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Investigating the potential of producing eco-friendly liquid biofertilizer from domestic wastewater

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INVESTIGATING THE POTENTIAL OF PRODUCING ECO-FRIENDLY LIQUID BIOFERTILIZER FROM DOMESTIC WASTEWATER

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A Dissertation Submitted in Partial Fulfillment of the Requirements for the Degree of Master's in Hydrology and Water Resources Engineering of the Nelson Mandela African Institution of Science and Technology

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ABSTRACT

Water bodies experience environmental challenges such as eutrophication due to poor management of domestic wastewater in developing countries. Meanwhile, the explosion in population of the world has resulted in a 1.8 percent annual increase in demand for fertilizers that contain important nutrients. Although domestic wastewater contains nutrients (nitrogen, phosphorus, and potassium) that can be used in agriculture, its recovery is still a challenge. Some of the potential methods, such as the use of struvite precipitate in recovering nutrients from wastewater, are not only costly but also introduce a second pollutant into the ecosystem. The ion exchange method can recover phosphorus from wastewater; however, its effectiveness is limited by the presence of competing anions, such as sulfates. Freeze concentration method is one of the potential techniques for recovering nutrients from wastewater. However, its optimal condition such as temperature and time in recovering nutrients from domestic wastewater is not well known by researchers. In this study, method of freeze concentration was studied to establish its optimal condition in recovering nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in anaerobic digester. Frozen liquid and unfrozen concentrated liquid are produced. The effect of freezing temperature from -10°C to -80°C, freezing time from 1 hour to 8 hours and energy consumption on the nutrient recovery were investigated. Freezing temperature of -20°C, cooling time of 7 hours and energy consumption of 0.197 kWh/L resulted in the highest nitrate-nitrogen and phosphate nutrient recovery value of 1.114 and 4.667 respectively at the inlet of anaerobic digester 1, 1.325 and 4.975 respectively at the outlet of anaerobic digester 1, 1.099 and 4.859 respectively at the inlet of anaerobic digester 2, 1.132 and 4.755 respectively at the outlet of anaerobic digester 2 and for gravel filter at the outlet the values where 1.111 and 4.861 respectively. This study shows that, when the freeze concentration method is used with the right temperature, time, and energy, a significant amount of nutrients may be recovered from domestic wastewater that can be used as biofertilizer.

DECLARATION

I, Elly Chimoto Muga, do hereby declare to the Senate of the Nelson Mandela African Institution of Science and Technology that this dissertation is my original work and that it has neither been submitted nor being concurrently submitted for degree award in any other institution.

Elly Chimoto Muga

Name of candidate   **Signature**   **Date**

The above declaration is confirmed by

Prof. Hans C. Komakech

Name of supervisor 1   **Signature**   **Date**

Prof. Yusufu A. C. Jande

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CERTIFICATION

The undersigned certify that they have read and found the dissertation acceptable by the Nelson Mandela African Institution of Science and Technology.

Prof. Hans C. Komakech

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DEDICATION

This work is a tribute to my devoted father, Engineer Zacharia Chimoto Muga, for his unwavering commitment to and sacrifices made on my behalf during my whole educational career, from elementary school through my master's degree.

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LIST OF ABBREVIATIONS AND SYMBOLS

°C	Degrees of Centigrade
KW-hr	Kilowatt hour
NM-AIST	Nelson Mandela African Institution of Science and Technology
MSc	Masters of Science
HESLB	Higher Education Students Loans Board
NH4MgPO46HH20	Magnesium ammonium phosphate
WWTP	Waste water treatment plant
P	Phosphorus
NH_4^+	Ammonium
DAP	Diammonium phosphate
UN	United Nations
SP1	Sample point one
SP2	Sample point two
SP3	Sample point three
SP4	Sample point four
SP5	Sample point five

CHAPTER ONE

INTRODUCTION

1.1.   Background of the Problem

Pollution of water resources with excessive nutrient loads, most commonly phosphate and nitrogen, is a major environmental problem that disproportionately affect developing countries. The problem is exacerbated by population growth, which increases improper disposal of nutrients correspondingly, usually through wastewater discharges into various aquatic environments. Nutrients promote growth and reproduction of aquatic plants, which eventually becomes a serious problem because water becomes oxygen-depleted (Mavhungu *et al*., 2021).

Lack of dissolved oxygen in an aquatic ecosystem create bad environmental condition to aquatic life (Peng *et al.*, 2018). Furthermore, organic compounds affects the ecosystem's aesthetic value (Masindi *et al*., 2016).

Meanwhile, the explosion in population of the world has resulted in a 1.8% annual increase in demand for fertilizers which contain important nutrients (Yan *et al.*, 2018). According to studies, domestic wastewater contain nutrients that can be used in agriculture (Abdel-Shafy & Mansour, 2020; Bonvin *et al*., 2015). Nitrogen, phosphorus, and potassium are all important nutrients that can be recovered, concentrated, or eliminated from wastewater to create fertilizer for agricultural development. It has been reported that nitrogen and phosphorus can be recovered from both man-made and real wastewater throughout a wide range of nitrogen and phosphorous concentrations, including very low concentrations (Mehta *et al*., 2015). Williams *et al*. (2015) proved that nitrogen and phosphorus nutrients can be recovered from synthetic wastewater for fertilizer formation.

Researchers are now investigating or assessing various ways for recovering nutrients from wastewater for re-use as agricultural chemical fertilizer or biological fertilizer as a solution to the problems of improper nutrient-rich wastewater discharge, low fertilizer access in developing nations, as well as the impact of rising fertilizer costs on agricultural productivity (Wang *et al.*, 2019; Saliu *et al.*, 2020; van der Grift *et al.*, 2016). However, many of the previous researchers' methods are inaccessible and introduce a second pollutant into the ecosystem. Once they have been utilized, you will need to spend money on removing the

second pollutant (Siciliano *et al.,* 2020; Lai *et al.*, 2016; Khan *et al.*, 2019). Alternative methods such as struvite precipitation, ion exchange, electrochemical, algae, and freeze concentration have all been researched, but each has its own setbacks and drawbacks.

Struvite precipitation is a method of recovering nutrients such as phosphorus. Struvite can be recovered and used in crop production as a solid fertilizer. However, this technique uses chemicals which add second pollutant to the environment (Sena *et al.*, 2021).

Algae have been used to produce solid or liquid biofertilizer since they grow very well in wastewater while absorbing nitrogen and phosphorus nutrients which are important for crop production (Huo *et al.*, 2020). However, there are numerous challenges and technical flaws, including the appearance of second pollutant in wastewater, the high price of algal compounds, algal pollution, low availability of algae, potential cyanobacteria threats to the environment and high-water consumption as highlighted by Zou *et al.* (2020).

Ion exchange method is one of the methods that can remove and recover phosphorus to form solid fertilizer. However, presence of competing anions such as sulfates in wastewaters provide a major bottleneck of limiting selection of phosphorus (Ownby *et al.*, 2021).

Freeze concentration is reported as a promising technique for recovering nutrients from wastewater. It is also a clean technology with no secondary pollutant production, no chemical addition and low equipment erosion (Chen *et al.*, 2021). Freezing concentration technique is a physical method where a solution is concentrated by freezing out water content in the form of ice crystals (Samsuri *et al.*, 2015). The freezing method because of its low latent heat of fusion consume less energy compared to evaporation method (Moharramzadeh *et al.*, 2021). A saturated liquid and a solid crystalline phase is generated as a result of the freezing concentration technique (Lu *et al.*, 2017). This technology has numerous advantages over other procedures such as high rate of recovery, simultaneously recovering of both water and valuable minerals, and the absence of any additional supplemental information (Lu *et al.*, 2017). Freezing concentration technique has been used in wastewater treatment for solutions where the solubility of the solute is substantially dependent on temperature (Ab Hamid & Jami, 2019). Researchers have previously employed the freezing concentration technique to remediate wastewater from the pharmaceutical, chemical, fluoride removal, and chromium (VI) removal industries (Ab Hamid & Jami, 2019). However, limited studies have been done on establishing the optimal operating conditions for the freeze concentration method to

recover nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in an anaerobic digester (Ghernaout, 2020).

As a result, the goal of this research was to establish optimal conditions for the freeze concentration method to recover important nutrients from wastewater processed in an anaerobic digester. The freezing concentration performance was evaluated using nitrate-nitrogen and phosphate nutrient recovery values. At various coolant temperatures, freezing time and energy consumption, the performance of freeze concentration was examined.

1.2.   Problem Statement

Water bodies experience environmental challenges such as eutrophication due to poor management of domestic wastewater in developing countries. (Sena *et al*., 2021; Theregowda *et al.,* 2019; Sánchez*, 2020).* Meanwhile, the explosion in population of the world has resulted in a 1.8 percent annual increase in demand for fertilizers which contain important nutrients *(Yan et al.,* 2018*).* Past studies have proven that, among other techniques, the freezing method is a tidy and effective way to recover nutrients from the domestic wastewater to form liquid biofertilizer (Chen *et al.,* 2021; Mazli *et al.*, 2021). However, there are few studies to establish optimal conditions, such as temperature and time, for the freeze concentration method for recovering nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in an anaerobic digester. As a result, the goal of this research was to establish optimal conditions for the freeze concentration method to recover nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in an anaerobic digester.

1.3.   The Rationale of the Study

This research enables linkage between the agriculture sector and the sanitation sector, which will reduce or remove the problem of polluting our environment due to population growth. Also, provides good management of domestic wastewater to protect our environment and increases availability of fertilizers.

1.4.   Research Objectives

1.4.1   General Objective

To investigate the potential of producing eco-friendly liquid bio-fertilizer from domestic wastewater for crop production.

1.4.2   Specific Objectives

(i)   To establish the optimal condition such as temperature and time for freeze concentration   method to recover nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in an anaerobic digester.

(ii)   To evaluate the energy consumption for freeze concentration method to recover nitrate nitrogen and phosphate nutrients from domestic wastewater processed in anaerobic digester.

1.5.   Research Questions

(i)   What is the optimal condition of freeze concentration method to recover nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in an anaerobic digester?

(ii)   What is the energy consumption for freeze concentration method to recover nitrate-nitrogen and phosphate nutrients from domestic wastewater processed in anaerobic digester?

1.6.   Significance of the Study

The results from present study will contribute to Tanzanian government to achieve the Goal number two of Zero Hunger and Goal number six of Sanitation and Clean Water of the United Nations Sustainable Development Goals by 2030.

1.7.   Delineation of the Study

This study focused on the establishment of optimal conditions for the freeze concentration method to recover nutrients from domestic wastewater. Such optimal conditions are cooling temperature and freezing time

CHAPTER TWO

LITERATURE REVIEW

2.1.   Nutrient Recovery Methods

2.1.1   Struvite Precipitation Nutrient Recovery Method

Struvite is useful fertilizer made from phosphorus and nitrogen recovered from wastewater. Ammonium extraction and recovery through struvite precipitation from wastewater is a long-term solution that generates useful fertilizer while lowering environmental effect. In treatment plants, the use of Visual MINTEQ simplifies struvite recovery from wastewater by predicting the precipitation process (Jia *et al.*, 2017).

As inputs to the model, Jia *et al*. (2017) used different magnesium molar ratio to phosphate and a pH control mechanism. Their findings reveal that the magnesium to phosphate molar ratio has a considerable impact on ammonium recovery efficiency, with a magnesium to phosphate ratio of 2: 1 yielding over 96% ammonium recovery. Using chemicals and reagents that pollute the environment, these researchers were able to make struvite fertilizer.

Phosphorus in the form of commercial fertilizers can be recovered through struvite method. This can benefit municipal infrastructures, minimize eutrophication in urban rivers, and make further wastewater refinement for reclaimed water production easier (Sánchez, 2020).

Sánchez (2020) evaluated the viability of recovering phosphorous in a big metropolitan region's treatment plants. Using chemicals, he was able to collect phosphorus from wastewater and turn it into usable fertilizer. Phosphorus in wastewater is derived through human food metabolites and crop fertilizer uptakes, both of which are produced from phosphate rock. Using chemicals and reagents that pollute the environment, these researchers were able to make struvite fertilizer.

Theregowda *et al*. (2019) compared struvite fertilizer output to commercial fertilizer output like diammonium phosphate (DAP). The findings revealed that producing one unit of fertilizer with struvite takes one order of magnitude less energy than producing DAP, showing that struvite production is a more efficient technique. Despite the fact that struvite manufacture consumes less energy than DAP, it still involves the use of chemicals that degrade the environment.

In order to recover nitrogen from liquid side streams, Boehler *et al*. (2015) used two approaches. They demonstrated the first approach at a wastewater treatment facility (WWTP) in Kloten-Opfikon, where they treated 5 m^3/h to 7 m^3/h sludge water. They introduced a third column in addition to the scrubbing columns and standard stripping to extract carbon dioxide, lowering the sodium hydroxide requirement because of the following ammonia removal.

Boehler *et al*. (2015) began by operating and optimizing the stripping operation without any supernatant pre-treatment. Following that, activation of the carbon dioxide stripper columns and changed using gas estimation to reduce energy use, free ammonia losses, and heat losses, as well as examine important functioning parts. Finally, the stripping facility was able to feed up to 1.4 m^3/h of source-separated urine.

For the second way of ammonia removal, In 2012 and 2013, Boehler *et al*. (2015) used hydrophobic nanofibers in two small prototype systems built by different vendors at WWTP Neugu. For this method, free ammonia gas in the sewage liquid disperses in to the sulfuric acid passing through the hollow fibers of the microporous hydrophobic membrane, resulting in ammonium sulfate. Although the first approach is preferable to the second, both techniques require a lot of energy and expensive ingredients to produce ammonium sulfate, such as sodium hydroxide and sulfuric acid.

Sena *et al*. (2021) were able to recover phosphorous and nitrogen by precipitating struvite (magnesium ammonium phosphate) as shown on Fig. 1, to create a useful alternative fertilizer. This method needs the use of energy and chemicals, both of which are costly and polluting the environment.
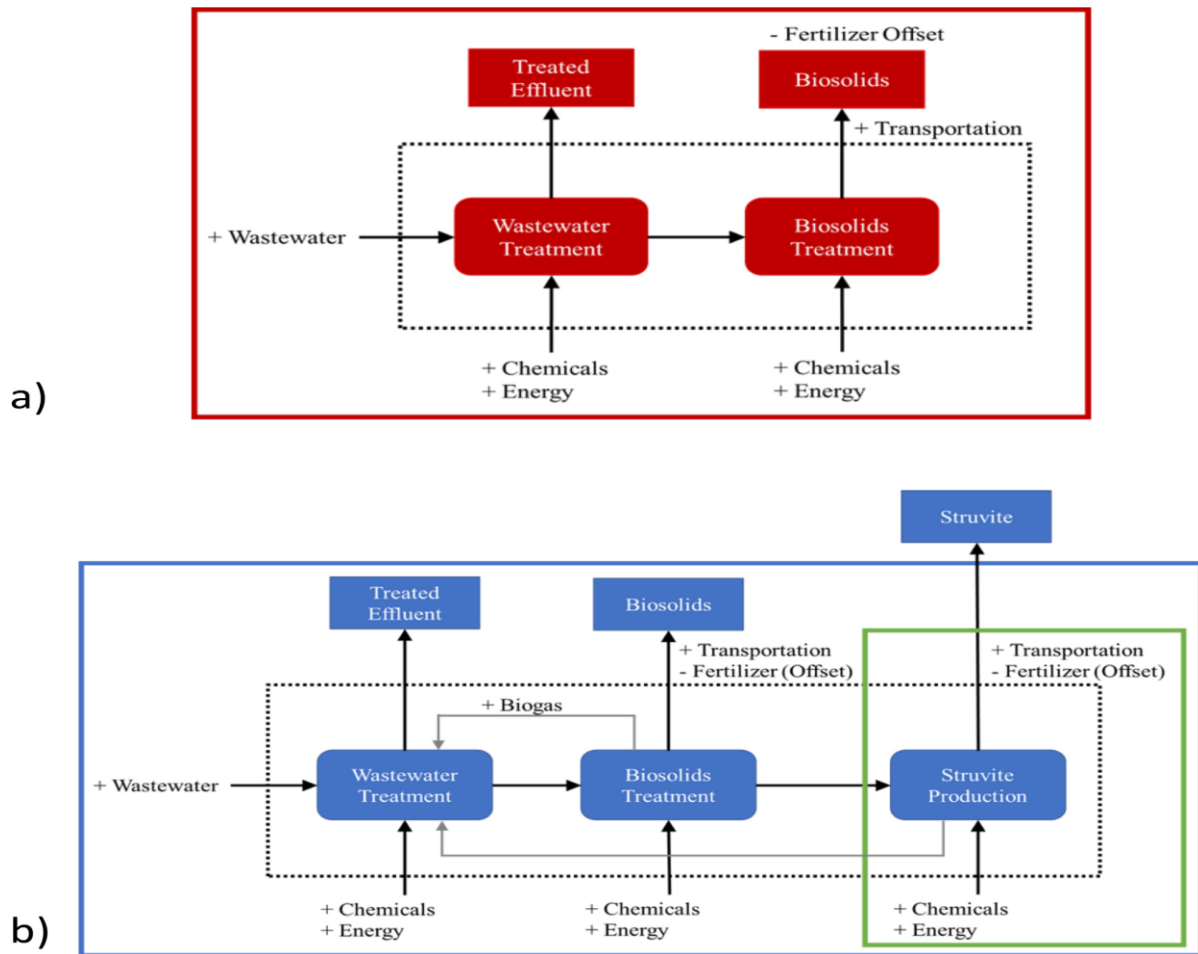
Figure 1: a) The base b) The Waste Water Treatment Plant's Struvite Recovery System (Sena *et al*., 2021)

2.1.2 Algae Nutrient Recovery Method

Due to the obvious benefits of algae biotechnology in soil improvement and nutrient recovery, Algal-based wastewater treatment and algal organic fertilizer manufacturing have recently gained popularity. Zou *et al.* (2020) emphasized the advancements made in the area of algae growing in wastewater produces biomass and algal bio-fertilizer application.

Three varieties of algal organic fertilizers are introduced, including gradual release organic fertilizers, nitrogen fixation cyanobacteria, and liquid organic fertilizers, all of which have been extensively tested and used in agriculture. However, there are numerous challenges and technical flaws, including the appearance of second pollutant, the high price of algal compounds, algal compound pollution in wastewater, low productivity of algae, potential cyanobacteria threats to the environment and high water consumption, Zou *et al.* (2020) highlights.

It has been reported by Chu *et al.* (2021) that microalgae and HTC technology as shown on Fig. 2 were used to an agricultural plot to recycle phosphorus (P) from wastewater. With starting amount of 41.3 mg/kg of P, Microcystis sp (MS) and Chlorella vulgaris (CV) were cultivated in chicken farm effluent. MS was better than CV in removing 88.4 percent phosphorus from effluent.

Hydrochars formed from MS and CV were made at 200°C or 260°C in solutions containing citric acid or distilled water. After HTC, 1% citric acid solution with the MS derived hydrochar at 260°C (MSHCA260) removed more P (91.5 percent). Charring boosted conversion of the P from a soluble and exchangeable state to a fairly accessible state. (Fe/Al-bound P) in this work, and employing citric acid solution as feedwater boosted P removal and Fe/Al-bound P production.

Chu *et al.* (2021) carried out microalgal culture in a batch experiment with a fixed start P amount of 41.3 mg/kg, which does not reflect large-scale outdoor conditions with changing P concentration.
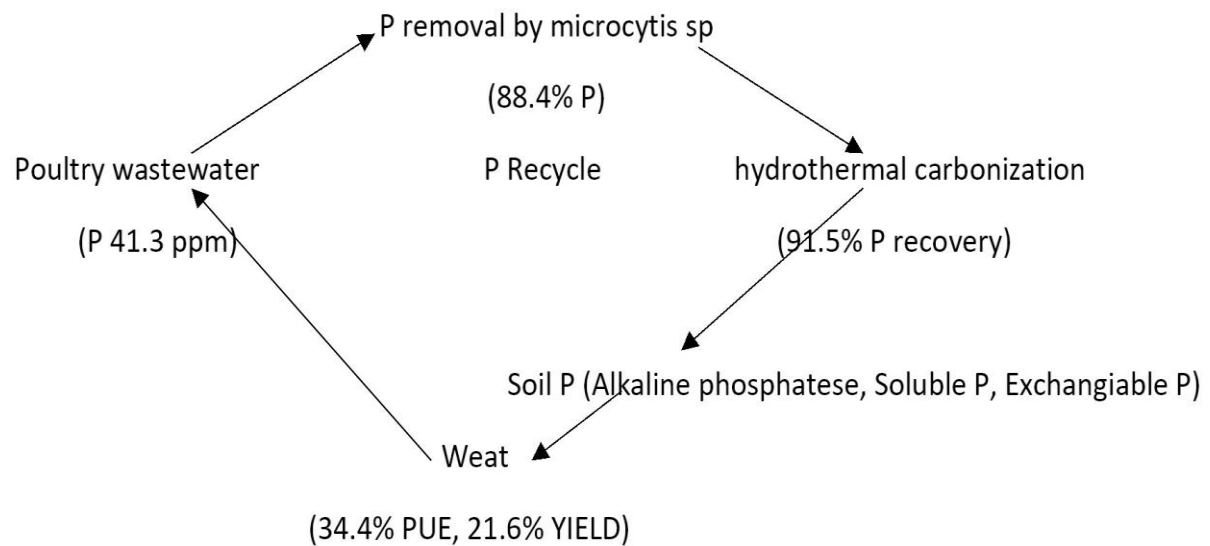
Figure 2: Phosphorus recovery and microalgae biomass conversion into slow fertilizer (Chu *et al.*, 2021)

Khan *et al.* (2019) recommended combining wastewater-based algae production with biomass harvesting as a bio-fertilizer. For nutrient removal and biomass synthesis, Nostoc muscorum Scendesmus sp and Chlorella minutissima, were cultivated in sewage effluent, and biomass with additional value was harvested to manufacture organic fertilizer.

Khan *et al*. (2019) confirmed that cultivating algae in wastewater to provide organic fertilizer with additional value is feasible. This new approach has not been widely implemented in agriculture due to the high cost of algal organic compounds, pollution of algal biomass, low output of algae and useful parameters, water loss and consumption, and effective cyanobacteria dangers to the environment.

2.1.3 Electroactive Bacteria Nutrient Recovery Method

For ammonium and phosphate removal electroactive bacteria can create an electric field. Additionally, these bacteria can change nitrate and nitrite to ammonium directly, increasing the amount of nitrogen ready for recover, according to the Li *et al*. (2020).

Electroactive bacteria as shown on Fig. 3 were used in this method to reduce nitrate and iron to create bioelectric field generators. More research is needed to figure out under the availability of nitrate and nitrite, how electroactive bacteria control their electron dispersion, how reactor design can be developed to remove ammonia gas from concentrated catholyte according to these researchers.
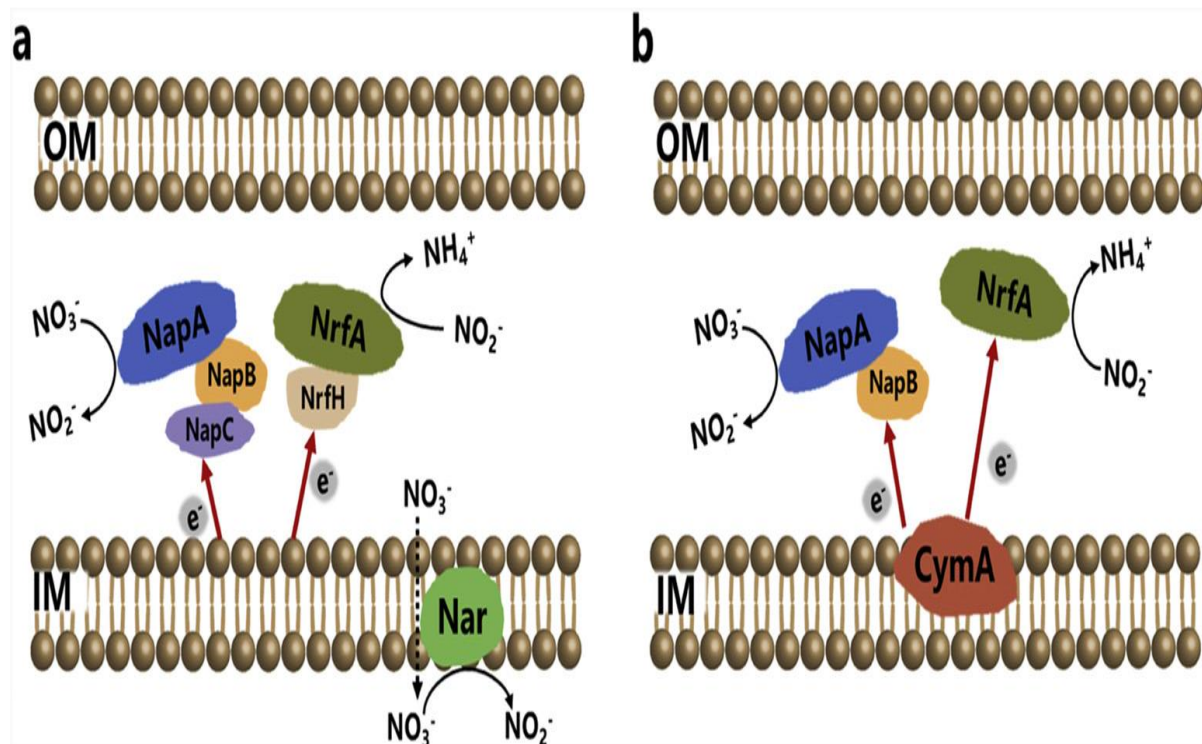
Figure 3: Electron transmission in Geobacter (a) and Shewanella (b) (Li *et al.*, 2020)

2.1.4 Magnetic Micro Sorbents Nutrient Recovery Method

Phosphate-loaded particles could well be magnetically separated from the water phase. The functionalized improved nanocomposite magnetic particles with ZnFeZr-adsorbent were created, characterized, and evaluated for direct phosphate separation and recovery from spiked secondary wastewater effluent (10 mg/L $PO_4^{3-}P$) (Drenkova-Tuhtan *et al*., 2017).

In multiple adsorption/desorption tests, Drenkova-Tuhtan *et al.* (2017) proved its reusability and durability, obtaining over 90 percent total P-recovery efficiency under optimum conditions. After treating 1.5 m^3 wastewater in 20 cycles, they conducted pilot experiments to confirm the proof-of-concept by scaling up the process and retaining recovery efficiency.

Drenkova-Tuhtan *et al.* (2017) discovered that in treated wastewater, effluent values of less than 0.05 mg/L $PO_4^{3-}P$ can be reached. They also employed a repeated application to add phosphate ions to the recovered desorption solution, achieving up to 38-fold enrichment (380 mg/L $PO_4^{3-}38$ P) relative to the original wastewater concentration. The P-rich eluate was used to initiate the struvite recovery. These authors, on the other hand, utilized extremely alkaline chemicals to regenerate adsorption material.

Lai *et al.* (2016) coated $Fe_3O_4@SiO_2$ core/shell magnetic nanoparticles with hydrous lanthanum oxide to create a readily separable adsorbent for effective phosphate recovery. Their research focused on Fe–Si–La, which was studied using XRD, TEM, XRF, magnetization and specific surface area as well as their capacity to remove phosphate.

The core/shell structure of $Fe_3O_4@SiO_2$ was verified, and it was successfully loaded with hydrous lanthanum oxide. According to their research, the newly manufactured adsorbent has a magnetization of 51.27 emu/g. By adding approximately 1 mmol lanthanum per gram of magnetite, the Langmuir adsorption capacity of phosphate by Fe–Si–La was 27.8 mg/g.

Lai *et al*. (2016) observed that adsorption was rapid, with 99% of the phosphate being removed in within 10 minutes, and that the pH range 5.0 to 9.0 was the best. When the dosage of adsorbent was greater than 0.2 kg/ton, phosphorus recovery efficiency of better than 95% was achieved for actual effluent from treatment plant.

Adsorbed phosphate may be reused with NaOH solution, according to Lai *et al.* (2016). However, challenging production processes, the use of expensive graphene as a substrate, and weak sorption capacities (27.8 mg P/g for $Fe_3O_4@SiO_2@La_2O_3$) have limited the study.

Nodeh *et al.* (2017) in their study for the phosphate and nitrate recovery from rivers and sewage systems, designed and evaluated a doped novel nanocomposite adsorbent based on nanosized lanthanum hydroxide on magnetic reduced graphene oxide. Using Fourier transform infrared spectroscopy, X-ray diffraction, and field emission scanning electron microscopy, the influence of significant factors on the efficacy of the removal process, such as adsorbent dosage, salt addition, solution pH, contact duration, and analyte concentration, was examined.

Finally, Nodeh *et al.* (2017) proved that in genuine sewage and river water samples, field application of newly produced graphene oxide offered significant phosphate and nitrate ion removal efficiency (74% to 90%). However, this research has been hampered by sophisticated synthesis techniques, the use of graphene as a substrate, which is very pricey and low sorption capabilities.

2.1.5   Ion Exchange or Adsorption Precipitation Nutrient Recovery Method

In their study, Williams *et al.* (2015) concentrated nutrients in dilute (domestic) wastewater using ion exchange (IX), then recovered them using precipitation of struvite. This study examines phosphorus recovery then after nitrogen removal and precipitation utilizing clinoptilolite IX columns and mixed regenerants.

In Williams *et al.* (2015) study phosphate recovery prior was 0.5 g P L/media to 2.0 g P L/media, yielding effluent values less than 0.1 mg /L PO_4^{3-}P and less than 0.2 mg/ L NH_4-N for greater or equal to 80 bed volumes. In the regeneration eluate, Dow-FeCu resin achieved effective P elimination, fast neutral pH production, and 560 mg P/L⁻ Prior to break-through, the exchange capacity of clinoptilolite in column mode was 3.9 N L/media to 6.1 g N L/media. The capability of exchange of clinoptilolite in column mode was 3.9 g N L/media to 6.1 g N L/media before to break-through. Using Dow-FeCu, they discovered that precipitation with mixed anion and cation producers resulted in a maximum of 74% P elimination with Al^{3+}, Ca^{2+}, and Fe impurities.

In Williams *et al.* (2015) analysis, the IX-precipitation removal procedure eliminated approximately 98% phosphorus and 95% nitrogen, with precipitates containing 13% phosphorus and 1.6% nitrogen. However, for media regeneration, they need chemicals and reagents like NaCl, MgCl, and NaOH.

Ion-exchange membrane materials made of conductive polymers could be employed. For potassium ion removal from water, Zhou *et al.* (2020) developed a unique redox transistor electrodialyzer with two chambers split by a polypyrrole membrane electrode **as** shown on Fig. 4. Polypyrrole membrane electrodes were created in their study by electrochemically depositing polypyrrole on a stainless-steel wire mesh. The polypyrrole membrane showed electrodialysis selectivity for K^+ in the presence of Na^+, with a K^+/Na^+ split factor of 2.10, based on ion-exchange data. When compared to the original polypyrrole membrane,

Zhou *et al.* (2020) added changed active carbon to it. However, these authors employed an excessive amount of energy, 3.80 kW h/kg K.
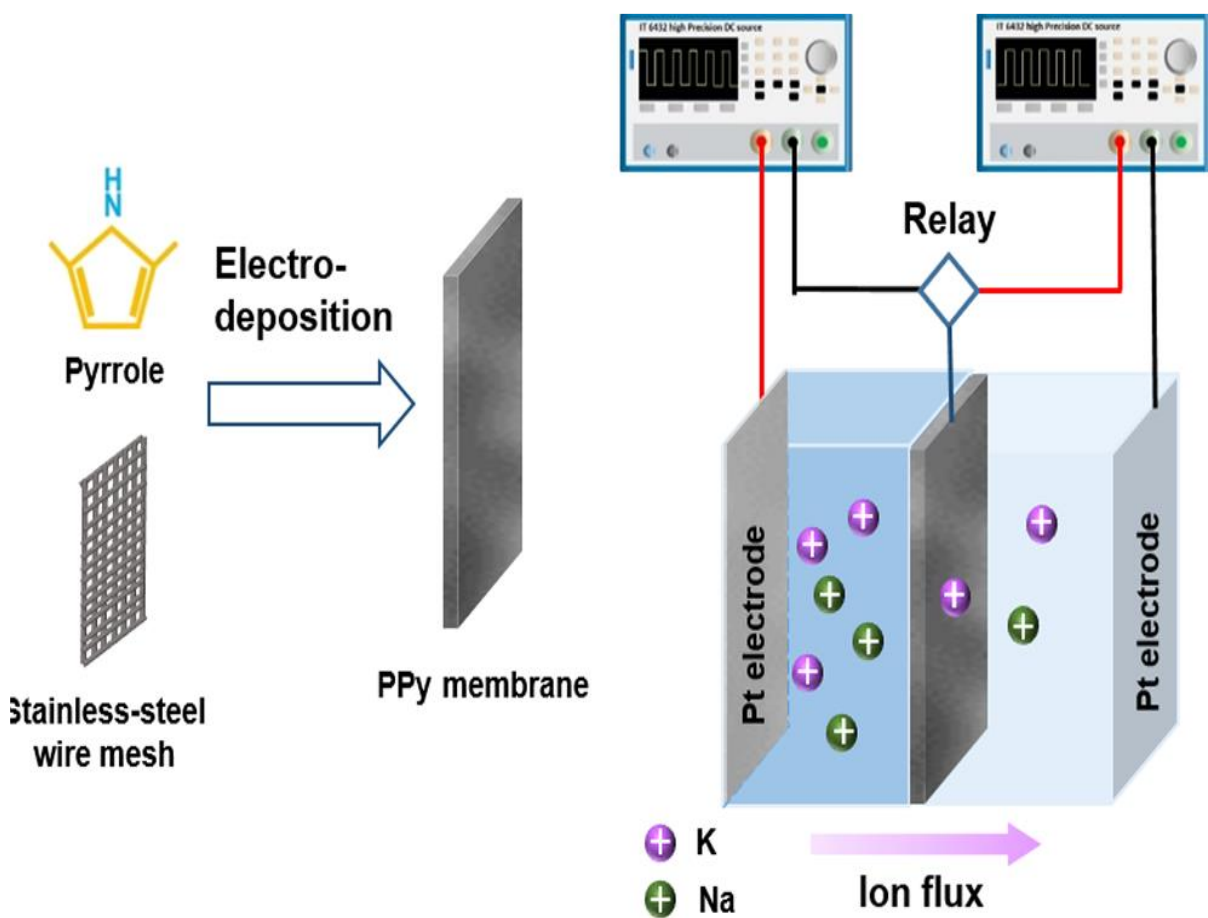

Figure 4:   Redox transistor electro dialyzer schematic (Zhou *et al*., 2020)

Liu *et al.* (2020) showed in their work that electrochemical removal is a mixture of electrodialysis and membrane removal and it may be used to selectively recover the fertilizer ammonium sulphate. These authors investigated influence of wastewater (30 mgN/L, 300 mgN/L, and 3000 mgN/L) on nitrogen elimination and recovery by electrochemical method

Liu *et al.* (2020) also constructed and tested a model of nitrogen mass transport to explain the experimental findings, offering a mechanistic explanation for the observed impacts of changing operating parameters. While modifying operational parameters did have an impact on performance, electrochemical stripping performed well throughout three gas permeable membranes, three orders of wastewater concentration, and a wide range of actual ambient temperatures.

These findings show that electrochemical stripping is feasible over a wide spectrum of waste streams and is resistant to nitrogen concentration and temperature variations, as well as establishing operational trade-offs between energy usage and residence time according to Liu *et al.* (2020). Electrochemical stripping has progressed from concept to practice as a result of this study and it has provided lessons for the development of additional resource recovery technologies. The effect on performance when the catholyte temperature is outside of these ranges (15°C, 23°C, and 35°C) is unknown.

2.1.6 Electrochemical Nutrient Recovery Method

Using electrode and intermediate chamber, ammonium (NH^4) and phosphate (PO_4^{3-}) may also be separated and concentrated from synthetic wastewater using a new reactor electrochemical with circulating cathode and anode. Based on idea of capacitive deionization, Ren *et al.* (2017) constructed a new reactor electrochemical with cyclic electrolyte flow in chambers for electrodes to continuously and selectively separate ammonium and concentrate phosphate from wastewater as shown on Fig. 5.

Phosphate and ammonium in residential wastewater concentration levels were eliminated and at a concentration ratio of 25:1 of NaCl solution, struvite was recovered continuously and between the electrode chambers and the center chamber selectively. Ren *et al.* (2017) also demonstrated that, operation for a long time suggested the ability to continue the reaction indefinitely and confirmed long-term stability.

Ren *et al.* (2017) used chemicals such as $MgCl_2 6.H_2O$ when it comes to long-term operations, which are expensive and pollute the environment.
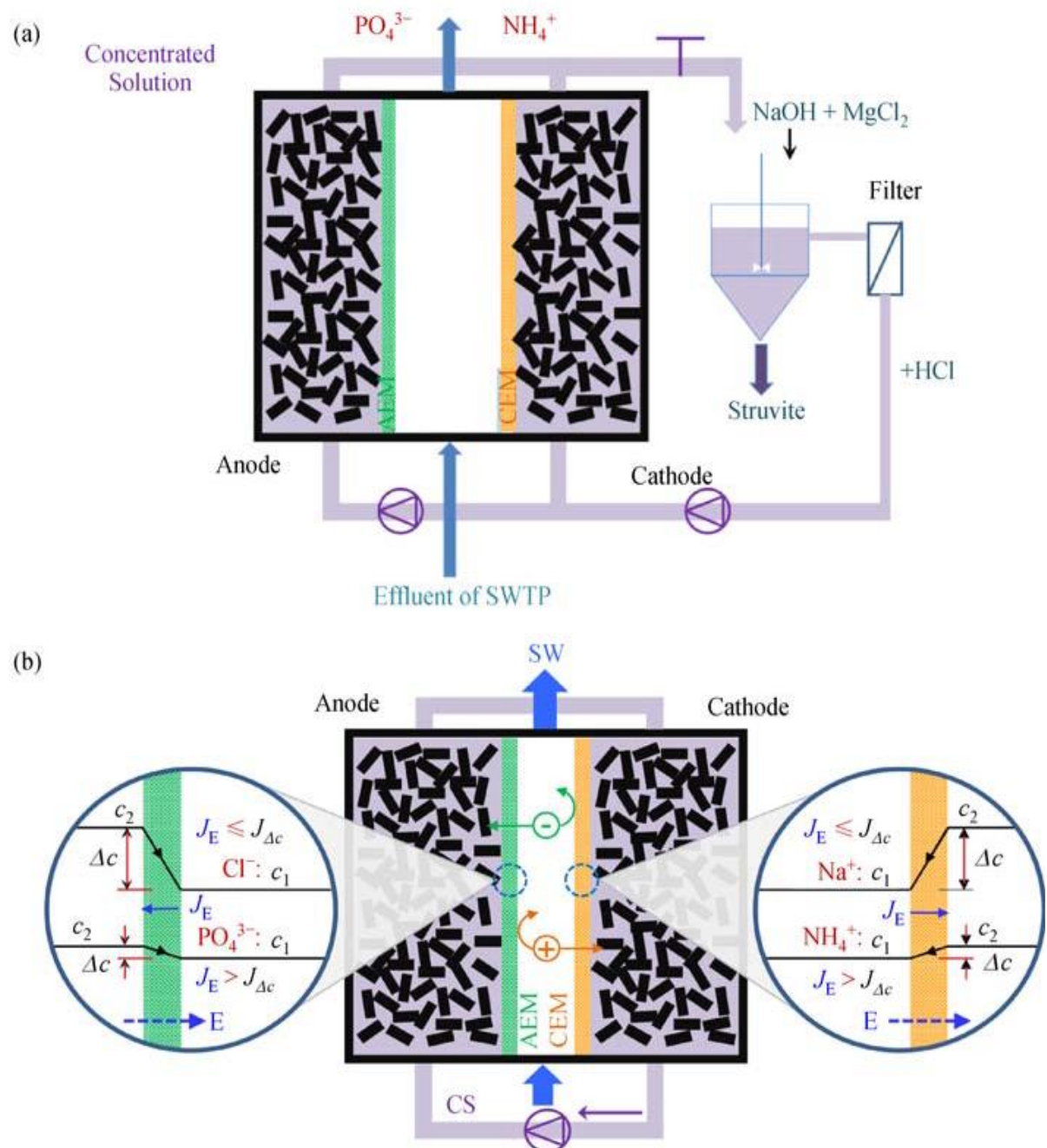
Figure 5 **(a)** **Schematic diagram of an electrochemical reactor-based laboratory-scale ammonia and phosphate recovery system.** **(b) The difference in concentration between the middle chamber and the electrode chamber (Ren *et al*., 2017)**

CHAPTER THREE

MATERIALS AND METHODS

3.1.   Wastewater Samples

The feed water for the freezing concentration trials in this investigation was real domestic wastewater effluent from two anaerobic biodigester and gravel filter treatment plants put in sequence located at The Nelson Mandela African Institution of Science and Technology in Arusha Tanzania for establishing optimal condition (temperature and time) and for comparison purposes. The wastewater for the tests was collected from the inlet and outlet of each treatment plants as shown on Fig. 6.
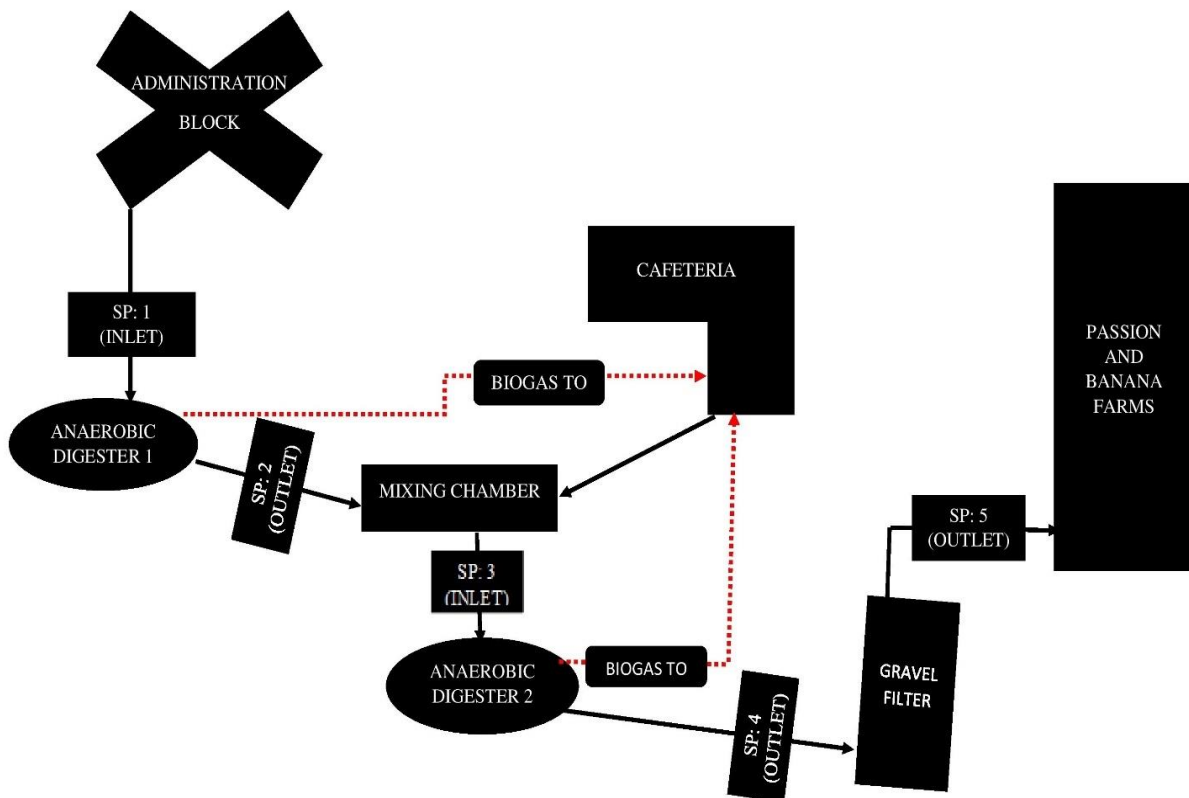
Figure 6:   Process flow of anaerobic biodigesters and gravel filter treatment plants

Domestic wastewater is a complicated mixture of accumulated chemicals and contaminants in water. Due to varying flow rates induced by water usage and precipitation, the quality and composition of the wastewater also varies on a regular basis.

3.2. Experimental Setup

The setup of the apparatus for the Freezing concentration process is shown in Fig. 7. Thermal scientific freezer model number 713 CD and 813 CV were used for temperature of -10℃ to -40℃ and -50℃ to -80℃ respectively. This freezer provides power and control temperature. The average energy consumption of the coolant were 8.1 kWh/day and 11.6 kWh/day for temperature of -10℃ to -40℃ and -50℃ to -80℃ respectively.

Figure 7:   Experimental setup for freezing concentration

3.3.   Experimental Procedure

At the inlet and outlet of each anaerobic biodigesters and a gravel filter treatment plant, wastewater samples were collected. Samples were filtered by using Cellulose Nitrate Filter, pore size 0.45 µm before being placed in the sample vessel during the experiment. Following the sample filtering and measurement of initial concentration as shown on Table 1, 400 mL of wastewater added to the sample plastic vessel for freezing concentration process.

Table 1:   Average initial physical and chemical characteristics of the sample

Sample points	Physical parameters			Chemical parameters	
	pH	EC (µS/cm)	TDS (mg/L)	Nitrate-N (mg/L)	Phosphate (mg/L)
SP1	7.33	856	428	5.8	10.1
SP2	7.17	881	441	6.1	10.1
SP3	7.19	759	381	5.3	8.5
SP4	7.30	1005	503	4.1	8.4
SP5	7.09	980	467	3.8	8.1

Vessels containing the sample solution were placed in the freezer and the operating parameters were adjusted as needed. The coolant temperature and time tested in this study were as shown on Table 2. The sample vessel was removed after the freezing concentration procedure was finished under the specified operating conditions. The volume and concentration of the concentrate (unfrozen liquid) and melt solution (solution from melted ice) were measured and recorded by using standard techniques for the Examination of Water and Wastewater (APHA, 2012). Value of nutrient recovery efficiency was used to determine efficiency of freezing concentration process (Ab Hamid & Jami, 2019).

Table 2:   Values of coolant temperature and freezing time tested

Freezing Time (Hours)	Coolant Temperature (°C)
1 to 8.00	-10
1 to 7.00	-20
1 to 5.50	-30
1 to 4.50	-40
1 to 4.25	-50
1 to 4.00	-60
1 to 3.75	-70
1 to 3.50	-80

3.4. Analytical Procedure

Using graduated cylinders (500 mL), the volumes of the initial input water sample (collected water samples before freezing method), unfrozen liquid (samples of concentrate formed after freezing method), and melting ice samples (samples of melted ice formed after freezing method), were collected and measured. Standard techniques for the Examination of Water and Wastewater were used to measure nitrate-nitrogen (NO_3-N) and phosphate (PO_4^{3-}) nutrients (APHA, 2012).

The analysis of Nitrate-Nitrogen (NO_3-N) concentration was measured by using DR 900 UV-Visible spectrophotometer method number 355 HR. Phosphate (PO_4^{3-}) was measured by using the same spectrophotometer method 490 HR.

3.5. Data Analysis

Research information have been analysed via way of means of one of a kind statistical software program inclusive of Origin Pro nine and Excel. Descriptive information has been accomplished to summarize the information obtained, to set up the overall performance of the remedy structures in phrases of nutrient recovery value and to attract consultant graphs of the device overall performance in nutrient recovery.

3.6. Nutrient Recovery Efficiency

Nutrient recovery is the ratio of nutrient amount in the unfrozen liquid to that in the original solution (Ab Hamid & Jami, 2019). To get the nutrient recovery, Eq (1) was used.

$$\text{Nutrient recovery} = \frac{c_L}{c_O} \tag{1}$$

Where, C_L is the amount of nutrient in concentrated solution (mg/L), while C_O is the amount in the original solution (mg/L). A higher value of nutrient recovery gives a good efficiency of the freezing concentration process.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1. General Information

By examining nutrient recovery, coolant temperature effect, time and energy consumption on the freezing concentration process was explored. Freeze concentration efficiency in the system was given by measuring variations in value of nutrient recovery from the wastewater. Ice crystals were seen growing on the vessel once the freezing concentration process was completed under the specified operating conditions.
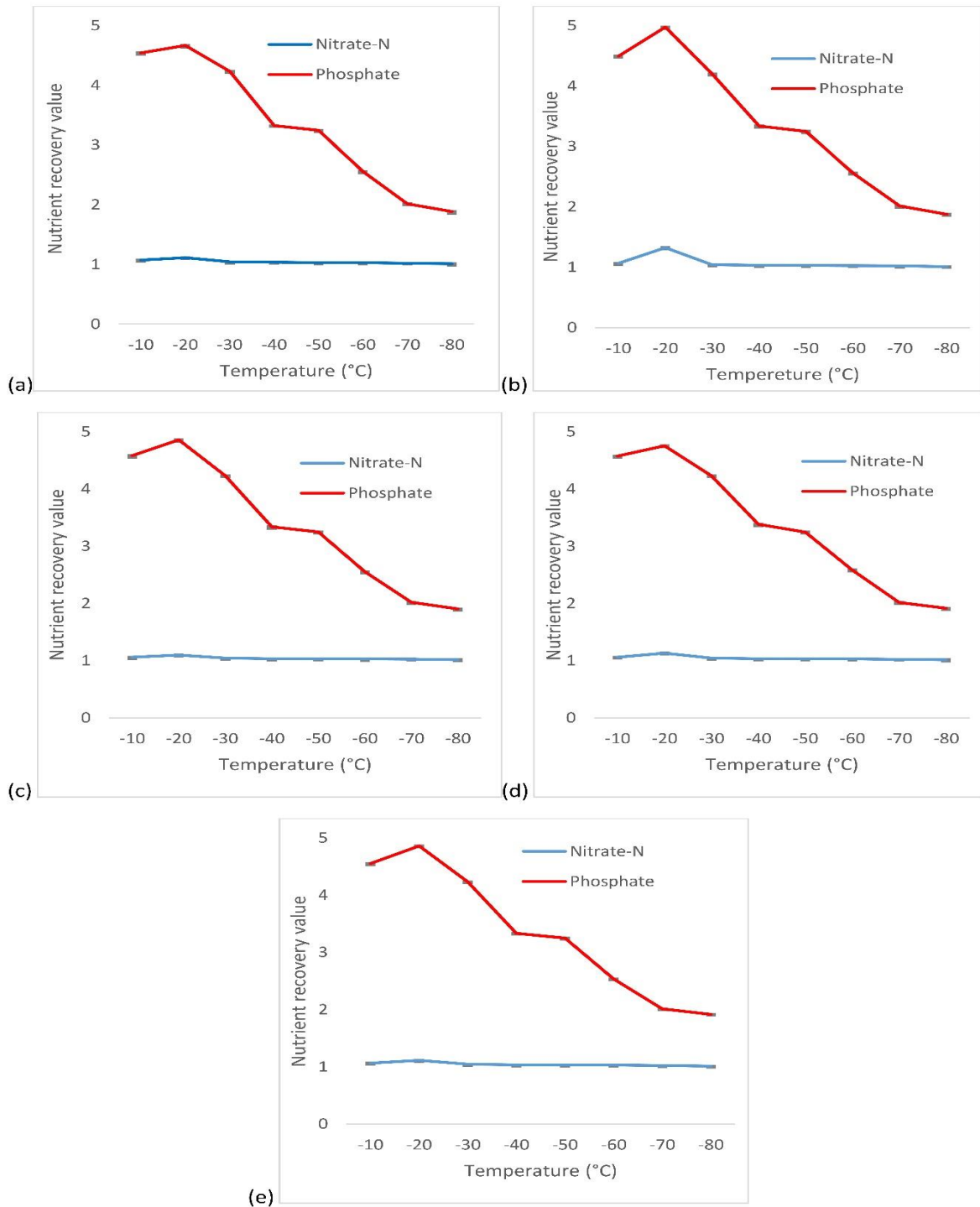
4.2. Effect of Coolant Temperature

Since it gives a freezing rate, the freezing temperature is important in controlling the process (Melak *et al.*, 2016). Amount of temperature among the freezing temperature, the surface temperature, and the temperature of the wastewater causes transfer of heat between freezer and wastewater solution across the vessel wall surface during the freezing concentration process (Ab Hamid & Jami, 2019). Difference in temperature between the freezer and wastewater solution is directly related to the heat transfer rate (Samsuri *et al*., 2015). The heat transfer rate increases as freezing temperature is lowered. As a result, a lower coolant temperature is better, as this improves transfer of heat between the freezer and wastewater. A lower temperature is observed at the surface when there is lower freezing temperature providing an acceptable supercooling environment for ice formation.

The effect of freezing temperature on the operation of a freezing concentration system was investigated using nutrient recovery values as a metric. Figure 8 shows the relationship between temperature of coolant and the average value of nutrient recovery at different points of treatment plants. It can be observed that nutrient recovery value was increasing with decreasing in coolant temperature from -10°C to -20°C and the maximum nutrient recovery was reached at coolant temperature of -20°C for samples collected from inlet and outlet of both anaerobic digesters and gravel filter treatment plants. Minimum temperature will result in higher nutrient recovery value (Samsuri *et al*., 2015; Amran & Jusoh, 2016). As the freezer temperature reduced, difference in temperature between freezer and wastewater became high, implying the increase in the heat transfer rate which results to a better efficiency of the freeze concentration performance (Ab Hamid & Jami, 2019).

However, as the freezer temperature was reduced further from -30°C to -80°C, the trend shifted, and the nutrient recovery value declined marginally. This circumstance suggests that the process' efficiency has decreased. The discrepancy could be due to the supercooling effect, which occurs when the freezing temperature is at its lowest (Ab Hamid & Jami, 2019) . The lower nutrient recovery value was caused by the supercooling phenomena which reduced the effectiveness of the freezing concentration process. The supercooling effect speeds up the creation of an ice crystal layer, resulting in more solute inclusion in the side of the ice. Furthermore, depending on the findings of Ab Hamid & Jami (2019) minimum temperatures gave a higher growth rates of ice crystals, resulted into poor purity of ice produced. It's also important to note that temperature of -20°C was chosen as the optimal temperature for this system for both sample points because of its better nutrient recovery value.

The observed disparities in nutrient recovery of nitrate-nitrogen and phosphate by freezing concentration could be due to the morphology of the ice created, which was a multi-crystalline dendritic ice structure that held more nitrate-nitrogen than phosphate. Also, it could possibly be due to molecular weight and size discrepancies between nitrate-nitrogen and phosphate (Gu, 2016).

Figure 8: Effect of coolant temperature on nitrate-nitrogen and phosphate nutrient recovery at (a) Anaerobic digester 1inlet (SP1) (b) Anaerobic digester 1 outlet (SP2) (c) Anaerobic digester 2 inlet (SP3) (d) Anaerobic digester 2 outlet (SP4) (d) Gravel filter outlet (SP5)

4.3.   Effect of Freezing Time

In general, a longer freezing duration could increase the freezing concentration process's efficiency (Safiei *et al.*, 2017; Amran & Jusoh, 2016). With the occurrences of dendrites structure, the amount of crystallinity of the generated ice was low when the freezing process began. By extending the freezing period, the ice area thickens and causing the unfrozen fluid in a state close to saturation (Yang *et al.*, 2017). Figure 9 and Fig. 10 show the trends of nitrate-nitrogen and phosphate nutrient recovery respectively at different freezing time from the samples collected at different treatment plants.

Nutrient recovery value increases as the freezing time increases from 1 hour to 8 hours,1 hour to 7 hours, 1 hour to 5.5 hours, 1 hour to 4.5 hours, 1 hour to 4.25 hours, 1 hour to 4 hours, 1 hour to 3.75 hours and 1 hour to 3.5 hours at coolant temperature of -10°C, -20°C, -30°C, -40°C, -50°C, -60°C, -70°C and -80°C respectively for both samples.

In this analysis, 7 hours freezing time at -20°C coolant temperature was enough to give good results of the process together with highest nutrient recovery value. At this condition, the average value of nutrient recovery of nitrate-nitrogen and phosphate were 1.114 and 4.667 respectively at the inlet of anaerobic digester 1, 1.325 and 4.975 respectively at the outlet of anaerobic digester 1, 1.099 and 4.859 respectively at the inlet of anaerobic digester 2, 1.132 and 4.755 respectively at the outlet of anaerobic digester 2 and for gravel filter at the outlet the values where 1.111 and 4.861 respectively. Nutrients at outlet of anaerobic digester 1 where highly recovered compared to other sampling points.

However, at -30°C, -40°C, -50°C, -60°C, -70°C and -80°C the maximum freezing time were reached at 5.5 hours, 4.5 hours, 4.25 hours, 4 hours, 3.75 hours and 3.5 hours respectively because of supercooling effect as described by Ab Hamid and Jami (2019). This result is in agreement with the findings of Moussaoui *et al.* (2021), Safiei *et al.* (2019) and Azman *et al.* (2018).
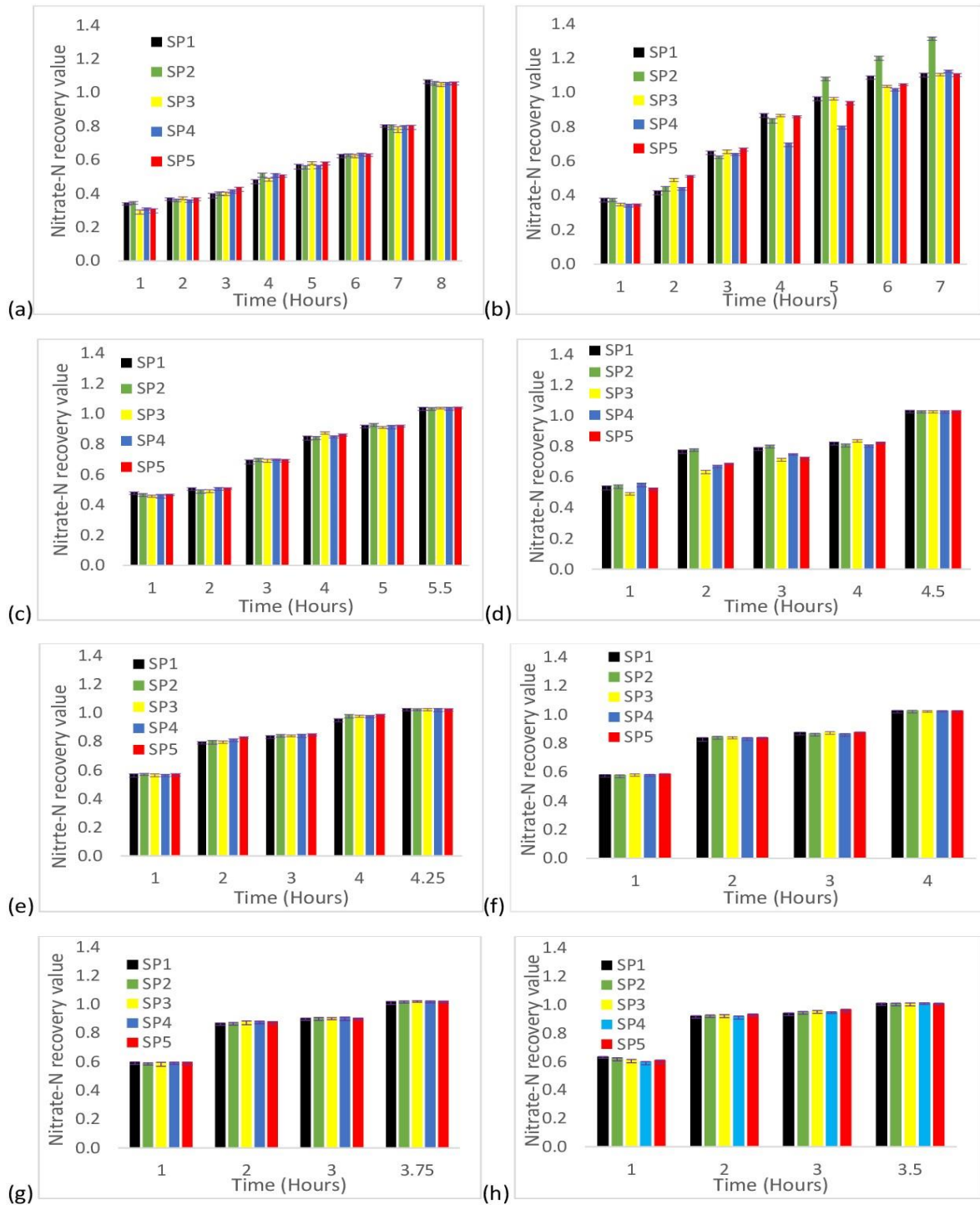
Figure 9: **Nitrate-nitrogen nutrient recovery value at different freezing time at a coolant temperature of (a) -10°C (b) -20°C (c) -30°C (d) -40°C (e) -50°C (f) -60°C (g) -70°C (h) -80°C**
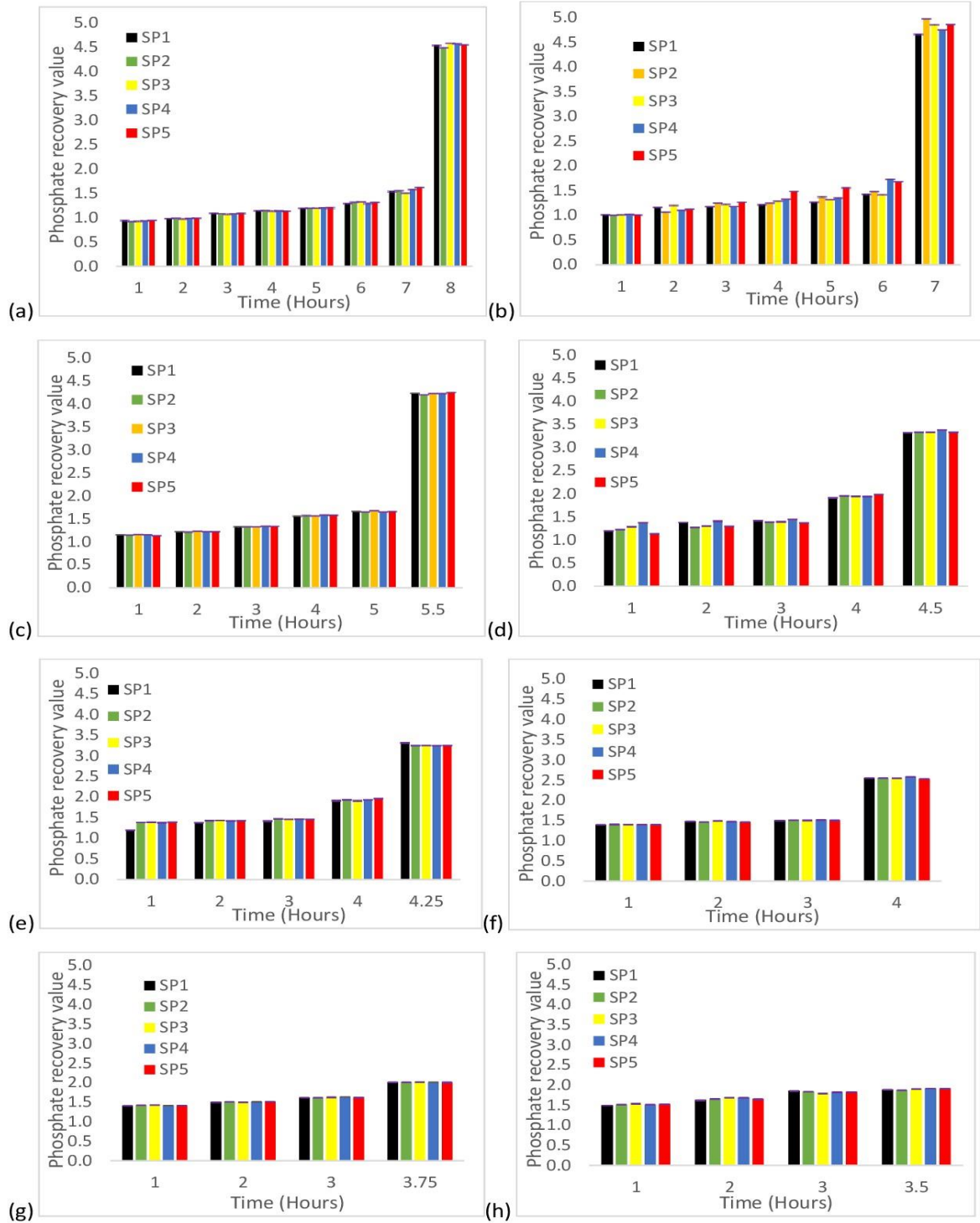
Figure 10: Phosphate nutrient recovery value at different freezing time at a coolant temperature of (a) -10℃ (b) -20℃ (c) -30℃ (d) -40℃ (e) -50℃ (f) -60℃ (g) -70℃ (h) -80℃

4.4. Analysis on Energy Consumption

One of the major problems in several industries is to reduce energy consumption and operational costs (Ghannadzadeh & Sadeqzadeh, 2016). For innovative resource recovery techniques to be adopted and established in the agricultural sector, it is essential to evaluate their economic implications. As a result, the present study examined the energy consumption of the entire process as an extra criterion to establish optimal condition for the freezing concentration process to recover nutrients from domestic wastewater. Heat transfer and energy utilized per unit volume of the wastewater sample processed were used to analyse energy consumption. A comparison of energy consumption and coolant temperature is shown on Table 3.

Table 3: Energy consumption at different coolant temperatures

Freezing Time (Hours)	Energy consumption (kWh/L)	Coolant Temperature (°C)
1 to 8.00	0.225	-10
1 to 7.00	0.197	-20
1 to 5.50	0.155	-30
1 to 4.50	0.127	-40
1 to 4.25	0.171	-50
1 to 4.00	0.161	-60
1 to 3.75	0.151	-70
1 to 3.50	0.141	-80

When compared to membrane method, which is widely used to concentrate wastewaters and recover particular components, freezing concentration process uses the same amount of energy during operation, if not less. Comparing freeze concentration to heating and evaporation procedures, there is a great potential for energy savings (Uald-Lamkaddam *et al.,* 2021). The amount of energy used depends on the technology being used, the feed solution being used, the ambient temperature, the desired recovery rate, and the electricity cost.

In comparison to the energy consumption of the most effective evaporation systems there is savings of up to 30% of energy while using a freeze concentration process, recovering

nutrients, and combining with the film method (Pazmiño *et al.,* 2017*)*. Mtombeni *et al.* (2013) further claimed that its use of freeze concentration technique to recover nutrients resulted in the lowest energy consumption of 0.39 kWh. This study's findings indicate that employing freezing concentration system, 1 litre of a domestic wastewater will require 0.197 kWh energy to recover maximum nutrients. However, one of the most important considerations for separation and concentration technologies is the capital and operating expenses (Uald-Lamkaddam *et al.*, 2021). While other separation techniques like ammonia stripping, thermal treatment, ion exchange, and adsorption may need more energy input and be influenced by factors like pH and aeration, freeze concentration method is recognized as an environmentally friendly method, simple to operate, uses low energy and has high rate of rejection (Shi *et al.*, 2018). However, freezing facilities for -20°C to -80°C are mostly practical at lab scale.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

5.1. Conclusion

The possibility of using a freezing concentration method to recover nutrients from domestic wastewater was proven in this study. This method, which is based on the freezing concentration mechanism, has been used in separation liquid and solid e.g., in food and pharmaceuticals industries and it has been effectively used for seawater desalination. Nutrient recovery values were used to assess freezing temperature, cooling time and energy consumption on the effectiveness of the freezing concentration method. It is critical to run this process to get the optimal nutrient recovery value. The freezing temperature of −20°C and cooling time of 7 hours gave the highest nutrient recovery value during the process. The amount of energy consumed by the coolant at this particular condition was 0.197 kWh/L. In general, the results show the possibility of the freezing concentration method and give conditions that can be used to recover nutrient from domestic wastewater. The recovered nitrate-nitrogen and phosphate nutrient can be used as the biofertilizer in agricultural production while solving the environmental problems.

5.2. Recommendations

Further analysis is needed on the use of this method in recovering of heavy metals, emerging compounds and other organic compounds from domestic wastewater. Cost benefit analysis should be done on the applicability of this method for commercial or large scale and how the recovered nutrients can be used in agriculture and stored. Finally, the set-up of the equipment and stability of the produced nutrients should be checked in the future studies.

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RESEARCH OUTPUT

Publication Paper

Muga, E. C., Komakech, H. C., & Jande, Y. A. C. (2023). Establishing the Optimal Condition for Nutrient Recovery from Domestic Wastewater Using the Freeze Concentration Method. *Journal of Water Practise and Technology*, 111, 1–10. http://dx.doi.org/10.2166/wpt.2023.111

Poster Presentation titled

Establishing the Optimal Condition for Nutrient Recovery from Domestic Wastewater Using the Freeze Concentration Method.