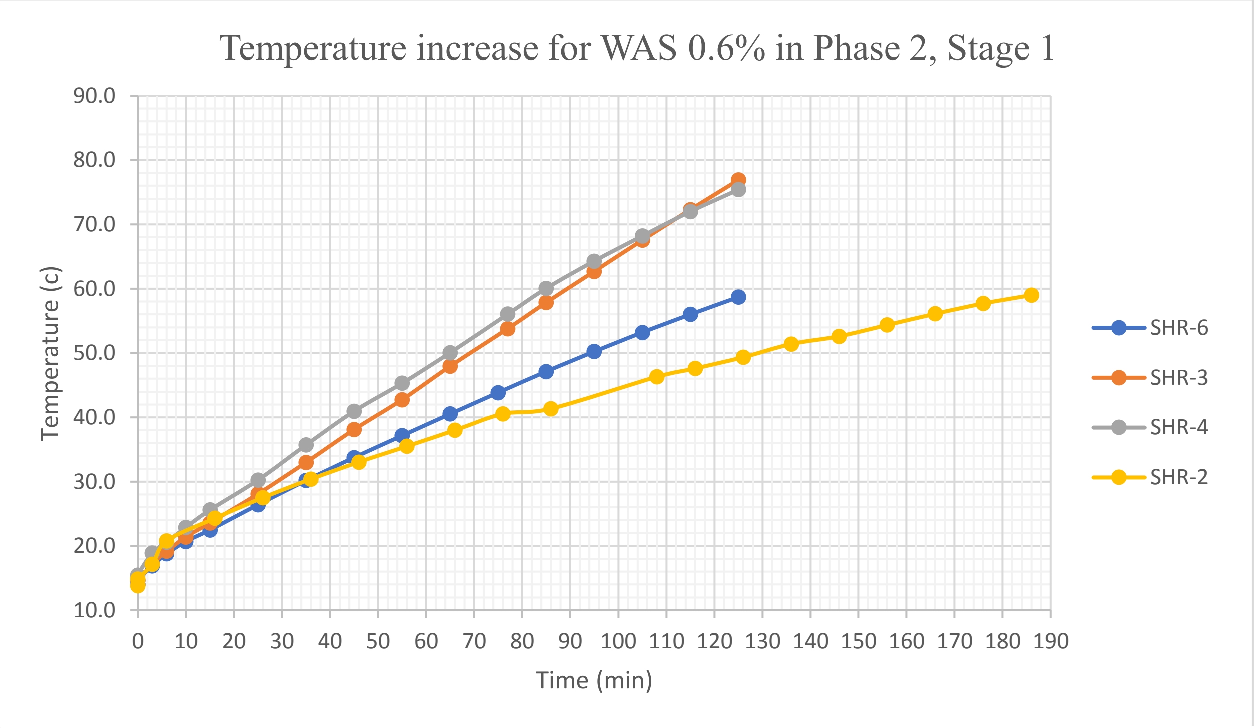


Figure 4.9 Temperature increase for Phase 2, Stage 1 (WAS 0.6% TS), T_i= 15 °C, at low voltage. SHR-2=[2(g/L BS)/6(g/L)AN], SHR-3=[4/8], SHR-4=[8/8], SHR-6=[3/6].

Once again, in Figure 4.10, the contribution of AN can be observed to the temperature increase; the slope for SHR-7 is double the slope for SHR-8, when the AN concentration was double.

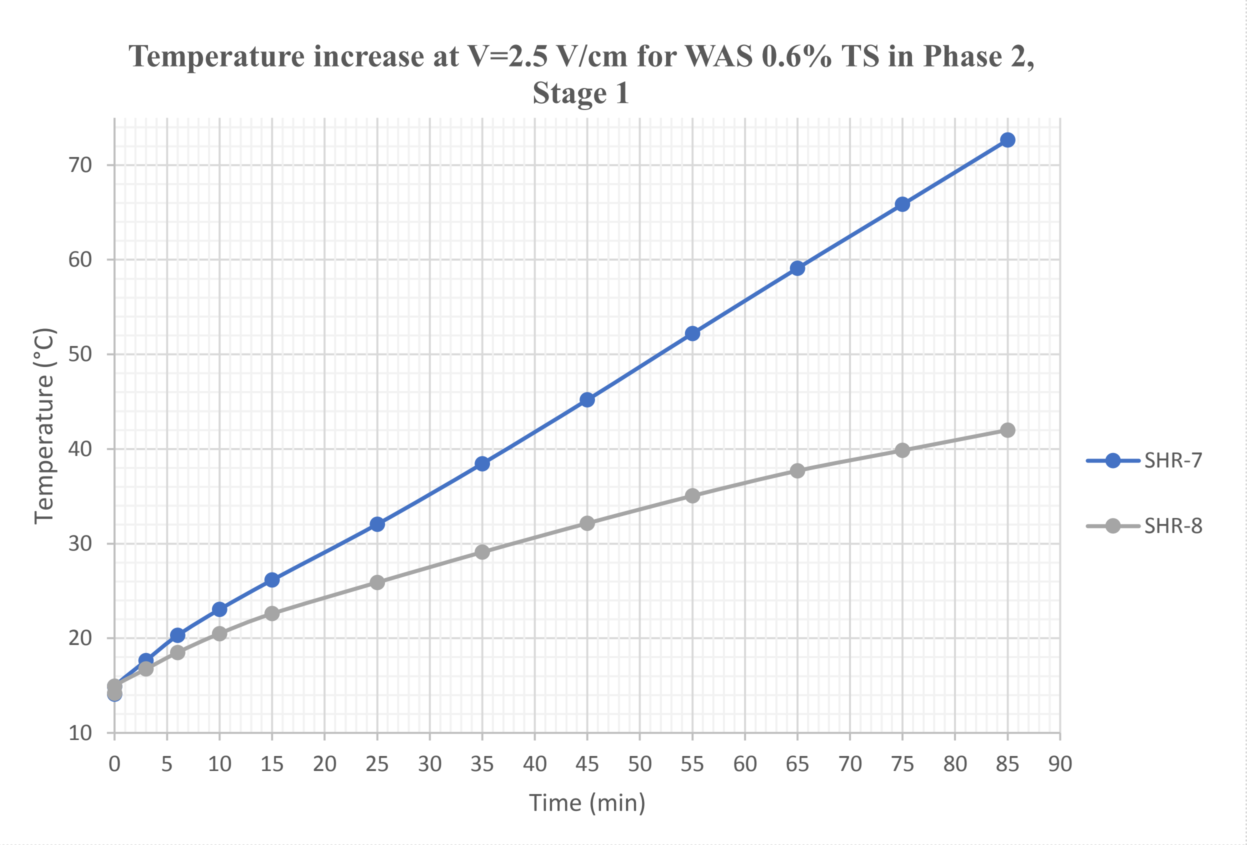


Figure 4.10. Temperature increase Phase 2, Stage 1 (WAS 0.6% TS), T_i=15C, high voltage, SHR-7= [2/6], SHR-8= [2/1].

Figure 4.11 shows that higher voltage gradient can have a great impact on the treatment time required. By increasing the voltage gradient by 0.5 V/cm, the required treatment time might decrease by about 100 minutes since the rate of temperature rise is 3 times higher. It was also observed that a ratio of AN:BS=3:1 would provide more stable increase of temperature due to the effect of the BS at the cathode. At high concentration of BS (>8g/L) an unstable temperature increase at the cathode area was observed.

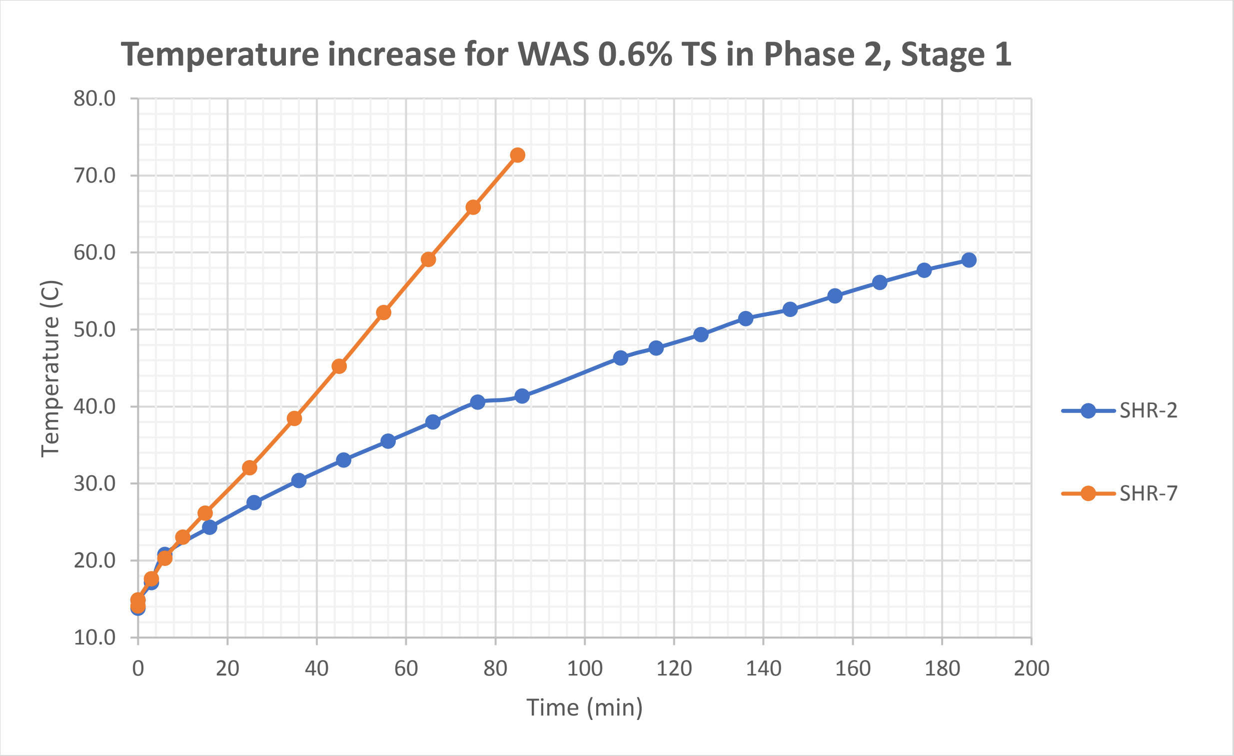


Figure 4.11. Temperature increase Phase 2, Stage 1(WAS 0.6% TS), for T_i= 15°C: comparison of low and high voltage. , SHR-7= [2/6]-2.5V/cm, SHR-2= [2/6]-2.0V/cm.

At a high voltage, the temperature at the anode (for sample SHR-7) reached 65°C 10 minutes faster than at the cathode (Fig 4.12). At low voltage, the temperature at the cathode (for sample SHR-2) reached 60°C about 15 min faster than at the anode (Fig. 4.12)

The study showed that the sludge characteristics is a determining factor for conditioners needs. As compared to WAS 5% TS and combined sludge from Phase 1, the sludge used in Phase 2, Stage 1 (WAS 0.6% TS) would need more time to reach the required temperature for Class A biosolids. This can be rectified by adding a higher concentration of enhancers in the reactor.

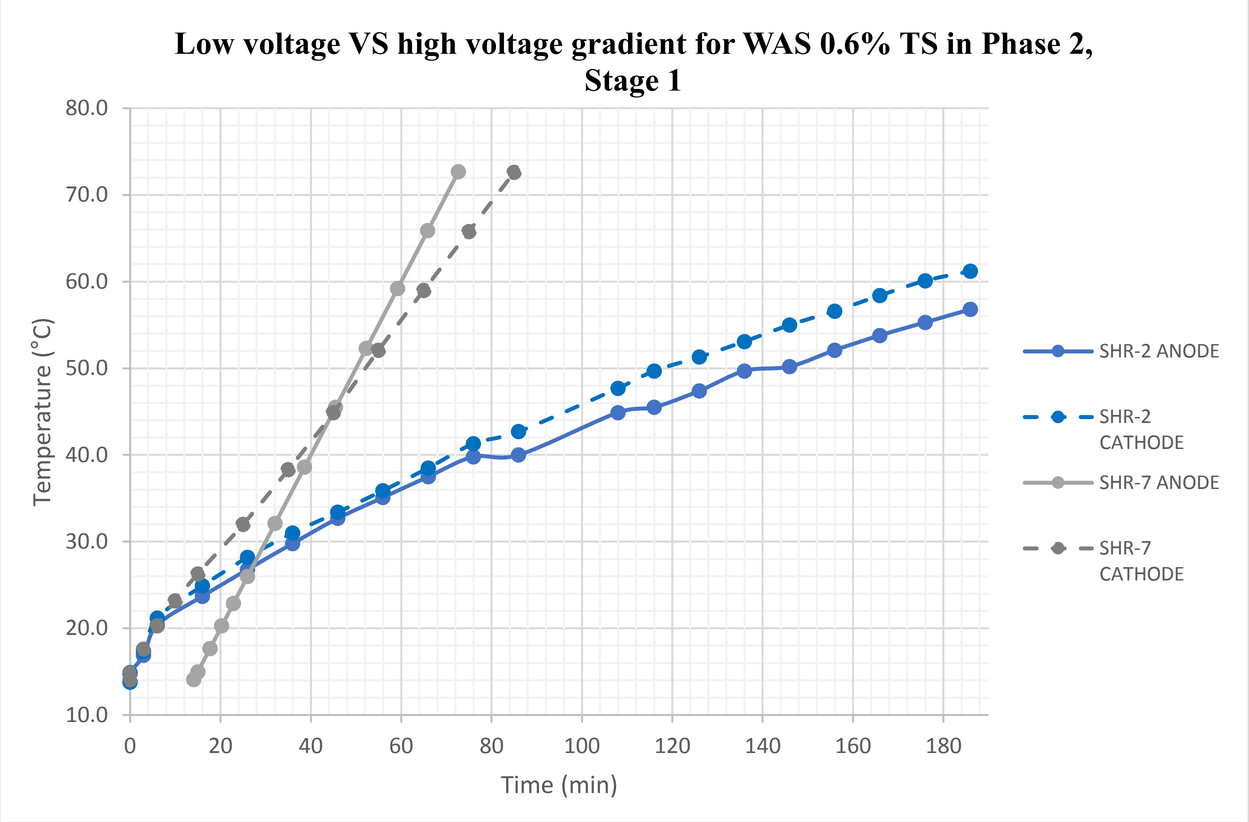


Figure 4.12. Temperature increase in Phase 2, Stage 1 (anode and cathode). Low voltage gradient vs high voltage gradient. BS=2, AN=6g/L.

4.1.4 Phase 2, Stage 2

In Stage 2, thickened WAS was used. The WAS was centrifuged with coagulants and at collection time it had 6% TS. Different concentrations of conditioners and two different voltage gradients were used to compare the stability of the temperature increase.

Samples SH-1 (BS=1g/L and AN=3 g/L) and SH-2 (BS=2g/L and AN=6 g/L) underwent EK treatment at a higher voltage (2.5V/cm). Sample SH-2 reached 65°C in 55 minutes, while sample SH-1 reached 65°C in 75 minutes. It was concluded that higher concentrations of conditioners (Fig. 4.13) will influence the temperature rise in the EK reactor, e.g. SH-2 reached 65°C 20 minutes faster than SH-1. Figure 4.15 shows that when the concentration doubled (from 3g/L to 6 g/L), the temperature maintained a linear temperature rise, reaching 84°C in 75 minutes.

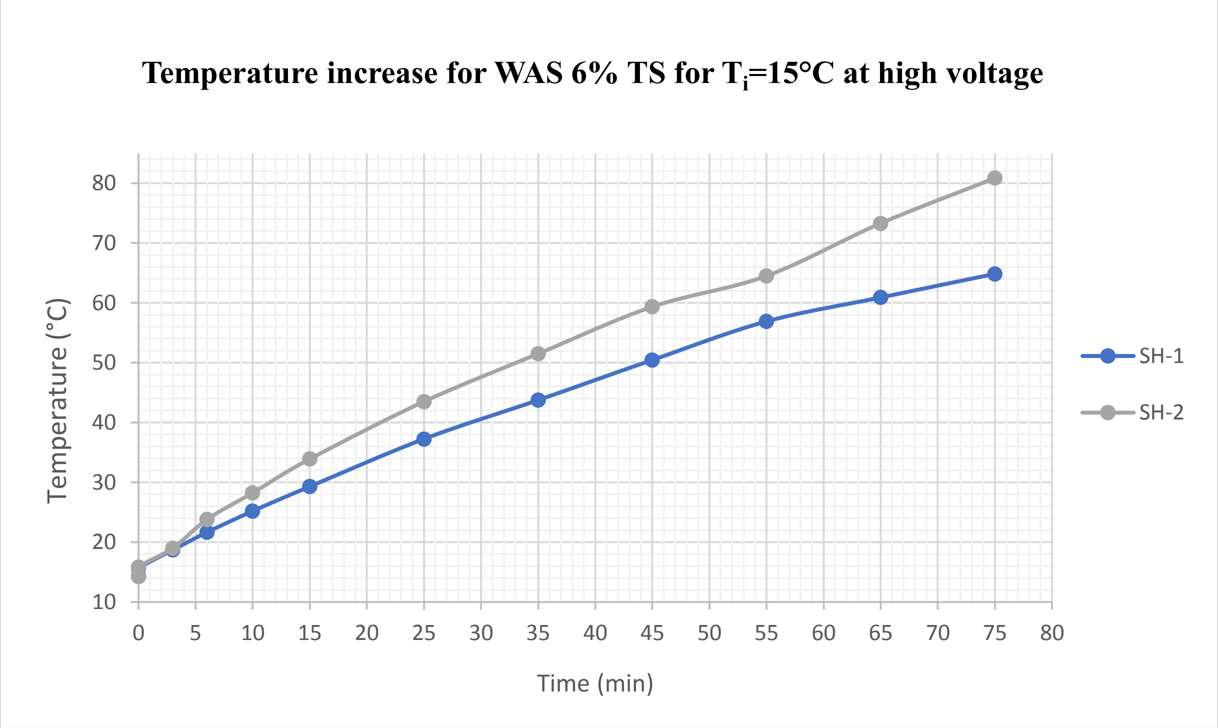


Figure 4.13 Temperature increase in Phase 2, Stage 2 for T_i=15°C at high voltage. SH-1=[1g/L BS/3g/L AN], SH-2=[2/6].

Samples SH-4 (BS=1g/L; AN= 3g/L) and SH-5 (BS=2g/L; AN= 6g/L) underwent EK treatment at 2.0V/cm. SH-4 reached 65°C in 140 minutes, while SH-5 in 170 minutes (Figure 4.14). It was also observed that at a lower voltage gradient, the rise in temperature at cathode was also higher than the rise at the anode when the concentration was doubled (Figure 4.15).

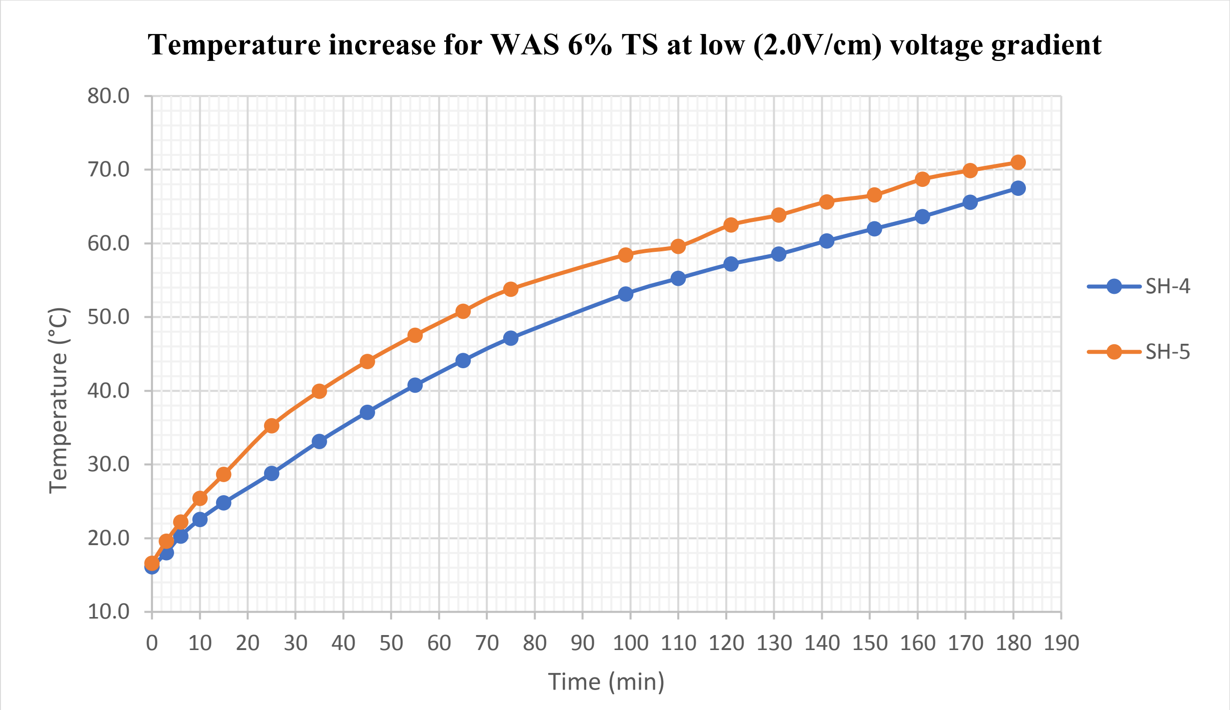


Figure 4.14 Temperature increase in Phase 2 Stage 2 (WAS 6% TS) at low voltage (2.0V/cm). SH-4=[1g/L BS/3g/L AN], SH-5=[2/6].

By comparing SH-2 and SH-5, it can be concluded that the temperature at the cathode at the end of the experiment was higher than at the anode (Figure 4.15). At a lower voltage gradient, the temperature at the anode for samples SH-4 and SH-5 had very similar values after 2 hours of treatment. However, the higher temperature at cathode for sample SH-5 increased the average temperature in the reactor to 72°C at the end of the experiment (t=180 minutes). By comparing the results shown in Figures 4.3, 4.5 and 4.15, it can be observed that the temperature gradient between electrodes depends on the properties of the sludge. It seems that a lower TS content in the sludge will give a more uniform temperature between the anode and cathode.

Overall, the voltage gradient had the biggest impact (at lower concentrations of conditioners) on the rate of temperature increase. As shown in Figure 4.16, for the same concentrations (BS =2g/L; AN =6g/L), the temperature in sample SH-2 reached 65°C about 80 minutes faster than sample SH-5.

EK treatment proved to be very successful for thickened WAS with enhancers at concentrations of AN=6g/L and BS=2g/l and voltage gradient of 2.5V/cm (Figure 4.13). In this case, the time required to achieve the desired treatment temperature of 65°C was 55 minutes and it only took 75 minutes to reach 80°C.

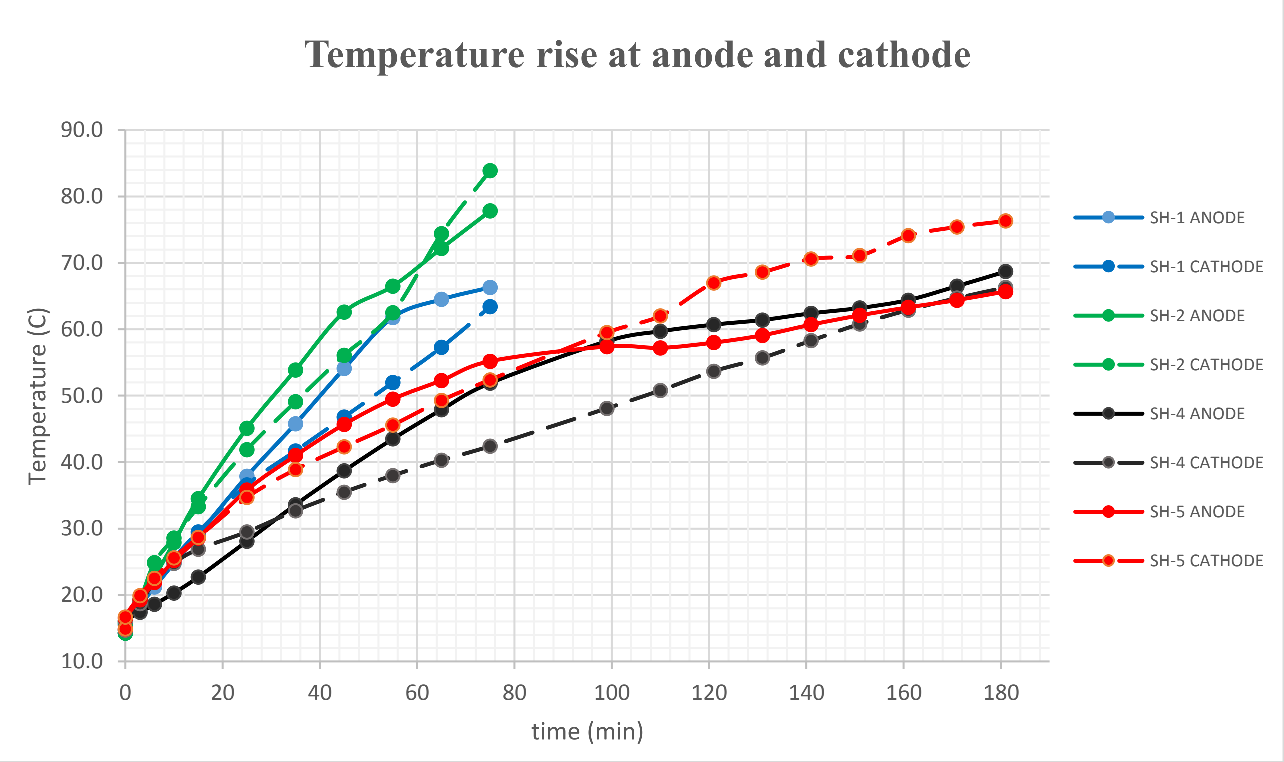


Figure 4.14. Temperature rise at anode and cathode, 2 different concentrations, 2 different voltage gradients (SH-1=[1/3], SH-2=[2/6]-2.5V/cm, SH-4=[1/3], SH-5=[2/6]-2.0V/cm).

Thickened WAS (6% TS) is more suitable for electrokinetic treatment using enhancers due to the characteristics acquired after addition of coagulants. The results presented in Figure 4.11 and Figure 4.16 showed that thickened activated sludge reached 65°C, 20 minutes faster than activated sludge from the return (55 minutes vs 75 minutes) under the same conditions. Therefore, it can be concluded that sludge properties are very important in achieving a fast rise of temperature.

In conclusion, waste activated sludge from the Saint Hyacinthe WWTP could successfully undergo electrokinetic treatment using the BioElectro configuration. As it is shown in Figures 4.13 to 4.16, thickened WAS was more suitable for the EK treatment due to its characteristics, including a high TS content. Thus, the BioElectro system was able to reach the temperature required in order to achieve Class A in less than one hour at a voltage gradient of 2.5V/cm and enhancer concentrations of AN=6g/L and BS=2g/L.

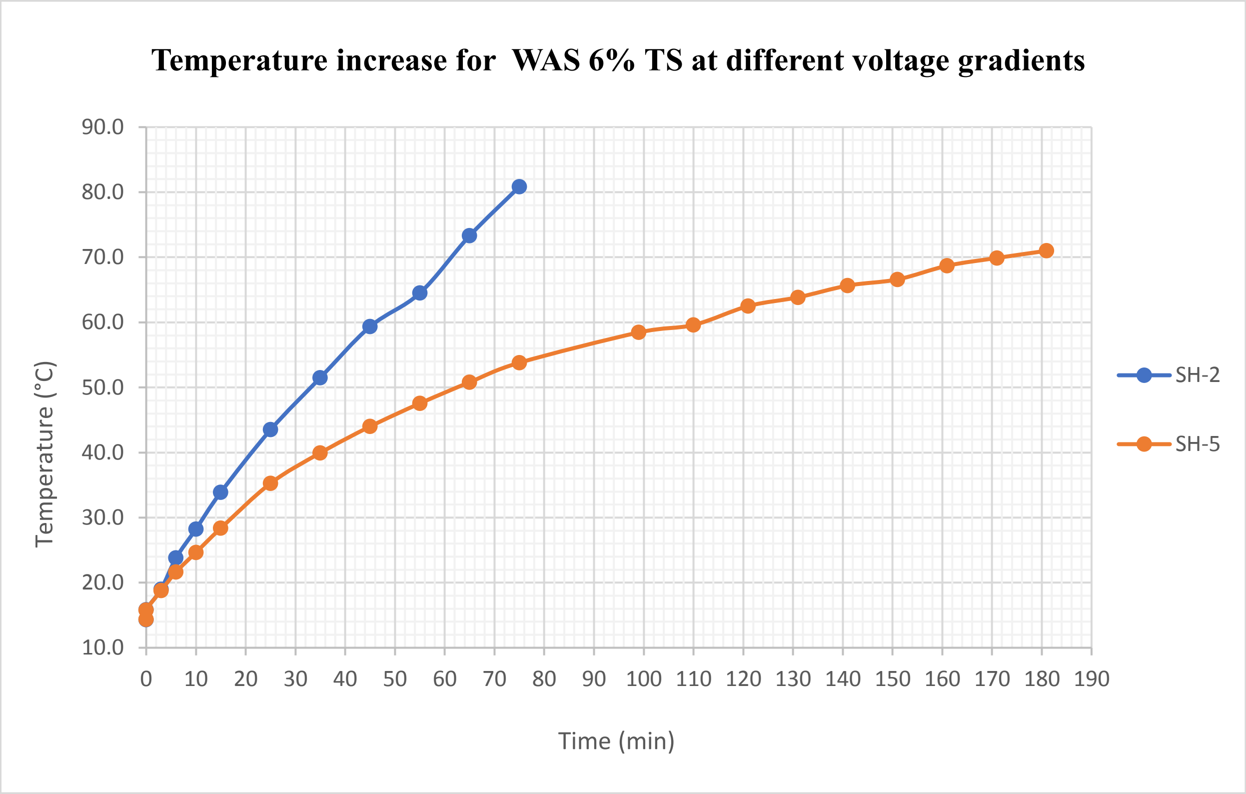


Figure 4.16 Temperature increase in Phase 2, Stage 2 for T_i=15°C: comparison of low and high voltage [BS]=2g/L and [AN]=6g/L.

4.1.5 Conclusion on temperature rate

In this experimental study, electrokinetics was used to achieve thermal treatment of waste activated sludge. According to the equation provided in Regime D Class A pathogen reduction, sludge containing 7% or less solids can follow a time-temperature relationship (USEPA, 2012). Therefore, at a temperature of 65°C, the treatment time required for sludge is just under 2 hours.

Four types of sewage sludge were used in order to compare its behavior in the BioElectro reactor. The sludge underwent EK treatment at different concentrations of ammonium nitrate and BioxyS and two different voltage gradients: 2.0V/cm and 2.5V/cm. After assessing the rate of temperature rise at different concentrations, a ratio of AN:BS=3:1 where AN=6g/L and BS=2g/L was recommended. Table 4.1 presents the relationship between time (t) and temperature (T) which is defined based on experimental data. As seen in Figures 4.1 to 4.17, the temperature rise is not exclusively linear, the thickened sludges (WAS 5% and WAS 6%) are represented by second degree equations (Table 4.1).

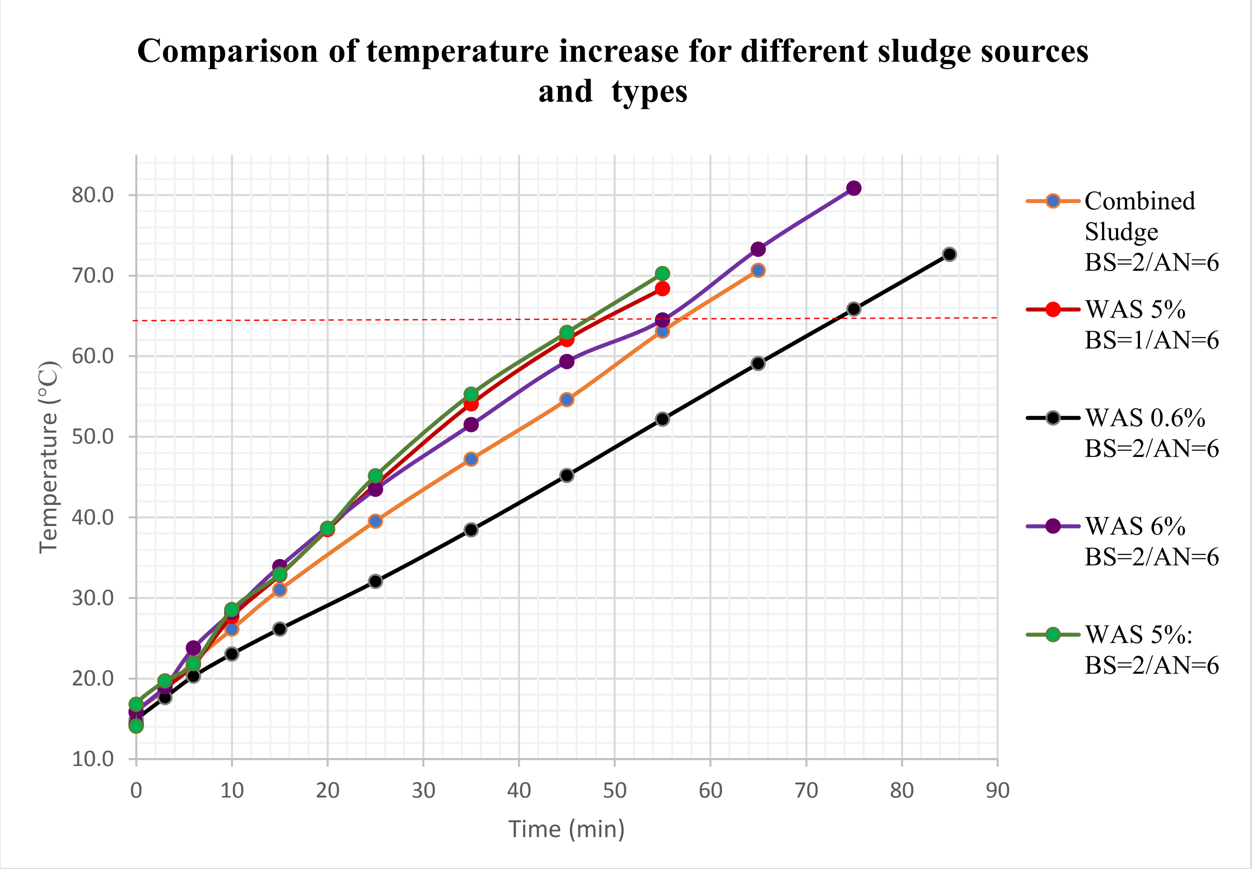


Figure 4.17 Comparison of temperature increase for Phase 1 and Phase 2 at different concentrations at higher voltage gradient (2.5V/cm).

Table 4.1 Temperature rise equations for Phase 1 and Phase 2. T=temperature, t=time.

	Voltage Gradient					
	2.0 V/cm			**2.5 V/cm**		
	Ref #	**Equation**	**Regression coefficient (R^2)**	**Ref #**	**Equation**	**Regression coefficient (R^2)**
Phase 1 Stage 1				**OM-10**	$T = 2.0855t + 23.212$	0.993
				OM-9	$T= 1.0686t + 22.292$	0.995
				OM-8	$T = 0.4449t + 21.762$	0.995
				OM-7	$T = 0.3766t + 22.1$	0.994
				OM-6	$T = 0.849t + 16.684$	0.988
				OM-5	$T = 0.7943t + 15.96$	0.910
				OM-3	$T = 0.539t + 7.7545$	0.990
				OM-2	$T = 0.4045t + 8.1994$	0.989
Phase 1 Stage 2	**O-7**	$T = -0.0029t^2 + 0.7308t + 14.408$	0.997	**O-2**	$T= 1.2148t + 16.85$	0.992
	O-10	$T = -0.0021t^2 + 0.6753t + 15.759$	0.998	**O-3**	$T = -0.006t^2 + 1.2314t + 14.146$	0.999
				O-4	$T = -0.0048t^2 + 1.0695t + 14.61$	0.999
				O-5	$T = -0.0033t^2 + 1.0606t + 14.854$	0.999
Phase 2 Stage 1	**SHR-2**	$T = 0.2288t + 19.765$	0.966	**SHR-7**	$T = 0.6717t + 15.402$	0.999
	SHR-3	$T = 0.4948t + 15.582$	0.992	**SHR-8**	$T = 0.3227t + 16.464$	0.982
	SHR-4	$T = 0.4854t + 17.517$	0.994			
	SHR-6	$T = 0.3521t + 16.602$	0.993			
Phase 2 Stage 1	**SH-4**	$T = -0.0016t^2 + 0.5633t + 19.805$	0.990	**SH-1**	$T = 0.6835t + 17.547$	0.992
	SH-5	$T = -0.0011t^2 + 0.4701t + 17.535$	0.997	**SH2**	$T = 0.8673t + 18.203$	0.997

Thickened WAS (5% TS) from the Ottawa WWTP reached 65°C in approximately 130 minutes at lower voltage and in 54 minutes at higher voltage gradient. Thickened WAS (6% TS) from the Saint Hyacinthe WWTP reached 65°C in about 133 minutes at low voltage gradient and in about 56 minutes at higher voltage gradient.

WAS collected at the return (0.6% TS) from the Saint Hyacinthe WWTP required a longer time (200 minutes) to reach 65°C at a lower voltage gradient; however, it took 75 minutes at a higher voltage gradient.

As mentioned previously, the coagulants added to the WAS at the WWTP, were an important factor in determining an adequate concentration of enhancers. Coagulants might change the characteristics of sludge by increasing its conductivity, and therefore less enhancers would be required for EK treatment.

4.2 pH

The pH was measured in each sample as described in the Methodology section 3.6.1. The pH changes occurred in the electrokinetic reactor and the values were influenced by the concentration of conditioners used, voltage gradient and treatment time (Tables 4.2- 4.5).

4.2.1 Phase 1, Stage 1

The initial pH of combined sludge was 6.3. After a series of experiments with different conditions, the pH was measured again in the anode and cathode areas, as well as in a combined (reactor contents were mixed) sludge itself. The lowest pH at anode was 4.3 for OM-1 with [BS]=0.5 g/L and [AN]= 0.5g/L and the highest was 6.2 for OM-4 with [BS]=8 g/L and [AN]=8 g/L (Table 4.2). At cathode, the lowest pH measured was 8.1 for OM-4, while the highest was for OM-1. For the mixed samples, the lowest pH measured was 6.5 for OM-8 with [BS]=1 g/L and

[AN]=1 g/L and the highest was 8.0 for OM-6 with [BS]=2 g/L and [AN]=6 g/L. Overall, the pH increased at the end of the experiment and the pH change can be attributed to the electrokinetic treatment and the enhancers present in the reactor.

Table 4.2 pH measurements for Phase 1, Stage 1. Combined sludge: primary + WAS (5% TS) 1:1 ratio. + is for anode, - is for cathode, M is for mixed contents.

Phase 1 Stage 1						
Reference #	**pH**					
	+	$\sigma_{\bar{x}}$	**-**	$\sigma_{\bar{x}}$	**M**	$\sigma_{\bar{x}}$
Initial					6.30	0.58
OM-1	4.30	0.15	8.60	0.25		
OM-2	-		-		6.70	0.26
OM-3	-		-		7.60	0.15
OM-4	6.20	0.26	8.10	0.31	-	
OM-5	4.60	0.35	8.70	0.46	-	
OM-6	4.50	0.57	8.70	0.20	8.00	0.36
OM-7	-		-		6.90	0.14
OM-8	-		-		6.50	0.26

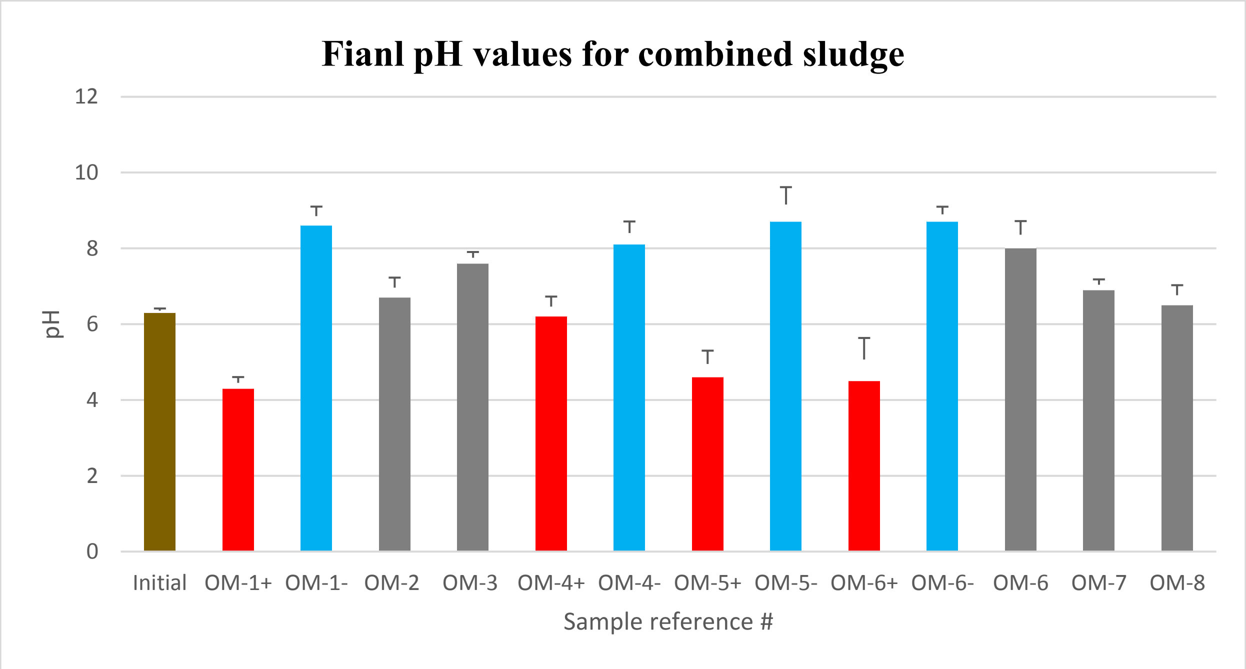


Figure 4.18 pH measurements for Phase 1, Stage 1 at the end of the experiment. Brown is the initial pH value Red represents anode (+), blue represents cathode (-), grey represents the mixed contents of the reactor. The error bars represent the standard deviation. OM-8=[1/1], OM-7=[0.5/0.5], OM-6=[2/6], OM-5=[1/6], OM-4=[8/8], OM-3= [2/2], OM-2= [1/1], OM-1=0.5 g/L BS/ 0.5g/L AN].

4.2.2 Phase 1, Stage 2

The initial pH value for the WAS (5% TS) was 6.9. This was measured when the sample reached the lab- one hour after collection. Then pH was measured for all samples after EK treatment was completed (Table 4.3).

Table 4.3. pH measurements for Phase 1, Stage 2. WAS with 5% TS. + is for anode, - is for cathode, M is for mixed contents.

Phase 1 Stage 2						
Reference #	**pH**					
	+	$\sigma_{\bar{x}}$	**-**	$\sigma_{\bar{x}}$	**M**	$\sigma_{\bar{x}}$
Initial					6.9	0.06
O-1	4.9	0.25	8.5	0.17	6.7	0.40
O-2	6.0	0.55	9.2	0.42	7.6	0.50
O-3	4.6	0.45	9.2	0.64	7.3	0.60
O-4	-		-		6.3	0.55
O-5	5.4	0.45	8.9	0.61	7.4	0.35
O-6	-		-		6.0	0.49
O-7	4.2	0.21	9.5	0.66	6.0	0.31
O-8	4.4	0.25	7.8	0.59	6.1	0.31
O-9	4.3	0.50	9.4	0.67	8.4	0.59
O-10	5.2	0.47	8.9	0.50	6.8	0.45

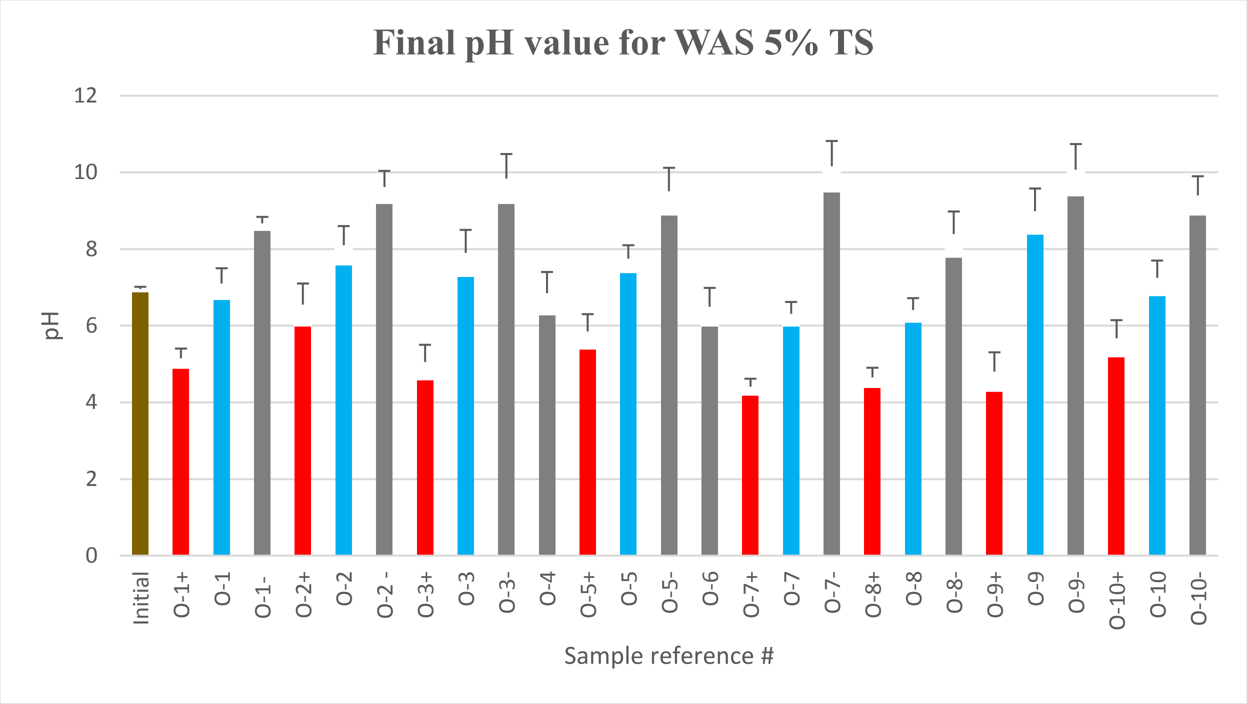


Figure 4.19. pH measurements for Phase 1, Stage 2 at the end of the experiment. **Brown is the initial pH value Red represents anode (+), blue represents cathode (-), grey represents the mixed contents of the reactor. The error bars represent the standard deviation O-10=[2/6], O-9=[0/6], O-8=[1/3], O--7=[1/6], O-6=[2/6]-2.0V/cm; O-5=[2/6], O-4=[0.5/3], O-3= [1/6], O-2= [8/8], O-1=0 g/L BS/ 0g/L AN].**

The lowest pH at anode was 4.2 and the highest was 6.0. At cathode, the lowest value measured at 7.8 and highest at 9.4. However, the mixed mass of samples had a pH ranging from 6.0 – 8.4. Figure 4.19 shows the influence of the concentration of enhancers and the voltage gradient on the pH value of sludge. As it can be observed, there is a significant difference in pH values at anode and cathode. The biggest pH differences are for the concentration combinations that contain little or no BioxyS (O-3, O-7, O-9). Therefore, a ratio of 3:1 (AN: BS) would insure a more uniform reaction.

In this experiment setting, the voltage gradient did not play a significant role in the pH distribution in EK cell. According to the results, at a voltage gradient of 2.0V/cm, the pH at anode tends to be on average 6% lower than at a voltage gradient of 2.5V/cm. At the cathode, the voltage gradient did not seem to have a significant impact (Table 4.3).

After the biosolids were removed from the BioElectro, and mixed thoroughly, the pH was measured again. The pH for samples treated with a voltage gradient of 2.0V/cm were slightly acidic on average (pHavg $_{[2.0\text{V/cm}]}$ =6.3) while those treated at 2.5V/cm were found to be slightly basic (pHavg $_{[2.5\text{V/cm}]}$ = 7.1) (Table 4.3 and Figure 4.19).

4.2.3 Phase 2, Stage 1

In return sludge (0.6% TS), the lowest pH value determined at the anode was for a sample that underwent treatment without any enhancing agents (SHR-1), while the highest pH was 9.8. The measurement of pH in the matrix of mixed samples ranged from 7.0 to 9.0. Table 4.4 presents all average pH value in Phase 2, Stage 1. Compared to the WAS from Ottawa (5% TS), the return WAS from Saint Hyacinthe had much higher pH values after EK treatment.

Table 4.4 pH measurements for Phase 2, Stage 1. + is for anode, - is for cathode, M is for mixed contents.

Phase 2, Stage 1						
Reference #	**pH**					
	+	$\sigma_{\bar{x}}$	-	$\sigma_{\bar{x}}$	M	$\sigma_{\bar{x}}$
Initial					7.0	0.06
SHR-1	5.6	0.29	9.8	0.45	7.4	0.31
SHR-2	7.9	0.19	8.8	0.34	8.4	0.45
SHR-3	8.3	0.13	9.0	0.16	8.7	0.26
SHR-4	8.2	0.51	9.8	0.47	9.0	0.36
SHR-5	7.1	0.4	8.9	0.32	8.0	0.35
SHR-6	6.0	0.41	9.2	0.3	7.6	0.17
SHR-7	6.8	0.25	8.9	0.17	7.9	0.35
SHR-8	-		-		7.5	0.25

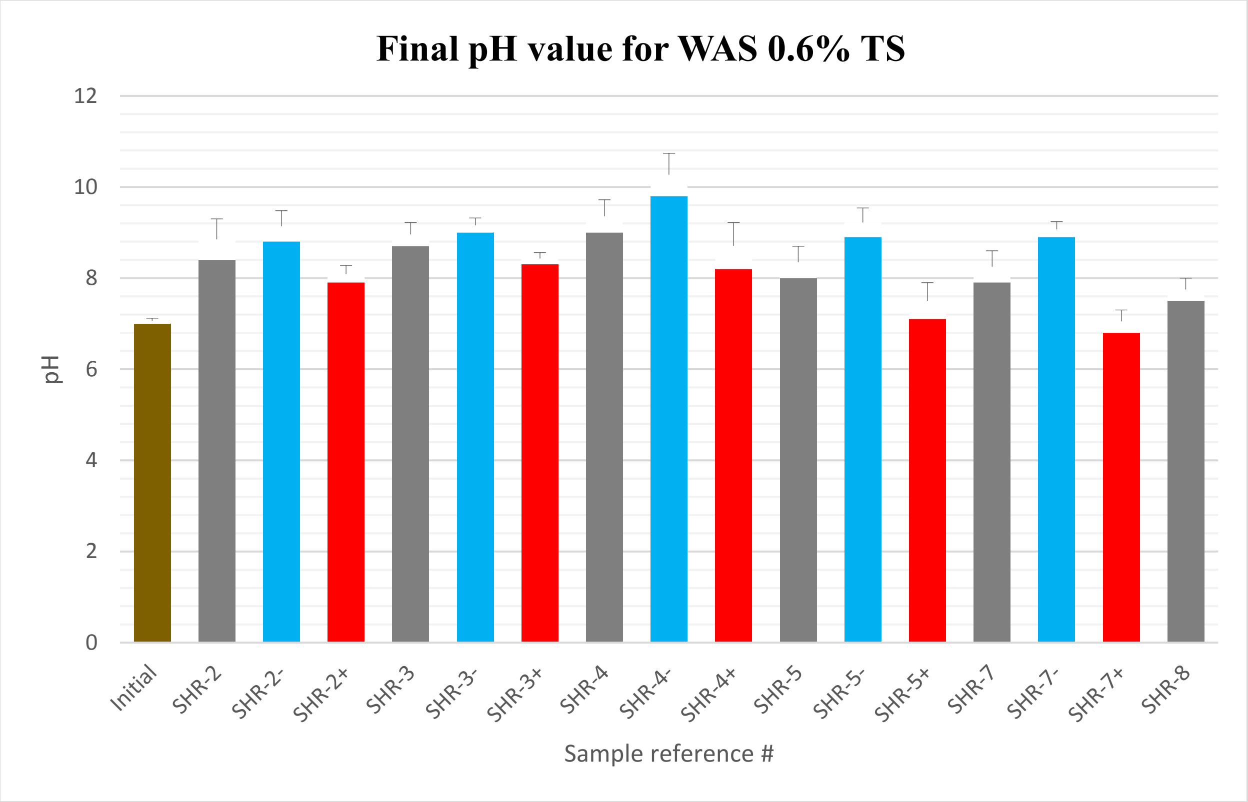


Figure 4.20. **pH measurements for Phase 2, Stage 1 at the end of the experiment. Brown is the initial pH value Red represents anode (+), blue represents cathode (-), grey represents the mixed contents of the reactor. The error bars represent the standard deviation SHR-2=[2(g/L BS)/6(g/L)AN], SHR-3=[4/8], SHR-4=[8/8], SHR-5=[3/8], SHR-7= [2/6], SHR-8= [2/1]**

Overall, the pH gradient between anode and cathode was smaller compared to thickened sludge from the Ottawa WWTP. The results show that batches with higher BS concentration at a high ratio with ammonium nitrate concentration will have a lower pH at anode, approaching acidity while having a basic pH at cathode (Figure 4.20). After mixing, the sludge had close to neutral pH value of 7.4.

4.2.4 Phase 2, Stage 2

After EK treatment of St Hyacinthe thickened sludge (6%), an entire content of each EK cells was mixed and underwent pH measurement. The lowest value was measured in sample SH-1, which underwent treatment at a voltage gradient of 2.5V/cm and [BS] =1g/L and [AN] =3g/L. The highest pH of 6.1 was measured in SH-4 sample. Table 4.4 and Figure 4.21 presents the pH measurements for Phase 2, Stage 2.

Table 4.5 pH measurements for Phase 2, Stage 2. M is for mixed contents.

Phase 2, Stage 2		
Reference #	**pH**	
	M	$\sigma_{\bar{x}}$
initial	6.9	0.05
SH-1	5.9	0.26
SH-2	5.8	0.14
SH-3	6.8	0.17
SH-4	6.1	0.35
SH-5	6.0	0.25

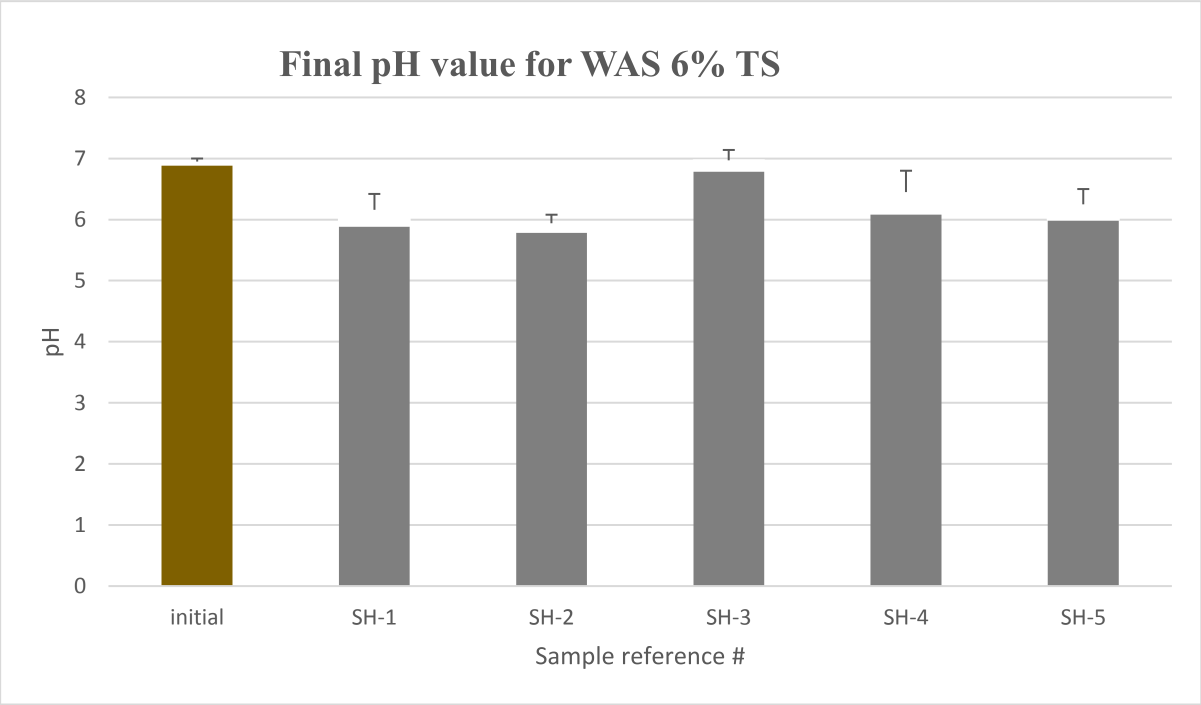


Figure 4.21 pH measurements for Phase 2, Stage 2 at the end of the experiment. Brown is the initial pH value. Grey represents the mixed contents of the reactor. The error bars represent the standard deviation. SH-1=[1/3], SH-2=[2/6]-2.5V/cm, SH-3=[0/0], SH-4=[1/3], SH-5=[2/6]-

Overall, the pH distribution was as predicted. The biosolids had a final pH ranging from 5.9 – 9.0. It was observed that the pH would be increased at a higher concentration of ammonium salts, while increasing the BioxyS concentration would decrease the pH. Therefore, as discussed previously, a ratio of 3:1 (AN:BS) is suggested in order to keep the pH value close to neutral (6.6-7.2).

4.3 ORP

Additional measurements were also done on some sludge samples with respect to oxidation-reduction potential, ORP at the end of experiments. As it was observed by Safaei (2007), the ORP was highest in the anode and lowest in the cathode area. The values shown in Table 4.6 represent the average ORP measured over a five-minute period. The lowest ORP (-14.2mV) at the cathode in the EK reactor with sample SHR-2 (return sludge from St Hyacinthe), while the highest value 12.1mV was observed in the mixed sludge O-5 (Ottawa, 5% TS). Same as in Safaei (2007), the ORP has the tendency to increase with a higher voltage gradient. As shown in Table 4.5, SHR-2 has negative values at cathode, this can be attributed to the fact that the ammonia present in the reactor has not oxidized and instead has been present in a gas form.

Table 4.6. ORP measurements for both Phase 1 and 2. (+ is anode, - is cathode and M is mixed reactor contents).

Reference number	**ORP (mV)**		
	+	-	**M**
O-3= [1/6] -2.5 V/cm	-	-	9.6
O-4= [0.5/3]- 2.5 V/cm	-	-	0.4
O-5= [2/6]- 2.0 V/cm	4.6	1.1	12.1
O-7= [1/6]- 2.0V/cm	-	-	4.1
SHR-2= [2/6]- 2.0V/cm	1.5	-14.2	9.1
SHR-7= [2/6]- 2.5V/cm	4.1	0.3	10.1
SH-1= [1/3]- 2.5V/cm	-	-	8.4
SH-2= [2/6] -2.5V/cm	9.1	2.4	3.4
SH-5= [2/6] -2.0V/cm	-	-	3.9

Among the samples measured for conductivity after EK treatment, the lowest value (1.13mS/cm) was in sample O-4 and the highest (16.5 mS/cm) was in sample O-3. No relationship was observed between conductivity at anode or at cathode. However, the measurements show that conductivity tends to increase with a higher concentration of ammonium salts.

4.5 Total suspended solids and volatile suspended solids

The TSS and VSS were determined using the method described in the methods chapter (Section 3.6.5) for all the samples. Since the experiment took place in a closed no drainage of the system was possible. Three different types of WAS were used and the effects of the EK treatment on the TSS and VSS were assessed.

The VSS/TSS ratio is an important characteristic which was used as an indicator of biodegradability of wastewater. A high ratio indicates that the sludge would be successfully digested. TSS/VSS was determined for each of the samples below. Overall, the ratios VSS/TSS found to be relatively high.

4.5.1 Phase 1, Stage 2

The initial concentration of total solids (TS) provided by the wastewater treatment plant, was measured the day the sludge was collected and it was 5.2%. The total suspended solids and volatile suspended solids were determined in triplicates (Table 4.7). Before treatment, the TSS was 5.25% and the VSS was 3.73% (+/- 0.01%). The TSS for sludge with treatment but without addition of enhancers was 3.98% at anode and the VSS was 2.27%. The average TSS and VSS for WAS that underwent electrokinetic treatment at different concentrations of enhancers is 3.08% and 1.81 % respectively.

Table 4.7. Measurement of total suspended solids and volatile suspended solids at the end of the electrokinetic treatment. + is for anode and M is for mixed contents.

Reference number	TSS (%)		VSS %		**VSS/TSS**	
	+	**M**	+	**M**		
Before treatment		5.25		3.73	0.71	
O-1= [0/0]	3.98		2.27		0.57	
O-2= [1/1]	2.34		1.56		0.67	
O-3= [2/2]	2.99	3.84	1.80	2.20	0.60	0.57
O-4= [0.5/3]		2.40		1.50	0.63	
O-5= [2/6]		4.67		2.53	0.54	
O-6= [0.5/3]		3.07		2.45	0.80	
O-7= [1/6]	2.22		1.01		0.45	

A significant decrease in VSS can be observed with an average VSS change of 51%. For the most cost efficient and viable concentration ratio (O-5) the VSS change was 32%. Although O-2, O-3, O-4 used lower concentration, it is important to keep into account that a longer treatment time is required in order to reach the target temperature for Class A biosolids.

The VSS/TSS ratio for this WAS was high, with the lowest value of 0.45 and the highest of 0.80. This shows that the sludge underwent treatment successfully and that is has a lot of potential for anaerobic treatment.

4.5.2 Phase 2, Stage 1

The initial concentration of total solids provided by the water plant, measured the day the sludge was collected was 0.6%. The total suspended solids and volatile suspended solids were determined in triplicates (Table 4.8). Before treatment, the TSS was 0.61% and the VSS was 0.37% (+/- 0.01%). The TSS for sludge with treatment but without addition of enhancers was 0.58% at anode and the VSS was 0.21%. The average TSS for WAS that underwent electrokinetic treatment at different concentrations of enhancers is 0.44% and for VSS is 0.21%.

Table 4.8 Measurement of total suspended solids and volatile suspended solids at the end of the electrokinetic treatment. + is for anode and M is for mixed contents.

Reference number	TSS (%)			VSS %			VSS/TSS		
	+	-	M	+	-	M	+	-	M
Initial			0.61			0.37	0.61		
SHR-1= [0/0]	0.58			0.21			0.36		
SHR-2= [2/6]	0.36	0.54		0.19	0.33		0.53	0.61	
SHR-3= [4/8]		0.28			0.14		0.5		
SHR-4= [8/8]	0.53			0.11			0.21		
SHR-5= [3/8]	0.30	0.26		0.20	0.26		0.67	1	
SHR-6= [3/6]		0.54			0.54		1		
SHR-7= [2/6]	0.64	0.47		0.18	0.13		0.28	0.28	
SHR-8= [2/1]			0.11			0	0		

From the results obtained, a significant change in VSS can be observed. The average VSS change was about 43%. The concentrations of most interest, represented by SHR-2 and SHR-7 showed a VSS decrease of 29.7% and 56.7% respectively.

The ratio for WAS collected from the return, shows an overall lower biodegradability potential, with the lowest ratio being at 0.28 (excluding the 0) and the highest at 1. The average ratio was however at 0.55.

4.5.3 Phase 2, Stage 2

The initial concentration of total solids provided by the water plant, measured the day the sludge was collected was 6.1%. The total suspended solids and volatile suspended solids were determined in triplicates (Table 4.9). Before treatment, the TSS was 4.57% and the VSS was 3.78% (+/- 0.01%). The average TSS for WAS that underwent electrokinetic treatment at different concentrations of enhancers was 4.15% and for VSS was 2.72%.

Table 4.9 Measurement of total suspended solids and volatile suspended solids at the end of the electrokinetic treatment. + is for anode and M is for mixed contents.

Reference number	**TSS %**	**VSS %**	**VSS/TSS**
	mixed	**mixed**	
Before Treatment	4.57	3.78	0.83
SH-1= [1/3]	4.27	3.20	0.75
SH-2= [2/6]	5.82	3.45	0.59
SH-4= [0/0]	3.62	2.28	0.63
SH-5= [2/6]	2.90	1.95	0.67

Results showed that the change in VSS was about 31% in Stage 2. The concentrations of most interest, represented by SH-2 and SH-5 showed a VSS decrease of 17 % and 48.4 %, respectively. A smaller change in VSS for SH-2 could be associated with the treatment time. For sample SH-2 the treatment was shorter than in most other cases. The sample SH-5 had a longer treatment since it was operated at a lower voltage.

All ratios of VSS/TSS for thickened WAS were high, with the highest value being at 0.75 and the lowest at 0.54. The average ratio was 0.66. SH-2 having the best concentrations and voltage gradient and a ratio of 0.59 showed great potential for degradability.

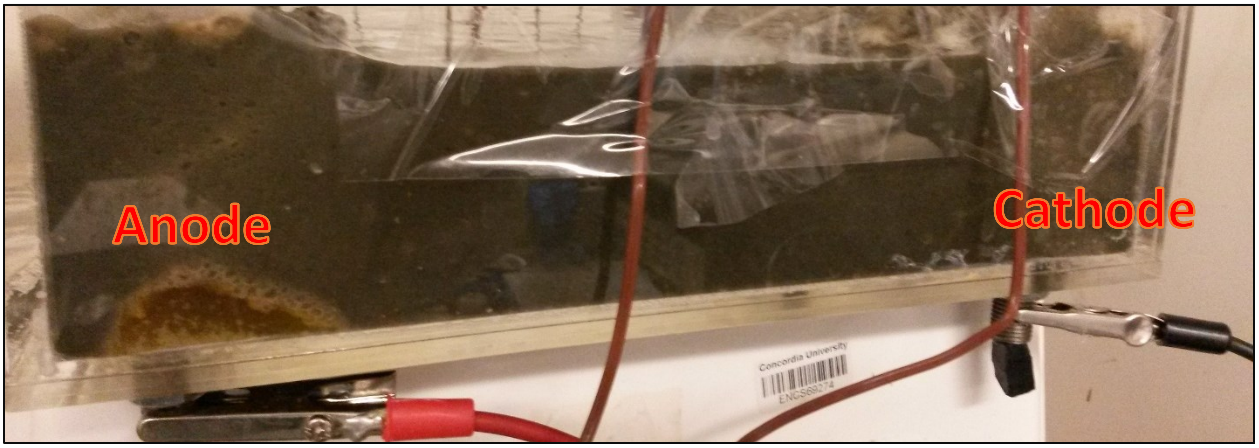


Figure 4.22. BioElectro reactor set-up. Time= 20 minutes in the experiment

Figure 4.23 BioElectro reactor set-up. Time= 20 minutes in the experiment. Closeup at anode.

Figure 4.24. BioElectro reactor set-up. At the end of the experiment.

In conclusion, most EK treated samples showed a decrease in VSS, and the variation in VSS change could be attributed to several factors. The sludge had different provenance and characteristics which may vary day by day. Although industrial wastewater represents a fraction of the sludge, this might still affect its properties. The treatment time plays an important factor as well; which is related to the voltage gradient and concentration of enhancing agent. BS plays an important role in the change of VSS due to its oxidant action. The coagulants added to thicken the sludge would also influence VSS change during electrokinetic treatment because of its important role in the conductivity of the system. The formation of bigger flocs contributes to the change of VSS from effluent (supernatant). The percentage of TSS was in average higher around the cathode area. Due to the specific particle charges and electrokinetic reactions, the TSS were observed to aggregate around the cathode.

4.6 Particle size distribution

Particle size and distribution is an important factor that can influence the dewaterability of sludge. According to Turovskiy and Mathai (2006), the greater the number of small particles, the greater the surface area/volume ratio which results in greater hydration and increased resistance to dewatering. Due to the extra layer of water, the aggregation of solids will be decreased and, in addition, sludge solids are negatively charged and thus tend to be mutually repulsive (Turovskiy and Mathai, 2006).

Zeta potential, a parameter used in characterizing the electrochemical equilibrium on interfaces is important for aggregation of particles and their stability. The electric potential at the slipping plane is called zeta potential. A high positive or negative zeta potential means that the particles are stable and hard to coagulate. A low positive or negative zeta potential means that the particles are unstable and easy to coagulate. Therefore, this can affect the dewaterability of sludge. Ideally, the zeta potential should be around zero.

Colloidal particles are expected to have a positive or a negative zeta potential, above 30 mV or below -30 mV. This will make dewatering more difficult, and high quantities of chemicals/conditioners are required. As a result, the particle size needs to be measured in order to determine if sludge treatment was successful.

The change in particle size was determined using the Horiba Particle size analyzer. In order to determine the distribution of particle size, the phi-scale and the ASTM mesh was used. As mentioned in the methodology, three different types of sludge underwent such measurements. The same refractive index (1.52) was used for all three types of sludge. The PSA results will help analyze and compare the changes in particles size before and after treatment in order to determine the effects of different concentrations, treatment times and voltage potential.

4.6.1 Phase 1, Stage 2

As Table 4.10 and Figure 4.25 show, before application of EK treatment the sludge particles had a mean size of 43 µm with a standard deviation of 38 µm and a geometric mean size of 31 µm with a Skewness of 2.1.

Table 4.10 Particle size distribution Phase 1, Stage 2.

Diameter Size (μm)	**Frequency %**				
	Initial WAS Ottawa	**O-2**	**O-3**	**O-4**	**O-5**
20	0.9	1.3	1.3	1.3	1.3
25	5.3	5.2	6.2	5.1	3.9
32	10.1	8.7	9.6	9.2	7.5
38	10.8	9.3	9.7	10.3	9.5
45	7.9	7.6	7.6	8.0	7.8
53	6.7	6.9	7.0	6.7	6.1
63	5.7	5.9	6.2	5.4	4.3
75	5.0	4.7	5.1	4.1	2.9
90	3.7	3.0	3.3	3.1	2.2
106	3.0	2.5	2.8	3.2	2.5
125	1.6	1.3	1.9	2.3	1.4
150	1.7	1.2	1.2	1.5	1.4
180	1.1	0.9	0.8	0.2	1.1
212	0.2	0.2	0.5	0.0	0.3
250	0.0	0.0	0.1	0.0	0.0

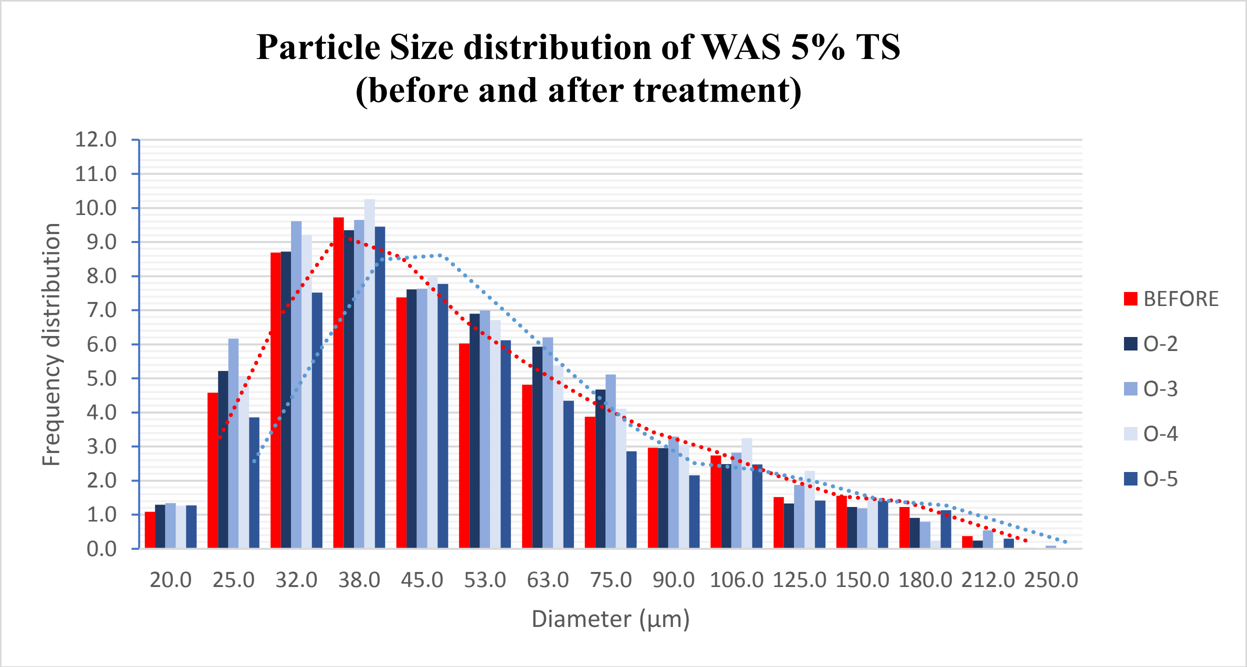


Figure 4.25. Particle size distribution: before and after treatment. Phase 1, Stage 2 at high voltage O-5=[2/6], O-4=[0.5/3], O-3= [1/6], O-2= [8/8]

As shown in Figure 4.25, the distribution of particle size will change after treatment. The distribution becomes less skewed as the trendlines indicate. Samples having the highest concentration of enhancers showed the most change in particle size distribution at the end of the experiment. A higher concentration of AN with a higher voltage gradient will show the most impact on the sample at the end of the treatment, for example, sample O-5 achieved the highest frequency of larger diameter size- flocculation. Electrocoagulation process was successful in all applied conditions

4.6.2 Phase 2, Stage 1

Table 4.11shows the different samples chosen in this experiment and the distribution of particle size. As Table 4.11 and Figure 4.26 show, before treatment was applied to the sludge, the particles had a mean size of 67 µm with a standard deviation of 45 µm and a geometric mean size of 55 µm with a Skewness of 1.9

Table 4.11. Particle size distribution Phase 2, Stage 1

Diameter (µm)	**Frequency (%)**					
	BEFORE	**SHR-2**	**SHR-4**	**SHR-5**	**SHR-6**	**SHR-7**
20	4.5	2.6	1.1	2.2	1.4	3.4
25	5.2	7.4	6.8	7.6	7.4	7.2
32	5.2	8.8	10.5	9.9	9.4	8.3
38	7.5	9.3	10.7	10.0	8.3	9.7
45	11.1	9.6	9.7	9.3	7.4	10.5
53	14.6	10.7	10.1	10.0	8.4	11.4
63	15.1	10.4	9.7	9.8	9.0	10.5
75	11.1	7.9	7.6	7.8	7.9	7.7
90	4.6	4.4	3.9	4.1	4.6	4.2
106	3.0	3.9	3.0	3.2	3.8	3.8
125	1.9	2.8	2.1	2.4	2.7	2.7
150	2.4	3.7	2.7	3.4	3.8	3.2
180	2.7	3.9	3.1	3.5	5.2	3.5
212	1.5	2.0	1.6	1.4	4.0	2.2
250	0.6	0.4	0.5	0.1	1.5	0.7
300	0.0	0.0	0.0	0.0	0.4	0.0

WAS from the return has 0.6% of total solids compared to thickened WAS ranging from 5-6%.

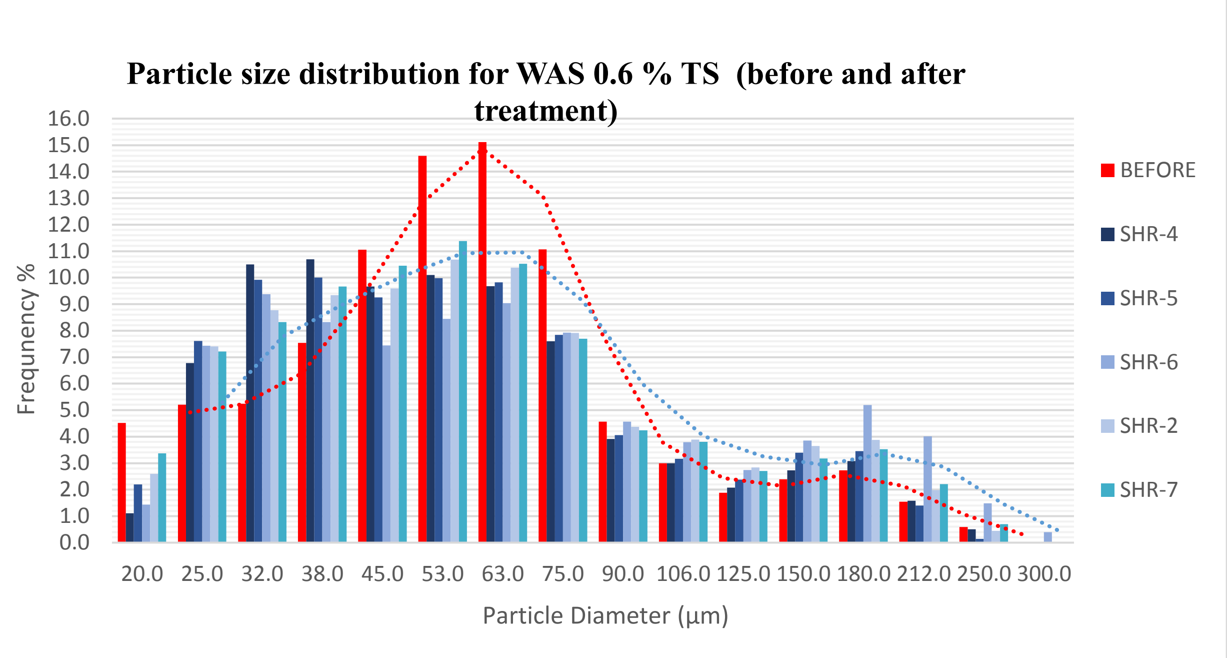


Figure 4.26. Particle size distribution: before and after treatment. Phase 2, Stage 1. SHR-2=[2(g/L BS)/6(g/L)AN], SHR-6=[3/6], SHR-4=[8/8], SHR-5=[3/8], SHR-7= [2/6]

After treatment, the size of particles became more evenly distributed (Figure 4.26). SHR-2 and SHR-6 underwent the longest treatment time ranging from two to three hours. It is worth reminding

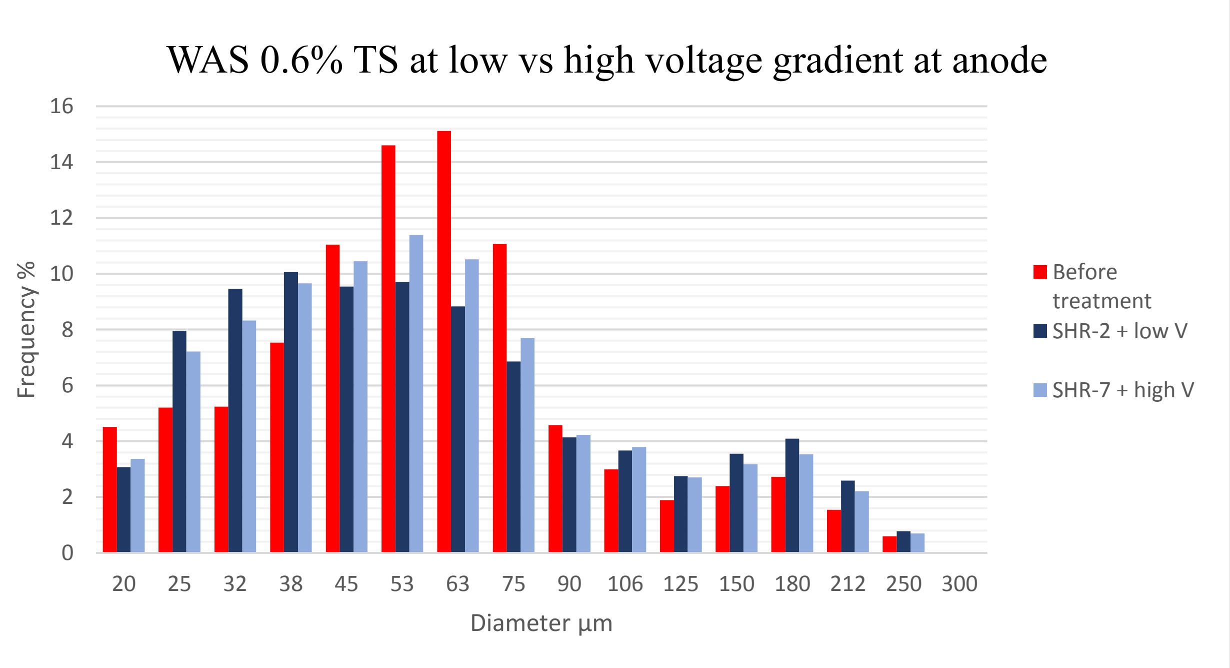


Figure 4.27. Particle size distribution at anode: comparison of low and high voltage. Phase 2, Stage 1 SHR-2= [2/6]- 2.0V/cm, SHR-7= [2/6]-2.5 V/cm.

that these runs took place at a lower voltage potential along with lower concentration of conditioners and thus required a longer time to reach the target temperature.

Figure 4.27 compares the differences in distribution for low and high voltage at anode. Both samples had the same initial concentration of conditioners, but the reaction time for SHR-7 was 2.2 times shorter than SHR-2. From Figure 4.26 it can be observed that a higher voltage gradient had more impact the particle size distribution, which becomes less skewed at the end of treatment. This could be the result of electroosmotic phenomena. In a EK treatment study on sludge in reactor, it was found that the mean particle size change over time depended on electrical field Ibeid et al. (2015). According to Ibeid et al. (2015), the water associated with bio- flocs was removed by electroosmosis, which, aided with the the reduction of the repulsive forces between the flocs, more specifically due to the decrease of zeta potential. This phenomenon will therefore insure the successful flocculation process in a EK reactor.

4.6.3 Phase 2, Stage 2

Table 4.11 shows the different samples chosen in this experiment and the distribution of particle size. As Table 4.12 and Figure 4.28 show, before treatment was applied to the sludge, the particles had a mean size of 42 µm with a standard deviation of 27 µm and a geometric mean size of 35 µm with a Skewness of 1.8.

Table 4.12 Particle size distribution Phase 2, Stage 2.

Diameter (µm)	Frequency (%)		
	Before	SH-2	SH-5
20	10.3	4.6	6.6
25	10.5	7.9	8.5
32	9.3	9.1	8.9
38	11.0	10.4	10.4
45	10.9	9.9	10.3
53	9.6	9.1	9.2
63	6.6	7.1	6.6
75	3.6	4.5	3.6
90	1.7	2.2	1.4
106	1.6	1.7	1.2
125	1.4	1.1	0.8
150	0.9	1.5	1.4
180	0.1	1.8	1.6
212	0.0	1.1	0.8
250	0.0	0.2	0.3

As seen with the previous sludge types, the distribution becomes less skewed, although not significantly. Figure 4.28 shows the difference in the distribution for a longer treatment time, compared to a shorter treatment time at the same voltage and the same concentration. In this case, both runs were done at a lower voltage, and again, the longer treatment time will increase the size of the particles, thus making their distribution slightly more uniform.

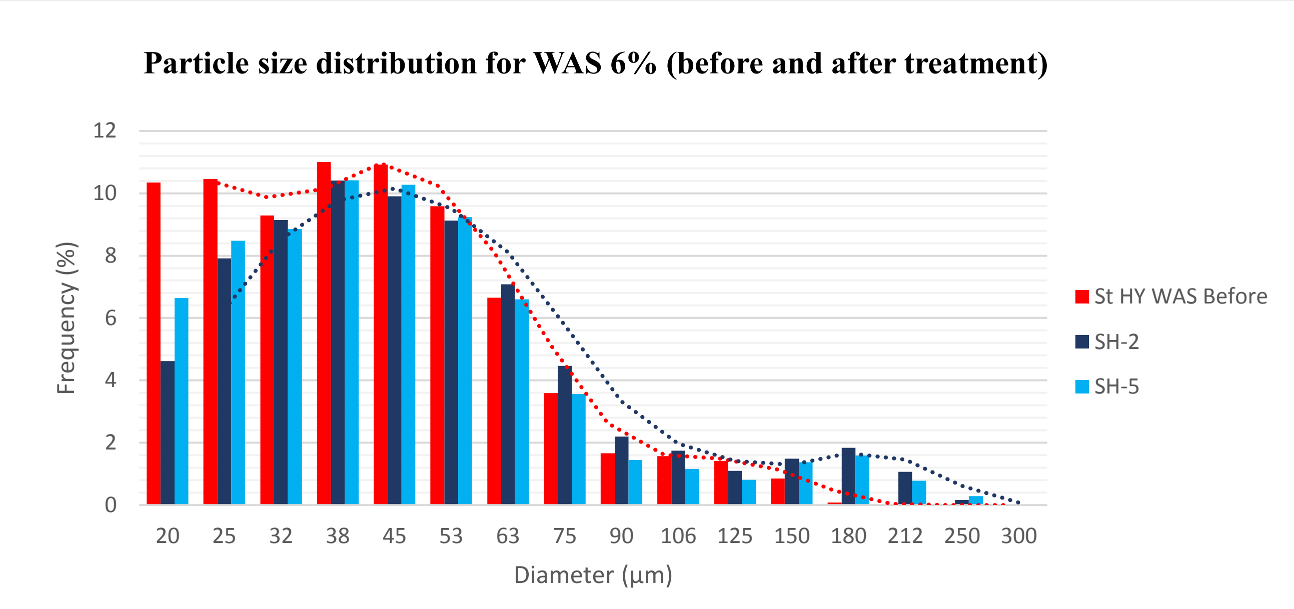


Figure 4.28. Particle size distribution: before and after treatment. Phase 2, Stage 2 SH-2=[2/6]-2.5V/cm, SH-5=[2/6]-2.0V/cm.

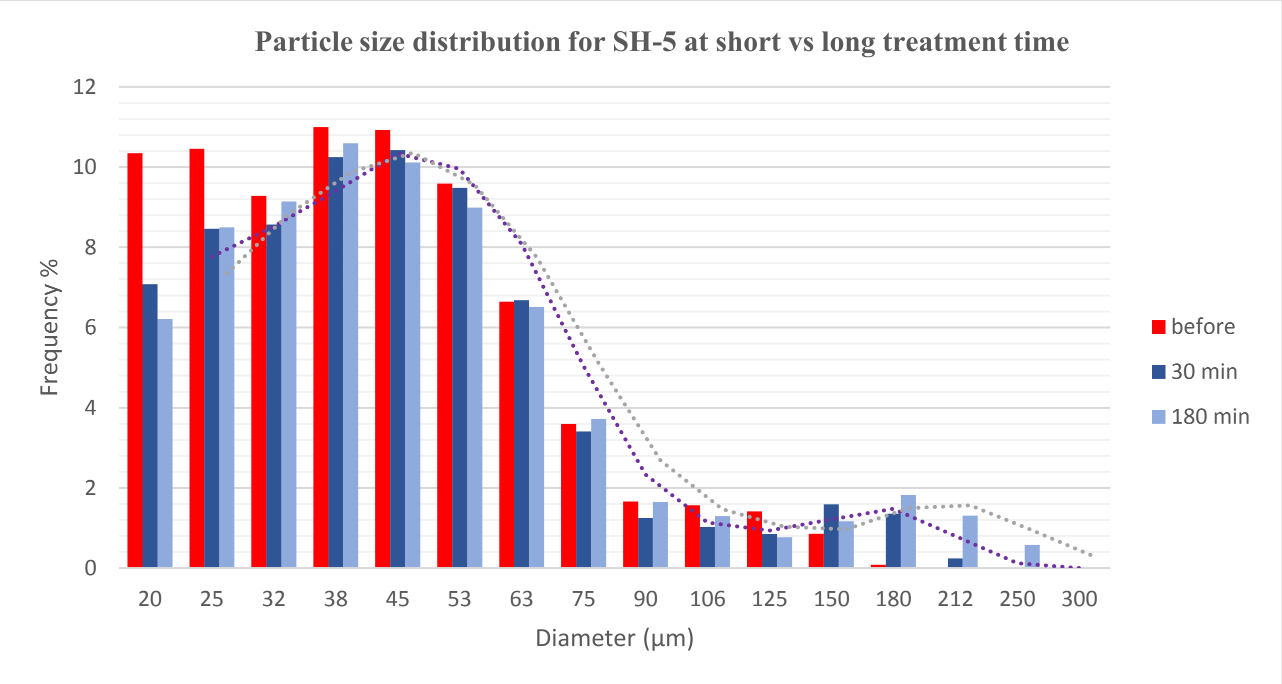


Figure 4.29. Particle size distribution: short vs long treatment time. Phase 2, Stage 2. SH-5=[2/6]-2.0V/cm.

Figure 4.29 compares the change in particle size for sludge undergoing different treatment time in order to determine the effects of electrokinetic treatment on particle size distribution. As shown by the graph, there is a tendency for a longer treatment time to increase the particle diameter by aggregation. For example, with a longer treatment time, the frequency of particles with a size larger than 180 μm increased by 25% in the case of SH-5 (WAS 6% TS).

In conclusion, electrokinetic treatment using the BioElectro will have an impact on particle size distribution. Overall, the particles show an increase in diameter size. The particles will thus become more easily removable during the dewatering process.

4.7 Nitrates and Ammonia (NO_3 and NH_3)

Nitrate and ammonia analyses permitted to assess their distribution in EK cells after the BioElectro treatment with respect to each particular sludge type. Results showed the highest concentrations of NH_3 were detected in the cathode area. The highest concentration of 700mg/l, was measured in sample SHR-2, and the same sample had the lowest concentration of NO_3. The lowest concentration of NH_3 had the highest concentration of NO_3. For example, in the case of WAS 6% TS treated with BS=2g/L and AN=6g/L at 2.5V/cm, from St Hyacinthe (SH-2), NH_3 and NO_3 was 0.4mg/L and 376mg/L (Table 4.13). All analysed samples showed similar behavior related to the oxidation of ammonia to NO_3. Therefore, the highest concentration of NO_3 was observed at the anode. Nitrate is a form of available nitrogen that can be easily absorbed by plants in aerobic soils. In case of anaerobic conditions (e.g. Soils), ammonia would predominate (Miller et al. 2012). Usually, sludge produced by other treatments does not have sufficient amount of ammonia (with respect to phosphorous) to be recognized as a valuable fertilizer. Therefore, BioElectro approach not only disinfects the sludge but also improves its fertile properties.

Table 4.13. Nitrate and ammonia measurements from samples of different concentration combinations and voltage gradient. + is for anode and M is for mixed contents.

Reference number	NO3 (mg/L)			NH3 (mg/L)		
	+	-	M	+	-	M
O-4= [1/6]			224			5.1
O-5= [2/6]			240			19.1
SHR-2= [2/6]- 2.0V/cm	147	7.5		189	708	
SHR-7= [2/6]- 2.5V/cm	244	6.3		7.2	270	
SH-1= [1/2]- 2.5V/cm			189			0.3
SH-2= [2/6]- 2.5V/cm			376			0.4

4.8 Bacterial analysis: fecal coliform

As described in the methodology (Chapter (3.6.8), the fecal coliform were determined by the membrane filtration technique using MI Agar, a chromogenic/fluorogenic medium.

In spite of satisfactory fulfilment of USEPA (2006) regulation to generate Class A biosolids, some validation tests were conducted. Then, to validate the efficiency of the EK treatment with respect to pathogens, random collected samples were submitted to bacteriological analysis of fecal coliform. Selected samples treated with 2.5V/cm and conditioners (AN and BS) underwent the membrane filtration procedure. The samples were diluted 100, 200, 400 or 500 times depending on the concentration of TS. After incubation at 35°C, the *E. coli* was counted using the naked eye, while the other CFU was counted under the UV light.

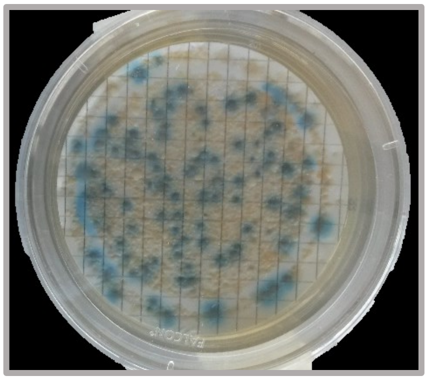


Figure 4.30. FC test before treatment. WAS 5% TS. 1/400 dilution.

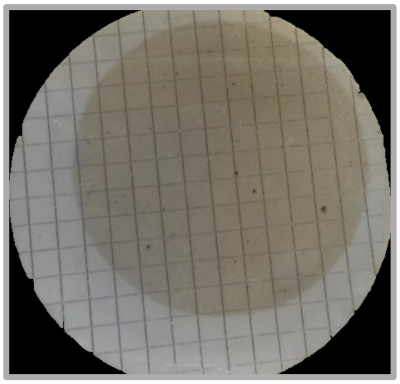


Figure 4.31. FC test for O-5. 1/200 dilution.

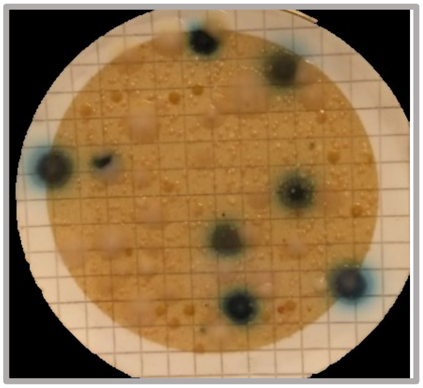


Figure 4.32. FC test for O-5. 1/100 dilution.

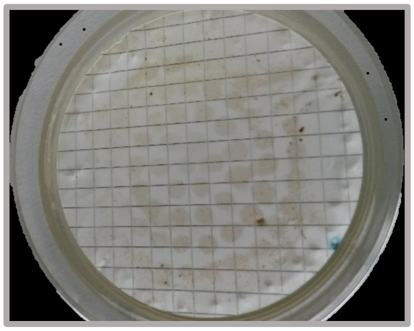


Figure 4.33. FC test for O-4. 1/200 dilution.

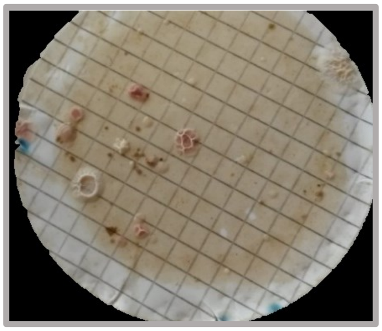


Figure 4.34. FC test for SH-2. 1/500 dilution.

Table 4.14. CFU count in Stage 3.

Sample	**CFU/100ml**		**CFU/ dry g**
	E. coli	**Other**	
Ottawa WAS before (1/200 dilution)	Too many to count		
St Hyacinthe WAS before (1/200 dilution)	Too many to count		
O-4 (1/200 dilution)	ND	17	**340**
O-5 (1/100 dilution)	8	64	**1353**
O-5 (1/200 dilution)	ND	9	**263**
SHR-7 (1/200 dilution)	3	13	**354**
SH-2 (1/200 dilution)	ND	21	**978**
SH-5 (1/200)	13	2	**699**
SHR-2 (1/200)	ND	6	**133**

Table 4.14 shows that most samples are within the limits required by USEPA (2012) in order to categorized them as Class A biosolids. The photos (Figures 4.30-4.34) show a substantial change in CFU at the beginning and at the end of the experiment. Samples that did not undergo EK treatment, showed too many CFU colonies that were indistinguishable from one another. As shown in Table 4.14, the samples containing a BS concentration of 2g/L showed to be the most successful at reducing the CFU count. In order to accomplish similar results with a lower concentration of BS, a longer treatment time might be required, depending on the initial sludge characteristics.

CHAPTER 5: CONCLUSION, CONTRIBUTION, FUTURE WORK

5.1 Final conclusion

This study achieved to apply successfully the BioElectro system for treatment of combined and waste activated sludge (WAS) of low (0.6%) and higher (5%, 6%) total solids contents (TS). The process showed possibility to transform sludge into Class A biosolids with respect to pathogens content. It was achieved through the application of multiple stressors, where the main one was rising temperature using a combination of electrokinetics enhanced by ammonium salt (AN) and BioxyS (BS) producing PAA.

The results showed that the initial characteristics of sludge were an important factor in determining the appropriate treatment time, the concentration of enhancers and the voltage gradient. Sludge underwent treatment at different concentrations of enhancing agents at two voltage gradients: 2.0V/cm and 2.5V/cm. After assessing the rate of temperature increase at different concentrations, it was concluded that a sustainable combination of AN:BS is 3:1, i.e. AN=6g/L; BS=2g/L, while sludge achieved 65°C within less than an hour - nevertheless, much lower concentrations can be also successful when the retention time can be expanded.

The treatment Alternative (1) under Part 503 requires at least 30 minutes of treatment at 65°C, in order to achieve Class A biosolids (USEPA, 2012). The study also permitted to assess the differences in response to four different sludge types to the BioElectro process. The results showed that under the same technological conditions, the temperature of 65°C was achieved in the following order: [combined sludge] < [WAS 5%TS (Ottawa)] & [WAS 6% TS (St. Hyachinthe)] < [return sludge 0.6% TS (St Hyacinthe)]; i.e. [54 min] < [56 min] < [75 min], respectively.

Sludge which underwent treatment at a lower voltage gradient 2.0V/cm required a longer exposure time to reach 65°C. Therefore, WAS (5% TS) of Ottawa WWTP achieved a temperature of 65°C around 136% slower at a lower voltage gradient. The WAS (6% TS) from St-Hyacinthe achieved it 140% slower, while the return WAS (0.6% TS) from St-Hyacinthe reached 65°C 166% slower at lower voltage (Chapter 4.1).

Changes in sludge properties were observed in all samples by the end of the experiments. When temperature increased at an efficient rate, a gradient between electrodes in pH and ORP was observed. Simultaneously, changes in VSS content, as well as particle aggregation (determinant factor of sludge dewaterability) were detected. Furthermore, the fecal coliform reduction was also confirmed.

The pH value decreased at anode due to the formation of H^+ ions and increased at cathode due to formation of OH^- ions. The ORP values were higher at anode and lower at cathode, while value deepened on voltage gradient.

Depending on the sludge type, the change in VSS averaged from 31% to 51%. The VSS/TSS ratio also showed a potential of the application of the final BioElectro product for anaerobic digestion. For the thickened sludge, the average VSS/TSS ratio was 0.64 (Chapter 4.4).

The measurements of particle size at the end of the experiment showed that electro-coagulation phenomena took place in all the reactors. The aggregation of particles depended on the voltage gradient, the temperature and the exposure time (Chapter 4.5). All tested sludge types showed a potential for better dewatering than conventional WAS. It would be achieved by either a higher voltage gradient or a longer exposure time in the EK cell.

Fecal coliform colonies were also counted for a number of random samples in order to validate the quality of the biosolids. Disinfection was achieved in the majority of the samples; the count was within the limits specified by Class A requirements (Chapter 4.6). The combination of stressors acting on the system had a significant contribution in the inactivation of pathogens, it might therefore be permitting a shorter treatment process.

Higher voltage gradient enhanced chemical reactions, taking place in the presence of conditioners such as AN and BS. As such, the enhancers dissociated at a faster rate, which in turn produced increased oxidation at anode, a higher pH gradient between the electrodes and increased redox potential. A higher voltage gradient also showed increase of ohmic heat.

The EK reactor has the advantage of controlling the stressors (heat, exposure time and conditioners), which in turn generated stressor inactivating pathogens. Moreover, the ammonia present in the final biosolids product improved the fertile properties of biosolids as an agriculture soil amendment.

Overall, the BioElectro reactor produced disinfected, fertile biosolids with improved dewaterability that can be used in different beneficial applications.

5.2 Contributions

The study made several contributions to the advancement of electrokinetic sludge treatment.

First of all, this bench scale experiment was the first assessment of the waste activated sludge (WAS) responding to BioElectro process.

Furthermore, WAS underwent EK treatment with BioxyS and ammonium salts as conditioners using a low voltage gradient (2.0 V/cm and 2.5 V/cm). As such, thermal treatment was achieved as exothermic reactions permitted a high rise of temperature within the sludge matrix.

The study proved a direct conversion of Class B to Class A biosolids without digestion but using electrokinetics to create multi-stressor conditions in WAS. Technological conditions, which have been suggested in this study is a ratio of BS:AN=1:3 since it generates a more homogeneous rate of temperature increase in the BioElectro reactor.

5.3 Future works

In order to better understand the processes generated in the EK system, more research at a larger scale is required. Considering thermodynamic phenomena, it is speculated that the system would be more efficient at a larger scale. Particular interest would be the development of a continuously working system.

Feasibility of application of the obtained biosolids into various non-fertile types of soil as a fertilizer.

Future experiments also require an in-depth assessment of the microbial survival and stabilization re-survival in already treated sludge.

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Appendix 1

The TSS were determined using the EPA method 160.2.

Steps:

1. A glass fiber filter disc was inserted in a Gooch crucible with the wrinkle side up. Then washed with three successive 20-mL portions of distilled water. Using vacuum and placed in an oven 105°C for 1 h to dry.
2. After, the filter was ignited at 550°C for 15 min in a muffle furnace, then cooled in a desiccator, and weighted. The cycle of igniting, cooling, desiccating, and weighting was repeated until a constant weight was obtained or until weight changes was less than 0.5mg of the previous weighting.
3. Samples chosen to be filtered had a volume of 40 mL in order to obtain between 5.5 and 200 mg of residue.
4. The sample is well shaken and then filtered through the filtering assembly using suction.
5. After all the sample passed through, the graduated cylinder, filter, non-filterable residue and filter funnel wall are washed with three portions of distilled water. All traces of water are removed by continuing to apply vacuum after water has passed through.
6. Then, the filter is removed and dried at 103-104 C for 24 h.
7. The filter is then cooled in a desiccator for 30 minutes and weighed.

The TSS were determined using the following equation:

$$\%\ \text{total solids} = \frac{(A-B)\times 100}{(C-B)}$$

A= mass of filter and crucible combination+ dried residue, (mg)

B= mass of filter and crucible combination (mg)

C= mass of wet sample+ filter and crucible combination (mg).

Appendix 2

The VSS were determined by using the same sample from the TSS.

Steps:

1. After the TSS was determined for the desired sample, the filter was ignited at 550 for 1 hour
2. Then, the filter was cooled in the desiccator for 30 min. The cycle of igniting, cooling, desiccating, and weighting was repeated until weight change was less than 0.5mg of the previous weight.

The TSS were determined using the following equation:

$$\% \, VSS = \frac{A-B}{D} \, x \, 100$$

Where:

A= (mass of residue+ filter, and crucible combination) before ignition, [mg]

B= (weight of residue+ filter, and crucible combination) after ignition, [mg].

Appendix 3

The probe used for measuring the NO_3-N concentration is the ISENO 3181. It was calibrated according to the steps shown in the user manual:

1. The probe is first connected to the meter, secured and then the meter is turned on.
2. 3 different Nitrate Nitrogen standards were prepared and then Nitrate ionic strength powder was added. The probe was rinsed with deionized water.
3. Calibrate button pressed
4. A stir bar is added to the standard solution and put the probe immersed. Then added to the electromagnetic stirrer.
5. The Read function was then used to display the standard value.
6. The steps are then repeated for the other standards.

The measurements were then done by the direct method, as shown in the user manual:

1. The probe is first connected to the meter, secured and then the meter is turned on.
2. 40 ml of each sample was prepared for measurement by adding the Nitrate ionic strength powder. The probe was rinsed with deionized water.
3. A stir bar is added to sample and put the probe immersed. Then added to the electromagnetic stirrer.
4. The Read function was then used to display the value once stabilized.
5. The steps are then repeated for all the samples measured

Appendix 4

The probe used for measuring the NH_3-N concentration is the ISENH 3181. It was calibrated according to the steps shown in the user manual:

1. The probe is first connected to the meter, secured and then the meter is turned on.
2. 3 different Ammonia standards were prepared and then Ammonia ionic strength powder was added. The probe was rinsed with deionized water.
3. Calibrate button pressed
4. A stir bar is added to the standard solution and put the probe immersed. Then added to the electromagnetic stirrer.
5. The Read function was then used to display the standard value.
6. The steps are then repeated for the other standards.

The measurements were then done by the direct method, as shown in the user manual:

1. The probe is first connected to the meter, secured and then the meter is turned on.
2. 40 ml of each sample was prepared for measurement by adding the Ammonia ionic strength powder. The probe was rinsed with deionized water.
3. A stir bar is added to sample and put the probe immersed. Then added to the electromagnetic stirrer.
4. The Read function was then used to display the value once stabilized.
5. The steps are then repeated for all the samples measured