**ADDIS ABABA UNIVERSITY**

**SCHOOL OF GRADUATE STUDIES**

**ADDIS ABABA INSTITUTE OF TECHNOLOGY (AAiT)**

**CENTER FOR RENEWABLE ENERGY**

# Investigation of combined treatment methods on biogas slurry (BGS) concentration and nutrient recovery

A THESIS SUBMITTED TO THE SCHOOL OF GRADUATE STUDIES OF ADDIS ABABA UNIVERSITY IN PARTIAL FULFILLMENT OF THE DEGREE OF MASTER OF SCIENCE IN RENEWABLE ENERGY TECHNOLOGY

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# DECLARATION

I, **Eniyew Abebaw Tsegaye**, declare that this thesis is the result of my own work and that all source and material used for this thesis have been duly acknowledged. This thesis is submitted in partial fulfillment of the requirement for Master of Science in Renewable Energy Technology at Addis Ababa University and to be made available at the at the university's library under the role of the library. I confidently declare that this thesis has not been submitted to any other institutions anywhere for the award of any academic degree, diploma, or certificate.

Name: Eniyew Abebaw Tsegaye

Signature: ------------------

Date: 06/12/2023

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## Investigation of combined treatment methods on biogas slurry (BGS) concentration and nutrient recovery

By**:** Eniyew Abebaw Tsegaye

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# ABSTRACT

Biogas slurry (BGS) is generated as byproduct during biogas production and can be used as organic fertilizer. However, Biogas slurry (BGS) application is limited due to its bulkiness and nutrient volatilization in the form of ammonia.

Hybrid treatment methods which include chemical treatment, physical separator and evaporation process were used to concentrate bulky biogas slurry. Principal nutrients (P, and $NH_4$-N) distribution in solid and liquid fraction of BGS was examined after chemically treated with the addition of coagulant and flocculant and physical separations by using vibrating screen. The result showed that significant portion of readily available soluble ammonium ($NH_4$-N) was presented in liquid fraction with distribution share of 91% and a significant amount of phosphorous, P (68.2%) found in solid fraction.

The influences of heating time (30, 45, and 60 minutes), heating temperature (65, 75 and 85 $^0$C) and pH (7.76, 7 and 6) on water removal (WR) efficiency during evaporation of BGS liquid fraction was investigated. The highest WR efficiency (55%) was obtained at 75 $^o$C, 45 minute and pH of 7. The effectiveness of $NH_4$-N recovery was examined at different pH (6, 7 and 7.76) and maximum $NH_4$-N (95.1% ) recovery were achieved at heating time of 45 min., temperature of 75 $^o$C and pH of 6.

The final concentrate biogas slurry (CBGS) had a higher nutritional concentration and was less bulky than the raw biogas slurry. This effort helps to prevent the challenge faced associated with transporting of bulky BGS from biogas digester to application sites, reduce imported chemical fertilizer dependency and promote sustainable farming.

**Key words:** Biogas slurry, Energy security, Nutrient recovery, Agricultural productivity

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# LIST OF ABERVATIONS

AAU- Addis Ababa University
AD-Anaerobic digestion
BGS- Biogas slurry
C/N- Carbon nitrogen ratio
CBGS- Concentrated biogas slurry
CEC-Cations exchange capacity
CHP - Combined heat and power
CO-Carbon monoxide
$CO_2$- Carbon dioxide
$C_t$- Condensate
DAF-Dissolved air floatation
DM-Dry matter
EC-Electrical conductivity
$FeCl_3$-Iron chloride
$H_2S$- Hydrogen sulfide
$H_2SO_4$- Sulfuric acid
HBGD-House hold biogas digester
Km- Kilo meter
Kwh- Kilo watt hour
LBGD-Larger biogas digester
LF- Liquid fraction
$LF_1$- Concentrating liquid fraction
$LF_2$- Evaporator concentrate
MSBGD-Medium size biogas digester
$NH_4$-N- Ammonium
NPK-Nitrogen, phosphorous and potassium
NR- Nutrient recovery
OC-Organic carbon

RBGS- Raw biogas slurry

SF- Solid Fraction

$SF_1$- Solid fraction

S-L-Solid-liquid

TS-Total solids

VOC-Volatile organic carbon

VS-Volatile solid

WHC- Water holding capacity

WR-Water removal

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# 1. INTRODUCTION

Biogas slurry (BGS) is a nutrient rich residues generated during anaerobic digestion (AD) of organic wastes (food waste, cow manure, agricultural wastes) during biogas production process in a biogas digester. The slurry comprised of essential plant nutrients and significant amount of water (Bonten et al., 2014). This resource is known as "brown gold" due to its use as organic fertilizer, bio pesticide and seed dresser (de Groot and Bogdanski, 2013). Nutrients in biogas slurry (nitrogen) are more readily available in the form of $NH_4$-N which result significant short term fertilization effect for plant growth (Kumar et al., 2022). However, its application is hindered by its bulkiness (high water content up to 90%), and difficult to transport from biogas plant to the point of application. Recent study indicated that biogas slurry (BGS) management mostly in the form of transport and spreading costs, account up to 40% of the entire cost of a biogas plant (Bonten et al., 2014). Research to save transport cost have been done by removing water and reduce the volume of the slurry while enhancing its nutritional content (Wang et al., 2022).

BGS can be separated into liquid fraction with a greater mineral nitrogen (N) and potassium (K) content and a solid fraction with a higher organic matter and phosphorous (P) content (Verbeke et al., 2021). The liquid fraction is further treated to reduce its bulkiness and creating an end product with higher nutrient concentrations (that contain both minerals and organics) than the raw biogas slurry. Biogas slurry evaporation was presented as one of the efficient methods in reducing volume of water for its operational reliability, ease of integration with existing biogas plant, water reuse, excess biogas and waste heat utilization (Vondra, Máša, and Bobák, 2018). Evaporation is usually accompanied with other pretreatment technologies such as introducing flocculants and coagulant prior to mechanical filtration (Drosg, 2013 and Vaneeckhaute et al., 2017). These techniques are useful to retain nutrient, to improve evaporation efficiency by reducing fouling at heat exchange surface, prevent fibers possibly clogging evaporators, reduce viscosity, low pH that ensure low ammonia concentration and decrease ammonia volatilization (Monlau et al., 2015; Vaneeckhaute et al., 2017; Vondra, Máša, Touš, et al., 2018; Verbeke et al., 2021).

However, the effectiveness of water removal (WR) efficiency and nutrient concentration depend on different conditions including type of additive used, additive dosage, filtration mechanisms, heating temperature, time and acidification. Different research has been conducted on the effect of these parameters on BGS concentration (Devarenjan et al., 2019; Rafiee et al., 2021). Bonmati et al, (2022) conducted evaporation of pig slurry at different pH (4, 5, 6) to evaluate nutrient concentration, condensate quality and economic feasibility of the system and showed that parameters were strongly dependent on acid treatment ( Kumar et al., 2022). With the complexity of anaerobic digestion, feedstock type and location of the digester, the characteristics of the BGS varies accordingly. Hence, site specific investigation is needed to evaluate the effect of process parameters such as additive dosage, solid-liquid separation and evaporation operating conditions on essential nutrient recovery, water removal efficiency and condensate quality. Despite few Biogas slurry (BGS) chain streaming efforts as part of biogas dissemination Program are going on, still further research work has to perform for sustainable BGS utilization in Ethiopia.

Therefore, the current study will make an attempt to evaluate the effect of various treatment methods and operational conditions for maximum water removal and nutrients recovery efficiency. Hereby the BGS is treated with coagulants and flocculants at various dosages and separated into solid and liquid fractions using screens having different mesh sizes. Rotary evaporator operating at different operating conditions was used for BGS concentration purpose. The end product is expected to have higher nutrient concentrations (that contain both minerals and organics) than the raw biogas slurry.

## 1.1.Problem Statement

Addressing the challenges of BGS in biogas plants in a sustainable and an efficient way is indispensable. Despite of its proven use as organic fertilizers, pesticides and seed dresser, BGS handling and application is hindered by its high water content and essential nutrient volatilization in the form of ammonia gas. BGS is bulky because it contains a lot of water (90%), which makes it more difficult to store, transport and apply it to fields not close to the digester. Moreover, the bulkiness of BGS reduces its ability to meet the full nutrients need in agricultural fields (Bonten et al., 2014 and (A. Kumar et al., 2022). Nitrogen loss via volatilization of ammonium is the main draw backs of BGS treatment and application which observed at the time of drying or soon after its application in agriculture fields (Selvaraj et al., 2022). Moreover, the piling of BGS at the proximity of the plant causes environmental problems, occupy space and create economic challenge instead of being source of income especially in medium and large size biogas plants.

Different researches have been conducted on BGS water volume reduction and minimize essential nutrient loss using different treatment conditions. However, due to the nature of BGS is dependent on feedstock, places and other operational conditions, investigating the effect of treatment conditions which is specific to the study taking into consideration is important. This particular study is, thus, to analyze the effect of different treatment conditions for maximum water removal efficiency and essential nutrient recovery. For this purpose, the BGS sample was taken from medium size biogas digester (MSBGD) installed at Sululeta, Ethiopia.

To this end, the research helps for understanding the appropriate technologies, optimizing conditions, energy recovery alternatives and water recycling options, enhances biogas slurry main streaming efforts and foster biogas dissemination strategies.

## 1.2. Objectives

### 1.2.1. General objectives

The aim of this study is to develop concentrated biogas slurry (CBGS) without the loss of essential nutrient ($NH_4$-N) using combined treatment methods. These hybrid treatment methods include chemical additives, mechanical solid-liquid separation and thermal evaporation of water. The volatilization of nutrient during evaporation is minimized by treated inlet liquid fraction with acid. The overall goal is to produce less bulky, higher nutrient concentration end product that can be used as sustainable organic fertilizer.

### 1.2.1. Specific objectives

- To collect and characterize raw biogas slurry (RBGS)
- To pretreat RBGS using chemical additives (flocculent and coagulant)

- To separate treated BGS into solid and liquid fraction by vibrating screens physically
- To investigate the effect of different operating conditions (temperature, time and pH) on WR efficiency and nutrient recovery ($NH_4$-N) during evaporation
- To characterize the various end products during pretreatment and concentration of BGS

## 1.3. Significance and Scope of the Study

The value of BGS is fairly high as it contains readily available nutrient ($NH_4$-N). In Ethiopia, farmers are getting positive results by using bio-slurry as an organic fertilizer, bio pesticide and seed dresser. Acknowledging the benefit of seeds dressing for quick returns, high coverage and ease of management, the Ethiopian Ministry of Agriculture urges farmers to adopt bio-slurry for seeds dressing.

The reduction of the bulkiness of the BGS helps for ease of transportation to the point of application, minimize $CO_2$ emissions from transportation raw BGS and used as tool for biogas technology dissemination strategies. Sustainable use of value added BGS as affordable organic fertilizer reduces consumption of natural gas that could be used as raw materials for making chemical fertilizers and increase biogas digester owner’s income.

The scope of the research was partially removing the water content of the BGS with minimum essential plant nutrient loss using combined treatment methods. The evaporation-condensation process of BGS concentration was accompanied with pretreatments (both chemical and physical). Cooled water content was also examined for its ammonium content as it indicate at what extent the essential nutrient is lost with the water vapor. The goal is providing a lab-based reliable research output for further BGS value addition effort. This effort enhances soil fertility, agricultural productivity, reduce imported chemical fertilizer dependency and promote sustainable farming.

The economic and environmental feasibility of the topic is not included in this study due to time and financial limitations. Furthermore, due to financial limitations, the impact of chemical dosage and screen mesh sizes on the distribution of nutrients in the solid and liquid fraction was not analyzed.

# 2. LITERATURE REVIEW

## 2.1. Anaerobic Digestion (AD)

An aerobic digestion (AD) is a process of converting organic materials into gas mixture and biogas slurry (BGS) without the presence of oxygen.

Biogas is mainly comprised of methane and carbon dioxide and other traces of water vapor, hydrogen sulfide, ammonia, nitrogen, hydrogen and oxygen (Awe et al., 2017) (Martin et al., 2020). Typical value for a biogas composition is presented in Table 2.1. The concentration of gases had crucial importance to its energy content and effective utilization. The presence of impurities is undesired as it reduce the calorific value of biogas and create various operational problems on machines (Struk et al., 2020). Higher content of methane is an indication of higher energy value. Whereas presence of higher concentrations of other trace impurities such as $H_2S$ severely damage downstream processes. Hence, these impurities have to be cleaned by using different upgrading technologies before utilize to different applications (Struk et al., 2020)(Andriani et al., 2020).

Table 2-1Biogas composition (Siyal et al., 2023) and Martin et al., 2020)

| Component | Content (%) |
|---|---|
| Methane ( $CH_4$ ) | 50-75 |
| Carbon dioxide ( $CO_2$ ) | 25-50 |
| Ammonia ($NH_3$) | 1-2 |
| Nitrogen ($N_2$) | 0.5-3 |
| Hydrogen ($H_2$) | 1-10 |
| Water vapor ($H_2O$) | 2-8 |
| Carbon monoxide (CO) | 1 |
| Hydrogen sulfide ($H_2S$) | 0-3 |

The efficiency of the anaerobic digestion (AD) is dependent on feedstock quality, pretreatment type and operating conditions. Usually feedstock quality is determined in terms of its organic matter and digestibility conditions (Struk et al., 2020). Treatment methods such as mechanical, thermal, chemical and biological enhance digestibility of the substrate (Gikas et al., 2018).Operating conditions including pH, temperature, Carbon to nitrogen (C:N) ratio, hydraulic retention time (HRT) and organic loading rate (OLR) highly affect biogas yield and biogas slurry quality (Zamri et al., 2021).

The acidity or alkalinity (pH) of the process determines the stability of the activity of microorganism inside the digester and ideally neutral pH range is considered as the optimum value for AD process (Mao et al., 2015). Temperature plays a major role in biogas production through the stability of enzymes and co-enzyme activity. AD process can be operated in psychrophilic (10–30 ◦C), mesophilic (30–40 ◦C) or thermophilic (50–60 ◦C) conditions. Commonly, mesophilic temperature condition is applied as it is optimal for microbial stability and diversity (Labatut et al., 2014). Moreover, the C: N ratio of present in organic materials and an optimum C: N ratio is generally required for an effective AD process. Ideally, the optimum C:N ratio for the AD process is within the range of 20–35 (D. Li et al., 2015). Organic loading rate (OLR) is detrimental to maximum biogas yield and typically, a decrease in biogas yield indicates an excessive degradation capacity of the reactor due to high OLR. Usually, in organic digestion, the OLR range from 1.2 to 12 kg of VS/m$^3$/day (Li et al., 2015; Martin et al., 2020).

The process of AD takes place in sealed vessel called biogas digester (BGD) which is designed and constructed with different size and shapes depending on feedstock conditions and specific to the sites (Obileke et al., 2021). The biogas digester has the following main components (Deng et al., 2020) (Figure 2-1).

- Inlet tank: It is a tank where feedstock is diluted with water before feeding into the digester. The feedstock source might be various organic wastes (Manure, municipal waste, agricultural, industrial) and latrine connection.

- Digester: It is a chamber where organic materials are decomposed by microorganisms in an oxygen free environment and generate biogas and biogas slurry.
- Gas holder: It is a gas storage dome or membrane either integrated to the digester or separated container which biogas is stored until it gets consumed.
- Slurry collection pit: It is a collection pit where excess biogas slurry is collected and stored before application. The biogas slurry is expelled from the digester as a result of gas pressure developed inside the system.

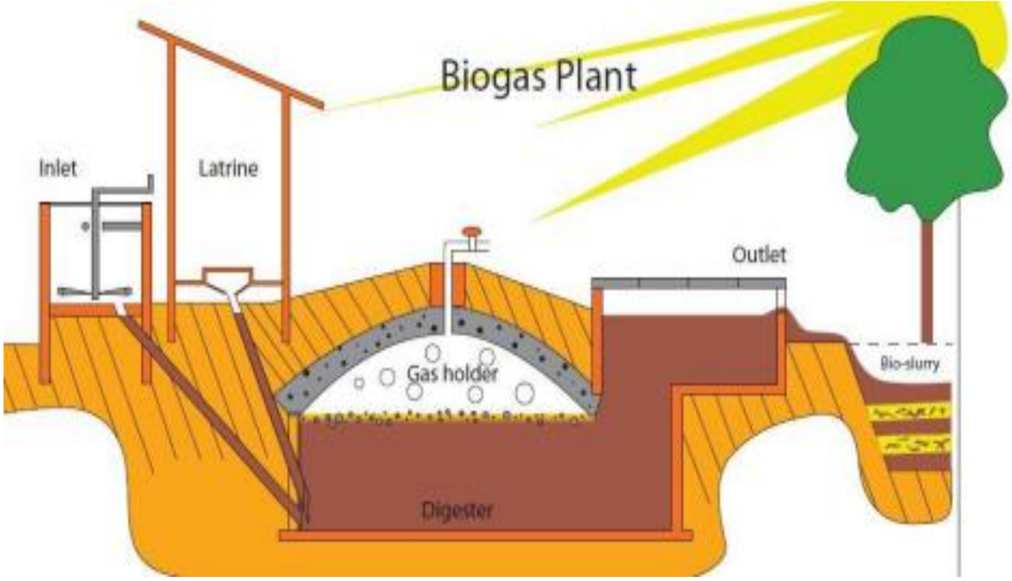

Figure 2-1Biogas digester (Smith, 2014)

## 2.2. Biogas Energy: Opportunities and Challenges

Biogas technology has a number of benefits. It addresses the growing problems related to energy security, organic waste management, and agricultural productivity and greenhouse gas emissions. Huge amounts of organic residues from municipals, agriculture and industry are converted into useful energy and biogas slurry (BGS) at moderate to high temperatures (Bhajani, 2022; Ghysels et al., 2020). The content of total solids (TS), also known as dry matter, decreases during AD process and the BGS can contain 50-80% less TS compared to the incoming substrate (Drosg et al., 2015). With this, 20-50% of waste reduction could be attained at landfills only by AD of organic waste in biogas digester. The biogas technology also contributes to green electric power production, off-grid lighting alternatives, indoor pollution free cooking, greenhouse gas reduction, nutrient recycling and sustainable farming (Ahmed et al., 2021). It enhances smaller holder farmers' income, reduce vulnerability to women and children and encourage sustainable natural resource use. Moreover biogas technology creates an option to the reduction of petroleum and large-scale use of fossil fuels and associated greenhouse emissions (Awe et al., 2017).

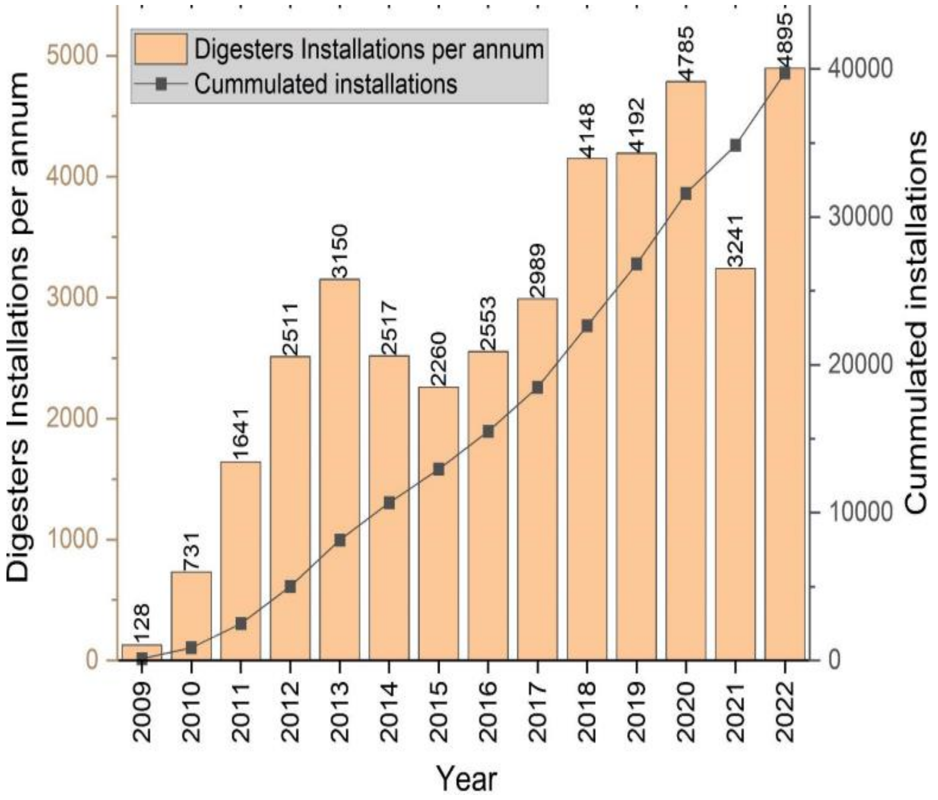

Figure 2-2 Biogas digester installation progress in Ethiopia (Mohammed et al., 2023)

Though, the biogas technology is endowed with all these above benefits, its dissemination efforts are still at infancy stage especially in developing countries such as Ethiopia (Mohammed et al., 2023). In Ethiopia, Biogas technology started in 1960's and up to now only 40,000 biogas digesters were constructed (Mohammed et al., 2023). Lack of access to finance, the lack of awareness among potential customers about the benefits of the technology (biogas and biogas slurry), low functionality of installed digesters, limited expertise on the sector and limited private sector participation are some of the reason behind its unsatisfactory expansion ( Mohammed et al., 2023).

Usually biogas digester installation is encouraged on the basis of its clean energy for cooking and lighting (de Groot and Bogdanski, 2013). However, BGS main streaming can be taken as one tool to promote biogas technology. The technology contributes to sustainable agricultural practice, soil fertility improvement, water recycling, waste management and better sanitation (de Groot and Bogdanski, 2013). Chemical fertilizer shortage and its increased prices, government support and carbon revenues are opportunities to promote biogas technology in perspective of biogas slurry. Recently the Ethiopia’s Ministry of Agriculture and other non-governmental organization (such as SNV) includes biogas slurry in agricultural extension services and an entry point for biogas dissemination effort (Mohammed et al., 2023).

### 2.3. Biogas Slurry (BGS)

Biogas slurry (BGS) is generated as byproduct during anaerobic decomposition of organic matter at biogas digester. Typically, 25-30% of the organic matter (volatile solids) is converted into biogas during AD process whereas the remaining 70-75% is generated as biogas slurry (BGS)(Drosg et al., 2015). BGS is a mixture of water (93%) and solid matter (7%) (Bonten et al., 2014). The BGS contains essential plant nutrients (macro and micro) and bioactive compounds that plays an important role in promoting overall soil and plant health management ( Kumar et al., 2022; Vondra, Máša, Touš, et al., 2018).

The composition of biogas slurry (BGS) strongly depends on the characteristics of the feedstock (biodegradable wastes) and type (cow dung, chicken manure, food waste) and AD process parameters (Vaneeckhaute et al., 2017). Recent study reported that BGS contains 2.55% N, 0.57% P, and 1.77% K (Kumar et al., 2022). Moreover, sun dried BGS reported C, N, P, and K content of 41.6±2.1%, 0.72±0.12%, 0.59±0.02%, and 0.91±0.04% respectively (Sharma et al., 2021). It is clear that the content of nitrogen significantly affected during BGS drying.

Ammonium ($NH_4$-N) is one of the most important and readily available plant nutrient contained in BGS. It is released during the degradation of organic nitrogen compounds during AD process. The concentration of ammonium depends on substrate type, type of digestion (co- or mono- digestion), amount of fresh water and degree of recirculation ( Wang et al., 2022). On the other hand, the content of phosphorous (P) is not affected by AD process. It completely depends on the substrate content (Fouda, 2011).

The pH value of fresh BGS typically ranges from 7.5 to 8.0. This is little higher than the average pH (7.1) of undigested manure (Drosg et al., 2015). This increment of pH is justified by the removal of $CO_2$ as result of transformation of $CO_3^{2-}$ and $H_3O^+$ to $CO_2$ and $H_2O$ (Hjorth et al., 2009). The concentration of cations such as $Ca^{2+}$ and $K^+$ also reported to contribute to the rise of pH in biogas slurry (Hjorth et al., 2009). Consequently, soluble ammonium volatilization (nitrogen lost) favored in the form of ammonia increase with alkaline conditions. Acid treatment of liquid fraction is reported as major factor to reduce nutrient loss during evaporation (Wang et al., 2020). That why BGS injection, incorporation with soil, impermeable bottom and covered storage tanks are recommended to prevent N loss during storage and application (Bonten et al., 2014). The average content of BGS obtained from typical household and larger biogas digester is given at Table 2.2.

Study showed that 2 $m^3$ of biogas digester produce up to 50 Liters of BGS per day. With this regard, 0.16 to 1.05 kg of nitrogen which is equal to approximately 0.35 to 2.5 kg of urea can be prepared per $m^3$ of BGS ( Herbertet al., 2019). Recent data revealed that 2 kg of Nitrogen, 0.4 kg of phosphorus, 0.78 kg of potassium and 0.0019 kg of boron can be obtained per $m^3$ from house hold derived biogas slurry in Ethiopia (Abera et al, 2022).

Table 2-2 Biogas slurry composition (Abera et al., 2022)

| Parameter | Unit | HBGD | LBGD |
|---|---|---|---|
| pH | | 7.33 | 7.15 |
| Total nitrogen (TN) | % | 2.94 | 3.58 |
| Phosphorous (P) | mg/L | 395.04 | 459.54 |
| Potassium (K) | mg/L | 7888.86 | 1066.34 |
| Calcium (Ca) | mg/L | 1064.17 | 484.09 |
| Magnesium (Mg) | mg/L | 281.31 | 219.5 |
| Sodium (Na) | g/kg | 157.34 | 447.50 |
| Sulphur (S) | mg/L | 204.15 | 162.84 |
| Iron (Fe) | mg/L | 349.43 | 127.98 |
| Manganese (Mn) | mg/L | 44.74 | 21.84 |
| Copper (Cu) | mg/L | 0.96 | 0.69 |
| Zinc (Zn) | mg/L | 1.56 | 1.11 |
| Boron (B) | mg/L | 1.91 | 1.36 |
| Organic carbon (OC) | % | 39.45 | 45.15 |
| Dry matter (DM) | % | 6.78 | 4.92 |

### 2.4.Uses of Biogas Slurry (BGS)

Biogas slurry (BGS) is a residual biomass, which hosts a diverse microbial population, and carrying essential micro/macro-nutrients. The use of BGS as fertilizers depends on its nutrient contents. The beneficial effects of biogas slurry in agriculture are summarized as follow ( Kumar et al., 2022):

- Biogas slurry is a source of macro (N, P, Ca, Mg, Na, S) and micro nutrients (Zn, B, Fe, Cu…)
- It uses as bio pesticides, anti-fungal, termite, repellant, nematicidals
- It promotes growth of essential microbes (nitrogen fixing bacteria, phosphate solubilizing bacteria)
- It maintains physiochemical properties of soil (Soil structure, air permeability, cations exchange capacity (CEC), water holding capacity (WHC))

- Improve level of biochemical in plants (amino acids, proteins, soluble sugars, plant pigments)

Moreover, BGS has the potential of improving soil structure through input of inert organic matter and fibers (primarily lingo-cellulose), which contributes to the formation of humus in the medium to long term (Fuchs & Drosg, 2013).

BGS can be applied as a foliar spray, diluted liquid, and dry composted form and be used directly or added with other organic materials and synthetic fertilizers (Godoi et al., 2021). Though, farmers are getting positive results by using bio-slurry as an organic fertilizer, pesticide and in recent times as a seed dresser, there are still challenges regarding its management due to its bulkiness and nutrient volatilization.

### 2.5. Limitation of Biogas Slurry (BGS)

In spite of its fertigation profile, the handling and application of biogas slurry (BGS) is hindered due to its bulkiness, high pH, ammonium loss as ammonia volatilization, reduced rates of C/N transformation (Kumar et al., 2022). The bulkiness in BGS is because of high water content (~93%), which affect its storage, transport and utilization of biogas slurry (Bonten et al., 2014); (Kumar et al., 2022). The large volume and low concentration of BGS create storage, transport and application costs burden to farmers. A study indicated that BGS management mostly in the form of transport and spreading costs, account up to 40% of the entire cost of a biogas plant (S. Kumar et al., 2015). BGS have pH values usually in the alkaline range and the higher pH (>7),the higher it promotes N loss (Bonten et al., 2014). In BGS, risk of contamination is not considerable for pathogens are killed during anaerobic digestion at mesophilic or thermophilc conditions. However, the presence of heavy metals in the substrate (sewage and industrial substrate) more than the threshold level, persistent organic pollutants and other chemical pollutants may be a barrier for marketing of BGS products (Fuchs & Drosg, 2013). Hence, investigating the risk of BGS contamination before further application is necessary (Kumar et al., 2022).

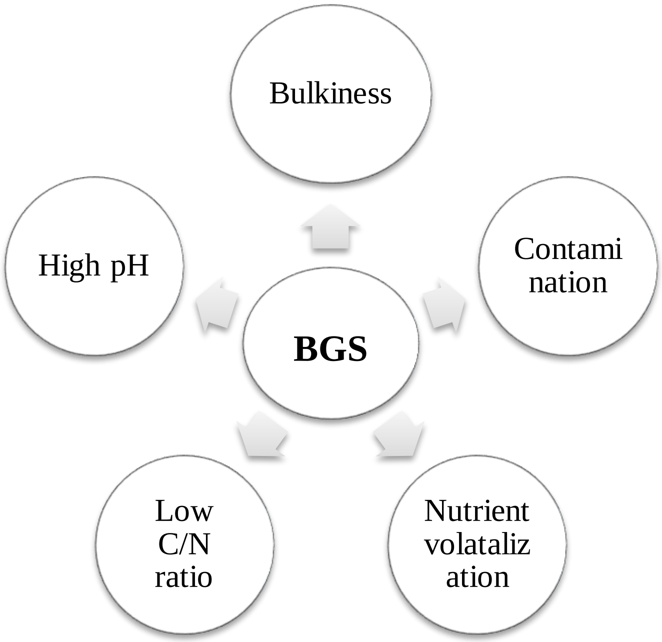

Figure 2-3Limitation of biogas slurry adopted from Kumar et al. 2022 (A. Kumar et al., 2022)

### 2.6.Biogas Slurry (BGS) Treatment

There are different technologies employed to concentrate biogas slurry (BGS). These technologies include solid-liquid separation, nitrogen stripping-scrubbing, evaporation and condensation, phosphorus stripping and precipitation and drying (Verbeke et al., 2021). Each method varies in terms of separation efficiency, nutrient recovery rates and costs (Verbeke et al., 2021).Solid-liquid separation is used to separate solid and liquid fraction and it is the first steps in biogas slurry treatment. All technologies except (whole biogas slurry drying) employ solid-liquid separation technology as prior steps (Drosg et al., 2015). The solid–liquid separation techniques include screw press, belt filters, centrifuge, floatation, screens and filters and the technologies are chosen based on their investment cost and separation efficiency (Fuchs & Drosg, 2013).

After solid-liquid separation, the solid fraction of BGS can be further stabilized by composting and drying (Verbeke et al., 2021). On the other hand, the liquid fraction is concentrated by membrane or evaporation technologies (Devarenjan et al., 2019). Nitrogen and phosphorus can be recovered by using nitrogen stripping-scrubbing and phosphorus stripping-precipitation respectively (Drosg et al., 2015). These processing technologies differ from each other in terms of their simplicity, technology maturity, investment and operation cost. Cost wise, membrane is the most expensive technology with high optimization possibilities in large scale application. Whereas evaporation and condensation is interesting options, if excess energy is available (Herbes et al., 2020).

Evaporator and condensation was presented as one of the efficient methods for BGS concentration. It is preferred due to its operational reliability, ease of integration with existing biogas plant, water reuse, excess biogas and waste heat utilization (Vondra & Bob, 2018). Evaporation and condensation approaches are usually accompanied with other pretreatment technologies such as chemical and physical treatment (Fuchs & Drosg, 2013)(M. Hjorth et al., 2009)( Figure 2-4).

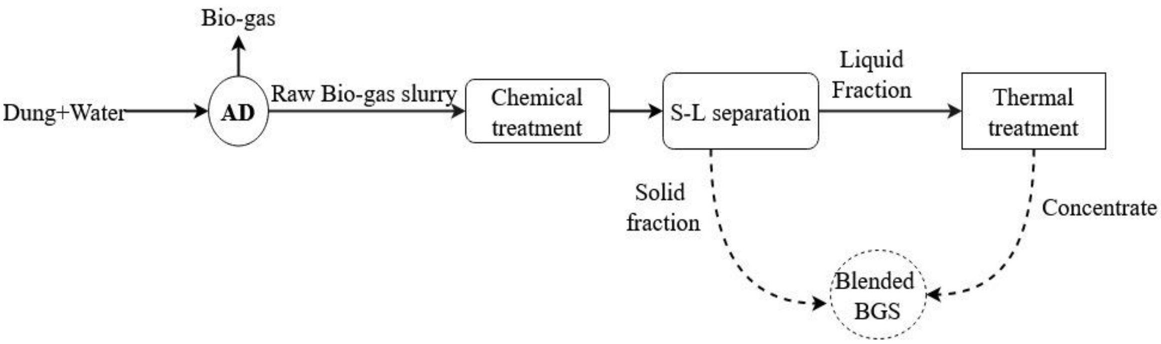

Figure 2-4Selected biogas slurry treatment routes

The effectiveness of biogas slurry (BGS) concentration depends on various factors. These factors include physical and chemical characteristics of the slurry, solid-liquid separation technologies efficiency and operating conditions. The physical characteristics of biogas slurry (BGS) comprises of particle size, dry matter, moisture content, density and viscosity (Fuchs & Drosg, 2013). Particularly BGS particle size distribution is the major factor which determines the solid-liquid separation efficiency (Dottorato, 2013). Likewise, biogas slurry dry matter content and viscosity affects the thermal efficiency of evaporator. The chemical characteristics refer nutrient content of BGS such as nitrogen, phosphorous, potassium, magnesium, calcium, organic carbon, electrochemical properties and micro elements (Zn, B, Fe, Mn…)(Dottorato, 2013).

Different methods of separating BGS into solid and liquid fractions have been developed (Vaneeckhaute et al., 2017). The separation efficiency of these methods is affected by type of additive used, additive dosage and type of separators. Once the solid fraction of BGS is separated, further processing of liquid fraction and essential nutrient recovery efficiency depends on different factors.

### 2.6.1. Chemical and Physical Treatment

Raw biogas slurry (RBGS) consists of colloid and fine particles, which have a negative charge and are stable in water. Hence, the physical separation of BGS is usually conducted together with chemical treatment by adding coagulant and flocculants.

Coagulation and Flocculation are chemical pre-treatments process that improves the mechanical solid–liquid separation of many suspensions. The solid-liquid separation of BGS is usually accompanying by adding coagulant and flocculants. These additives are used to retain soluble nutrient in the solid fraction of BGS during filtrations. Additives are also useful to retain nutrient, to improve evaporation efficiency by reducing fouling at heat exchange surface, prevent fibers possibly clogging evaporators, reduce viscosity, low pH that ensure low ammonia concentration and decrease ammonia volatilization (Monlau et al., 2015; Vondra, Máša, & Bobák, 2018).

These are accomplished by the formation of particle aggregate between the negatively charged BGS particles and the positive charge additives. The positive charged coagulant ($Fe^{3+}$) neutralize by adsorbing the negatively charged particles in the BGS. Ions such as carbonate ($CO_3^{2-}$) and Hydroxyls ($OH^-$) contribute to the negative charge of slurry (Maibritt Hjorth et al., 2009). Because of this, cationic polymer-based biogas slurry flocculation has a greater impact on the effectiveness of solid-liquid separation (Maibritt Hjorth et al., 2009).

The addition of polyelectrolyte polymers to slurry induces flocculation by polymer bridging reaction mechanism (M. Hjorth et al., 2009). Hence, an enhanced separation of solid and liquid fractions at separator can be achieved by selecting appropriate additive type used and dosage. Maximum coagulation and flocculation with minimum chemical dosage require an optimization process (Arachchige et al., 2023).

Large numbers of coagulants are commercially available. A few examples of these coagulants include multivalent cations like $FeCl_3$, $Al_2$ (SO4)$_3$, CaO (Maibritt Hjorth et al., 2009). It is reported that addition of $FeCl_3$ and $Al_2$ ($SO_4$) is more preferable as they decrease pH and hinder ammonium volatilization ($NH_3$ emissions)(M. Hjorth et al., 2009). Multivalent cations causes coagulation of BGS particle by neutralize negatively charged ions and removing the electrostatic barrier that prevents aggregation (Hjorth et al., 2009). In this regard, optimum dose rates are essential to prevent counter acting aggregation. The optimal amount of coagulant added varied between 50-350 mg/L of BGS and this value depend on the type of biogas slurry (Verbeke et al., 2021).

Similarly, Polyelectrolyte polymer (PEP) facilitate flocculation and contain anion or cation groups of varying molecular weights, charge densities and molecule shapes polymer ( Hjorth et al., 2009 and Verbeke et al., 2021). Study indicates that a cationic polymer is superior to anionic which correlates well with the fact that the particles in BGS are mainly negatively charged (Hjorth et al., 2009). Polymers of medium charge density (20–40 mol%), branched polymer and larger molecular weight have been shown to be most efficient in flocculation of BGS ( Hjorth et al., 2009).

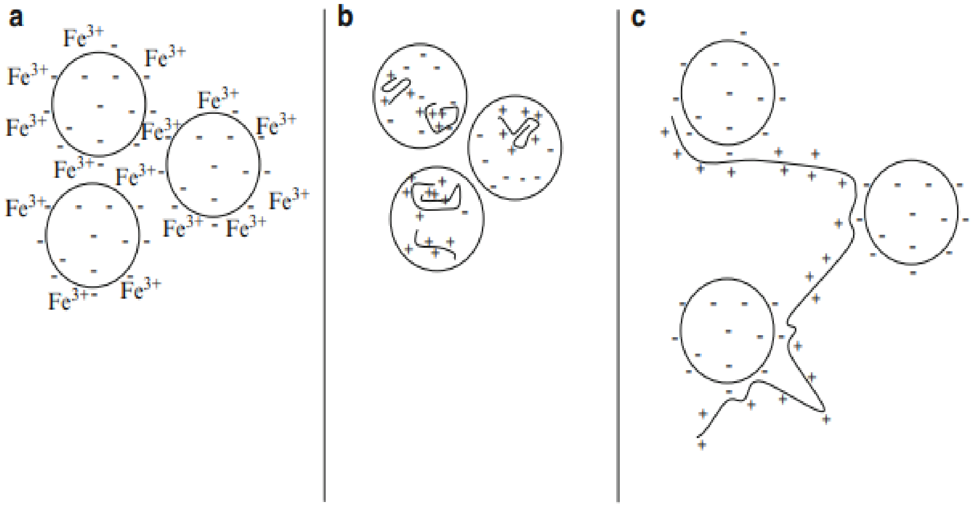

Figure 2-5 Diagrammatic representations of (a) coagulation, (b) patch flocculation and (c) polymer bridging (adapted from( Hjorth et al., 2009)

For good aggregate formation, the addition of coagulant and flocculent is held separately in which the former is added first and the latter followed (Hjorth et al., 2009). Moreover, short time vigorous and several minutes slow stirring are necessary to ensure ions homogenous distribution and to form coagulation and patch flocculation. As a result, chemically treated BGS can be transferred to physical separator so that liquid fraction is separated from the solid fraction.

Physical treatment is employed to separate BGS into solid and liquid fraction. Examples of separators include decanter centrifuge, screw press, belt press, dissolved air flotation, metal edge separator, vacuum filter press, chamber filter press, rotary sieve, sieve drum, vibration screens (Verbeke et al., 2021). The selection for appropriate technologies depends on initial investment cost, separation efficiency, and particle size that they able to retain (Dottorato, 2013).

A recent report on scenario’s and schemes of proven nutrient recovery and reuse (NRR) techniques estimated energy consumption and capital cost of selected separator (Verbeke et al., 2021). According to this report, screw press and belt press separator were found cheapest and most expensive, respectively. In terms of energy, centrifuge was found more energy intensive followed by dissolved air floatation. Based on mesh size, centrifuge separates smaller particles and gives greater separation efficiency of soluble nutrients in solid fraction (Fuchs and Drosg, 2013 and  Verbeke et al., 2021).

Table 2-3Summary of different solid-liquid separators (Meixner et al., 2015)(Verbeke et al., 2021)

| Separator | Mesh size | Capital Cost (Euro)** | Energy use |
|---|---|---|---|
| Screw press | 0.5–1 mm | 25,625±14,402 | 0.4–0.5 kWh/m$^3$ |
| Centrifuge | 0.45-10 µm | 86,333±22,965 | 3–5 kWh/m$^3$ |
| Belt press | - | 107,500±40,697 | 0.9±1.3kWh/m$^3$ |
| Screens | 100-300 µm | - | - |
| DAF* | - | 375,760 | 1.36 kWh/m$^3$ |

*Dissolved Air Flotation (DAF), ** the capital cost was estimated for all separator (except DAF) with 2 ton/hr. and 15 ton/hr. for DAF.

Vibrating screens are also used in BGS processing for its simplicity and wider particle size range. The biogas slurry is put on the top of the screens and the liquid fraction passes through the filter pores (usually between 100-300 µm). The solid fraction remained on the top or moves to the screen edge. Screens are operated under vibration to prevent clogging (Meixner et al., 2015).

Followed by solid and liquid separations, the distributions of principal constituents between typical solid and liquid fractions were analyzed and presented at Figure 2.6 (Drosg et al., 2013). Generally, BGS separation results a solid fraction with a higher organic matter and P content and a liquid fraction with a higher mineral N and K content (Bonten et al., 2014 and Verbeke et al., 2021). The $NH_4$-N is a readily available nutrient to plant growth and majority of it is found in the liquid fraction. However, loses of $NH_4$-N should be minimized during storage, transporting, further processing and application. This can be achieved by using cover storage, acid treatment and injection into the soil during application (Tao and Ukwuani, 2015).

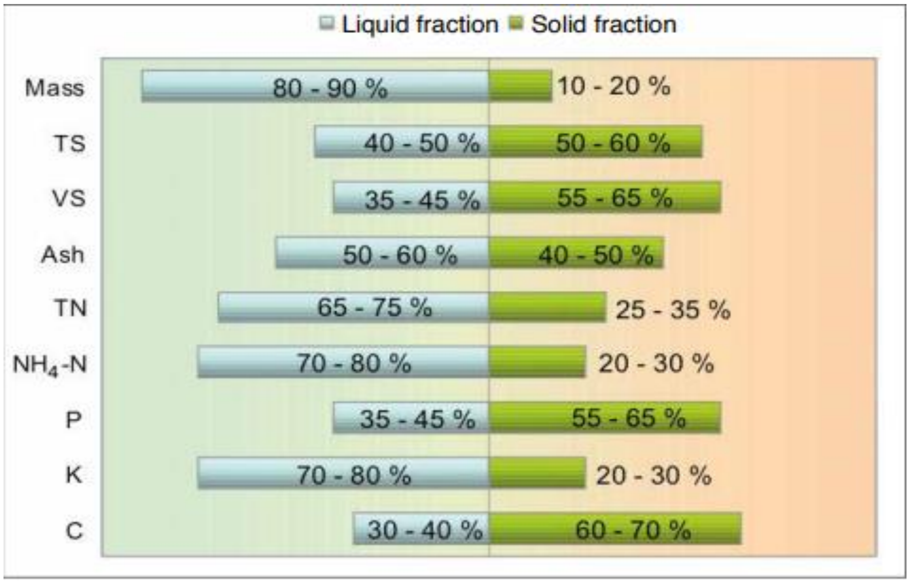

Figure 2-6 Distribution of the principal constituents after solid–liquid separation (Fuchs & Drosg, 2013)

### 2.6.2. Thermal Treatment

After solid-liquid separation the liquid fraction of BGS contain considerable amount of dissolved nutrients. The fate of this fraction may be either directly discharges to receiving streams or used for diluting substrate or further processed for nutrient and water recovery. Direct disposal of liquid fraction poses environmental problem such eutrophication in the water bodies. Partial utilization of liquid fraction to mix substrate can be considered. However, this option should be implemented with careful investigation of ammonia and salt concentration so that inhibition in the digester can be avoided (Drosg et al., 2015). Among these alternative, the most common practice is further concentrating of liquid fraction to recover and reuse nutrients and water (Pantelopoulos et al., 2016). Concentration of liquid fraction reduces its bulkiness by removing water under different treatment technologies.

Evaporation and condensation were presented as one of the efficient methods in reducing water volume of BGS for its operational reliability, ease of integration with existing biogas plant, water reuse, excess biogas and waste heat utilization (Vondra, Máša, Touš, et al., 2018; Vondra, Máša, and  Bobák, 2018). Evaporation vaporizes up to 80% of the water and utilizes it as process water. Moreover, evaporation is usually accompanied with other technologies such as chemical and physical treatment and it is possible to create concentrate BGS which have up to 10- 12% DM (max.30%) (Verbeke et al., 2021). The incorporation of additive and physical filtration techniques with evaporation are useful to retain nutrient, to improve evaporation efficiency by reducing fouling at heat exchange surface, prevent fibers possibly clogging evaporators, reduce viscosity, low pH that ensure low ammonia concentration and decrease ammonia volatilization (Vondra and Bob, 2018).

Factors' affecting nutrient recovery and water removal (WR) efficiency includes heating temperature, time, pH and pressures. Wang et, al. (2022) investigated the effect of heating temperature and time on concentration of biogas slurry. The result showed that WR efficiency increased from 12% to 53.49% when the temperature increased from 50 to 90 $^0$C (Wang et al., 2022). Increasing temperature increases the vapor pressure and this accelerated evaporation process. It is also observed that the increased temperature led to an increase in the percentage of free ammonia ($NH_3$), which was easily lost through volatilization (Wang et al., 2022). To this effect, the higher the heating temperature, the lower the $NH_4$-N recovery rate during evaporation ( Tao and Ukwuani, 2015; Awiszus et al., 2018). On the other hand, pretreatment of influent liquid fraction with acid reduces ammonium volatilization in the form of ammonia (Pantelopoulos et al., 2016).

When evaporation is operated under vacuum pressure, vaporization and better concentration efficiency can be achieved at a lower temperature (Verbeke et al., 2021). However, after reaching at boiling temperature, the water removal efficiency may be diminishing with increasing of temperatures. Likewise, with an increase in heating time at various temperatures, the water removal (WR) efficiency increased. Wang et al. (2022) observed rapid increase of WR efficiency at heating time from 40-90 min ( Wang et al., 2022). On the contrary, $NH_4$-N recovery and electric conductivity (EC) decreased with the increase of heating time. The amount of soluble salts in solution was indicated by the value of EC and its recovery rate decreased almost linearly with the increase of heating temperature (Wang et al., 2022). Otherwise, the potassium and phosphorus in the BGS were in a constant state and seldom evaporated.

To prevent volatilization of ammonia during evaporation, the pH of the influent of the evaporator can be adjusted by adding acid (Pantelopoulos et al., 2016). This will lower the pH, pushing the equilibrium of $NH_4^+/NH_3$ to ammonium in solution. The amount of acid required to get to this pH, is again depend on the buffer capacity of the BGS solution (Verbeke et al., 2021). This approach will cause only the water (and some volatile components) to evaporate and create a more concentrated BGS which still includes the ammonia (Vondra, Máša, and Bobák, 2018; Vondra, Máša, Touš, et al., 2018). When the pH is more than 6, the $NH_4$-N recovery rate decreased. With this regard, decrements of $NH_4$-N recovery from 96.8% to 68.3% were observed when pH increased from 6 to 7. When pH increased to 8, only 52.48 % $NH_4$-N recovery rate was obtained (Wang et al., 2022). Similarly, the EC has inverse relation with the pH. In a similar study the recovery rate of EC was found 56.16% when the pH was 8 (Wang et al., 2022). Phosphorous recovery rate was found independent of pH (Wang et al., 2020 and Xiao et al., 2022).

The pH of condensate is expected to increase with pH due to the escape of ammonia with water at alkaline condition. The condensate (ammonia) water does not contain concentrated nutrients, but is not clean enough for discharge. According to B 18596- 2001 "Discharge standard of pollutants for livestock and poultry breeding", a daily discharge standard of $NH_4$-N is reported 80 mg/ L (Wang et al., 2022).

The use of evaporator for BGS processing is highly dependent on the energy source. As evaporation is energy intensive, it is only interesting for biogas plants where excess heat is available in sufficient amounts (Drosg et al., 2015). Different sources of heat such as solar radiation, greenhouse and heat from combined heat-and-power (CHP) units can be employed as energy source (Wang et al., 2022; Herbes et al., 2020). Residual heat from CHP from existing biogas plant is commonly utilized for concentrating biogas slurry. Based on experience, a thermal energy demand of about 300–350 kWh per ton of water evaporated was reported (Drosg et al., 2015). In other study, the energy consumed estimated to as high 670 kWh per metric ton of water evaporated ( Hjorth et al., 2009). However, varied data was reported for different evaporator conditions and configuration.

## 2.7.Biogas Slurry (BGS) Value Addition and Energy

Bulky biogas slurry (BGS) management demands energy for transporting from biogas plant to farm site and land application. This energy can be explained in terms of specific cost/$m^3$ of biogas slurry and the specific cost is directly correlated to transport distance(km)(Drosg et al., 2015). Especially long distance unprocessed BGS transportation and application increase the burden considerably. Hence, volume reduction of BGS by evaporation could bring potential reduction in energy consumption. With this, energy consumption reduction for spreading and transporter vehicle and associated $CO_2$ emissions mitigation can be attained. BGS concentration also decreases the use of chemical fertilizers and by this it reduces natural gas consumption, non-renewable raw materials for making chemical fertilizers (de Groot and Bogdanski, 2013).

Moreover, BGS processing (with evaporator) gives an opportunity to recycle water for different application and reduce freshwater use (Vaneeckhaute et al., 2017). As far as the concentration of ammonia is not high, the condensate can be reused for mashing/diluting feedstock to optimal dry matter and to mix flocculants. Residual heat from CHP can also be transferred to process water and used again (Guan et al., 2014) (Wang et al., 2022).

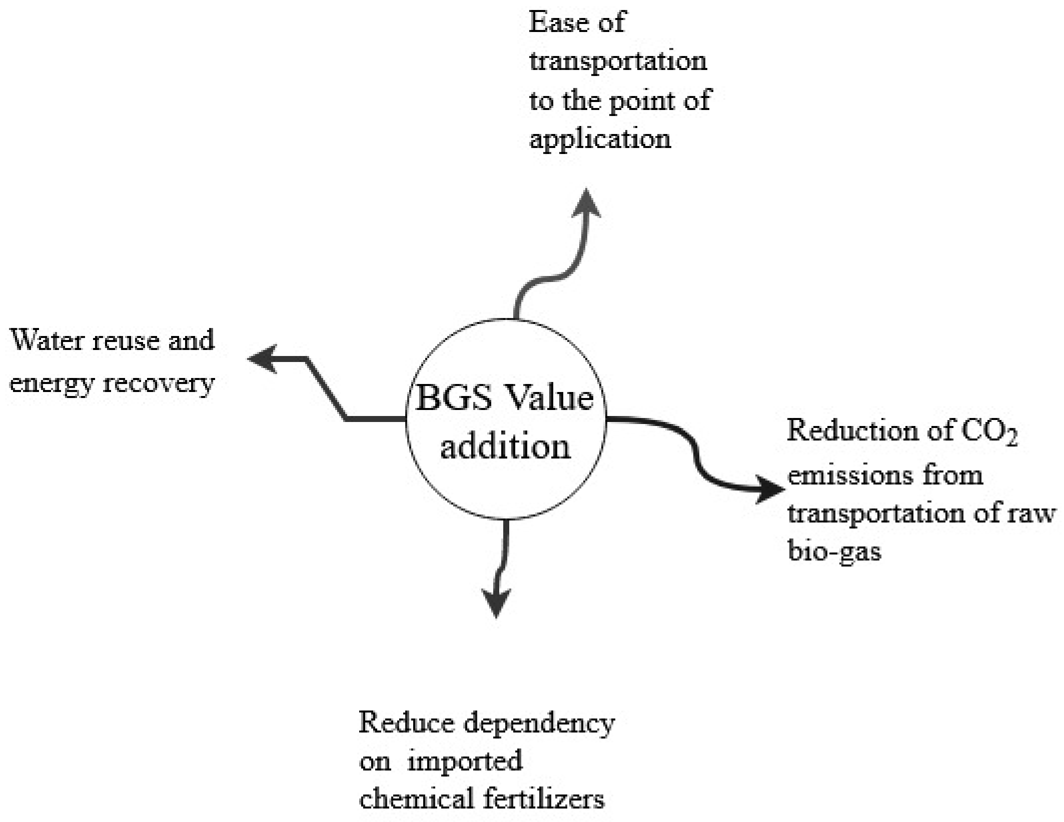

Figure 2-7 Significance of BGS concentration (Guan et al., 2014)

BGS Value addition is a gate way to better promote biogas technology dissemination. By this it indirectly helps to reduce the use of fire wood, charcoal and kerosene. The concentrated slurry also contributes for the reduction of imported chemical fertilizer, transporting fuel and $CO_2$ emissions. Moreover, value addition of BGS using evaporation and condensation process create an opportunity of energy recovery and water reuse.

## 2.8. Biogas Digester and Dairy Farm

Building biogas digester integrated with the existing dairy farm helps to reduce greenhouse gas emissions during both manure storage and open digstate pile, enhance sustainable rural development, increase farm's income and energy self-sufficiency (Torquati et al., 2014; Guan et al., 2014; Wahyudi et al., 2015). Moreover, the integration of biogas plant with the existing dairy farm able to provide an opportunity to convert organic matter rich milk byproducts into biogas energy under aerobic digestion process (Traversi et al., 2013).

In Ethiopia very few dairy farm integrated biogas plant with the existing systems. Zagol dairy farm and milk processing plant is among few farms which generate biogas and biogas slurry from the anaerobic digestion of cow manure. At Zagol dairy farm and milk processing plant, a medium size biogas digester (MSBD) with a capacity of 80 $m^3$was installed. The digester was used cow manure (from 100 milked cows) as a feedstock for mesophilic anaerobic fermentation and daily about 36 $m^3$ of biogas was generated. In the farm the biogas is converted into 16 kw of electric power by using generator set and was utilized for different energy application demanded by dairy farm which includes refrigeration and hot water especially during power outage.

The daily biogas slurry (BGS) generation at Zagol diary is estimated to be 1320 kg and portion of the slurry was used as organic fertilizer to grow fodder grass inside the compound. This is carried out by moving the slurry from the slurry pit to the field using diesel drive pump. Limited efforts have been carried out to use slurry to the surrounding farmers as organic fertilizer in nursery and garden. However, its wider application is hindered due to the high moisture content of BGS (expensive to transport to the point of application) and lack of public awareness about the BGS use as organic fertilizer. To this end, it is not uncommon to observe that larger volumes of BGS were piled near the biogas digester and occupy larger space and poses environmental problem. Hence, further study is necessary to effectively transform this "bulky waste" into a more concentrated resource.

# 3. MATERIAL AND METHODS

A hybrid Physico-chemical and thermal treatment techniques was employed to concentrate biogas slurry. Chemical and physical treatments were used to minimize water soluble and volatile nutrients loss whereas the thermal treatments applied to reduce its water content. For this purpose, 10 Liters of raw biogas slurry (RBGS) was collected from biogas digester at Zagol (Melkam) dairy farm and milk processing industry, Sululeta, Oromia, Ethiopia.

Table 3-1Biogas digester at Zagol dairy farm and milk processing (Tesfaye et al., 2011)

| Parameters | Descriptions |
|---|---|
| Digester type | Fixed dome (concrete bricks) |
| Location | Sululeta, Oromia region, Ethiopia |
| Owner | Melkam dairy farm and milk processing plc. |
| Estimated cost of bio-digester | 190, 500 birr |
| Capacity | 80 $m^3$ |
| Daily biogas production | 36 $m^3$ (41.4 kg methane) |
| Biogas storage bag size | 30 $m^3$ |
| Daily feed to digester | 1200 kg of fresh dung with water and cattle urine |
| Daily BGS production | 1320 kg/ day |
| Biogas uses | The biogas is used in milk chiller and the incubation room, cooking and lighting |
| Biogas slurry uses | The biogas slurry is used as organic fertilizer to be used for fodder production, nursery and garden |

The raw biogas slurry took and kept in a refrigerator at $4^oC$ before further experiments, this was done to in inactivate the microorganism and prevent further degradation of carbon. Then the sample was treated with additives to maximize water removal efficiency and nutrient recovery rate in the subsequent treatment processes. The chemical composition of the raw biogas slurry (RBGS) collected at Zagol diary reported in Table 3.2.

Table 3-2Composition of raw biogas slurry (RBGS) (Mekuria et al., 2021)

| Parameter | Amount | Parameters | Amount |
|---|---|---|---|
| pH | 7.04 | Mn(mg/l) | 28.85 |
| Ca(mg/l) | 561.5 | Cu(mg/l) | 0.67 |
| Mg(mg/l) | 160.46 | Zn(mg/l) | 1.03 |
| K(mg/l) | 723.8 | B(mg/l) | 1.69 |
| Na(mg/l) | 94.28 | Fe(mg/l) | 163.08 |
| P(mg/l) | 253.38 | TN (%) | 26.23 |
| S(mg/l) | 128.23 | OC (%) | 52.03 |

### 3.1. Physico-Chemical Treatment

Physico-chemical treatment of raw biogas slurry (RBGS) was conducted prior to thermal treatment. The RBGS was chemically treated with the addition of coagulant and flocculant for retaining water soluble nutrients and maximize solid-liquid separation and thermal operation efficiency (Hjorth et al., 2009). The coagulant and flocculants used for this experiment were Ferric chloride ($FeCl_3$) and cationic medium charge density poly electrolyte polymer, PEP, (20-40 mol %), respectively. A dosage rate of 50 mg/L, 100, mg/L, and 150 mg/L each chemicals were added into the RBGS and mixed with a stirrer (EURO-ST40, 30-2000 rpm, Germany) at AAiT, in the school of Chemical and Bioengineering laboratory (Verbeke et al., 2021). It is indicated that cationic polymer has superior performance to anionic as BGS are mainly negatively charged (Hjorth et al., 2009). The optimal amount of coagulant and flocculants dosage reported in the range 50-350 mg/L of BGS (Verbeke et al., 2021).

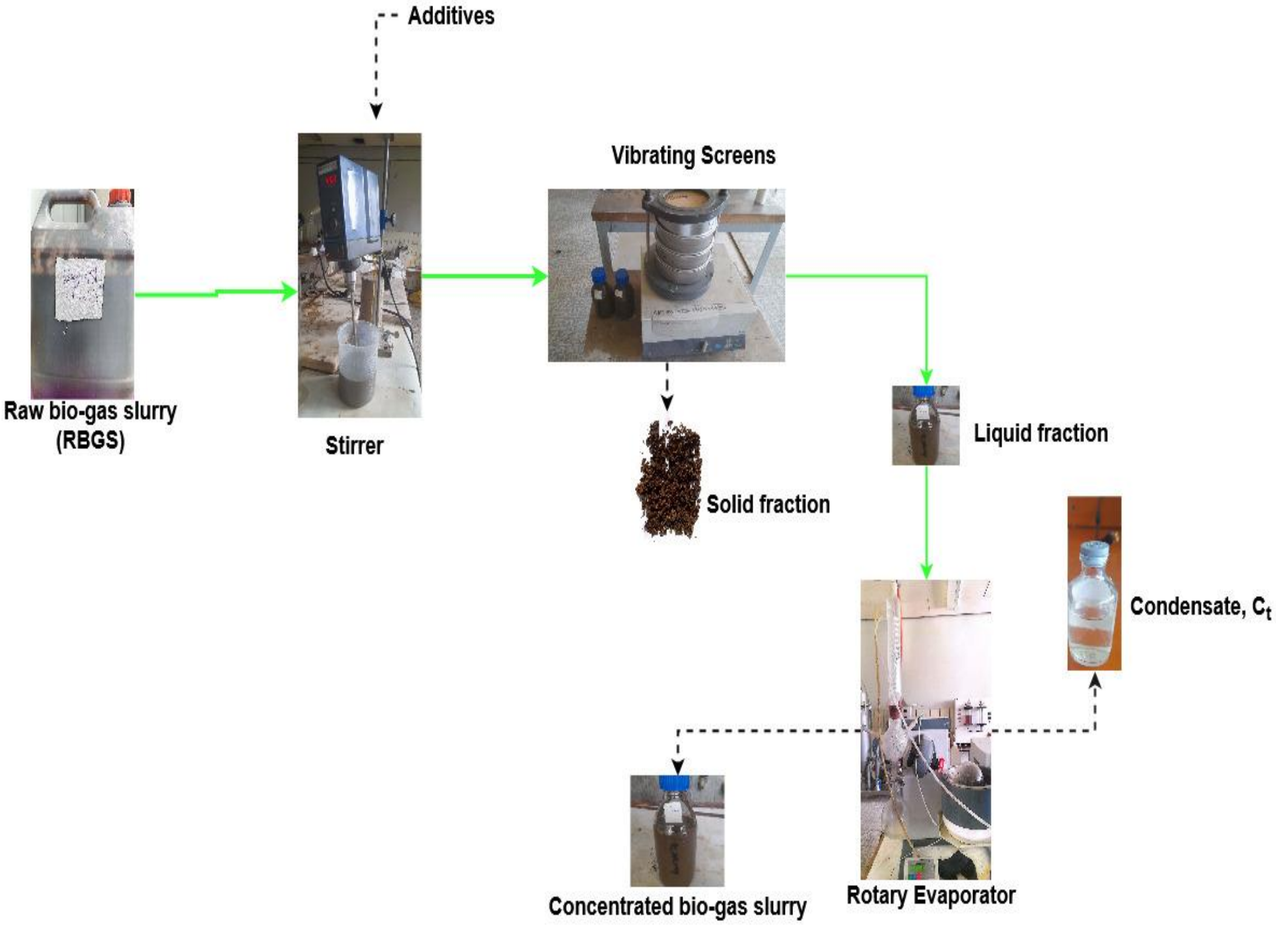

Figure 3-1Experimental Procedure for Concentrating Biogas slurry (BGS)

A rigorous stirring velocity of 450 rpm for 5 minutes for coagulation and slow stirring of 30 rpm for duration of 15 minutes were employed for flocculation. For proper aggregation of suspensions $FeCl_3$ was added first and PEP added later as suggested by (Hjorth et al.,(2009).

The treated biogas slurry was then sent to physical separator where it was separated into solid and liquid fractions. The mechanical filtrations tests were done by tradition cotton cloth (as preliminary experiment) and vibrating screen (Retsch-GmbH, 4278, 230V, 50HZ, Amplitude 0-100, 2A, Germany) systems at AAiT, School of chemical and bioengineering laboratory. To investigate principal nutrient distribution at solid and liquid fractions, various screen meshes sizes (0.25 mm, 0.5mm and 0.71 mm) were employed. Vibration of screen was induced at amplitude of 60 for duration of 20 minutes to avoid clogging at mesh.

## 3.2.Thermal Treatment

Thermal treatments of liquid fraction were performed by evaporation and condensation process. These techniques were presented as one of the efficient methods in reducing volume of biogas slurry for its operational reliability, ease of integration with existing biogas plant, water reuse, excess biogas and waste heat utilization (Vondra et al., 2018). For this experiment, rotary evaporator (RVO 400, no revolution=15454, 230 V, 10A, 50 HZ, IKA, Czech Republic)which is coupled with vacuum pump (No-0099, 2015, 330V, 50HZ,Czech Republic) and condenser were used at Chemical Engineering Laboratory, School of chemical and bio Engineering, AAiT. The liquid fraction from the filtration system then subjected to thermal treatment to partially remove water with minimum nutrient loss at different heating time, temperature and pH. The vacuum pump (with relative pressure of 60-70 mbar) was used to create lower pressure conditions so as to decrease the temperature requires to vapor the water (Bonmatí et al., 2010).

The water vapor and other volatile compounds were condensed by a condenser. To prevent volatilization of ammonium in the form of $NH_3$ during evaporation, the liquid fraction was treated with $H_2SO_4$ (98%).

## 3.3.Experimental Design

The effect of different variables for water removal (WR) and nutrient recovery (NR) efficiency were investigated at each treatment stages (Figure 3-2). These includes:-

- Effects of additives dosage
- Effects of screen mesh size
- Effects of heating temperature
- Effects of heating time
- Effects of pH

Principal nutrients (P, K, $NH_4$-N) distribution in solid and liquid fraction was examined at various chemical dose rates of 50 mg/L, 100 mg/L and 150 mg/L. The treated BGS is designated as $LF_{0.}$ The $LF_o$ was then separated into solid and liquid fraction by vibrating screens with at different mesh size of 0.25 mm, 0.5 mm and 0.71 mm. The resulted solid and liquid fractions of BGS are designated as $SF_o$ and $LF_1$ respectively.

At the rotary evaporator, the liquid fraction ($LF_1$) was further concentrated at different operating conditions. These operating parameters include heating temperatures, $T^0$, (65, 75, 85 and 95 $^oC$), heating time, t, (30, 45, and 60min.) and pH (6, 7 and 7.76) on water removal (WR) efficiency was investigated. The pH value of RBGS was measured and found to be 7.76. The effect of pH (6, 7 and 7.76) on essential nutrient recovery was examined. A pH of 7 and 6 was maintained by adding 8 drops (0.4 ml) and 16 drops (0.8 ml) of $H_2SO_4$ (98%) per 100 ml liquid fraction. Pretreatment of liquid fraction was carried to minimize ammonium loss in the vapor in the form of $NH_3$. Concentrated biogas slurry and condensate was generated as result of evaporation of the liquid fraction. The concentrated biogas slurry is designated as CBGS and condensate as $C_t$. Finally the CBGS (rich in nutrients, salts and organic matter) was mixed with solid fraction ($SF_o$) to form NPK rich end product. Overall experimental design is presented in Figure 3.2.

### 3.4.Chemical Analysis

The nutrient recovery (NR) efficiency indicators in the sample include $NH_4$-N, P, K, Ca, S, Mg, Zn, and B, pH, electric conductivity (EC). The macro and micro nutrients content were tested by Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES)(Plasma Power: 1400 W, Pump Speed: 30rpm, Coolant Flow: 13 L/min, Auxiliary Flow: 0.8 L/min, Nebulizer Flow: 0.73 L/min) at Horticop, Bishoftu, Ethiopia. The pH and EC were measured using a pH meter (3505 pH, TENWAY meter, input=100-240 V, output= 24v, 1.33A) and an electrical conductivity meter. The concentration of ammonium in the condensate was analyzed by using UV spectrometer at chemistry department, AAU.

The water removal (WR) efficiency of biogas slurry during evaporation was determined by equation 1 as indicated by Wang et al., (2020).

$$\mathrm{WR} = \frac{\mathrm{V_o} - \mathrm{V_i}}{\mathrm{V_0}} * 100 \qquad (1)$$

Where, WR is the water removal efficiency (%), $V_i$ is the volume of biogas slurry at time $t_i$ (mL), and $V_0$ is the initial volume of biogas slurry (mL).

$$\text{Nutrient recovery rate} = 1 - \frac{C_{LF} - C_{CBGS}}{C_{LF}} * 100 \qquad (2)$$

Where $C_{LF}$ is the nutrient concentration in the liquid fraction and $C_{CBGS}$ is the nutrient concentration in the concentrate at different conditions.

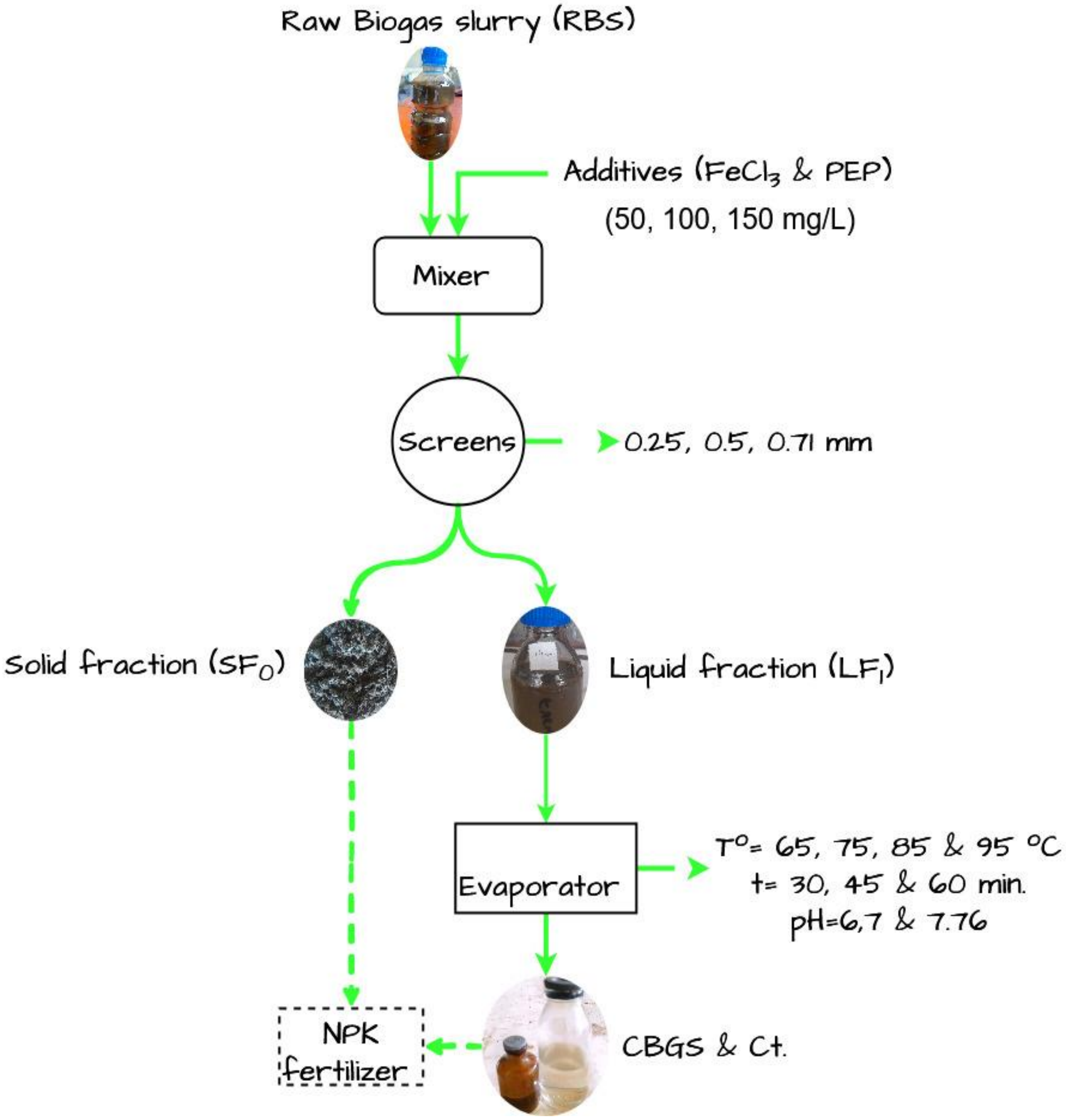

Figure 3.2 Experimental designs for Biogas slurry (BGS) concentration

# 4. RESUST AND DISCUSSION

This section provides result and discussion of the main factors studied, including the effects of chemical, physical, and thermal treatments. Specifically, it focuses on the impact of thermal treatment on water removal efficiency and nutrient recovery, considering variables such as heating time, temperature, and pH. The results analyzed and interpreted to address the research objectives and provide insights into the effectiveness of thermal treatment in concentrating biogas slurry while maximizing water removal and nutrient recovery.

## 4.1.The Effects of Chemical Coagulant and Flocculants

For this particular study $FeCl_3$ and medium charge density polyelectrolyte polymer (20-40 %) as coagulant and flocculants respectively, were used. The effect of additive dosages (50, 100, 150 mg/L) on the principal nutrient distribution between solid and liquid fractions (at 100 mg/L) was examined and presented (Figure 4.1).

The result showed that the liquid fraction was consisted of 29.8% of ammonium ($NH_4$-N) and 23.2% of potassium (K). Whereas the amount of $NH_4$-N and K share in solid fraction were found 2.9% and 17.3%. Significant portion of readily available soluble $NH_4$-N and K were presented in liquid fraction with distribution share of 91% and 57.3% respectively. The distribution of phosphorous (P), calcium (Ca), magnesium (Mg) and sulfur in liquid fraction were determined 10.1%, 10.8%, 7.7% and 3.8% respectively. A significant amount of P share (68.2%) was resulted in solid fractions and this value is comparable with the earlier works which presented the content of P as high as 65% (Drosg et al., 2015). Besides the distribution range of P in solid fraction of BGS was reported between 55-65 % (Drosg et al., 2015). Higher level of Ca’ Mg’ Na and S were found in the solid fraction. This is justified by the fact that the addition of cationic coagulant and flocculent facilitates aggregation of these ions to be retained in the solid fraction. Other trace elements (Fe, Mn, Cu, Zn and B) almost showed similar distribution between liquid and solid fraction.

Table 4-1Composition of solid and liquid fraction after solid-liquid separation

| Parameter | Unit | $LF_1$ | $SF_1$ | Parameter | unit | $LF_1$ | $SF_1$ |
|---|---|---|---|---|---|---|---|
| $P^H$ | | 7.76 | - | P | mg/l | 543 | 1,167 |
| OC | % | 36.66 | 48.75 | S | mg/l | 200 | 372 |
| $NH_4$-N | mg/l | 1594 | 153 | Cu | mg/l | 1.60 | < 0.04 |
| Ca | mg/l | 572 | 1,085 | Zn | mg/l | 10.83 | 14.55 |
| Mg | mg/l | 410 | 709 | B | mg/l | 0.64 | < 0.1 |
| K | mg/l | 1,243 | 925 | Fe | mg/l | 214 | 209 |
| Na | mg/l | 536 | 664 | Mn | mg/l | 25.31 | 37.43 |

In this work the organic carbon (OC) in the liquid fraction and solid fraction determined as 36.66% and 48.75%, respectively. Approximately similar organic carbon content of the liquid fraction (35.2%) and solid fraction (50.1%) of biogas slurry obtained from anaerobic digestion of cow manure was reported (Slepetiene et al., 2023). The organic carbon content of the liquid and solid fraction of biogas slurry obtained from cow manure can vary depending on various factors, including the composition of the manure, the anaerobic digestion process conditions, and the efficiency of the digestion process. The accumulation of organic carbon enhances soil structure and it is presented as carbon sink approach to mitigate greenhouse gas emissions ( Li et al., 2017).

Generally, higher levels of organic matter, phosphorus, magnesium and calcium are characteristics of the solid fraction. Whereas, high concentrations of nitrogen, potassium, zinc, and copper are present in the liquid fraction (Chuda and Ziemin, 2021).Solid-liquid separations with chemical pretreatment of biogas slurry enhance its filterability between solid and liquid fractions. Better separation efficiency of components and organic matter between solid and liquid separations were obtained due to the addition of flocculants and coagulant (Chuda and Ziemin, 2021). However, the use of the resulted solid fraction for possible agricultural application requires careful attention and should be consistence with national and international heavy metals concentration level (Bona et al., 2020).

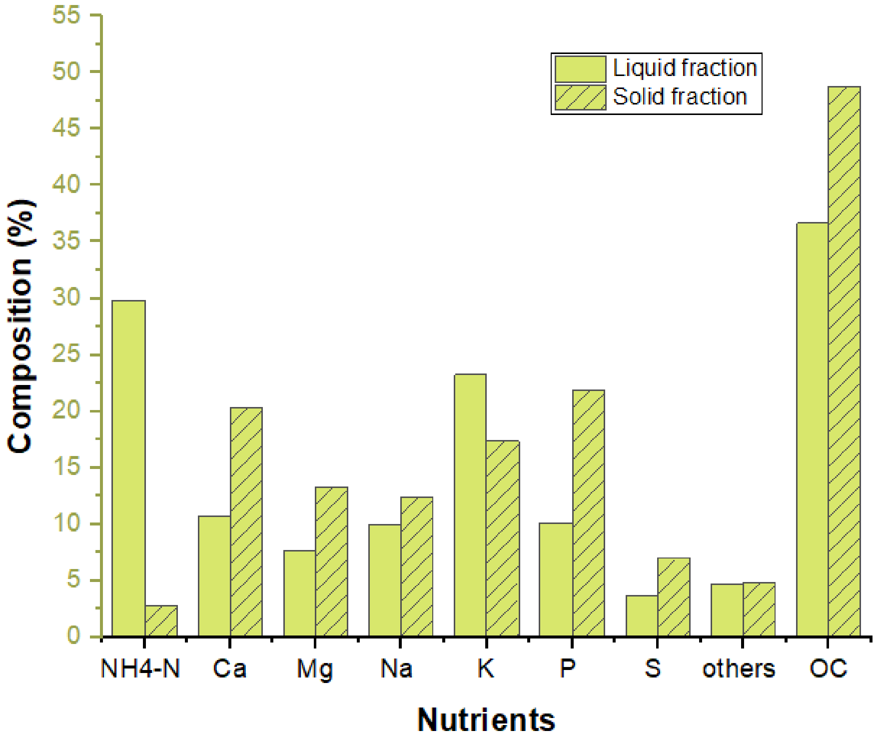

Figure 4-1 Distribution of nutrients between solid and liquid fraction

### 4.2.The Effects of Physical Treatment

Handling, transportation and application of raw biogas slurry pose management challenge for biogas plant owner due to its bulkiness. Separation of biogas slurry into liquid and solid fraction is employed as the first step in biogas slurry processing. Because there is less water in the separated solid fraction, it can be easily stored in simpler conditions, transported and used immediately for agricultural uses at a significant cost savings (Drosg et al., 2015). Usually the liquid fractions are subjected to various treatment and nutrient recovery options (Chuda and Ziemin, 2021). The effectiveness of solid and liquid separation with sieve is affected by parameters such mesh sizes (Jing et al., 2016).

Vibrating screens with different mesh sizes (0.25, 0.5, 0.71 mm) were employed to carry out the solid-liquid separations. The screen was chosen because it allows for the filtration of a wider spectrum of BGS particle sizes (0.1-0.3 mm) (Meixner et al., 2015). To avoid clogging, vibration was induced by applied rapid motion at amplitude of 60 for duration of

20 minutes. The outcome demonstrated that the most solid material was retained when the treated BGS was filtered through a mesh size of 0.5 mm. When using small aperture screen mesh (0.25 mm), effective separations of liquid and solid was not achieved and this obstruction is caused by due to the clogging of smaller screen size (Jing et al., 2016). The least solid fraction was retained on the surface of the screen with a mesh size of 0.71 mm as most of the BGS particle sizes might be less than 0.71 mm. It has been reported that about 84% of anaerobically digested slurry has particle size of less than 0.01mm (M. Hjorth et al., 2009).It is anticipated that after the biogas slurry has been chemically treated with coagulant and flocculent, the particle size grow in the form of floc and it will be effectively separated by screen size greater than its particle size (Bona et al., 2020). Thus, the physical separation of biogas slurry with screen is required the control of clogging (Zhan et al., 2018). Effects of screen sizes for separations efficiency and nutrient recovery distribution between solid and liquid fractions was not investigated as it is out of the scope of the study.

### 4.3.Effect of Heating Time on WR Efficiency

The predominant components of typical bulky biogas slurry (BGS) are water (93%) and other organic and inorganic matters (7%) (S. Kumar et al., 2015). The bulkiness of BGS makes it difficult and expensive to store, transportation to the field. Few researches have been conducted on ways to decrease the amount of water without losing its essential plant nutrient during BGS processing (Vondra, Máša, Touš, et al., 2018; Menkveld and Broeders, 2018). Evaporation and condensation are one of the most efficient and commonly used method to partially reduce BGS water volume due to its operational reliability, simplicity of integration with the existing biogas plant, water reuse, and utilization of extra biogas and waste heat (Vondra, Máša, and Bobák, 2018). The major determinant of BGS water volume reduction operations are presented as heating time, temperature and pH (Pantelopoulos et al., 2016).

The influences of heating time with different temperature on WR efficiency during evaporation of liquid fraction were examined and presented in Figure 4.2. WR efficiencies of 27%, 40% and 35% were obtained for 65, 75, and 85 $^{o}$C at same heating time of 45 min under vacuum pressure. The highest WR efficiency of 70% was obtained at 60 minutes and 75 °C. WR efficiency increased with the increase of heating time. At lower heating time, the WR efficiency showed constant trend for about 10-15 minutes and then increased with heating time. This is justified by the fact that at a given temperature vapor formation requires sufficient time to heat and to start vapor. At 45 minutes of heating time and 65 °C of heating temperature, the slurry's water removal efficiency showed its lowest point. This might have happened as a result of increased pressure.

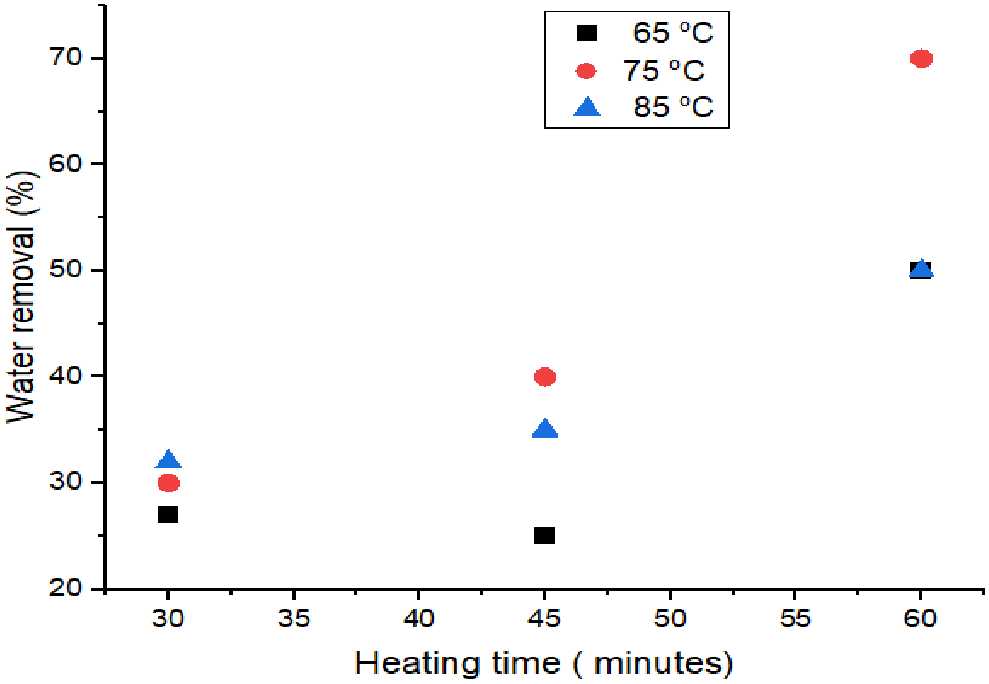

Figure 4-2Effects of heating time on bio-slurry concentration

On the other hand, a slightly higher WR efficiency was achieved with higher temperature at same heating time as vapor formation and boiling process is favored at higher temperature. This is because of higher drying temperatures increase the system's energy input and speed up moisture diffusion (Pantelopoulos et al., 2016)(Pantelopoulos et al., 2016)(Pantelopoulos et al., 2016). WR effectiveness and heating temperature revealed a nonlinear relationship as drying time increased. Higher WR efficiency was attained for heating temperatures of 75 $^0$C starting at heating times of 37.5 min. and onward (Horttanainen, 2018). This is because the vacuum operating pressure causes the slurry boiling temperature to be lower than the typical boiling point (Ukwuani and Tao, 2016). The biogas slurry's boiling point was reduced to 65 $^0$C in the current investigation, which ran at vacuum pressures of 60 to 70 mbar. This outcome was consistent with the pattern seen in the biogas slurry boiling point vacuum-temperature curve (Tao and Ukwuani, 2015). Therefore, because vacuum pressure operation requires less heat, it uses less energy, which is adverse to the viability of the evaporation process for biogas concentration (Yan et al., 2010).

### 4.4.Effects of Temperature on WR Efficiency

The effect of heating temperatures on WR efficiency of BGS were examined at 65, 75 and 85 $^0$C under constant heating time of 45 minutes and constant vacuum pressure of 65 mbar. The effect of temperature on BGS water volume reduction was also investigated at 95 $^0$C under atmospheric condition. Figure 4.3 shows the findings that how heating temperature and WR efficiency were related. WR efficiencies of 28%, 40% and 35% were obtained at 65, 75 and 85$^o$C respectively. The WR efficiency showed non-linear relation with temperatures. As depicted in Figure 4.3, WR increased initially as a function of temperature and reach maximum with temperature of 75$^o$C then progressively declined. Moisture content removal enhanced at higher temperature and lower pressure. However, WR efficiency decrease after an optimum boiling temperature reached due to the diminishing bulk water and water vapor generation. The dynamics of water removal efficiency over the heating temperatures coincide with the result reported in literature (Ukwuani and Tao, 2016). Additionally, as the temperature rises, the escape of ammonium in the form of ammonia increase as its volatilization highly favored with temperature and pH rise (Cerrillo et al., 2023). It is also indicated that the vacuum's suction effect enhances the mass transfer of ammonia during concentration of biogas slurry. Enhancement of ammonia mass transfer is enabled by liquid turbulence and vapor current (Ukwuani and Tao, 2016).

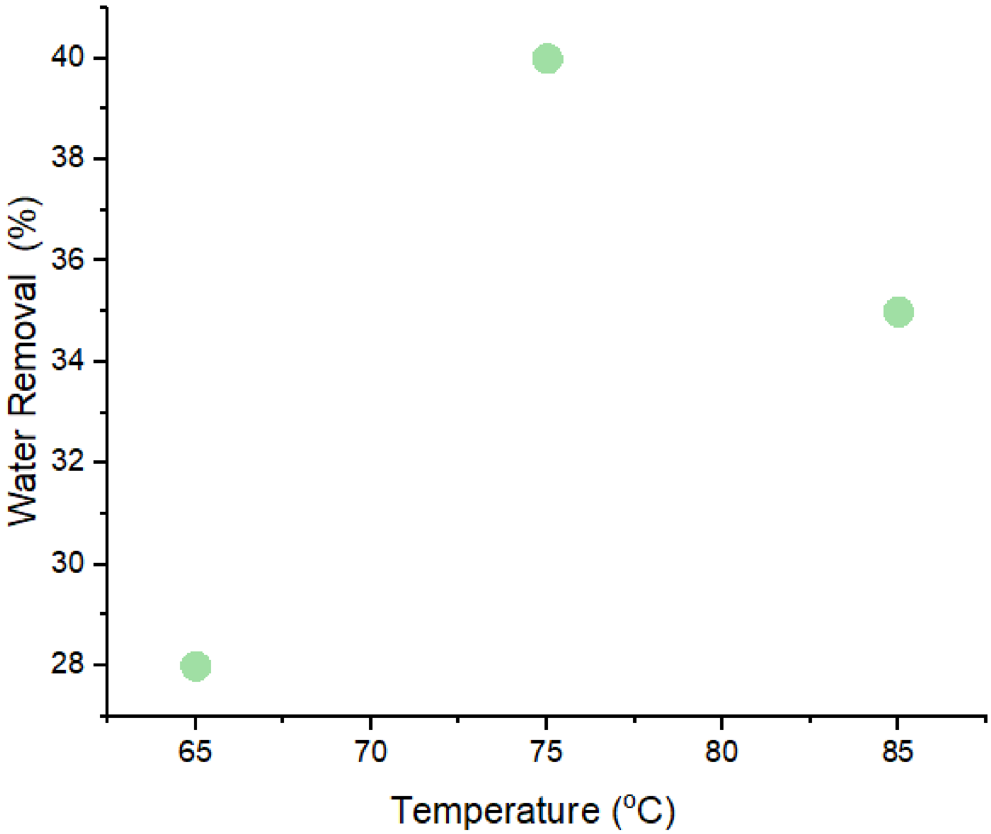

Figure 4-3Effect of heating Temperatures on bio-slurry concentration

Furthermore, the pressure conditions have a significant impact on the biogas slurry concentration: Operating under negative pressure have been shown to lower heating temperature (Ukwuani and Tao, 2016). A maximum WR efficiency of 40% was achieved at 75 °C under vacuum conditions (60-70 mbar) whereas the lowest WR efficiency (18%) was found at 95 °C at atmospheric conditions. It was observed that creating lower pressure highly determine the effectiveness of evaporation operations (Ukwuani and Tao, 2016; Cerrillo et al., 2023). Since evaporator systems consume a lot of energy, it's important to concentrate BGS while using energy efficiently. By lowering the pressure conditions at the evaporator reduces the energy or boiling temperature required to remove the moisture content of biogas slurry. According to a study, energy demand can be lowered by 56% while running at 65 °C as opposed to 102 °C (Ukwuani and Tao, 2016). Operating at relatively lower temperature also enables utilization of waste heat from combined heat and power (CHP) which is usually not higher than 80 °C and excess biogas energy from the existing biogas plant (Pintari et al., 2020).

### 4.5.Effects of pH on WR Efficiency

The effect of acid pretreatment on WR efficiency was examined and presented in Figure 4.4. The investigation was conducted at heating temperature and time of 75 °C and 45 minutes respectively. The liquid fraction without treatment (pH=7.76), neutral (pH=7) and acidic (pH=6) conditions were used for the analysis. As indicated in the Figure 4.4, a maximum WR efficiency of 55% was obtained at pH value of 7. The WR efficiencies at pH 6 and 7.76 were found 40% and 35% respectively.

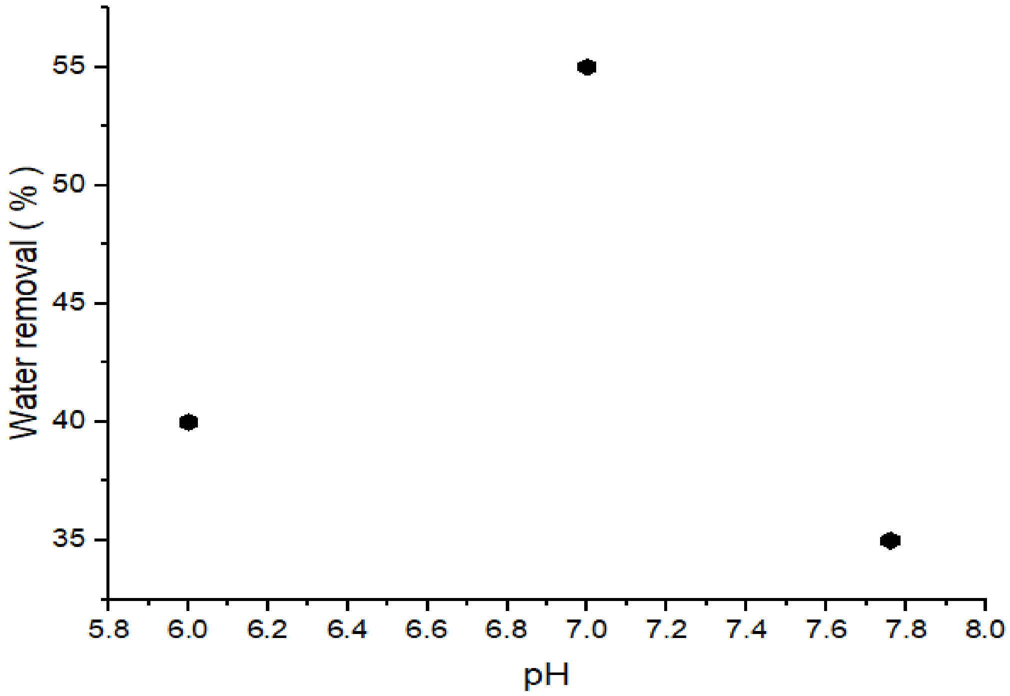

Figure 4-4Effect of pH on bio-slurry concentration

Vaporization of water more favored in pure state than in a mixture. A study comparing the boiling points of various types of biogas slurry to pure water at various vacuum pressures and heating temperatures was carried out and the results were presented as vacuum-temperature curves (Ukwuani and Tao, 2016). The comparison was conducted for varies slurry with different pH condition including diary manure digestate, food waste digestate, sludge digestate, pH 9 water and pure water. The outcome shows that digestates has slightly higher boiling temperatures and vacuum pressures than pure water. This result showed that lower moisture content reduction is achieved both at lower and higher pH due to the interference of ions and other volatile components occurred during evaporation of BGS (Zhang et al., 2020). Highest WR efficiency (55%) was obtained at pH of 7. Generally the level of water removal efficiency is substantially influenced by total solid concentrations.

### 4.6.Effect of Acid Treatment on Nutrient Recovery (NR) Efficiency

Acid treatment of liquid fraction is one of the efficient methods to reduce essential nutrient volatilization during evaporation process (Pantelopoulos et al., 2016). Powerful acidifying chemicals such as $H_2SO_4$ have been shown to be effective as $NH_3$ emission inhibitors for keeping the pH level low. The nutrient recovery (NR) efficiency was affected by the type of BGS, pH value, temperature, time and operating pressure (Wang et al., 2020, 2022). The pH conditions during evaporation of liquid fraction have reported as a major factor which determines nutrient recovery rate (Battista et al., 2021).

The effect of pH (7.76, 7 and 6) on NR efficiency was investigated and presented (Figure 4.5). The $NH_4$-N concentration was found 647.44 mg/L, 922.21 mg/L and 1,516.5 mg/L at pH 7.76,7 and 6 respectively. The concentration of $NH_4$-N was increased when pH decreased from 7.76 to 6. The highest $NH_4$-N recovery efficiency (95.1%) was obtained at pH=6. $NH_4$-N recovery rate was lowest with 40.6% at pH of 7.76.The $NH_4$–N recoveries improvement from 40.6% to 95.1% was when the raw biogas slurry (pH of 7.76) acidified to 6. The $NH_4$-N recovery rates were determined by subtracting percent of $NH_4$-N losses during evaporation from 100% (Equation 2). Thus, it is evident that acidifying the biogas slurry increased the rate of $NH_4$-N recovery also stated in literature (Pantelopoulos et al., 2016). This is explain by the dynamic equilibrium between ammonium and ammonia ($NH_4^+ = NH_3\,(g) + H^+$) which is highly dependent on the temperature and pH. A greater ammonia concentration gradient between the biogas slurry and the surrounding environment results when pH rises (P. Wang et al., 2020). This causes the equilibrium to shift to the right, favoring $NH_4$-N volatilization (Menkveld and Broeders, 2018; Morey et al., 2023).

Studies have also revealed that after acid treatment, other physicochemical changes take place in biogas slurry (Maibritt Hjorth et al., 2009; Fangueiro et al., 2015). As presented in Table 4-2, as the pH dropped from 7.76 to 6, the sulfur concentration changed dramatically and the level increased from 220.38 mg/L to 700 mg/L. The presence of $SO_4^{2-}$ and $HS^-$ ions as a result of the dissociation of $H_2SO_4$ during acidification serves as justification for this (Fangueiro et al., 2015). In the present study, the inorganic content (dissolved in organic compounds) of the slurry such as P, Fe, Zn, Cu, Mg and Ca showed little or no significant differences relative to untreated slurry as they were non-volatile substances (P. Wang et al., 2020).

Table 4-2 Composition of concentrate BGS at different pH

| Parameter | Unit | pH=7.76 | pH=7 | pH=6 |
|---|---|---|---|---|
| pH | | 7.48 | 7.22 | 6.68 |
| EC | mS/cm | 10.5 | 18.2 | 20.5 |
| OC | % | 39 | 37.83 | 34.32 |
| $NH_4$-N | mg/l | 647 | 922 | 1,516 |
| Ca | mg/l | 674 | 1,019 | 986 |
| Mg | mg/l | 444 | 637 | 670 |
| Na | mg/l | 717 | 1,099 | 590 |
| K | mg/l | 1,202 | 1,191 | 1100 |
| P | mg/l | 506 | 498 | 542 |
| S | mg/l | 220 | 656 | 700 |
| Fe | mg/l | 185 | 224 | 200 |
| Mn | mg/l | 28.73 | 35.57 | 20 |
| Zn | mg/l | 11.72 | 14.9 | 8.9 |
| B | mg/l | 0.68 | 0.39 | 0.1 |
| Cu | mg/l | 1.95 | 1.13 | 0.09 |
| Mo | mg/l | 2.33 | 0.74 | 0.34 |
| Si | mg/l | 1.74 | 0.85 | 0.16 |

The decrease and increase of some species may be caused by the creation and dissociation of compounds during reactions in the system. $K^+$ concentrations, for instance, drop when the pH falls below 7.76-6. This might be because the cation and $SO_4^-$ react to form metallic sulphate.

The OC content of biogas slurry was determined to be 39%, 37.83%, and 34.32% at pH of 7.76, 7 and 6 respectively. As shown in Table 4.2, the organic carbon (OC) content of the treated biogas slurry is lower than that of the untreated slurry. Another study proved that this is due primarily as a result of C losses from $HCO_3/CO_3^{2-}$ conversions to $H_2CO_3$ and subsequent gaseous release as $CO_2$ (Fangueiro et al., 2009).

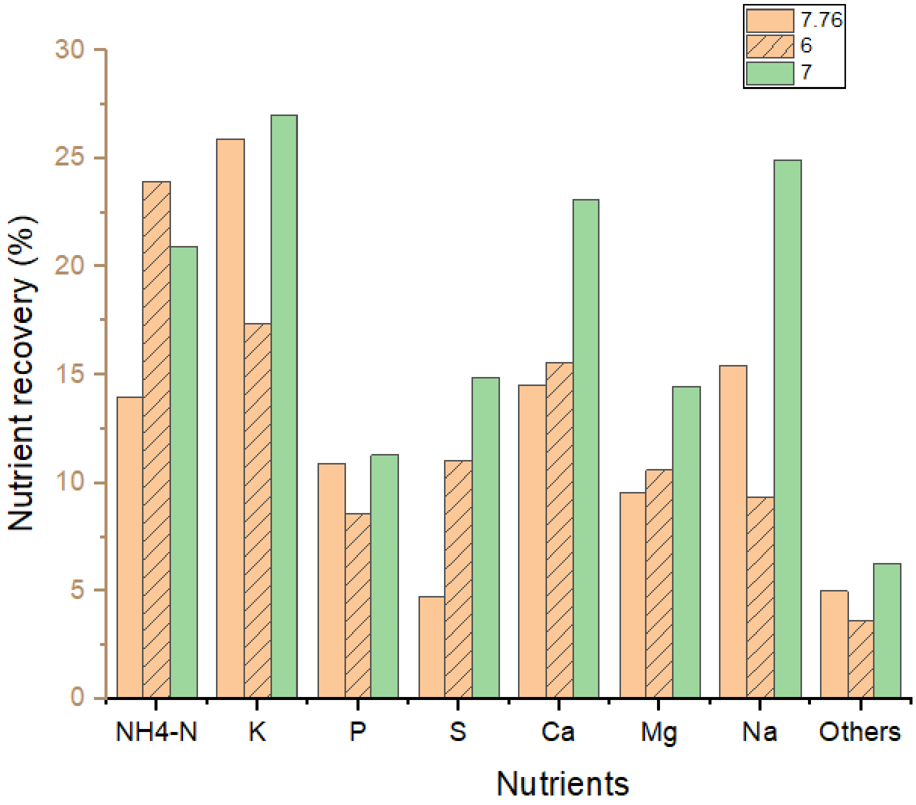

Figure 4-5 The effect of pH on nutrient recovery

The value of EC indicates the accumulation of $NH_4^+$and soluble salts in solution. The electrical conductivity (EC) increased from 10.5 mS/cm to 18.2 mS/cm when pH changed from 7.76 to 7. The highest electrical conductivity of 20.5 mS/cm was obtained at a pH of 6 (Table 4.2). The EC recovery trend and effect primarily paralleled with the $NH_4$-N content as other salt ions like $Na^+$ and $K^+$ were non-volatile substances and their concentrations changed little during evaporation unlike the biogas slurry's $NH_4^+$ content ((Pantelopoulos et al., 2016;  P. Wang et al., 2022).

To this end, the concentration of $NH_4$-N was 1,516.5 mg/L when the pH was 6, and it steadily dropped as the pH rose. When the pH reached 7.76, the concentration of $NH_4$-N decreased to 647.44 mg/L. Likewise the EC increased from 10.5 mS/cm to 20.5 mS/cm when the pH changed from 7.76 to 6.

Table 4-3Ammonium composition and pH level of condensate

| Liquid fraction pH value | $NH_4$-N (mg/L) | Condensate pH value |
|---|---|---|
| 6 | 14.88 | 7.28 |
| 7 | 42.91 | 9.06 |
| 7.76 | 90 | 9.4 |

Likewise, the concentration of $NH_4$-N in the condensate was investigated. The investigation is useful to determine the extent of ammonium volatilization (in the form of $NH_3$) during evaporation of biogas slurry. This is also important  to check whether the condensate complies with pollution standards before it  discharged to water bodies or used for  different application (Wang et al., 2020).

In the present study the concentration of $NH_4$-N and condensate pH were analyzed and reported in Table 4.3. The result showed that the concentrations of ammonium were 14.88 mg/L, 42.9 mg/L and 90 mg/L at pH of 6, 7, and 7.76 respectively. Maximum concentration value (90 mg/L) was obtained at alkaline conditions as ammonia volatilization is favored at higher pH. Lowest $NH_4$-N concentration (14.88 mg/L) in condensate was reported at pH of 6. This result also testify also that acid treatment of liquid fraction reduce $NH_4$-N loss by shifting the equilibrium towards $NH_4$-N (Fangueiro et al., 2015).

Moreover, at acidic, neutral, and basic conditions, the pH of the condensate was measured to be 7.28, 9.06, and 9.4, respectively. The higher the pH of the condensate indicates the higher amount of $NH^{+}_{4}$ lost with water vapor in the form of $NH_3$ during evaporation (Morey et al., 2023).

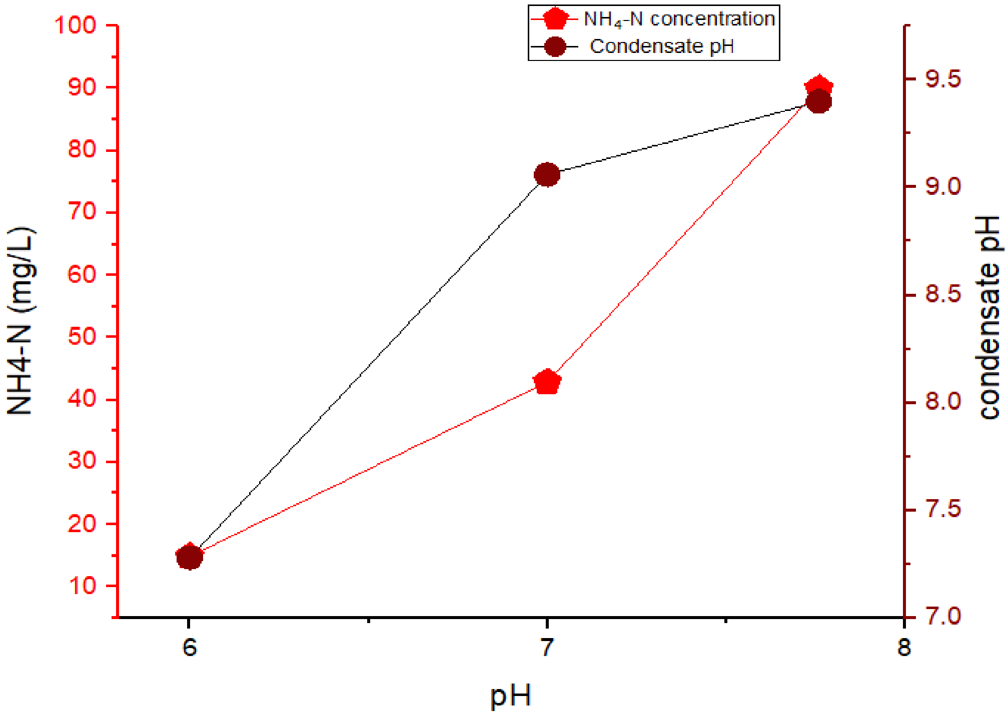

Figure 4-6. Effect of acid treatment on $NH_4$-N loss during evaporation

# 5. CONCLUSIONS AND RECOMMENDATIONS

## 5.1.Conclusions

In this study hybrid treatment methods were employed to concentrate biogas slurry (BGS) without losing its essential nutrient. The raw biogas slurry (RBGS) was chemically treated with the addition of Ferric chloride ($FeCl_3$) and polyelectrolyte polymer, PEP for retaining water soluble nutrients, maximize solid-liquid separation and thermal operation efficiency. Separation of BGS into liquid and solid fractions was performed by using vibrating screen and their nutrient distribution was investigated. While a larger amount of P was identified in the solid fraction, a significant component of the readily available soluble $NH_4$-N and K were exhibited in the liquid fraction.

Heating time, temperature and operating pressure were presented as major determinant during evaporation of liquid fraction of BGS. Maximum water removal efficiency was (55%) found at 45 min., 75 $^oC$ and pH of 7. This enables to utilize the waste heat from the biogas plant's combined heat and power (CHP) system, which is typically not higher than 80 $^oC$.

Optimum conditions for maximum $NH_4$-N recovery efficiency (95.1%) were obtained at heating time of 45 min., temperature of 75$^oC$ and pH of 6. Volatilization $NH_4$-N with water vapor hindered at lower pH condition.

The resulted concentrated biogas slurry (CBGS) is less bulky and has higher macro nutrient concentration than the raw biogas slurry. As a result, the CBGS can be delivered with less fuel and effort to the point of application and efficiently employed as organic fertilizers, insecticides, and seed dressers.

## 5.2.Recommendations

Due to time, finance and facilities constraints, this study only focus on concentrating of BGS without losing its essential nutrient using physico-chemical and thermal treatment. As biogas slurry concentration with thermal treatment is an energy intensive process, wise use of heat utilization has a prime importance. Hence for economic and sustainable BGS management strategies, further research on the heat consumption and optimization of evaporation of liquid fraction process is required. This may include investigating the effect of temperature and vacuum on water removal and nutrient recovery efficiency. Moreover, the technological and environmental feasibility of concentrating BGS using combined treatment methods needs further research.

The effectiveness of concentrating biogas slurry as organic fertilizer also requires further evaluation and investigation for its agronomic application. The concentration of nutrient content and trace elements concentration should be evaluated according to the national and international regulation. Therefore, both the raw BGS and concentrated products should be thoroughly investigated for its heavy metal concentration and presence of pathogen before agricultural application.

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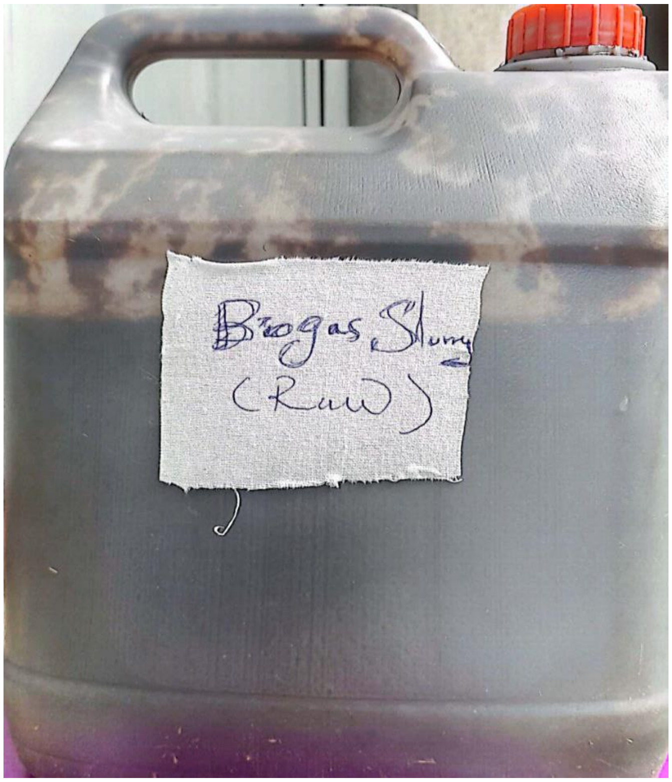
# APPENDICES

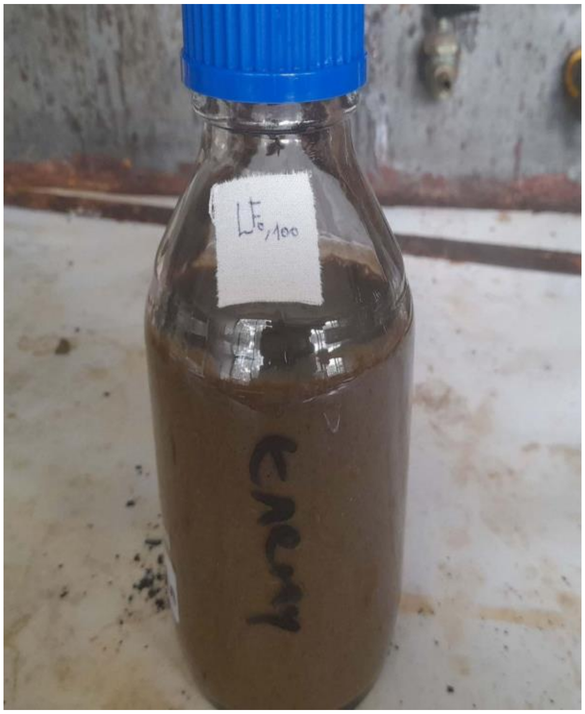
During biogas slurry sample collection



Raw biogas slurry (RBGS)

Coagulant and flocculant

Strirrer



**Chemically treated slurry ($LF_o$)**

**Vibrating Screens**

**Liquid fractions ($LF_1$)**

**Solid fraction (SF)**

Rotary Evaporator

Concentrate and condensate