Electrokinetic Remediation of Organic Soil Polluted with Petroleum Products in Temperate and Cold Regions

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Abstract For MASc

Electrokinetic Remediation of Organic Soil Polluted with Petroleum Products in Temperate and Cold Regions

Shayan Ghanami

Global warming leads to the thawing of ice caps in cold regions like Canada's northern territories, which are predominantly covered with permafrost. Its active top layer contains different fractions of organic matter and clay. More frequent use of shipping routes increases the risk of northern soil pollution. One group of common pollutants, originating from pipeline spills or various leakage, are light hydrocarbons. Soil pollution affects the sensitive northern environment. Effective soil remediation necessitates a comprehensive understanding of soil properties to implement an appropriate remediation method tailored to the specific soil characteristics and the challenging climatic conditions in northern territories.

A series of laboratory tests were conducted to evaluate the feasibility of electrokinetic (EK) soil remediation on organic soils in cold-tempered regions. The study involved four distinct soil compositions to determine the efficacy of EK transport within organic matter (3%, 16%, and 35% w/w) containing soil. The soil was polluted with toluene and exposed to a constant low DC voltage gradient. To optimize electrokinetic remediation, a daily injection of surfactant was conducted. The tests were carried out continuously over a period of four days without anolyte, and one day including electroosmotic discharge collecting both anolyte and catholyte. The study was conducted in ambient (20°C) and cold temperatures (7°C) to simulate temperate and cold environments.

The results showed the feasibility of light hydrocarbons removal from organic soils, while EK transport was more effective under low temperatures. Soil changed properties and pH gradient was observed between the anode and cathode. Furthermore, extracted liquids indicated some dissolutions of humic substances present in organic soil.

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Dedication

To my parents,

For their endless support and love, for making me who I am today.

In memory of Nika Shakarami

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List of Abbreviations

BTEX	Benzene, toluene, ethylbenzene, xylene
DI	Deionized water
EK	Electrokinetics
EM	Electromigration
EO	Electroosmosis
EP	Electrophoresis
GC	Gas chromatography
HPLC	High-performance liquid chromatography
MEKC	Micellar electrokinetic chromatography
SOC	Soil organic carbon
SPE	Solid-phase extraction
SVE	Soil vapor extraction
UNESCO	The United Nations Educational, Scientific and Cultural Organization

1 Introduction

1.1 Problem statement

The inevitable result of climate change, global warming, is accelerating the annual ice cap thawing at an alarming rate. This development is especially worrying for Canada's northern areas, where permafrost predominates. This permafrost's thawing raises serious concerns because it may cause pollutants to accumulate and spread across the active layer.

Among the diverse range of pollutants, BTEX (benzene, toluene, ethylbenzene, and xylene) is one of the most common ones, which can be found in water, air, and soil. Sources of BTEX contamination in natural water systems primarily include sewage discharge, oil leaks, and petroleum products transportation (Yu et al., 2022).

One notable characteristic of the northern Canadian regions is the significant presence of organic matter in the soil, often in the form of peat. Organic soils exhibit distinct behaviors in contact with BTEX, often retaining the pollutants longer than other soil components (Zhenghu & Honglang, 2000).

Various studies have explored different methods for soil remediating, including phytoremediation, soil washing, bioremediation, and soil bioventing (Bolan et al., 2011; Gouma et al., 2014; Kostecki et al., 2005; Rathfelder et al., 1995). A particular problem with remediation presents a clay fraction in polluted soil. This study aims to build upon previous efforts in soil remediation using electrokinetics (Hakimipour & Elektorowicz, 2001; Hatem & Elektorowicz, 1999), which have primarily focused on clayey soils and ambient temperature conditions. In contrast, this study specifically addresses the challenges posed by the harsh climatic conditions of cold regions and soil containing a fraction of organic soils. Exploring the application of electrokinetic remediation in these contexts offers a promising alternative to traditional techniques for the effective and sustainable treatment of BTEX-contaminated organic soils. Moreover, from an economic perspective, electrokinetic remediation presents advantages such as reduced excavation and disposal costs. Additionally, the potential for on-site treatment minimizes the need for off-site transportation and associated expenses, further enhancing its economic viability.

During the electrokinetic (EK) treatment process, three essential electrokinetic phenomena come into play: electroosmotic flow, electrophoresis, and electromigration. Each of these phenomena

plays a crucial role in facilitating the remediation process. Furthermore, electrokinetics generates a gradient of pH between the anode and cathode.

To achieve optimal outcomes during the EK treatment, various parameters can significantly influence the results, such as a voltage gradient, conditioning agents (along with their concentration), and electrode configurations. Conditioning agents, typically liquids containing surfactants, can enhance the mobilization and extraction of contaminants (Riekkola, 2000).

1.2 Objectives

The principal objective of this study is to comprehensively investigate the applicability of electrokinetics for remediating light hydrocarbon-contaminated soils containing an organic fraction. The research aims to delineate the optimal criteria and test conditions necessary to achieve efficient and effective soil remediation outcomes.

A secondary objective of this study is to expand the scope of the investigation to encompass cold regions, thereby evaluating the viability and efficacy of electrokinetic remediation under challenging environmental conditions prevalent in such areas.

1.3 Thesis structure

The thesis comprises five chapters. Chapter 1 provides an introduction, presenting the problem statement and research scope. In Chapter 2, a comprehensive literature review is conducted, examining Canadian soil properties and exploring technologies to mitigate the environmental impact of BTEX in permafrost. Chapter 3 details the methodology, including materials, sample properties, experimental setup, and analytical methods. The experimental results and discussions are presented in Chapter 4. Finally, Chapter 5 concludes the thesis, summarizing the findings, discussing research contributions, and offering recommendations for future work.

2 Literature Review

2.1 Soil classification in Canada

The classification of soil varies depending on the needs of a particular region or country. Four primary classification systems for soil are widely used across different parts of the world, each offering a unique perspective on the properties and characteristics of soil: the National system, French system, Soil Taxonomy, and WRB FAO UNESCO. Soil Taxonomy is used in the United States, the western regions of South America, the northern parts of Africa, and the western regions of Asia.

The National system is another widely used classification system applied in Canada, Russia, China, Australia, some European countries, and some countries in the southern regions of Africa. This system considers the specific characteristics and properties of soils within a particular country or region based on factors such as climate, vegetation, parent material, and time.

The French system and WRB FAO UNESCO classifications are used in Mexico, Greenland, and some European countries. The French system focuses on soil's morphological and physical properties, such as color, texture, and structure. The WRB FAO UNSECO classification considers the properties of soil and the environment in which it occurs, including climate, vegetation, and geology (University of Idaho, 2023).

Before 1955, soil testing in Canada relied on classification methods similar to those used in the United States, the soil Taxonomy system (Pidwirny, 2023).

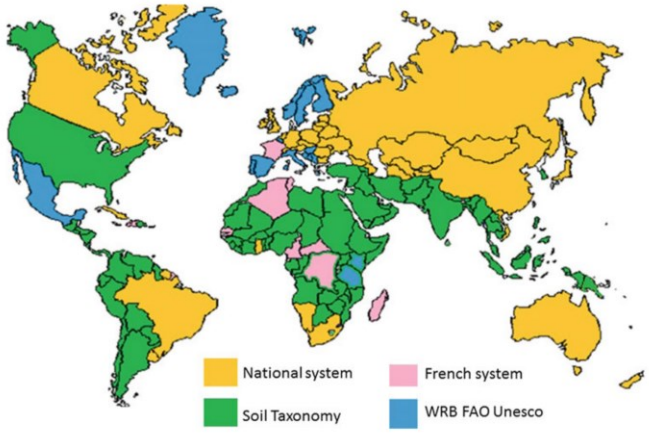


Figure 2.1 Soil classification over the world (West et al., 2016)

In the most recent version of the classification system for Canadian soils, a hierarchical structure consisting of five categories arranged from general to specific: Orders, Great Groups, Subgroups, Families, and Series (Krasil′nikov, 2009).

The Canadian soil classification has nine soil orders: Cryosolic, Organic, Vertisolic, Podzolic, Gleysolic, Solonetzic, Chernozemic, Luvisolic, Brunisolic, and Regosolic. The geographical distribution and locations of each soil order are visually presented in Figure 2.2, introduced by the Canadian society of soil science.

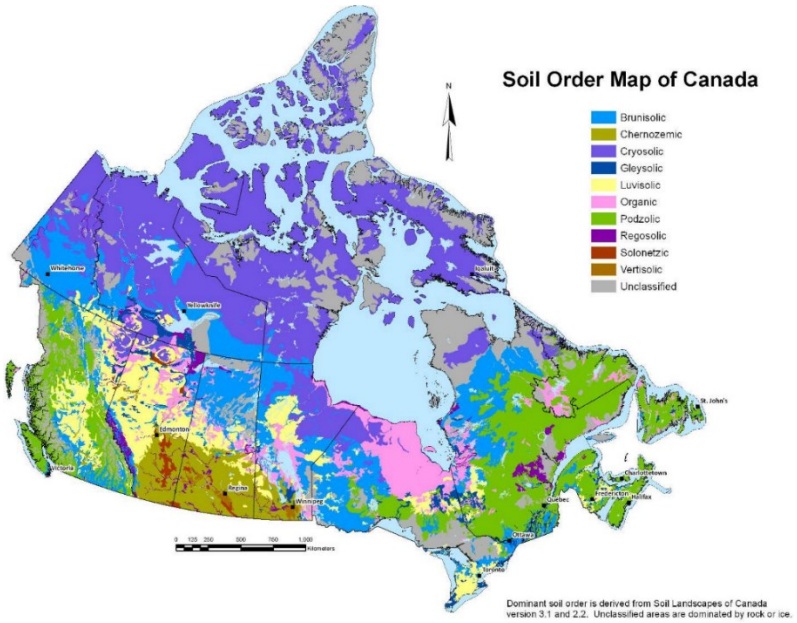


Figure 2.2 The soil order map of Canada (Canadian Society of Soil Science, 2020)

2.2 Organic soil

The distribution of organic matter in Canadian soils exhibits variation influenced by several factors, including soil type, climate, vegetation, and land use practices (Gorham 1957). Forested areas and wetlands demonstrate comparatively high levels of organic matter, often ranging from 50% to 90%. In contrast, arid and semi-arid regions generally exhibit a lower organic matter content, typically not exceeding 1%. Soils containing approximately 17% soil organic carbon (SOC) are commonly classified as organic soils (Troeh and Thompson 2005).

Organic soils can be observed throughout all provinces and territories of Canada, with a notable concentration in proximity to forested regions (Clayton 1977). Wetland organic soils cover every province and territory encompassing an approximate area of 755,568 km^2, representing 8.4% of Canada's territory (Kroetsch et al. 2011).

Organic soils are very important in Canada because of their enormous impact on climate change issues. They actively participate in carbon sequestration and influence greenhouse gas dynamics, thus necessitating their inclusion in climate change mitigation and adaptation strategies (Kroetsch et al. 2011). Therefore, it is essential to protect organic land against pollution.

2.2.1 Peat

Bogs, mires, moors, or muskegs develop peat, which is a collection of partially decomposed vegetation or organic matter (Joosten et al. 2002). It forms when the decomposition process fails to keep up with organic matter production, producing dark fibrous material (Waksman 1943). Peat formation occurs in acidic and anaerobic conditions when plant material does not fully decay, often in waterlogged environments with low oxygen or nutrient levels or low temperatures (Sahu and Singh 2019). Peat develops in wetland environments when floods or standing water obstructs the passage of oxygen from the atmosphere, reducing the decomposition process (Smardon 2014).

Peat primarily comprises sphagnum moss, also known as peat moss, although other plant species can also contribute (Walker 2019). Globally, peat stores an estimated 550 gigatons (Gt) of carbon, accounting for 42% of all soil carbon. This exceeds the carbon stored in all other vegetation types, including forests, despite peat covering only 3% of the land's surface. Peat is essential to the global carbon cycle and attempts to combat climate change because of the substantial carbon reservoir that it contains.

Peat deposits can be found in various regions worldwide, with notable concentrations in northern Europe and North America. In North America, significant peat deposits are principally located in Canada and the Northern United States. Some of the world's largest peatlands include the West Siberian Lowlands, the Hudson Bay Lowlands, and the Mackenzie River Valley (Jean 2006). In Canada, sphagnum-dominated peatlands predominantly exist within the permafrost zone, where evapotranspiration levels remain lower than precipitation (Kroetsch et al., 2011).

The average annual temperature within the specified geographic regions of Canada has been monitored and reported by the nation's environmental authorities. These findings indicated a temperature range of 7 to 8 degrees Celsius during the period spanning from June to July. This comprehensive dataset is drawn from a thirty-year observational period from 1981 to 2010 (Canada, 2023). While the average temperatures during the remaining months of the year exhibit a distinct and notably cooler pattern in comparison to the observed range in the aforementioned three months, forward-looking projections suggest the potential for a substantial temperature shift. Specifically, there is an anticipated increase of approximately 10 degrees Celsius in the average temperature over the next half-century (Kirkey, 2017).

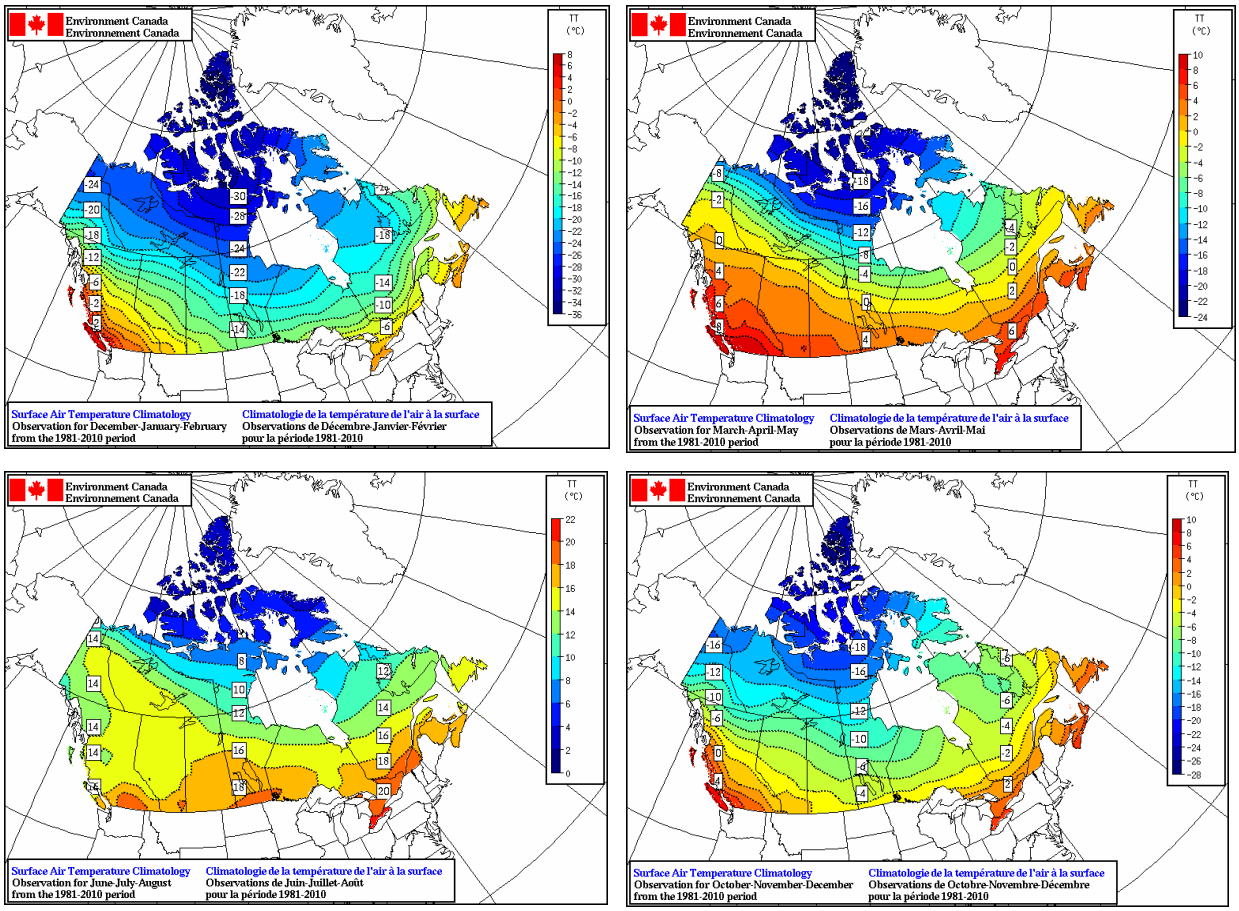


Figure 2.3 Temporal patterns of average annual temperature in Canadian regions (1981-2010), (Canada, 2023).

2.3 Permafrost

Permafrost is a critical component of Canada's northern landscape and has significant implications for infrastructure, transportation, natural resource development, and climate change. Permafrost is a frozen ground for two or more consecutive years and covers a vast area of Canada's northern regions (Osterkamp and Burn, 2003; Ford et al., 2010). Large amounts of carbon and other greenhouse gases are held back by permafrost, which can regulate the climate and ecosystems of the planet (Mackelprang et al. 2016).

A range of ground ice types, including segregated ice, pore ice, and massive ice, characterizes permafrost in Canada. These ice types can significantly impact the mechanical properties of permafrost, and their distribution can vary across different regions and landscapes.

Climate change has caused permafrost in Canada to degrade more rapidly in recent years, which has caused the permafrost to thaw and altered its physical and hydrological characteristics. This degradation has significant implications for infrastructure, transportation, natural resource development, and Indigenous peoples' traditional ways of life in the North (Kokelj and Jorgenson 2013).

2.3.1 Permafrost active layer

The active layer, which is the soil, rock, or sediment layer that freezes and thaws annually above the permafrost, plays a crucial role in the Arctic environment. According to Anisimov et al. (1997), the thickness of the active layer varies with the season, ranging from 0.3 to 4 meters. This layer serves as a conduit for the transfer of heat to/from the underlying permafrost. (Osterkamp and Burn 2003).

Global warming has triggered significant consequences, including thawing permafrost soils in the Arctic. As climate changes persist, the underlying permafrost undergoes detrimental effects, expanding the depth of the seasonally thawed active layer. This thawing process not only results in the release of previously confined liquid water but also fosters an increase in microbial activity (Zhang et al. 2021). Consequently, the potential exists for the dispersion of soil pollution to adjacent regions. A comprehensive understanding of the active layer's characteristics and its relationship to permafrost is crucial for devising effective strategies to mitigate these changes' environmental and societal impacts (Muhilan and Chattopadhyay, 2022; Anisimov et al., 1997).

2.4 Toluene

2.4.1 Introduction to BTEX

In the realm of petroleum refining and petrochemical industries, the acronym BTEX encompasses a group of aromatic hydrocarbons known as benzene, toluene, ethylbenzene, and the three isomeric forms of xylene, distinguished as ortho – (or o –), meta – (or m –), and para – (or p –). The structural diagrams of the BTEX hydrocarbons can be observed in Figure 2.3 (Bolden et al., 2015). Additionally, certain properties of these compounds at room temperature and a temperature of 7°C are presented in Table 2.1 (Kieffer 1975).

Table 2.1 BTEX properties in 20°C and 7° C (Kieffer 1975)

	Benzene	**Toluene**	**Ethylbenzene**	**P-Xylene**	**M-Xylene**	**O-Xylene**
Molecular formula	C_6H_6	C_7H_8	C_8H_{10}	C_8H_{10}	C_8H_{10}	C_8H_{10}
Molecular Mass, g.mol^{-1}	78.12	92.15	106.17	106.17	106.17	106.17
Log Kow	2.13	2.73	3.15	3.15	3.2	3.12
Log Koc	1.79	2.15	2.31	2.49	2.29	2.38
Dynamic viscosity, mPa.s, 20° C	0.66	0.59	0.67	0.65	0.64	0.81
Dynamic viscosity, mPa.s, 7° C	0.81	0.69	0.79	0.79	0.74	0.97

Toluene, classified as a substituted aromatic hydrocarbon, is a colorless liquid with distinct characteristics resembling paint thinners. It exhibits limited solubility in water but is miscible with various organic solvents, including ethanol, benzene, diethyl ether, acetone, chloroform, glacial acetic acid, and carbon disulfide (Benignus 1981; Filley et al., 2004). Toluene is present in many basic fuels, e.g., diesel fuel, which is still used in heavy machinery, trucks, trains, ships, etc.

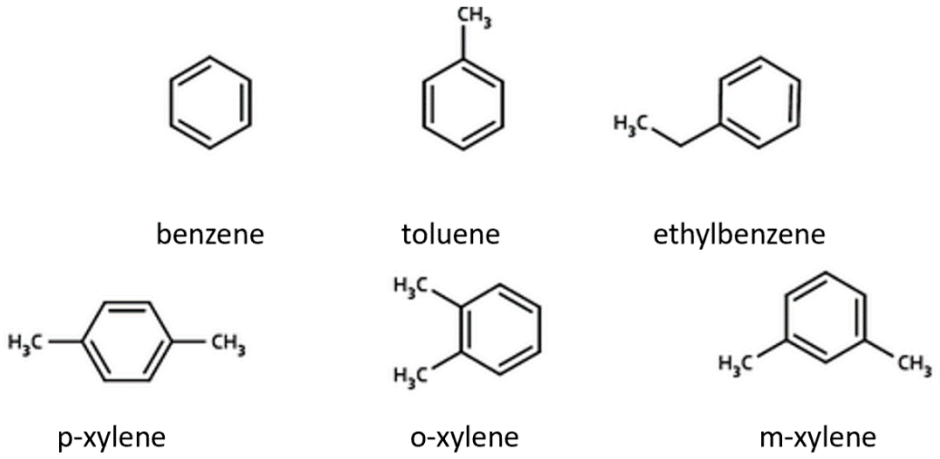


Figure 2.4 BTEX structural diagrams (Bolden et al., 2015)

2.4.2 Sources and exposure ways

BTEX sources are crude oil, seawater near natural gas, and petroleum deposits. These compounds are widely utilized in various industries, such as gasoline production, printing, leather, and rubber manufacturing. Therefore, spills in these areas are a common source of environmental pollution. Subsequently, light hydrocarbons like BTEX can find their way into the soil through various pathways, including pipeline leakages, corrosion, and spills. For instance, the most significant leak in the province in 35 years occurred in April 2011 when a massive spill occurred close to the Little Buffalo First Nations hamlet in northwest Alberta (CBC 2015).

Historical data reveals that 1973 and 1993 witnessed the highest reported spills, with 27 and 26 incidents, respectively. Among these spills, the most significant one was reported near Swan Lake, Manitoba, in October 1967, when over 5.2 million liters of crude oil spilled into the surrounding environment (Kheraj, 2020).

An incident such as the Exxon Valdez oil spill from an oil tanker in Prince William Sound, Alaska, on March 24, 1989, released 260,000 to 750,000 barrels of crude oil (WWF 214).

Improper disposal practices can also contribute to soil contamination by BTEX compounds. When these substances are not handled and disposed of correctly, they can seep into the soil, causing environmental harm.

It is vital to highlight Canada's strategic geographical position alongside three major oceans, which positions the country as a significant player in the global shipping industry. Canada's proximity to the Arctic region has become particularly noteworthy due to the melting ice cap, leading to increased shipping activity and the transportation of goods and resources through the Arctic Bridge Gateway and the Northwest Passage. However, this heightened shipping activity also brings a heightened risk of pollution. Harsh weather conditions, narrow shipping channels, and winter ice cover further amplify the likelihood of accidents and incidents that can result in pollution.

Figure 2.5 illustrates Canada's two significant shipping routes: The Arctic Bridge Gateway and the Northwest Passage. The Arctic Bridge Gateway encompasses seven potential routes, with the most viable options being M'Clure Strait, Prince of Wales Strait, and Peel Sound, with the latter connecting Canada to Russia.

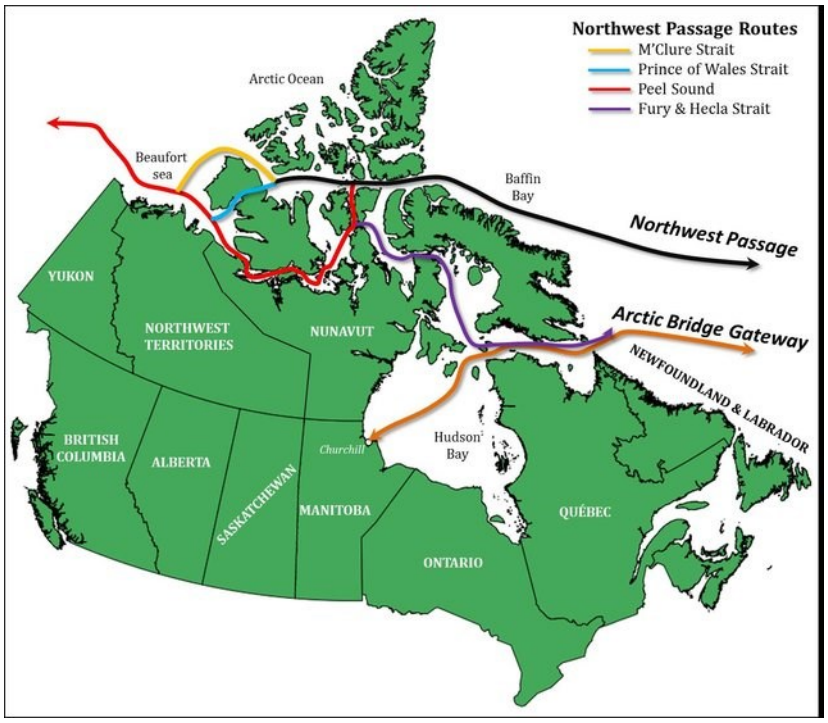


Figure 2.5 Main Canadian shipping routes (Gavrilchuk and Lesage 2014)

2.4.3 Environmental and health issues related to BTEX

Significant adverse impacts of toluene, as another BTEX compound, on the environment and human health exist. Toluene's volatilization affects air quality and may result in inhalation exposure, which adds to air pollution. Toluene's toxicity towards soil organisms disrupts soil processes, reducing soil fertility and hindering nutrient cycling. It also pollutes ecosystems,

impairs groundwater quality, and disrupts ecological balance (Chae et al. 2021). Human exposure to toluene can result in various health issues, ranging from mild symptoms to severe conditions such as skin irritation, liver damage, and kidney damage (Benignus 1981).

2.4.4 The fate and behavior of BTEX components in soil

Once released into the soil, BTEX compounds undergo various fate processes influenced by factors such as soil type, microbial activity, and environmental conditions. Two significant fate mechanisms for BTEX compounds are volatilization and biodegradation.

Volatilization is a prominent fate process where BTEX compounds evaporate from the soil into the atmosphere. Highly volatile compounds like benzene and toluene exhibit more pronounced volatilization. However, organic matter or clay in the soil can slow the volatilization rate (Zhenghu and Xiao, 2000). Nevertheless, soil polluted with diesel preserves the toluene content during aging process. It still might be a good representative of such pollution in conducted studies.

Biodegradation might be another crucial fate mechanism for BTEX compounds in topsoil. Microorganisms present in the soil have the ability to metabolize and break down these compounds under suitable conditions. BTEX compounds generally tend to be more biodegradable under aerobic conditions where oxygen is present in topsoil (Morgan et al., 1993). However, toluene undergoes biodegradation process in anaerobic conditions either (Edwards & Grbić-Galić, 1994).

Temperature, diffusion, and distance from the source significantly influence the levels of BTEX compounds in the environment (Yu et al., 2022). Generally, lower concentrations are observed in warmer months. For instance, in Beijing, BTEX concentrations in the air were 1.5 times higher in November compared to July (Liu et al. 2013). Another example from a semi-urban environment in Orleans, France, shows the average concentration order of BTEX compounds as follows: summer (337 ppt), autumn (682 ppt), spring (724 ppt), and winter (823 ppt) (Yu et al. 2022).

2.4.5 BTEX soil pollution regulation in Québec

Table 2.2. displays the acceptable soil pollution criteria specific to Quebec concerning the maximum permissible concentration of BTEX in each kilogram of dry soil. Table 2.2. is divided into three sections, representing the standards for agricultural, residential, and commercial or industrial zones.

Table 2.2 Regulation respecting contaminated soil storage and contaminated soil transfer stations(Québec 2023)

	Agricultural (mg/ kg of dry soil)	Residential (mg/ kg)	Commercial / Industrial (mg/ kg)
Benzene	0.5	5	100
Toluene	3	30	100
Ethylbenzene	5	50	100
Xylene	5	50	300

BTEX removal can be achieved through a variety of methods. Some of these practical approaches include Soil Vapor Extraction (Amin et al. 2014), bioremediation (Prenafeta-Boldú et al. 2004), surfactant washing (Liang et al. 2016), phytoremediation (Boonsaner et al., 2011), and electrokinetics (Maini et al. 2000; Hatem & Elektorowicz, 1999; Hakimipour & Elektorowicz, 2001).

2.5 Common remediation methods of petroleum-polluted soil in cold regions

The choice of remediation method for petroleum-polluted soil in cold regions depends on several factors, including the extent and type of contamination, the soil properties, and the available resources. A combination of remediation methods may be necessary for optimal results. The highest challenge of the most cost-effective soil remediation in-situ is time limitation to 4 -5 months.

2.5.1 Soil washing

Soil washing is an ex-situ and solvent extraction process for soil treatment which consists of two processes. In the first process, which is physical separation, by washing the soil, the small particles of clay and silt are separated to avoid increase of soil volume due to clay expansion behavior. Thus, soil washing is applied to coarse material with the highest efficiency. Secondly, chemical leaching is achieved by adding a surfactant, where its hydrophilic tail is associated with the petroleum fractions, make them soluble in water.(Fernández Rodríguez et al. 2014; Kostecki et al., 2005)

The washing method of soil containing clay fraction is not cost-effective since it increases the volume of material to manage. Furthermore, a considerable amount of water required treatment in

addition to a significant space reserved for equipment. Moreover, due to a short summer period, the use of water for soil washing systems is limited.

2.5.2 Phytoremediation

Phytoremediation is a term for a wide variety of technologies with the same base, and it is the removal of contaminants, especially hazardous wastes, and clean-up by using different kinds of high plants in the contaminated sites. Their main difference is the type of plants used for remediation or the type of target contaminant. For instance, phytoextraction, rhizoextraction, and phytofiltration are used to remove organic or inorganic contaminants (Bolan et al., 2011; McCutcheon and Jørgensen, 2008). However, this process is slow, and the application of phytoremediation in cold climates is limited to a short vegetation period and types of plants.

2.5.3 Bioremediation

Microorganisms using carbon from pollutants for their metabolism are helpful for soil bioremediation (Shackelford, 2013). It can be carried out based on bioaugmentation of indigenous or inoculated microbial cultures. In frigid climates, where conventional treatments could be ineffective, bioremediation might be a viable strategy for soil treatment (Sarkar et al. 2005); however, its effectiveness is influenced by many variables such as the type of contamination, characteristics of the soil, temperature, availability of nutrients, and moisture content. Bioremediation provides a long-term and economical solution for soil contamination problems by enhancing microbial activity through nutrient supplementation and environmental changes (Gouma et al. 2014); however, in cold regions is significantly limited, unless conducted in a closed area.

2.5.4 Soil vapor extraction

Volatile organic compounds (VOCs) can be removed from polluted soil using a process known as soil vapor extraction (SVE). It involves applying a vacuum to the soil, which induces the transfer of VOCs from the solid to the gas phase in soil pores. Heating the soil can increase this process. SVE is effective in the vadose zone and requires a network of extraction wells connected to a vacuum pump. The extracted air is treated to remove contaminants. However, SVE may face limitations like low permeability soils and low volatility contaminants. To overcome these limitations, a combination of SVE with other in-situ remediation techniques, such as air sparging,

thermal desorption, or chemical oxidation, can be used (Rathfelder et al., 1995). This approach is not necessarily cost-effective when applied energy is high, particularly in cold regions.

2.5.5 Electrokinetic soil remediation

Electrokinetic soil remediation, initially developed in the late 1980s (Reddy and Cameselle 2009), is an in-situ and ex-situ methodology utilized for soil remediation (Elektorowicz, 1994; Kariminezhad and Elektorowicz 2018). This approach involves the application of a low-voltage direct current (DC) to a complex colloidal clay-medium, resulting in the induction of electroosmosis, electrophoresis, and electrolytic migration (Elektorowicz & Hatem, 2000; Elektorowicz, 2009). Its primary purpose is the extraction of radionuclides, heavy metals, and specific organic compounds, as well as mixed inorganic species (Elektorowicz, 2009) and organic wastes from soils and slurries (Habibi & Elektorowicz, 2004). Electrokinetic remediation has been employed worldwide for soil decontamination purposes, mostly for metal remediation (Acar et al. 1995).

When combined with facilitating agents such as surfactants, co-solvents, nanoparticles, and oxidizing chemicals, electrokinetics becomes capable of remediating organic contaminants in soil (Reddy and Cameselle 2009). These agents assist in the removal or degradation of contaminants in-situ (Hakimipour & Elektorowicz, 2001; Hatem & Elektorowicz, 1999; Rajaei & Elektorowicz, 2023).

2.5.5.1 Electrokinetic phenomena

Electrokinetic phenomena encompass a range of effects within the electrokinetic process, including electroosmotic flow (EO), electrophoresis (EP), and electromigration (EM), as illustrated in Figure 2.6. These phenomena collectively describe the movement and manipulation of particles and fluids under the influence of an electric field.

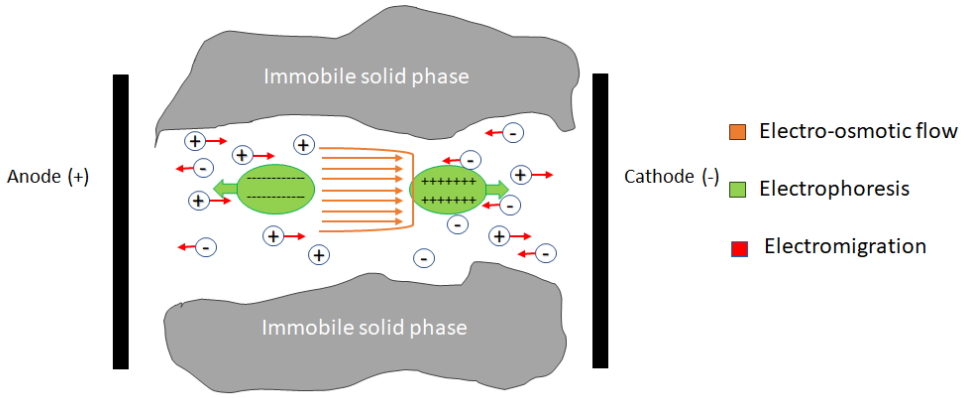


Figure 2.6 Electrokinetic phenomena (Hakimipour & Elektorowicz, 2001)

2.5.5.1.1 Electroosmotic flow (EO)

Electroosmotic (EO) flow refers to the liquid movement within a porous material, capillary tube, membrane, microchannel, or some other fluid conduit, typically toward the cathode (when the medium has a negative charge) under the influence of an applied potential (Jennings and Mansharamani 1999). It is also known as electroosmosis, which describes the electrically driven motion of liquid through a charged porous solid medium (Delgado et al. 2007). This phenomenon occurs due to Coulomb force exerted by an electric field on a solution's overall mobile electric charge (Tiflidis et al. 2021).

The electroosmotic flow is typically directed toward the cathode in the clayey soils' electrokinetic remediation. However, in the electrokinetic remediation of organic soils, it has been observed that organic matter resulted in liquid absorption restraining the flow (Figure 2.7.b) (Ghanami et al., 2023). By introducing a liquid booster to enhance the EO flow, significant movement from the anode to the cathode can be achieved.

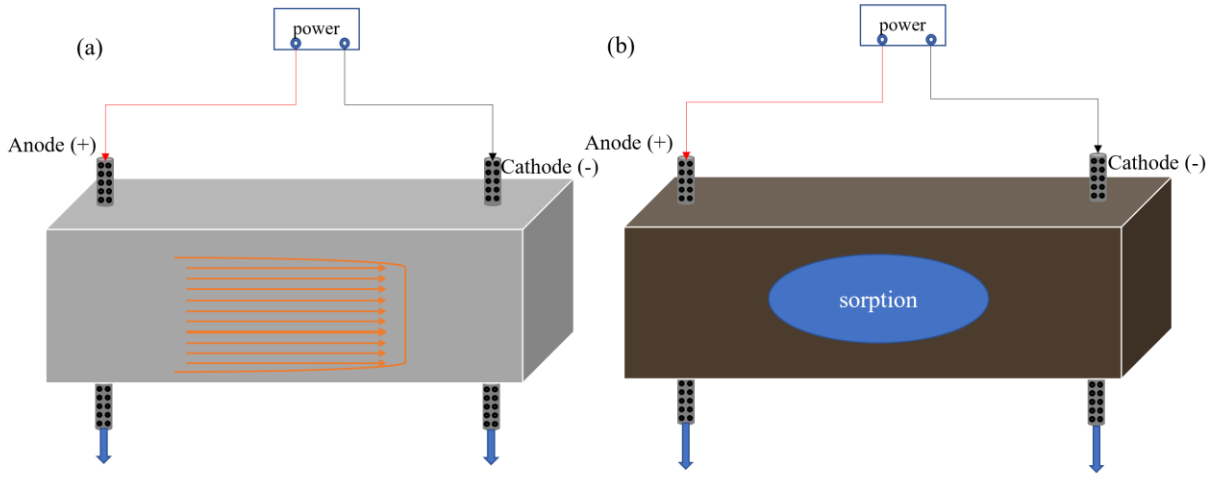


Figure 2.7 Electroosmotic flow, a) in a clayey soil matrix, and b) in an organic-soil matrix

2.5.5.1.2 Electrophoresis (EP)

Electrophoresis (EP) involves dispersed charged particles moving in relation to a fluid while being affected by an electric field that is uniform in space (Anderson 1989). It involves migrating mobile solid phase particles driven by their net surface charge within an applied DC field, typically directed towards the anode (Jennings and Mansharamani 1999). This process describes the electrical migration of charged colloidal particles or polyelectrolytes immersed in a liquid medium (Delgado et al., 2007).

2.5.5.1.3 Electromigration (EM)

Electromigration (EM) is the phenomenon wherein individual ions migrate within a conductor under an applied DC field (Jennings and Mansharamani 1999). This process involves the directional movement of ions toward their corresponding electrodes, i.e., cations toward the cathode and anions toward the anode. Inorganic pollution is driven by the momentum transfer between conducting electrons and diffusing metal atoms, resulting in the slow transit of material (Oh et al. 2017). Electromigration occurs when an electric field, created by a voltage difference, is present (Claisse 2016).

2.5.5.2 Advantages, disadvantages, and possible limits of electrokinetics

The electrokinetic (EK) remediation technique offers various advantages for treating contaminated soils. Firstly, it demonstrates exceptional effectiveness in addressing fine-grained soils with low permeability, which proves challenging for conventional remediation methods (Fernández

Rodríguez et al. 2014). Additionally, one of the notable features of EK is its silent operation, which allows for environmentally friendly and inconspicuous remediation processes. An important advantage of electrokinetic remediation is its application in-situ. Also, its temperature dependence is also low; thus, it is suitable for cold regions (Raihan & Elektorowicz, 2006).

However, the implementation of EK is not without its drawbacks. One significant concern involves the possibility of electrode corrosion, thus, anode should be periodically exchange to avoid a negative impact on the system efficiency. Another limitation arises from introducing external fluid into the soil during the process, potentially hindering the extraction of certain contaminants if they cannot be dissolved, but external conditioners are also required for other remediation methods (Acar et al. 1995). Moreover, the electric potential of the system may experience a decline, posing another challenge to the effectiveness of EK remediation.

To address these limitations, several solutions can be considered. The injection of appropriate chemical stabilizers represents a promising approach to maintaining the electrical potential within the system. Furthermore, use multifunctioning perforated cylindrical electrodes can serve as a drainage wells. their combination with inert perforated wells might effectively manage the external fluid introduced during the remediation process.

2.6 Surfactant

Commercial surfactants have garnered significant attention as potential environmental hazards for soils and groundwater, primarily due to their substantial environmental emissions (Eriksson et al., 2002). These compounds can significantly affect the mobility and availability of potentially hazardous organic compounds in soil, posing a threat to ecological systems (Mulligan et al., 2001; Hernández-Soriano et al., 2007). Moreover, carboxylic acids, a specific type of surfactant, have been found to influence the structure of organic matter and induce the dispersion of humic aggregates (Oades, 1984). Subsequently, their use in soil has to be closely controlled. Electrokinetics controls the fate of surfactant distribution, managing the liquid to follow the current lines.

Anionic surfactants, such as those with sulfate, sulfonate, phosphate, and carboxylates as their head groups, have become common surfactant types (Hernández-Soriano et al., 2011; Hatem & Elektorowicz, 1999). Examples of notable anionic surfactants include ammonium lauryl sulfate,

sodium lauryl sulfate (SLS or SDS), sodium laureth sulfate (SLES), and sodium myreth sulfate. These surfactants have effectively removed dirt, clay, and certain oily stains from materials (Williams, 2007).

Particularly, micellar electrokinetic chromatography (MEKC) techniques have preferred anionic surfactant systems. This preference arises from the unique characteristics of anionic surfactants, as they allow micelles' electrophoretic migration in the direction that is the opposite of the electroosmotic flow. Additionally, the micelles do not interact with the negatively charged walls of fused silica capillaries, enhancing the separation efficiency in MEKC (Riekkola 2000). However, micelles can follow this transport toward the cathode when the electroosmotic flow is strong enough.

3 Methodology

3.1 Methodological approach

This study considered different soil compositions to accomplish the research objectives, focusing specifically on the organic matter content. These compositions included 0%, 3%, 16%, and 35%. The remaining portion of the soil comprised a mixture of coarse and fine sand, along with clay (bentonite (montmorillonite).

The experimental work in this study comprised four stages, as illustrated in the flowchart shown in Figure 3.1.

The research progression comprised several stages aimed at achieving the study's objectives. The initial stage served as an exploratory phase, where multiple variable factors were examined to ascertain the most optimal testing conditions. These factors included the voltage gradient, water injection ratio, presence or absence of surfactants, moisture levels of the initial soil components (dry or wet), and the methodology employed for effluent discharge.

Subsequently, the study progressed to the primary phase (Phase 1), known as the second stage. Building upon the knowledge obtained from the exploratory stage, the soil was meticulously mixed to generate four distinct compositions, varying in the percentage of organic matter content. This investigation encompassed a range of organic carbon percentages from 0% to 35%. The ambient room temperature (20°C) was maintained throughout this phase to ensure consistency.

Continuing the research trajectory, Phase 2 was conducted under colder conditions to simulate real-world scenarios relevant to the study. The samples employed in Phase 2 were identical to those utilized in Phase 1, ensuring consistency and comparability of results. A cooling chamber was appropriately regulated to achieve the desired lower temperature, maintaining the ambient temperature at 7 degrees Celsius, while temperature inside of soil fluctuated between 3 to 7 degrees Celsius.

Furthermore, a supplementary stage was conducted under the specific cold temperature of 7°C, specifically focusing on the variable factors of toluene and surfactant. The tests were performed on a soil mixture containing 35% organic carbon. Manipulating these variables resulted in four distinct experimental conditions, classified by the presence or absence of toluene and surfactant.

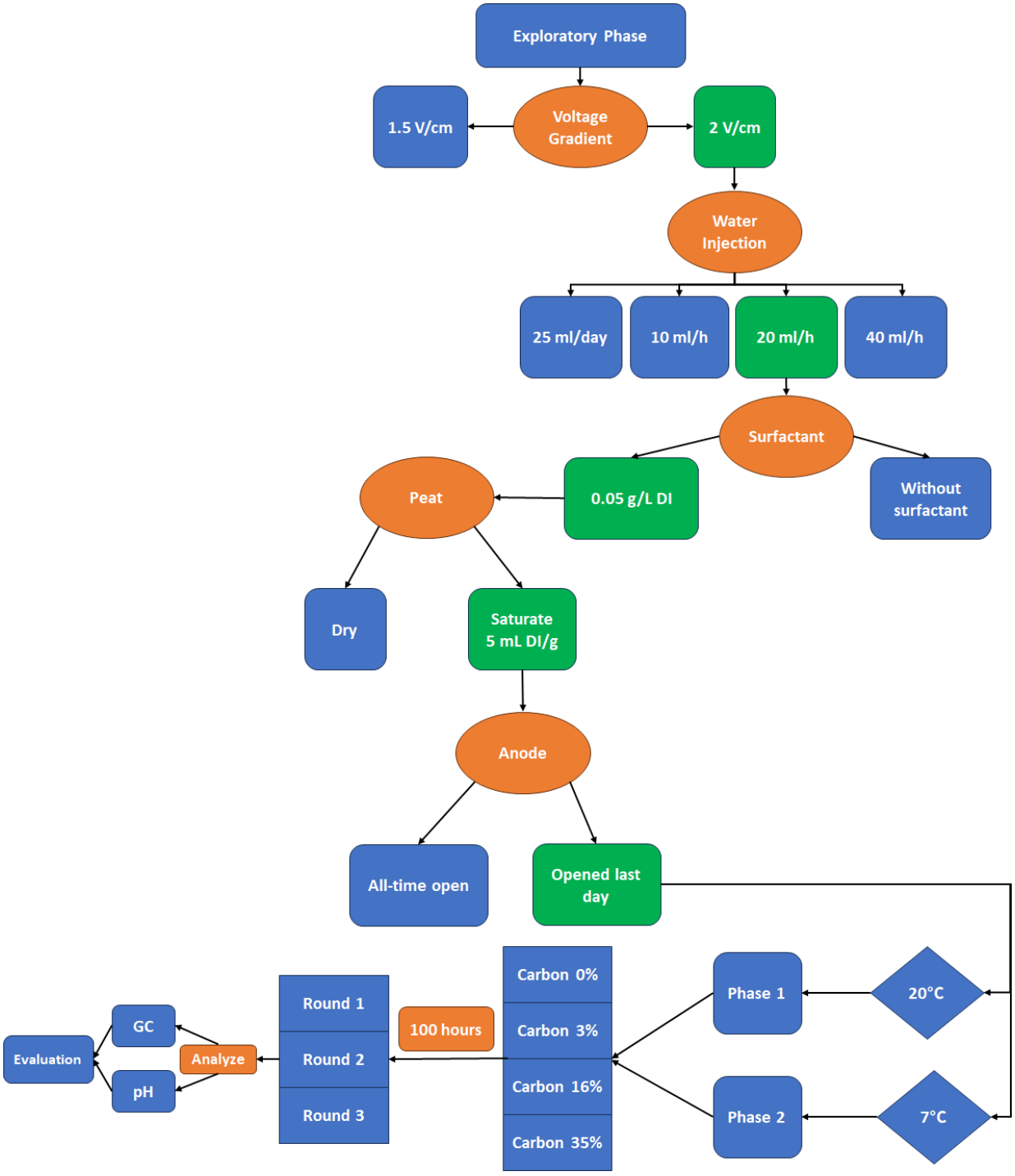


Figure 3.1 The methodology used for the experimentation

3.2 Soil-sample description

3.2.1 Pollutant properties

Toluene, representing light hydrocarbons pollution, was obtained from the Fisher Scientific Group for use in this study. It has a molecular weight of 92.14 g/mol. This colorless liquid is characterized by its high purity, with a minimum guaranteed percentage purity of 99.5%. Toluene is liquid and exhibits a low water content, with a maximum concentration of 0.03%. The viscosity of toluene is measured at 0.6 mPa/s and 0.7 mPa/s at a temperature of 20°C and 7°C, respectively.

The concentration of toluene in the soil samples was intentionally set higher than the residential standards established by the Quebec government (Table 2.2). Each kilogram of the dry mass soil mixture contained 40 mg of toluene.

3.2.2 Soil samples properties

The soil used in this study aimed to replicate the conditions which can be found in the northern territories of Canada. It was composed of a combination of sand, clay, and peat, serving as the organic matter component. The distribution of these soil constituents varied as the tests were conducted using four different organic matter concentrations. Specifically, 0%, 3%, 16%, and 35% of organic matter were incorporated into the soil to facilitate comparative analysis and assess the suitability of this method.

A constant parameter was maintained in all tests, ensuring a consistent presence of 3% clay content. The remaining portion of the soil composition consisted of sand. The following table presents the diverse distributions of samples used in the experimentation:

Table 3.1 Characteristics of soil samples in the experimentation

	Sample 1	**Sample 2**	**Sample 3**	**Sample 4**
Peat (%)	0	3	16	35
Sand (%)	97	94	81	62
Clay (%)	3	3	3	3

Screening Floral ("Poussière de Pierre") was selected as the sand component for the soil mixtures in this study. Sand used in the experiments was procured from Charbonneau Floral LTÉE (Laval, QC, Canada).

The sand composition consisted of a blend of 70% medium sand with a diameter range of 0.425-2.00 mm and 30% fine-sized sand with a diameter range of 0.002-0.075 mm (Rajaei & Elektorowicz, 2023). Combining these sand types ensured a diverse grain size distribution, contributing to the desired characteristics of the soil mixtures used in the research (ASTM, 2014).

In this study, the clay component was represented by bentonite containing mostly montmorillonite minerals. The bentonite used in this research was from BPM Minerals, LLC (Houston, TX), packaged in Calgary, AB. Bentonite, known for its remarkable water-swelling capability and a surface area approaching 1000 square meters per gram, boasts a high cation exchange capacity (CEC) (Huang et al., 2017).

Activated sphagnum peat, obtained from Sphag Sorb (SS-8Q) located in Edmonton (Alberta), was used as the organic carbon component in this study. Peat, being hydrophilic, was saturated with water prior to the experimentation. For every gram of activated peat, 5 mL of water was required to achieve full saturation.

3.3 Sample preparation

The soil mixtures were prepared in a methodical manner to ensure consistency and homogeneity for the experimental investigations.

Firstly, the sand mass was divided into eight equal portions. These portions were systematically mixed in pairs, repeating until only one portion remained, ensuring thorough distribution and homogenization.

Secondly, the well-mixed sand was further divided into eight equal portions, and the clay component was added in equal proportions to each portion. Employing a similar pairwise mixing approach as in the previous step, the sand and clay components were meticulously blended until a singular well-incorporated portion was obtained.

Finally, the water-saturated peat, vital for the desired composition, was incorporated into the eight equal portions of sand and clay. Employing the same method as in the prior steps, the combination of sand, clay, and moist peat was meticulously mixed to obtain a uniformly distributed soil mixture.

The soil mixtures were deemed ready for subsequent testing following this rigorous preparation process. The final step involved the introduction of the targeted pollutant, toluene, into the prepared soil. A controlled quantity of 40 mg of toluene per kilogram of dry mixed soil was added. To ensure the pollutant's comprehensive dispersion within the soil matrix, the polluted soil mixture was allowed to rest for one day.

3.4 Electrokinetic parameters

3.4.1 Description of installation

3.4.1.1 Electrokinetic cells

In this study, the preferred material for the electrokinetic cells (EK) was polyethylene due to its resistance to petroleum products. The EK cells were designed with a top-open rectangular structure, facilitating direct soil access and convenient measurements and variations. A parafilm layer, Bemis^{TM} ACAPM996, was applied to the surface of every cell to mitigate the volatilization of toluene. Both Phase 1 and Phase 2 of the study employed cells with identical dimensions. Each cell possessed a length of 10 cm, a width of 5.5 cm, and a depth of 4.5 cm. These standardized dimensions ensured consistency and comparability between the two phases.

3.4.1.2 Electrodes

Each cell was equipped with two multifunctioning electrodes to enable the essential electrochemical phenomena. Cylindrical perforated stainless-steel electrodes served as an anode and as a cathode. Simultaneously, they served as drainage wells, enabling anolyte and catholyte collection from EK cells. The electrodes used in the experiment were positioned strategically at the midpoint of the cell's width to ensure optimal electrical current line distribution. Electrodes with a diameter of 1 cm were installed at 7cm from each other. This positioning and spacing configuration facilitated effective electrical field distribution and efficient performance of the EK cell (Figure 3.2).

To comprehensively investigate the electrokinetic cells' behavior and ensure the results' replicability, three parallel cells were simultaneously operated under identical conditions. This

concurrent arrangement enabled extensive observations and analyses, leading to distinct rounds denoted as round 1, round 2, and round 3 within the ongoing study. Figure 3.2 presents a schematic representation of one of the cells in the study.

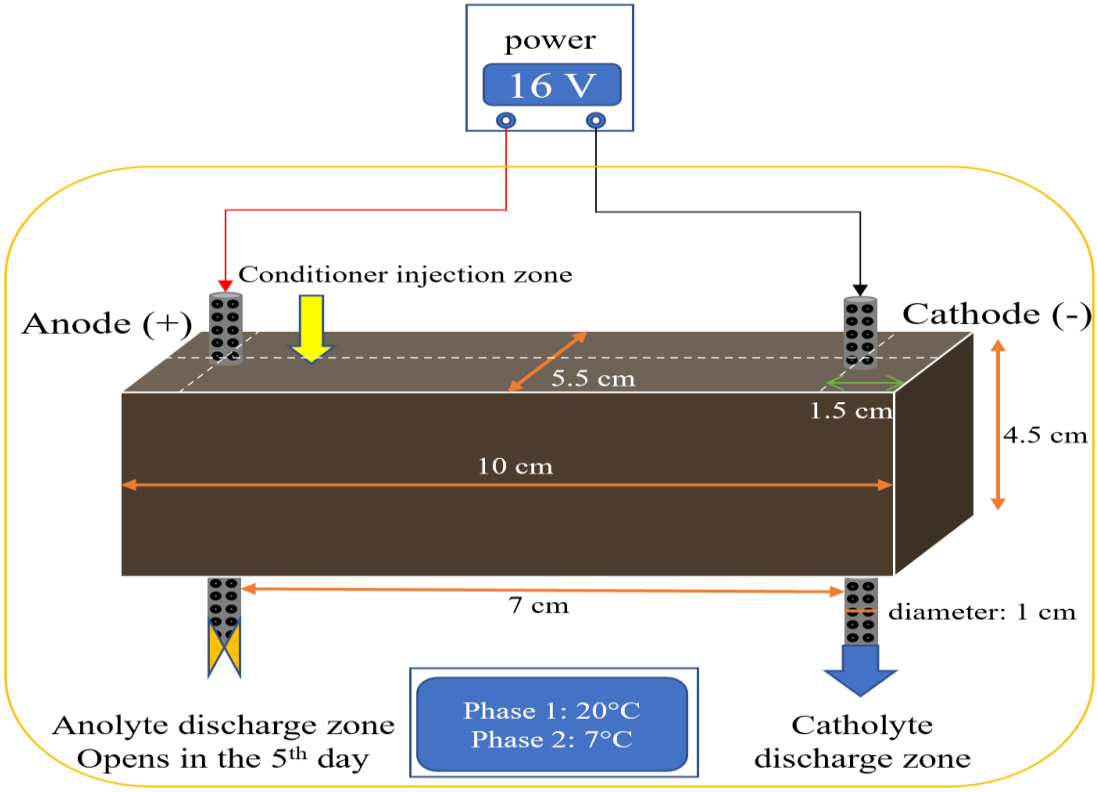


Figure 3.2 Configuration of electrokinetic cells

3.4.1.3 Power supply

The experiment was powered by two distinct power supplies, enabling the simultaneous application of electrical voltage to all three electrokinetic cells. The first power supply utilized was the AB-5300, a linear DC power supply acquired from ABRA Group. With its two channels connected to the first and second cells, the AB-5300 model provided a stable and precise power source. In addition, the TES 6230, a regulated DC power supply from TES company, was employed in the experiment. Both power supplies could deliver a voltage range of 0 to 30 Volts, ensuring flexibility in meeting the experimental requirements. Three high-quality couple wires cables, featuring banana plugs to alligator clips, were utilized to establish a seamless connection between the power supplies and the electrodes.

Based on previous studies (Hakimipour & Elektorowicz, 2001; Hatem & Elektorowicz, 1999) on clayey materials, a constant voltage gradient was applied in all three channels to promote electrokinetic phenomena motions.

The assessment of EK cell amperage was facilitated using the DM6000AR multimeter (AstroAI group).

3.4.1.4 Temperature measurement

The temperature measurements in this research were conducted using thermocouples located inside the soil matrix. IPB-16S PID Temperature Controller provided the temperature. This model, renowned for its precision and reliability, was selected by the INKBIRD company. The thermometer boasts a temperature range of -50 to 125℃ and offers an accuracy level of ±0.1℃.

3.4.1.5 Environmental chamber

The VBV-60-FRP-E fume hood, supplied by Bedcolab company, served as the environmental chamber during Exploratory stage (Phase 0) and Phase 1 of the test, maintaining a constant temperature of 20-21°C.

The HOTPACK 305210 environmental chamber played a crucial role in Phase 2 of the study. This particular model offers a temperature range of 2 to 55°C. During this phase of the tests, the chamber temperature was set to 7°C, creating conditions akin to the cold regions of Canada.

3.5 Application of a conditioner

To promote changing of the toluene partitioning from solid phase to liquid phase, a conditioner in the form of a surfactant solution was applied. An anionic surfactant (Hostapur® SAS 60) was chosen due to its potential biodegradability (Chemicals 2020). Its chemical name is Sodium C14-17 Alkyl Sec Sulfonate. The following reaction is involved in its production:

$$R\text{-}H + 2SO_2 + O_2 + H_2O \rightarrow RSO_3H + H2SO_4 \text{ (Chemicals 2020)}$$

The solution used for the experiment was prepared by combining 2 liters of deionized (DI) water with 0.1 g of the anionic surfactant. Then after a careful evaluation, an injection rate of 10 mL of surfactant solution per 30 minutes or 20 mL per hour would yield the most effective results. To determine the optimal rate, the behavior of the soil mixture and the discharge amount of the solution from the electrodes were considered. A higher tendency of micelles to carry contaminants

towards the cathode zone has been seen after the injection of an anionic surfactant due to the electroosmotic flow.

3.6 Experimental procedure

3.6.1 Exploratory Phase- Responding of various soil parameters to different electrokinetic parameters

During this phase [Phase 0], an initial investigation was conducted to establish optimal experimental conditions for the subsequent study phases. This preliminary stage aimed to determine the most influential parameters for the electrokinetic remediation process. Five main criteria were considered, and various factors were thoroughly examined and refined to achieve optimal results. These five criteria were applied to all the toluene-polluted samples with various percentages of organic matter (Table 3.1); the following criteria were challenged wherever an optimum condition was found.

3.6.1.1 Voltage gradient

Optimizing the voltage gradient was the first step in the experimental procedure. After evaluating two options - a voltage gradient of 1.5 V/cm and 2 V/cm - it was determined that a voltage gradient of 2 V/cm provided better removal of organic acids (Rajaei & Elektorowicz, 2023). This choice was confirmed visually by observing more vibrant and colorful discharges.

3.6.1.2 Water injection

Various ratios for water injection in the anode area were tested. Initially, water injection occurred only when the amperage reached 1 mA or lower, with a mere 25 mL of water added to the anode zone. However, this ratio proved insufficient for generating a significant amount of catholyte or anolyte. Subsequent investigations implemented a continuous watering system for the cells.

After evaluating three different watering systems, at rates of 10 mL/h, 10 mL/30 min, and 20 mL/30 min, it was determined that the most efficient ratio was 10 mL/30 min. Additionally, it was observed that 10 mL/30 min and 20 mL/h yielded similar behavior. Thus, it can be inferred that water injection follows a linear relationship.

3.6.1.3 Surfactant

Initially, the washing system only consisted of DI water. However, further experimentation revealed that adding 0.05 g of anionic surfactant per 1 L of water improved pollutant removal (Rajaei & Elektorowicz, 2023).

3.6.1.4 Peat

The soil mixtures initially comprised dry peat, clay, and sand. However, it was observed that there was no discharge for several hours after initiating the tests. Since peat is highly hydrophilic, it was more time-efficient to saturate it before mixing it with other soil components. This adjustment also made the soil mixture more representative of real-life scenarios where the active layers are recently thawed and saturated with water. It was determined that each gram of peat required 5 mL of water for saturation.

3.6.1.5 Electrodes

The in-solution injection was placed in the vicinity of the multifunctional anode. Consequently, most discharges were collected as anolyte shortly after the solution injection. However, to allow for electroosmotic flow toward the cathode collecting on the way the pollutants, the anode was blocked for four days, and it was unblocked on the last day of tests to collect anolyte.

3.6.2 Phase 1- Response of organic soil to electrokinetics in mild temperature

Phase 1 was conducted at an ambient temperature of around 20°C, with the EK cells placed under a fume hood. The procedure for this phase was as follows:

- Three cells were filled with the initial soil mixture of 3% w/w clay, where organic fraction w/w changed from 0 to 35% (Table 3.1 and 3.2) while the remaining portion comprised sand. Each kilogram of dry soil mixture was polluted by 40 mg of toluene.

The cells were filled with various soil mixtures. Since the samples exhibited distinct distributions of organic matter (Table 3.1), their masses differed despite occupying the same volume, equivalent to cell volume (Table 3.2).

- The anode electrode's bottom was blocked to prevent any discharge from that section.

- A constant voltage gradient of 2V/cm was supplied to electrodes.

- After a 30-minute stabilization period, the first conditioner injection was introduced near the anode. This liquid injection occurred every 30 minutes with a volume of 10 mL or every hour with a volume of 20 mL.

- The injection process was repeated for a total of 5 hours per day (equivalent to 100 mL per day).

-After that, no additional injections were given for the rest of the day while keeping the system's voltage at 16 V constant after the 5-hour conditioner injection was finished. The conditioner injection started again the following day.

- This conditioner injection procedure with the blocked anode was repeated for four consecutive days, and the discharges from the cathode section (catholyte) were collected in a 50 mL vial.

- On the last day of the test (Day 5), the anode was opened, and discharges were collected from both the anode and cathode electrodes.

- Finally, 100 mL of the solution was injected, and after this injection, the cells and systems were closed and cleaned.

-Tests were repeated three times.

- Furthermore, during Phase 1, an additional set of experiments was conducted. This included a round of experiments using non-polluted soil samples and three rounds involving toluene-polluted soil mixtures. The purpose of including the non-polluted sample was twofold: first, to facilitate comparing the physical results obtained from the experiments (as discussed in Chapter 4), and second, to assess the suitability and applicability of gas chromatography (GC) tests for analyzing the experimental data.

Table 3.2 List of samples tested in Phase 1

Organic Matter	Round N	Round 1	Round 2	Round 3	Mass (g)
0%	C0_1N_TNSY	C0_11_TYSY	C0_12_TYSY	C0_13_TYSY	300
3%	C3_1N_TNSY	C3_11_TYSY	C3_12_TYSY	C3_13_TYSY	250
16%	C16_1N_TNSY	C16_11_TYSY	C16_12_TYSY	C16_13_TYSY	200
35%	C35_1N_TNSY	C35_11_TYSY	C35_12_TYSY	C35_13_TYSY	120

Note: The table utilizes a CQ_WE_TNSY code to provide information about various soil samples. The code breakdown is as follows:

-"C" is the referring to organic matter.

- "Q" represents the soil sample's organic matter percentage.

- "W" indicates the phase of testing, with "1" representing room temperature (Phase 1) and "2" representing cold temperature (Phase 2).

- "E" denotes the round of testing conducted.

- "T" is the referring to toluene in the sample, with "Y" indicating its presence and "N" indicating its absence.

- "S" signifies the surfactant in the conditioner used, with "Y" indicating its presence and "N" indicating its absence.

For example, the code "C35_13_TYSY" indicates that the soil sample has 35% organic matter. It is the third round of testing at room temperature (Phase 1). The sample was polluted with toluene, while surfactant was used as a conditioner.

3.6.3 Phase 2 – Response of organic soil to electrokinetics in cold temperature

Phase 2 followed the same procedure as Phase 1, with the only difference being the temperature. The EK cells were placed in a cold environmental chamber with a temperature set at 7°C. The tests were repeated three times (Tab. 3.3). The samples and experiments used in this phase were created to closely resemble the permafrost active layer's actual circumstances in the spring and summer when temperatures are higher than 0°C (Osterkamp and Burn 2003).

Table 3.3 List of samples tested in Phase 2

Organic Matter	Round 1	Round 2	Round 3	Mass (g)
0%	C0_21_TYSY	C0_22_TYSY	C0_23_TYSY	300
3%	C3_21_TYSY	C3_22_TYSY	C3_23_TYSY	250
16%	C16_21_TYSY	C16_22_TYSY	C16_23_TYSY	200
35%	C35_21_TYSY	C35_22_TYSY	C35_23_TYSY	120

3.7 Analytical equipment

3.7.1 pH meter

The pH measurements were conducted utilizing the accumet AB200 pH meter (Fisher Scientific) with an accuracy of ±0.002. This versatile instrument excels in pH measurement and assessing other parameters such as conductivity, TDS, salinity, resistivity, and temperature.

3.7.2 GC system

The GC system employed in the study was the Agilent 8890 GC. This instrument offers high-performance capabilities for accurate and reliable gas chromatographic analysis.

The DB-1 column, acquired from Agilent Technologies, was selected to separate and analyze volatile organic compounds. The column has a diameter of 0.250 mm and a film thickness of 0.25 µm. This capillary column, known for its non-polar nature, is highly suitable for a wide range of analytes, including alkanes, aromatics, alcohols, ketones, esters, and more. The DB-1 column demonstrates excellent thermal stability, ensuring reliable performance across a wide temperature range from -60 to 325°C. With its superior inertness, the column minimizes the potential for analyte interactions, thereby ensuring accurate and precise results. Additionally, the DB-1 column exhibits low bleed characteristics, effectively reducing background noise and enhancing the system's sensitivity.

3.7.2.1 Calibration curve

A 95% n-hexane solution was employed in the liquid-liquid extraction method to establish a calibration curve for quantification purposes. This calibration curve, constructed using n-hexane as a reference standard, enabled accurate quantification of the target compounds during the GC analysis.

Fisher Scientific provided n-hexane, which has a chemical formula of C_6H_{14}, for utilization in the study. With a molecular weight of 86.18 g/mol, this liquid compound is transparent and exhibits a purity level of 95%. In its liquid state, n-hexane has minimal water content, with a maximum concentration of 0.01%. At a temperature of 20°C, the viscosity of n-hexane measures 0.31 mPa/s.

Multiple standard solutions were prepared to establish an effective method for analyzing toluene content using gas chromatography (GC). These solutions were utilized to generate a standard curve, aiding in quantifying toluene levels.

The standard curve was constructed using solutions consisting of different concentrations of toluene in hexane. Specifically, solutions of 1000 ppm, 500 ppm, 250 ppm, and 100 ppm of toluene in hexane were meticulously prepared. These concentrations served as reference points for calibration during the subsequent GC analysis of toluene content in the samples.

Following the establishment of the calibration curve, a consistent hexane-to-liquid discharge ratio of 1:1 was employed to mix the effluent and hexane. The resulting mixture obtained from the previous step was agitated using a shaker. The agitation process occurred in a New Brunswick Scientific™ Innova™ 42 Incubator Shaker, which offers a speed range of 25 to 400 rpm. For this experiment, the shaker operated at 300 rounds per minute, ensuring consistent and thorough mixing. The agitation was carried out for at least 12 hours at room temperature (20°C), allowing for complete dissolution and uniform distribution of the components.

Once the shaking process was completed, the prepared mixtures were ready to be collected. For subsequent analysis using the gas chromatography (GC) system, a volume of 2 mL from each mixture was deemed sufficient for the testing vial. Each sample was subjected to double filtration using syringe filters sourced from Thermo Fisher Scientific. The filters were constructed from PVDF (hydrophilic) material, ensuring efficient filtration of the samples. With a pore size of 0.45 μm and a diameter of 13 mm, these filters were designed specifically for GC, HPLC (high-performance liquid chromatography), and SPE (solid-phase extraction) applications.

4 Results and discussion

The results presented encompass a range of parameters, including the discharge volumes of both the catholyte and anolyte (measured in milliliters), the volume of solution added during the experimental process (also measured in milliliters), variations in amperage (measured in milliamperes), and the cell temperature (measured in degrees Celsius) throughout the duration of the tests (measured in hours).

Furthermore, pH levels and toluene concentrations (in parts per million) are documented. The results are categorized into two distinct sections: Phase 1, which corresponds to room temperature conditions, and Phase 2, which pertains to cold temperature conditions.

4.1 Electrokinetic treatment under ambient temperature

In Phase 1, the experiments were conducted in a repeated manner, with three rounds of testing. An additional round of experimentation was introduced to further investigate the impact of toluene as a pollutant in the soil mixture. Consequently, the obtained results are categorized into two distinct subgroups. The initial subgroup consists of Round N and Round 1. Round N represents the non-polluted sample, whereas Round 1 and Rounds 2 and 3 comprise the toluene-polluted samples. The second subgroup included Rounds 2 and 3. The inclusion of the unpolluted mixture was essential to verify the identity of the observed peaks in the gas chromatography (GC) analysis, specifically in relation to their correspondence with toluene.

4.1.1 Pollutant extraction rate in the moderate temperature

4.1.1.1 Organic matter content: 0%

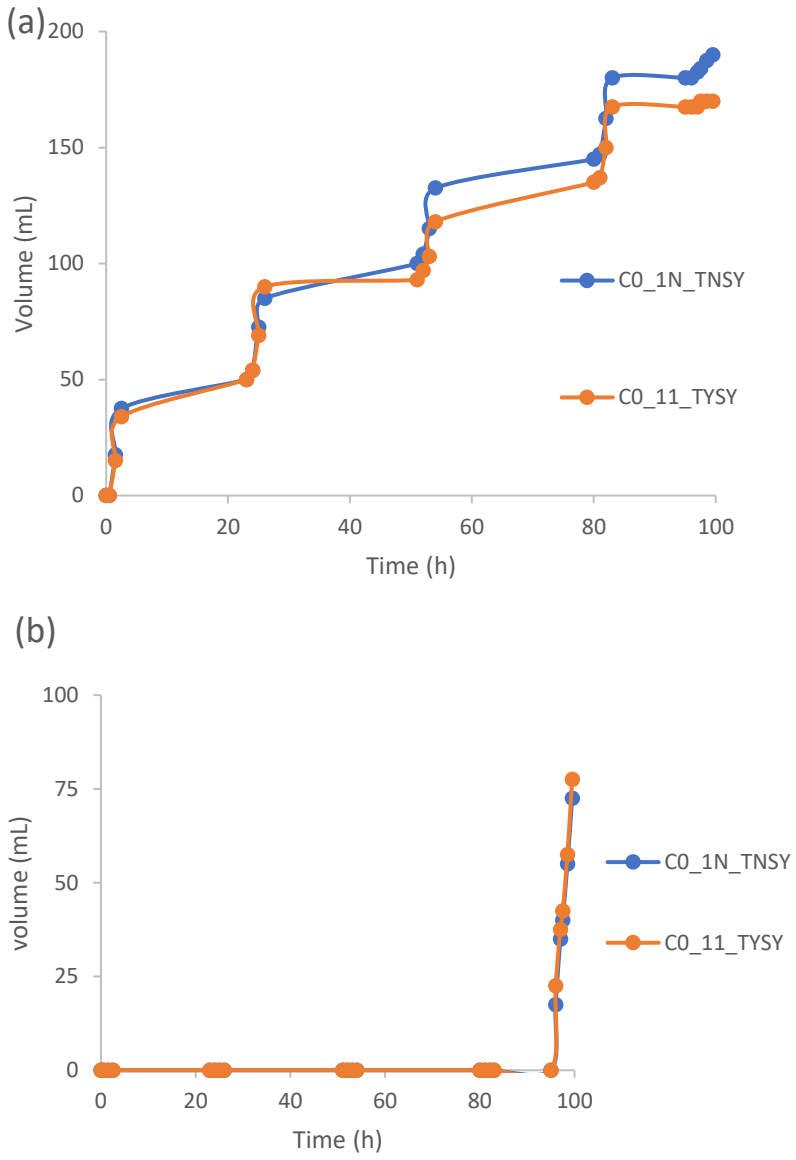
4.1.1.1.1 First subgroup: Round N vs. Round 1

Figure 4.1.a illustrates the discharge of the catholyte and anolyte over a duration of 99.5 hours during the testing process. In the case of the non-polluted sample, 190 mL of catholyte and 72.5 mL of anolyte were discharged. For Round 1, the corresponding values were 170 mL for catholyte and 77.5 mL for anolyte, as depicted in Figure 4.1.a-b.

Figure 4.1.c depicts the fluctuation of amperage throughout the testing period. Both samples exhibited a maximum amperage of 26 mA during the initial hours of the first day. As time progressed, the amperage gradually decreased, with daily peaks indicating the initiation of the

conditioner injection into the system. Notably, the amperage variation followed a similar trend in both cells, reaching 1 mA by the end of the test.

Figure 4.1.d presents the ambient temperature profiles observed over the 99.5-hour duration. The temperature remained relatively stable, ranging from 17 to 20 degrees Celsius for both EK cells throughout the testing period.



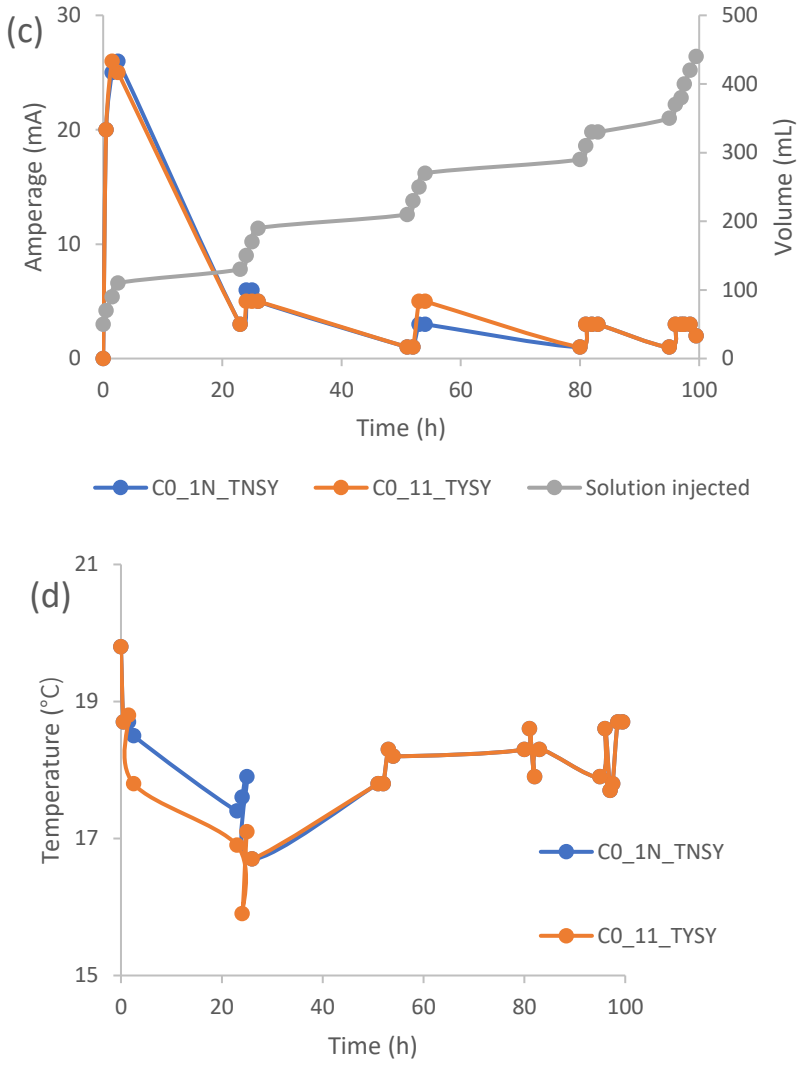


Figure 4.1 Phase 1 (Rounds 0 and 1) soil without organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and amperage, and d) Cell temperatures

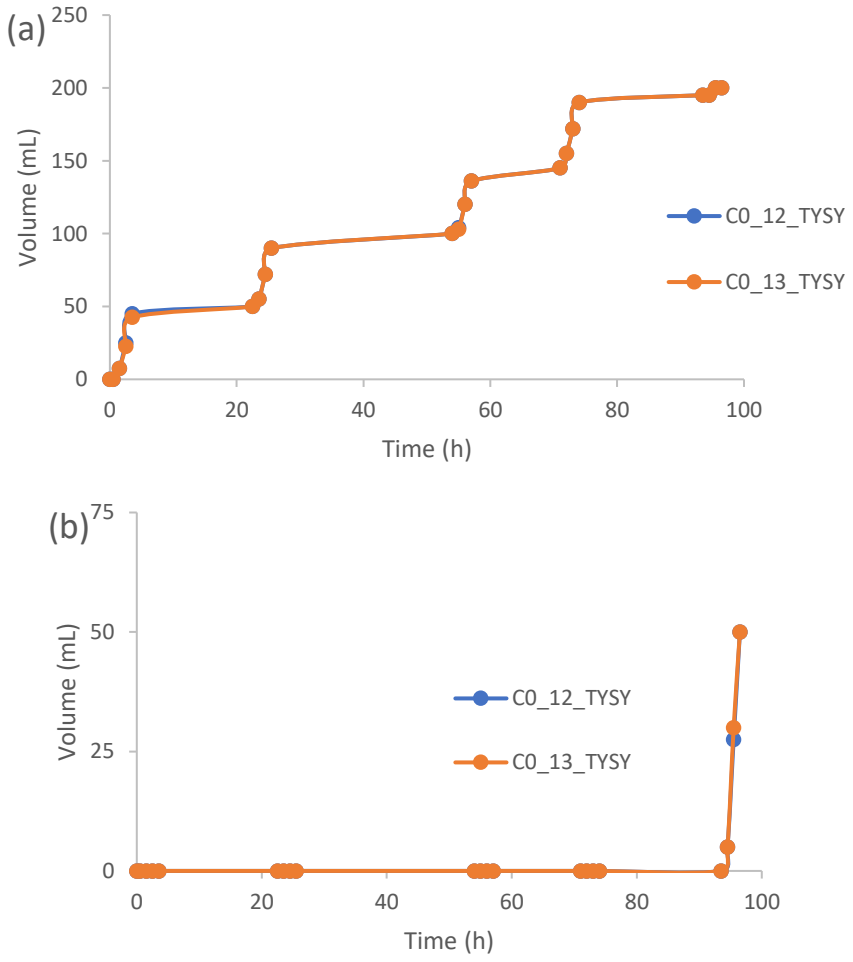
4.1.1.1.2 Second subgroup: round 2 and round 3

Figures 4.2.a and 4.2.b present the discharge behavior of the catholyte and anolyte over a duration of 96.5 hours during the testing. In Rounds 2 and 3, 200 mL of catholyte and 50 mL of anolyte were discharged in each round. Additionally, Figure 4.2.c illustrates the cumulative volume of injected solution for these cells, which amounted to 380 mL.

Figure 4.2.c further showcases the fluctuations in amperage observed throughout the testing period. Initially, both samples exhibited a maximum amperage of 20 or 21 mA, respectively, during the early hours of the first day. As the experiment progressed, the amperage gradually

decreased, with daily peaks indicating the introduction of the conditioner solution into the system. It is worth noting that the amperage variation followed a consistent trend in both cells, ultimately reaching 1 mA by the conclusion of the test.

Moreover, Figure 4.2.d provides insights into the temperature profiles recorded over the 96.5-hour testing period. The temperature remained within the 17 to 19.5 degrees Celsius range in both cells, demonstrating a consistent thermal behavior throughout the experiment.



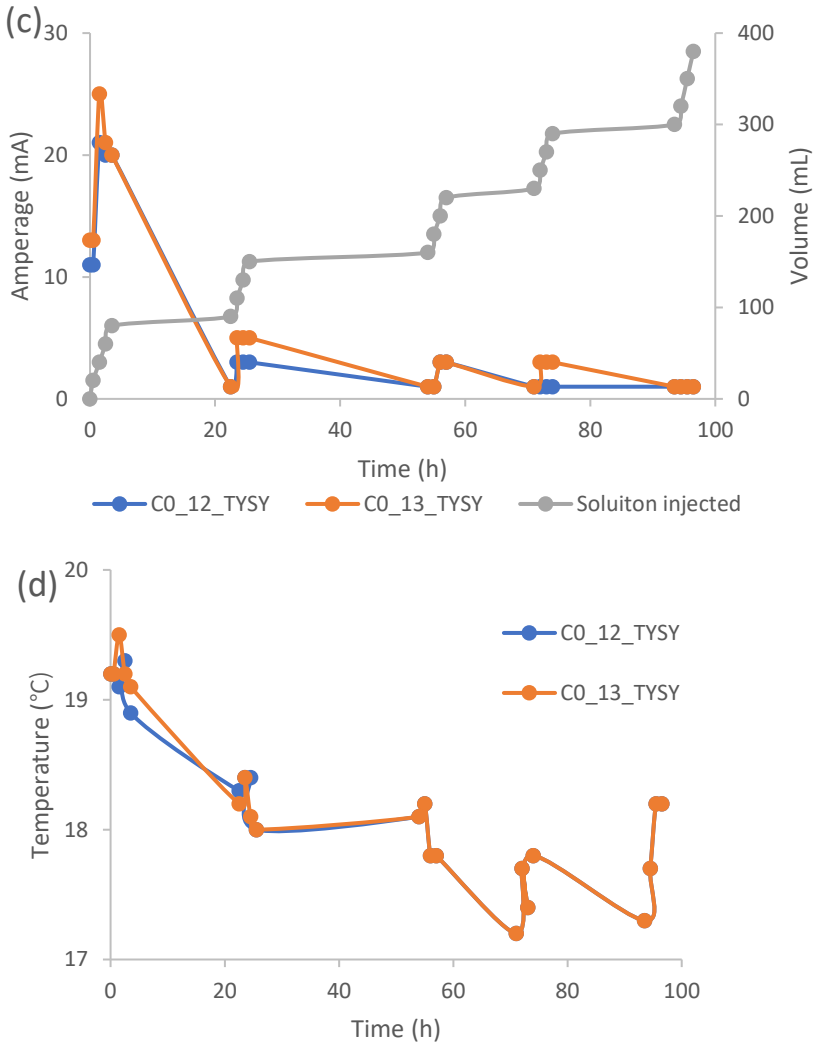


Figure 4.2 Phase 1 (Rounds 2 and 3) soil without organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.1.1.2 Organic matter content: 3%

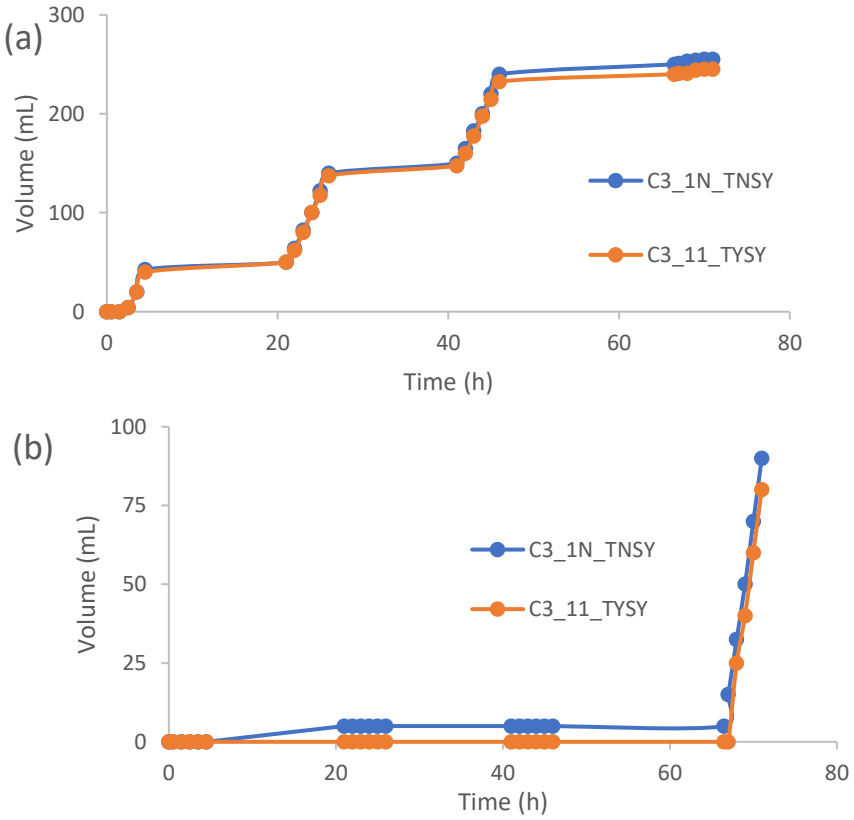
4.1.1.2.1 First subgroup: Round N vs. Round 1

Figure 4.3.a showcases the discharge behavior of the catholyte and anolyte over a duration of 71 hours during the testing phase. These two cells were filled with 120 g of soil mixture, occupying approximately one-third of the cell volume. The experiment was terminated at the 71-hour due to understanding the necessity of maintaining continuous electrical conduction by keeping the electrodes in contact with the soil mixture so the cells for the following tests were filled.

In the case of the non-polluted sample, the discharge volume of the catholyte was measured at 255 mL, while the anolyte discharge amounted to 90 mL. For Round 1, the corresponding values were 245 mL for the catholyte and 80 mL for the anolyte, as indicated in Figure 4.3.a-b. Additionally, Figure 4.3.c provides an overview of the total volume of injected solution, which reached 430 mL for these cells.

The fluctuations in amperage throughout the testing period are depicted in Figure 4.3.c. Initially, both samples exhibited a maximum amperage of 10-11 mA during the initial hours of the first day. The amperage gradually decreased to 1 mA, with daily peaks indicating the injection of the solution into the system.

Furthermore, Figure 4.3.d presents the temperature profiles recorded over the 71-hour duration. The temperature remained relatively stable throughout the testing period, ranging from 18.5 to 20.7 degrees Celsius in both cells.



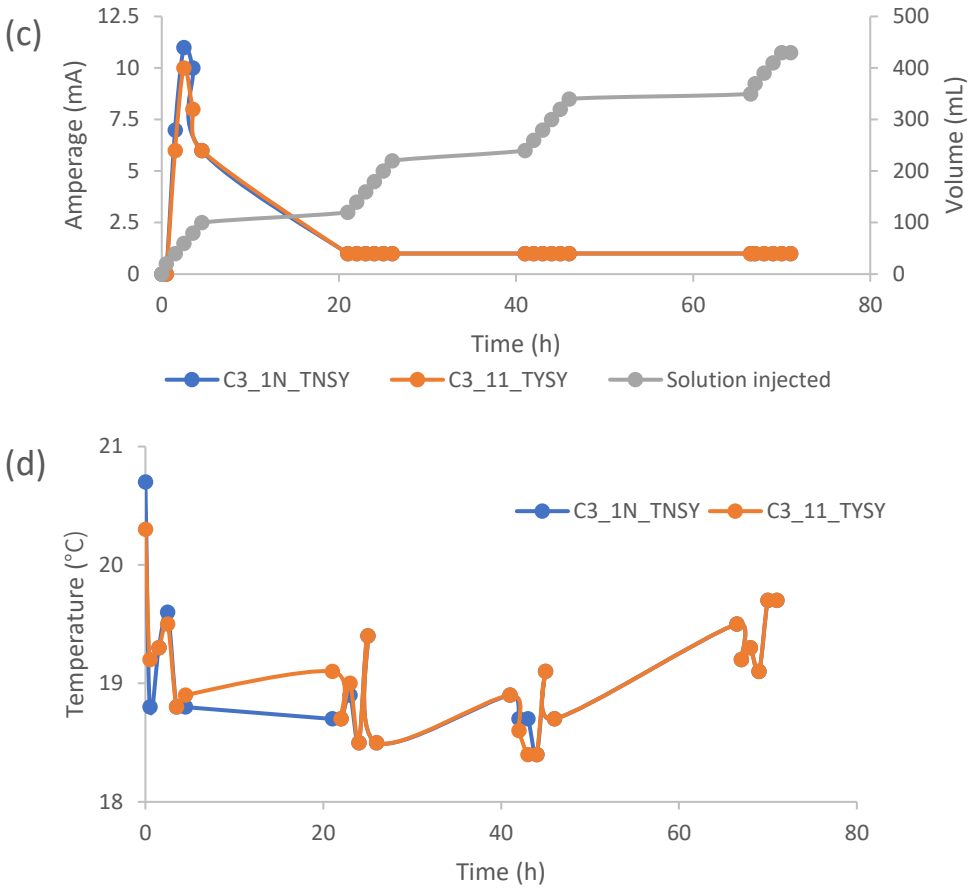


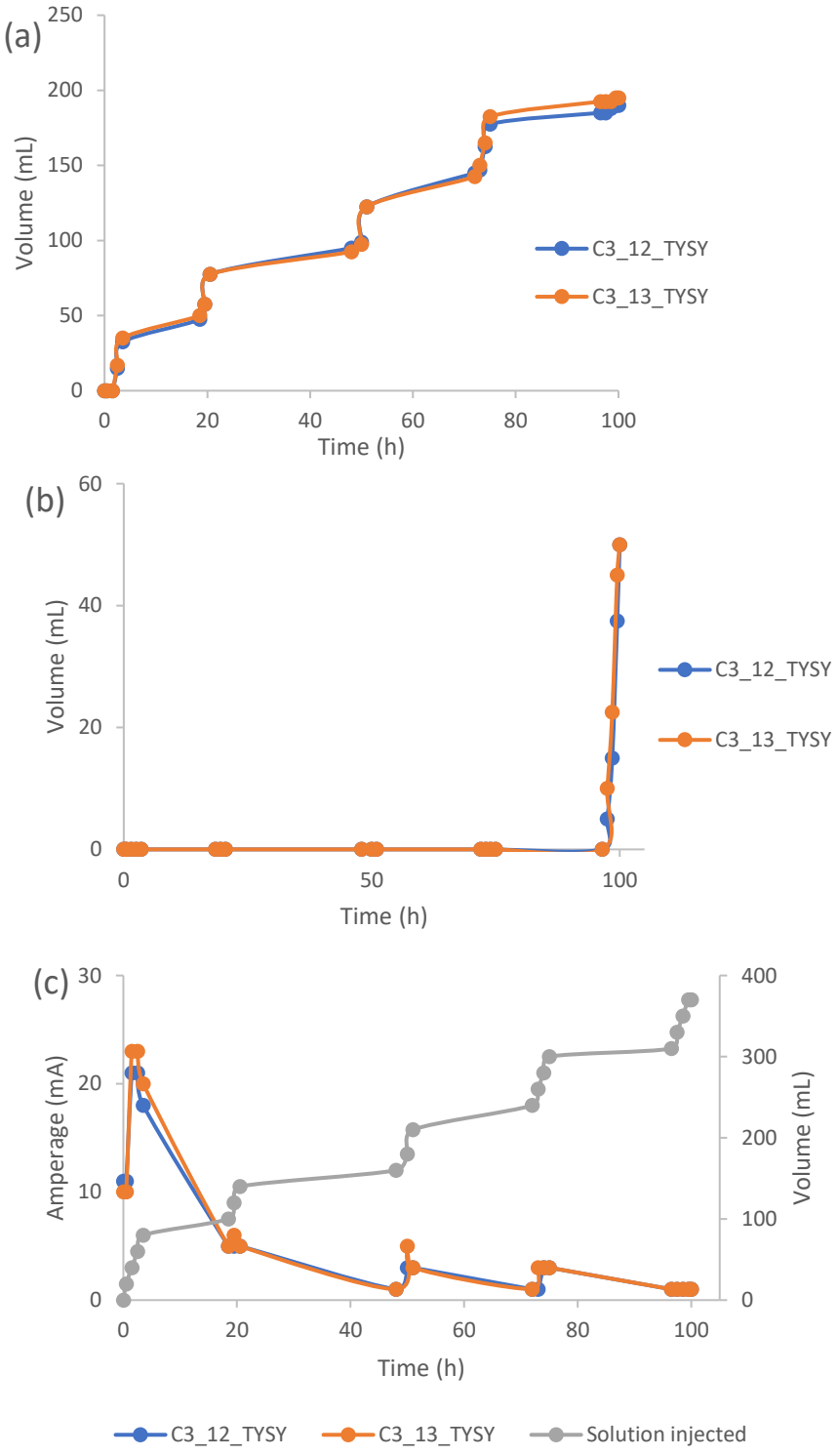
Figure 4.3 Phase 1 (Rounds 0 and 1) soil with 3% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.1.1.2.2 Second subgroup: round 2 and round 3

Figures 4.4.a and 4.4.b provide an illustration of the discharge patterns of the catholyte and anolyte over a duration of 100 hours during the testing phase. In Round 2, a discharge volume of 190 mL for the catholyte and 50 mL for the anolyte was observed, while in Round 3, 195 mL of catholyte and 50 mL of anolyte were discharged. These findings are visually represented in Figures 4.4.a and 4.4.b, respectively. The cumulative volume of injected solution for these cells amounted to 370 mL, as depicted in Figure 4.4.c.

The amperage fluctuation throughout the testing period is graphically presented in Figure 4.4.c. Both samples exhibited a maximum amperage of +20 mA during the initial hours of the first day. Consistent with previous tests, the amperage gradually decreased over time, ultimately reaching 1 mA by the conclusion of the test.

Moreover, Figure 4.4.d showcases the temperature profiles observed over the 100-hour duration. The temperature trend followed a similar pattern in both cells, with values ranging between 17 and 20 degrees Celsius. This consistent temperature behavior is graphically represented in Figure 4.4.d.



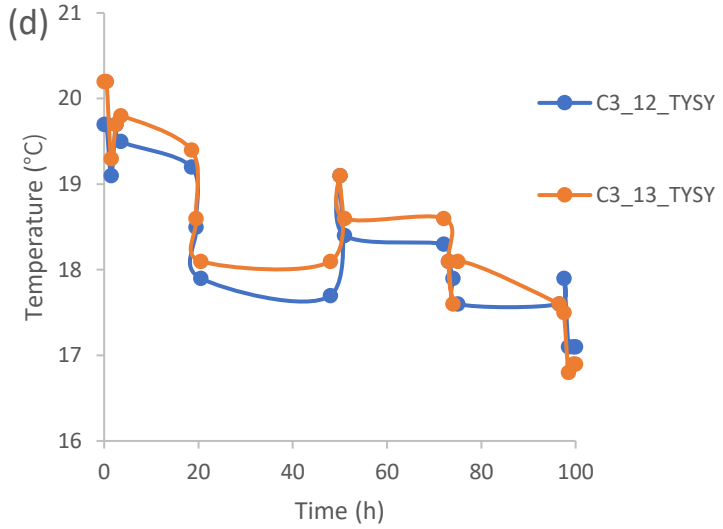


Figure 4.4 Phase 1 (Rounds 2 and 3) soil with 3% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

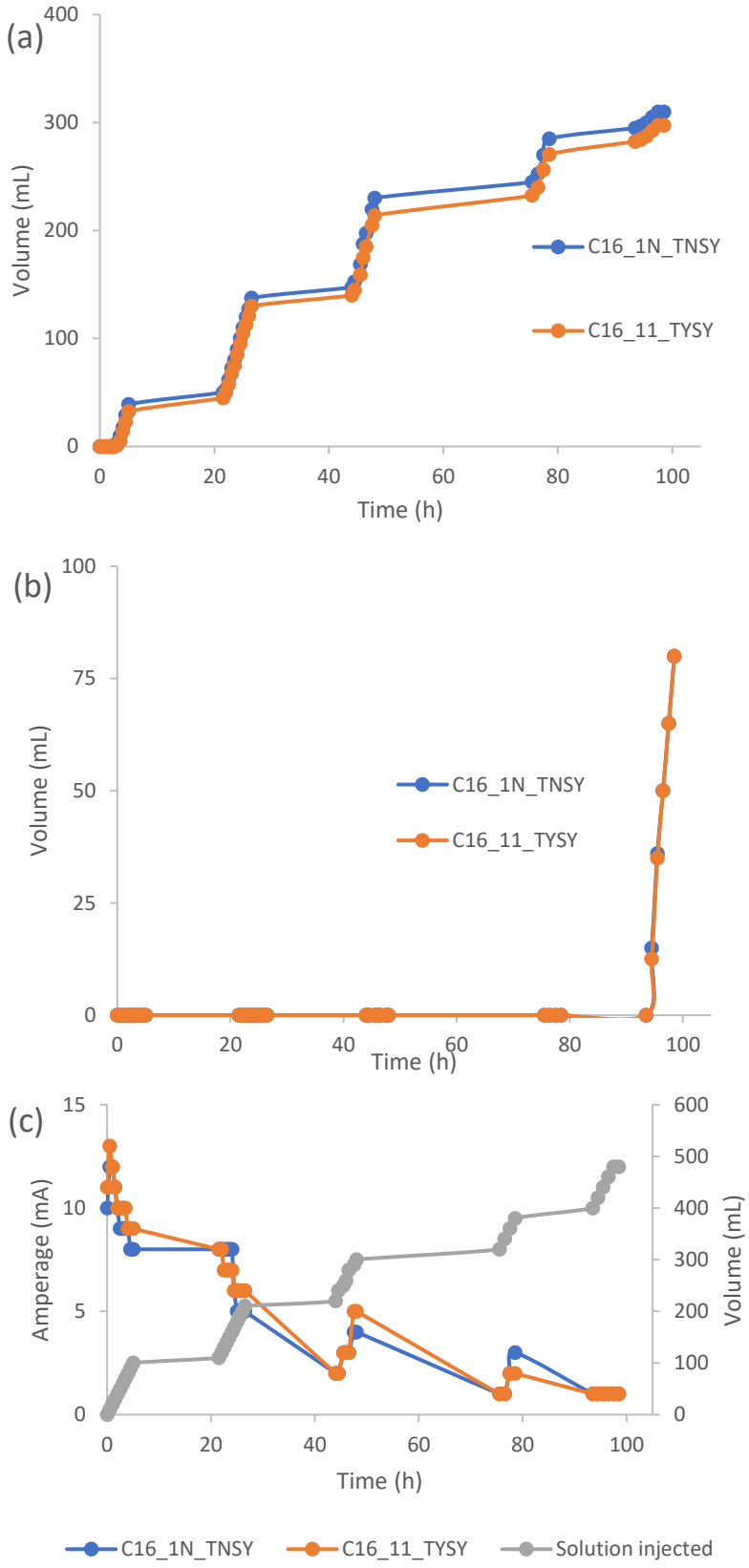
4.1.1.3 Organic matter content: 16%

4.1.1.3.1 First subgroup: Round N vs. Round 1

Figure 4.5.a provides an insightful representation of the discharge behavior of the catholyte and anolyte over a duration of 98.5 hours during the testing phase. In the case of the non-polluted sample, a discharge volume of 310 mL of catholyte and 80 mL of anolyte was observed. Similarly, for Round 1, the respective values were 297.5 mL for catholyte and 80 mL for anolyte, as visually depicted in Figure 4.5.a-b. The cumulative volume of injected solution for these cells amounted to 480 mL, as highlighted in Figure 4.5.c.

Figure 4.5.c illustrates the fluctuation of amperage throughout the testing period. Both samples exhibited a maximum amperage of +12 mA during the initial day. As time progressed, the amperage gradually decreased and eventually stabilized at 1 mA.

Furthermore, Figure 4.5.d provides an overview of the temperature profiles recorded during the experiment, ranging from a minimum of 17 degrees Celsius to a maximum of 20 degrees Celsius. This temperature range, demonstrated in Figure 4.5.d, reflects the stability observed in both cells throughout the testing period.


(a)
Volume (mL)
Time (h)
C16_1N_TNSY
C16_11_TYSY
(b)
Volume (mL)
Time (h)
C16_1N_TNSY
C16_11_TYSY
(c)
Amperage (mA)
Volume (mL)
Time (h)
C16_1N_TNSY
C16_11_TYSY
Solution injected

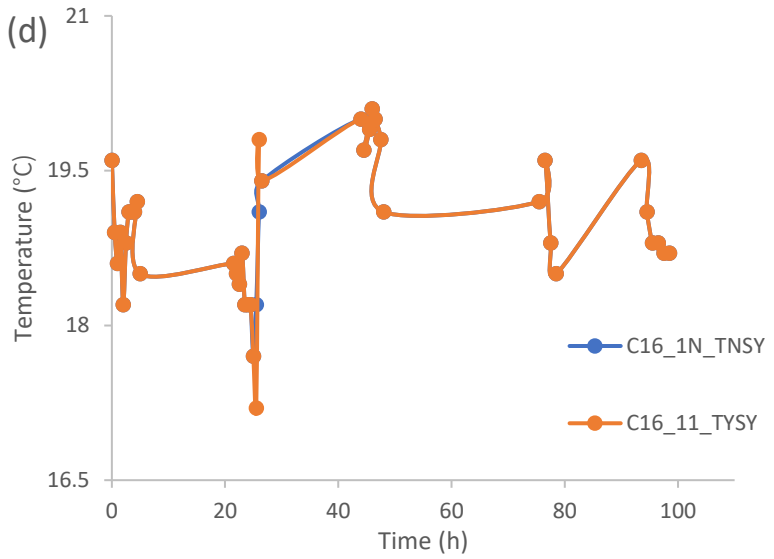


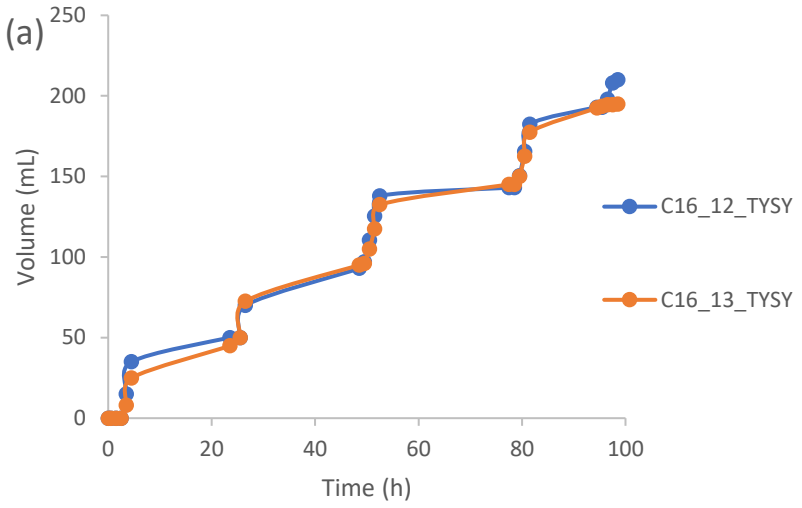
Figure 4.5 Phase 1 (Rounds 0 and 1) soil with 16% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

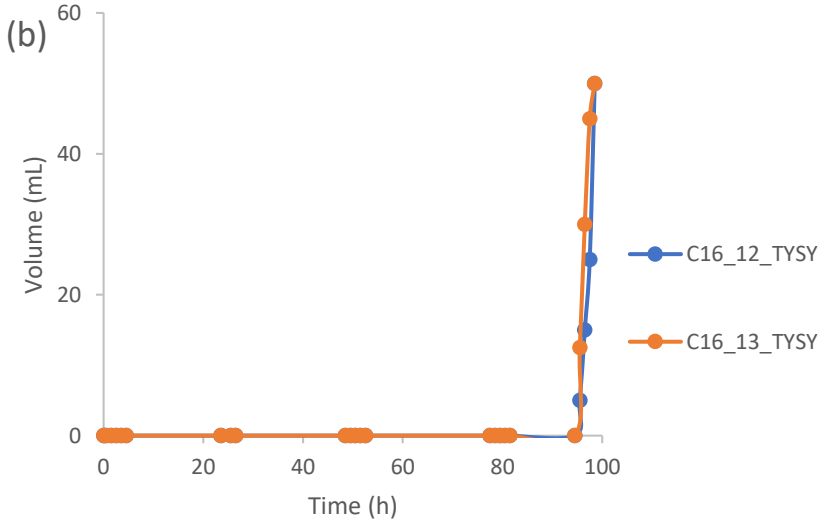
4.1.1.3.2 Second subgroup: round 2 and round 3

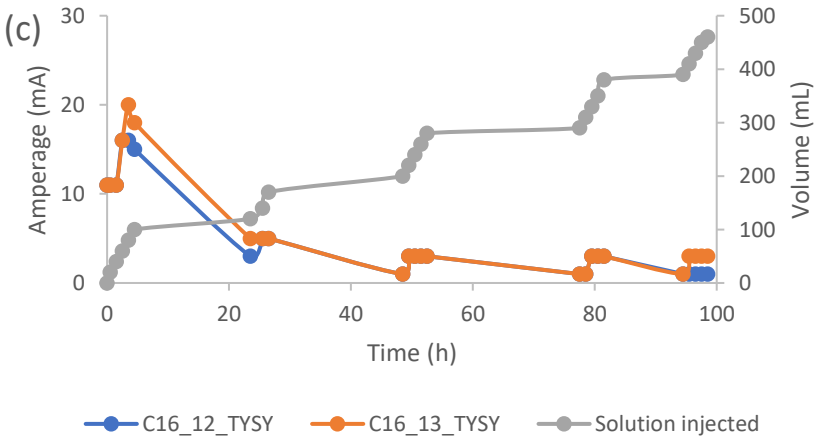
During the 98.5-hour duration of this scenario, 460 mL of surfactant solution was injected, as depicted in Figure 4.6.c. The temperature profiles exhibited similar behavior in both cells, ranging between 17.5 and 20 degrees Celsius.

In Round 2, the catholyte production reached 210 mL, accompanied by an anolyte production of 50 mL, as shown in Figures 4.6.a and 4.6.b. Similarly, in Round 3, 195 mL of catholyte and 50 mL of anolyte were produced, indicating the discharge behavior of the cells.

The amperage measurements during the first 24 hours of the test demonstrated approximately 5 mA higher value in Round 3 compared to Round 2, as illustrated in Figure 4.6.c. The maximum amperage reached 16 mA in Round 2 and 20 mA in Round 3. However, as time progressed, the amperage gradually decreased in both rounds and eventually stabilized at 1 mA.


(a)
Volume (mL)
250
200
150
100
50
0
0
20
40
60
80
100
Time (h)
C16_12_TYSY
C16_13_TYSY


(b)
Volume (mL)
60
40
20
0
0
20
40
60
80
100
Time (h)
C16_12_TYSY
C16_13_TYSY


(c)
Amperage (mA)
30
20
10
0
Volume (mL)
500
400
300
200
100
0
0
20
40
60
80
100
Time (h)
C16_12_TYSY
C16_13_TYSY
Solution injected

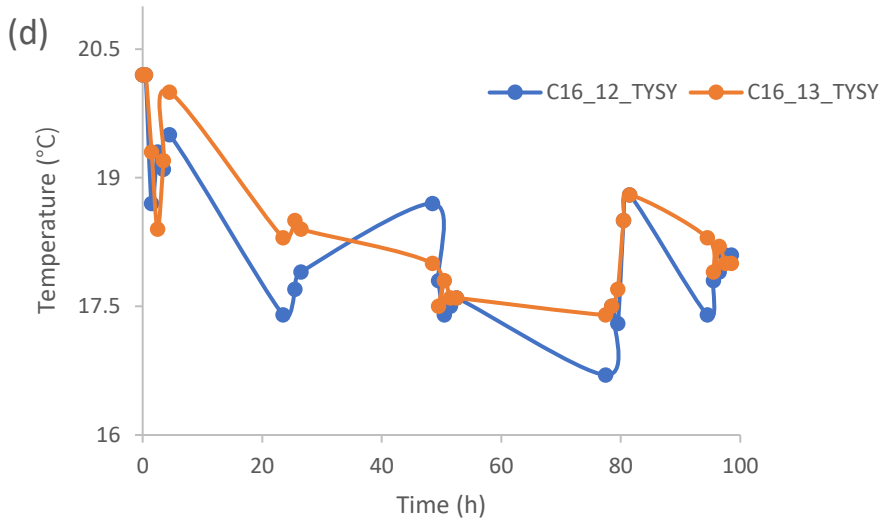


Figure 4.6 Phase 1 (Rounds 2 and 3) soil with 16% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

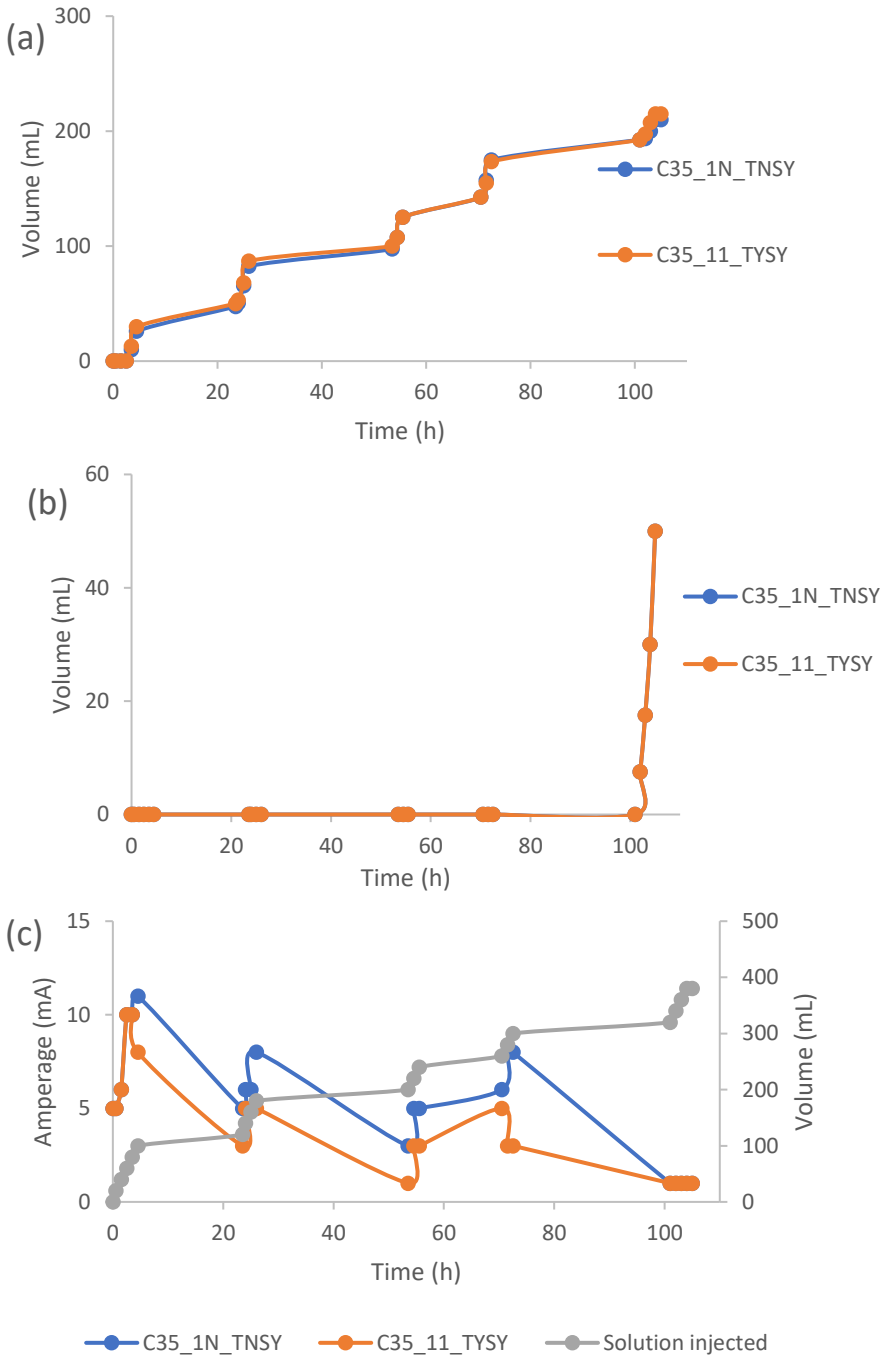
4.1.1.4 Organic matter content: 35%

4.1.1.4.1 First subgroup: Round N vs. Round 1

Figure 4.7 a provides a graphical representation of the discharge behavior of the catholyte and anolyte over a duration of 105 hours during the testing process. In the case of the non-polluted sample, a discharge volume of 210 mL of catholyte and 50 mL of anolyte was observed. For Round 1, the respective values were 215 mL for catholyte and 50 mL for anolyte, as illustrated in Figure 4.7 a-b. The total volume of injected solution for these cells amounted to 380 mL, as shown in Figure 4.7 c.

Figure 4.7 c presents the fluctuation of amperage throughout the testing period. Both samples exhibited a maximum amperage of +10 mA during the first day. Notably, the non-polluted sample displayed an amperage of 3 mA higher than that of Round 1. Over time, the amperages gradually decreased in both samples, stabilizing at 1 mA.

Furthermore, Figure 4.7.d provides an overview of the temperature profiles observed during the test, ranging from a minimum of 17.5 degrees Celsius to a maximum of 20.5 degrees Celsius.


(a)
Volume (mL)
300
200
100
0
0
20
40
60
80
100
Time (h)
C35_1N_TNSY
C35_11_TYSY
(b)
Volume (mL)
60
40
20
0
0
20
40
60
80
100
Time (h)
C35_1N_TNSY
C35_11_TYSY
(c)
Amperage (mA)
15
10
5
0
Volume (mL)
500
400
300
200
100
0
0
20
40
60
80
100
Time (h)
C35_1N_TNSY
C35_11_TYSY
Solution injected

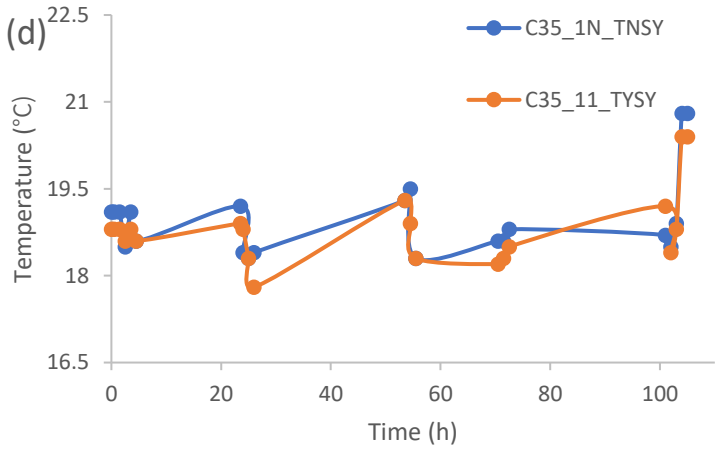


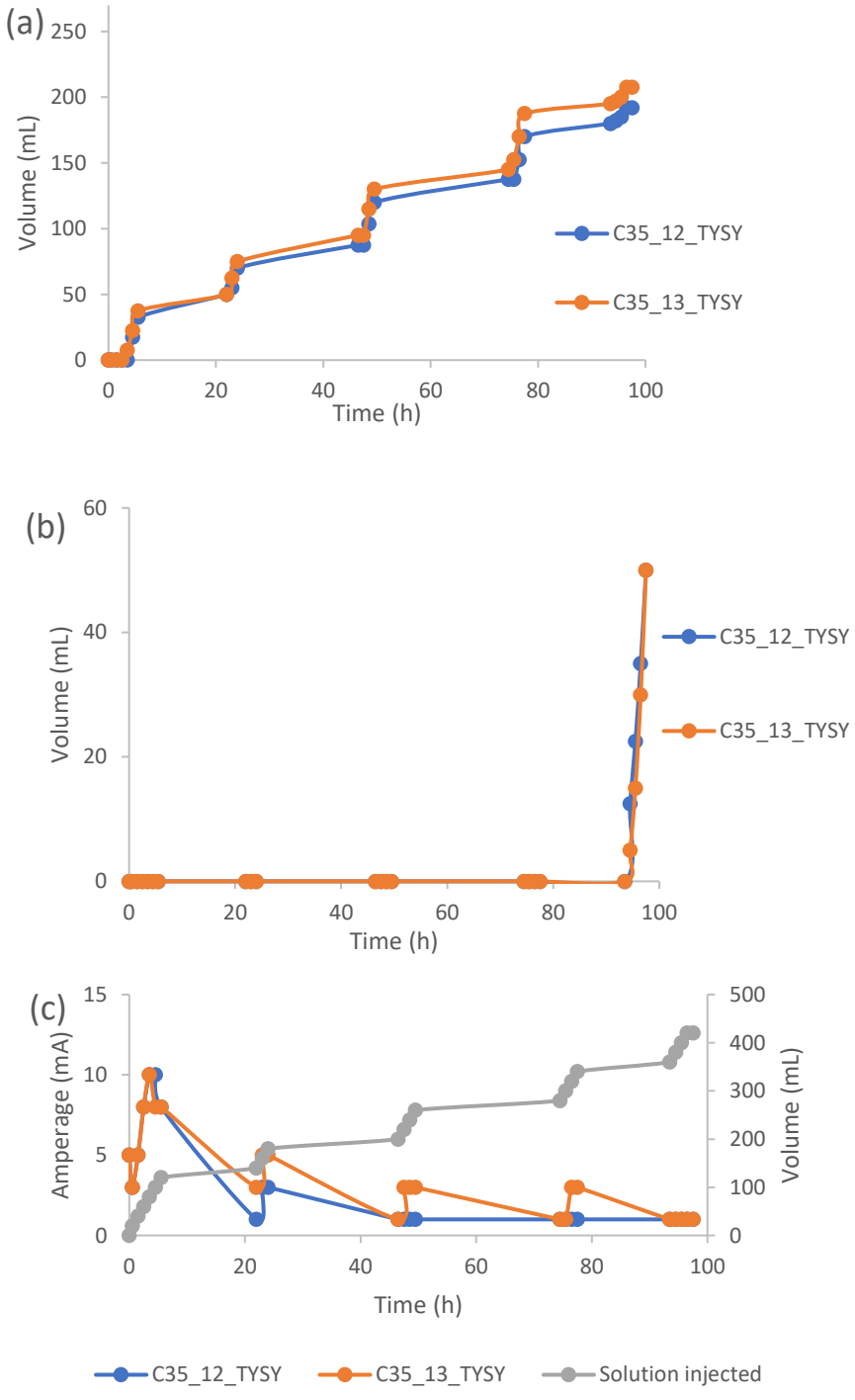
Figure 4.7 Phase 1 (Rounds 0 and 1) soil with 35% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.1.1.4.2 Second subgroup: round 2 and round 3

During the 97.5-hour test duration, 420 mL of surfactant solution was injected, as depicted in Figure 4.8.c. The temperature remained within the range of 17.5 to 20 degrees Celsius, as observed throughout the test.

In Round 2, the catholyte discharge amounted to 192 mL, accompanied by an anolyte discharge of 50 mL, as illustrated in Figures 4.8.a and 4.8.b. Similarly, in Round 3, 207.5 mL of catholyte and 50 mL of anolyte were produced, indicating the discharge behavior of the cells.

As shown in Figure 4.8.c, the amperage measurements reached a maximum value of 10 in both rounds. The amperage remained consistent throughout the test duration for both rounds.


(a)
Volume (mL)
Time (h)
C35_12_TYSY
C35_13_TYSY
(b)
Volume (mL)
Time (h)
C35_12_TYSY
C35_13_TYSY
(c)
Amperage (mA)
Volume (mL)
Time (h)
C35_12_TYSY
C35_13_TYSY
Solution injected

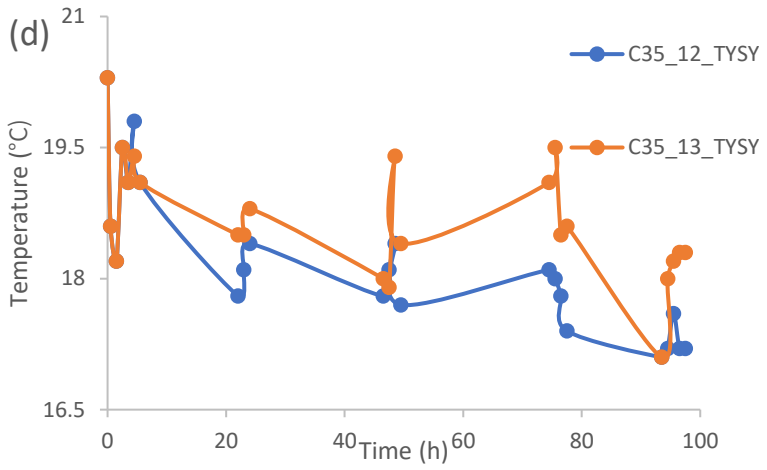


Figure 4.8 Phase 1 (Rounds 2 and 3) soil with 35% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.1.2 Toluene removal

4.1.2.1 Organic matter content: 0%

Table 4.1 displays the results from the sample analysis conducted using a Gas Chromatography (GC) system to examine the catholyte and anolyte samples over five consecutive days (Day 1 to Day 5), encompassing three experimental rounds. The initial day's sample demonstrated the highest observed toluene removal, averaging 11.95 parts per million (ppm). However, a sudden decline in toluene removal was noted in the subsequent days. Intriguingly, detectable toluene concentrations were absent in both the catholyte and anolyte samples on Day 4 and Day 5.

Table 4.1 Toluene concentration in discharged liquid at various time intervals in Phase 1 in 0 % organic matter samples

Type of sample	**Catholyte (ppm)**				**Anolyte (ppm)**
Discharge order	1	2	3	4	1
Round 1	11.87	2.13	0.64	0	0
Round 2	11.66	1.55	0.53	0	0
Round 3	12.33	2.88	0.70	0	0
Average	11.95	2.19	0.62	0	0

4.1.2.2 Organic matter content: 3%

The data presented in Table 4.2 demonstrates that the highest toluene removal, with an average concentration of 7.03 ppm, was achieved on the first analysis day. Subsequently, a gradual decrease in toluene removal was observed across the remaining days. Moreover, the average concentration of toluene in the anolyte samples was 0.77 ppm.

Table 4.2 Toluene concentration in discharged liquid at various time intervals in Phase 1 in 3% organic matter samples

Type of sample	**Catholyte (ppm)**				**Anolyte (ppm)**
Discharge order	1	2	3	4	1
Round 1	7.72	3.11	2.05	0.12	1.04
Round 2	5.34	3.04	1.58	0.11	1.27
Round 3	8.04	3.23	2.41	0.12	0
Average	7.03	3.13	2.02	0.11	0.77

4.1.2.3 Organic matter content: 16%

Upon closer examination of Table 4.3, it becomes apparent that there is no significant difference in toluene removal between the first and second days. Instead, a relatively consistent toluene removal trend is observed throughout the scenario outlined in the table. This consistency is evident in all three rounds, as each round shows an anolyte sample with a toluene concentration of 0.42 ppm.

Table 4.3 Toluene concentration in discharged liquid at various time intervals in Phase 1 in 16% organic matter samples

Type of sample	**Catholyte (ppm)**				**Anolyte (ppm)**
Discharge order	1	2	3	4	1
Round 1	4.36	3.12	1.32	1.29	0.42
Round 2	5.04	2.51	0.97	1.03	0.42
Round 3	3.43	3.68	1.93	1.46	0.42
Average	4.28	3.10	1.41	1.26	0.42

4.1.2.4 Organic matter content: 35%

Unexpectedly, the second set of samples exhibited the highest toluene removal, with an average concentration of 1 ppm more than the first-day samples. Notably, a consistent and sustained toluene removal pattern was observed in the soil mixture characterized by a high distribution of organic matter. On average, the anolyte samples demonstrated a toluene concentration of 1 ppm (Table 4.4).

Table 4.4 Toluene concentration in discharged liquid at various time intervals in Phase 1 in 35% organic matter samples

Type of sample	**Catholyte (ppm)**					**Anolyte (ppm)**
Discharge order	1	2	3	4	5	1
Round 1	4.97	6.52	2.36	1.4	0.16	2.02
Round 2	6.9	3.1	2.94	1.21	0.19	0.94
Round 3	2.99	6.02	3.52	1.32	0.13	0.17
Average	4.95	5.23	2.94	1.31	0.16	1.04

4.1.3 Impact of organic matter

4.1.3.1 Organic matter content: 0%

Due to the absence of organic soil components in the soil mixture, it was anticipated that the samples would exhibit a basic pH. As expected, all samples, including the catholyte and anolyte, fell within the basic pH range. The pH values ranged from 11.97 to 8.33 for the various samples. Notably, the first anolyte sample from round 1 deviated from the trend and had a slightly lower pH of 6.77. This variation could be attributed to specific experimental conditions or factors unique to the sample.

Table 4.5 pH values during different time intervals in Phase 1 with 0% organic matter samples

Type of sample	Catholyte				Anolyte	
Discharge order	1	2	3	4	1	2
Round 1	9.9	9.45	10	9.6	6.77	8.88
Round 2	10.28	9.37	10.47	10.14	8.48	-
Round 3	11.97	10.76	9.97	9.9	8.33	-
Average	10.72	9.86	10.15	9.88	7.86	8.88

4.1.3.2 Organic matter content: 3%

Table 4.6 provides an overview of the pH measurements obtained from the samples. One notable observation is the significant pH difference of approximately 2 units between the catholyte and anolyte samples. This difference can be attributed to the presence of peat, which is an organic matter, in the soil mixture.

Table 4.6 pH values during different time intervals in Phase 1 with 3% organic matter samples

Type of sample	Catholyte						Anolyte	
Discharge order	1	2	3	4	5	6	1	2
Round 1	9.76	11.25	10.39	8.04	8.44	7.92	7.51	7.62
Round 2	8.41	8.91	8.78	9.05	9.17	-	7.9	-
Round 3	11.62	12.02	9.25	10.5	-	-	8.35	-
Average	9.93	10.73	9.47	9.20	8.81	7.92	7.92	7.62

4.1.3.3 Organic matter content: 16%

According to the data presented in Table 4.7, all the samples exhibited a basic nature initially. Over time, the catholyte samples showed an increase in pH, indicating a shift towards a more alkaline environment. On average, the anolyte samples maintained a pH close to neutral, with an average pH of 7.45.

Table 4.7 pH values during different time intervals in Phase 1 with 16% organic matter samples

Type of sample	Catholyte							Anolyte	
Discharge order	1	2	3	4	5	6	7	1	2
Round 1	10.25	11.26	10.9	11.26	10.68	10.9	10.6	6.9	7.43
Round 2	7.59	7.82	7.9	8.05	8.36	-	-	7.78	-
Round 3	7.45	7.73	11.33	11.72	-	-	-	7.84	-
Average	8.43	8.94	10.04	10.34	9.52	10.9	10.6	7.51	7.43

4.1.3.4 Organic matter content: 35%

The catholyte samples exhibited a basic pH range from 7.31 to 11.81. Conversely, the anolyte samples had an average pH of 5.99. The significant pH gradient, observed between the catholyte and anolyte indicates organic acids' presence and subsequent removal.

Table 4.8 pH values during different time intervals in Phase 1 with 35% organic matter samples

Type of sample	Catholyte					Anolyte
Discharge order	1	2	3	4	5	1
Round 1	7.31	7.6	8.73	8.78	8.82	6.19
Round 2	8.8	9.01	8.16	8.69	8.42	5.09
Round 3	8.56	7.76	11.57	11.81	9.37	6.68
Average	8.22	8.12	9.49	9.76	8.87	5.99

4.2 Electrokinetic treatment under cold temperature

This section presents and evaluates the results of Phase 2, wherein the cells were placed in an environmental chamber set at 7 degrees Celsius.

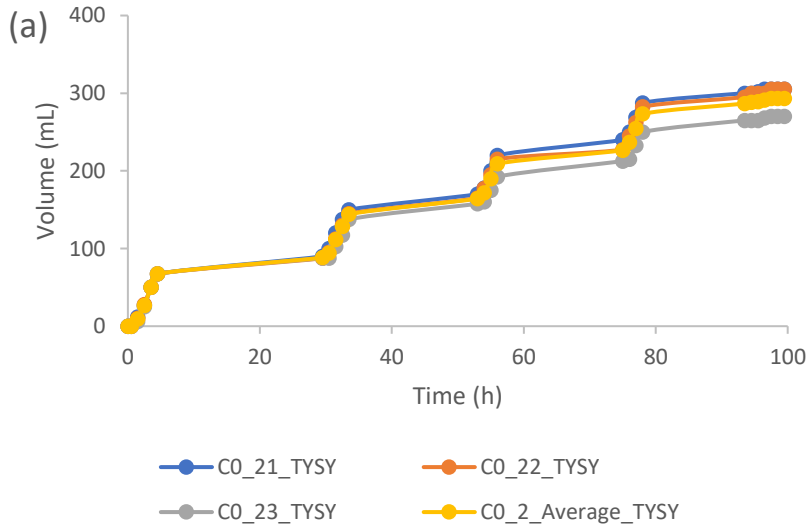
4.2.1 Pollutant extraction rate in the cold temperature

4.2.1.1 Organic matter content: 0%

In Figure 4.9.a, the cumulative generated catholyte volumes for Rounds 1, 2, and 3 were 305 mL, 305 mL, and 270 mL, respectively. Similarly, in Figure 4.9.b, the cumulative generated anolyte volumes were 100 mL, 97.5 mL, and 100 mL for Rounds 1, 2, and 3, respectively.

During the 99.5-hour test duration (Figure 4.9.c), 480 mL of solvent was added. Notably, the temperature experienced a sudden decrease from 20 degrees Celsius to 3 degrees Celsius, remaining stable throughout the test (Fig 4.9.d).

The amperage values exhibited a maximum of 18 in all three rounds, as illustrated in Figure 4.9.c. Moreover, the amperage trends were similar across all three rounds, indicating consistent behavior throughout the testing process.



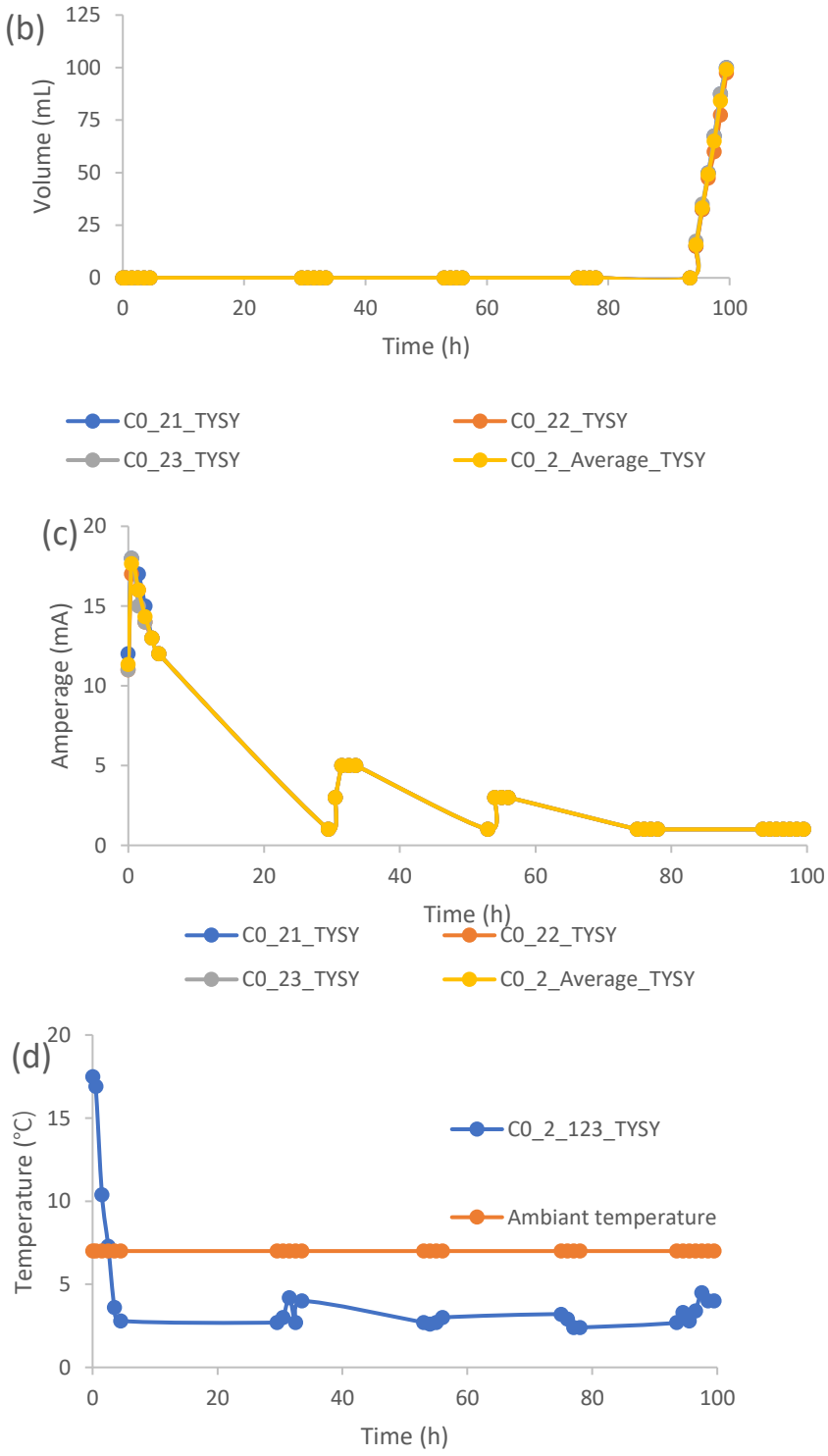


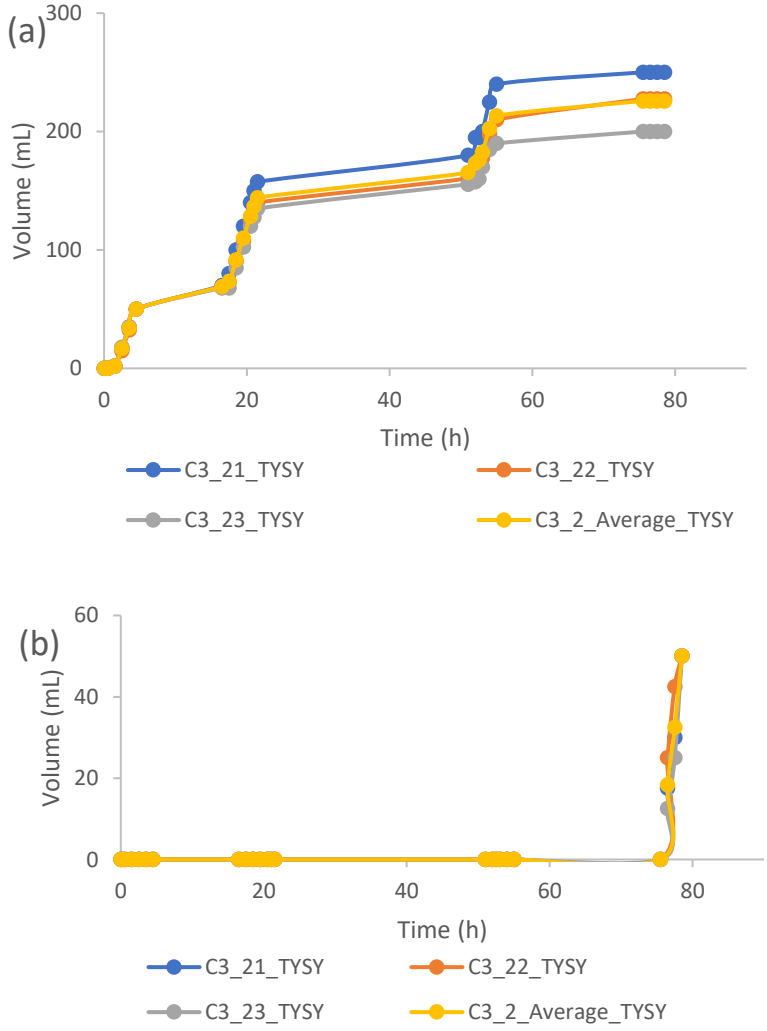
Figure 4.9 Phase 2 (all rounds) soil without organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.2.1.2 Organic matter content: 3%

Figure 4.10.a. presents the cumulative generated catholyte volumes for Rounds 1, 2, and 3, which amounted to 250 mL, 227.5 mL, and 200 mL, respectively. Similarly, Figure 4.10.b showcases the cumulative generated anolyte volumes of 50 mL for all three rounds.

Throughout the 78.5-hour test duration, 420 mL of solvent was added, as depicted in Figure 4.10.c. During this time, the temperature suddenly decreased from 20 degrees Celsius to 3 and remained stable throughout the test, as shown in Figure 4.10.d.

The amperage values reached a maximum of 18 to 23 mA in all three rounds, as observed in Figure 4.10.c. It is worth noting that the amperage trends exhibited similar patterns across all three rounds, indicating consistent behavior throughout the testing process.



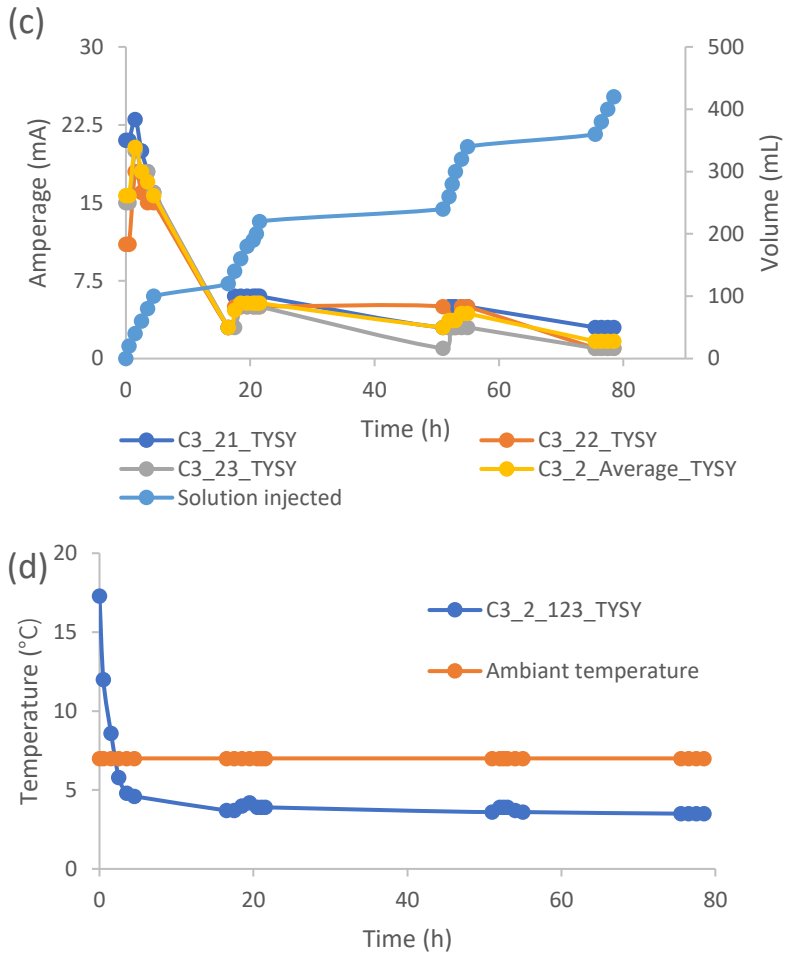


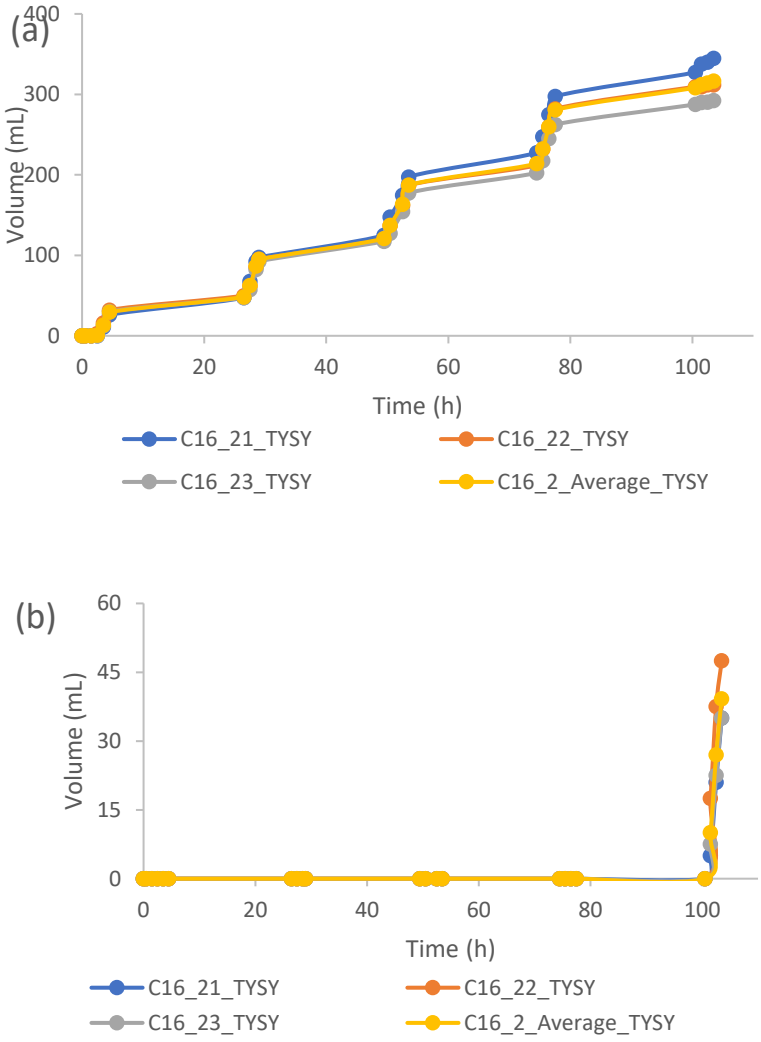
Figure 4.10 Phase 2 (all rounds) soil with 3% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.2.1.3 Organic matter content: 16%

Figure 4.11.a. displays the cumulative generated catholyte volumes for Rounds 1, 2, and 3, which amounted to 345 mL, 312 mL, and 292.5 mL, respectively. Similarly, Figure 4.11.b illustrates the cumulative generated anolyte volumes of 35 mL, 47.5 mL, and 35 mL for Rounds 1, 2, and 3, respectively.

Throughout the 103.5-hour test duration depicted in Figure 4.11.c, a total of 520 mL of solvent was added. It is worth noting that the temperature experienced a sudden decrease from 20 degrees Celsius to 3 degrees Celsius, and it remained stable throughout the test, as indicated in Figure 4.11.d.

The amperage values reached a maximum of 15 mA, as observed in Figure 4.11.c. Additionally, the amperage trends exhibited similar patterns across all three rounds, indicating consistent behavior throughout the testing process.



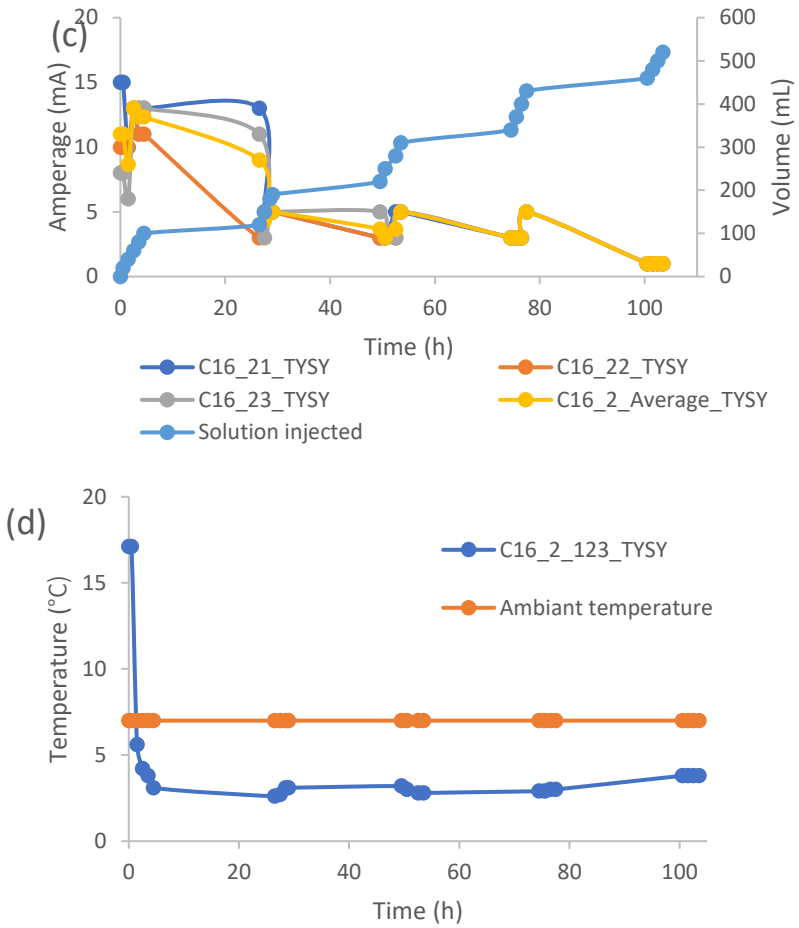


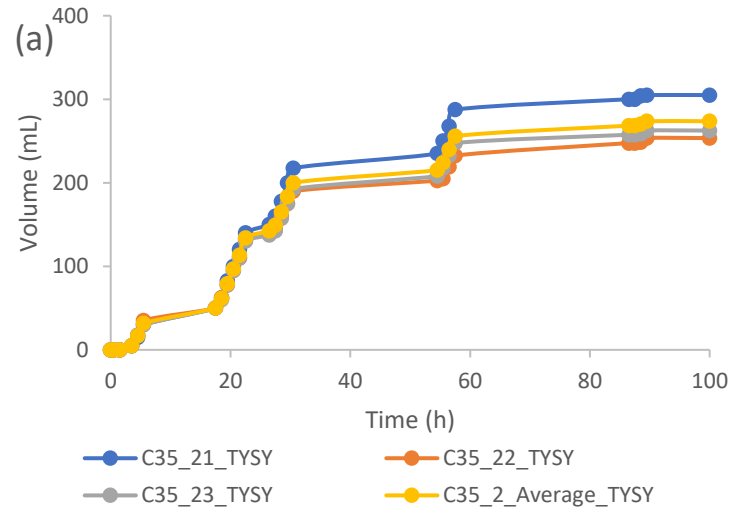
Figure 4.11 Phase 2 (all rounds) soil with 16% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

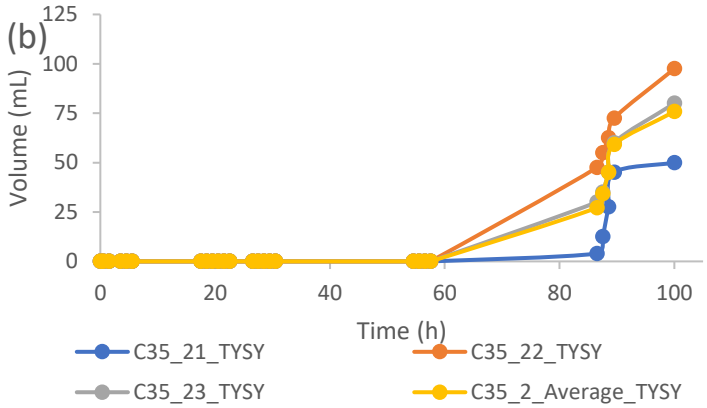
4.2.1.4 Organic matter content: 35%

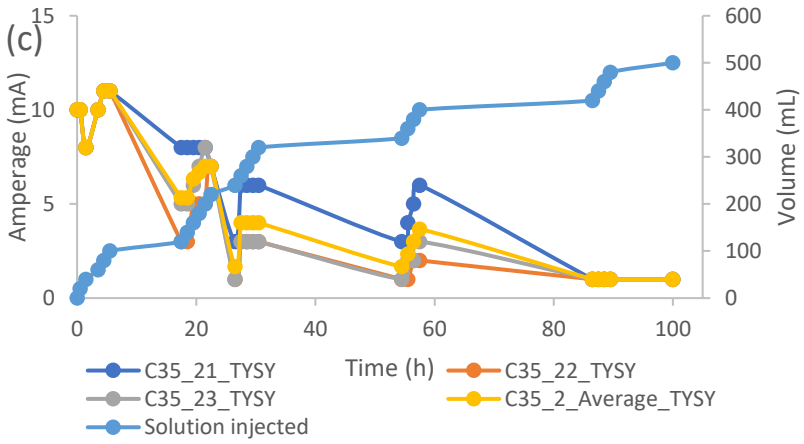
Figure 4.12.a. presents the cumulative generated catholyte volumes for Rounds 1, 2, and 3, which were recorded as 305 mL, 253.5 mL, and 262.5 mL, respectively. Similarly, Figure 4.12.b depicts the cumulative generated anolyte volumes of 50 mL for each round.

Throughout the 100-hour test duration displayed in Figure 4.12.c, a total of 500 mL of solvent was added. Notably, there was a sudden decrease in temperature from 20 degrees Celsius to 3 degrees Celsius, which remained constant throughout the test, as shown in Figure 4.12.d.

The amperage values reached a maximum of +10 mA, as illustrated in Figure 4.12.c. Additionally, it is noteworthy that by the end of the test, the amperage levels had converged to 1 mA for all three rounds.


(a)
Volume (mL)
Time (h)
C35_21_TYSY
C35_22_TYSY
C35_23_TYSY
C35_2_Average_TYSY


(b)
Volume (mL)
Time (h)
C35_21_TYSY
C35_22_TYSY
C35_23_TYSY
C35_2_Average_TYSY


(c)
Amperage (mA)
Volume (mL)
Time (h)
C35_21_TYSY
C35_22_TYSY
C35_23_TYSY
C35_2_Average_TYSY
Solution injected

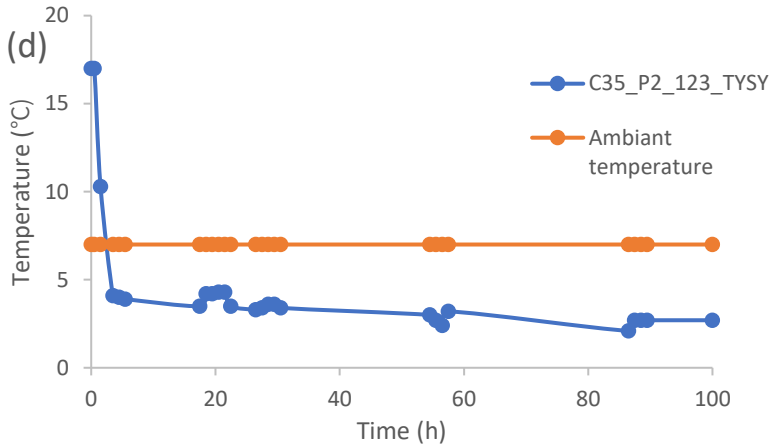


Figure 4.12 Phase 2 (all rounds) soil with 35% organic matter, a) Cumulative catholyte generation and b) Cumulative anolyte generation, c) Solution injection and Amperage, and d) Cell temperatures

4.2.2 Toluene removal

4.2.2.1 Organic matter content: 0%

The second sample displayed the highest toluene removal rate, averaging 8.31 ppm. Notably, the toluene concentration in the second anolyte sample was three times higher compared to the first sample. Overall, an average of 34 ppm toluene was successfully removed after 100 hours of treatment, according to Table 4.9.

Table 4.9 Toluene concentration in discharged liquid at various time intervals in Phase 2 in 0% organic matter samples

Type of sample	**Catholyte (ppm)**						**Anolyte (ppm)**	
Discharge order	1	2	3	4	5	6	1	2
Round 1	0.17	14.88	13.99	13.52	6.74	4.21	2.09	3.66
Round 2	5.87	4.19	3.54	2.55	2.00	1.34	1.19	5.93
Round 3	5.17	5.84	0	2.85	1.41	1.21	0	0
Average	3.73	8.31	5.85	6.30	3.39	2.26	1.09	3.19

4.2.2.2 Organic matter content: 3%

Throughout the test, an average toluene concentration of 35 ppm was collected. The first sample exhibited the highest toluene removal among all the samples. Regarding the anolyte samples, an average of approximately 3 ppm of toluene was collected from each sample.

Table 4.10 Toluene concentration in discharged liquid at various time intervals in Phase 2 in 3% organic matter samples

Type of sample	Catholyte (ppm)					Anolyte (ppm)
Discharge order	1	2	3	4	5	1
Round 1	16.70	14.24	12.91	11.88	3.01	7.71
Round 2	6.74	3.04	3.43	3.01	2.03	2.67
Round 3	8.52	2.55	2.02	0.87	3.67	1.07
Average	10.65	6.61	6.12	5.25	2.91	3.82

4.2.2.3 Organic matter content: 16%

Table 4.11 demonstrates a consistent and continuous toluene removal pattern. The highest average toluene removal, reaching 7 ppm, was observed in the first sample. Following the opening of the anode electrode, the anolyte samples contained approximately 1.5 to 2 ppm of toluene.

Table 4.11 Toluene concentration in discharged liquid at various time intervals in Phase 2 in 16% organic matter samples

Type of sample	Catholyte (ppm)							Anolyte (ppm)
Discharge order	1	2	3	4	5	6	7	1
Round 1	16.47	13.92	13.51	12.77	2.21	5.52	0.71	2.42
Round 2	4.77	2.67	3.45	0.04	2.76	4.42	0.84	1.41
Round 3	0.06	2.52	2.48	2.25	2.00	1.42	0.62	1.01
Average	7.10	6.37	6.48	5.02	2.33	3.79	0.72	1.61

4.2.2.4 Organic matter content: 35%

The second sample exhibited a notable peak in toluene removal, surpassing other samples (table 4.12). Throughout the test duration, this sample consistently demonstrated superior toluene

removal, outperforming the rest until the last day of the experiment. An average reduction of 90 ppm toluene was achieved after 100 hours of testing.

Table 4.12 Toluene concentration in discharged liquid at various time intervals in Phase 2 in 35% organic matter samples

Type of sample	**Catholyte (ppm)**						**Anolyte (ppm)**	
Discharge order	1	2	3	4	5	6	1	2
Round 1	3.17	20.09	18.39	20.84	23.05	14.86	5.04	0.73
Round 2	16.95	19.40	15.24	16.22	14.50	17.52	3.84	0.71
Round 3	7.09	14.34	14.00	4.92	5.46	7.42	2.94	0.69
Average	9.07	17.93	15.88	13.99	14.33	13.27	3.94	0.71

4.2.3 Impact of organic matter on pH distribution

4.2.3.1 Organic matter content: 0%

The data obtained from Table 4.13 indicates that all samples exhibited alkaline properties. The pH values decreased from a maximum of 10.51 to 9.84. Additionally, the anolyte samples had pH values above 7, indicating their alkaline nature. The relatively small gradient in pH suggests that there was limited removal of organic acids, which is expected considering the absence of organic matter in the soil mixture.

Table 4.13 pH values during different time intervals in Phase 2 with 0% organic matter samples

Type of sample	**Catholyte**						**Anolyte**	
Discharge order	1	2	3	4	5	6	1	2
Round 1	9.87	9.48	9.48	9.67	9.94	10.21	8.7	8.67
Round 2	11.39	11.57	10.86	10.56	9.76	10.57	8.57	8.6
Round 3	10.26	10.19	9.35	8.95	8.77	8.74	8.65	8.58
Average	10.51	10.41	9.90	9.73	9.49	9.84	8.69	8.48

4.2.3.2 Organic matter content: 3%

Table 4.14 presents pH measurements of samples containing 3% organic matter in the soil. All samples remained alkaline throughout the analysis. However, it is worth noting that the pH increased after the first day, indicating a slight shift towards a higher pH range. Although the pH gradient observed in this scenario was still insignificant, it was relatively higher than the samples with 0% organic matter in the soil. These results suggest that the presence of organic matter may have influenced the pH dynamics to some extent, albeit without a substantial impact on the overall alkaline nature of the samples.

Table 4.14 pH values during different time intervals in Phase 2 with 3% organic matter samples

Type of sample	**Catholyte**					**Anolyte**
Discharge order	1	2	3	4	5	1
Round 1	10.24	11.82	11.88	11.8	11.76	8.72
Round 2	8.75	10.56	10.39	11	9.03	8.24
Round 3	8.19	9.64	9.18	8.92	8.45	8.12
Average	9.06	10.67	10.49	10.57	9.75	8.36

4.2.3.3 Organic matter content: 16%

Based on the data presented in Table 4.15, the initial samples displayed a slightly basic nature with an average pH of 7.42. However, throughout the experiment, the pH increased and reached a higher value of 10.37. On the other hand, the anolyte samples exhibited a pH close to 7, indicating a weak basic nature.

Table 4.15 pH values during different time intervals in Phase 2 with 16% organic matter sample

Type of sample	Catholyte							Anolyte
Discharge order	1	2	3	4	5	6	7	1
Round 1	7.28	10.25	8.13	7.78	10.91	11.6	9.23	8.02
Round 2	7.48	10.62	10.9	10.23	10.17	11.1	9.02	7.98
Round 3	7.49	7.92	7.98	7.91	8.03	8.42	8.62	7.52
Average	7.42	9.60	9.00	8.64	9.70	10.37	8.96	7.84

4.2.3.4 Organic matter content: 35%

Intriguingly, the initial samples exhibited an unexpectedly acidic nature, with an average pH of 6.94. However, as time progressed, there was an increase in pH, reaching 10.21. Conversely, the anolyte samples maintained a pH of 5.49. The significant pH gradient observed in this scenario suggests organic acids' presence and subsequent removal.

Table 4.16 pH values during different time intervals in Phase 2 with 35% organic matter samples

Type of sample	Catholyte						Anolyte
Discharge order	1	2	3	4	5	6	1
Round 1	7.39	7.11	7.93	11.1	11.11	10.84	5.8
Round 2	6.61	8.91	6.97	11.18	10.39	10.13	5.97
Round 3	6.8	6.9	7.22	7.44	7.57	9.66	4.71
Average	6.94	7.64	7.37	9.91	9.69	10.21	5.49

4.3 Comparative analyses

The comparison of the results can be divided into two distinct subgroups. The first subgroup separately analyzes the results of analytical parameters, such as pH and GC analysis, for each phase. This comparison allows for examining differences and similarities between the two environments with respect to climatic conditions.

The second subgroup compares the results based on their behavior under different temperature conditions using the same soil matrix components. This comparison enables the assessment of how temperature variations affect the observed outcomes, providing insights into the temperature-dependent dynamics of the system.

4.3.1 Intra-Phase analysis

4.3.1.1 Phase 1 (temperate climate)

Table 4.17 presents the average results obtained from Phase 1, where several tests were conducted at an ambient temperature of 20°C. The table reveals interesting findings regarding the relationship between the soil mixture's initial organic matter content (peat) and the corresponding toluene removal rates. Samples with lower initial organic matter content exhibited higher toluene removal in their first sample. On the other hand, samples with higher organic matter content, such as the sample with 35% peat, still had detectable levels of toluene even after 100 hours, while the sample without any organic matter (0%) showed complete toluene removal by the 4th day.

Furthermore, samples with a higher organic matter content were observed to have a higher concentration of toluene in their anolyte discharges. This suggests a correlation between the presence of organic matter and the release of toluene into the anolyte.

Lastly, as indicated in the last column, the total toluene removal in ppm demonstrates that soils with a higher organic matter component resulted in greater overall toluene removal. The results imply that the electrokinetic cells exhibit improved performance when there is a higher presence of organic matter in the soil mixture.

Table 4.17 Comparison of toluene removal in different soil samples in Phase 1

Type of sample	**Catholyte (ppm)**					**Anolyte (ppm)**	**Total (ppm)**
Discharge order	1	2	3	4	5	1	
OM 0%	11.95	2.19	0.62	0	-	0	14.76
OM 3%	7.03	3.13	2.02	0.11	-	0.77	13.06
OM 16%	4.28	3.1	1.41	1.26	-	0.42	10.47
OM 35%	4.95	5.23	2.94	1.31	0.16	1.04	15.63

Table 4.18 provides a comparison of the acidity or alkalinity of the samples. It is observed that soil mixtures with a higher initial organic matter content produced liquid discharges with lower pH values than those with lower organic matter content. This can be attributed to the presence of organic acids in the soil, which contribute to the acidity of the liquid discharges.

Interestingly, all catholyte samples at room temperature were found to be basic, regardless of the organic matter content in the soil mixture. This suggests that the organic matter content less influences the catholyte pH and remains predominantly alkaline.

Furthermore, more organic matter in the soil mixture resulted in less basic anolyte discharges. This can be attributed to the release of organic acids from the soil, which lower the pH of the anolyte. Notably, the anolyte of the soil mixture with 35% organic matter exhibited a pH below 7, indicating an acidic nature.

The pH gradient observed across the samples suggests a potential removal of organic acids, such as humic acid or/and fulvic acid, which is more pronounced in soil mixtures with higher organic matter content. This suggests that the electrokinetic process contributes to the removal of these organic acids, leading to a decrease in the overall acidity of the samples.

Table 4.18 Comparison of pH values in different soil samples in Phase 1

Type of sample	**Catholyte**							**Anolyte**	
Discharge order	1	2	3	4	5	6	7	1	2
OM 0%	10.72	9.86	10.15	9.88	-	-	-	7.86	8.88
OM 3%	9.93	10.73	9.47	9.2	8.81	7.92	-	7.92	7.62
OM 16%	8.43	8.94	10.04	10.34	9.52	10.9	10.6	7.51	7.43
OM 35%	8.22	8.12	9.49	9.76	8.87	-	-	5.99	-

4.3.1.2 Phase 2 (cold climate)

Table 4.19 presents the average results of the analysis conducted by the GC system in Phase 2, which was carried out at a colder temperature of 7°C. Like the previous section, the influence of organic matter on toluene removal is observed, whereby a higher initial organic matter content in the soil leads to greater toluene removal.

Interestingly, it is observed that the second discharge generally exhibits high levels of toluene removal. In some cases, such as the samples with 0% and 35% organic matter, it represents the highest amount of toluene removed in the test. This can be attributed to stabilizing the electrokinetic cells and samples after the first day, allowing for more efficient toluene removal.

Furthermore, it is notable that samples with 0%, 3%, and 16% organic matter demonstrate very similar toluene removal by the end of the tests. However, the sample with 35% organic matter exhibits three times more toluene removal than the other samples.

Table 4.19 Comparison of toluene removal in different soil samples in Phase 2

Type of sample	**Catholyte (ppm)**				**Anolyte (ppm)**			**Total (ppm)**		
Discharge order	1	2	3	4	5	6	7	1	2	
OM 0%	3.73	8.31	5.85	6.3	3.39	2.26	-	1.09	3.19	34.12
OM 3%	10.65	6.61	6.12	5.25	2.91	-	-	3.82	-	35.36
OM 16%	7.1	6.37	6.48	5.02	2.33	3.79	0.72	1.61	-	33.42
OM 35%	9.07	17.93	15.88	13.99	14.33	13.27	-	3.94	0.71	89.12

Table 4.20 presents the average pH data for the samples in the 2nd phase of the test. Similar to the previous phase, a consistent trend is observed, where samples with a higher initial organic matter content in the soil mixture exhibit less alkaline pH values.

Notably, all samples with 0%, 3%, and 16% organic matter content remain basic. However, the pH values for the samples with 35% and 16% organic matter content are less basic and, in some cases, even acidic in some cases. This indicates that a higher organic matter content in the soil mixture leads to a decrease in pH, potentially due to the release of organic acids.

Furthermore, a greater pH gradient is observed in the samples with organic soil mixtures, indicating a higher level of organic acid potential removal. The electrokinetic process contributes to removing these organic acids, leading to a decrease in the overall alkalinity of the samples.

Table 4.20 Comparison of pH values in different soil samples in Phase 2

Type of sample	Catholyte							Anolyte	
Discharge order	1	2	3	4	5	6	7	1	2
OM 0%	10.51	10.41	9.9	9.73	9.49	9.84	-	8.69	8.48
OM 3%	9.06	10.67	10.49	10.57	9.75	-	-	8.36	-
OM 16%	7.42	9.6	9	8.64	9.7	10.37	8.96	7.84	-
OM 35%	6.94	7.64	7.37	9.91	9.69	10.21	-	5.49	-

4.3.2 Temperature-dependent analysis

This section focuses on discussing and comparing the results of the effects of varying operational temperatures.

4.3.2.1 Organic matter content: 0%

Table 4.21 presents a comparison of toluene removal from a soil mixture in the absence of any organic matter. The results indicate that the removal of toluene occurred at a faster rate during Phase 1, which involved room temperature conditions. However, more continuous toluene removal was observed in Phase 2, characterized by colder temperatures.

Furthermore, it is worth noting that after 100 hours, no toluene was detected in the anolyte in Phase 1. In contrast, toluene removal was observed in the anolytes of Phase 2.

Overall, the data reveals that the extent of toluene removal was more than twice as high in Phase 2 compared to Phase 1.

Table 4.21 Comparison of toluene removal in Phase 1 and 2: Soil with 0% organic matter

Type of sample	Catholyte (ppm)						Anolyte (ppm)		Total (ppm)
Discharge order	1	2	3	4	5	6	1	2	
Phase 1	11.95	2.19	0.62	0	-	-	0	-	14.76
Phase 2	3.73	8.31	5.85	6.3	3.39	2.26	1.09	3.19	34.12

Table 4.22 displays the pH values of samples from two phases. The data indicates that there is no significant difference in pH between the two phases, as both exhibit a similar trend in pH variation.

Table 4.22 Comparison of pH values in Phases 1 and 2: Soil with 0% organic matter

Type of sample	**Catholyte**						**Anolyte**	
Discharge order	1	2	3	4	5	6	1	2
Phase 1	10.72	9.86	10.15	9.88	-	-	7.86	8.88
Phase 2	10.51	10.41	9.9	9.73	9.49	9.84	8.69	8.48

4.3.2.2 Organic matter content: 3%

Table 4.23 presents the results obtained from the GC system for both phases of the soil sample tests, specifically for samples containing 3% organic matter. The data reveals that colder temperatures resulted in a longer process for removing toluene. Additionally, Phase 2 exhibited a significantly higher toluene removal than Phase 1, with approximately three times more toluene removed.

On the 5th day of the test in Phase 1, a toluene concentration of 0.77 ppm was observed in the anolyte sample. In contrast, in Phase 2, the anolyte exhibited a significantly higher toluene concentration, measuring 3.82 ppm, approximately five times higher than in Phase 1.

Table 4.23 Comparison of toluene removal in Phase 1 and 2: Soil with 3% organic matter

Type of sample	**Catholyte (ppm)**					**Anolyte (ppm)**	**Total (ppm)**
Discharge order	1	2	3	4	5	1	
Phase 1	7.03	3.13	2.02	0.11	-	0.77	13.06
Phase 2	10.65	6.61	6.12	5.25	2.91	3.82	35.36

The data reveals a mixed behavior based on the results presented in Table 4.24 for the test conducted on soil samples containing 3% organic matter. However, all the samples exhibited a basic behavior. This suggests that the pH values of the samples generally leaned towards the alkaline side, although there might have been some variations or inconsistencies in the specific pH values observed.

Table 4.24 Comparison of pH values in Phases 1 and 2: Soil with 3% organic matter

Type of sample	Catholyte						Anolyte	
Discharge order	1	2	3	4	5	6	1	2
Phase 1	9.93	10.73	9.47	9.2	8.81	7.92	7.92	7.62
Phase 2	9.06	10.67	10.49	10.57	9.75	-	8.36	-

4.3.2.3 Organic matter content: 16%

Table 4.25 compares toluene concentrations in samples containing 16% organic matter under cold and ambient conditions. The results indicate that the colder temperature led to a threefold increase in toluene removal compared to ambient temperature conditions.

Like the previous mixtures with lower organic matter content, the data demonstrate that a longer duration was required for toluene removal in the cold temperature setting. This suggests that lower temperatures may contribute to a more prolonged toluene degradation or removal process.

Table 4.25 Comparison of toluene removal in Phase 1 and 2: Soil with 16% organic matter

Type of sample	Catholyte (ppm)						Anolyte (ppm)		Total (ppm)
Discharge order	1	2	3	4	5	6	7	1	
Phase 1	4.28	3.1	1.41	1.26	-	-	-	0.42	10.47
Phase 2	7.1	6.37	6.48	5.02	2.33	3.79	0.72	1.61	33.42

According to Table 4.26, there is a notable similarity observed in most cases. However, on average, the samples from Phase 2 exhibited a slightly lower alkalinity level than those from Phase 1. This indicates that, on average, the pH values of the samples in Phase 2 were slightly less basic than those in Phase 1.

Table 4.26 Comparison of pH values in Phases 1 and 2: Soil with 16% organic matter

Type of sample	Catholyte							Anolyte	
Discharge order	1	2	3	4	5	6	7	1	2
Phase 1	8.43	8.94	10.04	10.34	9.52	10.9	10.6	7.51	7.43
Phase 2	7.42	9.6	9	8.64	9.7	10.37	8.96	7.84	-

4.3.2.4 Organic matter content: 35%

Based on the findings in Table 4.27, the test conducted with a soil mixture containing 35% organic matter demonstrates a significant increase in toluene removal, approximately six times higher than previous scenarios. Furthermore, the data indicate a decreasing trend in the toluene removal process at room temperature after the initial sample. In contrast, a higher amount of toluene was removed in the cold temperature setting compared to the initial sample, suggesting a more effective removal process under colder conditions.

Table 4.27 Comparison of toluene removal in Phase 1 and 2: Soil with 35% organic matter

Type of sample	Catholyte (ppm)						Anolyte (ppm)		Total (ppm)
Discharge order	1	2	3	4	5	6	1	2	Total
Phase 1	4.95	5.23	2.94	1.31	0.16	-	1.04	-	15.63
Phase 2	9.07	17.93	15.88	13.99	14.33	13.27	3.94	0.71	89.12

Table 4.28 reveals that both phases resulted in the production of acidic anolyte. Furthermore, the pH gradient observed in the samples is significant. Furthermore, Phase 2 exhibited a larger pH gradient than Phase 1, indicating a more pronounced removal of organic acids under colder temperatures. This suggests that the colder temperature facilitated a more effective removal of organic acids, leading to a greater pH reduction in Phase 2.

Table 4.28 Comparison of pH values in Phases 1 and 2: Soil with 35% organic matter

Type of sample	**Catholyte**						**Anolyte**
Discharge order	1	2	3	4	5	6	1
Phase 1	8.22	8.12	9.49	9.76	8.87	-	5.99
Phase 2	6.94	7.64	7.37	9.91	9.69	10.21	5.49

5 Conclusion, Contribution, Future Work

5.1 Conclusion

1) Four phases of this study permitted to better understand the applicability of electrokinetic organic soil remediation in cold regions, encompassing Canadian northern territories, permafrost lands, boreal forests, and wetlands, all of which exhibit the presence of organic soil.
2) The results of each phase showed that a higher fraction of organic matter in the soil mixture led to better electrokinetic toluene removal in spite of a high potential sorption of the organic pollutant to soil organic fraction.
3) Phase 1, representing temperate climate conditions, demonstrated that soils with higher organic matter content retained toluene in their matrix for a longer period compared to clayey matrix only, which exhibited a faster rate of toluene removal.
4) A comparison between Phase 1 and Phase 2 (representing cold climate conditions) revealed that colder temperatures had a higher toluene removal. For soil matrices containing 0%, 3%, and 16% of organic matter, three times more toluene was removed at cold temperatures. Toluene removal was six times higher in the soil with 35% organic matter compared to temperate temperature conditions.
5) The results also demonstrated that the initial discharges from organic soils had lower pH values than those from clayey soil alone, showing attenuation behavior of organic soil.
6) Potential dissolution and discharge of organic soils fractions such as humic and/or fulvic acids was also observed contrary to clayey soil matrix only.
7) The temperature did not significantly affect the humic fractions removal in samples with 0%, 3%, and 16% of organic matter.
8) However, in the sample with 35% organic matter content, a more significant pH gradient was observed at colder temperatures, indicating probably an increase in organic acids dissolution and removal.
9) This study initiated comprehensive investigation of soil organic fraction response to electrokinetic phenomena, and opened the door to further application of electrokinetic remediation of polluted soil in temperate and cold climates in Canada.
10) Due to the shared physicochemical attributes, such as log Koc and log Kow, between toluene and the other constituents of the BTEX group, this work has the potential to offer applicability to a wider range of BTEX compounds in addition to toluene.

11) The study confirmed feasibility to apply the electrokinetic remediation to organic soils in cold regions during a short summer period in-situ and ex-situ during remining periods, which gives an advantage over other remediation technologies.

5.2 Contribution

This thesis significantly contributes to the elektrokinetics (EK) field and soil remediation. Firstly, it successfully applies EK to organic soil, expanding the technique's scope and providing insights into organic soil behavior during remediation. Additionally, the study reveals the impact of organic components on EK remediation, enhancing our understanding of their influence and informing tailored strategies for organic-rich soils. Furthermore, the research demonstrates the effectiveness of EK in cold regions, offering a viable solution for contaminated sites in challenging climates. These contributions advance the knowledge and application of EK, benefiting soil remediation practices and opening new possibilities for environmental cleanup.

5.3 Future works

Future research directions in electrokinetic soil remediation encompass the following aspects. Firstly, the investigations can focus on determining the feasibility of implementing electrokinetic organic soil remediation on a larger scale.

Secondly, it is recommended to focus research efforts on developing and optimizing a suitable the injection system for conditioners. By refining the injection methodology, the efficiency and effectiveness of the electrokinetic remediation process could be enhanced. This involves refining contaminant distribution, improving electroosmotic flow patterns, and maximizing removal rates, thus pushing the boundaries of electrokinetic soil remediation.

Additionally, it is imperative to expand the research scope beyond removing toluene and address other pollutants belonging to the benzene, toluene, ethylbenzene, and xylenes (BTEX) group as well as polyaromatic hydrocarbons (PAHs). Considering the removal of crude oil contaminants in conjunction with the targeted toluene removal is essential.

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Appendices

Appendix A: Comprehensive datasheet for the tests

Phase 1

pH for samples without toluene

Table A.1 pH values in different not-polluted samples in Phase 1

Type of sample	Catholyte							Anolyte	
Discharge order	1	2	3	4	5	6	7	1	2
OM 0%	10.7	10.63	9.1	9.28	-	-	-	8.47	8.28
OM 3%	8.03	10.6	9.21	10.79	9.18	-	-	8.51	8.5
OM 16%	9.25	7.9	7.8	10.1	8.26	9.69	10.04	8.25	8.33
OM 35%	7.86	7.65	8.44	7.92	11.3	-	-	8.01	-

Pollutant extraction datasheet

Organic matter content: 0%
Not polluted round

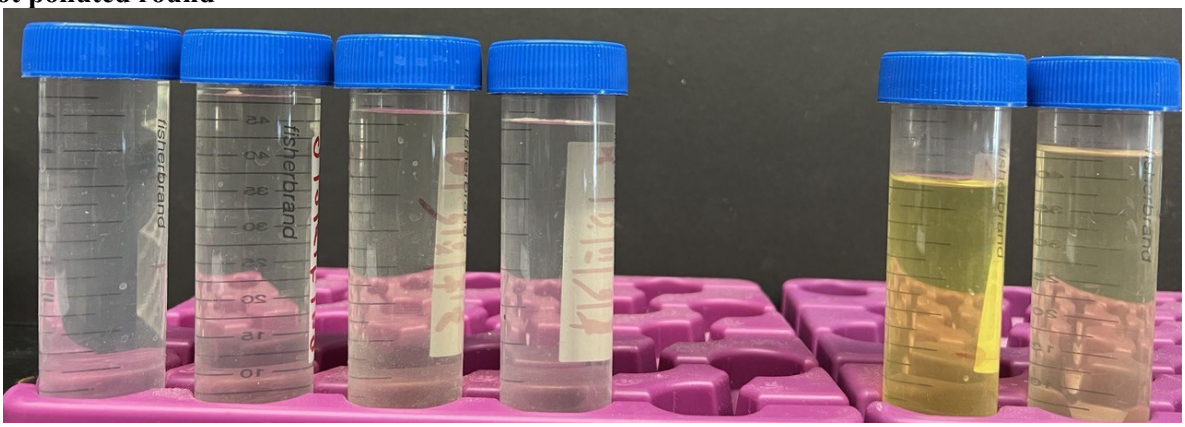


Figure A.1 Catholytes and anolytes (in order from left to right) for C0_1N_TNSY

Table A.2 Comprehensive datasheet for C0_1N_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	50	19.8	0	0	0
0.5	70	18.7	20	0	0
1.5	90	18.7	25	0	17.5
2.5	110	18.5	26	0	37.5
23	130	17.4	3	0	50
24	150	17.6	6	0	54
25	170	17.9	6	0	72.5
26	190	16.7	5	0	85
51	210	17.8	1	0	100
52	230	17.8	1	0	104
53	250	18.3	3	0	115
54	270	18.2	3	0	132.5
80	290	18.3	1	0	145
81	310	18.6	3	0	147
82	330	17.9	3	0	162.5
83	330	18.3	3	0	180
95	350	17.9	1	0	180
96	370	18.6	3	17.5	180
97	380	17.7	3	35	182.5
97.5	400	17.8	3	40	184
98.5	420	18.7	3	55	187.5
99.5	440	18.7	2	72.5	190

Round 1

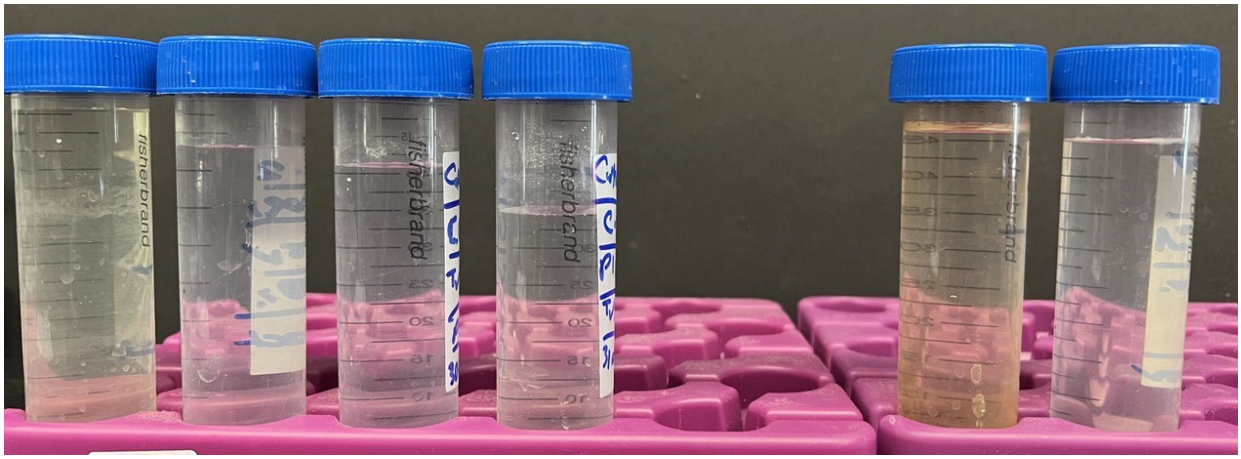


Figure A.2 Catholytes and anolytes (in order from left to right) for C0_11_TYSY

Table A.3 Comprehensive datasheet for C0_11_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	50	19.8	0	0	0
0.5	70	18.7	20	0	0
1.5	90	18.8	26	0	15
2.5	110	17.8	25	0	34
23	130	16.9	3	0	50
24	150	15.9	5	0	54
25	170	17.1	5	0	69
26	190	16.7	5	0	90
51	210	17.8	1	0	93
52	230	17.8	1	0	97
53	250	18.3	5	0	103
54	270	18.2	5	0	118
80	290	18.3	1	0	135
81	310	18.6	3	0	137
82	330	17.9	3	0	150
83	330	18.3	3	0	167.5
95	350	17.9	1	0	167.5
96	370	18.6	3	22.5	167.5
97	380	17.7	3	37.5	167.5
97.5	400	17.8	3	42.5	170
98.5	420	18.7	3	57.5	170
99.5	440	18.7	2	77.5	170

Round 2

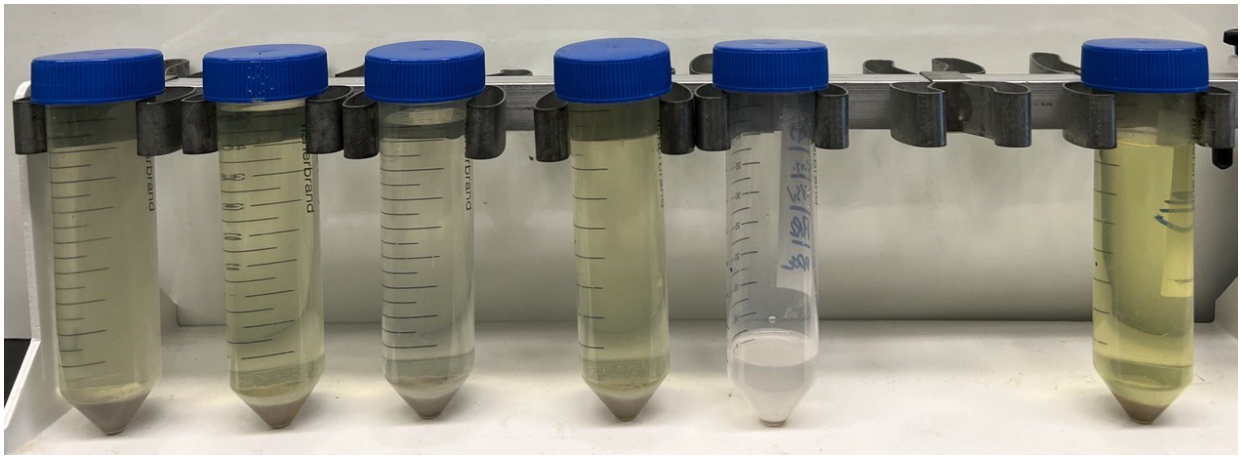


Figure A.3 Catholytes and anolyte (in order from left to right) for C0_12_TYSY

Table A.4 Comprehensive datasheet for C0_12_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.2	11	0	0
0.5	20	19.2	11	0	0
1.5	40	19.1	21	0	7.5
2.5	60	19.3	20	0	25
3.5	80	18.9	20	0	45
22.5	90	18.3	1	0	50
23.5	110	18.4	3	0	55
24.5	130	18.4	3	0	72
25.5	150	18	3	0	90
54	160	18.1	1	0	100
55	180	18.2	1	0	104
56	200	17.8	3	0	120
57	220	17.8	3	0	136
71	230	17.2	1	0	145
72	250	17.7	1	0	155
73	270	17.4	1	0	172
74	290	17.8	1	0	190
93.5	300	17.3	1	0	195
94.5	320	17.7	1	5	195
95.5	350	18.2	1	27.5	200
96.5	380	18.2	1	50	200

Round 3

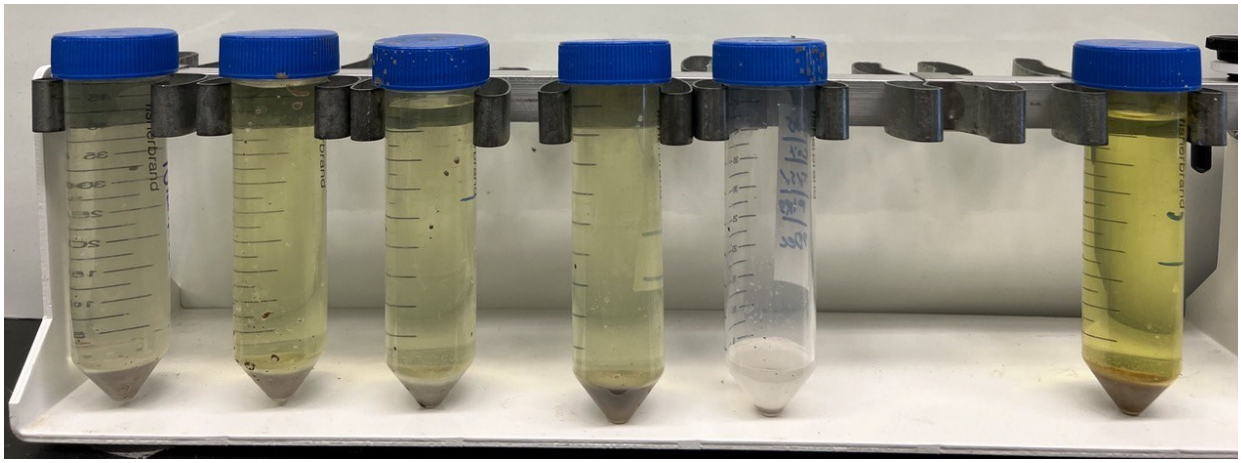


Figure A.4 Catholytes and anolyte (in order from left to right) for C0_13_TYSY

Table A.5 Comprehensive datasheet for C0_13_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.2	13	0	0
0.5	20	19.2	13	0	0
1.5	40	19.5	25	0	7.5
2.5	60	19.2	21	0	22.5
3.5	80	19.1	20	0	42.5
22.5	90	18.2	1	0	50
23.5	110	18.4	5	0	55
24.5	130	18.1	5	0	72
25.5	150	18	5	0	90
54	160	18.1	1	0	100
55	180	18.2	1	0	103
56	200	17.8	3	0	120
57	220	17.8	3	0	136
71	230	17.2	1	0	145
72	250	17.7	3	0	155
73	270	17.4	3	0	172
74	290	17.8	3	0	190
93.5	300	17.3	1	0	195
94.5	320	17.7	1	5	195
95.5	350	18.2	1	30	200
96.5	380	18.2	1	50	200

Organic matter content: 3%

Not polluted round

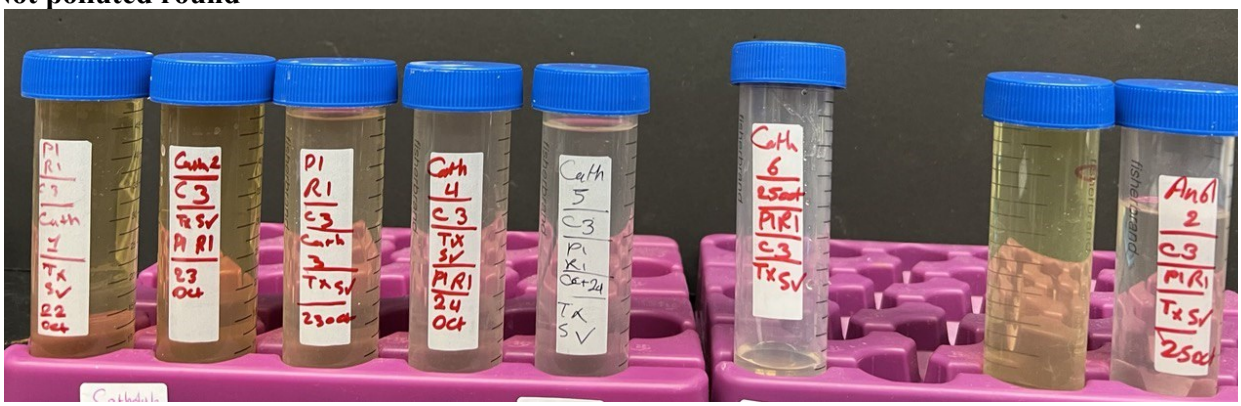


Figure A.5 Catholytes and anolytes (in order from left to right) for C3_1N_TNSY

Table A.6 Comprehensive datasheet for C3_1N_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.7	0	0	0
0.5	20	18.8	0	0	0
1.5	40	19.3	7	0	0
2.5	60	19.6	11	0	4
3.5	80	18.8	10	0	20
4.5	100	18.8	6	0	42.5
21	120	18.7	1	5	50
22	140	18.7	1	5	64
23	160	18.9	1	5	82.5
24	180	18.5	1	5	100
25	200	19.4	1	5	122
26	220	18.5	1	5	140
41	240	18.9	1	5	150
42	260	18.7	1	5	165
43	280	18.7	1	5	182.5
44	300	18.4	1	5	200
45	320	19.1	1	5	220
46	340	18.7	1	5	240
66.5	350	19.5	1	5	250
67	370	19.2	1	15	251
68	390	19.3	1	32.5	253
69	410	19.1	1	50	254
70	430	19.7	1	70	255
71	430	19.7	1	90	255

Round 1

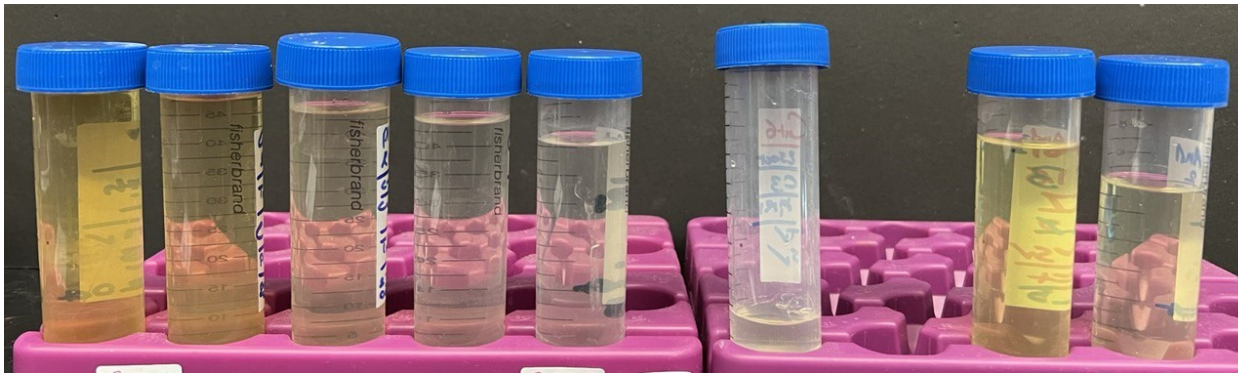


Figure A.6 Catholytes and anolytes (in order from left to right) for C3_11_TYSY

Table A.7 Comprehensive datasheet for C3_11_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.3	0	0	0
0.5	20	19.2	0	0	0
1.5	40	19.3	6	0	0
2.5	60	19.5	10	0	4
3.5	80	18.8	8	0	20
4.5	100	18.9	6	0	40
21	120	19.1	1	0	50
22	140	18.7	1	0	62
23	160	19	1	0	80
24	180	18.5	1	0	100
25	200	19.4	1	0	117.5
26	220	18.5	1	0	137.5
41	240	18.9	1	0	147.5
42	260	18.6	1	0	160
43	280	18.4	1	0	177.5
44	300	18.4	1	0	197.5
45	320	19.1	1	0	214.5
46	340	18.7	1	0	232.5
66.5	350	19.5	1	0	240
67	370	19.2	1	0	241
68	390	19.3	1	25	241
69	410	19.1	1	40	244
70	430	19.7	1	60	245
71	430	19.7	1	80	245

Round 2

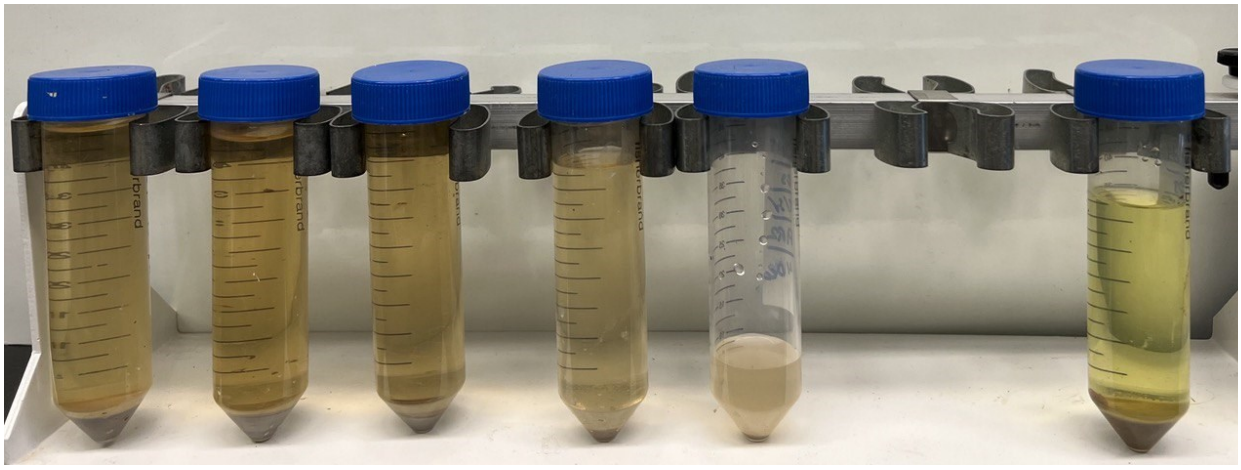


Figure A.7 Catholytes and anolyte (in order from left to right) for C3_12_TYSY

Table A.8 Comprehensive datasheet for C3_12_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.7	11	0	0
0.5	20	19.7	11	0	0
1.5	40	19.1	21	0	0
2.5	60	19.7	21	0	15
3.5	80	19.5	18	0	32.5
18.5	100	19.2	5	0	47.5
19.5	120	18.5	5	0	57.5
20.5	140	17.9	5	0	77.5
48	160	17.7	1	0	95
50	180	19.1	3	0	99
51	210	18.4	3	0	122.5
72	240	18.3	1	0	145
73	260	18.1	1	0	147
74	280	17.9	3	0	162.5
75	300	17.6	3	0	177.5
96.5	310	17.6	1	0	185
97.5	330	17.9	1	5	185
98.5	350	17.1	1	15	188
99.5	370	17.1	1	37.5	190
100	370	17.1	1	50	190

Round 3

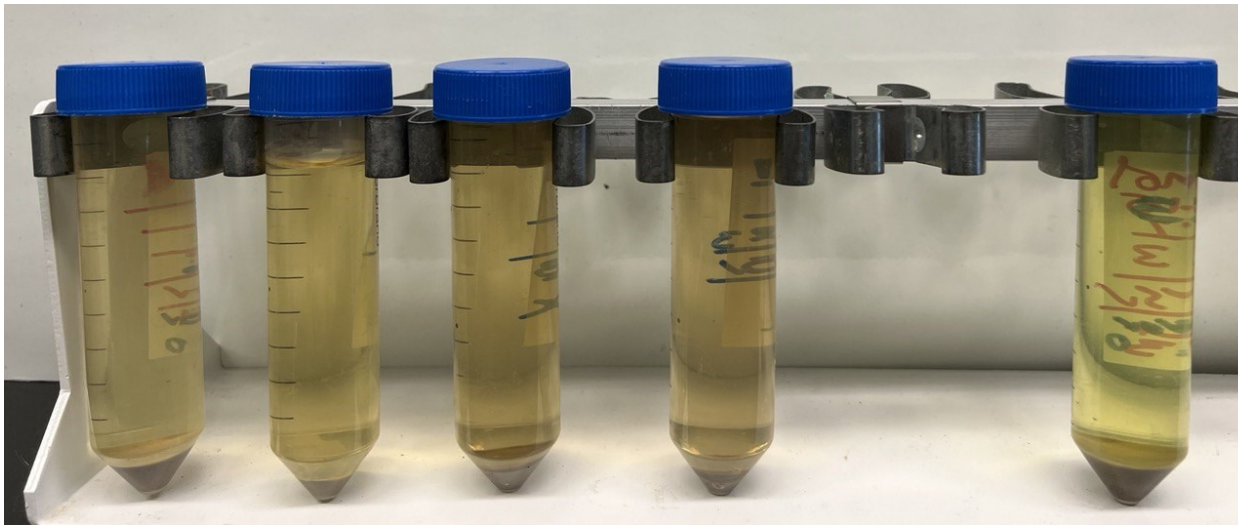


Figure A.8 Catholytes and anolyte (in order from left to right) for C3_13_TYSY

Table A.9 Comprehensive datasheet for C3_13_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.2	10	0	0
0.5	20	20.2	10	0	0
1.5	40	19.3	23	0	0
2.5	60	19.7	23	0	17
3.5	80	19.8	20	0	35
18.5	100	19.4	5	0	50
19.5	120	18.6	6	0	57.5
20.5	140	18.1	5	0	77.5
48	160	18.1	1	0	92.5
50	180	19.1	5	0	97.5
51	210	18.6	3	0	122.5
72	240	18.6	1	0	142.5
73	260	18.1	3	0	150
74	280	17.6	3	0	165
75	300	18.1	3	0	182.5
96.5	310	17.6	1	0	192.5
97.5	330	17.5	1	10	192.5
98.5	350	16.8	1	22.5	192.5
99.5	370	16.9	1	45	195
100	370	16.9	1	50	195

Organic matter content: 16%

Not polluted round

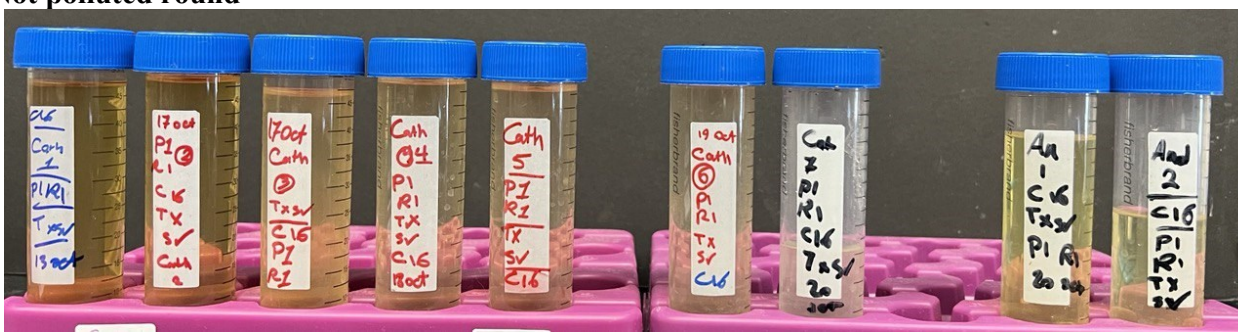


Figure A.9 Catholytes and anolytes (in order from left to right) for C16_1N_TNSY

Table A.10 Comprehensive datasheet for C16_1N_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.6	10	0	0
0.5	10	18.9	12	0	0
1	20	18.6	12	0	0
1.5	30	18.9	11	0	0
2	40	18.2	10	0	0
2.5	50	18.8	9	0	0
3	60	19.1	9	0	3
3.5	70	19.1	9	0	10
4	80	19.1	9	0	17.5
4.5	90	19.2	8	0	29
5	100	18.5	8	0	39
21.5	110	18.6	8	0	50
22	120	18.5	8	0	53
22.5	130	18.4	8	0	62
23	140	18.7	8	0	72.5
23.5	150	18.2	8	0	80
24	160	18.2	8	0	90
24.5	170	18.2	6	0	100
25	180	17.7	5	0	110
25.5	190	18.2	5	0	120
26	200	19.1	5	0	127.5
26.5	210	19.4	5	0	137.5
44	220	20	2	0	147.5
44.5	240	19.7	2	0	152.5
45.5	250	19.9	3	0	168.5

46	260	20.1	3	0	187.5
46.5	280	20	3	0	197.5
47.5	290	19.8	4	0	219.5
48	300	19.1	4	0	230
75.5	320	19.2	1	0	245
76.5	340	19.6	1	0	252.5
77.5	360	18.8	2	0	270
78.5	380	18.5	3	0	285
93.5	400	19.6	1	0	295
94.5	420	19.1	1	15	297
95.5	440	18.8	1	36	300
96.5	460	18.8	1	50	305
97.5	480	18.7	1	65	310
98.5	480	18.7	1	80	310

Round 1

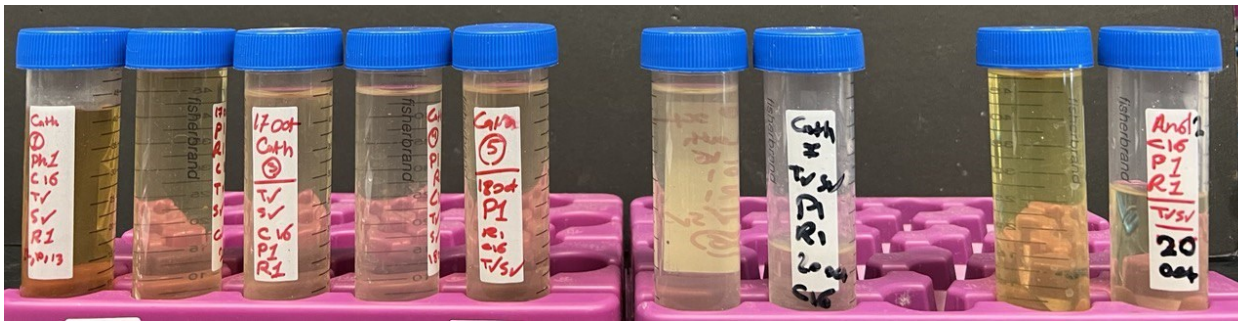


Figure A.10 Catholytes and anolytes (in order from left to right) for C16_11_TYSY

Table A.11 Comprehensive datasheet for C16_11_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.6	11	0	0
0.5	10	18.9	13	0	0
1	20	18.6	12	0	0
1.5	30	18.9	11	0	0
2	40	18.2	10	0	0
2.5	50	18.8	10	0	0
3	60	19.1	10	0	1
3.5	70	19.1	10	0	5
4	80	19.1	9	0	14
4.5	90	19.2	9	0	22.5
5	100	18.5	9	0	32.5
21.5	110	18.6	8	0	45

22	120	18.5	8	0	50
22.5	130	18.4	7	0	57.5
23	140	18.7	7	0	67.5
23.5	150	18.2	7	0	75
24	160	18.2	7	0	85
24.5	170	18.2	6	0	95
25	180	17.7	6	0	105
25.5	190	17.2	6	0	112.5
26	200	19.8	6	0	121
26.5	210	19.4	6	0	130
44	220	20	2	0	140
44.5	240	19.7	2	0	145
45.5	250	19.9	3	0	159
46	260	20.1	3	0	175
46.5	280	20	3	0	185
47.5	290	19.8	5	0	205
48	300	19.1	5	0	214
75.5	320	19.2	1	0	232.5
76.5	340	19.6	1	0	240
77.5	360	18.8	2	0	256.5
78.5	380	18.5	2	0	270.5
93.5	400	19.6	1	0	282.5
94.5	420	19.1	1	12.5	284.5
95.5	440	18.8	1	35	287.5
96.5	460	18.8	1	50	292.5
97.5	480	18.7	1	65	297.5
98.5	480	18.7	1	80	297.5

Round 2

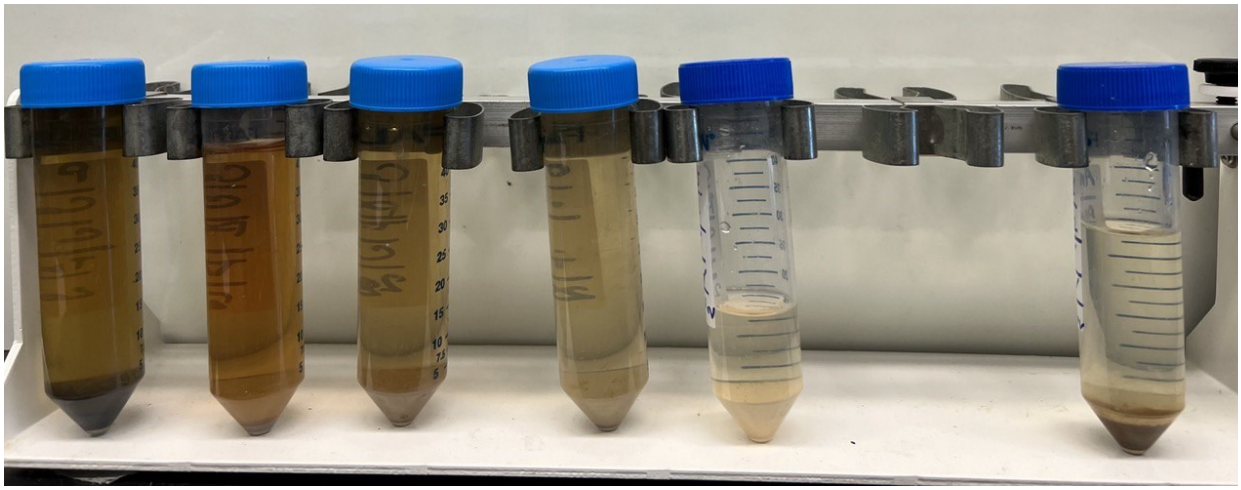


Figure A.11 Catholytes and anolyte (in order from left to right) for C16_12_TYSY

Table A.12 Comprehensive datasheet for C16_12_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.2	11	0	0
0.5	20	20.2	11	0	0
1.5	40	18.7	11	0	0
2.5	60	19.3	16	0	0
3.5	80	19.1	16	0	15
4.5	100	19.5	15	0	35
23.5	120	17.4	3	0	50
25.5	140	17.7	5	0	50
26.5	170	17.9	5	0	70
48.5	200	18.7	1	0	93
49.5	220	17.8	3	0	97
50.5	240	17.4	3	0	110.5
51.5	260	17.5	3	0	125.5
52.5	280	17.6	3	0	138
77.5	290	16.7	1	0	143
78.5	310	17.5	1	0	143
79.5	330	17.3	3	0	150.5
80.5	350	18.5	3	0	165.5
81.5	380	18.8	3	0	182.5
94.5	390	17.4	1	0	193
95.5	410	17.8	1	5	193
96.5	430	17.9	1	15	198
97.5	450	18.1	1	25	208
98.5	460	18.1	1	50	210

Round 3

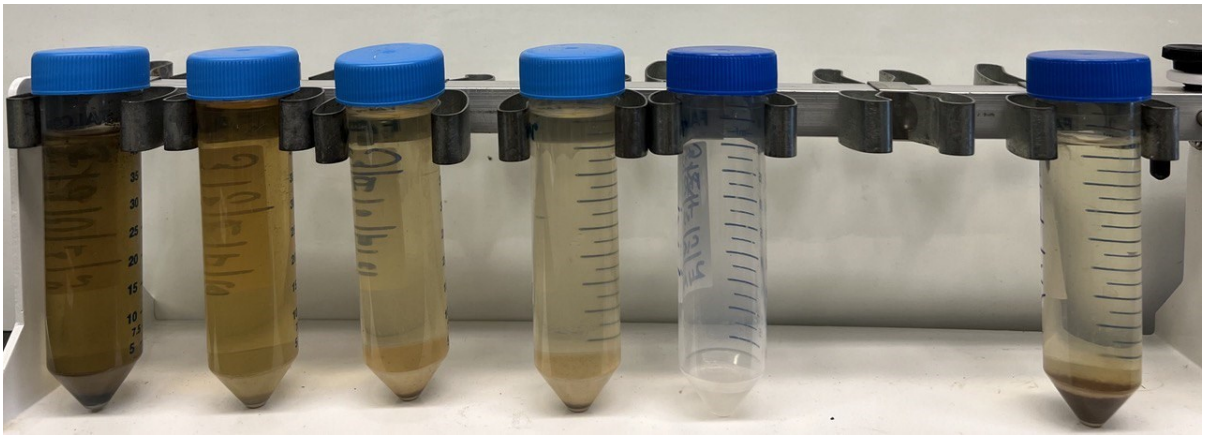


Figure A.12 Catholytes and anolyte (in order from left to right) for C16_13_TYSY

Table A.13 Comprehensive datasheet for C16_13_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.2	11	0	0
0.5	20	20.2	11	0	0
1.5	40	19.3	11	0	0
2.5	60	18.4	16	0	0
3.5	80	19.2	20	0	8
4.5	100	20	18	0	25
23.5	120	18.3	5	0	45
25.5	140	18.5	5	0	50
26.5	170	18.4	5	0	72.5
48.5	200	18	1	0	95
49.5	220	17.5	3	0	96
50.5	240	17.8	3	0	105
51.5	260	17.6	3	0	117.5
52.5	280	17.6	3	0	132.5
77.5	290	17.4	1	0	145
78.5	310	17.5	1	0	145
79.5	330	17.7	3	0	150
80.5	350	18.5	3	0	162.5
81.5	380	18.8	3	0	177.5
94.5	390	18.3	1	0	192.5
95.5	410	17.9	3	12.5	193.5
96.5	430	18.2	3	30	194.5
97.5	450	18	3	45	194.5
98.5	460	18	3	50	195

Organic matter content: 35%

Not polluted round

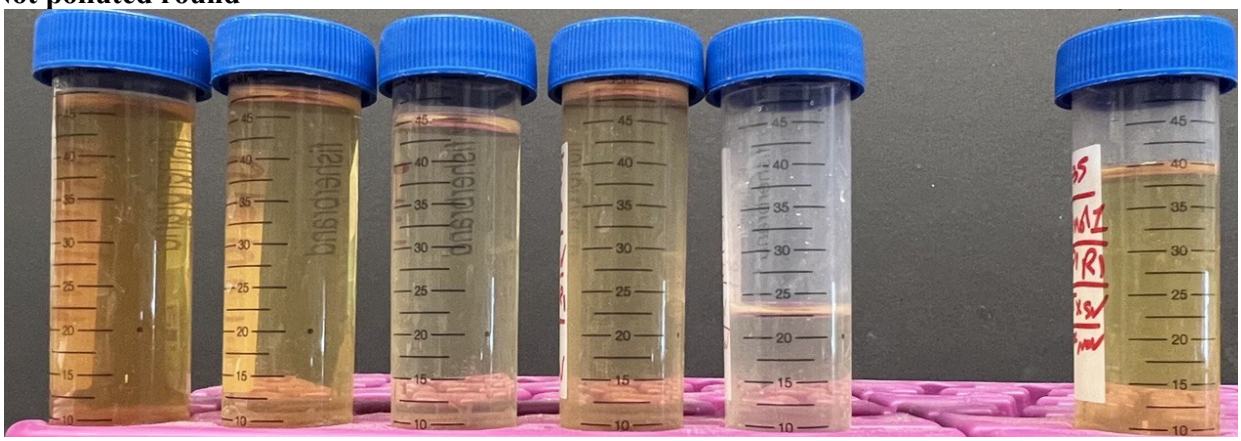


Figure A.13 Catholytes and anolyte (in order from left to right) for C35_1N_TNSY

Table A.14 Comprehensive datasheet for C35_1N_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.1	5	0	0
0.5	20	19.1	5	0	0
1.5	40	19.1	6	0	0
2.5	60	18.5	10	0	0
3.5	80	19.1	10	0	10
4.5	100	18.6	11	0	26
23.5	120	19.2	5	0	47.5
24	140	18.4	6	0	50.5
25	160	18.3	6	0	65.5
26	180	18.4	8	0	82.5
53.5	200	19.3	3	0	97.5
54.5	220	19.5	5	0	107.5
55.5	240	18.3	5	0	125
70.5	260	18.6	6	0	142.5
71.5	280	18.6	8	0	157.5
72.5	300	18.8	8	0	175
101	320	18.7	1	0	192.5
102	340	18.5	1	7.5	193.5
103	360	18.9	1	17.5	200
104	380	20.8	1	30	210
105	380	20.8	1	50	210

Round 1

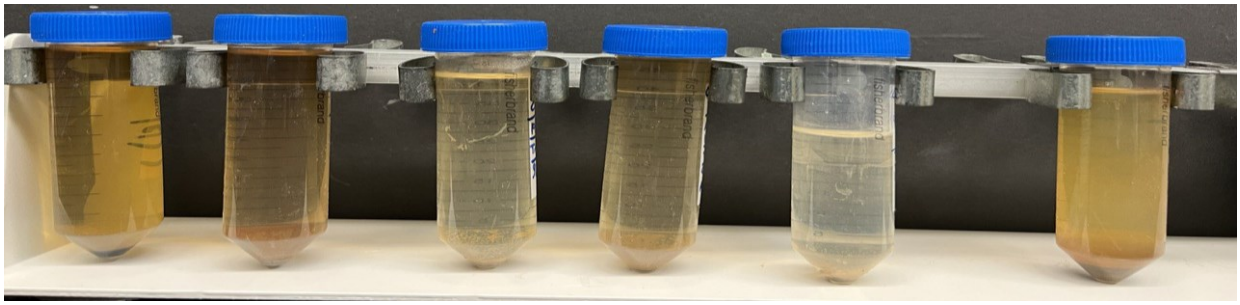


Figure A.14 Catholytes and anolyte (in order from left to right) for C35_11_TYSY

Table A.15 Comprehensive datasheet for C35_11_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	18.8	5	0	0
0.5	20	18.8	5	0	0
1.5	40	18.8	6	0	0
2.5	60	18.6	10	0	0
3.5	80	18.8	10	0	13
4.5	100	18.6	8	0	30
23.5	120	18.9	3	0	50
24	140	18.8	5	0	53
25	160	18.3	5	0	68
26	180	17.8	5	0	87
53.5	200	19.3	1	0	100
54.5	220	18.9	3	0	107.5
55.5	240	18.3	3	0	125
70.5	260	18.2	5	0	142.5
71.5	280	18.3	3	0	155
72.5	300	18.5	3	0	173.5
101	320	19.2	1	0	192.5
102	340	18.4	1	7.5	197.5
103	360	18.8	1	17.5	207.5
104	380	20.4	1	30	215
105	380	20.4	1	50	215

Round 2

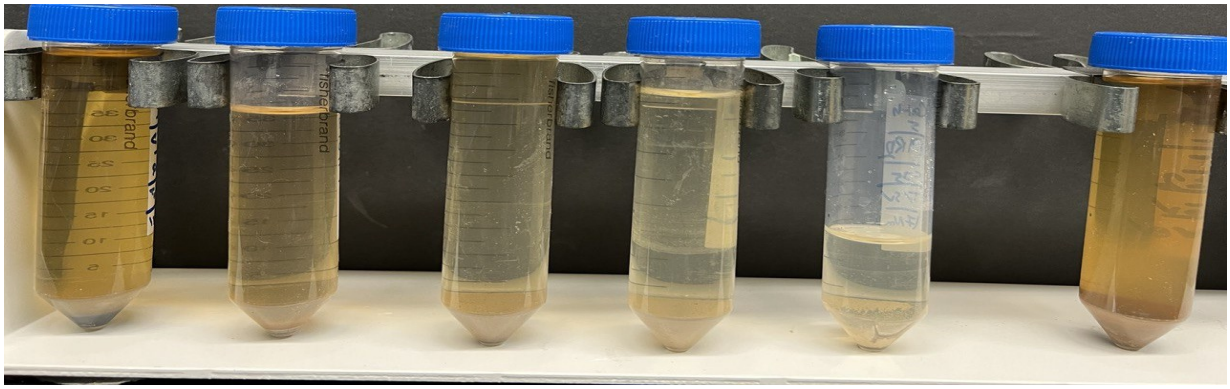


Figure A.15 Catholytes and anolyte (in order from left to right) for C35_12_TYSY

Table A.16 Comprehensive datasheet for C35_12_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.3	5	0	0
0.5	20	18.6	3	0	0
1.5	40	18.2	5	0	0
2.5	60	19.5	8	0	0
3.5	80	19.1	10	0	0
4.5	100	19.8	10	0	17.5
5.5	120	19.1	8	0	32.5
22	140	17.8	1	0	50
23	160	18.1	3	0	55
24	180	18.4	3	0	70
46.5	200	17.8	1	0	87.5
47.5	220	18.1	1	0	87.5
48.5	240	18.4	1	0	103.5
49.5	260	17.7	1	0	120
74.5	280	18.1	1	0	137.5
75.5	300	18	1	0	137.5
76.5	320	17.8	1	0	152.5
77.5	340	17.4	1	0	170
93.5	360	17.1	1	0	180
94.5	380	17.2	1	12.5	182
95.5	400	17.6	1	22.5	185
96.5	420	17.2	1	35	192
97.5	420	17.2	1	50	192

Round 3

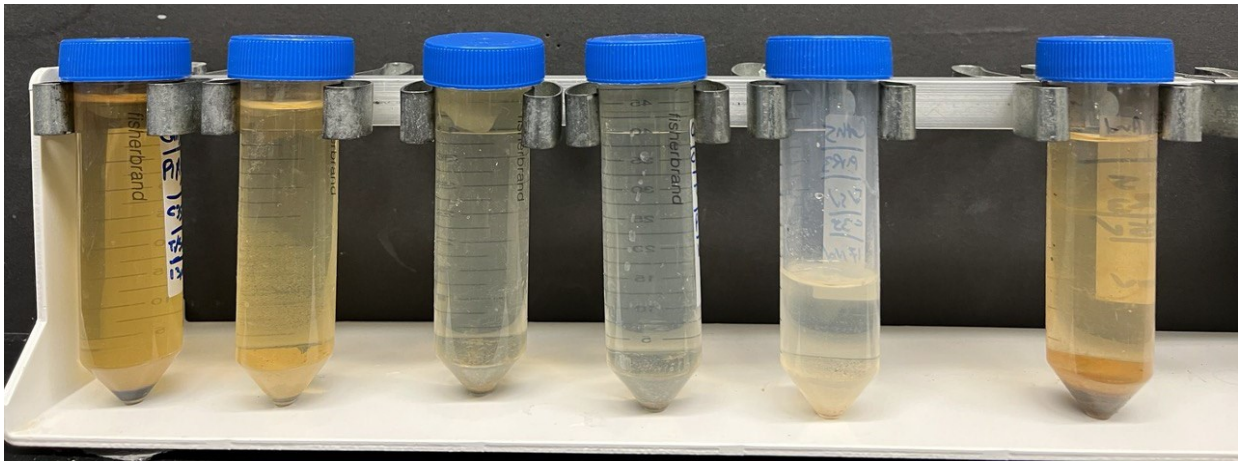


Figure A.16 Catholytes and anolyte (in order from left to right) for C35_13_TYSY

Table A.17 Comprehensive datasheet for C35_13_TYSY

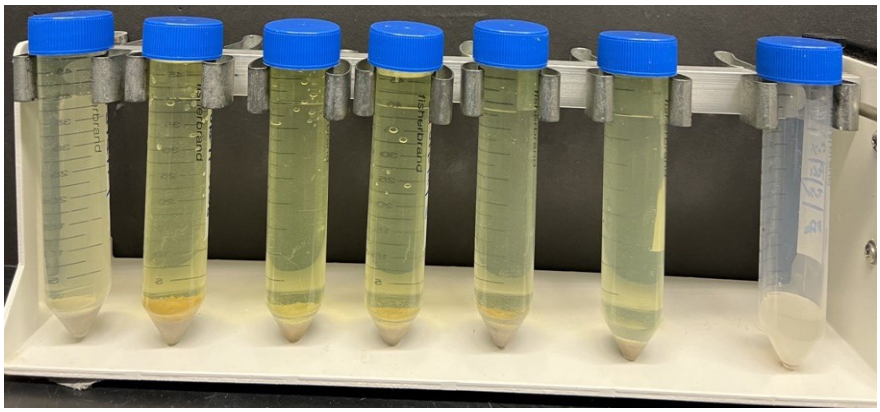
Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.3	5	0	0
0.5	20	18.6	3	0	0
1.5	40	18.2	5	0	0
2.5	60	19.5	8	0	0
3.5	80	19.1	10	0	7.5
4.5	100	19.4	8	0	22.5
5.5	120	19.1	8	0	37.5
22	140	18.5	3	0	50
23	160	18.5	5	0	62.5
24	180	18.8	5	0	75
46.5	200	18	1	0	95
47.5	220	17.9	3	0	95
48.5	240	19.4	3	0	115
49.5	260	18.4	3	0	130
74.5	280	19.1	1	0	145
75.5	300	19.5	1	0	152.5
76.5	320	18.5	3	0	170
77.5	340	18.6	3	0	187.5
93.5	360	17.1	1	0	195
94.5	380	18	1	5	197
95.5	400	18.2	1	15	200
96.5	420	18.3	1	30	207.5
97.5	420	18.3	1	50	207.5

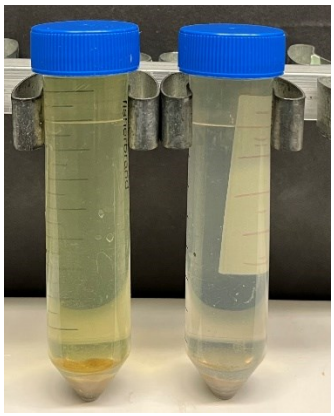
Phase 2

Organic matter content: 0%

Round 1

(a)





(b)

Figure A.17 a) Catholytes and b) anolytes (in order from left to right) for C0_21_TYSY

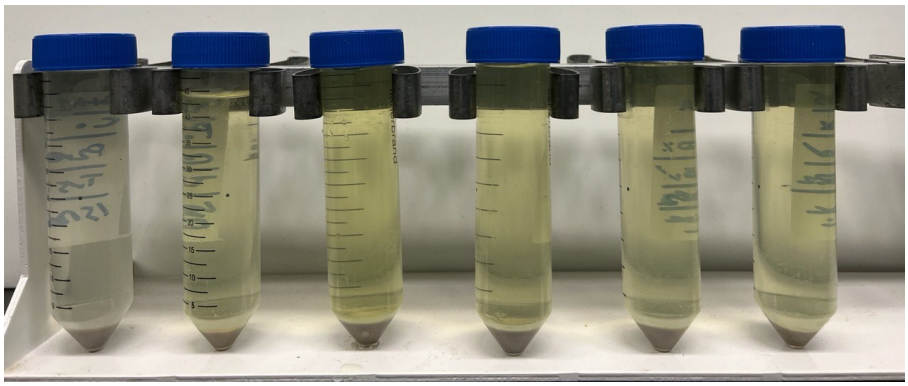
Table A.18 Comprehensive datasheet for C0_21_TYSY

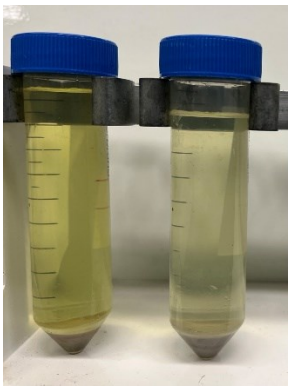
Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.5	12	0	0
0.5	20	16.9	18	0	0
1.5	40	10.4	17	0	12
2.5	60	7.3	15	0	27
3.5	80	3.6	13	0	50
4.5	100	2.8	12	0	67.5
29.5	120	2.7	1	0	90
30.5	140	3	3	0	100
31.5	160	4.2	5	0	120
32.5	180	2.7	5	0	137.5
33.5	200	4	5	0	150
53	220	2.7	1	0	170
54	240	2.6	3	0	178
55	260	2.7	3	0	200
56	280	3	3	0	220
75	300	3.2	1	0	240
76	320	2.9	1	0	250
77	340	2.4	1	0	269
78	360	2.4	1	0	287.5
93.5	380	2.7	1	0	300
94.5	400	3.3	1	15	300
95.5	420	2.8	1	32.5	302

96.5	440	3.4	1	50	305
97.5	460	4.5	1	17.5	305
98.5	480	4	1	37.5	305
99.5	480	4	1	50	305

Round 2

(a)





(b)

Figure A.18 a) Catholytes and b) anolytes (in order from left to right) for C0_22_TYSY

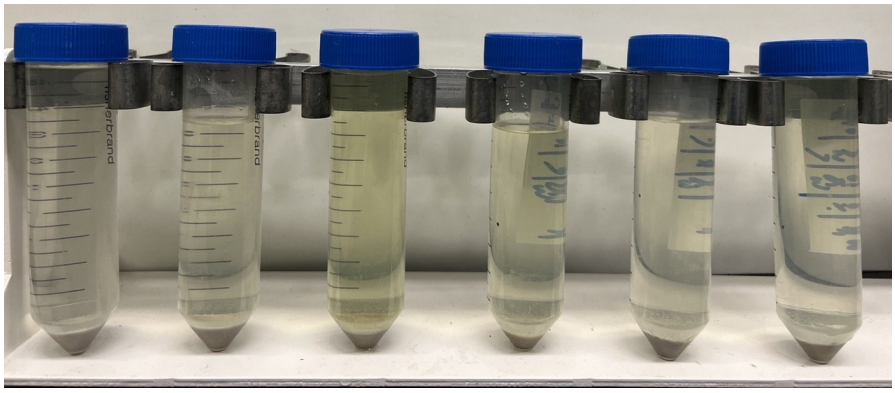
Table A.19 Comprehensive datasheet for C0_22_TYSY

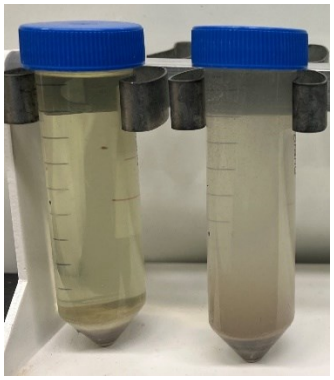
Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.5	12	0	0
0.5	20	16.9	18	0	0
1.5	40	10.4	17	0	12
2.5	60	7.3	15	0	27
3.5	80	3.6	13	0	50
4.5	100	2.8	12	0	67.5
29.5	120	2.7	1	0	90
30.5	140	3	3	0	100
31.5	160	4.2	5	0	120
32.5	180	2.7	5	0	137.5
33.5	200	4	5	0	150
53	220	2.7	1	0	170
54	240	2.6	3	0	178
55	260	2.7	3	0	200
56	280	3	3	0	220
75	300	3.2	1	0	240
76	320	2.9	1	0	250
77	340	2.4	1	0	269

78	360	2.4	1	0	287.5
93.5	380	2.7	1	0	300
94.5	400	3.3	1	15	300
95.5	420	2.8	1	32.5	302
96.5	440	3.4	1	50	305
97.5	460	4.5	1	17.5	305
98.5	480	4	1	37.5	305
99.5	480	4	1	50	305

Round 3

(a)





(b)

Figure A.19 a) Catholytes and b) anolytes (in order from left to right) for C0_23_TYSY

Table A.20 Comprehensive datasheet for C0_23_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.5	11	0	0
0.5	20	16.9	18	0	0
1.5	40	10.4	15	0	6
2.5	60	7.3	14	0	25
3.5	80	3.6	13	0	50
4.5	100	2.8	12	0	67.5
29.5	120	2.7	1	0	87.5
30.5	140	3	3	0	87.5
31.5	160	4.2	5	0	102.5
32.5	180	2.7	5	0	117.5
33.5	200	4	5	0	137.5
53	220	2.7	1	0	157.5
54	240	2.6	3	0	160
55	260	2.7	3	0	175
56	280	3	3	0	192.5

75	300	3.2	1	0	212.5
76	320	2.9	1	0	215
77	340	2.4	1	0	232.5
78	360	2.4	1	0	250
93.5	380	2.7	1	0	265
94.5	400	3.3	1	17.5	265
95.5	420	2.8	1	35	265
96.5	440	3.4	1	50	268
97.5	460	4.5	1	17.5	270
98.5	480	4	1	37.5	270
99.5	480	4	1	50	270

Organic matter content: 3%

Round 1

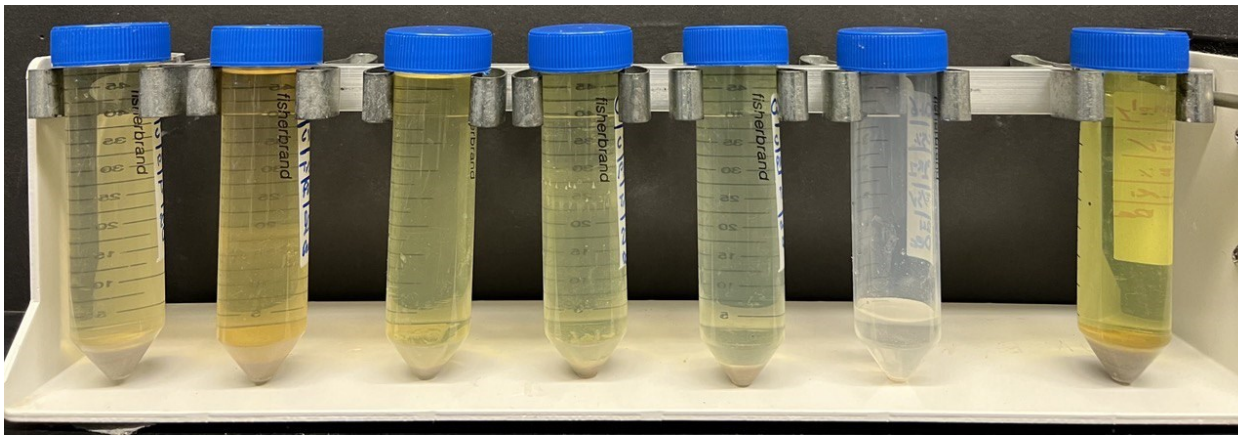


Figure A.20 Catholytes and anolyte (in order from left to right) for C3_21_TYSY

Table A.21 Comprehensive datasheet for C3_21_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.3	21	0	0
0.5	20	12	21	0	0
1.5	40	8.6	23	0	2
2.5	60	5.8	20	0	17.5
3.5	80	4.8	18	0	35
4.5	100	4.6	16	0	50
16.5	120	3.7	3	0	70
17.5	140	3.7	6	0	80
18.5	160	4	6	0	100
19.5	180	4.2	6	0	120
20.5	190	3.9	6	0	140
21	200	3.9	6	0	150
21.5	220	3.9	6	0	157.5
51	240	3.6	3	0	180
52	260	3.9	5	0	195
52.5	280	3.9	5	0	195
53	300	3.9	5	0	200
54	320	3.7	5	0	225
55	340	3.6	5	0	240
75.5	360	3.5	3	0	250
76.5	380	3.5	3	17.5	250
77.5	400	3.5	3	30	250
78.5	420	3.5	3	50	250

Round 2

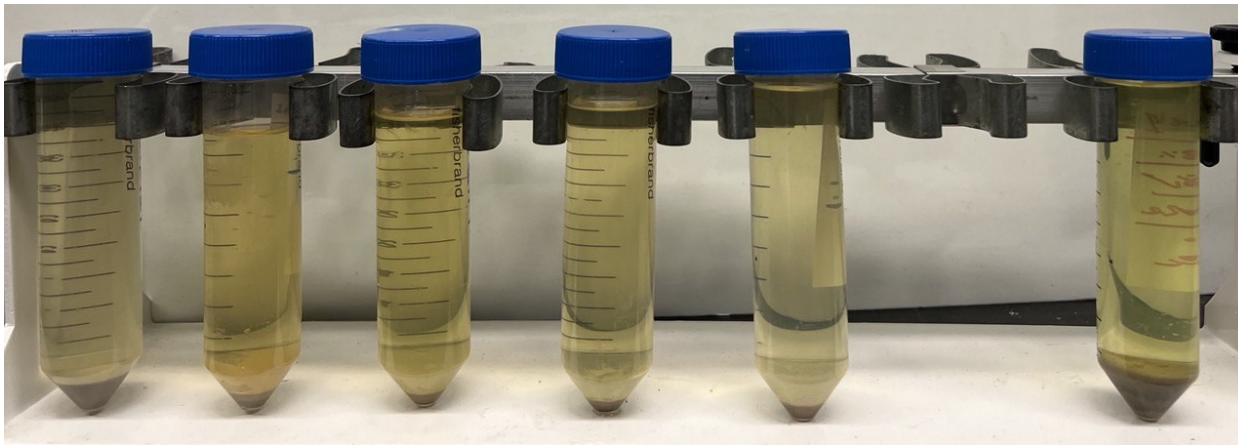


Figure A.21 Catholytes and anolyte (in order from left to right) for C3_22_TYSY

Table A.22 Comprehensive datasheet for C3_22_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.3	11	0	0
0.5	20	12	11	0	0
1.5	40	8.6	18	0	2
2.5	60	5.8	16	0	15
3.5	80	4.8	15	0	32.5
4.5	100	4.6	15	0	50
16.5	120	3.7	3	0	69
17.5	140	3.7	5	0	72.5
18.5	160	4	5	0	90
19.5	180	4.2	5	0	107.5
20.5	190	3.9	5	0	125
21	200	3.9	5	0	132.5
21.5	220	3.9	5	0	140
51	240	3.6	5	0	160.5
52	260	3.9	3	0	167.5
52.5	280	3.9	3	0	172.5
53	300	3.9	3	0	177.5
54	320	3.7	5	0	197.5
55	340	3.6	5	0	210
75.5	360	3.5	1	0	227.5
76.5	380	3.5	1	25	227.5
77.5	400	3.5	1	42.5	227.5
78.5	420	3.5	1	50	227.5

Round 3

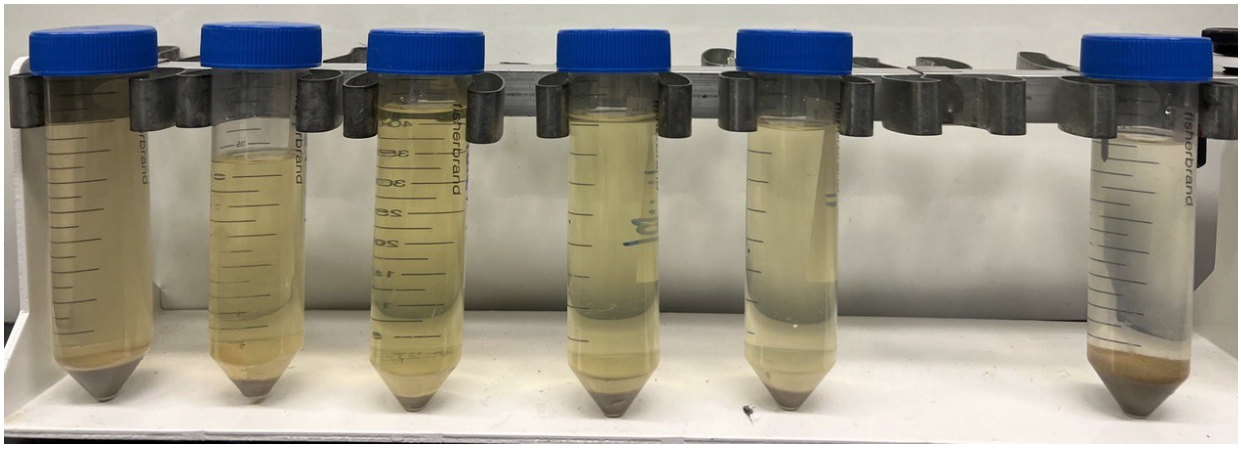


Figure A.22 Catholytes and anolytes (in order from left to right) for C3_23_TYSY

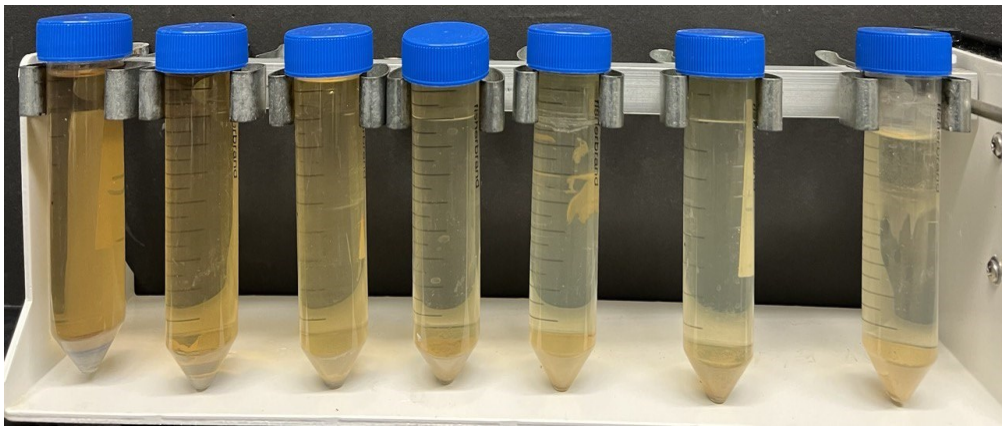
Table A.23 Comprehensive datasheet for C3_23_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.3	15	0	0
0.5	20	12	15	0	0
1.5	40	8.6	20	0	2
2.5	60	5.8	18	0	17.5
3.5	80	4.8	18	0	35
4.5	100	4.6	16	0	50
16.5	120	3.7	3	0	68
17.5	140	3.7	3	0	68
18.5	160	4	5	0	85
19.5	180	4.2	5	0	102.5
20.5	190	3.9	5	0	120
21	200	3.9	5	0	127.5
21.5	220	3.9	5	0	135
51	240	3.6	1	0	155.5
52	260	3.9	3	0	157.5
52.5	280	3.9	3	0	160
53	300	3.9	3	0	170
54	320	3.7	3	0	185
55	340	3.6	3	0	190
75.5	360	3.5	1	0	200
76.5	380	3.5	1	12.5	200
77.5	400	3.5	1	25	200
78.5	420	3.5	1	50	200

Organic matter content: 16%

Round 1

(a)



(b)

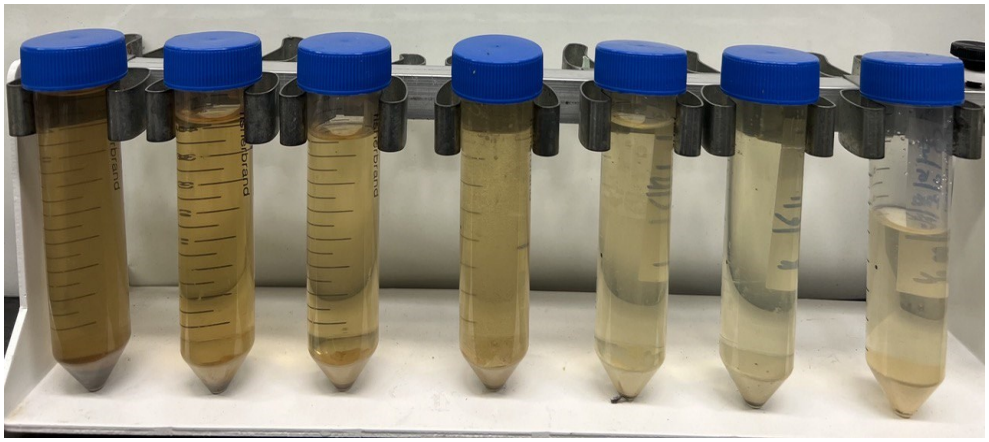
Figure A.23 a) Catholytes and b) anolytes (in order from left to right) for C16_21_TYSY

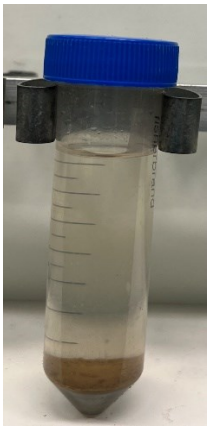
Table A.24 Comprehensive datasheet for C16_21_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.1	15	0	0
0.5	20	17.1	15	0	0
1.5	40	5.6	10	0	0
2.5	60	4.2	13	0	0
3.5	80	3.8	13	0	11
4.5	100	3.1	13	0	26
26.5	120	2.6	13	0	47.5
27.5	150	2.7	5	0	67.5
28.5	180	3.1	5	0	92.5
29	190	3.1	5	0	97.5
49.5	220	3.2	3	0	125
50.5	250	3	3	0	147.5
52.5	280	2.8	5	0	174.5
53.5	310	2.8	5	0	197.5
74.5	340	2.9	3	0	227.5
75.5	370	2.9	3	0	247.5
76.5	400	3	3	0	275
77.5	430	3	5	0	297.5
100.5	460	3.8	1	0	327.5
101.5	480	3.8	1	5	337.5
102.5	500	3.8	1	21	340
103.5	520	3.8	1	35	345

Round 2

(a)





(b)

Figure A.24 a) Catholytes and b) anolyte (in order from left to right) for C16_22_TYSY

Table A.25 Comprehensive datasheet for C16_22_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.1	10	0	0
0.5	20	17.1	10	0	0
1.5	40	5.6	10	0	0
2.5	60	4.2	13	0	3
3.5	80	3.8	11	0	16
4.5	100	3.1	11	0	32
26.5	120	2.6	3	0	50
27.5	150	2.7	5	0	62
28.5	180	3.1	5	0	85
29	190	3.1	5	0	95
49.5	220	3.2	3	0	120
50.5	250	3	3	0	137
52.5	280	2.8	3	0	162
53.5	310	2.8	5	0	187
74.5	340	2.9	3	0	212
75.5	370	2.9	3	0	232
76.5	400	3	3	0	259.5
77.5	430	3	5	0	282
100.5	460	3.8	1	0	309.5
101.5	480	3.8	1	17.5	309.5
102.5	500	3.8	1	37.5	312
103.5	520	3.8	1	47.5	312

Round 3

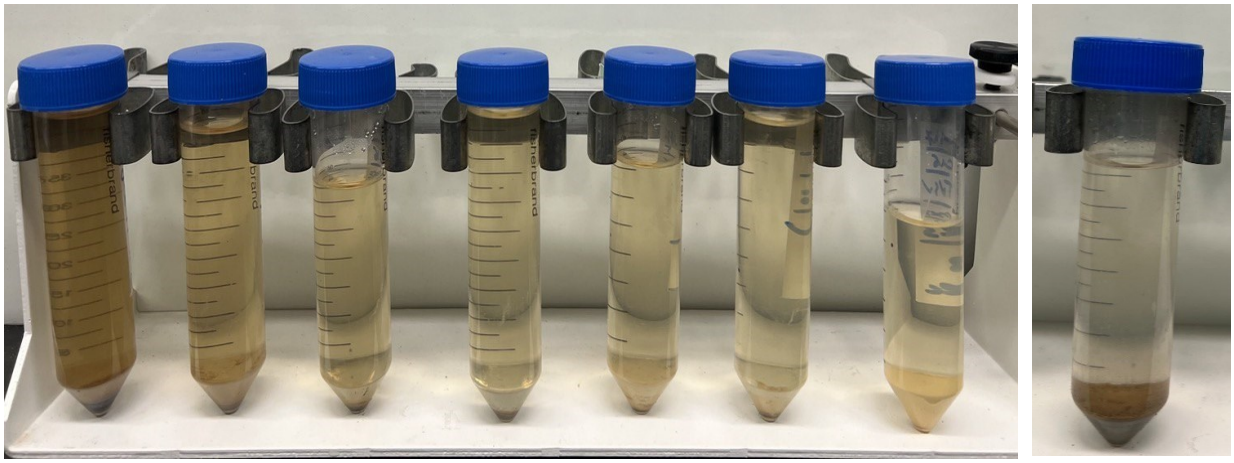


Figure A.25 a) Catholytes and b) anolyte (in order from left to right) for C16_23_TYSY

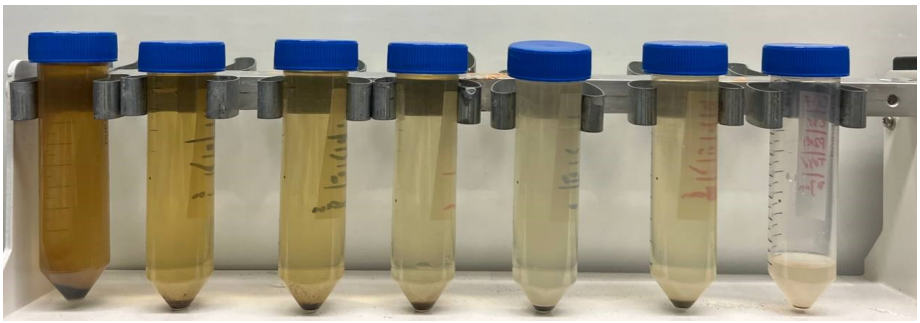
Table A.26 Comprehensive datasheet for C16_23_TYSY

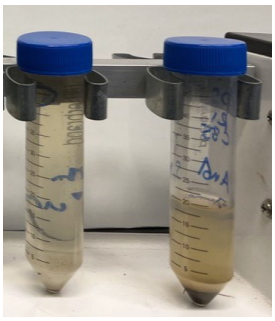
Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17.1	8	0	0
0.5	20	17.1	8	0	0
1.5	40	5.6	6	0	0
2.5	60	4.2	13	0	0
3.5	80	3.8	13	0	12.5
4.5	100	3.1	13	0	30
26.5	120	2.6	11	0	47.5
27.5	150	2.7	3	0	57.5
28.5	180	3.1	5	0	82.5
29	190	3.1	5	0	92.5
49.5	220	3.2	5	0	117.5
50.5	250	3	3	0	127.5
52.5	280	2.8	3	0	154.5
53.5	310	2.8	5	0	177.5
74.5	340	2.9	3	0	202.5
75.5	370	2.9	3	0	217.5
76.5	400	3	3	0	245
77.5	430	3	5	0	262.5
100.5	460	3.8	1	0	287.5
101.5	480	3.8	1	7.5	290
102.5	500	3.8	1	22.5	290.5
103.5	520	3.8	1	35	292.5

Organic matter content: 35%

Round 1

(a)





(b)

Figure A.26 a) Catholytes and b) anolytes (in order from left to right) for C35_21_TYSY

Table A.27 Comprehensive datasheet for C35_21_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17	10	0	0
0.5	20	17	10	0	0
1.5	40	10.3	8	0	0
3.5	60	4.1	10	0	5
4.5	80	4	11	0	15
5.5	100	3.9	11	0	30
17.5	120	3.5	8	0	50
18.5	140	4.2	8	0	62.5
19.5	160	4.2	8	0	82.5
20.5	180	4.3	8	0	100
21.5	200	4.3	8	0	120
22.5	220	3.5	7	0	140
26.5	240	3.3	3	0	150
27.5	260	3.4	6	0	160
28.5	280	3.6	6	0	177.5
29.5	300	3.6	6	0	200
30.5	320	3.4	6	0	217.5
54.5	340	3	3	0	235
55.5	360	2.7	4	0	250
56.5	380	2.4	5	0	267.5
57.5	400	3.2	6	0	287.5
86.5	420	2.1	1	4	300
87.5	440	2.7	1	12.5	300
88.5	460	2.7	1	27.5	304
89.5	480	2.7	1	45	305
100	500	2.7	1	50	305

Round 2

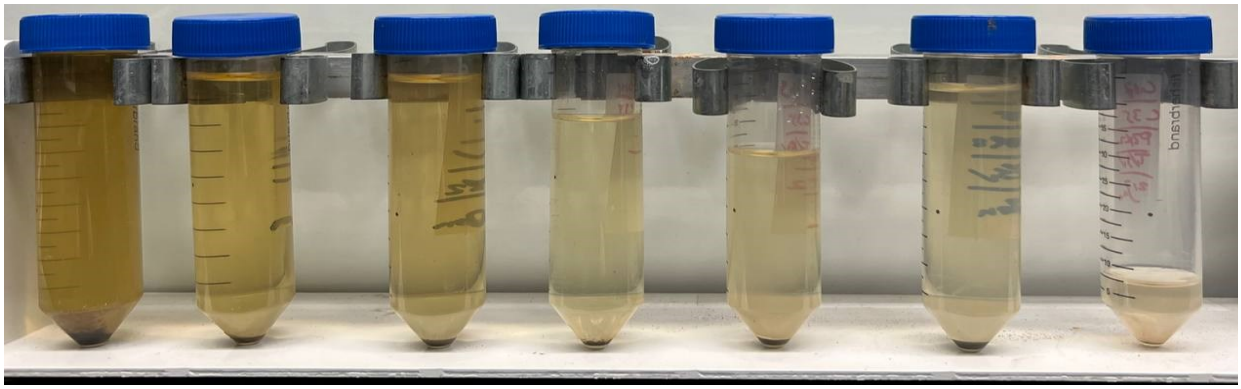


Figure A.27 Catholytes (in order from left to right) for C35_22_TYSY

Table A.28 Comprehensive datasheet for C35_22_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17	10	0	0
0.5	20	17	10	0	0
1.5	40	10.3	8	0	0
3.5	60	4.1	10	0	5
4.5	80	4	11	0	17.5
5.5	100	3.9	11	0	35
17.5	120	3.5	3	0	50
18.5	140	4.2	3	0	62.5
19.5	160	4.2	5	0	77.5
20.5	180	4.3	5	0	95
21.5	200	4.3	5	0	110
22.5	220	3.5	7	0	132.5
26.5	240	3.3	1	0	140
27.5	260	3.4	3	0	145
28.5	280	3.6	3	0	160
29.5	300	3.6	3	0	175
30.5	320	3.4	3	0	190
54.5	340	3	1	0	202.5
55.5	360	2.7	1	0	205
56.5	380	2.4	2	0	219
57.5	400	3.2	2	0	232.5
86.5	420	2.1	1	47.5	247.5
87.5	440	2.7	1	55	247.5
88.5	460	2.7	1	62.5	248.5
89.5	480	2.7	1	72.5	253.5
100	500	2.7	1	97.5	253.5

Round 3

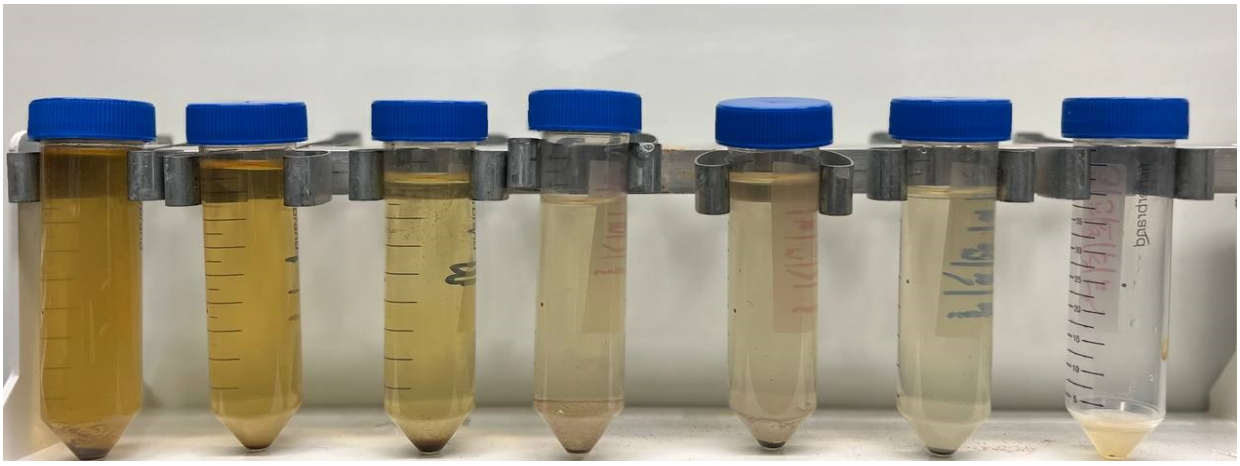


Figure A.28 Catholytes (in order from left to right) for C35_23_TYSY

Table A.29 Comprehensive datasheet for C35_23_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	17	10	0	0
0.5	20	17	10	0	0
1.5	40	10.3	8	0	0
3.5	60	4.1	10	0	5
4.5	80	4	11	0	17.5
5.5	100	3.9	11	0	30
17.5	120	3.5	5	0	50
18.5	140	4.2	5	0	60
19.5	160	4.2	6	0	77.5
20.5	180	4.3	7	0	95
21.5	200	4.3	8	0	110
22.5	220	3.5	7	0	130
26.5	240	3.3	1	0	137.5
27.5	260	3.4	3	0	142.5
28.5	280	3.6	3	0	157.5
29.5	300	3.6	3	0	175
30.5	320	3.4	3	0	192.5
54.5	340	3	1	0	208
55.5	360	2.7	2	0	217.5
56.5	380	2.4	2	0	232.5
57.5	400	3.2	3	0	247.5
86.5	420	2.1	1	30	257.5
87.5	440	2.7	1	35	257.5
88.5	460	2.7	1	45	258.5
89.5	480	2.7	1	60	262.5
100	500	2.7	1	80	262.5

Appendix B: Phase 3: Supplementary study on organic soil remediation in cold temperatures

Phase 3 followed the same procedure as Phases 1 and 2, serving as a supplementary stage. The soil samples contained approximately 35% organic matter and were exposed to a temperature of 7°C during this phase. The first and second soil mixtures were contaminated with toluene, while the third mixture remained free from any pollution (Table B.1,2). For the first and third mixtures, a surfactant solution was injected, while the second mixture received DI water injections.

Table B.1 Variables of Phase 3

	Toluene	Surfactant
1	Present	Present
2	Present	Absent
3	Absent	Present

Table B.2 List of samples tested in Phase 3

	Round 1	Round 2	Round 3
1	C35_P31_TYSY	C35_P32_TYSY	C35_P33_TYSY
2	C35_P31_TYSN	C35_P32_TYSN	C35_P33_TYSN
3	C35_P31_TNSY	C35_P32_TNSY	C35_P33_TNSY

Round 1

C35_P31_TYSY

Table B.3 Comprehensive datasheet for C35_31_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	12.9	5	0	0
0.5	10	8.4	6	0	0
1	20	8.1	6	0	0
1.5	30	6.7	6	0	0
2	40	5.5	8	0	0
2.5	50	5.5	9	0	2
3	60	5	10	0	5
3.5	70	5.4	10	0	12
4	80	5.5	10	0	19.5
4.5	90	5.5	10	0	26
5	100	5.1	9	0	37.5
5.5	110	4.8	9	0	47.5
6	120	4.9	9	0	57.5

6.5	130	5	9	0	67.5
7	140	4.9	9	0	73.5
7.5	150	5.3	9	0	87.5
8	160	5.4	8	0	97.5
8.5	170	5.5	8	0	109.5
9	180	4.9	8	0	117.5
9.5	190	4.7	8	0	127.5
20	200	4.5	5	0	140
20.5	210	5	6	0	147.5
21	220	4.8	6	0	157.5
21.5	230	4.8	6	0	167.5
22	240	5.2	6	0	178
22.5	250	4.7	6	0	187.5
23	260	4.2	6	0	198.5
24	270	4.2	6	0	209.5
25.5	280	4.2	6	0	220
26.5	290	4.2	6	0	230
27.5	300	4.2	6	0	240
28	310	4.2	6	0	250
28.5	320	4.3	6	0	260
29	330	5.2	6	0	270
29.5	340	5	6	0	280
30	350	4.8	6	0	292
43.5	360	3.6	4	0	305
44	370	4.8	5	0	312.5
44.5	380	5.4	5	0	324
45	390	4.9	6	0	334
45.5	400	4.9	6	0	346.5
46	410	4.6	6	0	359
46.5	420	5.3	6	0	371.5
47.5	430	6.8	7	0	384
48	440	5.7	7	0	394
48.5	450	5.6	7	0	406.5
49	460	4.8	7	0	416.5
49.5	470	4.5	7	0	426.5
50.5	480	4.3	7	0	436.5
51.5	490	4.4	7	0	446.5
52	500	4.5	7	0	457.5
52.5	510	4.1	6	0	466.5
53	520	4.4	6	0	476.5
53.5	530	3.4	6	0	486.5
67.5	550	5	3	0	511.5
68	560	5.7	4	0	516.5
68.5	570	6	4	0	529

69	580	5.1	5	0	539
69.5	590	5.5	5	0	551.5
70	600	5.5	5	0	561.5
70.5	610	5	5	0	574
71	620	3.9	5	0	584
73	630	3.9	5	0	601.5
74.5	640	3.9	5	0	611.5
75.5	650	3.9	5	0	623.5
78.5	670	4	5	0	643.5
91.5	680	3.5	3	0	656.5
94	690	4.6	3	0	658.5
94.5	700	4.7	4	6	663.5
95	710	4.6	4	12.5	666
96	720	4	4	20	668.5
96.5	730	4	3	27.5	671.5
118	740	2.7	3	35	676

C35_P31_TYSN

Table B.4 Comprehensive datasheet for C35_31_TYSN

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	14.5	5	0	0
0.5	10	8.3	5	0	0
1	20	8.1	6	0	0
1.5	30	7.1	8	0	0
2	40	5.1	9	0	0
2.5	50	5.3	10	0	0
3	60	5.6	12	0	0
3.5	70	5.5	11	0	5
4	80	5.5	10	0	12.5
4.5	90	5.5	10	0	20
5	100	5.2	9	0	30
5.5	110	4.8	9	0	38
6	120	4.9	9	0	47.5
6.5	130	4.9	9	0	59.5
7	140	4.9	8	0	70
7.5	150	5.4	8	0	77.5
8	160	5.4	8	0	85
8.5	170	5.7	7	0	95
9	180	4.9	7	0	104
9.5	190	4.7	7	0	112.5

20	200	4.7	5	0	122.5
20.5	210	5	6	0	127.5
21	220	5	6	0	135.5
21.5	230	5.2	6	0	145
22	240	5.2	6	0	155
22.5	250	4.8	6	0	164.5
23	260	4.5	6	0	173.5
24	270	4.7	6	0	181
25.5	280	4.8	6	0	188.5
26.5	290	4.6	6	0	196
27.5	300	4.4	6	0	206
28	310	4.7	6	0	213.5
28.5	320	4.8	6	0	223.5
29	330	4.8	6	0	233.5
29.5	340	4.9	6	0	241.5
30	350	5	6	0	251
43.5	360	3.9	4	0	258.5
44	370	4.9	5	0	263.5
44.5	380	5.66	6	0	271.5
45	390	5.1	6	0	279
45.5	400	5.1	6	0	286.5
46	410	4.6	6	0	296.5
46.5	420	5.3	6	0	304
47.5	430	6.6	7	0	314
48	440	6.2	7	0	324
48.5	450	6.1	7	0	333
49	460	5.3	7	0	341
49.5	470	5.1	7	0	349
50.5	480	4.9	7	0	358.5
51.5	490	4.5	7	0	368.5
52	500	4.4	7	0	376
52.5	510	4.3	6	0	383.5
53	520	4.3	6	0	391
53.5	530	4.3	6	0	406
67.5	550	4.3	6	0	426
68	560	4.3	3	0	431
68.5	570	5	4	0	438.5
69	580	5.4	4	0	446
69.5	590	5.7	5	0	456
70	600	5.5	5	0	466
70.5	610	5.7	5	0	473.5
71	620	5.8	5	0	482
73	630	5.8	5	0	498.5
74.5	640	5.2	5	0	506

75.5	650	4.3	5	0	516
78.5	670	4.5	5	0	531
91.5	680	4	6	0	541
94	690	3.8	4	0	542
94.5	700	4.6	4	5	543
95	710	4.7	4	12.5	543.5
96	720	4.4	4	20	543.5
96.5	730	4	4	26	543.5
118	740	4.5	4	32.5	546

C35_P31_TNSY

Table B.5 Comprehensive datasheet for C35_31_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	12.1	5	0	0
0.5	10	9.7	5	0	0
1	20	7.8	5	0	0
1.5	30	6.1	5	0	0
2	40	6.2	6	0	0
2.5	50	6.6	7	0	0
3	60	5.2	8	0	0
3.5	70	4.9	8	0	5
4	80	5.5	8	0	11
4.5	90	5.3	8	0	17.5
5	100	4.9	7	0	26
5.5	110	4.6	7	0	35
6	120	4.6	7	0	45
6.5	130	4.9	6	0	55
7	140	4.9	7	0	67.5
7.5	150	5.2	7	0	80
8	160	5.2	7	0	87.5
8.5	170	5.3	7	0	97.5
9	180	4.3	7	0	106.5
9.5	190	4.9	6	0	115
20	200	5.1	4	0	127.5
20.5	210	5	4	0	131.5
21	220	4.4	4	0	139
21.5	230	4.7	4	0	148.5
22	240	4.8	5	0	158.5
22.5	250	4.7	5	0	168.5

23	260	3.9	5	0	177.5
24	270	3.9	5	0	181.5
25.5	280	4	5	0	190
26.5	290	4	5	0	198.5
27.5	300	4.2	5	0	208.5
28	310	4.2	5	0	217.5
28.5	320	4	5	0	227.5
29	330	4.7	6	0	237.5
29.5	340	5	6	0	247.5
30	350	4.7	6	0	255
43.5	360	4.8	3	0	263.5
44	370	4.9	4	0	265.5
44.5	380	4.8	4	0	275
45	390	4.7	5	0	284
45.5	400	4.8	6	0	293
46	410	4.2	6	0	303
46.5	420	4.2	6	0	312.5
47.5	430	7.2	7	0	325
48	440	5.5	6	0	335
48.5	450	5.5	6	0	344
49	460	4	6	0	355
49.5	470	5.1	6	0	365
50.5	480	4.2	6	0	375
51.5	490	4.2	6	0	384
52	500	4.1	5	0	392.5
52.5	510	3.9	5	0	402.5
53	520	4.4	6	0	411
53.5	530	4.2	6	0	416
67.5	550	3.4	3	0	434
68	560	5	3	0	441
68.5	570	6.8	4	0	446
69	580	6	5	0	456
69.5	590	4.9	5	0	466
70	600	5.5	5	0	476
70.5	610	5.4	5	0	487
71	620	5	5	0	496
73	630	4.5	5	0	513.5
74.5	640	3.9	5	0	523.5
75.5	650	3.9	5	0	532.5
78.5	670	3.6	4	0	550.5
91.5	680	3.4	3	0	561
94	690	4.6	3	5	562
94.5	700	4.4	3	12.5	563
95	710	4.6	3	20	564

96	720	4	3	30	565.5
96.5	730	3.9	3	37.5	565.5
118	740	3.1	2	37.5	565.5

Round 2

C35_P32_TYSY

Table B.6 Comprehensive datasheet for C35_32_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.8	7	0	0
0.5	10	15.7	7	0	0
1.5	20	8.6	8	0	0
2	30	6.7	8	0	0
2.5	40	5.5	7	0	0
3	50	5.1	7	0	2
3.5	60	5.1	7	0	5
4	80	5.1	7	0	12.5
5	90	5.4	7	0	27.5
5.5	100	5.1	7	0	38
6.5	110	4.5	7	0	49
7	130	4.4	7	0	59
25.5	140	4	4	0	81
26	150	4.3	4	0	89
26.5	160	4.3	5	0	96.5
27	170	4.5	5	0	105.5
27.5	180	5.9	5	0	114
28	190	6	6	0	122.5
29.5	210	6	6	0	143.5
30.5	220	4.4	6	0	154.5
54	240	4.1	4	0	173.5
54.5	250	5.3	4	0	177.5
55	260	5.1	4	0	186.5
55.5	270	4.8	4	0	193.5
56	280	4.8	4	0	203.5
57	290	4.8	4	0	213.5
57.5	300	4.3	5	0	223.5
58	310	5	5	0	233.5
58.5	320	4.6	5	0	243.5
59	330	4.7	5	0	253.5
59.5	340	4.5	5	0	264.5
60	350	5.1	5	0	273.5
74.5	360	3.5	4	0	283.5
75	370	4.7	4	0	288.5
75.5	380	5	4	0	298.5
76	390	5.3	4	0	308.5

76.5	400	5.3	5	0	318.5
77	410	5.3	5	0	328.5
77.5	420	5.1	5	0	338.5
78	430	5.2	5	0	348.5
78.5	440	4.7	5	0	358.5
79	450	5.2	5	0	366.5
122	460	3.3	3	0	378.5
123	470	5.2	3	10	378.5
123.5	480	5.2	3	15	381
124	490	5.5	3	20	383.5
124.5	500	5.4	3	30	386
125	510	5.1	3	35	388.5

C35_P32_TYSN

Table B.7 Comprehensive datasheet for C35_32_TYSN

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19	8	0	0
0.5	10	15.7	8	0	0
1.5	20	8.6	8	0	0
2	30	6.7	8	0	0
2.5	40	5.6	8	0	0
3	50	5.4	8	0	0
3.5	60	5.4	8	0	1
4	80	5.4	8	0	7.5
5	90	5.9	8	0	20
5.5	100	5.4	8	0	30
6.5	110	5	8	0	39
7	130	5	8	0	49
25.5	140	4.2	4	0	68
26	150	4.3	4	0	76.5
26.5	160	4.4	5	0	86.5
27	170	4.5	6	0	96.5
27.5	180	5.7	6	0	106.5
28	190	5.9	7	0	117.5
29.5	210	5	8	0	134
30.5	220	4.4	8	0	144
54	240	4.5	4	0	166.5
54.5	250	5.4	4	0	171
55	260	5.5	4	0	180
55.5	270	5.6	5	0	189

56	280	5.2	5	0	199
57	290	5	6	0	209
57.5	300	5	6	0	217
58	310	5	6	0	226.5
58.5	320	5.1	7	0	234
59	330	5.5	7	0	244
59.5	340	5.6	7	0	255
60	350	5.7	7	0	264
74.5	360	3.6	4	0	274
75	370	4.7	4	0	281.5
75.5	380	5	4	0	291.5
76	390	5.5	5	0	301.5
76.5	400	5.3	5	0	311.5
77	410	5.8	5	0	321.5
77.5	420	5.4	6	0	331.5
78	430	5.4	6	0	341.5
78.5	440	5.1	6	0	351.5
79	450	5.2	7	0	361.5
122	460	3.7	3	0	371.5
123	470	5.2	3	10	371.5
123.5	480	5.2	3	17.5	374
124	490	5.5	3	24	376.5
124.5	500	5.5	3	31	379
125	510	5.5	3	35	381.5

C35_P32_TNSY

Table B.8 Comprehensive datasheet for C35_32_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	19.1	6	0	0
0.5	10	14.1	5	0	0
1.5	20	6.5	5	0	0
2	30	5.7	5	0	0
2.5	40	4.6	6	0	0
3	50	5	7	0	0
3.5	60	5.1	8	0	2
4	80	5.3	8	0	7.5
5	90	5	7	0	22.5
5.5	100	4.6	7	0	31
6.5	110	4	7	0	41
7	130	4.2	7	0	51

25.5	140	3.5	3	0	68.5
26	150	4.3	4	0	71
26.5	160	3.9	5	0	81
27	170	4.5	6	0	89
27.5	180	5.5	7	0	99
28	190	5.8	7	0	109
29.5	210	4.7	6	0	126
30.5	220	4.6	6	0	136.5
54	240	3	3	0	149
54.5	250	5.4	4	0	152
55	260	5.1	5	0	158
55.5	270	4.9	6	0	166.5
56	280	4.6	6	0	176.5
57	290	4.5	6	0	184
57.5	300	4.2	6	0	194
58	310	5	6	0	204
58.5	320	4.5	6	0	214
59	330	4.8	6	0	221.5
59.5	340	4.4	6	0	233
60	350	4.9	6	0	244
74.5	360	3.5	3	0	254
75	370	4.7	4	0	256.5
75.5	380	4.6	5	0	265
76	390	5.3	6	0	274
76.5	400	5.3	6	0	284
77	410	5.6	6	0	294
77.5	420	5	6	0	304
78	430	5.1	6	0	312
78.5	440	4.5	6	0	321.5
79	450	5.1	6	0	332
122	460	3.3	2	0	344
123	470	5.1	3	10	345
123.5	480	5.2	3	15	346
124	490	5.5	3	20	346
124.5	500	5.4	3	27.5	346
125	510	5.1	3	35	346

Round 3

C35_P33_TYSY

Table B.9 Comprehensive datasheet for C35_33_TYSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.1	8	0	0
0.5	10	18.9	8	0	0
1	20	15.5	7	0	0
1.5	30	11.4	7	0	0
2	40	8.4	8	0	0
2.5	50	7.3	8	0	0
3	60	6.6	8	0	5
3.5	70	5.9	9	0	10
4	80	5.2	9	0	17.5
4.5	90	5.5	9	0	25
24	115	3.7	4	0	45
24.5	125	5.2	4	0	50
25	135	5.1	4	0	60
25.5	145	5.6	5	0	67.5
26	155	5.7	5	0	70
26.5	165	5.3	6	0	77.5
27	175	5.1	6	0	87.5
27.5	185	5.9	7	0	97.5
28	195	5	7	0	97.5
28.5	205	5.2	8	0	107.5
29	215	5.9	8	0	115.5
47.5	225	4.5	4	0	127.5
48	235	5	4	0	135
48.5	245	5.2	5	0	137.5
49	255	6.1	5	0	150
49.5	265	6	5	0	165
50	275	6.7	6	0	177.5
50.5	285	6.2	6	0	187.5
51	300	6.5	7	0	192.5
51.5	310	6.6	7	0	200
52	320	6.6	7	0	207.5
52.5	330	6.6	7	0	215
72.5	340	4.4	3	0	235
73	350	4.5	3	0	240
73.5	360	5.4	4	0	247.5
74	370	5.2	4	0	257.5
74.5	380	4.9	5	0	267.5

75.5	390	4.5	5	0	275
77	400	4	6	0	285
95.5	410	4.2	3	0	297.5
96	420	4.8	3	0	315
96.5	430	4.7	3	30	315
97	440	4.5	3	30	317.5
97.5	450	4.2	3	32.5	320
98	460	4.2	3	35	322.5
98.5	470	4.2	3	37.5	325
99	480	4.2	3	40	327.5
99.5	490	4.2	3	42.5	327.5

C35_P33_TYSN

Table B.10 Comprehensive datasheet for C35_33_TYSN

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.1	5	0	0
0.5	10	19.2	5	0	0
1	20	15.9	6	0	0
1.5	30	12.2	6	0	0
2	40	8.4	7	0	0
2.5	50	7.7	7	0	0
3	60	6.9	8	0	0.5
3.5	70	6.3	8	0	5
4	80	5.8	9	0	12.5
4.5	90	5.8	9	0	19
24	115	3.7	4	0	37.5
24.5	125	4.6	4	0	45
25	135	5.3	5	0	55
25.5	145	5.4	5	0	62.5
26	155	6	6	0	75
26.5	165	5.3	6	0	82.5
27	175	5.6	6	0	92.5
27.5	185	6.2	7	0	102.5
28	195	5.7	7	0	112.5
28.5	205	5.6	7	0	122.5
29	215	5.9	7	0	130
47.5	225	5.6	4	0	142.5
48	235	5	4	0	150
48.5	245	5.4	4	0	162.5
49	255	6	5	0	177.5

49.5	265	6	5	0	190
50	275	6.8	5	0	205
50.5	285	6.7	6	0	215
51	300	6.6	6	0	222.5
51.5	310	6.7	7	0	227.5
52	320	6.6	7	0	232.5
52.5	330	6.7	7	0	240
72.5	340	4.3	3	0	267.5
73	350	5.2	3	0	272.5
73.5	360	5.6	4	0	282.5
74	370	5.2	4	0	290
74.5	380	4.9	5	0	300
75.5	390	4.4	5	0	310
77	400	4	5	0	320
95.5	410	5.1	3	0	335
96	420	4.9	3	10	337.5
96.5	430	4.8	3	17	337.5
97	440	4.8	3	26	342.5
97.5	450	4.5	3	32.5	346.5
98	460	4.5	3	40	347.5
98.5	470	4.5	3	47.5	347.5

C35_P33_TNSY

Table B.11 Comprehensive datasheet for C35_33_TNSY

Time (h)	Total solution added (mL)	Cell Temp. (°C)	Amperage (mA)	Cumulative anolyte (mL)	Cumulative catholyte (mL)
0	0	20.1	6	0	0
0.5	10	18.5	6	0	0
1	20	14.5	6	0	0
1.5	30	10	6	0	0
2	40	7.6	6	0	0
2.5	50	5.5	6	0	0
3	60	5.9	7	0	0.5
3.5	70	5.6	8	0	4
4	80	4.4	8	0	7.5
4.5	90	5.2	8	0	13
24	115	3.7	4	0	35
24.5	125	5.2	4	0	37.5
25	135	5.1	6	0	45
25.5	145	5.6	7	0	55
26	155	5.8	7	0	65

26.5	165	5.5	7	0	75
27	175	5.1	7	0	85
27.5	185	5.8	7	0	95
28	195	5.2	7	0	105
28.5	205	5.1	8	0	115
29	215	5	4	0	123
47.5	225	4.5	5	0	135
48	235	5.5	7	0	140
48.5	245	5	8	0	145
49	255	6.1	8	0	157.5
49.5	265	6	8	0	172.5
50	275	7	8	0	185
50.5	285	6.2	9	0	195
51	300	6.3	9	0	202.5
51.5	310	6.3	9	0	210
52	320	6.3	9	0	217.5
52.5	330	6.2	8	0	225
72.5	340	4.4	4	0	240
73	350	4.5	4	0	241
73.5	360	5.4	4	0	247.5
74	370	5.1	6	0	251
74.5	380	4.9	6	0	260
75.5	390	4.5	7	0	269
77	400	4	8	0	277.5
95.5	410	4.1	4	0	287.5
96	420	4.5	4	7.5	305
96.5	430	4.4	4	12.5	305
97	440	4.4	4	20	306.5
97.5	450	4.5	4	27.5	310
98	460	4.4	4	35	312.5
98.5	470	4.4	4	42.5	312.5
99.5	480	2.9	2	50	315

Appendix C: GC8890 setup

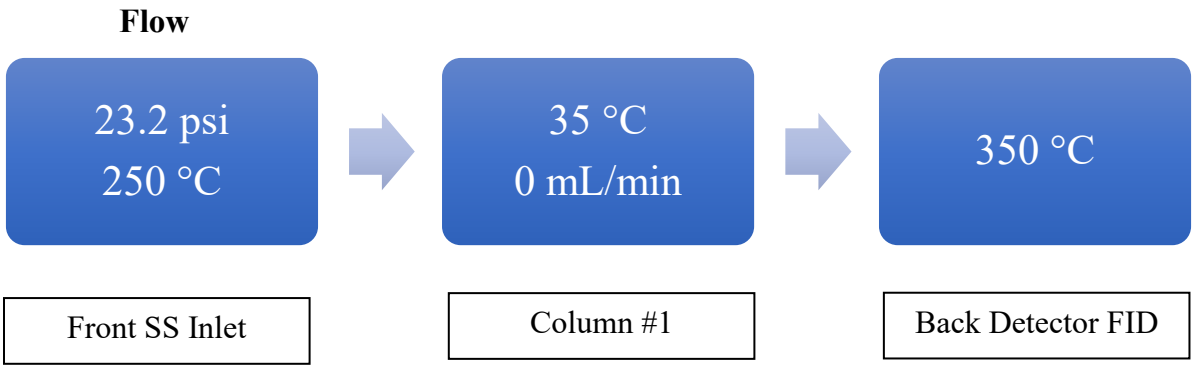


Figure C.1 Front inlet flow path in GC

Columns

Table C.1 GC column settings

Flow	Pressure	Average velocity	Holdup time	Flow type
3 mL/min	16.371 psi	68.279 cm/sec	0.36615 min	constant

Oven

Table C.2 GC oven settings

Equilibration time	Maximum oven temperature
2 min	350 °C

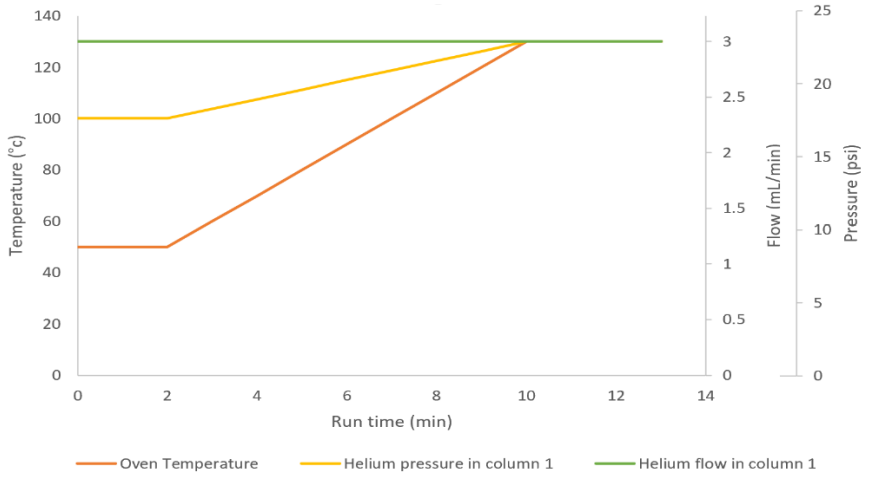


Figure C.2 GC oven setup

Detectors

Back detector FID

Table C.3 Back detector settings

Heater	Airflow	H_2 fuel flow	Makeup flow (He)
300 °C	300 mL/min	30 mL/min	30 mL/min