FMH606-1 24V Master's Thesis

Process Technology

Designing a lab-scale apparatus for reclaiming amines

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Summary:

Reactive absorption in amines stands as a mature and economically viable technology for CO_2 capture in industrial processes. However, the efficiency of this method declines over time due to the degradation of the amine solvent, necessitating reclaiming processes to maintain performance. This thesis investigates amine reclaiming technologies by employing a wide range of scientific papers and focuses on developing a lab-scale apparatus for efficient amine reclamation. Among thermal, electrodialysis (ED) and ion exchange technologies, electrodialysis emerges as an appropriate option, particularly when targeting heat stable salts (HSS) removal. It has lower energy demand than thermal reclamation and lower maintenance and chemical costs in comparison with ion exchange. Additionally, it has high HSS removal efficiency and low amine loss. By investigating the ED configurations, the suggested lab-scale ED design, which has the flexibility to be used under the laboratory hood, is presented. It is a two-stage ED unit with C-A and BP-A stack configurations. This apparatus demonstrates significant HSS removal efficiency, reaching over 96%, while minimizing amine loss to less than 4% after approximately 6 hours of operation. By increasing operation time, cell pairs, HSS concentrations, and current intensity, along with the use of reinforced homogeneous membranes with moderate thickness, this apparatus can be optimized. A cost analysis for this apparatus estimates an investment of $ 24,365.43 with yearly fixed costs of $ 4,364.99.

Preface

This master's thesis is submitted to the University of South-Eastern Norway (USN) to fulfill partial requirements for the degree of Master in Process Technology (PT). The research presented herein was conducted under the supervision of Kjell-Arne Solli, associate professor, and in collaboration with the USN research group 'Energy and CO_2 Capture (ECC)', a part of URGENT.

The thesis comprises seven chapters in which a review and comparison of amine reclamation technologies are conducted with the aim of selecting an appropriate method that can be used for construction a lab-scale apparatus with optimal efficiency. Throughout this work, a variety of valuable scientific papers were employed for literature reviews, analytical methods, apparatus design, and process optimization.

I extend my sincere gratitude to Kjell-Arne Solli for his invaluable guidance and support throughout this endeavor. I would also like to thank the members of the USN research group 'Energy and CO_2 Capture (ECC)' for their collaboration and insights.

Additionally, I am deeply grateful to my husband for his unwavering support, understanding, and encouragement during the course of this research.

It should be noted that the picture on the cover page shows the solvent's color shifting rapidly upon exposure to flue gas, progressing from clear to yellow, then orange, and finally dark brown. After reclamation, it returned to a color resembling its fresh state at the campaign's start [1].

Porsgrunn, 10th of May 2024

Nasrin Hosseini

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Nomenclature

MEA	Monoethanolamine
HSS	Heat stable salt
ED	Electrodialysis
PCC	Post-combustion carbon dioxide capture
CEM	Cation exchange membrane
AEM	Anion exchange membrane
BPM	Bipolar membrane
C-A	Repeated units of a cation and an anion exchange membrane
C-A-A	Repeated units of a cation and two anion exchange membranes
BP-A	Repeated units of a bipolar and an anion exchange membrane
DC	Direct electrical potential

1 Introduction

This chapter provides an overview of the background, objectives, methods, and report structure of this master's thesis, aimed at providing the reader with a good understanding of the project.

1.1 Background

The most mature technology for CO_2 capture is reactive absorption in amine. This method has proven to be effective and economically feasible, with operational plants successfully capturing carbon dioxide from various industrial processes. However, a significant challenge arises over time due to the degradation of the amine solvent. As the solvent comes into contact with CO_2, it undergoes chemical reactions, leading to the formation of degradation products that reduce its efficiency over time.

To address this issue and maintain the performance of CO_2 capture plants, reclaiming process is implemented. Reclaiming involves the separation of these degradation products from the amine solvent and restoring its effectiveness. This process could be essential for the long-term viability and efficiency of CO_2 capture technologies. By utilizing lab-scale equipment, researchers can conduct experiments to better understand the dynamics of amine degradation and the efficient methods for amine reclamation.

1.2 Objectives

This study aims to conduct research on amine reclamation technologies and identify an efficient solution that offers high amine recovery and contaminants removal efficiency at a laboratory scale.

- The primary goal of this investigation is to develop a lab-scale apparatus for amine reclamation. This apparatus will be designed to fit conveniently under laboratory hoods, facilitating the easy installation of analytical sensors for measuring spectroscopy data, pH levels, conductivity, and etc. Moreover, it will allow for the withdraw of samples for further analysis.

The scope of this project includes several important aspects:

- First, selecting an appropriate amine reclamation technology. This involves evaluating available options to determine a suitable setup based on factors such as energy efficiency, contaminants removal capabilities, high amine recovery, and cost-effectiveness.
- Following the technology selection, the project will focus on designing a lab-scale apparatus unit and creating detailed equipment drawings.
- Another aspect of the project involves estimating the efficiency of the separation process and amine recovery, as well as investigating the effects of some parameters on the process.
- Lastly, the project will include a cost estimation for the required equipment. This will involve considering the costs of equipment, chemicals, and any other fixed expenses associated with the implementation of the selected technology. The goal is to provide a

clear understanding of the financial implications and feasibility of the selected approach.

1.3 Methods

In this study, a diverse range of valuable research papers, which serve as the foundation for the literature review and analytical framework, are employed. Additionally, CorelDRAW and PowerPoint software are used for drawing some figures.

1.4 Report structure

In chapter 1, the introduction sets the stage by providing a brief background on the absorption of CO_2 in amine and emphasizes the significance of lab-scale amine reclamation. This chapter delves into the objectives, methods, and report structure.

In chapter 2, the greenhouse effects, global warming's causes, and efforts to mitigate carbon emissions through technologies like post-combustion carbon dioxide capture (PCC) plants are discussed.

In chapter 3, alkanolamines' role in CO_2 absorption and their challenges with thermal and oxidative degradation, leading to the formation of heat-stable salts (HSS) are discussed. It also delves into amine reclaiming technologies like thermal reclamation, ion exchange, and electrodialysis, highlighting their processes, advantages, and drawbacks in amine reclamation.

In chapter 4, the focus shifts to explores electrodialysis (ED) as a method for amine reclamation. It delves into ion-exchange membrane types and configurations, including C-A, C-A-A, and BP-A setups.

Chapter 5 presents a proposed apparatus design specifically tailored for laboratory use, and the detail design considerations, components, and specifications of the lab-scale apparatus.

Chapter 6 delves into important parameters influencing ED processes, including HSS removal efficiency, amine loss, CO_2-loading effects, current density, pH impact and cost estimation.

Chapter 7 provides a summary and drawing the key insights and conclusions from the previous chapters.

2 The global warming challenge

Greenhouse gases like carbon dioxide absorb radiation from the Earth's surface, which leads to an increase in temperature. This greenhouse effect naturally warms the earth by around 30°C on average. Without this greenhouse effect, the average surface temperature on Earth would be below 0°C. Global warming is caused by a rise in greenhouse gases in the atmosphere due to human activities like burning fossil fuels. Even a small increase in the global average temperature from this human-caused rise in greenhouse gases is thought to have serious consequences for ecosystems around the world [2]. Researchers are currently focusing on the development of low-emission technologies in order to reduce the carbon footprint of existing energy and industrial infrastructure. This shift creates space for the replacement of aging and polluting systems with more sustainable alternatives.

2.1 Post-combustion carbon dioxide capture (PCC)

One of the key technologies being developed is post-combustion carbon dioxide capture (PCC) plants [3]. PCC is recognized as the technologically mature and effective method to prevent CO_2 emissions into the atmosphere from fossil fuel-fired power plants. The chemical absorption technology, based on amines, has been researched for decades and was the initial technology to be commercialized for gas purification [4]. Amine scrubbing is the process of capturing CO_2 using an aqueous amine solution. In a typical amine scrubbing system designed for a power plant, as depicted in Figure 2.1, flue gas is passed through a scrubber containing the aqueous amine. In this stage, the amine absorbs the CO_2. The CO_2-rich amine is then transferred to a stripper, where it is heated, typically using steam from the plant's steam cycle. This step releases the CO_2 from the solution, which is subsequently compressed for transportation and sequestration [5]. Increasing the temperature in the desorber can enhance the separation of amine from CO_2, but it also leads to thermal and thermal-oxidative degradation. This degradation occurs at temperatures above 110-150°C [6].
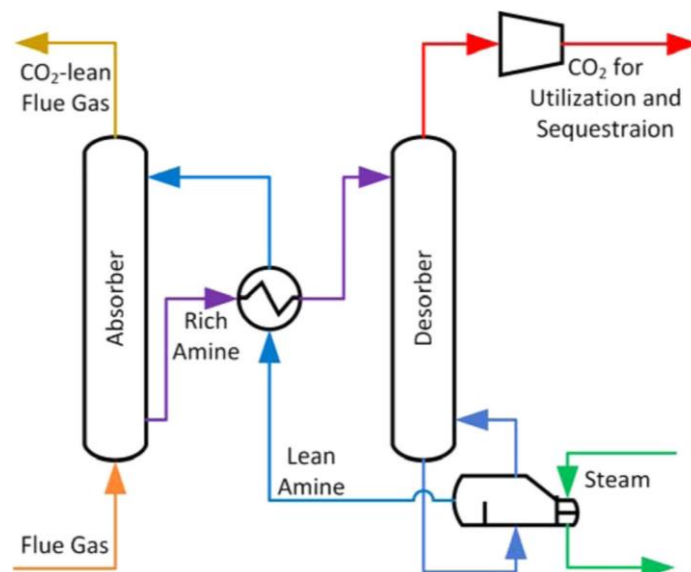

Figure 2.1: Amine-based CO_2 capture plant [5].

3 Amine reclaiming technologies

Ammonia (NH_3) stands as the simplest form of amine, wherein the nitrogen atom at its core is linked to three hydrogen atoms, each sharing identical chemical properties. Alkanolamines are substances derived from ammonia where one or more hydrogen atoms have been substituted with alkyl (R) or aryl (Ar) groups. They are categorized into three types, as illustrated in Figure 3.1, based on the number of hydrogen atoms attached to the nitrogen atom. Primary amines (e.g., monoethanolamine (MEA), 2-amino-2-methyl-1-propanol (AMP)) have nitrogen atoms bonded to two hydrogen atoms, secondary amines (e.g., diethanolamine (DEA), di-isopropanolamine (DIPA)) have nitrogen atoms bonded to one hydrogen atom, and tertiary amines (e.g., N-methyldiethanolamine (MDEA), triethanolamine (TEA)) have nitrogen atoms bonded to no hydrogen atoms [2], [7].
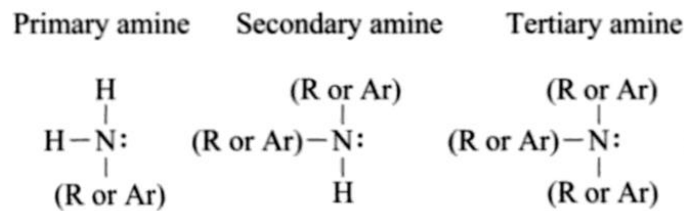
Figure 3.1: Illustrates the structures of primary, secondary and tertiary amines [8].

The chemical process of absorbing CO_2 into amine solutions at low temperatures and then releasing it through heating is complex and varies depending on the type of alkanolamine used. Unhindered (primary or secondary) amines typically create stable carbamates, resulting in the absorption of only half a mole of CO_2 per mole of amine (Reaction (3.1)). Upon heating, these carbamates break down to release CO_2 and regenerate the amine. However, breaking the stable carbamate bonds requires a considerable amount of heat energy [7].

$$2R^1R^2R^3NH + CO_2 \rightarrow R^1R^2NCOO^- + R^1R^2NH_2^+ \qquad 3.1)$$
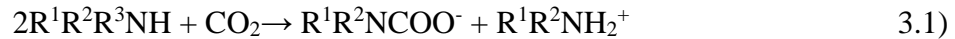
Hindered (tertiary) amines, on the other hand, form unstable carbamates, and an alternative reaction generates bicarbonate ions. This allows tertiary amines to theoretically absorb one mole of CO_2 per mole of amine (Reaction (3.2)) with less heat energy required for regeneration compared to unhindered amines. However, the slow absorption rates of tertiary amines make them less practical for CO_2 removal [7].

$$R^1R^2R^3N + CO_2 + H_2O \rightarrow R^1R^2R^3NH_3^+ + HCO_3^- \qquad (3.2)$$

The chemical reactivity is commonly observed to decrease as follows: primary ($-NH_2$) > secondary (>NH) > tertiary (>N$-$) [2]. The most commonly employed and economically viable absorbent is MEA, which is a primary alkanolamine. MEA is preferred for its quick reaction kinetics in CO_2 absorption, a relatively high capacity to absorb CO_2, strong selectivity for CO_2 over other gases, and the low viscosity of its aqueous solution [4], [9], [10].

Alkanolamine are subject to solvent degradation due to heat exposure (thermal degradation) and oxygen presence (oxidative degradation) [11]. In the following paragraphs, they are investigated in more detail.

Thermal degradation

Investigations into the thermal degradation of alkanolamines during post-combustion carbon capture have been conducted in recent decades. This degradation, known as carbamate polymerization, primarily occurs in the stripper section at elevated temperatures, but it can also happen in the heat exchanger and reclaimer sections. Although the exact temperature for these reactions is not precisely determined, studies generally report temperatures between 100 and 200°C, which tend to rise with increasing CO_2 levels.

In MEA solvents, common thermal degradation products include 2-oxazolidinone (OZD), MEA urea, 1-(2-hydroxyethyl) imidazolidone (HEIA), N-(2-hydroxethyl) ethylenediamine (HEEDA), N-(2-hydroxyethyl) imidazole (HEI), N-(2-aminoethyl)-N'-(2-hydroxyethyl) imidazolidin-2-one (AHEIA), and N-(2-hydroxethyl) ethylenediamine (HEEDA) [11], [12].

Oxidative degradation

Based on this [12] research, oxidative degradation during CO_2 absorption processes, typically at temperatures around 55°C under oxidizing conditions, is a significant cause of solvent loss in flue gas with 3%–5% oxygen content. Alongside oxygen, NO_x, SO_x, free radicals, and metal ions also contribute to this degradation. It leads to the breakdown of alkanolamine molecules and the formation of HSS. HSS are formed when alkanolamines react with acids that are notably stronger than CO_2. The primary mechanisms involve two processes: direct oxidation of alkanolamines into organic acids and the indirect interaction of oxygen and other oxidizing species, leading to the formation of intermediate products. These intermediates then react with amines to produce additional degradation compounds. The degradation process begins with the radical loss of electrons from nitrogen in alkanolamines or the loss of hydrogen from carbon atoms to nitrogen.

The resulting degradation products of alkanolamines fall into two categories: primary and secondary species. Primary products include aldehydes, ammonia, amines, amides, carboxylic acids, and organic acids. Secondary products, formed from primary ones, consist of formats, oxalates, acetates, succinates, and glycolates, such as N-(2-hydroxyethyl) glycine, N,N'-bis (2-hydroxyethyl) oxalamide, N-(2-hydroxyethyl) acetamide, 1-(2-hydroxyethyl) piperazine-2-one, 2-hydrox-y-N-(2-hydroxyethyl) acetamide, and 1-(2-hydroxyethyl) piperazine-2-one. Acetate and formate are products of MEA, while N-(2-hydroxyethyl) acetamide and N-(2-hydroxyethyl) formamide derive from acetic and formic acids, respectively. Glycolic acid aids in the formation of HSS with MEA, although its amide form tends to hydrolyze into glycolates and MEA. Oxalic acid acts primarily as an intermediary, breaking down into formic acid and producing stable N-(2-hydroxyethyl) formamide. Similarly, succinic acid transforms through an intermediary species of N-(2-hydroxyethyl) succinamide into a stable N-(2-hydroxyethyl) succinimide.

HSSs cannot naturally be removed from the stripper during regular operation. Consequently, they accumulate in the solvent loop over time. Based on these researches [13]. [9], This accumulation necessitates specific actions due to several reasons:

- The amine solvent's ability to absorb CO_2 decreases as HSS build up, affecting the overall performance of the system.

- The properties of the solvent change, notably its viscosity increases, which reduces mass transfer efficiency and raises solvent circulation costs.

- HSS has a chelating effect, leading to equipment corrosion. Additionally, dissolved metals like iron, chromium, and nickel can accelerate oxidative degradation.

- HSS and corrosion byproducts create stable solvent foam, decreasing the gas-liquid surface area.

To mitigate the impact of unwanted impurities and minimize operational and maintenance issues, several options exist for controlling contaminants like solution purging known as bleed and feed, neutralization, and amine reclamation. Bleed and feed, a common method in solvent management, involves the periodic purging of contaminated solvent and its replacement with fresh solvent. While this approach helps in controlling degradation, it comes with drawbacks such as wastage of amine and associated disposal costs. Neutralization, on the other hand, is a technique used to convert amine HSS into sodium or potassium HSS by introducing NaOH or KOH. While this process does maintain the capacity of the amine, it does not address the reduction of salt content in the solvent. As time progresses, the amine becomes increasingly contaminated with these salts, which can lead to issues such as increased solvent density and viscosity [11].

Despite the usefulness of bleed and feed and neutralization, they fall short in providing long-term solutions to contamination problems. This has led to the exploration of more sustainable approaches, such as amine reclamation. The focus of amine reclamation is to purify solvents by effectively separating degradation products from the amine.

Some significant methods of amine reclamation have been developed, including thermal reclamation (distillation), ion exchange, and electrodialysis.

3.1 Thermal reclamation

In thermal reclamation technology, the aim is to separate the amine from other high-boiling degraded components and impurities using heat energy. As shown in Figure 3.2, a slipstream is fed into the reclaimer, typically with the addition of high-pressure steam and either soda ash or caustic soda to the system. Soda ash aids in releasing amines from HSS. The purified amine exits the reclaimer and returns to the process, while waste is extracted through the drain at the bottom of the reclaimer. Before the degraded amine enters the reclaimer, it can be treated with a strong base like sodium carbonate or sodium hydroxide to prevent the distillation of any stronger acids and to liberate the amine from the degraded solution. The amount of the strong base should be slightly more or less than stoichiometric; if it exceeds this amount, it can cause an increase in waste. In the conventional reclamation method that relies solely on heating and evaporation, a portion of the absorbent component may persist in the reclaimer bottom sludge. The bottom liquid, primarily recycled, is reheated and returned for further processing [10]. Operating conditions vary with solvent types, in the Table 3.1 the decomposition temperatures and boiling points for MEA, DEA and MDEA are shown [14].

Modern thermal reclaimers typically operate at atmospheric pressure or under vacuum conditions. Vacuum distillation is specifically designed for reclaiming high-boiling-point amine solvents, which are prone to decomposition at high temperatures [10]. While degraded secondary and tertiary amines can be thermally recovered under vacuum pressure, with operating temperatures kept below their decomposition thresholds to prevent further

degradation, primary amines are normally recovered under atmospheric pressure or slightly above conditions [14].
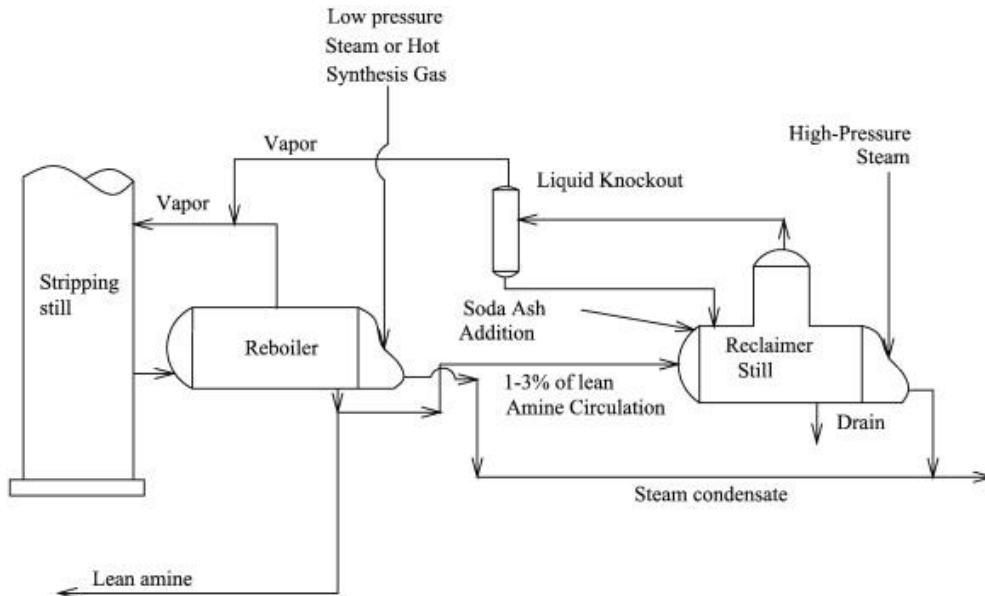
Figure 3.2: Classic amine thermal reclamation setup [10].

Table 3.1: Comparative data on amines' decomposition temperatures and boiling points [14].

Amines	Decomposition temperature (°C)	Boiling point (°C)
MEA	147	171
DEA	204	268
MDEA	182	247

3.2 Ion exchange

Ion exchange is a reversible process that involves exchanging ions from a solid to a liquid without appreciably changing the solid's structural makeup. This solid substance usually comprises of particles or resins for ion exchange. Ion exchange takes place between a liquid and a solid phase, and its process is similar to neutralization with a strong base [10]. The contaminated solution passes through resin beds where cation beds remove positively charged impurities (like ferrous and sodium ions) by exchanging them with protons. Anion beds then remove anions such as chloride, acetate, and sulfate by exchanging them with hydroxide ions [15].

Equations (3.3) and (3.4) describe the loading and regeneration step, which are the two primary processes in a typical ion exchange process. During loading, unwanted HSS anions undergo bonding with the ion exchange resin, typically pre-treated with a strong base like sodium

hydroxide (NaOH), resulting in the liberation of corresponding amines and the generation of water. Subsequently, in the regeneration phase, the introduction of a strong base (e.g., sodium hydroxide) aids in releasing the bonded resin anion, effectively removing HSS anions from the resin system [10].

Loading: $AmineH^+ + Resin^+OH^- + HSS^- \rightarrow Amine + H_2O + Resin^+HSS^-$ (3.3)

Regeneration: $Resin^+ HSS^- + NaOH \rightarrow Resin^+OH^- + Na^+ HSS^-$ (3.4)
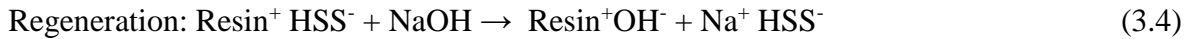
A common anion exchange resin is a mixture of styrene and divinyl benzene. Strong base anion (SBA) resins are usually treated with chloromethylation followed by amination. This creates a quaternary ammonium group as the fixed functional group, with hydroxide as the mobile ion that exchanges with the desired anion during absorption. Type I SBA resins use trimethylamine for amination, while Type II SBA resins use dimethylethanolamine. Studies have found that Type II SBA resins are easier to regenerate than Type I. Weakly acidic cationic resins perform better but are less stable than strongly acidic ones. Strong cation exchange resins are prone to irreversible fouling by ferrous iron, unlike weak cation ones. The process of ion exchange works best at lower temperatures (<45 °C), high temperatures degrade ion exchange resins, especially anionic ones, whose breakdown relates to the counterion type [15].

3.3 Electrodialysis

Electrodialysis units eliminate ionic degradation products through ion-exchange membranes while using direct current as a driving force. In an electrodialysis setup as depicted in Figure 3.3, each cell consists of cation-exchange and anion-exchange membranes separated by a spacer gasket along with two electrodes and a power supply. This forms the core of the system's operation.
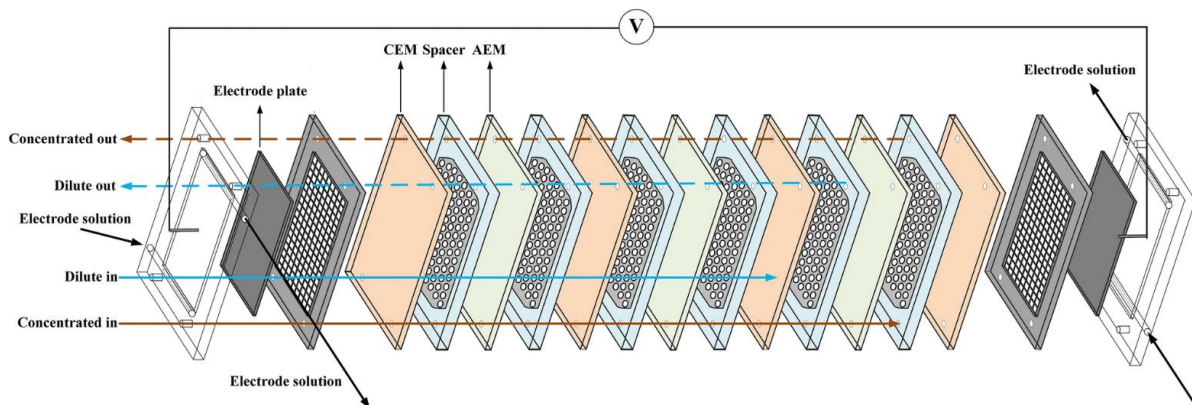
Figure 3.3: Provides a visual representation of the structure of an conventional ED stack [16].

As shown in Figure 3.4, when an electric potential is applied, positively charged ions (cations) in solution migrate towards the cathode, passing through the cation-exchange membrane but are blocked by the anion-exchange membrane. Negatively charged ions (anions) move towards the anode, passing through the anion-exchange membrane but are obstructed by the cation-exchange membrane. Through this selective mechanism, the solution in one compartment loses its ionic components, while the solution in another compartment accumulates more ionic components and becomes more concentrated. A cell pair, with a cation-exchange membrane, a

dilute cell, an anion-exchange membrane, and a concentrate cell, defines this setup. This method offers several benefits of membrane-based processes, including compact equipment size, modular design, operational flexibility, and scalability [9]. This technology will discuss more in details in the next chapter.
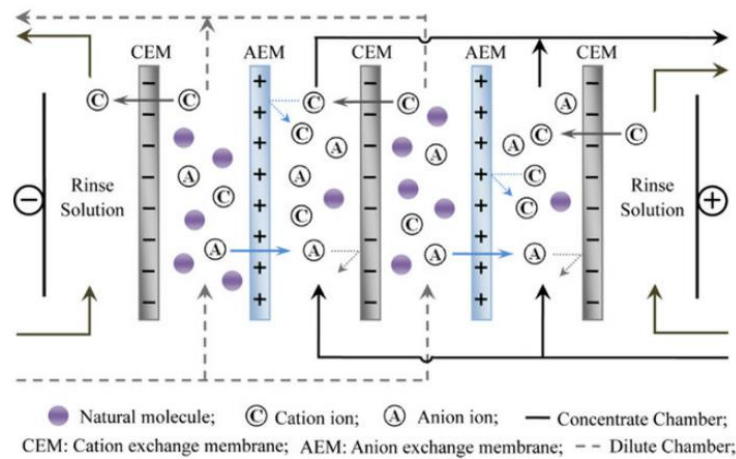
Figure 3.4: Presents the procedure happening within a typical ED stack [17].

3.4 Comparison of technologies

There are some advantages and disadvantages associated with the three common amine reclamation technologies mentioned, some of which are outlined in Table 3.2. According to articles [9], [10], [14], [15], thermal reclamation offers the advantage of thoroughly eliminating contaminants from amine solutions, yet its energy consumption constitutes a substantial portion of operational costs in CO_2 capture plants. Conversely, ion exchange and ED operate at lower temperatures and are less energy-intensive techniques. Nonetheless, they come with certain drawbacks compared to thermal reclamation. For instance, the need to cool down the hot lean amine solution before applying it to ion exchange and ED. Moreover, both methods struggle with handling non-ionic contaminants and generate aqueous waste, necessitating disposal.

Ion exchange, though chemically straightforward and with low energy requirements, is most effective when HSS concentration is minimal. Achieving low salt concentrations is possible, but regenerating it requires significant amounts of acid and base, which adds to the overall cost. On the other hand, ED consumes fewer chemicals and less water than ion exchange, and it does not affect the concentration in the feed, but it demands a higher energy input.

ED technology stands as a competitive option alongside thermal technique for reclaiming high boiling point amines. One challenge with ED lies in the selectivity of ion exchange membranes operating under high pH conditions. Additionally, membrane fouling due to solid impurities in the amine solution presents an operational difficulty for ED. However, the resulting waste from the ED treatment method is non-toxic, unlike the waste produced by thermal reclamation processes.

Table 3.2: Comparison of three main technologies for amine reclamation.

Parameters	Thermal reclamation	Ion exchange	Electrodialysis	Ref.
Operation temperature	120-140°C	40-60°C	40-60°C	[14]
Waste	Small volume of concentrated waste, hazardous	A large volume of aqueous waste	A moderate volume of aqueous waste, non-hazardous	[10], [14]
Chemical usage	NaOH	NaOH and H_2SO_4 for resin regeneration	NaOH	[10]
Energy demand	High	Low	Moderate	[10]
Overall amine recovery	85%–95%	99%	98%	[10]
Advantages	Enabling the removal of all contaminants, highly concentrated waste	Ideal for low feed salt concentration, capable of achieving very low product salt concentrations, maintains low energy consumption levels	Efficient for charged species; not affected by concentration in feed; lower chemical consumption	[10]
Challenges	Purification of high boiling point amines, high cost associated, low amine recovery rate, energy-intensive process, vacuum required for most amines, risk of further thermal degradation	Unable to remove non-ionic contaminants, high salt levels result in frequent bed exhaustion and regeneration, expensive when scaled up to large plant sizes, prone to resin fouling	Inability to eliminate non-ionic contaminants, considerations for membrane selection and durability, risk of membrane fouling and damage due to exposure to high pH levels	[10] [14]

4 Electrodialysis fundamentals and stack configurations

Based on the findings from the previous chapter and with a focus on the removal of HSS, electrodialysis is selected as the amine reclaiming technology for this thesis due to some advantages over other techniques, both practical and financial. Unlike solvent change-out or bleed and feed methods, ED offers greater efficiency and cost-effectiveness. Concerns about high utility and chemical consumption, as well as potential hazardous waste disposal associated with ion exchange and thermal reclamation, along with its compact equipment size, modular design, and scalability, led to the selection of ED as the appropriate technology for amine reclamation.

In section 3.3, the main parts and principles of an ED were discussed. In this chapter, the role of ion-exchange membranes and their configurations within the electrodialysis unit are investigated to provide a detailed understanding of this ED technology.

4.1 Ion-exchange membranes

There are two main types of ion-exchange membranes: cation-exchange membranes (CEM) and anion-exchange membranes (AEM). CEM typically consist of sulfonic acid groups, phosphoric acid groups, sulfonamides, and azole derivatives. A range of polymer materials, such as poly (ether sulfone) (PES), poly (ether ketone) (PEK), polybenzimidazole (PBI), polyimide (PI), poly (phenylene), polyphosphazene, and polyvinylidene fluoride (PVDF), have been studied as the main structures for CEMs [17]. With negative charge groups fixed to the polymer matrix, CEMs allow the movement of mobile cations, known as counter-ions, while excluding anions, making them selectively permeable to cations [18]. AEMs typically consist of quaternized ammonium (QA) groups and tertiary amines [17]. AEMs, with their fixed positive charges on the polymer matrix, exclude cations and promote the permeability of mobile anions, known as co-ions.

As discussed in this [18] article, cation- and anion-exchange membranes are grouped into different types: strong acid (include sulfonic acid as their charged groups), weak acid (include carboxylic acid as their charged groups), strong base (like quaternary amines), or weak base (like tertiary amines). Membranes can also be sorted by their structure: homogeneous membranes have integrated ion-exchange groups for evenness, while heterogeneous membranes mix ion-exchange resin powder with a binder polymer, leading to uneven distribution (see Figure 4.1). Ion-exchange membranes have specific properties influenced by factors like polymer network density, matrix polymer's affinity for water (hydrophobic or hydrophilic character), the type and amount of fixed charges, and overall structure. Ideal properties include high selectivity for certain ions, low electrical resistance, good durability, high chemical and thermal stability, and affordability. Heterogeneous membranes have higher electrical resistance due to longer ion pathways and less selectivity, allowing some ions through unintended gaps. Despite their lower production costs, heterogeneous membrane structures typically show reduced permselectivity.

In membrane separation processes, concentration polarization arises when the rate at which solutes move toward the membrane exceeds their rate of transport through it. The concentration

gradient in the solution, driving diffusive transport. In electrodialysis, for example, the cation-exchange membrane facing the dilute solution reduces ion concentration at its surface, while the other side facing the concentrate solution increases ion concentration. Concentration polarization in electrodialysis can lead to ions accumulating on the membrane surface facing the concentrate cell, potentially causing salt precipitation if concentrations exceed solubility limits. In the dilute cell, concentration polarization results in ion depletion at the membrane surface, determining the limiting current density, where ion concentration at the membrane surface approaches zero. Once this limit is reached, further voltage increase doesn't significantly increase current, as shown in Figure 4.2. The curve in the diagram illustrates that the current and resistances change drastically with applied voltage. Initially, resistance is constant, increasing linearly with voltage (Ohm's law). At the limiting current density, cell resistance suddenly increases, and further voltage increase does not significantly raise current density until a certain voltage, termed overlimiting current density apply, where current density increases again. This overlimiting current density is caused by electroconvection, partly due to H^+ and OH^- ions generated at the membrane and solution interface by water dissociation. This water dissociation affects current usage and can lead to drastic pH changes; decrease in the dilute, and increase in the concentrate solutions.
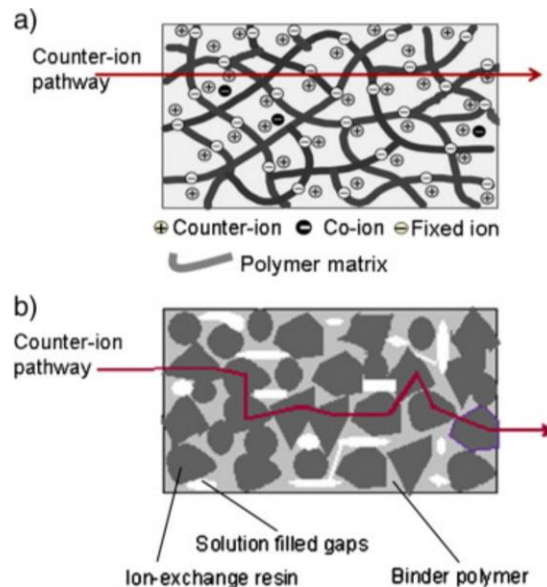
Figure 4.1: Showing a) cation-exchange membrane structured homogeneously and b) an ion-exchange membrane formed heterogeneously using ion-exchange resin powder and a binder polymer [18].
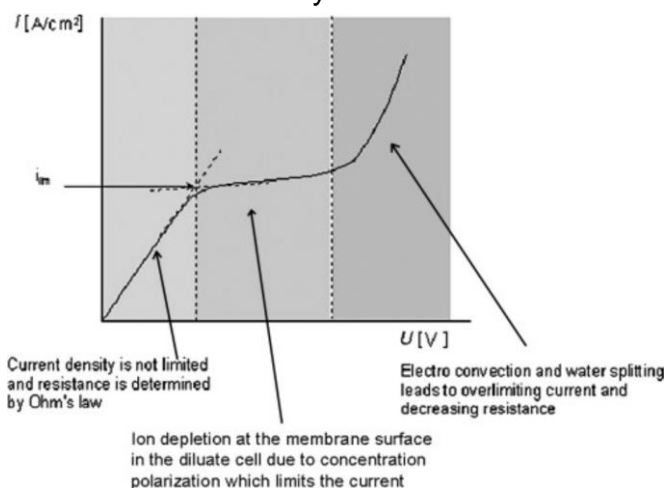
Figure 4.2: Showing current vs. voltage for an electrodialysis stack with constant flow and concentrations, marking limiting and overlimiting current density points [18].

4.2 ED stack configurations

The arrangement of the electrodialysis stack is a crucial factor that influences its efficiency and effectiveness in various applications, especially in processes like amine reclamation. Researchers have identified three common types of ED stack configurations that are employed for amine reclamation due to their distinct advantages and functionalities:

- C-A configuration (repeated units of a cation and an anion exchange membrane)
- C-A-A configuration (repeated units of a cation and two anion exchange membranes)
- BP-A configuration (repeated units of a bipolar and an anion exchange membrane).

In the following subchapters, we will delve deeper into each of these configuration.

4.2.1 C-A configuration

The typical configuration of ED stacks involves repeating units comprising a CEM and an AEM, as depicted in Figure 4.3, known as the C-A configuration. Upon applying voltage, cations like protonated amines and anions such as HSS^-, carbamates, and carbonates migrate from the dilute to the concentrate compartment, while purified amines move in the opposite direction.

However, within the C-A ED setup, the migration of amine in protonated and carbamate forms to the concentrate compartment leads to amine loss, raising concerns about process efficiency and cost-effectiveness. Additionally, the ion concentration in the compartments remains low due to the absence of supplemental ions, resulting in a consistent but relatively low current density. This low ion concentration presents challenges to ED efficiency as the driving force for ion migration relies on the ion concentration gradient [19].

One way to increase the efficiency of conventional ED using the C-A configuration is by using a two-stage ED unit, as presented in this [20] publication. The findings discuss a novel two-stage electrodialysis method for extracting heat-stable salts from monoethanolamine solvent, which is utilized in a post-combustion carbon capture pilot plant in Heilbronn, Germany. Two ED modules in an electrodialysis unit were evaluated in MEA solvent slipstreams with different

pilot plant operating times. Up to 65% of HSS anions were eliminated by the ED unit after 1 hour for the first stage and 1.5 hours for the second stage.
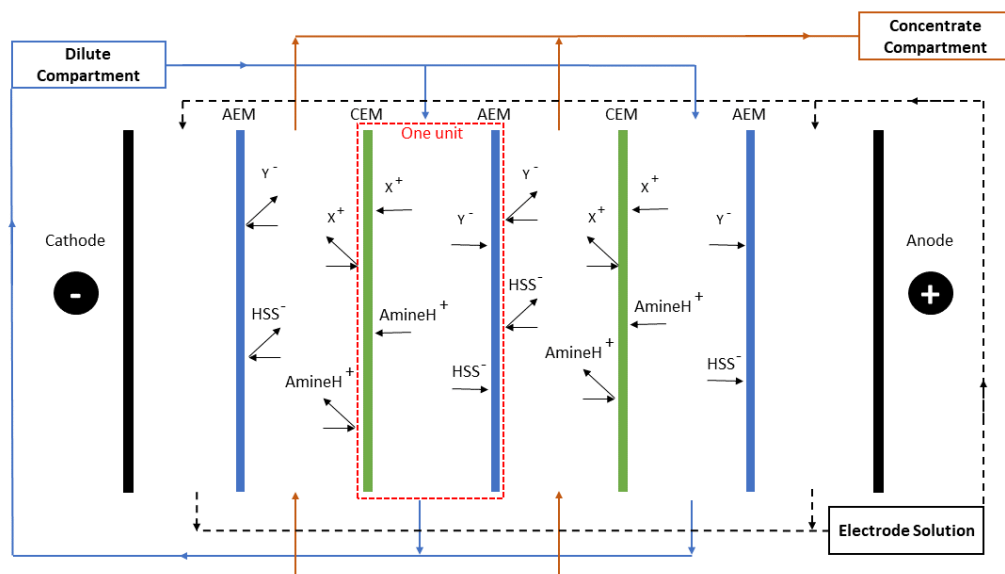
Figure 4.3: ED with C-A stack configuration.

4.2.2 C-A-A configuration

In response to the considerable loss of amine in the protonated amine and carbamate forms, researchers have explored alternative configurations to mitigate this issue. One such configuration is the C-A-A stack arrangement, which aims to address the loss of amine. As depicted in Figure 4.4, the C-A-A stack configuration introduces an additional AEM, resulting in a repeated unit consisting of a CEM, two AEMs, and two electrodes. This modified configuration introduces base compartments within the stack to neutralize the amine loss. The base compartments serve a crucial role in the process by providing hydroxide ions (OH^-) to react with the protonated amine. This reaction, as illustrated in Reaction (4.1), forms amine, thereby reducing the loss of amine in its protonated form [21].

$$OH^- + AmineH^+HSS^- \quad \rightarrow \quad HSS^- + Amine + H_2O \tag{4.1}$$

By introducing these base compartments and facilitating the conversion of protonated amine to amine, the C-A-A stack configuration shows promise in reducing the loss of amine.

A practical application of this setup is exemplified by Electros Technologies Inc. This company developed the ElectroSepTM technique, which selectively removes only anions from contaminated amine solutions. This is achieved by injecting a base into the electrodialysis stack rather than directly into the amine solution. Studies have shown that neutralizing the amine within the ED stack reduces amine loss and enables 99.5% overall amine recovery [10].

However, it is important to note that while this configuration addresses the loss of amine in its protonated form, amine in the carbamate form still faces the risk of being lost during the electrodialysis process. Despite this limitation, the C-A-A stack configuration represents a step forward in optimizing the efficiency [21].
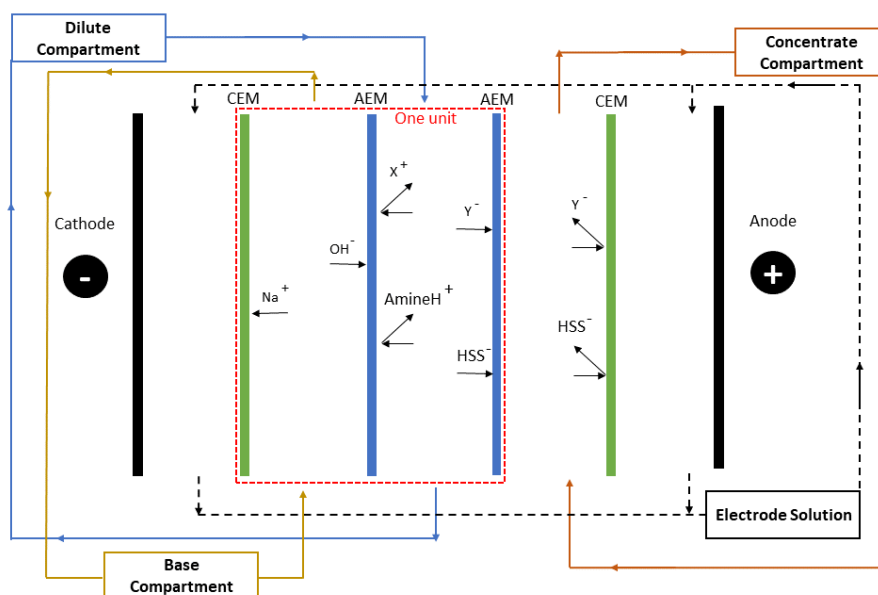
Figure 4.4: ED with C-A-A stack configuration.

4.2.3 BP-A configuration

In the ED stack configuration, it can be used a bipolar membrane (BPM), which dissociates water into H^+ and OH^- by applying a direct electrical potential (DC). The configuration of the BPM within the stack can vary based on the applications, but one commonly observed arrangement is the A-C-BP-A-C configuration, where the bipolar membrane is positioned between two units of anion and cation exchange membranes. In this standard arrangement, acid-base reactions occur. On one side of the BP-A segment, the H^+ ions interact with anions, forming acids as a result. Conversely, on the opposite side of the bipolar exchange membrane (C-BP), the OH^- ions interact with cations, giving rise to bases [18].

Among the practical stack configurations utilizing a bipolar membrane, the BP-A setup stands out for reducing amine loss. As shown in Figure 4.5, the OH^- ions migrate to the dilute compartment, where they react with protonated amines to yield amines and water. Simultaneously, the H^+ ions travel to the concentrate compartment, where they react with HSS^- to generate acids, following reactions (4.2 and 4.3) [21]:

$$OH^- + AmineH^+ \rightarrow Amine + H_2O \tag{4.2}$$

$$H^+ + HSS^- \rightarrow HSS \tag{4.3}$$
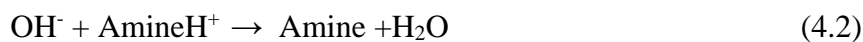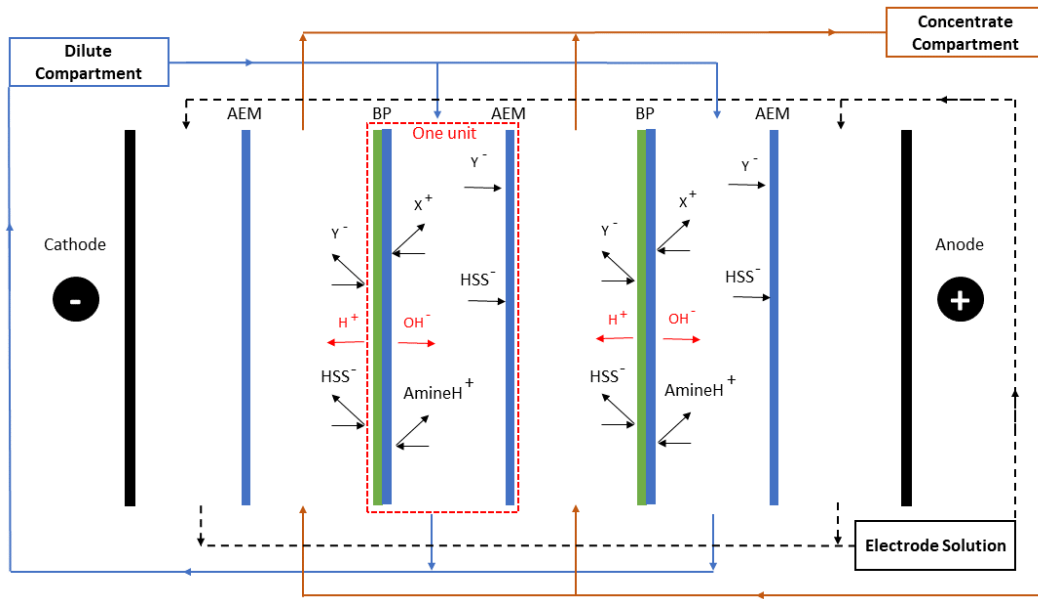
Figure 4.5: ED with BP-A stack configuration.

5 Design a lab-scale apparatus for amine reclamation

In reference to the experimental research outlined in this [21] article, a two-stage ED setup was developed for the reclamation of amine. The apparatus details are expounded upon in the following sections. As depicted in the Figure 5.1, the first stage employs an ED unit with a C-A membrane configuration, followed by the utilization of an ED unit with a BP-A configuration in the second stage.

In the initial stage, two beakers are allocated for distinct purposes—one for the dilute compartment and another for the concentrate compartment. Given that the electrode solution are same across both stages, a single beaker containing the electrode solution could be sufficed for both compartments. Additionally, six peristaltic pumps (three in first stage and three in second stage) facilitate operations in both stages. Moving to the second stage, separate beakers are designated for the dilute and concentrate (or acid compartment). Data collection can be efficiently managed with the use of a data logger, providing detailed insights into the process parameters and performance.

A degraded amine is introduced into the dilute compartment in the first stage, while deionized water is utilized in the concentrate compartment. For the electrode solution, 0.5 mol/L Na_2SO_4 is employed. Prior to applying voltage, the solutions circulate for 20 minutes, during which any observable gas bubbles are purged from each compartment. Subsequently, the electrodes are supplied with the appropriate voltage. The operation time is determined by the conductivity sensor, ceasing when the conductivity of the dilute solution reaches less than 1 µS/cm, at which point the pumps are deactivated and the primary purified amine is obtained.

For the next step and to mitigate CO_2-loading in the concentrate solution in the first stage, the addition of a strong acid is recommended. This treated solution then serves as the initial feed for both the dilute and concentrate compartments in the second stage. Following the application of voltage, the operation of the second stage commences. In this stage, as the concentrate compartment is utilized as feed, the final conductivity of the dilute solution should settle at a higher number, approximately less than 8 µS/cm, to complete the process.

It should be noted that ensuring the uniformity of solutions requires equipping each beaker with suitable laboratory stirrers and also the dilute beakers in the first and second stages should have sufficient space to accommodate analytical sensors. Moreover, covering all beakers with lids is recommended to safeguard against impurities and inaccuracies in data collection

The secondary stage's objective is to optimize the initial stage, minimize waste, and convert waste into a valuable material, specifically, a strong acid capable of regenerating the membranes and releasing CO_2-loading from the concentrate compartment in the second stage.
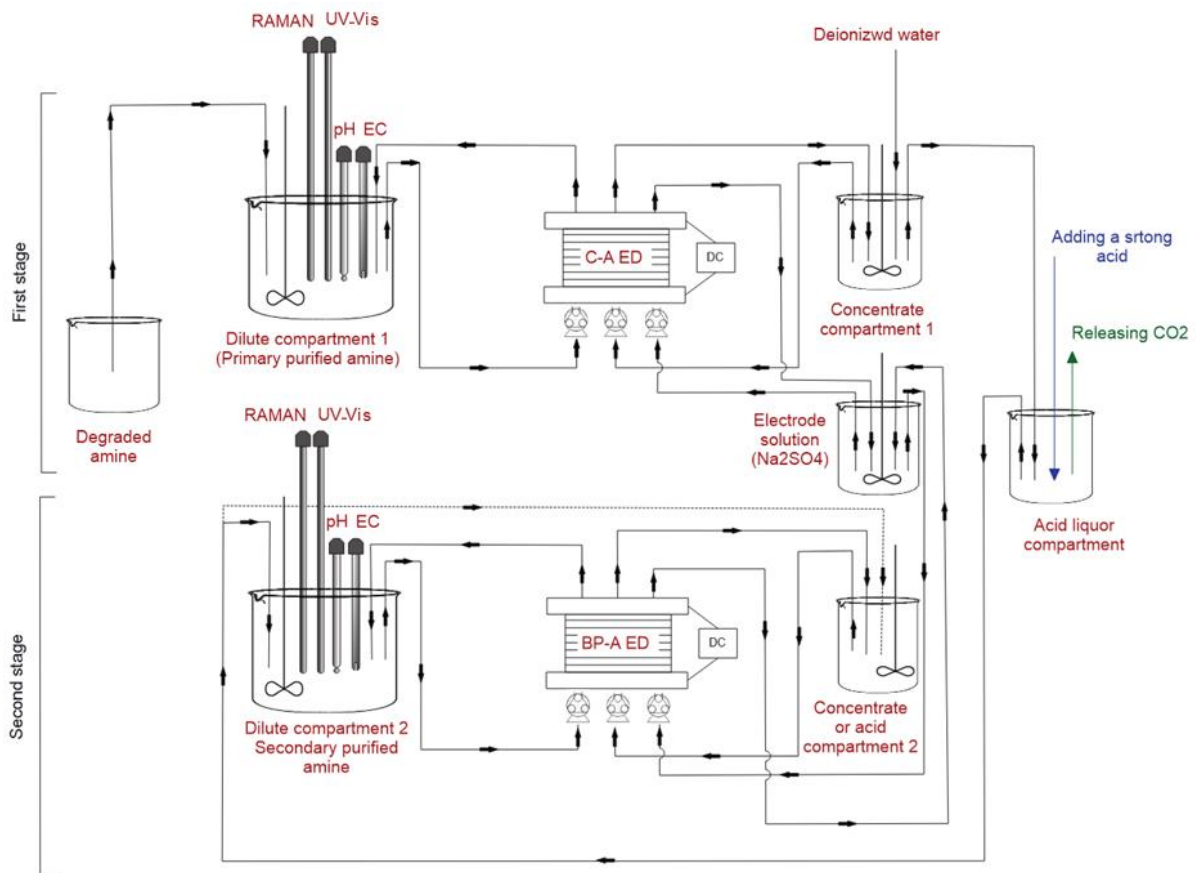
Figure 5.1:Illustrates a schematic of a two-stage laboratory ED unit.

5.1 Material

This section delves into the materials utilized in the proposed ED unit, such as membrane stacks, pumps, chemicals, tubes, beakers, and the DC power supply. Subsequent subchapters provide a deeper explanation of each component.

5.1.1 ED stack setup

Based on this [21] experimental research, the ED setup, configures with both C-A and BP-A arrangements, consist of several components. In each stack configuration, there are primarily two main plates: a cathode plate and an anode plate, both constructed using iridium material coated with titanium. Spacers are employed to separate the membranes, and these spacers are equipped with Viton gaskets to ensure proper sealing. In the C-A setup, there are a total of 11 anion exchange membranes and 10 cation exchange membranes, each covering an effective area of 55 cm^2. Additionally, there are 10 cells in this configuration, divided into 5 dilute cells and 5 concentrate cells. For the BP-A setup, there are also a total of 11 anion exchange membranes and 10 bipolar membranes. Apart from this difference, all other components and specifications remained the same as in the C-A setup. The membranes' specifications are described in Table 5.1.

Table 5.1: Membrane specifications for the designed apparatus [21].

Membrane	Thickness (μm)	Area (m^2)	Resistance (Ω.cm^2)	Burst strength (Mpa)	Operating temperature (° C)	Operating pH
CEM	150	0.0055	1.9	> 0.5	25-40	2-10
AEM	180	0.0055	2.3	> 0.5	25-40	2-10
BPM	280	0.0055	-	> 1.0	25-40	0-14

5.1.2 Pumps

As mentioned in the article [21], six peristaltic pumps with a maximum flow rate of 500 ml/min each are used for circulating the solutions through the membrane stack. Since the apparatus is intended to be installed under the laboratory hood, it is advisable to use one or two multi-channel pumps due to space limitations. It is recommended to maintain uniform flow rates across all streams to ensure the stable operation of the electrodialyzer.

5.1.3 Chemicals

According to [21], In the first dilute compartment, an initial volume of 1 liter of the lean blended amine solvent is utilized. It originates from samples taken at the absorber inlet of a 150 Kt/y coal-fired flue gas CO_2 capture demonstration conducted in Yulin, China. This solvent comprises approximately 30.55% amines and has a total HSS concentration slightly above 2000 ppm. Its CO_2-loading is 0.38 mole (CO_2) per mole (amine). The initial conductivity of this solvent measured 14.01 mS/cm, with a pH level recorded at 10.02. These parameters collectively characterize the composition of the solvent, detailed in Table 5.2.

Table 5.2: Indicates the compositions of initial degraded amine [21].

Components	Concentration
Amine I	6.95 wt.%
Amine II	23.60 wt.%
Glycolate (HOCH^{2}COO$^-$)	1465 ppm
Formate (HCOO$^-$)	366ppm
Chloride (CL$^-$)	14 ppm
Sulfate (SO$_4^{2-}$)	189 ppm
Oxalate (C2O$_4^{2-}$)	~5 ppm
Iron ion (Fe$^{2+/3+}$)	36 ppm
water (H$_2$O)	Bal

In the first concentrate compartment, an initial volume of 500 ml of deionized water is employed. For the electrode solution, 1000 ml (500 ml for the C-A stack and 500 ml for the BP-A stack) of 0.5 mol/L Na_2SO_4 is used.

5.1.4 Tubes and beakers

The suggested ED unit requires six beakers, each with a capacity of 1L, made of glass to ensure durability and chemical compatibility. To establish a closed system, it is recommended that all beakers used in the process be equipped with tightly sealed lids and customized with slots for sensors and stirrer insertion.

When selecting materials for the tubes that transport the degraded amine solution, Teflon stands out as the suitable choice due to its chemical resistance and inertness.

5.1.5 DC power supply

To select a suitable DC power supply, it is crucial to first determine the required ranges of current and voltage. Additionally, it should be considered whether it is more appropriate to control the current or the voltage. Drawing from the findings in research [21], the current should be controlled and the maximum current density for both CEM and AEM is 400 A/m², while for BPM, it stands at 800 A/m². Based on Table 5.1, the area for each membrane is 0.0055 m². Consequently, the maximum current density for the C-A stack configuration is calculated to be 2.2 A, and for the BP-A stack configuration, it is 4.4 A. the method of, further emphasizing the need for a power supply that can accommodate these specifications.

In the case of a stack comprised of uniform membranes, the voltage for each unit is 1V (in this research [20] it was set to 1.5 V). Conversely, for a stack of bipolar membranes, the optimal voltage per unit is 2 V. Given that each stack consists of 10 such units, in addition to 5 V from the electrodes, the total operating voltage for the uniform membrane and bipolar membrane stacks has been established at 15 V and 25 V, respectively. Hence, it becomes evident that an appropriate DC power supply should be capable of supporting currents ranging from 0 to 4.4 A, along with voltages spanning 0 to 25V for the specific design requirements.

5.2 Analytical methods

In discussing analytical methods, examples are drawn from other scientific papers that have undertaken similar work. It's worth noting that devices with specifications similar to those mentioned in the following subchapters may be suitable for conducting experimental analyses. Regarding sensor body materials, options include high-quality materials such as glass, polycarbonate, and polyoxometalates (POMs), along with other suitable materials designed to withstand the challenges posed by degraded amine solutions.

5.2.1 Ion Chromatography

Ion chromatography (IC) is employed for analyzing compounds resulting from amine degradation. Anion chromatography has proved effective in detecting HSS and making it the predominant method for assessing negatively charged compounds. On the other hand, cation chromatography was utilized to measure the depletion of amines in solution. [22].

The use of ion chromatograph devices for analyzing amine solvents is widespread across various research endeavors. For instance, in this research [23] outlines the chromatographic analysis of alkanolamines using a Thermo Scientific™ Dionex™ ICS-5000 system. Cations were separated using a Dionex IonPac™ CS19 analytical column and a guard column CG19. Samples were injected via a Dionex AS Autosampler and carried through with a dilute methanesulfonic acid (MSA) eluent. The CS19 column, renowned for its cation-exchange properties, facilitated the separation of cations and polar amines. The Dionex CSRS 300 2 mm suppressor reduced background conductivity, enhancing analyte response. Detection was performed using a Thermo Scientific conductivity detector, with Chromeleon® 7 software utilized for data analysis.

Similarly, in another experimental investigation [24], anion chromatography experiments were conducted using a Dionex DX-100 ion chromatograph with a conductivity detector. The analytical column employed was an IonPac CS14 (4 mm), accompanied by an IonPac AG14 (4 mm) guard column. The eluent consisted of 3.5 mM sodium carbonate/1 mM sodium bicarbonate, flowing at 1.2 mL/min. A self-regenerating ASRS−Ultra (4 mm) suppressor was utilized.

5.2.2 Raman spectroscopy

In the analytical sciences, Raman spectroscopy is one of in-situ monitoring tools and has been applied for the identification of unknown compounds. The energy difference between the incident and scattered photons provides a unique fingerprint for identifying different molecules [25]. Quantitative analyses in absorption spectroscopy are grounded in the Lambert-Beer law, expressed as equation (5.1):

$$\log \frac{I_0}{I} = \log \frac{1}{T} = A = abC \tag{5.1}$$

Here, I_0 represents incident light, I is light after it has passed through the sample, T is transmittance, A represents absorbance, a indicates absorption coefficient, b stands for sample thickness, and C denotes sample concentration. The absorption coefficient, specific to each material at a particular wavelength, remains invariant. In scenarios adhering to the Lambert-Beer law, absorbance demonstrates linearity concerning concentration (and path length), thus enabling both single component and multicomponent analyses [26]. The concentration of the absorbing species or the path length should remain within the linear range where the Lambert-Beer law holds true. Any deviation from this range could indicate potential issues, such as non-linear absorption behavior, scattering effects, or instrumental limitations.

Unfortunately, I could find a few articles that used Raman spectroscopy for analyzing amine degradation solutions online, however, in this experimental work [27], that aimed to create a fast and dependable analysis method for assessing the properties of gas loading and solvent strength in amine-based CO_2 capture processes, the Raman spectroscopy utilized was a RXN2 Kaiser 785 nm spectrometer Raman system. To minimize light sensitivity, samples were housed in black plastic holders and covered with aluminum foil to eliminate fluorescence interference. The immersion probe was cleaned with deionized water and acetone before each measurement to prevent contamination. The probe tip was inspected for air bubbles to ensure accurate readings.

M. Akram et al. [28] also used the same devise for exploring the effectiveness of Raman spectroscopy as a process analytical tool for real-time monitoring of CO_2 capture utilizing aqueous MEA solution.

5.2.3 Ultraviolet-Visible spectroscopy (UV-Vis)

Ultraviolet-Visible Spectroscopy (UV-Vis) is employed for analyzing degraded amine solvents by assessing their absorption of ultraviolet and visible light, revealing changes in electronic structure. By comparing the absorption spectrum of degraded amines to reference samples, shifts or new absorption bands indicating degradation products can be identified. UV-Vis spectroscopy enables both qualitative and quantitative analysis by detecting changes in absorption bands and correlating absorbance with concentration. The radiation in UV-Vis involves electronic transitions of bonding electrons, typically occurring within the wavelength range of 200 to 800 nm [29].

Most articles utilize laboratory UV-Vis spectroscopy for analytical work, and there is a lack of research employing in-situ UV-Vis sensors. For instance, in this paper [29], which focused on oxidative degradation of MEA, an HP 8452A Diode Array Spectrophotometer equipped with a deuterium lamp was used. Measurements were conducted with a 2 nm resolution, spanning from 200 nm to 800 nm, using 5 ml polycarbonate cuvettes with a path length of 1 cm. Each sample was analyzed within a time frame of 0.5 seconds. Samples registering an absorbance greater than 1.0 are deemed inappropriate for qualitative analysis, as deviations from the Lambert-Beer law (Equation 5.1) have been observed.

Alternatively, Reynolds et al. [30] conducted research into evaluating methods for monitoring MEA degradation, utilizing a Varian Cary 300 UV–vis spectrophotometer (Australia) to measure absorbance spectra ranging from 190 nm to 900 nm in a 1 cm quartz cell.

Drawing inspiration from these articles, one can select the appropriate UV-Vis spectrophotometer, considering budget constraints and enabling in-situ implications.

5.2.4 pH measurement

According to the information provided in this [31] reference, measuring pH involves various methods, including visual observation, photometric analysis, and potentiometric measurement using electrodes sensitive to pH changes. Potentiometric techniques offer superior sensitivity and adaptability compared to visual or photometric methods. These electrodes consist of several essential parts, including the electrode body, a pH-sensitive glass membrane, a reference electrode, a reference electrolyte, and a reference junction.

Glass electrodes, commonly utilized in pH measurement, feature a glass membrane housing a buffer solution with a known pH. This membrane generates an electric potential based on the concentration gradient of hydrogen ions between its interior and the sample solution. Various membrane shapes, such as spheres, cones, or flat tips, meet specific measurement requirements.

Reference systems are important in pH measurement, providing a stable electrical potential unaffected by sample composition or temperature. Different reference systems, such as silver/silver chloride, mercury chloride, or iodine/iodide, offer distinct properties and applications. The choice of reference electrolyte, typically potassium chloride (KCl), is crucial for maintaining electrical conductivity and stability within the electrode.

Electrolytes within electrodes can utilize gel, polymer, or liquid forms, each with unique benefits and drawbacks. Gel and polymer electrolytes prevent leakage and are easier to maintain but have a shorter lifespan compared to liquid electrolytes.

The role of the reference junction, also referred to as a diaphragm, is important in ensuring the accuracy of measurements by establishing electrical connectivity between the reference system and the solution. Its effectiveness relies on specific attributes, including a consistent outflow of reference electrolyte and controlled permeability to prevent rapid electrolyte escape. Junction types vary in their electrolyte outflow rates and electrical resistance, with the ideal characteristics being low resistance and chemical inertness. Various junction types, including ceramic, platinum, ground-joint, and plastic, offer differing outflow rates and are selected based on specific measurement conditions. Ceramic junctions make use of the porosity of unglazed ceramic, characterized by high electrical resistance and potential limitations in solutions with lower ionic strength. Platinum junctions ensure a steady electrolyte flow, though they may have limited applicability in highly reactive solutions. Ground-joint junctions utilize the narrow gaps in unlubricated ground glass for electrolyte outflow, offering low electrical resistance and suitability for measuring contaminated solutions. Plastic junctions fulfill specialized roles, such as integration into combination electrodes with plastic shafts or for measurements in fluoride-containing solutions.

When employing ED as a method for reclaiming amines, pH analysis plays an important role in various scientific applications, for instance, in the paper cited [21], which discusses the application of a novel two-stage electrodialysis process for removing HSS from degraded amine solvents in CO_2 chemical adsorption, a PHS-3C digital pH meter from Leici company was employed. This device accurately measures pH within the range of 0.00 to 14.00 (-1999 to 1999 mV) with an accuracy of ±0.01 pH.

In addition, the study referenced in article [9], which employed a two-step electrodialysis for the treatment of alkanolamine absorbent to eliminate heat stable salts in the presence of CO_2, utilized a Hanna HI 991003N small-size pH meter to determine pH values.

5.2.5 Conductivity measurement

The differences in concentration of HSSs in the dilute compartments can be determined through conductivity measurements. As discussed earlier, the operation time is influenced by the conductivity of dilute compartments. As expressed in [32], conductivity measurements are typically expressed in milliSiemens (mS) or microSiemens (μS), with the cell's geometry affecting readings. To standardize, units like mS/cm or μS/cm are used. The conductivity cell constant compensates for geometry differences, calculated by dividing the distance between plates (L) by their surface area (a = A × B) (shown in equation (5.2). Solution conductivity is found by multiplying measured conductivity (G) by the cell constant (k) (shown in equation (5.3)).

$$K = \frac{L}{a} \tag{5.2}$$

$$\text{Specific conductivity} = \text{measured conductivity (G) * cell constant (K)} \tag{5.3}$$

Ideally, a cell constant of 1.0 would match the solution's specific conductivity. However, adjustments are often needed. Low-conductivity solutions require closer plate placement

(reduced cell constant like 0.1 or 0.01), while high-conductivity ones need longer paths (higher constants like 10 or 100) for accurate readings.

In this [21] article, a DDS-11A conductivity meter from Leici company was used as an example, which can measure conductivity in the range of 0.00μS/cm to 199.9mS/cm.

It is also possible to use a multi-parameter tool for analyzing the conductivity and pH of the solution. For instance, in these experiments [35], which focused on the use of Electrodialysis for the Reclamation of MEA, a TPS WP-81 device from Thermo Fisher Scientific was utilized. This multi-parameter tool is capable of measuring pH, temperature, and conductivity.

5.2.6 Calculation of HSS removal efficiency

the efficiency of HSS removal efficiency (η, %) can be calculated by using the Equation (5.4):

$$\eta = \frac{\Delta C}{C_0} \times 100\% = \frac{C_{i,0} - C_{i,t}}{C_{i,0}} \times 100\%$$

(5.4)

Here, $C_{i,0}$ represents the initial concentration of anion i in the diluted amine solvent, while $C_{i,t}$ denotes its concentration at time t (measured in ppm (parts per million)) [21], [33].

In some research papers like [10] and [34], the HSS removal rate (r, g-eq/s) is calculated by the Equation (5.5):

$$r = \frac{E \times n \times I}{F}$$

(5.5)

where n is the number of membrane cell pairs, F is the Faraday constant (96480 Coulombs/g-eq), E is the efficiency factor, and I is the current (A).

5.2.7 Calculation of amine loss rate

Equation (5.6) [21] can be applied to calculate the rate of amine loss (λ, %):

$$\lambda = \frac{C_{d,0} - C_{d,t}}{C_{d,0}} \times 100\%$$

(5.6)

Where $C_{d,0}$ represents the initial concentration of amine in the diluted amine solvent, and $C_{d,t}$ denotes its concentration at time t (measured in wt.%).

5.2.8 Calculation of energy consumption:

The energy consumption (E, kW·h/g) of the ED process is determined using Equation (5.7) [21]:

5 Design a lab-scale apparatus for amine reclamation

$$E = \frac{\int_0^t UI\,dt}{C_0 V_0 - C_t V_t}$$

(5.7)

Where U represents the voltage drop across the ED stack (V), I denote the current (A), C_0 and C_t indicate the total HSS anion concentration of the dilute amine solvent (g/mL) at time t_0 and t respectively.

6 Discussion

In this chapter some important parameters that play important roles in electrodialysis process are discussed in the following subchapters.

6.1 HSS removal efficiency

During the electrodialysis process, HSS anions migrate through membranes into the concentrate solution. This study [33] developed dimensionless models for HSS removal efficiency via electrodialysis using RBF (radial basis function) and GP (genetic programming) intelligent approaches. Based on this research, various factors influence HSS removal efficiency. These factors include current intensity, time, HSS concentration, membrane type (homogeneous or heterogeneous ion exchange membrane), electrochemical membrane properties (ion exchange capacity (IEC), resistance, etc.), and the fraction of amines.

In part 4.1, the membrane's type and specifications were discussed. Regarding the effect of time, under identical conditions, there is an enhancement in ion removal efficiency with increasing time. This occurs because more anions transfer from the dilute to the concentrate compartment within the membranes as operation time extends. Similarly, as HSS concentration rises, there is a corresponding improvement in removal efficiency. This is because the potential difference acts as a driving force in the electrodialysis process. It is important to highlight that as HSS concentration rises, the applied current also increases due to the increasing limiting current density, consequently, a greater quantity of ions is transferred from the dilute solution to the concentrated solution. The amine fraction also plays a role in HSS removal efficiency, where an increase in the amine fraction leads to decreased efficiency. Anions' transport can be hindered by protonated amine presence, reducing the rate of HSS anion transport through the membrane.

Additionally, based on the Equation (5.5), the number of membrane cell pairs within the stack impacts HSS removal efficiency. A higher number of membrane cell pairs correspondingly enhances the efficiency of HSS removal at the same conditions.

When investigating the removal rate of each HSS ion, according to the findings of Y. Wang et al. [21], it was observed that the removal rates of HSS ions are influenced by factors such as their mobility, represented by the dissociation constant (pKa) of the corresponding acid. Additionally, initial concentration and molecular weight impact the ion migration process and final removal efficiency. For instance, in the C-A stack configuration presented in the designed ED unit, the concentration of formate anions, with a pKa of 3.75, decreased rapidly initially due to its lower molecular weight and higher initial concentration, while glycolate anions, despite a higher initial concentration, exhibited slower removal rates due to their larger molecular weight. Sulfate anions faced hindrance due to their large size and higher charge, resulting in limited removal efficiency. Chloride anions, with a low pKa value, exhibited low removal rates likely due to their low initial concentration. Table 6.1 displays the molecular weights and pKa values of mentioned HSS anions, while Figure 6.1 illustrates the corresponding removal rates.

Table 6.1: Molecular weight and pKa of the ions used in the ED process [21].

Anions	pKa	Molecular weight (g/mol)
Formate	3.75	46.03
Glycolate	3.83	76.05
sulfate (I , II)	I= -3, II=1.9	96.06
Chloride	-8	35.45
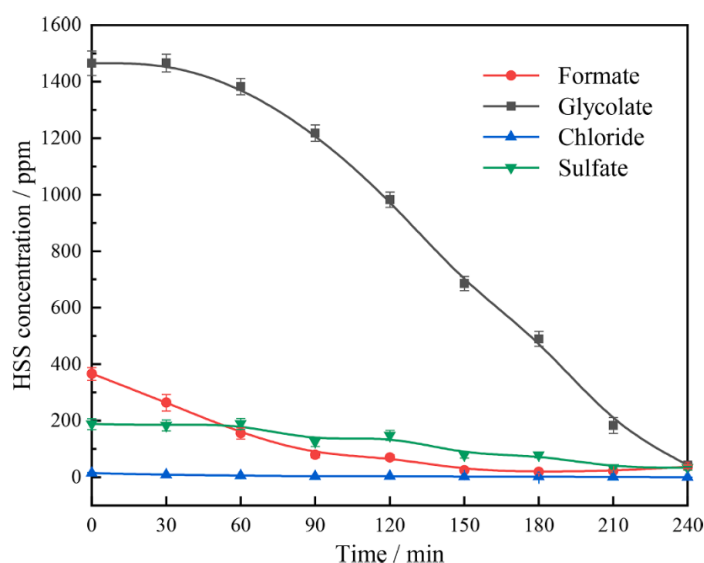
Figure 6.1:Illustrating the rate of migration for different HSS ions towards the concentrate compartments in C-A Stack Configuration presented in the designed ED unit [21].

The removal efficiency of HSS anions was investigated for the presented ED unit with C-A and BP-A stack configurations. Higher current density enhances the ion migration rate, making ion concentration in each compartment a critical factor limiting the efficiency of ED for HSS removal. In the C-A ED process, ion concentration remained low, resulting in a slow increase in removal efficiency. After 90 minutes, the removal efficiency is 30% and it reaches 94.35% after 240 minutes, when the dilution compartment's conductivity stabilizes. The BP-A ED process demonstrated the highest removal efficiency of 96.49% after 110 minutes due to its higher initial HSS concentration. However, this led to higher HSS concentrations in the recovered amine solution, notably with elevated levels of sulfate and formate anions. Both configurations yield reclaimed amine solutions with HSS concentrations below 500 ppm, meeting operational requirements.

Based on the provided data, the two-stage ED unit presented in Figure 5.1 needs 350 minutes (~ 6 h) to reach the efficiency of more than 96%.

6.2 Amine loss

Amine molecules have the capability to move through a membrane when there exists a concentration gradient of these amines between the dilute and concentrated compartments on both sides of the membrane. The specifications of the ion exchange membrane play an

important role in decreasing amine loss. Amine diffusion is particularly significant in membranes that are thin, typically around 50–100 μm and reinforced. For example, in homogeneous membranes with a reinforced structure and moderate thickness, the transfer of amines from the dilute solution to the concentrated effluent is greatly reduced, almost to zero [35].

Concerning the proposed ED design (Figure 5.1) and drawing from the insights of this research [21], Surprisingly, the amine concentration in BP-A's dilute compartment increases from 13.5wt.% to 16.9wt.% during secondary recovery, attribute to the selective passage of protonated amine cations and water electrolysis in the bipolar membrane. This process lead to decrease water content in the solution but maintains a relatively constant level of amine, resulting in increasing concentration. Despite a minor 3.44% decrease in total amine mass, amine loss is minimized. Conversely, in C-A, amine concentration in the dilute compartment decreases, with a 46.29% amine loss rate due to migration across the membrane.

In conclusion, to enhance amine recovery and since the current density is increased during the transfer of HSS components in the BP-A membrane stack compared to classical electrodialysis, it is advisable to incorporate the BP-A membrane stack in the second stage of the ED unit.

6.3 Effects of CO_2-loading

Based on the article [36], the concentration of CO_2 in the degraded amine solution affects the HSS removal rate and amine loss. By increasing the CO_2 concentration in the solution, the number of charged components increases, leading to increased competition between ions migrating through the membranes. As a result, the HSS removal rate decreases. The increase in CO_2 loading can also raise the amine loss rate. For example, as shown in Figure 6.2, increasing the CO_2 in the solution from zero loading to 0.2 mol (CO_2)/mole (amine) increases the amine loss from 4.4% to 29%, respectively.

In the suggested apparatus in the Figure 5.1, although adding the step for releasing CO_2-loading may seem to increase the operation time, it enhances the HSS removal rate in the second stage. Reclaiming amine solvent directly with a specific CO_2-loading through ED with a bipolar membrane can lead to CO_2 buildup within the membrane stack, causing blockages and operational issues. This occurs because the ongoing generation of hydrogen ions in the acid chamber triggers reactions with migrating anions, such as carbonate and bicarbonate, resulting in a continuous decrease in pH and the production of CO_2 gas [21].
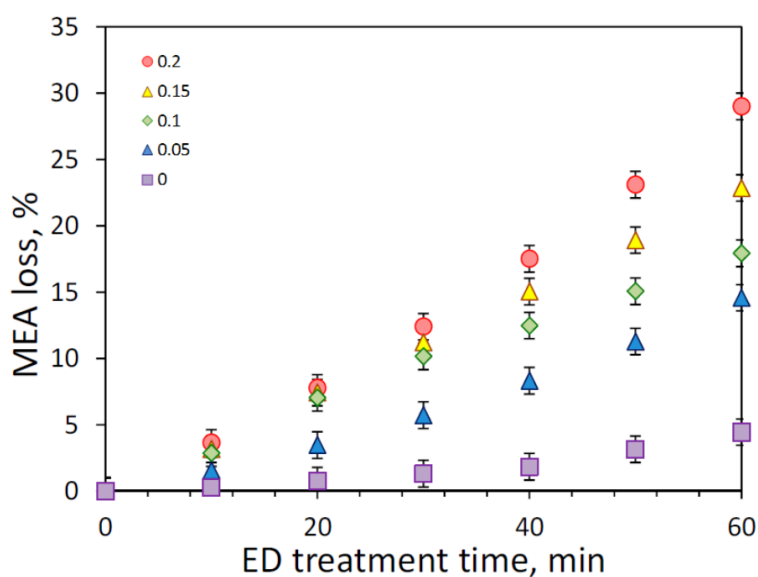
Figure 6.2: Effects of CO_2-loading on amine loss in ED process [36].

6.4 Effect of current density

The current density applied is important in determining the effectiveness of the ED process. Higher current densities not only reduce experimental duration, aiding in reaching desirable HSS removal efficiency faster and minimizing amine loss, but also necessitate less membrane area, thus reducing capital costs. However, this benefit comes with drawbacks; high current density can lead to membrane degradation, shortening their lifespan and compromising membrane ion selectivity. On the other hand, lower current densities provide benefits in terms of both current utilization and energy consumption. This is because reducing current density leads to decreased energy consumption. Practical applications of the ED process often involve a trade-off between high and low current densities. Higher current densities, while offering potential advantages, become less attractive due to their associated high energy costs. Conversely, lower current densities, while appealing in terms of energy efficiency, may not be recommended due to the greater membrane area required for a given plant [37].

Based on the [21] and for the suggested ED design presented in Figure 5.1, the current density of both membrane stack configurations initially increased and then decreased during the removal process, indicating ion flux changes through the ion-exchange membrane. Initially, high overall resistance due to deionized water in the concentrate compartment resulted in a low overall current. As the experiment progressed, ions migrated from the dilute to the concentrate chamber, increasing conductivity in the concentrate solution while decreasing it in the dilute solution. Consequently, current density peaked and then declined. The BP-A configuration showed significantly higher current density, reaching a limiting current density of 800 A/m2. This was attributed to its higher initial ion concentration and efficient ion transport across the membrane, resulting in increased energy consumption.

6.5 Effect of pH

Raising the pH level proves advantageous for amine recovery as shown in part 4.2.2, however the removal of HSS from the degraded amine solution is not heavily rely on the initial pH value. This is because HSS exist in an ionic state within aqueous solutions. The introduction of a base, increases the solution's ionic strength, thereby increasing experimental duration. Therefore, the final concentration of HSS is more closely tied to the solution's ionic strength rather than the initial pH of the amine solution. Moreover, energy consumption is not notably affected by the initial pH of the degraded amine solution. Despite the benefits of pH elevation for amine recovery, it entails the consumption of external chemicals and necessitates a larger membrane area in a given plant. Additionally, commercially available membranes typically advise operation below a pH of 12 to maintain membrane longevity, as higher pH levels can be detrimental [37].

6.6 Cost estimation

Assessing the costs of the presented electrodialysis unit (Figure 5.1) is important for evaluating its economic viability. In Table 6.2, estimated costs for each piece of equipment, and in Table 6.3, the chemicals used in the suggested apparatus are provided. These estimates serve as initial guidelines for understanding the investment required to set up and maintain the electrodialysis system. However, it is important to note that actual costs may vary depending on factors such as market fluctuations, technological advancements, and specific project requirements. Additionally, all technical specifications and other useful information for the equipment and chemicals in the following tables are indicated in Appendix B. As shown in this appendix, the size of the equipment can be selected from among other suitable equipment on the market, allowing the ED unit to be set up under the hood easily. I recommend using a stand with shelves suitable for use under the hood in terms of size when using two units simultaneously

The membrane price is calculated based on the area used in the membrane stack. For ED with a C-A stack configuration, there are 10 CEMs and 11 AEMs, each with an area of 0.0055 m². In ED with a BP-A stack configuration, there are 10 AEMs and 11 BPMs with the same effective area. Stack costs encompass various components essential for the functioning of electrodialysis stacks, including membranes, spacers, and frames, as its cost is vary, it is considered 1.5 times membrane cost. For peristaltic pumps, their costs can be calculated for 6 single-channel peristaltic pumps as shown in Table 6.2, or due to space limitations, it can be calculated for 1 six-channel peristaltic pump or 2 three-channel peristaltic pumps. However, using multi-channel pumps might be more expensive than using 6 single-channel pumps. For C-A ED and BP-A ED, the cost of a single-channel DC power supply is calculated, and for the suggested design, the cost of a dual-channel DC power supply is considered. 3 meters Teflon tubing is considered for each ED unit. Most of the cost of this ED design involves laboratory beakers because, in this work, an attempt is made to make the system a closed system, so all beakers should be equipped with a sealed lid that has space for all sensors and stirrers. 3 beakers are needed for each ED unit, and five beakers are needed for a two-stage ED unit. Each beaker has a 1 L capacity and is equipped with a stirrer. If using a closed system is not necessary, the total investment cost will decrease by about 50%.

Table 6.2: Shows the average cost of the equipment used in the designed apparatus.

Parameters	Average cost ($)	Cost for C-A ED unit ($)	Cost for BP-A ED unit ($)	Cost for the C-A+BP-A ED unit ($)
CEM	1816 ($ / m^2)	99.88 (10 Pcs)	0	99.88 (10 Pcs)
AEM	1816 ($ / m^2)	109.88 (11 Pcs)	109.88 (11 Pcs)	219.76 (22 Pcs)
BPM	5217 ($ / m^2)	0	286.93 (10 Pcs)	286.93 (10 Pcs)
Stack cost:	----	314.64	595.21	909.85
Peristaltic pump	787	2361 (3 Pcs)	2361 (3 Pcs)	4722 (6 Pcs)
DC power supply	2157	1947 (single-channel)	1947 (single-channel)	2157 (dual-channel)
Teflon tube	45$/m	135 (3 m)	135 (3 m)	270 (6 m)
Laboratory beaker	3098	9294 (3 Pcs)	9294 (3 Pcs)	15490 (5 Pcs)
Laboratory stirrer	42	126 (3 Pcs)	84 (2 Pcs)	210 (5 Pcs)

As indicated in Table 6.3, the suggested ED design requires minimal chemicals. Both ED units use Na_2SO_4 solution as an electrolyte, with a total volume of 1 L. According to this article [21], it needs a concentration of 0.5 mol/L Na_2SO_4. Considering the molecular weight of Na_2SO_4 (142.04 g/mol), the total amount required and its price can be calculated. Regarding deionized water, in C-A ED, the cost is accounted for 500 mL of deionized water for the electrode solution and another 500 mL for the concentrate solution. Similarly, in BP-A, the cost of deionized water can be calculated for 500 mL used in the electrode solution.

Table 6.3: Shows the average cost of the chemicals used in the designed apparatus.

Parameters	Average cost ($)	Cost for C-A ED unit ($)	Cost for BP-A ED unit ($)	Cost for the C-A+BP-A ED unit ($)
Sodium sulfate	162$/kg	5.75	5.75	11.50
Deionized water	17.5$/L	17.5	8.75	26.25

The cost estimation process involved a review of previous literature [21], [38], [39], which provided valuable insights into the financial aspects of electrodialysis processes. To calculate the total investment cost, it is necessary to consider both stack costs and equipment costs (exclude chemicals), as outlined in Table 6.4. Equipment costs encompass expenses associated with auxiliary equipment such as pumps, DC power supply and other required laboratory equipment that mentioned in Table 6.2.

Table 6.4: Estimation of investment cost of designed apparatus.

Parameters	Cost for C-A ED unit ($)	Cost for BP-A ED unit ($)	Cost for the C-A+BP-A ED unit ($)
Membrane cost	209.76	396.81	606.57
Stack cost	314.64	595.22	909.86
Equipment costs	13863	13821	22849
Total investment	**14387.40**	**14813.03**	**24365.43**

To calculate the total yearly fixed cost, one must consider various factors, including the assumed lifespan of membranes (typically set at 3 years), amortization schedules, interest rates in the operating region, and maintenance costs. The maintenance costs, amortization, and interest rates are assumed to be 10% of total investment costs, 3 years, and 4.5% (in Norway), respectively, as detailed in Table 6.5.

Table 6.5: Estimation of yearly fixed cost of designed apparatus.

Parameters	Cost for C-A ED unit ($)	Cost for BP-A ED unit ($)	Cost for the C-A+BP-A ED unit ($)
Amortization $/year	1797.80	2439.68	3041.81
Interest, $/year	242.70	329.36	410.64
Maintenance, $/year	539.34	731.90	912.54
Total fixed cost, $/year	**2579.84**	**3500.93**	**4364.99**

Consequently, the proposed lab-scale apparatus requires an investment of approximately $24,365.43 and a fixed yearly cost amounting to $4,364.99. It should be noted that the cost of energy is not factored into the estimation.

7 Conclusion

In conclusion, among thermal reclamation, ion exchange, and electrodialysis, electrodialysis emerged as an appropriate option due to its lower chemical and maintenance requirements. By focusing on heat stable salt removal, electrodialysis proved capable of effectively removing heat stable salts and achieving high amine recovery. The scalability of electrodialysis technology makes it suitable for laboratory use and adaptable for operation under laboratory hoods. Investigation into electrodialysis configurations led to the suggestion of a two-stage ED unit comprising C-A and BP-A stack configurations as the lab-scale apparatus for amine reclamation, achieving a HSS removal efficiency of over 96% with minimal amine loss of less than 4%. In this setup the lids of dilute beakers in both stages were considered to have places for inserting analytical sensors like pH, conductivity, Raman and UV-Vis to facilitate the monitoring process. A rough cost estimation conducted for the suggested apparatus showed that it required an investment of about $24,365.43 and yearly fixed costs of $4,364.99.

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Appendices

Appendix A: Project topic description

In this appendix, the signed final project topic description with no adjustments is attached.

University of South-Eastern Norway
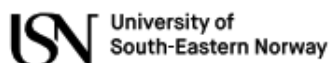
Faculty of Technology, Natural Sciences and Maritime Sciences, Campus Porsgrunn

FMH606 Master's Thesis

Title:
Designing a lab-scale apparatus for reclaiming amines

USN supervisor:
Kjell-Arne Solli

External partner:
In co-operation with USN research group 'Energy and CO2 Capture (ECC)', part of URGENT

Task background:
The most mature technology for CO2 capture is reactive absorption in amine. Effective and economically feasible plants are in operation, but over time degradation of the amine solvent is inevitable. Reclaiming is the process for separation of degradation products from the solvent. Lab-scale apparatus will enable research to fill the knowledge gap for a cost-effective operation, minimizing environmental impact.

Task description:
Known technologies for reclaiming includes (reduced pressure) distillation, membrane separation, ion exchange columns, and electrodialysis. A lab-scale apparatus must adhere to HMS requirements and the size limitations for placement in hood – and still enable withdrawal of samples for analysis as well as spectroscopic monitoring.

- Choose a preferred technology
- Design the apparatus, equipment drawings
- Estimate efficiency of separation
- Cost estimation of the equipment
- If possible within time limits, construction of the equipment and testing the performance using samples of degraded amine

Color change of amine solvent during CO2 capture and reclaiming. From Flø *et al.* (2017), "Results from MEA Degradation and Reclaiming Processes at the CO₂ Technology Centre Mongstad".
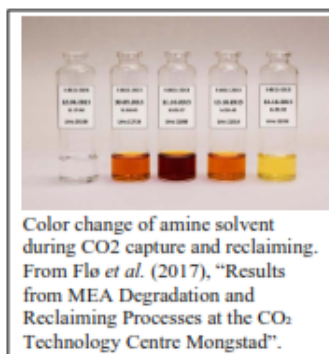
Student category:
EET or PT students

Is the task suitable for online students (not present at the campus)? No

Practical arrangements:
Work 'progress and guidance' meetings in co-operation with the research group. Finance of equipment and chemicals required (within limits).

Supervision:
As a general rule, the student is entitled to 15-20 hours of supervision. This includes necessary time for the supervisor to prepare for supervision meetings (reading material to be discussed, etc).

Signatures:

Supervisor (date and signature):   01.02.2024 *Kjell-Arne Solli*

Student (write clearly in all capitalized letters): NASRIN HOSSEINI

Student (date and signature):   31.01.2024 *Nasrin Hosseini*

Appendix B: Equipment and chemicals specifications

In this appendix, a list of the companies that supply the equipment and chemicals referenced in the cost estimation section will be found. Each entry includes the name of the supplier and corresponding prices, offering readers a detailed overview of the expenses associated with the procurement of necessary resources for the project.

Table A.1: Cation exchange membrane (CEM)

Supplier company: Membranes International Inc.	
Price ($/m^2):	194.45 (available online)
Type:	CXM-200S (CMI-7000S)
Functionality:	Strong Acid Cation Exchange Membrane
Polymer Structure:	Gel polystyrene cross linked with divinylbenzene
Functional Group:	Sulphonic Acid
Standard Size (m):	1.22 * 3.00
Electrical Resistance (Ohm.cm^2):	<30
Maximum Current Density (A /m^2):	<500
Permselectivity (%):	94
Standard Thickness (mm):	0.45±0.025
Total Exchange Capacity (meq/g):	1.6 ± 0.1
Water Permeability (ml/hr/ft^2) at 5psi:	<3
Mullen Burst Test strength (psi):	>80
Thermal Stability (ºC):	90
Chemical Stability Range (pH):	1-10
Source:	Membranes International Inc
Link:	https://ionexchangemembranes.com/cmi-7000-cation-exchange-membranes-technical-specifications/
Supplier company: Fumasep	
Price ($/m^2):	1816 (available online)
Type:	Fumasep FKB-PK-130
Standard Size (cm):	20*30
Thickness (μm):	110 - 140

Reinforcement:	PK
Electrical Resistance (Ohm.cm^2):	2.5-5.0
Specific Conductivity (mS/cm):	2-8
Selectivity (%):	96-99
Hydroxyl transfer rate (µmol/min cm^2):	5-100
Ion Exchange Capacity (meq/g)	0.8-1.0
Uptake in H$_2$O at 25°C (wt %)	10-30
Tensile Strength - max. (MPa)	50 - 80
Chemical Stability Range (pH):	Stable under acidic and caustic conditions.
Source:	Fuel cell store
Link:	https://www.fuelcellstore.com/fumasep-fkb?search=cation%20Exchange%20Membrane

Table A.2: Anion exchange membrane (AEM)

Supplier company: Membranes International Inc.	
Price ($/m^2):	194.45 (available online)
Type:	AXM-100S (AMI-7001S)
Functionality:	Strong base anion exchange membrane
Polymer Structure:	Gel polystyrene cross linked with divinylbenzene
Functional Group:	Quaternary ammonium
Standard Size (m):	1.22 * 3.00
Electrical Resistance (Ohm.cm^2):	<40
Maximum Current Density (A /m^2):	<500
Permselectivity (%):	90
Standard Thickness (mm):	0.45±0.025
Total Exchange Capacity (meq/g):	1.3 ± 0.1
Water Permeability (ml/hr/ft^2) at 5psi:	<3
Mullen Burst Test strength (psi):	>80
Thermal Stability (^{o}C):	90
Chemical Stability Range (pH):	1-10
Source:	Membranes International Inc
Link:	https://ionexchangemembranes.com/ami-7001-anion-exchange-membranes-technical-specifications/
Supplier company: Fumasep	
Price ($/m^2):	1816 (available online)
Type:	Fumasep FAB-PK-130
Standard Size (cm):	20*30
Thickness:	110 - 130 μm (microns)
Reinforcement	PET
Electrical Resistance (Ohm.cm^2):	10 - 20
Specific Conductivity (mS/cm):	10 - 20
Selectivity (%):	93 - 98

Proton Transfer Rate (µmol/min cm^2):	60-400
Ion Exchange Capacity (meq/g)	0.7-1.0
Uptake in H2O at 25°C (wt %)	5-15
Tensile Strength - max. (MPa)	40 - 80
Chemical Stability Range (pH):	0 - 14
Source:	Fuel cell store
Link:	https://www.fuelcellstore.com/fumasep-fab?search=Anion%20Exchange%20Membrane

Table A.3: Bipolar membrane (BPM)

Supplier company: Fumasep	
Price ($/m^2):	5216.6 (available online)
Type:	Fumasep FBM
Standard Size (cm):	20 * 30
Standard Thickness (µm):	130 - 160
Thermal Stability (ºC):	40 °C
Water Splitting Efficiency at 100 mA/cm^2	> 98 %
Water Splitting Voltage at 100 mA/cm^2	< 1.2 V
Chemical Stability Range (pH):	1 - 14
Source:	Fuel cell store
Link:	https://www.fuelcellstore.com/fuel-cell-components/membranes/membrane-type/bipolar-membranes/fumasep-fbm?sort=pd.name&order=DESC&limit=100

Table A.4: Peristaltic pump

Supplier company: Longer	

Price ($):	2374 (available online)
Model:	LP-L100-1F/DG-6
Number of channels:	6
Speed (rpm):	0.1-100
Flow rate:	0.16 μL/min – 500 mL/min
Dispensing volume:	100 μL to 9999 L (Accuracy of ±2% with calibration)
Calibration function:	Enter the measured pumping volume and automatically calibrate the flow rate and dispensing volume
Display:	7" HD color LCD with resolution of 1024×600
Work mode:	Programming
Communication function:	Modbus protocol, multiple baud rates (1200/2400/4800/9600/19200/38400 bps),USB and RS485 (RJ11) interface for connection
Control mode:	Touch screen control, foot switch control, external signal control, communication control
Parameter Memory:	Memorize the working parameters set before power off.
Power supply:	AC220V +20%, 50Hz/60Hz (or optional AC110V +20%, 50Hz/60Hz)
Operating temperature:	0-40°C
Dimensions (mm) ((Length × width × height):	180× 291 × 236
Source:	Drifton
Link:	https://www.drifton.eu/shop/10-dispensing-peristaltic-pumps/2458-lp-l100-1f-programmable-peristaltic-pump/

Price ($):	3240 (received the price by email)
Model:	BT100-1L/4*YZ1515X
Number of channels:	4
Speed (rpm):	0.1-100
Speed resolution (rpm):	0.1
Max flow rate (mL/min):	267
Calibration function:	Improve flow rate accuracy
Communication function:	RS485
Control mode:	Control panel, external signal control and communication control are available
Parameter Memory:	Return to previous status when power on
Power supply:	AC 220V +/- 10% / 50W, AC 110V +/- 10% / 50W (optional)
Operating temperature:	0-40°C
Source:	Drifton
Link:	https://www.drifton.eu/shop/10-dispensing-peristaltic-pumps/2458-lp-l100-1f-programmable-peristaltic-pump/

Price ($):	787 (received the price by email)
Model:	BT300-2J/YZ1515X
Number of channels:	1
Speed (rpm):	1-300
Max flow rate:	1500 mL/min
Display:	3-digit LED displays current speed (rpm)
Communication function:	RS485
Control mode:	Control panel, external signal control and communication control are available.
Parameter Memory:	Stores the running parameters automatically
Power supply:	AC 90V-260V/48W
Operating temperature:	0-40°C
Source:	Drifton
Link:	https://www.drifton.eu/shop/8-basic-peristaltic-pumps/329-lp-bt300-2j-peristaltic-pump/

Table A.5: Peristaltic pump tubing

Supplier company: Longer	
Price ($/m):	45 (received the price by email)
Model:	Peristaltic Pump Tubing Norprene Chemical
Specifications:	- Long flex life in peristaltic pumps
	- Temperature range of -75° F to 165° F
	- Superior chemical resistance
	- Plasticizer-free bore
	- Meets FDA criteria for food contact
	- Resists absorption adsorption of aqueous fluids
	- Virtually unaffected by chemical sanitizers and cleaners
	- Complies with FDA 21 CFR, 177.1520 criteria and is applicable for food contact applications
	- Unaffected by most commercial sanitizers and cleaners
	- Can be autoclaved for up to five cycle times without affecting its overall service life
Source:	Drifton
Link:	https://www.longerpump.com/index.php/PumpTubing/show/40.html

Table A.6: DC power supply

Supplier company: IKM Instrutek AS

Price ($/m):	2157 (received the price by email)
Model:	PSW-720L44 (Dual-Channel Switching DC Power Supply)
Voltage (V):	80
Current (A):	13.5
Power (W):	360
Constant voltage mode:	Line regulation:43 mV
	Load regulation: 45 mv
	Ripple and noise: 60 mV
	Temperature coefficient: 100 ppm/($^\circ$ C) of rated output voltage, after 30 minute warm-up.
Constant current mode:	Line regulation:18.5 mA
	Load regulation: 18.5 mA
	Ripple and noise: 27 mA
	Temperature coefficient: 200 ppm/($^\circ$ C) of rated output current, after 30 minute warm-up.
Voltage accuracy (mV):	0.1%+20
Current accuracy (mA)	0.1%+20
Interface:	USB/LAN/GPIB-USB (Option)
Operating temperature ($^\circ$ C):	0-50
Dimensions (mm)(W*H*D)	142 * 124 * 350
Source:	https://www.ikmwebshop.no/dc-stromforsyninger?parent=10359

Price ($/m):	1947 (received the price by email)
Model:	PSW80-40.5 (single cahnnel)
Voltage (V):	0-80
Current (A):	0-40.5
Power (W):	1080
Regulation (CV, load) (mV):	45
Regulation (CV, line) (mV):	43
Regulation (CC) (load) (mA):	45.5
Regulation (CC) (line) (mA)::	45.5
Measuring accuracy of voltage (mV):	0.1% +10
Measuring accuracy of current (mA):	0.1% +40
Operation temperature (° C):	0~50
Interface:	USB/LAN/GPIB-USB(Option)/RS232-USB(Option)
Power source:	85VAC~265VAC, 47~63Hz, single phase
Dimensions (mm)(W*H*D):	214 * 124* 350
Source:	IKM Instrutek AS
Link:	https://www.ikmwebshop.no/dc-stromforsyninger?parent=10359

Table A.7: Laboratory stirrer

Supplier company: JUSTOP	
Price ($):	42 (available online)
Model	JJ-1 Accurate electric stirrer
Max speed (rpm)	3000
Material	Stainless Steel
Voltage:	AC220V 50Hz
Timer (min)	0-120
Operation mode:	Continuous
Size (cm):	60
Link:	https://www.alibaba.com/product-detail/Laboratory-Stainless-Steel-High-Speed-Mixer_1600948305647.html

Table A.8: Laboratory beaker

Supplier company: IKA-Werke GmbH & Co. KG

Glass vessel:

Price ($)	1112 (received the price by email)
Model:	HA.gv.sw.1 glass vessel, single wall
Volume (L):	1.0
Type:	Single wall
Multi use:	Yes
Autoclavable:	Yes
Inner diameter (mm):	110
Ratio = height / inner diameter :	1.73
Useable volume (min):.	0.3 l
Useable volume (max.):	1.0 l
Material in contact with medium:	Borosilicate glass 3.3 DIN
ISO:	3585
Height (mm):	195
Source:	IKA
Link:	https://www.ika.com/en/Products-LabEq/Bioreactors-pg233/HAgvsw1-Glass-vessel,-single-wall-20106410/Technical-Data-cptd.html

Lid :	
Price ($)	1032 (received the price by email)
Model:	HA.lc.1 Reactor lid, complete (for 1L glass vessel)
Port sizes are available:	2 * M10, 3 * Pg13.5, 5 * 11 mm, M16, M27 (4in1 port).
Reference::	https://www.ika.com/en/Products-LabEq/Bioreactors-pg233/HAlc1-Reactor-lid,-complete-20106426/
Vessel holder	
Price ($)	954 (received the price by email)
Model:	HA.vh.1 Vessel holder (for 1 l glass vessels)
Source:	IKA
Link:	https://www.ika.com/en/Products-LabEq/Bioreactors-pg233/HAvh1-Vessel-holder-20106404/
Total price ($):	3098

Table A.9: Sodium sulfate

Supplier company: SIGMA	
Price ($/kg):	162 (available online)
Product name:	Sodium sulfate BioUltra, anhydrous, ≥99.0% (T)
CAS number:	7757-82-6

Molecular weight (g/mol):	142.04
Appearance (Form):	Powder or crystals
Impurities:	Insoluble matter, passes filter test
	≤0.0005% total nitrogen (N)
Source:	SIGMA
Link:	https://www.sigmaaldrich.com/NO/en/product/sigma/71959

Table A.10: Deionized water

Supplier company: Alphacool	
Price ($/L):	17.5 (available online)
pH value:	6.5-8.5
el. Conductivity (µs/cm):	Less than 0.1
Total dry residue (mg/L):	Less than 10
Chloride (mg/L):	Less than 0.01
Nitrate (mg/L):	Less than 0.002
Nitrate-Nitrogen (mg/L):	Less than 0.25
Sulfate (mg/L):	Less than 0.001
Magnesium (mg/L):	Less than 0.02
Sodium (mg/L):	Less than 0.05
Nitrite (mg/L):	Less than 0.002
Phosphate (mg/L):	Less than 0.002
SiO2 (mg/L):	Less than 0.01
Tin (mg/L):	Less than 0.01
Total hardness (mmol/L)	Less than 0,002
Source:	Aquatuning
Link:	https://www.aquatuning.com/en/watercooling/cooling-liquids/ready-mix/waz-alphacool-es-deionized-water-ultrapure-water-1000ml