OPTIMIZATION OF BIOGAS PURIFICATION USING WATER-SCRUBBING TECHNOLOGY FOR ENHANCED GAS QUALITY

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DECLARATION

I affirm that I am the author of the content in this report. The work presented herein is my own and has not been presented in or published by any other institution in whole or in part for any award except where due acknowledgment has been made.

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CERTIFICATION

The undersigned attest that they have reviewed and recommend the acceptance of a thesis entitled "**Optimization of Biogas Purification using Water-Scrubbing Technology for Enhanced Gas Quality"** at Masinde Muliro University of Science and Technology.

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DEDICATION

The work herein is dedicated to my family in appreciation of their prayers, unwavering support, continued encouragement, and understanding during my extended absence while undertaking the research.

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ABSTRACT

Biogas has been found to be a green source of power. It consists majorly of methane (CH4), carbon dioxide (CO2), hydrogen sulphide (H2S) and water vapour. Use of biogas has never reached its potential with the problem of inherent impurities that compromises its quality and restricts its usage. Purification of biogas is important in ensuring the gas is apt in a number of applications within the renewable energy sector. Amines scrubbing, physical scrubbing, cryogenic separation, pressure swing adsorption, membrane separation and water scrubbing are some of the methods which have been employed to upgrade biogas. Water scrubbing has, however, been highly applied in the enrichment of CH4 due to its nature of availability and ease of use. Various studies have applied water scrubbing to remove impurities from biogas; however, little has been done from these studies to establish the optimal working parameters applicable to small household operators. This study, therefore, focused on optimizing the water scrubbing process for optimal biogas quality using water scrubbing technology. The study optimized the following variables: water flow rate (WFR), the gas flow rate (GFR), pressure (P), and gas retention time (RT). WFR was varied at the rates of 3.5, 4.0, 4.5, 5.0, and 5.5 litres per hour (l/hr), while the GFR was varied at 7.5, 8.0, 8.5, 9.0, and 9.5 litres per minute (l/min). P was varied at 0.04, 0.05, 0.06, 0.07, and 0.08 bar, while RT was varied at 30, 45, 60, 75, and 90 seconds. A set of 30 runs was experimented and the percentage gas composition in the upgraded biogas recorded using a SKY2000-M4-WH multi-gas detector. The results of the experiment were analyzed and optimized using ANOVA, Response Surface Methodology, and Design Expert software version 13. The raw biogas of an initial composition of 80.66% Methane, 15.76% Carbon dioxide, and 10 mg/m^3 Hydrogen Sulphide was used as a control experiment. This study found that the optimal CH_4 enrichment of 91.45% was observed at the following values of WFR, GFR, P, and RT of 5.0 l/hr, 8.0 l/min, 0.07 bar, and 75 seconds, respectively. A combination of these factors gave the optimal results: CH_4 improved by 10.79% (from 80.66% to 91.45%), CO_2 had the lowest value of 6.91%, and H_2S was zero mg/m^3. The predictive models developed revealed that methane enrichment increases with the WFR and RT, while it decreases with an increase in the GFR. It is recommended that further research be conducted to establish the rank of influence of the factors for further optimization.

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LIST OF ABBREVIATIONS AND ACRONYMS

AD	Anaerobic Digestion
ANOVA	Analysis of Variance
BBD	Box-Behnken Design
CCD	Central Composite Design
CHP	Combined Heat and Power
CV	Coefficient of Variation
DoE	Design of Experiment
E	East
GB	Giga Byte
GFR	Gas Flow Rate
GHGs	Greenhouse Gases
hr	hour
kbar	kilo bar
LoF	Lack of Fit
MEA	Mono Ethanol Amine
mol	mole
MPa	Mega Pascal
P	Pressure of scrubber

PSA	Pressure Swing Adsorption
RSM	Response Surface Methodology
RT	Retention Time
Std. Dev	Standard Deviation
TM	Taguchi Method
VoCs	Volatile organic compounds
WFR	Water Flow Rate
2D	Two Dimensional
3D	Three-Dimensional
2FI	Two-factor-interaction

LIST OF SYMBOLS

Ɛ	Error term
%	Percent
β	Coefficient of regression
°C	Degree centigrade
CH_4	Methane
CO_2	Carbon dioxide
CO_3^{2-}	Carbonate ion
H	Hydrogen
H+	Hydrogen ion
HCO_3^-	Hydrogen Carbonate ion
H_2CO_3	Carbonic acid
H_2O	Water
H_2S	Hydrogen Sulphide
H_2SO_4	Sulphuric acid
K	Kelvin
K_H	Henry's coefficient of solubility
m	Metre
mm	Millimetre

N_2	Nitrogen
NH_3	Ammonia
O_2	Oxygen

CHAPTER ONE

1. INTRODUCTION

1.1 Background

Biogas is a renewable energy source that has been proven as a suitable substitute for fossil fuel-based energy systems, which are getting scarcer every day. It is a promising biofuel and a potential transition resource among renewable energy supplies. It is formed through the fermentation of organic as well as biodegradable materials like sewage, biomass, animal, and municipal wastes in an air-tight environment (Deepanraj *et al.*, 2014). Al Mamun and Torii (2015) noted that raw biogas is made up of methane (40 -70%), carbon dioxide (30 - 40%), hydrogen sulfide (0 – 1%), and inconsiderable quantities of water vapour and volatile organic compounds. However, Bharathiraja *et al.* (2018) observed that this composition does not always remain constant; it differs depending on several factors such as the kind of substrate or organic matter used and the decomposition process. Biogas purification is useful for cleaning the gas and making it suitable for various applications in the renewable energy sector (Daniel-Gromke *et al.*, 2018).

When the methane content exceeds 50%, biogas burns very well, making it a possible alternative to kerosene, charcoal, or firewood for cooking and lighting (Budzianowski, 2016). However, using biogas as a renewable energy source has not reached its full potential yet, mainly because of the impurities it contains, particularly the presence of carbon dioxide reduces the calorific value of the gas while the presence of hydrogen sulfide causes corrosion because of a chemical reaction with water vapor, forming sulfur dioxide, which is both corrosive and toxic to human health (Abdeen *et al.*, 2017). These impurities can make biogas unsuitable for certain important uses, like fuel for transportation or domestic purposes, which require high-quality standards.

Biogas, as a renewable energy source, is used in different applications both commercially and domestically. The commercial use includes: generation of power and electricity used to power factories, production of heat and steam, vehicle fuel, feedstock for the production of bio-based chemicals and substrate in fuel-cells, starting reactants in chemical processes, substitute for natural gas for industrial use, and gas grid injection. Similarly, biogas has been used domestically by medium-scale plant owners for lighting, cooking, and gas-powered incubators as well as refrigerators. Its versatility helps to use space heating, water heating, cooking, lighting, microturbines, gas engines, and fuel cells for continuous energy production (Ajay *et al.*, 2021; Lohani *et al.*, 2022).

Biogas purification is becoming crucial for making the gas suitable for various applications in the renewable energy sector, since there is a shift from conventional energy sources to renewable energy sources due to the environmental concerns associated with the increasing human population.

The initial biogas upgrading plants were built in the 1980s. Since then, there has been little modernization of biogas purification until the year 2006, when Germany focused on further study and development of biogas cleaning (Bond & Templeton, 2011). There are nearly 282 biogas stations in Europe, producing 1.3 billion cubic metres (m^3) of biogas annually. Biogas has tremendous potential to be used as a transportation fuel. However, the main obstacle in such a field is the presence of several contamination components that are responsible for making biogas less efficient as a transportation fuel. For this reason, in many countries, especially in Europe, several techniques are being used to remove the traces of moisture, Carbon dioxide, and Hydrogen Sulphide. Some profitable technologies related to biogas upgrading include water scrubbing (Al Mamun, 2018; Huqe *et al.*, 2018). Biogas improvement to enhance quality of biogas and its energy content is essential in ensuring efficient use of this renewable energy source.

Biogas has been upgraded using different techniques. These are cryogenic separation, pressure swing adsorption, membrane separation, chemical absorption and physical absorption (water scrubbing) (Andriani et al., 2014). The most prevalent of them is water scrubbing due to the accessibility of water that is easy, the simplicity and affordability of such system (Kapoor et al., 2017).

Water scrubbing is an approach to remove pollutants, contaminants or harmful gases in the industrial exhaust or flue gases (Nock et al., 2014). It operates on the principle of solubility differences between methane, carbon dioxide and hydrogen sulfide. Methane is not as soluble in water as carbon dioxide and hydrogen sulfide. It was found by Bauer et al. (2017) that the two gases, CO_2 and H_2S, are simple to separate with the help of water scrubbing because under room temperature conditions, methane is not very soluble in both gases, unlike the latter.

Vijay et al. (2006) developed a CO_2 cleaning system of the raw biogas which had approximately 40 per cent of CO_2. In their experiment, they used 1.8 and 2.0 m^3/h as the rates of water and they used 1.0 and 3.0 m^3/h as the rate of gas. Based on the results of the study they derived, they came to the conclusion that the amount of carbon dioxide that was absorbed was dependent on the rate of water and gas flow. The optimal percentage of CO_2 that was eliminated was 98.62. This was achieved at 1.8 m^3/h and 1.5 m^3/h flow rate of water and gas respectively.

The new technologies that are currently being developed to upgrade biogas focus on maximization of methane content at a minimum cost. These technologies, such as membrane separation, pressure swing adsorption (PSA), and biological methods, produce cleaner and high-quality biogas, which is utilized for various applications (Angelidaki *et al*., 2018; Das *et al*., 2022; Sahota *et al*., 2018; Salave and Desai, 2017; Sun *et al*., 2015). New strategies are looking into using affordable materials to capture pollutants and developing new catalysts to improve biogas conversion processes.

Work done by (Gantina *et al*., 2020; Kapoor *et al*., 2017; Nock *et al*., 2014; Vijay *et al*., 2006) indicates that most of the research previously undertaken focused mainly on water scrubbing as a biogas upgrading technology. These studies, however, concentrated mostly on purifying biogas using water scrubbing under high pressure, which consequently required high power to operate, limiting their application to commercial use. Few studies have focused on low-pressure water scrubbing operated at room temperature, such as the work done by Mugagga *et al*. (2022), who developed a system for optimizing and analyzing low-pressure water scrubbing. This study was conducted at a low pressure of 0.07 bar and an ambient temperature of 23 °C.

Despite water scrubbing being the most widely used purification technique, little effort has been invested in the optimization of water scrubbing technology in Kenya. This is evident in the work done by Mugagga *et al.* (2022), who discussed the optimization and analysis of a Low-Pressure water scrubbing biogas upgrading system using the Taguchi and Response Surface Methodology approaches. They maximized low pressure water scrubbing in their work and obtained the highest bio-methane concentration of 84.71 and 13.31 CO2 reduction.

The successful operation of a water scrubbing process relies on a number of factors, that is, the rate of water flow, gas flow rate, the retention period of the gas, the specification of the scrubber, the scrubber pressure, the composition of the raw biogas, and the purity of the water (Noorain et al, 2019). The above process parameters must be optimized to ensure that the system has an optimal performance.

The Taguchi Method and Response Surface Methodology (RSM) are some of the techniques that can be employed to find the optimal process parameter of a certain system according to Zabava et al. (2018). In spite of its simplicity, Taguchi method entails a three step process that includes system design, parametric design and tolerance design. This approach takes a lot of time since additional optimization procedures, including ANOVA will be needed to confirm the best scores (Okanminiwei and Oke, 2020). RSM is one of the statistical techniques that are applied to find out the impact of various independent variables on a specified process. Karmoker et al. (2019) have observed that RSM is a statistical tool that can be used effectively in calculating the optimal combination of factors to ensure the optimal system performance. RSM, in its turn, has the benefit of being less expensive to experiment with because fewer experimentation runs are required, and it can simultaneously optimize more than three independent variables (Kannan and Shanbhag, 2019; Kono et al., 2018).

The main aim of the research was to maximize the purification of bio gas in the application of water scrubbing technology in improving the quality of biogas. Water scrubbing being the most used method, little has been done to ensure that it is operated at optimal conditions. The study seeks to contribute to the pool of knowledge on the optimization of water scrubbing technology as a method of purifying biogas by optimizing the water scrubbing process to enhance the gas quality.

1.2 Problem Statement

Conventional fuel sources are currently facing extinction due to environmental concerns, which has necessitated the venture into alternative sources of energy that are recyclable. Biogas as a source of sustainable energy has significant potential for both domestic as well as industrial applications. However, the use of biogas as a sustainable energy source has not yet attained its full potential due to the challenge of inherent impurities presence. Studies depict that biogas primarily comprises 40% to 70% methane by volume (combustible), and 30% to 40% carbon dioxide, along with small amounts of other gases and water vapour ranging between 0% to 1% by volume. These impurities make biogas inappropriate for vital commercial and domestic applications, such as transport fuel, which requires strict quality demands, hence need for purification.

Various purification techniques have been used to enhance methane content in biogas; such as water scrubbing, pressure swing adsorption, membrane separation, cryogenic separation, and biofiltration of which, water scrubbing is the most popular with over 41% of entire techniques (Andriani *et al*., 2014). Despite water scrubbing being the most frequently used purification technique (due to its simplicity and availability), little work has been done in Kenya on the optimization of water scrubbing technology especially for small-scale applications. This is evident in the work done by Mugagga *et al.* (2022). It is based on this gap that this research was conducted to optimize biogas purification by the water scrubbing method for enhanced gas quality.

1.3 Objectives

1.3.1 Broad Objective

The main purpose of the study was to optimize the biogas purification process using water scrubbing technology for enhanced gas quality.

1.3.2 Specific Objectives

i. To determine the water flow rate and biogas flow rate that optimizes the biogas quality
ii. To find out the pressure in the scrubber at which the quality of upgraded gas is optimum.
iii. To determine the retention time of biogas in the scrubber for optimal removal of Carbon dioxide and Hydrogen Sulphide.
iv. To develop the predictive model for CH_4 enrichment, CO_2, and H_2S removal.

1.4 Research Questions

The study was guided by the following research questions;

i. Does the flow rate of water and biogas affect the optimization of biogas purification using water scrubbing technology?

ii. At what pressure should the scrubber operate to ensure optimal removal of CO_2 and H_2S?

iii. How long should the biogas be retained in the scrubber for maximum biogas quality?

iv. What are the predictive models that optimize the CH_4 enrichment, CO_2, and H_2S removal?

1.5 Justification

Biogas is suitable as a fuel for most applications. However, using it in its raw state has several limitations. If it is to be used for powering vehicles, however, the presence of CO_2 is unsatisfactory, because it lowers the power output from the engine, takes up space in the storage cylinders, and can cause problems of freezing at valves and metering points, where the compressed gas expands during running, refuelling, as well as in the compression and storage. All or most of the CO_2 must, therefore, be removed from the raw biogas to prepare it for use as a fuel for vehicles, in addition to compression of the gas into high-pressure cylinders, carried by the vehicle (Lohani *et al.*, 2022).

Hydrogen sulphide should also be removed to avoid the formation of corrosive gases, which combine with water vapours to form acids, and hence corrode all metal parts of the engine and gas system (Abdeen *et al*., 2018). These impurities make biogas inappropriate for vital commercial and domestic applications, such as transport fuel, which requires strict quality demands. The work done by Lumadede *et al*. (2021) revealed that Kenya has over 20,000 potential biomethane plants, and yet only 30% of these plants are functional because of impurities, implying that 70% are underutilized.

Carbon dioxide and hydrogen sulphide reduction have the effect of mitigating these issues of global warming by addressing the environmental concern of the emission of global warming gases. Biogas utilization has provided a great potential in the reduction of greenhouse gases, which cause global warming and climate change (Lippmann et al., 2003).

Even small households can use biogas to obtain light and it does not lead to air pollution. The big industrial companies can use biogas to generate electricity to support their processes and even sell the surplus energy to the National Grid (Rajendran et al., 2012).

Biogas is rich in calorific material, methane taking more than half of its structure, which makes it a potentially suitable alternative to kerosene, charcoal, and firewood as a cooking and lighting fuel (Budzianowski, 2016). Cooking activities in biogas help save women time that they would have used in harvesting wood thus enabling them to carry out other activities that are empowering economically. Additionally, it minimizes smoke emissions, which are a major cause of respiratory diseases and impaired vision among rural women and children who cook with firewood in poorly lit and ventilated spaces (Nzila *et al.*, 2015).

Gas-powered refrigerators and chicken incubators can use biogas to operate; this has been established in Kenya (Sibisi & Green, 2005). In the countries of India and Nepal, biogas systems are integrated with sanitation (toilets) facilities for lighting purposes (Batzias *et al.*, 2005).

Biogas' versatility can be used in space heating, water heating, to drive microturbines, and gas engines, as well as in fuel cells for continuous production of energy (Ajay *et al*., 2021; Lohani *et al*., 2022).

Optimization of factors affecting biogas purification assists in determining the level of combining the parameters that result in the optimal biogas quality (with the highest methane concentration).

1.6 Scope

The primary objective of the study was to establish the optimal operational parameters required to ensure optimal biogas quality using water scrubbing technology at Biogas International Ltd in Kisumu. The study encompassed biogas purification by water scrubbing technology and did not involve the digester installation and biogas production process. The sample biogas was extracted from the biogas plant installed at Biogas International Ltd, based in Kisumu.

1.7 Conceptual Framework

Figure 1.1 shows the conceptual framework for optimization of biogas purification by water scrubbing.

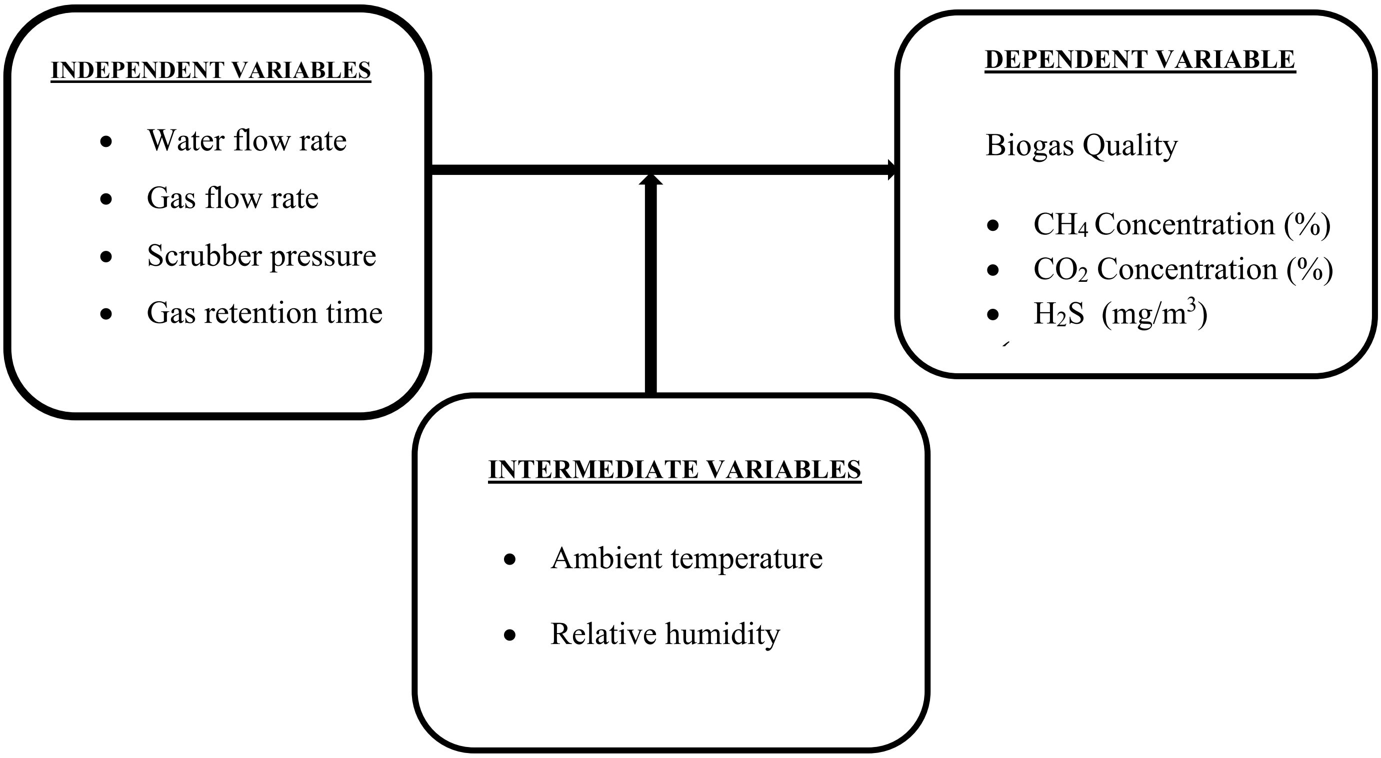


Figure 1.1: Conceptual flow for optimization of biogas purification by water scrubbing.

The water scrubbing of biogas purification consisted of three stages including sampling of raw biogas, dispensation procedure, and gathering of refined gas to be sampled. The sampling stage involved a sampling bag in which raw and purified biogas would be gathered to determine its composition, and a gas flow meter. Purification stage involved scrubbing chamber, water dripping down and a biogas entry at the bottom of the scrubber. The gas and water were counter-flowing in order to realize maximum absorption. The final step was the process of collection and sampling of upgraded biogas, which would then be analyzed by determining the composition of the biogas using SKY2000-M4-WH multi-gas detector.

CHAPTER TWO

2. LITERATURE REVIEW

The chapter reviews some of the available literature that relates to optimization of biogas purification with particular reference to the application of water purification technology.

2.1 Introduction

Biogas is a product of the bacterial decomposition of organic substance under the absence of air, by means of a process called anaerobic digestion. Nallamothu et al. (2013) report that biogas consists of methane (CH4), carbon dioxide (CO2), hydrogen sulphide and minute amounts of water vapour. The digestion of organic matter anaerobically (AD) involves the degradation of organic matter by various types of bacteria by a complex pathway of different steps. This occurs under a variety of conditions, including digestive system conditions, wetlands, marshes, sewage systems, rubbish dumps, and the Arctic Tundra (Lee et al., 2019). A biogas is the result of the degradation of organic substances, such as human feces, animal wastes, crop straws, kitchen waste, and industrial waste (Njogu et al., 2021). Organic wastes yield two main by-products, biogas and slurry produced by the AD. The biogas is then removed and the slurry is released out of the digester as the by-product of the process (Rasi et al., 2011). As Daniel-Gromke et al. (2018) claim, the main constituents of biogas are CH4 and CO2. Bio-gas may be utilized instead of kerosene, charcoal, and firewood because it burns highly well when the amount of CH4 is over 50 percent (Budzianowski, 2016).

2.2 Biogas Composition

Biogas is a mixture of methane (CH4) and carbon dioxide (CO2) with traces of water vapour and minor amounts of other gases including hydrogen sulphide (H2S). The composition of biogas depends on the type of feedstock as well as the rate of the digestion process. Table 2.1 provides the composition of biogas as discussed by Bharathiraja et al. (2018).

Table 2.1: Biogas composition

S/NO.	Gas	Chemical Symbol	Percentage (%)	Composition	Combustibility
1.	Methane	CH_4	40-70		Combustible
2.	Carbon dioxide	CO_2	30-40		Non-combustible
3.	Hydrogen	H_2	5-10		Combustible
4.	Nitrogen	N_2	1-2		Non-combustible
5.	Water Vapour	H_2O	0.3		Non-combustible
6.	Hydrogen Sulphide	H_2S	0-1		Non-combustible
7.	Ammonia	NH_4	0-3		Non-combustible

Table 2.1 shows that methane and hydrogen are the combustible constituents of biogas, whereas the other constituents are non-flammable under normal conditions. This lowers the quality of biogas (Bharathiraja *et al.,* 2018). Furthermore, only CH_4 is present in considerable quantities among these two gases; hence, it is taken into account in the majority of biogas cases. This indeed signifies that to enhance biogas quality, the methane content should be

increased while non-combustible elements should be eliminated. This has been done through various biogas upgrading technologies.

The major problem with the low uptake of biogas on a commercial scale is the reduced calorific content per unit volume attributed to the presence of impurities and its inability to liquefy under normal conditions. To commercialize biogas, it is imperative to make it transportable and adaptable to multiple commercial applications. To do this, the calorific value for a specific volume should be raised. This necessitates biogas purification to eliminate non-combustible components, lowering its quality (Al Mamun & Torii, 2015).

The general gaseous fuel properties are indicated in Table 2.2 (Bharathiraja *et al.*, 2018).

Table 2.2: General properties of gaseous fuels.

Gas	Calorific Value (MJ/m^3)	Specific Gravity	Boiling Point (K)	Ignition Temperature (K)	Flammability Limits in air (% Volume)
Biogas	23.1	0.80	-	923.15	8 - 18
Carbon dioxide	-	1.52	194.65	-	-
Methane	39.80	0.55	111.75	863.15	5 - 15
Natural Gas	38.70	0.65	14.45	901.15	-
Ethane	60.80	1.048	185.05	788.15	3 - 12
Propane	88.40	1.52	229.75	743.15	2 - 9

2.3 Thermo-Physical Properties of Biogas

Thermo-physical properties discussed are those thermodynamic parameters, including but not limited to the latent heat of vaporization and specific heat, pressure, density, temperature, as well as transport properties like thermal conductivity and viscosity, that have an impact on the transfer of heat and dynamics of pressure performance (Yaru *et al.*, 2014).

The thermo-physical characteristics of biogas are also changing according to the composition of gases and their concentration (Bharathiraj et al., 2018). These properties are very important in the places where biogas is applied including energy generation, heating, and industrial activities.

2.4 Equation of State of Biogas

It is a mathematical model that forecasts the nature of a gas when subjected to a set of given physical conditions. A unique correlation between variables of the gas state, including temperature, volume, pressure, gas density, and internal energy, is formed with the help of the equation (Marzuki and Wicaksono, 2017). The state behavior of biogas is imperative to comprehend so as to increase the safety of the person who uses biogas as well as the equipment utilized at biogas plants. This is done by determining the amount of biogas in the gas holder of a biogas plant under varying conditions of the state and studying the behaviour of biogas in the internal combustion engine (Travnicek et al., 2018).

According to Marzuki and Wicaksono (2017) the equations to compute the state quantities of biogas could be formulated by using two equations, the Redlich-Kwong and the Van der Waals equations as shown below;

a) Van der Waals Equation of State

The equation is described below;

$$\boldsymbol{p = \frac{r \cdot T}{v-b} - \frac{a}{v^2}} \tag{2.1}$$

where;

p ....the gas pressure [Pa]

r ....the gas constant [$J \cdot kg^{-1} \cdot K^{-1}$]

v ....the molar volume [$m^3 \cdot kg^{-1}$]

T ...... the absolute temperature [K]

a, and b .... are positive constants

b) Redlich-Kwong Equation of State

The Redlich-Kwong (Markočič & Knez, 2016) the equation is formulated as;

$$\boldsymbol{p = \frac{r \cdot T}{v-b} - \frac{a}{v^2} - \frac{a}{\sqrt{T} \cdot v(v+b)}} \tag{2.2}$$

where;

p ....the gas pressure [Pa]

r ....the gas constant [$J \cdot kg^{-1} \cdot K^{-1}$]

v ....the specific molar volume [$m^3 \cdot kg^{-1}$]

T …... the absolute temperature [K]

a and b .... are positive constants.

The constants **a**, and **b** are defined by the following equations;

$$\boldsymbol{a = \frac{27}{64} \frac{R^2 {T_c}^2}{p_c}} \text{ (cm}^6 \text{ bar)} \tag{2.3}$$

$$\boldsymbol{b} = \frac{\boldsymbol{1}}{\boldsymbol{8}} \frac{\boldsymbol{R T_c}}{\boldsymbol{p_c}} \ (\text{cm}^3) \tag{2.4}$$

where;

R …. the gas constant,

T_c …. the critical temperature,

p_c … the critical pressure

The constant ***a*** accounts for the attractive interactions between gas molecules, while the constant ***b*** accounts for the space or volume that the gas molecules occupy.

2.5 Biogas Purification

Purification of biogas refers to the extraction of the unwanted biogas components before the biogas can be used in the desired processes. The combustible element is the only methane (Zabava et al., 2019).

Other methods have been employed to increase the levels of methane in the biogas by removing large quantities of carbon dioxide (CO2) and hydrogen sulphide (H2S) (Zabava et al., 2018). The majority of the processes mentioned above were intended to be used in petrochemical, petroleum, and natural gas industries (Peluso et al., 2019). As a result, some of them might not be sufficient in biogas use unless it is necessary to have high flow rates (Haldar et al., 2023).

Vijay et al. (2006) note that in most processes that are applied in CO2 removal, H2S is removed. Such processes are: chemical conversion, membrane separation process, cryogenic separation, adsorption on solid substrates, physical and chemical absorption. The purification procedure that is selected is based on the purity required, the scale of operation, the composition of the biogas and the need to extract CO_2 in a given application (Andriani et al., 2014).

The biogas is a considerable biomass fuel that can be used to replace fossil fuel in heating and power production processes through the anaerobic digestion of organic compounds. Raw biogas should be processed in order to be utilized profitably or economically. As such, purification is done on unpurified biogas prior to its use, which gives it the properties required in the necessary application (Zabava et al., 2019).

Scholars such as Angelidaki et al. (2018), Kapdi et al. (2005), Salave and Desai (2017), Sun et al. (2015), Pareek (2021), and Sahota et al. (2018) and other scientists have explored the technological development of biogas purification in the literature appropriately. The researchers gave the various technology applied to improve the quality of biogas, how it works, the process, equipment involved, and the necessary requirements in the purification of the gas. As it has been mentioned in Angelidaki et al. (2018), there were special steps to upgrading a biogas, including cleaning of biogas, which removes harmful, corrosive, and dangerous biogas components, and can also upgrade biogas.

The technologies of biogas purifying are divided into two, depending on the gases to be cleared and the way of the gases separation. Kapdi et al. (2005) discussed some of the purification methods depending on the gas (CO_2 and H_2S) to be purified. These methods comprised of chemical conversion, membrane separation methods, cryogenic separation methods, adsorption on solid material and physical and chemical absorption. Agarwal and Shukla (2009) explained a process of to eliminate H_2S in biogas, which involves the application of an iron wool-based acidic scrubbing system and a lime or water scrubbing system.

The methods of biogas scrubbing are divided according to the kind of absorbents they use to purify the impurities. Bauer et al. (2013) explained a liquid scrubbing system as a system that uses absorbents, i.e., water, solvent, and pressure swing adsorption, amine scrubbing which involves the use of porous material as an absorbent, and membrane gas diffusions which involved separation of gases by the size of the molecules. The technological review offered by Sun et al. (2015) was an overview, based on comparable classifications. One of the new tendencies that Bauer et al. (2013) examined was the hydrate formation, the biological and in-situ membrane enrichment methods of upgrading biogas. The advancements were the basis on which the energy and financial costs of both technologies were compared. (Srichat et al. (2017), Akila et al. (2017), Vrbova and Ciahotny (2017), and Kosna (2018).

2.6 Biogas Purification Methods

2.6.1 Physical Purification Techniques

These include:

i. Adsorption

Adsorption refers to the adherence of materials to solid surface. When the biogas is filtered through an adsorbent, the contaminant molecules will be attached to the surface of the adsorbent and thus removes the contaminants in the gas stream. Successful adsorbents are normally characterized by a high surface area and high porosity, which consequently enhance their removal capacity to a large degree (Zabava et al., 2019).

Anceita Jepleting, Doricah Moraa, Dorcas Cheptoo, and Mecha, (2024) also compared the two adsorbents (iron oxide and activated carbon) in the removal of both CO2 and H2S in biogas. They discovered that the combination of the activated carbon-iron oxide achieved H2S and CO2 removals of 100 percent and

97.7 per cent respectively. This means that the adsorption technique can be used successfully.

ii. Absorption

The essence of biogas purification is absorption, which refers to the process of extracting the contaminants, primarily H_2S and CO_2, out of biogas to increase its quality to be used in certain applications (D Andriani et al., 2020). In the absorption process selective solvent is applied to absorb the impurities found in the biogas. The choice of solvent depends on the impurities that are supposed to be removed. As an example, using aqueous solutions of such chemicals as monoethanolamine (MEA), Potassium carbonate (K_2CO_3), or water is effective in CO_2 removal, whereas amine-based solvents such as diethanolamine (DEA) or methyldiethanolamine (MDEA) can be used in removing H_2S.

Absorption normally occurs in a packed column with the biogas being used as the feed to the system at the bottom of the column and the water which is used as the solvent being added to the system at the top of the scrubber. The water is mixed with biogas as the flow of water is in counter current allowing the impurities to have ample time to dissolve the water. The biogas that flows out of the opening at the top of the column does not contain Carbon dioxide and Hydrogen Sulphide. The water which includes the dissolved carbon dioxide and hydrogen sulphide spreads out of the scrubber and is gathered at the bottom to undergo further treatment to remove the dissolved gases.

The absorption process is dependent on a number of factors. These are: biogas contact time, water temperature, scrubber pressure, solvent chemical characteristics and concentration of impurities. The best operating conditions are arrived with in order to have maximum efficiency on the removal of impurities and minimum use of energy.

Altogether, absorption is a major process of biogas purification because it is one of the most efficient methods of removing the impurities, as well as enhancing the quality of the biogas to be utilized in other areas, such as electricity production, heating, or renewable fuel..

iii. Membrane Separation

Membrane separation is a set of technologies that make use of differences in the pressure of the solution entering contact with the membrane and the size of the pore of each material of the membrane to address the filtering and separation of substances. These technologies enable compounds to diffuse through membrane pore selectively (Chen et al., 2015).

Chen *et al.* (2015), noted that despite the membrane separation method having considerable advantages such as lower energy consumption, easily engineered modules, good selectivity, and high methane recovery efficiency (>96%), it requires multiple steps to reach high purity. According to Vijay *et al.* (2006), membrane separation technique has fouling effects as it operates near atmospheric pressure, and its robustness makes it expensive.

iv. Pressure Swing Adsorption

The process of segregating particles in a gas mixture at high pressure. Depending on the gas's molecular properties and affinity, different activated carbons, molecular sieves, or zeolites are frequently employed as adsorbing materials.

These materials preferentially adsorb CO_2 and H_2S from biogas, enhancing the methane in biogas. The adsorption rises with the increase in pressure. Reducing the pressure releases the adsorbed gas. This results in the segregation of gases since different molecules in a gas mixture are more or less strongly attracted to different solid surfaces. The regeneration process occurs when the adsorbed bed is almost saturated, significantly lowering pressure and releasing the adsorbed gases (Chen *et al.*, 2015).

v. Cryogenic Separation

Cryogenic separation involves a gradual diminution of the 2temperature with a constant pressure. This allows removal of various biogas pollutants such as H 2 S, water, siloxanes and CO2. The majority of the cryogenic system designs have a multi-phase alternating cooling and compression phase of gas. Biogas is pressed to pressure of more than 10 bar and slowly cooled to about -25 C to separate water, the removal of hydrogen sulphide, carbon dioxide and siloxanes occurs at temperatures of -55 o C down to -85 o C. Pure biogas is about 97 per cent pure. Nevertheless, cryogenic separation does not have wide application, and it captures 0.4% of the global biogas purification market (Bauer et al., 2013).

2.6.2 Chemical Purification Techniques

i. Water Scrubbing

Water scrubbing is a process involved in removing pollutants, contaminants, or other unwanted gases in the industrial exhaust gases or any other gas (Nock et al., 2014). This is achieved by having the biogas pass through water or some water solution whose main aim is to trap, dissolve and extract the soluble polluted substances (Carbon dioxide and hydrogen sulphide gases). The water used in the scrubbing process is called the scrubbing solution or absorbent in this case water is the scrubbing solution (Cynthia et al., 2009).

The most widespread technique of cleaning up biogas is water scrubbing due to the availability of the water factor, simplicity and cost-effectiveness of the system (Kapoor et al., 2017). It operates on the principle of solubility differences in the solubility of methane, carbon dioxide and hydrogen sulfide. Methane is not soluble in water as easily as carbon dioxide and hydrogen sulfide. Bauer et al. (2017) noted that water scrubbing is an easy separation of methane with either CO 2 or H 2 S since at the room temperature, methane is nearly insoluble with both CO 2 and H 2 S.

ii. Chemical Absorption

According to Ardolino et al. (2021), a scrubbing technique involves the use of chemical absorption as the solvents in which carbon dioxide and hydrogen sulphide are removed using organic amines as the solvents. These organic amines are diethanolamine (DEA), diglycolamine (DGA), monoethanolamine (MEA) and methyldiethanolamine (MDEA). The operation principle is similar to water scrubbing. However, the amine solvents have strong CO_2 absorption capacities compared to water, hence extracting more carbon dioxide per unit volume, and since the upgrading systems are smaller (Patterson et al., 2011). Moreover, amine solvents do not consume much electricity because they operate at pressure, which is almost equal to atmospheric pressure. Nevertheless, in order to avoid the decrease in the process efficiency, they require a certain amount of thermal energy to be regenerated within the stripper (Bauer et al., 2013). The Chemical Absorption technology provides the optimum methane concentration in enhanced biogas.

iii. Catalytic Conversion

Catalytic conversion refers to a chemical process in which a catalyst is used to facilitate and accelerate the conversion of reactants into desired products. The catalyst lowers the activation energy of the reaction, thereby increasing the rate of reaction and efficiency (Zain & Mohamed, 2018).

This technique, when applied to biogas purification, involves converting the main components of biogas, methane and carbon dioxide, into other products like biomethane, hydrogen, or biofuels (Bao & Yu, 2018). The methanation process is an example of catalytic conversion in which CO_2 is converted to CH_4 through reaction with hydrogen. This technique is the most widely used in the upgrading of biogas by catalytic conversion (Barz *et al*., 2024).

2.6.3 Biological Purification Techniques

a) Biogas Upgrading with Microorganisms

This process relies on the ability of microorganisms to convert CO_2 into beneficial products. Biological CO_2 fixing is a practical way of reducing CO_2 concentration in biogas since it is friendly to the environment and eradicates the step of captured CO_2 disposal (Adnan *et al.*, 2019).

The use of hydrogen for the bioconversion of carbon dioxide to methane based on the activity of hydrogenotrophic methanogens is one of the biological ways to utilize carbon dioxide in biogas.

b) Biofiltration

The biofiltration process makes use of the natural biological metabolic processes of sulfur-oxidizing bacteria species, to transform hydrogen sulfide into elemental sulfur or sulfate. These systems are designed in a manner that they favor dense population of microbes and maximum contact of the microorganisms with the feed gas (Zhu and Granick, 2001).

A biological filtration consists of biological desulfurization and water scrubbing. This biogas is blended with 4-6 percent of air in the atmosphere and then it is introduced into the filtration medium. The pollutants in a bio-scrubber system are absorbed in a liquid which passes counter-currently through an absorption column, similar to the working principles in a water scrubber. The liquid is then moved to a bioreactor where microbial organisms are used to degrade the contaminants. These pollutants contained in the biogas are absorbed and adsorbed onto the biofilm and then they interact with the microbial populations. Biogas is pumped through a biofilter which is composed of a high density of organic matter that encourages growth of biofilm.

The biofiltration systems most effectively upgrade low and high concentrations of hydrogen sulphide, 50-100ppm to 2000-4000ppm, with a removal efficiency of 89-99.9 percent at 20125 g H 2S /m 3/h. Most bacteria proliferate optimally in a pH of 7 and at 35 o C (Zabava et al., 2019).

2.7 Water Scrubbing Process

The process entails the use of water as a medium of eliminating contaminants or undesired gasses in biogas or any other gas (Nock et al., 2014). It entails the passage of the biogas stream by water or a water solution to dissolve the impurities. The impurities are captured by the water through which they can be removed off the gas stream (Ofori-Boateng and Kwofie, 2009).

Water scrubbing process is founded on the principle of mass transfer where the impurities available in the biogas are transferred to the liquid due to the difference in the concentration gradient. These contaminants dissolve or chemically react with the water and are therefore eliminated out of the biogas. The scrubbing process occurs in the scrubber. The figure 2

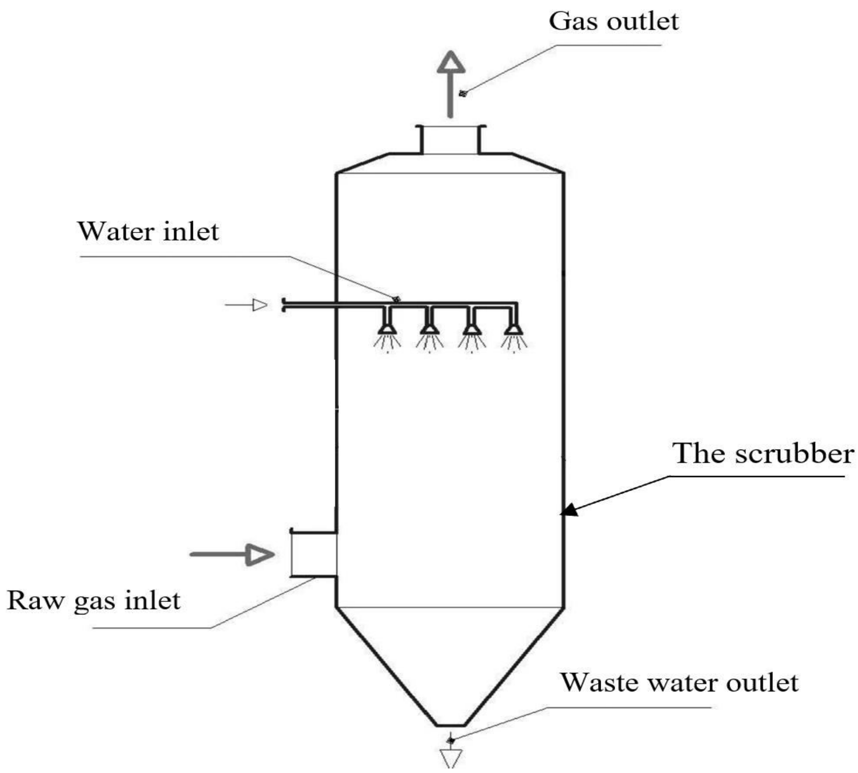


Figure 2.1: A schematic diagram of a water scrubber

The water scrubbing process involves the following steps;

a) Gas-Liquid Contact:

In this phase, raw biogas is fed to a scrubber whereby it is combined with a water stream flowing atop the water scrubber. The scrubber can be packed or it can be trayed with a specified media to ensure that there is maximum surface area between the gases and liquids.

b) Absorption of Impurities:

The biogas as it ascends up the scrubber tower goes through the water stream. The water is an absorber, and it selectively picks up pollutants like H 2 S, NH 3, and VOCs. The absorption is realized through chemical reactions or solubility basing on the nature of the impurities.

c) Scrubbing Solution Regeneration: The stream of water that has taken up the impurities, scrubbing solution, gathers at the bottom of the scrubbing tower. The solution is then sent to the regeneration procedure where the impurities are eliminated off the water phase. Various methods have been used during the regeneration process including chemical treatment, biological processes or adsorption.

d) Biogas Upgrading:

During this stage, a refined biogas is accumulated to the upper part of the scrubber. In cases where a more refined form of gas is needed, e.g. internal combustion engine, other biogas upgrading methods, e.g. cryogenic separation, membrane filtration, and pressure swing adsorption, can be combined with it to increase the concentration of methane..

2.7.1 Parameters for Optimization of Biogas Purification.

The important factors which are to be optimized in the purification of biogas through water scrubbing are the following:

a) Scrubbing System Design:

i) Scrubber Tower Design.

The construction of the scrubbing tower plays a very important role in the efficiency of contacts and transfer of mass between gases and liquids. The tower height, packing material, and tray design are some of the factors that should be optimized to give the maximum surface area where the interaction will take place and increase the absorption.

ii) Gas and Liquid Flow Rates

Biogas and water flow rates in the scrubber tower are supposed to be optimized in order to attain optimal contact time and absorption efficiency. The rate of flow of the gas and liquid is adjusted in such a way that it balances the scrubbing rates and the pressure drops are minimized.

iii) Scrubbing Solution Distribution.

The scrubbing solution should be distributed properly all over the tower to ensure that biogas is in physical contact. Rationing of the distribution systems including the placement of nozzles, and controlling of the flow prevents the channeling effect, and increases the absorption efficiency.

b) Residence Time

This is the duration of the time that the gas is in contact with the scrubbing liquid. This plays a significant role in the purity of gas determination. Lien et al. (2014) found that the greater the time of contact, the greater the efficiency of the removal. A paper by Gantina et al. (2020) employed the retention time of 60 seconds and reached the high $CO2$ removal rate of 99.5 percent and the $CH4$ concentration increase of 38.18 percent.

c) Parameters of Scrubbing Solution:

i) pH Control

The scrubbing solution pH has the ability to affect the usage efficiency of particular contaminants. A change in the pH within an optimum range can facilitate the process of scrubbing gases such as hydrogen sulphide (H2S) and ammonia (NH3).

ii) Composition Scrubbing Solution.

The scrubbing solution may contain different additives or chemicals to improve the absorption performance or to attack a specific impurity. Better results can be achieved by optimizing the make up of the scrubbing solution, say by choice of solvents or reactants.

iii) Temperature of Scrubbing solution.

The absorption capacity and reaction kinetics are dependent on the temperature of the scrubbing solution. The optimum temperature level can help enhance the cleaning ability of the impurities.

d) Removal of impurities and regeneration of scrubbing solution:

i) Efficiency of Removal of an impurity.

Gas composition, contact time, and chemical reactions are some of the factors, which determine the effectiveness of the impurities removal in biogas. By maximizing these parameters, one can ensure that one gets efficient elimination of target impurities, including H2S, NH3, and VOCs.

ii) Regeneration of Scrubbing Solution.

The procedure involves optimization of the regeneration process of the scrubbing solution in order to reduce the amount of energy used and maximize on the recovery of the impurity. Purification system cost-efficiency can also be enhanced by investigating effective regeneration techniques like chemical treatment, biological treatment, or adsorption.

e) Monitoring and Control:

i) Process Monitoring

This includes real-time monitoring of essential parameters including gas makeup, pressure dropped, temperature and flow rates which allow initiation of control and optimization of purification systems. This is achievable by use of sensor technology and automated control.

ii) Process Control

Using more sophisticated control methods, like feedback control loops, will assist in holding optimal conditions and changing the parameters in real time depending on the conditions on the process and the level of impurity. This will provide efficiency and uniformity in the working of the water scrubbing system.

f) Cost Optimization:

i) Energy Efficiency

Operational costs can be minimized by optimizing the use of energy in the purification system (reducing the amount of power needed to operate the pumps, or recovering the heat used in the regeneration process), and enhance sustainability.

ii) Scrubbing Solution Management.

Water can be minimized with the help of efficient management of the scrubbing solution including the circulation of the scrubbing solution, water needed to make it up, and the disposal of the spent solution.

In a study conducted by Vijay et al. (2006), the carbon dioxide in raw biogas was removed by using the water scrubbing method. The experimental system will be made up of the following devices: gas storage cylinder, three stage high pressure compressor, a water supply system, a gas supply system and a scrubbing column. The experiment was conducted with the pressure of 1.0 MPa and varying water and gas flow rates of 1.8 m3/hr to 2.0 m3/hr and 1.0 m3/hr to 3.0 m3 to the atmosphere respectively.

It was discovered in the study that gas and water flow rates influence the percentage of carbon dioxide absorption; the highest rate of removing carbon dioxide was 98.62 percent at water and gas flow rate of 1.8 m3 /hr and 1.5 m3 /hr respectively.

2.7.2 Carbon Dioxide Removal

According to Grando *et al.* (2017), Carbon dioxide is fairly soluble in water. Over 99% exists as dissolved gas while less than 1% is present in the form of carbonic acid (H_2CO_3), which undergoes partial dissociation to form H^+, HCO^-_3, and CO_3^{-2}. Upon reaction with water, Carbon dioxide forms carbonic acid. Water and carbon dioxide undergo chemical reactions as described by the equations below;

$$CO_{2(g)} + H_2O_{(l)} \rightleftharpoons H^+_{(aq)} + HCO^-_{3\,(aq)} \quad (2.5)$$

First, gaseous carbon dioxide dissolves into water

$$CO_2\,(g) \rightleftharpoons CO_{2(g)} \quad (2.6)$$

Carbon dioxide dissolves, forming carbonic acid as per equation 2.7 below;

$$CO_{2(g)} + H_2O_{(l)} \rightleftharpoons H_2CO^-_{3(aq)} \quad (2.7)$$

The carbonate deprotonates in solutions with a neutral pH to form bicarbonate.

$$H_2CO_{3(aq)} + H_2O_{(l)} \rightleftharpoons HCO^-_{3(aq)} + H_3O^+_{(aq)} \quad (2.8)$$

2.7.3 Removal of Hydrogen Sulphide

Hydrogen sulphide is an extremely harmful gas and causes corrosion of system components such as pipes and other parts. The hydrogen sulphide percentage composition in biogas may change depending on the type of raw materials used to produce biogas (Al Mamun & Torii, 2015).

To prevent harm to human health and downstream equipment and processes, H_2S in biogas must be reduced to a safe level (Moghadam & Banaei, 2022). Hydrogen Sulphide is the predominant contaminant in biogas, and its quantity varies from 100 to 10,000 ppm, depending on the kind of raw materials used in the biogas generation process. Consequently, it should be segregated before it is utilized since it is highly corrosive to pumps, pipes, and engine parts

The process of absorption of H_2S in the water scrubber is described by the following chemical reaction equation;

$$\boldsymbol{H_2S_{(g)} + 4H_2O_{(l)} \rightarrow H_2SO_{4\,(aq)} + 4H_{2(g)}} \quad (2.9)$$

$$\boldsymbol{H^+{}_{(aq)} + HO^-{}_{(aq)} \rightleftharpoons H_2O_{(l)}} \quad (2.10)$$

$$2H^+{}_{(aq)} + S^{2-}{}_{(aq)} \rightleftharpoons 2HS_{(l)} \quad (2.11)$$

2.7.4 Removal of Water Vapour

The biogas that is formed after decomposing various organic materials is usually wetted with water vapour (Persson et al., 2006). When this vapour condenses it leads to water or ice that further causes corrosion and blockages of parts of the system. Moreover, water makes available a medium through which Hydrogen Sulphide can be dissolved to form corrosive sulphuric acid. The elimination of water vapour is a prerequisite because in the majority of biogas applications, the gas needed is of high quality and relatively dry. Water vapour remover prevents the occurrence of clogging issues, corrosive acid, and ice during low temperatures (Karne et al., 2023).

A number of processes have been used to remove water vapour in biogas. They include;

a) Passive Cooling

This is done by applying biogas in underground pipes over a brief duration. Once the biogas cools down, the water separates out of the biogas and is either recycled into the system or released to the sewer.

b) Refrigeration and Pressurization.

This technique involves heat exchangers that cool down biogas with an intention of condensing the water vapour present in it. Biogas is subsequently pressed to get drier.

c) Absorption

This involves drying biogas using a drying agent like silica gel, aluminium oxide, glycol, and hygroscopic salts among others. The drying of such media at high pressure and temperatures is used to regenerate them. After some time, the drying medium must be changed.

2.8 Solubility of Gases in Water

Equation 2.12 illustrates Henry's law, which states that the saturated solubility of a gas in a liquid is directly proportional to the partial pressure of the gas, governing the solubility of gases in liquids at low pressures (Pruteanu *et al.*, 2017). However, the majority of gases deviate from this perfect behaviour in a sublinear manner, stabilizing at a pressure ranging between 0.1 to 0.5 GPa with a solubility of less than 1-mole percent (mol %). However, this is contrary to the fact that a rise in solubility of simple gases in water at high temperatures is linked to the critical point (647 K and 212 bar).

2.8.1 Solubility of Carbon Dioxide in Water

The solubility of CO_2 in aqueous solutions, including water, has been extensively investigated by employing a variety of instruments and methodologies (Cozma *et al.*, 2013; Ferrentino *et al.*, 2010). Despite the notable discrepancies identified in the database, as articulated by Diamond and Akinfiev (2003), investigations assessed both credible and unreliable experimental data that had been documented in the scholarly discourse. Comprehensive bibliographic studies concerning the solubility of CO_2 indicate that the solubility of CO_2 is enhanced by increasing pressure while lowering temperature (Calix *et al.*, 2008).

According to Olugasa and Oyesile (2015), the solubility of CO_2 is determined using Henry's Law. The carbon dioxide solubility in water at a pressure of 1 bar and a temperature of 298K is reported to be 2857 Pa.m^3/mol.

Henry's Law:

$$P_i = K_H C_{max} \tag{2.12}$$

where;

C_{max} - Saturation concentration of CO_2 in mol/m^3

K_H - Henry's Coefficient [Pa.m^3/mol] = 2857 Pa.m^3/mol

P_i - Partial pressure of the CO_2 component in biogas

Olugasa and Oyesile (2015) noted that Henry's coefficient for Methane is highest compared to the values of carbon dioxide and hydrogen sulphide, which are soluble. Henry's coefficient gives the representation of a gas's solubility in water. The higher the value of the constant, the lower the solubility, while a lower value signifies an increase in solubility, as shown by Equation 2.12.

Abas and Khan (2014) described the solubility of Carbon dioxide in water at a partial pressure of 101.325 kPa as shown in Figure 2.2

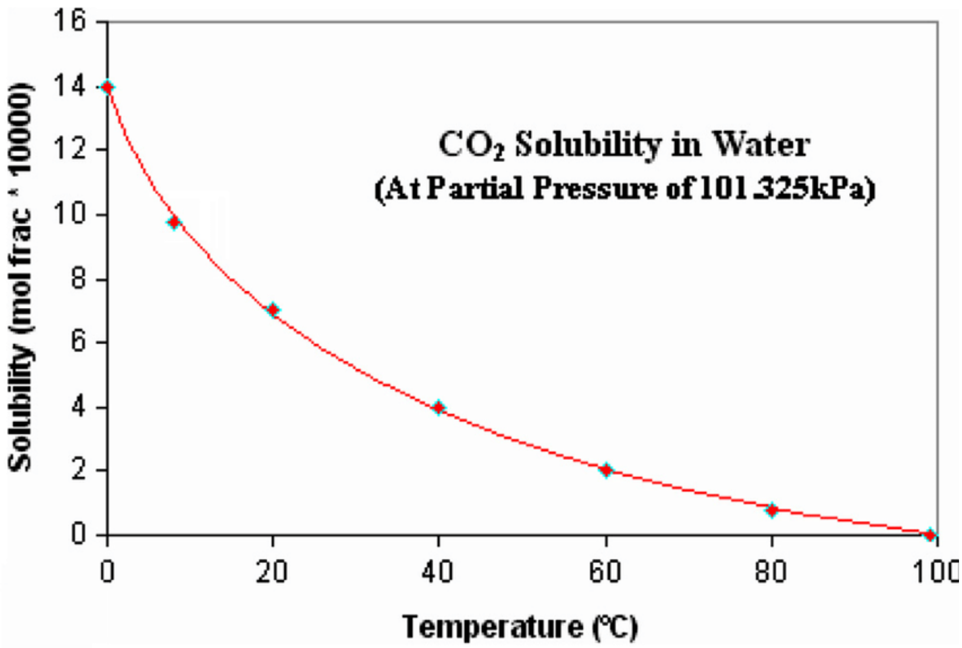


Figure 2.2: Solubility of Carbon dioxide in water

2.8.2 Solubility of Methane in Water

The solubility of methane from the gaseous state decreases with an elevation in temperature, indicating that the associated thermodynamic process is exothermic. In contrast, the solubility of methane in the hydrate phase rises as the temperature increases, implying that this process is endothermic (Grabowska *et al.*, 2022). This is demonstrated in Figure 2.3 as discussed by Velasco *et al*. (2018).

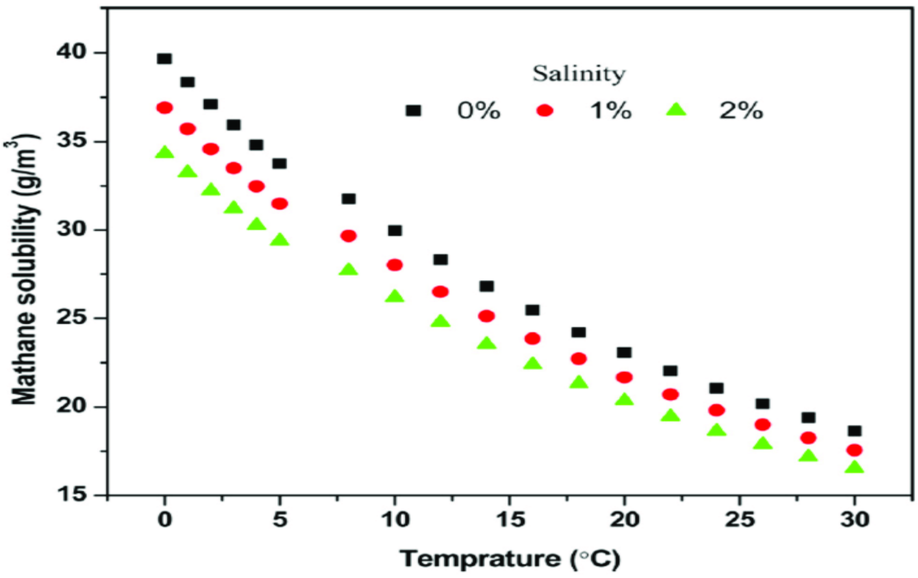


Figure 2.3: Solubility of Methane in Water

2.8.3 Solubility of Hydrogen Sulphide in Water

According to Jiang *et al.* (2020), Hydrogen sulfide (H_2S) exhibits a high degree of solubility in water, and upon its dissolution in water, an ionization equilibrium is attained that encompasses the following chemical processes; the dissociation of the molecular H_2S leading to the formation hydrosulphide ion (HS^-), the further dissociation of the hydrosulphide ion resulting in the production of sulphide ion (S^{-2}), and the ionization of water.

The chemical equations below illustrate this phenomenon;

$$H_2S_{(g)} \leftrightharpoons HS_{(aq)}^{-} + H^{+}_{(aq)} \tag{2.13}$$

$$HS^{-} \leftrightharpoons S^{2-}_{(aq)} + H^{+}_{(aq)} \tag{2.14}$$

$$H_2O_{(l)} \leftrightharpoons H^{+}_{(aq)} + HO^{-}_{(aq)} \tag{2.15}$$

Figure 2.4 shows the solubility of Hydrogen sulphide in water as discussed by Abas and Khan (2014).

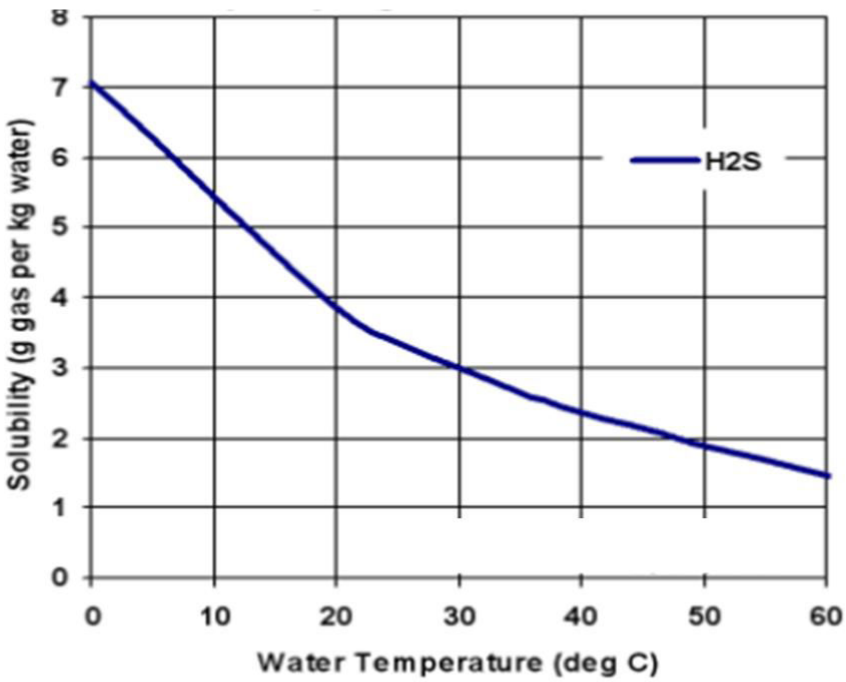


Figure 2.4: Solubility of Hydrogen sulphide in water

2.9 A Critical Review of Biogas Purification Optimization Techniques

Several techniques have been used to determine the optimal process parameters of a given system, as discussed by Zabava *et al.* (2018), such as the Taguchi Method and Response Surface Methodology (RSM).

2.9.1 Taguchi Method (TM)

The Taguchi method is defined as an optimization technique that utilizes the design of experiments to identify the best combination of parameters using orthogonal arrays and evaluates the signal-to-noise ratio to determine optimal levels. primarily applied in product development and industrial engineering (Canel & Baglan, 2023). TM is a powerful technique used to analyze the effects of the process parameters. Taguchi's technique of parametric optimization provides a systematic and efficient method for determining near-optimum design parameters for performance and cost. The objective is to select the best combination of control parameters so that the product or process is most robust with respect to noise factors (Li, Cheng, Hong, Lan, & Yin, 2020).

According to Mohamed, Alaa El Mokadem, and Osman (2022), the Taguchi method utilizes orthogonal arrays from the design of experiments to study a large number of variables with a small number of experiments. Using orthogonal arrays significantly reduces the number of experimental configurations to be studied.

TM offers several benefits, including considerable time and resource savings, determination of important factors affecting operation, performance, and cost, and quantitative recommendations for design parameters that achieve the lowest cost, high-quality solutions. However, it has several shortcomings, such as requiring a high level of expertise and knowledge in designing and analyzing experiments, which may not be available or affordable for medium and small-scale operators. Besides, it does not capture all the relevant factors or interactions that affect the quality of the process, especially if they are nonlinear, dynamic, or complex (Hisam *et al*., 2024).

2.9.2 Analysis of Variance (ANOVA)

Analysis of variance is a statistical measure that is utilized in the comparison of the means of two or more groups by examining the variance. It determines whether the differences that were observed between groups are statistically significant or the differences were randomly brought by chance. ANOVA does it by evaluating the difference between groups relative to the one within groups through an F-test, when the variation between-groups is found significantly greater than the variability within-groups, the means are differentiated (Mannan Bhuyean, 2025).

There are several parameters used in the ANOVA, which provide information about the variables and their responses, as well as their correlation. These parameters include: F-value, P-value, Adjusted R^2, Predicted $R^{2,}$ Adequate precision, mean, standard deviation, and the percentage of coefficient of variation. The p-value is critical in identifying the statistical significance of a given factor in relation to the chosen confidence interval.

R^2 represents the coefficient of determination of the linear regression model. It measures the variability of the actual response that can be defined by the corresponding independent factors (Mukhopadhyay *et al.*, 2013a). The predicted R^2 of 0.0936 indicated reasonable agreement with the Adjusted R^2 of 0.2978. The threshold is that the disparity between the adjusted R^2 and the predicted R^2 should be about 0.2 (P. Subha and Jayaraj, 2019).

The coefficient of variation percentage (C.V. %) is a measure of the amount of variation that the data has in relation to the mean size; the larger the value of CV the less reliable the experiment is (Šumica et al., 2016). In general, a model is reasonable to be reproducible in the case when CV does not exceed 10% (Rasouli et al., 2015).

Adequate precision, known as signal-to-noise (S/N) ratio is a value that is used to measure the quality of the experiment. The aim is to maximize the S/N ratio and hence reduce the variability (noise) whilst maintaining desired outcome (signal) within desired range.

2.9.3 Response Surface Methodology (RSM)

It is a statistical method of determining the relationships between two or more inputs/ factors (independent variables) on the one hand, and outputs/responses (dependent variables) on the other hand. As Okwunodulu et al. (2022) present, RSM is an empirical model that involves the use of statistical and mathematical methods to develop a correlation between inputs and outputs. It has certain advantages over other approaches that could be used such as theoretical models which are prevalently described as cumbersome in their application, requiring much time, inefficient, prone to errors, and unreliable. Even though such methodology is only an approximation, it is widely applicable as it is relatively easy to estimate and apply even in situations where there is limited knowledge about the process.

The optimal yield of a given output has been provided as a result of the optimization of working parameters by use of statistical techniques, including Response Surface Methodology (Karmoker et al., 2019). Unlike the traditional methods, RSM may be applied to identify the presence of any relationship between variables (Asadi and Zilouei, 2017). Response Surface Methodology (RSM) resembles a Fractional Factorial design or a Factorial experiment, and may be implemented on the response variable or variables of interest in order to optimize or attain a certain target (Bezerra et al., 2019).

As Karmoker et al. (2019) state, the major characteristics of the RSM are:

i. Orthogonality: It is a property which ensures that the effects of the k-factors can be estimated independently with little or no confounding. Besides, the orthogonality property provides estimates of the model coefficient that have the least variance, and they are not correlated..

ii. Rotatability: This is a property of rotating points of the design about the centre of the factor space. The moments of the distribution of the design points are constant. The concept of replication is taken into account.

iii. Uniformity: Central Composite Design (CCD) is used to manage the number of centre points with uniform precision. More runs are made at the centre of the dominant space.

RSM consists of two large designs that include central Composite Design (CCD) and Box-Behnken Design (BBD). BBD is an experimental design that is applied to examine each factor at one of three equal levels, typically coded as -1, 0, or +1; the exact response must have at least three levels (Buenaño et al., 2024). BBD is considered more competent and robust than other designs like three-level full factorial design, Central Composite Design (CCD), and Doehlert design even when it does not cover corners in the nonlinear design space adequately (Karmoker et al., 2019). The Central Composite Design (CCD) is an experimental design applied in RSM to approximate a second-order (quadratic) model of a specific response variable without having to perform a three-level factorial experiment (Asadi and Zilouei, 2017). The coded variables are usually utilized during the formulation of such an experimental design as is the case with the Box-Behnken Design (BBD). The design will have three sets of experimental runs (Bhattacharya, 2021) as follows:

i. Factorial points (controls) onto which the experiment is anchored.

ii. A set of center points, which are the experimental trials in which the values of each variable correspond to the medians of the values used in the factorial segment. This particular point is frequently replicated to enhance the precision of the experiment.

iii. A set of axial points, which are experimental trials identical to the central points except for a single factor, which assumes values both below and above the median of the two factorial levels, and generally extend beyond their respective range. All variables are manipulated in this way.

RSM, as an Optimal design, offers three advantages over sub-optimal experimental designs (Kannan and Shanbhag, 2019; Kono *et al.*, 2018):

i. Optimal designs lower experimentation costs by allowing statistical models to be estimated with fewer experimental runs.

ii. Optimal experimental designs are capable of integrating various categories of factors, which include process variables, mixture components, and discrete factors.

iii. Optimization of designs can be achieved when the design space is constrained, for instance, when the mathematical process space includes factor settings that are not practically feasible, such as a result of safety considerations.

From the above discussion, comparing the RSM and the Taguchi optimization methods, it is evident that RSM offers several advantages. This is because RSM, in contrast to the traditional methods, can be utilized to determine if there is any relationship between variables (Asadi and Zilouei, 2017). Besides, RSM can be used to do both mathematical modelling and statistical analysis at the same time and reveal the correlation between several control variables.

2.10 Research Gap

Based on observation and review of the literature on the current biogas purification techniques, it can be inferred that Pressure swing adsorption (PSA) requires a highly skilled operator and a lot of maintenance. On the other hand, a membrane separation system is costly and operates nearly at atmospheric pressure; fouling compromises its robustness. Despite being portable, the cryogenic process uses a lot of energy because it needs to maintain a CO_2 desublimation temperature of 194.5 K (-78.5 ^{o}C). This requirement necessitates the use of efficient condensers or heat exchangers, which may add to the system's complexity. The absorption method (water scrubbing) is the most used technique for purifying biogas because of the availability of water, simplicity, and environmental friendliness. However, optimization of biogas purification by this method is needed to improve system efficiency and effectiveness. It is not all about removing the contaminants, but also ensuring optimal biogas quality. Despite water scrubbing being the most frequently used purification technique little work has been done in Kenya on the optimization of the system especially for small-scale applications. The study, therefore, sought to determine the optimal process parameters required to ensure an optimal biogas purification process using water scrubbing technology.

CHAPTER THREE

3. MATERIALS AND METHODS

3.1 Materials

The following materials were utilized during stages of biogas sampling, data collection, process optimization, and gas analysis.

Materials for the Purification Process are given in Table 3.1.

Table 3.1: Materials for the purification process

S/No	Material Description	Purpose
1	Distilled water	To absorb the carbon dioxide and Hydrogen sulfide
2	Peristaltic pump	To record the water flow rate.
3	Gas flow meter	To record the flow rate of biogas
4	20 L and 5L Sampling bag	For the collection of biogas samples for the analysis of various parameters.
5	Pressure gauge	To record the pressure.
6	Storage bag/Gas sampling bag	For storing purified biogas
7	SKY2000-M4-WH multi-gas detector	For analyzing the gas composition
8	Delivery pipes	For the delivery of both biogas and water
9	20-liter water tank	For holding water before it flows to the scrubber
10	U-tube manometer	Determination of scrubber pressure
11	Stop Watch	For recording time
12.	Scrubber Column	The process chamber

The raw biogas used in the study was produced from municipal waste, wastes from hotel remains, water hyacinth, and wastes from fish processing on the shows of Lake Victoria at Dunga beach in Kisumu County. These materials were chopped into finer pieces and fed into the digester as the feedstock. The photograph below shows the feedstock used.

Photograph 3.1: Biogas feedstock

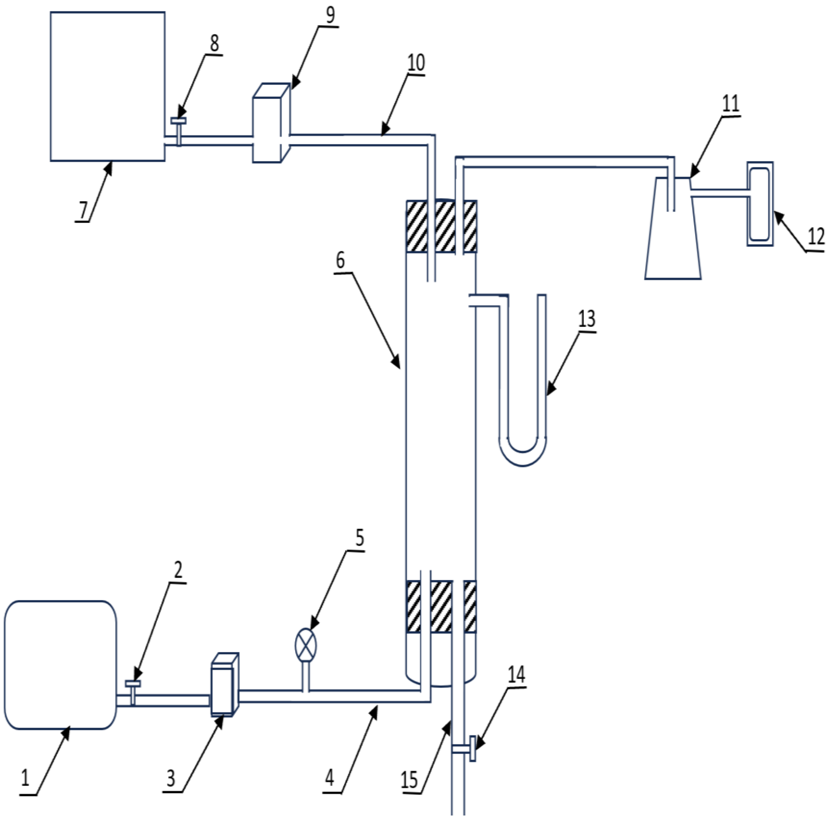
3.2 Methods

3.2.1 Experimental Setup

The process of optimizing biogas purification was designed to purify the raw biogas by eliminating incombustible gases from biogas mixtures, specifically carbon dioxide, and H_2S, using water scrubbing technology. The study was carried out using a water scrubber. The technical specifications and physical dimensions of the water scrubber were delineated as follows:

- Length of the column: 50 cm
- Diameter of the column: 10 cm.
- Composition of water Scrubber: Plastic material

The schematic diagram of the Experimental Setup is shown in Figure 3.3



1. Raw Biogas sampling Bag
2. Non return valve
3. Gas pump
4. Biogas inlet pipe
5. Gas flow meter
6. Scrubbing tower
7. Water reservoir
8. Valve
9. Peristatic pump
10. Water delivery pipe
11. Purified sampling bag
12. Gas Analyzer
13. U-tube manometer
14. Valve
15. Waste water outlet

Figure 3.1: Schematic diagram of the Experimental Setup

The experimental setup consisted of three sections or stages: Raw biogas collection and sampling, optimization scrubber, and collection unit. The first section consisted of raw gas sampling, analysis of raw gas composition, and a gas flow rate control meter. The second section involved the biogas purification process through the scrubber. Water was introduced into the scrubber through the water nozzle at the upper part of the scrubber, whereas biogas was supplied upwards from the base of the scrubber. This ensured the maximum gas-water contact for enhanced purification of biogas. H_2S and CO_2 dissolved in water were removed as wastewater through the exit pipe at the base of the scrubber. The methane-enriched biogas moved upwards due to its reduced density to the last stage. Finally, the third section involved the collection and sampling of the upgraded gas for composition analysis using the SKY2000-M4-WH multi-gas detector.

3.2.2 Design of Experiment

To achieve optimization, the parameters considered included: the rates of gas and water flow and the pressure of the scrubber, and the retention time of biogas in the scrubbing tower. The development of a matrix with the runs of experiments was done with the help of A Design of Experiment (DoE) and Central Composite Design (CCD) of Response Surface Methodology (RSM) (Asadi and Zilouei, 2017). The experiment was designed using the Design Expert version 13 software. Each run was collected with the help of upgraded biogas and the composition was analysed with the help of multi-gas detector SKY2000-M4-WH (Al Mamun and Torii, 2015).

3.2.3 Experimental Procedure

The process parameters that were used in determining the optimum biogas purity were: gas flow rate (GFR), water flow rate (WFR), pressure (P) in scrubber and gas retention time (RT) in scrubber.

Control Experiment: The biogas purification optimization system was operating with the following settings; WFR = 4.5 litres per hour, GFR = 8.5 litres per minute, RT = 60 seconds and P = 0.06 bar.

The experiment of constructing a matrix of experimental trials consisted of the adjustment of the parameters of the control facility based on CCD (Bezerra et al., 2019) as presented in Table 3.2.

Table 3.2: Variation of parameters

Factor	Name	Units	Lowest star point	Highest star point	Coded		
					Low (-1)	Mean (0)	High (+1)
A	WFR	l/h	3.50	5.50	4.00	4.50	5.00
B	GFR	l/min	7.50	9.50	8.00	8.50	9.00
C	RT	sec	30.00	90.00	45.00	60.00	75.00
D	P	bar	0.04	0.08	0.05	0.06	0.07

The star points are the minimum and maximum values that lie outside the given boundaries of low and high values, respectively (Li, *et al*., 2021).

Raw biogas was collected from Biogas International Co. Ltd and directly tested for the composition of CH_4, CO_2, and H_2S using a SKY2000-M4-WH multi-gas detector. The peristaltic pump was switched on to permit water into the scrubber to test for leakages. The gas pump was then switched on, and pressurized biogas was pumped up the scrubber to the top section, where it exited through the gas outlet. Water molecules mixed with the gas particles within the scrubber. CO_2 and H_2S, being soluble, dissolve and flow through the outlet pipe while the insoluble methane exits via the gas outlet at the top.

Optimization of process parameters was done by varying the water and gas flow rates, pressure, and gas retention time, as shown in Table 3.2. A set of 30 trials was experimented with, as illustrated in Table 3.3, to achieve the highest methane value in the upgraded biogas. The upgraded biogas was tested for every set, and the combination of parameters that gave the highest methane content was considered to be the optimal set of parameters that maximized methane quality.

According to Derringer (2016), the CCD developed the matrix shown in Table 3.3. The experiments were done based on this narrative.

Table 3.3: DoE Matrix

Run	WFR (l/h)	GFR (l/min	RT (sec)	P (bar)
1	4.5	8.5	60	0.06
2	4.5	8.5	60	0.06
3	4.5	8.5	60	0.06
4	3.5	8.5	60	0.06
5	5.0	9.0	45	0.05
6	4.5	8.5	60	0.06
7	4.5	8.5	60	0.08
8	4.5	8.5	30	0.06
9	4.0	9.0	45	0.05
10	4.0	8.0	45	0.05
11	5.0	9.0	75	0.05
12	4.5	7.5	60	0.06
13	5.5	8.5	60	0.06
14	5.0	8.0	45	0.07
15	4.5	8.5	90	0.06
16	5.0	8.0	75	0.07
17	4.0	9.0	45	0.07
18	4.5	8.5	60	0.04
19	4.0	9.0	75	0.05
20	4.0	8.0	75	0.07
21	4.0	9.0	75	0.07
22	4.5	8.5	60	0.06
23	4.0	8.0	45	0.07
24	4.5	9.5	60	0.06
25	5.0	8.0	45	0.05
26	4.5	8.5	60	0.06
27	5.0	9.0	75	0.07

28	5.0	9.0	45	0.07
29	4.0	8.0	75	0.05
30	5.0	8.0	75	0.05

3.2.4 Analytical Methods

The gas composition was analyzed offline by the SKY2000-M4-WH multi-gas detector. The SKY2000-M4-WH multi-gas detector was fitted directly to the gas delivery pipe from the scrubber to record the gas composition.

Photograph 3.2: SKY2000-M4-WH multi-gas detector; manufactured by Shenzhen Yuan Technology Co. Ltd

The rate of gas and water flow, scrubber pressure, and gas retention period were measured using gas and water flow meters, a U-tube manometer, a stopwatch, and a SKY2000-M4-WH multi-gas detector, respectively (Al Mamun and Torii, 2015).

3.3 Data Collection and Analysis

3.3.1 Data Collection

This process involved recording various parameters (as given in Table 3.3) in question with the aid of sensors, flow meters, gauges, and software. The raw biogas was sampled from the Biogas International Company Ltd and transported to the study area using 20 Litre sampling bags.

3.3.2 Data Analysis

a) Predictive Mathematical Modelling

The Linear regression was applied to ascertain the relationship between the independent and dependent variables (Urdampilleta *et al.*, 2023). The linearity assumption was addressed by Pearson Correlation Analysis. Normality was tested by the use of Skewness and Kurtosis.

The research adopted an ordinary least square regression model (Jhang *et al.*, 2004) as expressed below;

$$\boldsymbol{Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \varepsilon} \tag{3.1}$$

where;

Y = Dependent Variable (Quality of biogas)

x_1 = water flow rate

x_2 = biogas flow rate

x_3 = the gas retention time in the scrubber

x_4 = the pressure of the water jet

β_0 = the y-intercept

β_1, β_2, β_3, and β_4 are the regression coefficients

ε = Error term

b) Gas Composition Analysis

The gas composition was analyzed offline by the SKY2000-M4-WH multi-gas detector. This included the analysis of the composition of the raw biogas and purified biogas samples. The analysis of the results involved the following:

i) Determination of percentage CO_2 removal

The percentage of carbon dioxide removed was calculated as per the equation below;

$$\boldsymbol{C}_{\%} = \frac{\boldsymbol{y_P - y_r}}{\boldsymbol{y_r}} \tag{3.2}$$

where;

$C_{\%}$ ….. the percentage of carbon dioxide removed

y_P ..… the content of carbon dioxide in the upgraded biogas

y_r ..… the content of carbon dioxide in the raw biogas

ii) Determination of Methane enrichment

$$\boldsymbol{M}_{\%} = \frac{\boldsymbol{C_P - C_r}}{\boldsymbol{C_r}} \tag{3.3}$$

where;

$M_{\%}$ ….. the percentage of methane-enriched.

C_P ..… the content of methane in the upgraded gas.

C_r ..… the content of methane in the raw biogas.

iii) Determination of the Percentage removal of H_2S

$$\boldsymbol{H}_{\%} = \frac{\boldsymbol{h_P - h_r}}{\boldsymbol{h_r}} \qquad (3.4)$$

where;

$H_{\%}$ ….. the percentage of H_2S removed.

h_P ..… Hydrogen Sulphide content in processed gas.

h_r ..… Hydrogen Sulphide content in the raw gas.

c) Optimization using Response Surface Methodology

The optimization of purification parameters using RSM was achieved in two phases. The first phase involved designing an experiment using CCD. The Central Composite Design was employed to develop the experimental matrix, comprising thirty runs, which were used to determine the responses (Almeida *et al*., 2025).

The second phase involved the development of surface interaction graphs for each response (CH_4, CO_2, and H_2S composition). These graphs related two-factor interactions against the output of CH_4, CO_2, and H_2S composition, both two and three-dimensional. The two-dimensional contours and three-dimensional surface plots related to water flow rate and gas flow rate for the optimal responses are highlighted (Lamidi *et al*., 2022). For the 3D surface graphs, the response is shown on the vertical axis, while the two input factors are on the horizontal axes. They provide a visual representation of the relationship between the factors and the response (Lamidi *et al*., 2022). The optimal value for the input factors is determined at the point where the contour plots overlap or intersect (Yacout *et al*., 2025).

CHAPTER FOUR

4. RESULTS AND DISCUSSION

4.1 Introduction

The section provides the description and discussion of the obtained results, their implications, and their relevance to the study. It involves a statistical examination of the findings and the graphical presentation of the findings.

The table 4.1 depicts the experimental results that were obtained.

Table 4.1: Results of the Experiment.

Run	WFR (l/h)	GFR (l/min	RT (sec)	P (bar)	CH_4 (% Volume)	CO_2 (% Volume)	H_2S (mg/m^3)
1	4.5	8.5	60	0.06	78.06	17.32	11
2	4.5	8.5	60	0.06	78.23	17.40	11
3	4.5	8.5	60	0.06	77.82	17.26	11
4	3.5	8.5	60	0.06	76.54	17.82	12
5	5.0	9.0	45	0.05	72.94	19.24	15
6	4.5	8.5	60	0.06	81.07	17.16	11
7	4.5	8.5	60	0.08	78.12	17.04	11
8	4.5	8.5	30	0.06	74.38	18.03	14
9	4.0	9.0	45	0.05	78.02	16.28	10
10	4.0	8.0	45	0.05	81.36	15.02	10
11	5.0	9.0	75	0.05	86.36	12.05	9
12	4.5	8.5	60	0.06	80.66	15.76	10
13	5.5	8.5	60	0.06	83.65	16.22	10
14	5.0	8.0	45	0.07	86.14	14.73	10
15	4.5	8.5	90	0.06	78.02	17.29	11

16	5.0	8.0	75	0.07	91.45	6.91	0
17	4.0	9.0	45	0.07	82.85	14.60	8
18	4.5	8.5	60	0.04	71.68	21.52	18
19	4.0	9.0	75	0.05	82.69	17.22	11
20	4.0	8.0	75	0.07	89.82	10.12	7
21	4.0	9.0	75	0.07	86.20	13.68	9
22	4.5	8.5	60	0.06	79.01	17.35	11
23	4.0	8.0	45	0.07	84.63	15.84	10
24	4.5	9.5	60	0.06	78.47	17.24	11
25	5.0	8.0	45	0.05	83.67	16.25	10
26	4.5	8.5	60	0.06	84.52	15.41	10
27	5.0	9.0	75	0.07	88.63	10.95	8
28	5.0	9.0	45	0.07	83.84	15.94	17
29	4.0	8.0	75	0.05	85.28	14.58	10
30	5.0	8.0	75	0.05	87.12	11.82	9

4.2 Methane Maximization

Graphical representation, statistical analysis, diagnostics, and response surfaces are discussed.

4.2.1 Graphical Presentation

The highest percentage of purification was 91.45 percent, which is obtained when Table 4.1, run 16, with the experiment set as follows: a water flow rate (WFR) of 5.0 litres per hour, a gas flow rate (GFR) of 8.0 litres per minute, a retention time (RT) of 75 seconds, and pressure (P) of 0.07 bar. WFR of 4.5 litres per hour, GFR of 8.5 litre per minute, an RT of 60 seconds and P of 0.04 bar produced 71.68 percent of Methane. Figure 4.1 below provides a graphical representation..

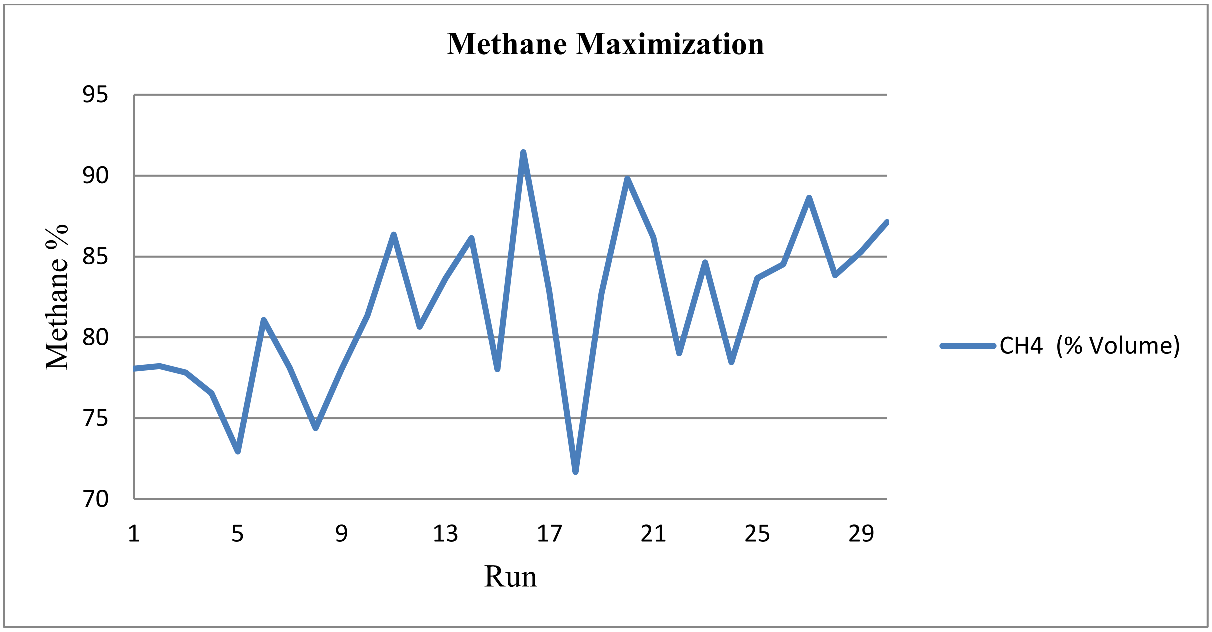


Figure 4.1: Methane Maximization

Run 12 was the control; it was characterized by a WFR of 4.5 litres per hour, GFR of 8.5 litres per minute, RT of 60 seconds and P of 0.06 bar. It gave a purification of 80.66%. This was an improvement of 10.79 percent to run 16.

Srichat et al. (2017) designed a biogas purification system based on a chemical scrubbing method: the flow rates of the biogas employed in the experimental process were 0.3 m3 /hour, 0.6 m3 /hour and 0.9 m3/ hour, and the flow rates of the solution were equal to 0.6 m3 /hour, 1.2 m3 /hour and 1.8 m3/hour. The experiment was performed on biogas with an initial composition of 51% methane. The maximum methane enhancement of 81.1% and 89.3% was recorded with calcium hydroxide having a concentration of 0.1 and 0.2 mol of biogas, and calcium hydroxide solutions flow rates of 0.3 m^3 /hour and 1.8 m^3 /hour, respectively.

Akila *et al.* (2017) studied the enrichment of CH_4 in biogas using the Pressure Swing Adsorption method. Raw biogas of 51 % methane was used. The study attained a maximum of 93.9% methane content at an optimal working pressure of 8 bar.

Vijay *et al.* (2006) observed that water scrubbing technology is a purification process that reduces the emission of harmful gases into the atmosphere, contributes to good air quality improvement, and has a low investment cost. This technology has a performance that is as good as the Chemical Scrubbing method and the Pressure Swing Adsorption method.

Mugagga *et al.* (2022) worked on the optimization and analysis of a Low-Pressure water scrubbing biogas upgrading system using the Taguchi and Response Surface Methodology approaches. In their work, they optimized water scrubbing at low pressure and achieved an optimum bio-methane concentration of 84.71% and 13.31% CO_2 reduction.

The purification percentages attained in the three outgoing observations compare very closely with what was achieved in this study. This study attained 91.45% methane and 6.91% carbon dioxide, which is higher than the work done by Mugagga *et al.* (2022) but slightly lower than the work done by Akila *et al.* (2017) who attained 93.9% methane, though at a high pressure of 8 bar.

4.2.2 Statistical Analysis

Design Expert Software (version 13), in which CCD of RSM was applied, gave the data that was used to do the statistical analysis. The probability that is less than 0.05 ($p < 0.05$), that is allowing an error of 5%, is significant.

a) Fit Summary

The Fit Summary is shown in Table 4.2

Table 4.2: Fit Summary for Methane

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2
Linear	0.0112	0.1223	0.2978	0.0936
2FI	0.9872	0.0702	0.1174	-0.7579
Quadratic	0.5830	0.0554	0.0650	-1.5766
Cubic	0.9858	0.0051	-0.6590	-49.7621

From Table 4.2, the sequential p-value of 0.0112 is significant since $p < 0.05$. A model F-value of 4.08 was established. This implies that there was a chance of less than 1.12% that a value this much could be caused by noise. Consequently, the linear model was selected and applied in this study. The two-factor-interaction (2FI), Quadratic, and Cubic models exhibit no statistical significance. and can't be used since they have $p > 0.05$.

b) Analysis of variance (ANOVA)

Table 4.3 shows the ANOVA.

Table 4.3: ANOVA for Linear Model of Methane

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	276.61	4	69.15	4.08	0.0112
A-WFR	23.05	1	23.05	1.36	0.2548
B-GFR	43.52	1	43.52	2.56	0.1218
C-RT	110.00	1	110.00	6.48	0.0174
D-P	100.04	1	100.04	5.90	0.0227
Residual	424.24	25	16.97		
Lack of Fit	390.31	20	19.52	2.88	0.1223
Pure Error	33.93	5	6.79		
Cor Total	700.85	29			

Other Statistics: Adjusted R^2 = 0.2978, Predicted R^2 = 0.0936, Adequate precision = 7.741, C.V. % = 5.04, Std. Dev. = 4.12, and Mean = 81.71. The percentage of the coefficient of variation (C.V. %) is computed as per equation 4.1.

$$C.V\ \% = \frac{\text{Std Dev}}{Mean}\ x\ 100 \tag{4.1}$$

The Analysis of Variance (ANOVA) Table 4.3 was employed to assess the statistical significance of the linear model (Nanduri *et al.*, 2008). A large F-value and a small P-value implied a more significant influence on the corresponding response variable (Quanhong and Caili, 2005). In this case, the linear effects of retention time (C), and pressure (D) were significant.

R^2 represents the coefficient of determination of the linear regression model. It measures the variability of the actual response that can be defined by the corresponding independent factors (Mukhopadhyay *et al.*, 2013a). The predicted R^2 of 0.0936 indicated reasonable agreement with the Adjusted R^2 of 0.2978. The threshold is that the disparity between the adjusted R^2 and the predicted R^2 should be about 0.2 (P. Subha and Jayaraj, 2019). The difference here was 0.2042. This showed a strong correlation between the observed and the predicted values.

The percentage of the coefficient of variation (C.V. %) serves as an indicator of the residual variation of the dataset relative to the size of the mean; the higher the value of CV, the lower the reliability of the experiment (Šumić *et al.*, 2016). A lower CV of 5.04% indicated a greater reliability of the experiment. As a general rule, a model can be considered reasonably reproducible if the CV is not greater than 10% (Rasouli *et al.*, 2015). Therefore, this model is reproducible.

Adequate precision is a measure of signal-to-noise ratio; a ratio greater than 4 is desirable (Mason *et al.*, 2003). In this case, the ratio was 7.741, which indicated an adequate signal. This model can therefore be used to navigate the design space.

From Table 4.3, the Lack-of-Fit (LoF) F-value of 2.88 had a p-value of 0.1223; it implied that it was not significant relative to the pure error. Non-significant lack of fit indicated a good fit of the model (Kang *et al.*, 2016). There was only a 12.23% chance that this magnitude of LoF F-value could occur due to noise. This is comparable to the work reported by Masinde *et al.* (2020). The model showed a standard deviation of 4.12, and a mean of 81.71.

4.2.3 Model Graphs for Methane

The two-dimensional contours and three-dimensional surface plots relating water flow rate and gas flow rate for the optimal responses are highlighted in Figures 4.2 and 4.3. The plots help visualize the relationship between the input variables and the responses.

The three-dimensional (3D) plots were produced by varying two variables within the experimental range, and maintaining the remaining two variables constant at their centre point values.

a) 2D Contour Plot

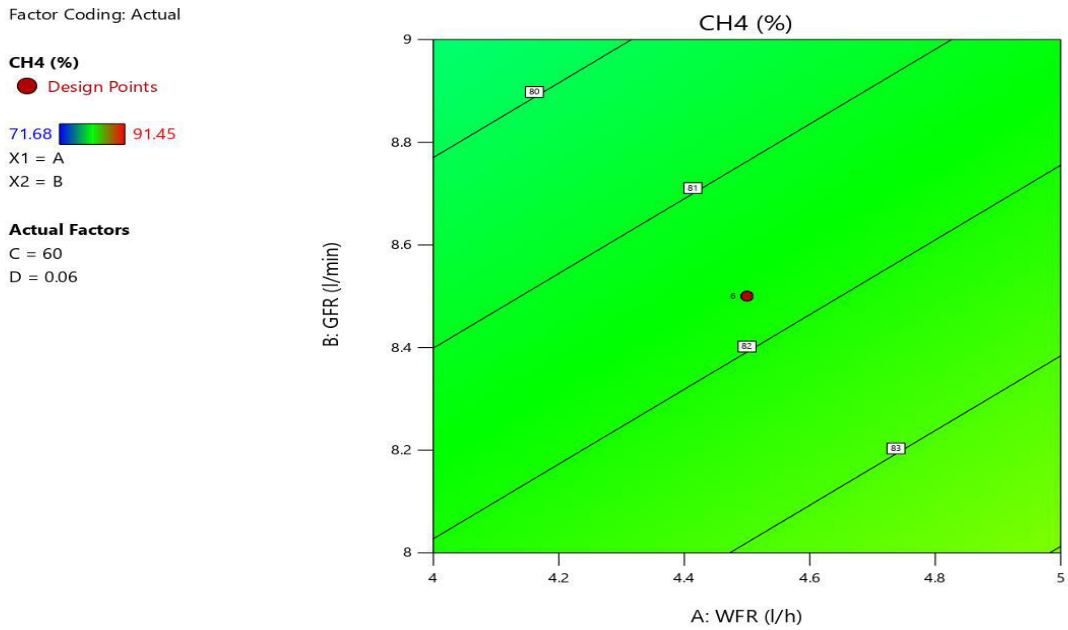


Figure 4.2: 2D Contour plot of WFR and GFR for CH_4

B) 3D Surface Plot

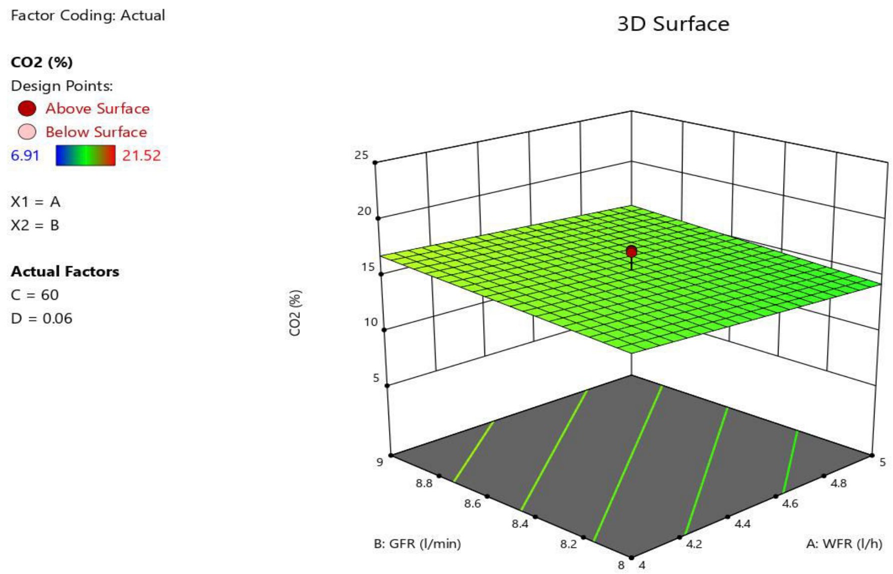


Figure 4.3: 3D surface plot of WFR and GFR for CH_4

4.3 Carbon Dioxide Minimization

4.3.1 Graphical Presentation

Table 4.1 shows that run 16 gave the highest minimization of Carbon Dioxide (CO_2) at 6.91%. The control experiment, represented by run 12, gave a CO_2 value of 15.76%. A graphical representation is shown in Figure 4.2.

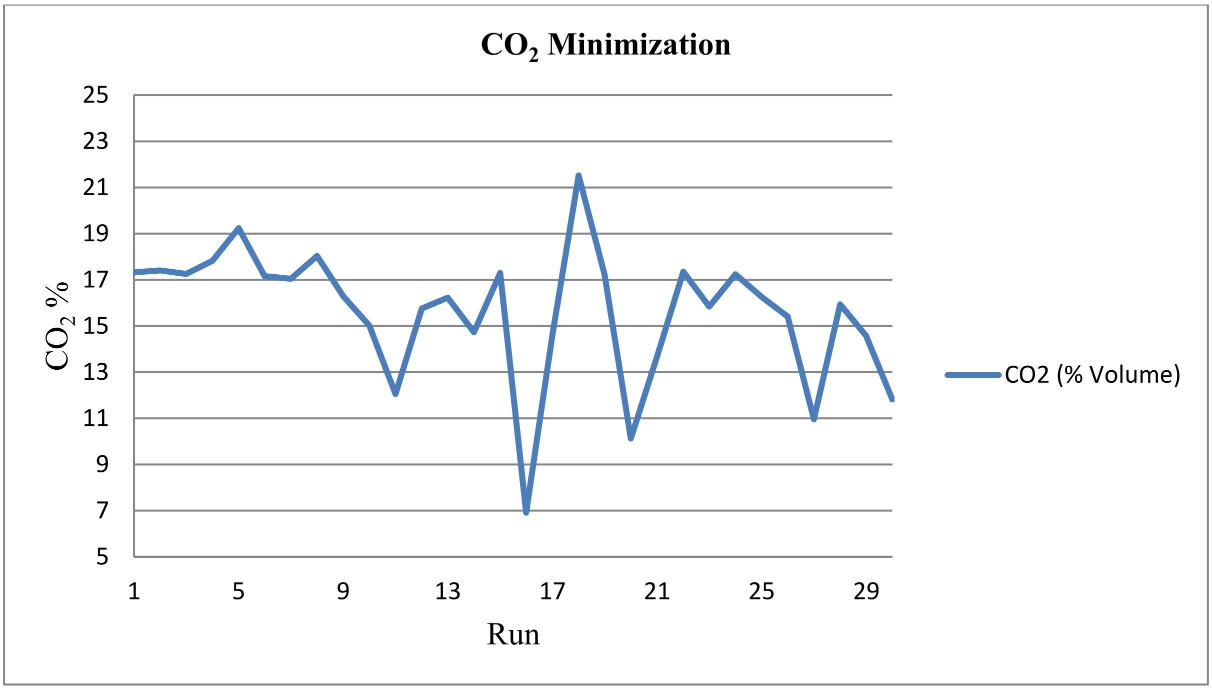


Figure 4.4: Carbon dioxide Minimization

The control experiment (run 12) reduced the CO_2 content to 15.76%, while run 16 gave the highest minimization of 6.91%. This showed an improved reduction of 8.85%.

Work done by Srichat *et al.* (2017), Akila *et al.* (2017), and Vrbova and Ciahotny (2017) concentrated on maximizing the CH_4 content; minimal attention was given to the CO_2 content. Andriani *et al.* (2014) recommended that water scrubbing is a better option for removing carbon dioxide when water is available in the required quantity.

4.3.2 Statistical Analysis

a) Fit Summary

Table 4.4 shows the Fit Summary. A sequential p-value of 0.0116 is significant since $p < 0.05$. The 2FI, Quadratic, and Cubic models are insignificant because their $p > 0.05$. Consequently, the linear regression model was selected.

Table 4.4: Fit Summary for Carbon dioxide

Source	Sequential p-value	Adjusted R^2	Predicted R^2
Linear	0.0116	0.2954	0.0798
2FI	0.6258	0.2480	-0.6099
Quadratic	0.4989	0.2286	-1.2456
Cubic	0.9167	-0.1789	-38.2384

b) Analysis of variance (ANOVA)

Table 4.5: ANOVA for the Carbon dioxide Linear Model

Source	Sum of squares	df	Mean square	F-value	p-value
Model	96.65	4	24.16	4.04	0.0116
A-WFR	6.67	1	6.67	1.11	0.3012
B-GFR	12.98	1	12.98	2.17	0.1532
C-RT	42.80	1	42.80	7.15	0.0130
D-P	34.20	1	34.20	5.72	0.0246
Residual	149.56	25	5.98		
Pure Error	3.00	5	0.6009		
Cor Total	246.21	29			

The work done by Nanduri *et al*. (2008) revealed that ANOVA is a crucial tool used to determine significant and non-significant variables. In this case, ANOVA was used to determine the significance of the linear model. According to Quanhong and Caili, (2005), the significance of a variable is determined by both F-value and P-value. The larger the F-value and the smaller the P-value the more the significant influence an input variable has on the respective response variable. In this case, from Table 4.5, the linear effects of retention time (C), and pressure (D) were significant. Also, an F-value of 4.04 has a chance of 1.16% to occur due to noise.

c) Fit statistics

Table 4.6: Fit statistics for Carbon dioxide

Std. Dev.	2.45	Adeq Precision	7.5945
Mean	15.60	Adjusted R^2	0.2954
		Predicted R^2	0.0798

R^2 represents the coefficient of determination of the linear regression model. It measures the variability of the actual response that can be described by the respective independent factors (Mukhopadhyay *et al.*, 2013a). The predicted R^2 of 0.0798 is closely in agreement with the Adjusted R^2 value of 0.2954. The difference here was 0.2156. The acceptable disparity between the adjusted R^2 and the predicted R^2 should be about 0.2 (P. Subha and Jayaraj, 2019). This consequently indicates a reasonable agreement between the observed and the predicted values.

Adequate precision is a measure of signal-to-noise ratio; a ratio greater than 4 is desirable (Mason *et al.*, 2003). In this case, the ratio was 7.5945; which indicated an adequate signal. This model can therefore be employed to navigate the design space. The model showed a standard deviation of 2.45, and a mean of 15.60.

4.3.3 Model Graphs for Carbon dioxide

The two-dimensional contours and three-dimensional surface plots relating water flow rate and gas flow rate for the optimal responses of CO_2 are highlighted in Figures 4.5 and 4.6. The plots help visualize the relationship between the WFR and GFR on the CO_2 composition.

a) 2D Contour Plot

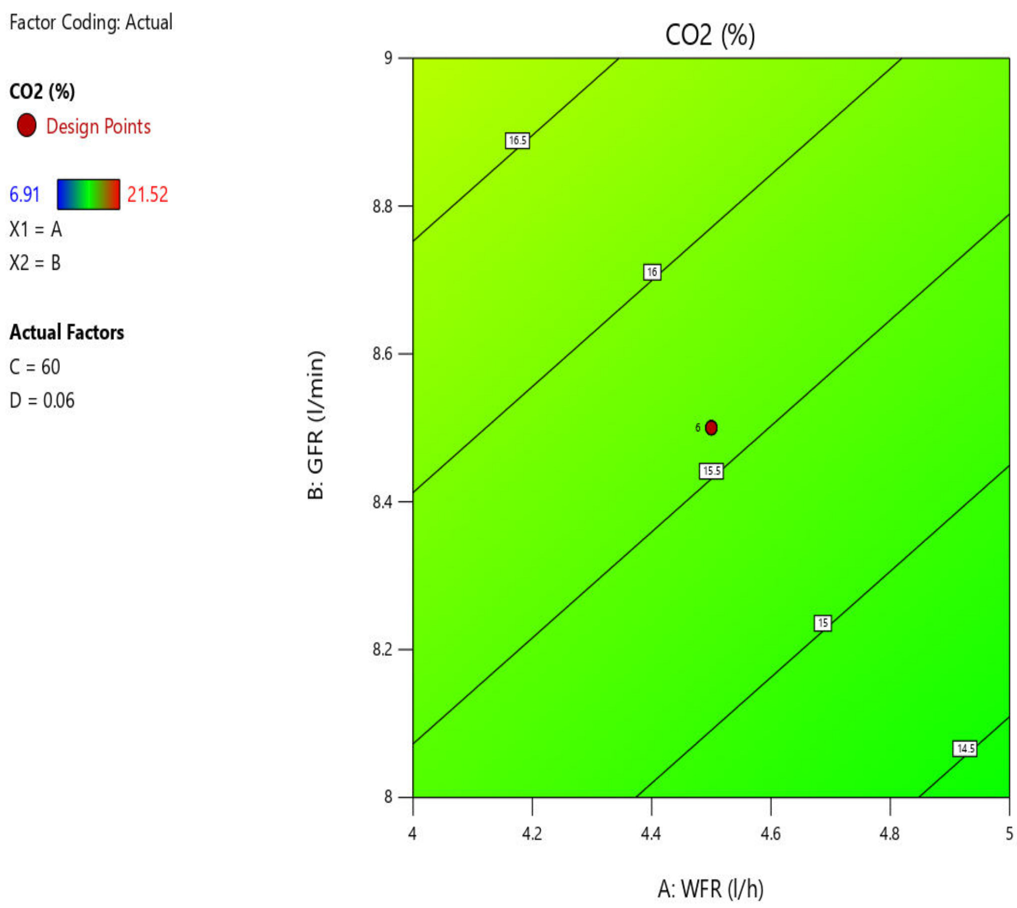


Figure 4.5: 2D Contour plot of WFR and GFR for CO_2

B) 3D Surface Plot

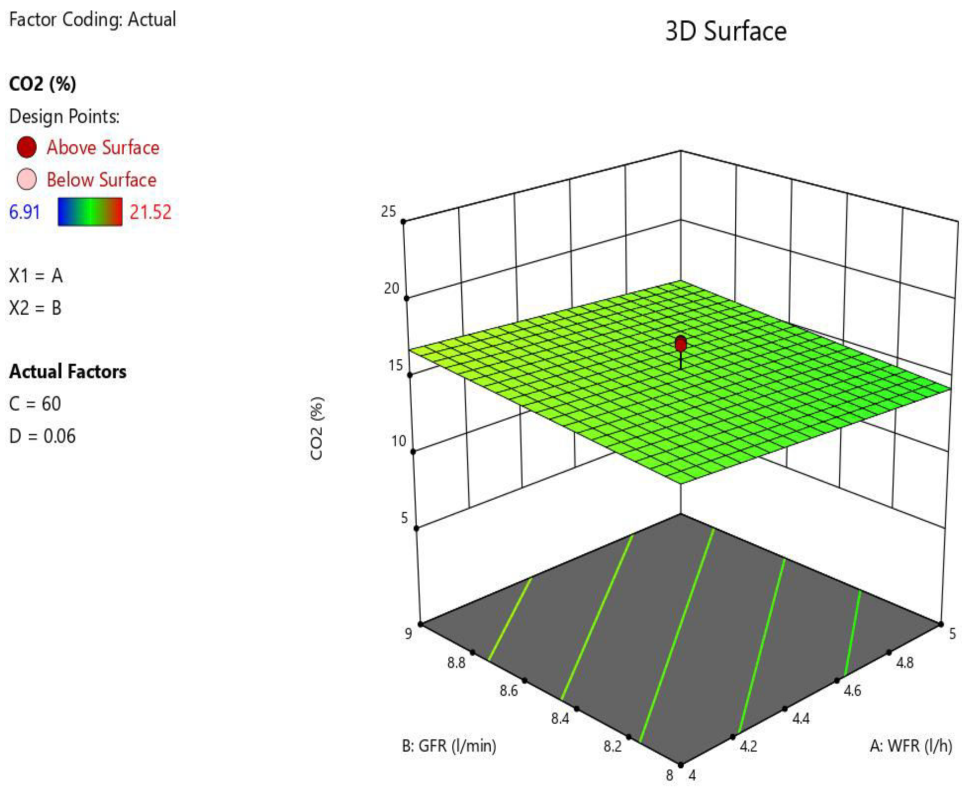


Figure 4.6: 3D surface plot of WFR and GFR for CO_2

4.4 Hydrogen Sulphide Minimization

4.4.1 Graphical Presentation

Table 4.1 shows that run 16 gave the highest minimization of Hydrogen Sulphide (H_2S) of 0% while the control experiment minimized to 10%. A graphical representation is shown in Figure 4.3.

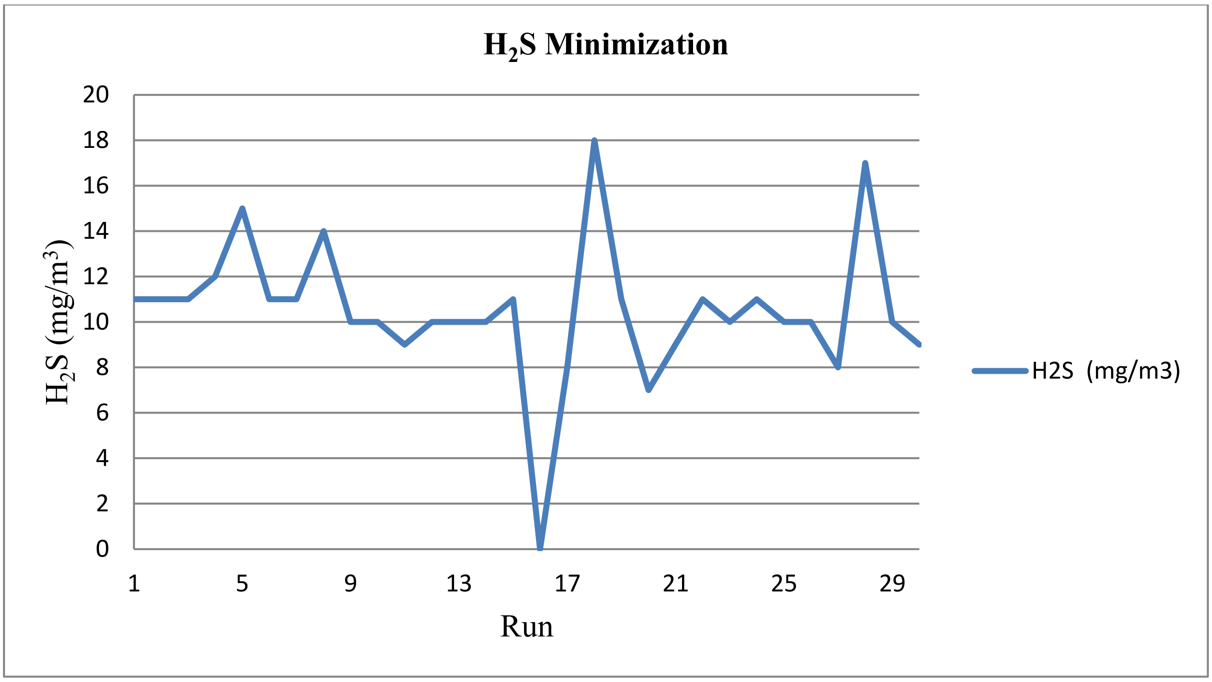


Figure 4.7: Hydrogen sulphide Minimization

According to Jiang *et al.* (2020), Hydrogen sulphide (H_2S) is highly soluble in water and establishes an ionization equilibrium once it is dissolved in water. Dissociation of the H_2S molecule forms a hydrosulphide ion (HS^-), which then dissociates to form the sulphide ion (S^{-2}) through the ionization of water. This explains the high efficiency of removing H_2S from biogas when water scrubbing technology is used.

4.4.2 Statistical Analysis

a) Fit Summary

Table 4.7 shows the Fit Summary. A sequential p-value of 0.0185 of the linear model is significant since its p-value is < 0.05. The 2FI has a p-value of 0.0477, but it is greater than that of the linear model. Quadratic and Cubic models can't be applied because their p-values are > 0.05. Consequently, the linear model was chosen.

Table 4.7: Fit Summary for Hydrogen Sulphide

Source	Sequential p-value	Adjusted R^2	Predicted R^2
Linear	0.0185	0.2654	0.0132
2FI	0.0477	0.4751	-0.1455
Quadratic	0.4389	0.4749	-0.5516
Cubic	0.5341	0.4602	-17.3370

b) ANOVA for H_2S Linear Model

Table 4.8: ANOVA for the linear model of Hydrogen Sulphide

Source	Sum of squares	df	Mean square	F-value	p-value	Remark
Model	183.38	10	18.34	3.62	0.0076	significant
A-WFR	0.0417	1	0.0417	0.0082	0.9286	
B-GFR	22.04	1	22.04	4.36	0.0506	
C-RT	45.38	1	45.38	8.97	0.0074	
D-P	35.04	1	35.04	6.93	0.0164	
AB	22.56	1	22.56	4.46	0.0482	
AC	39.06	1	39.06	7.72	0.0120	
AD	0.0625	1	0.0625	0.0124	0.9127	
BC	0.0625	1	0.0625	0.0124	0.9127	
BD	5.06	1	5.06	1.00	0.3297	
CD	14.06	1	14.06	2.78	0.1119	
Residual	96.12	19	5.06			
Pure Error	0.8333	5	0.1667			
Cor Total	279.50	29				

The Analysis of Variance (ANOVA) was applied to assess the significance of the linear regression model (Nanduri *et al.*, 2008). A large F-value and a p-value < 0.05 implied a more significant effect on the respective response variable (Quanhong and Caili, 2005). In this case, the linear influence of retention time (C), pressure (D), and interaction effects of WFR-GFR (AB), and WFR-RT (AC) were significant. This linear model is significant because the large F-value of 3.62 has a chance of 0.76% to occur due to noise.

c) Fit Statistics

Table 4.9: Fit statistics for Hydrogen Sulphide

Std. Dev.	Mean	Adeq. Precision
2.25	10.50	9.6672

Adequate precision is a measure of signal-to-noise ratio; a ratio greater than 4 is desirable (Mason *et al.*, 2003). In this case, the ratio was 9.6672; which indicated an adequate signal. This model can be used to navigate the design space. This linear model showed a standard deviation of 2.25 and a mean value of 10.50.

4.4.3 Model Graphs for Hydrogen Sulphide

The two-dimensional contours and three-dimensional surface plots relating water flow rate and gas flow rate for the optimal responses are highlighted in Figures 4.5 and 4.6. The plots help visualize the relationship between the input variables and the responses.

a) 2D Contour Plot

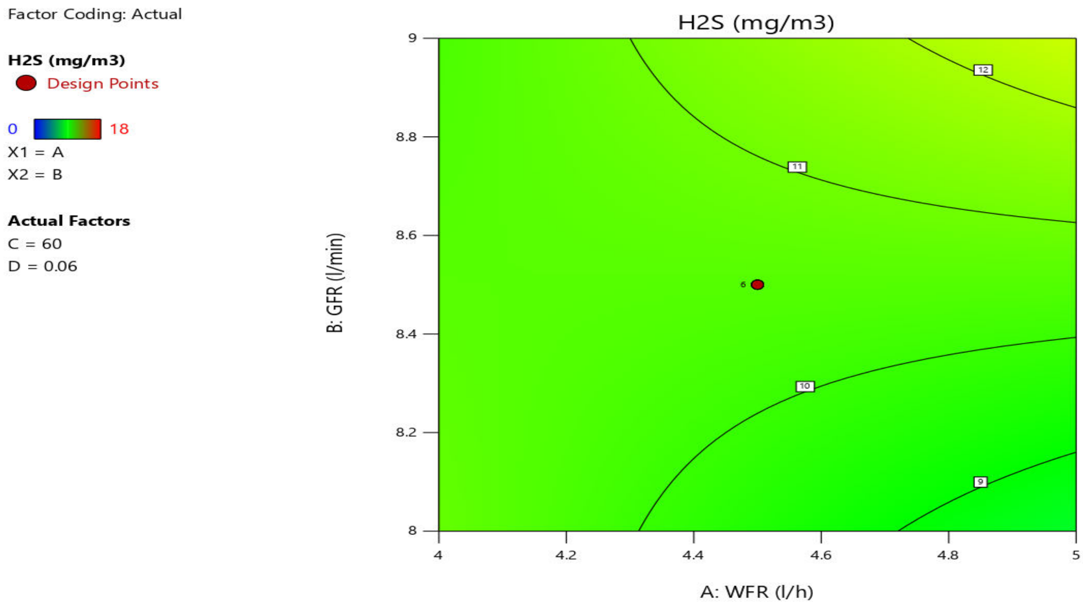


Figure 4.8: 2D Contour plot of WFR and GFR for H_2S

d) 3D Surface Plot

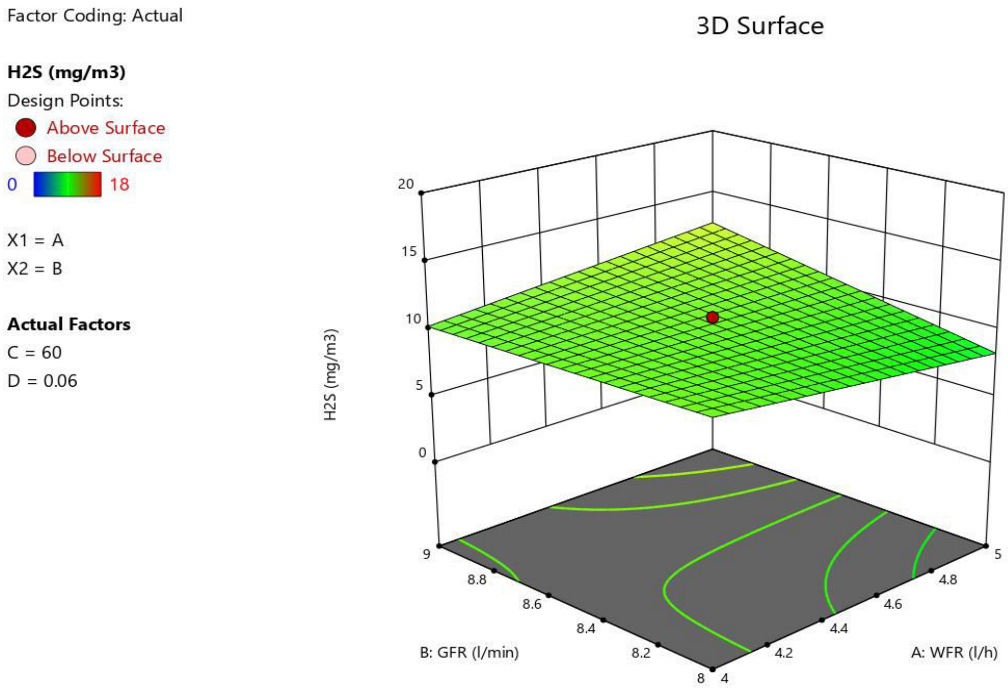


Figure 4.9: 3D surface plot of WFR and GFR for H_2S

4.5 Analysis of Prediction Models

The results of the experiment in Table 4.1 were fitted to the linear model and used to estimate the model parameters. The CCD was used to develop a matrix of 30 runs from which the responses were recorded and fed into the Design Expert version 13 software for the generation of the models for predicting the responses (CH_4, CO_2, and H_2S concentrations). The model equations for the three responses are illustrated by equations 4.2, 4.3, and 4.4, respectively.

4.5.1 The Prediction Model for Methane

The prediction model (equation) in terms of actual factors was given as in equation 4.2:

$$y = 1.96x_1 - 2.69x_2 + 0.14x_3 + 204.16x_4 + 74.96 \quad (4.2)$$

where;

y = percentage of methane (%)

x_1 = water flow rate (litres/hour)

x_2 = gas flow rate (litres/minute)

x_3 = retention time (seconds)

x_4 = pressure (bar)

If the values of the factors in run 10 are substituted in equation 4.1, that is 4.0, 8.0, 45, and 0.05 for x_1, x_2, x_3, and x_4, respectively, y is found to be 77.78%. The actual value of y is 81.36%. There is an error of 3.572%. This error is permissible since it is less than 5% (Reungsang *et al.*, 2012; Thanwised *et al.*, 2012). Hence, equation 4.2 can be used to predict the methane content by percentage in a similar experimental set-up.

4.5.2 The Prediction Model for CO_2

The model used to predict the responses for CO_2 was developed using the Design Expert version 13 as per the results indicated in Table 4.1. The results for every run were fed into the software, which generated the model shown in equation 4.3.

The prediction model for CO_2 in terms of actual factors is given by equation 4.3.

$$y = -1.056x_1 + 1.47x_2 - 0.08x_3 - 119.37x_4 + 20.34 \quad (4.3)$$

where;

y = percentage of CO_2 (%)

x_1 = water flow rate (litres/hour)

x_2 = gas flow rate (litres/minute)

x_3 = retention time (seconds)

x_4 = pressure (bar)

Taking run 14 randomly and substituting the independent factors: 5.0, 8.0, 45, and 0.07 replacing x_1, x_2, x_3, and x_4 respectively, y is predicted to be 14.89%. The actual value of y is 14.73%. There is an error of 0.16%. This error is permissible since it is less than 5% (B. Subha *et al.*, 2015; Urdampilleta *et al.*, 2023). Equation 4.3 can safely be used to predict the CO_2 content in a similar experimental set-up.

4.5.3 The Prediction Model for H_2S

The prediction model in terms of actual variables is given by equation 4.4.

$$y = -27.2x_1 - 26.7x_2 + 1.15x_3 - 645.83x_4 + 4.75x_1x_2 - 0.2x_1x_3 - 12.5x_1x_4 + 0.008x_2x_3 + 112.5x_2x_4 - 6.25x_3x_4 + 168.52 \quad (4.4)$$

where;

y = percentage of H_2S (mg/m^3)

x_1 = water flow rate (litres/hour)

x_2 = gas flow rate (litres/minute)

x_3 = retention time (seconds)

x_4 = pressure (bar)

If run 18 is randomly chosen and its independent factors; 4.5, 8.5, 60, and 0.04 are substituted for x_1, x_2, x_3, and x_4, respectively, y is predicted to be 15.1055%. The actual value of y is 18.0%. There is an error of 2.8945%. This error is permissible since it is less than 5% (B. Subha *et al.*, 2015; Urdampilleta *et al.*, 2023). Equation 4.4 can safely be used to predict the H_2S content in a similar experimental setup.

CHAPTER FIVE

5. CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSIONS

This research examined the purification of biogas using water scrubbing and compared the results through ANOVA, RSM, and CCD methods. An experimental matrix of thirty runs was done to examine the optimal performance of the system. The four control factors (Water Flow rate, gas flow rate, scrubber pressure, and gas retention time) were manipulated such as to achieve a combination of the factors that would lead to the best performance of the system.

It was found that a water flow rate (WFR) of 5 litres per hour, a gas flow rate (GFR) of 8.0 litres per minute, a retention time (RT) of 75 seconds and a pressure (P) of 0.07 bar gave the best results in terms of system performance. A combination of these factors gave the optimum level of methane of 91.45, the carbon dioxide of 6.91 and a 100 percent removal of hydrogen sulphide.

The purification of methane was increased by 10.79% -purifying 80.66% to 91.45% in comparison to the control experiment, carbon dioxide was decreased by 8.85% -purifying 15.76% to 6.91% and the purification of hydrogen sulphide had decreased by 0 - purifying 10 mg/m3 to 0 mg/m3.

Predictive models for the three responses (CH_4, CO_2, and H_2S) were developed. From these models, it can be deduced that there was an inverse relationship between methane concentration and the biogas flow rate, while water flow rate and retention were directly proportional to the methane concentration.

From the results, it can be concluded that optimizing the operating parameters of the water scrubbing process improves biogas quality. The simplicity of the system allows it to be easily adopted by medium and small-scale householders.

5.2 RECOMMENDATIONS

5.2.1 Policy and Practice Recommendations

1. The findings of this study show that optimizing the water scrubbing process yields a high methane content of 91.45% at low cost since it is operated at low pressure and at ambient temperature. It is therefore recommended that small-scale users of biogas can integrate this into their biogas plants in order to improve the calorific value of biogas and hence the combustion characteristics.

5.2.2 Recommendations for Further Research

1. Optimization of water scrubbing has proven to yield a high methane content; further research should be conducted to establish the rank of the influence of the parameters on the purification system and identify the most significant factor.
2. For environmental safety, the wastewater from this process is acidic because of dissolved CO_2 and H_2S, which are harmful to the environment. Therefore, there is a need to treat this waste before disposal or to simply develop a system for recycling the wastewater and minimizing wastage and environmental pollution.

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APPENDICES

APPENDIX 1: Methane

A1.1 Diagnostics

a) **Normal Plot**

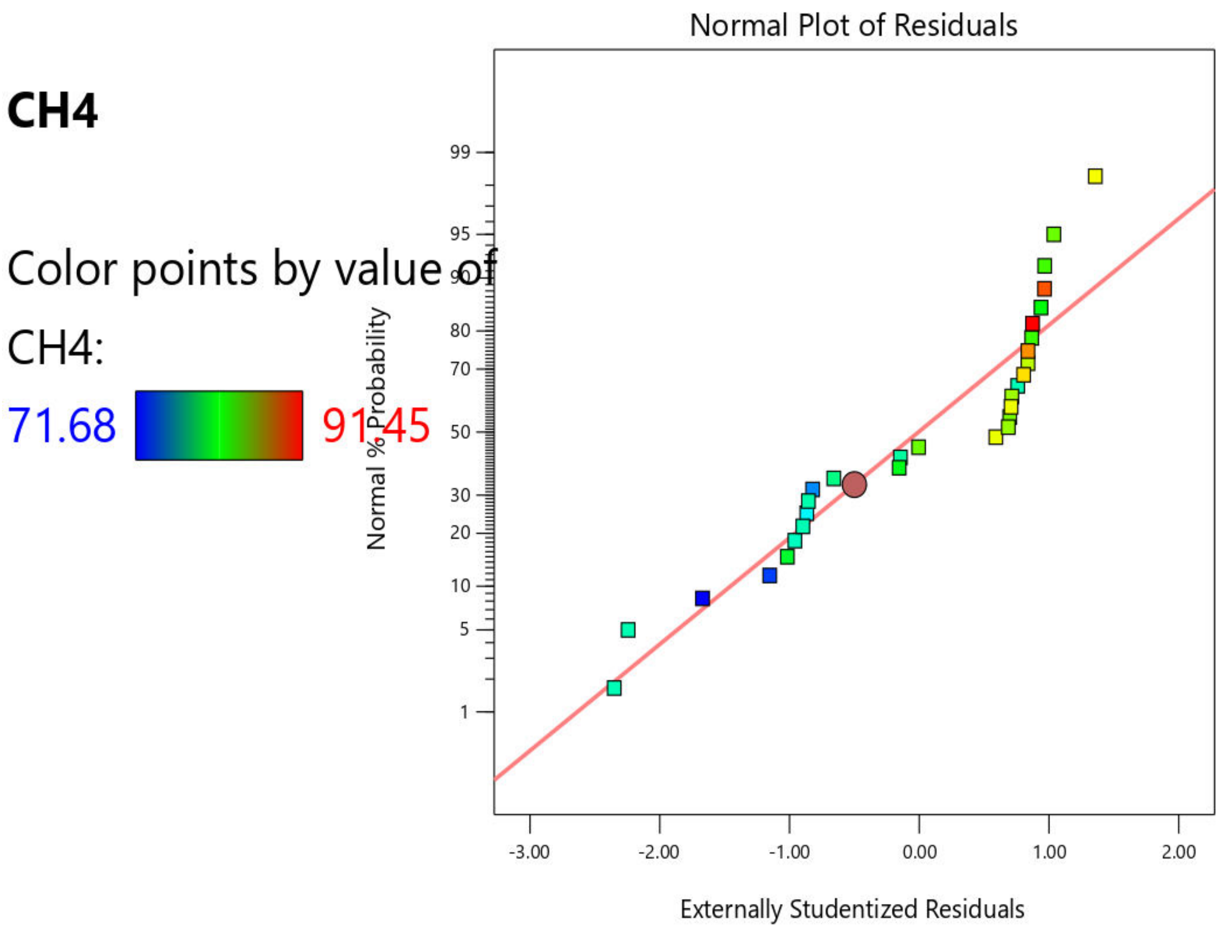

Figure A1.1.1: Normal plot of residuals

b) Predicted vs Actual

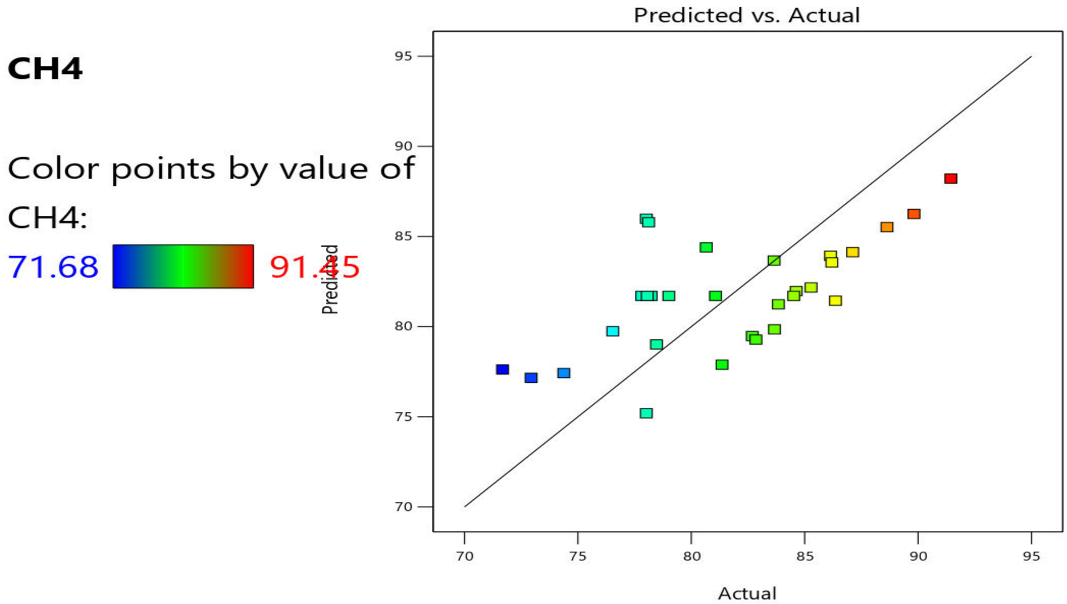

Figure A1.1.2: Predicted Vs actual methane

APPENDIX 2: Carbon Dioxide

A2.1: Diagnostics

a) Normal Plot

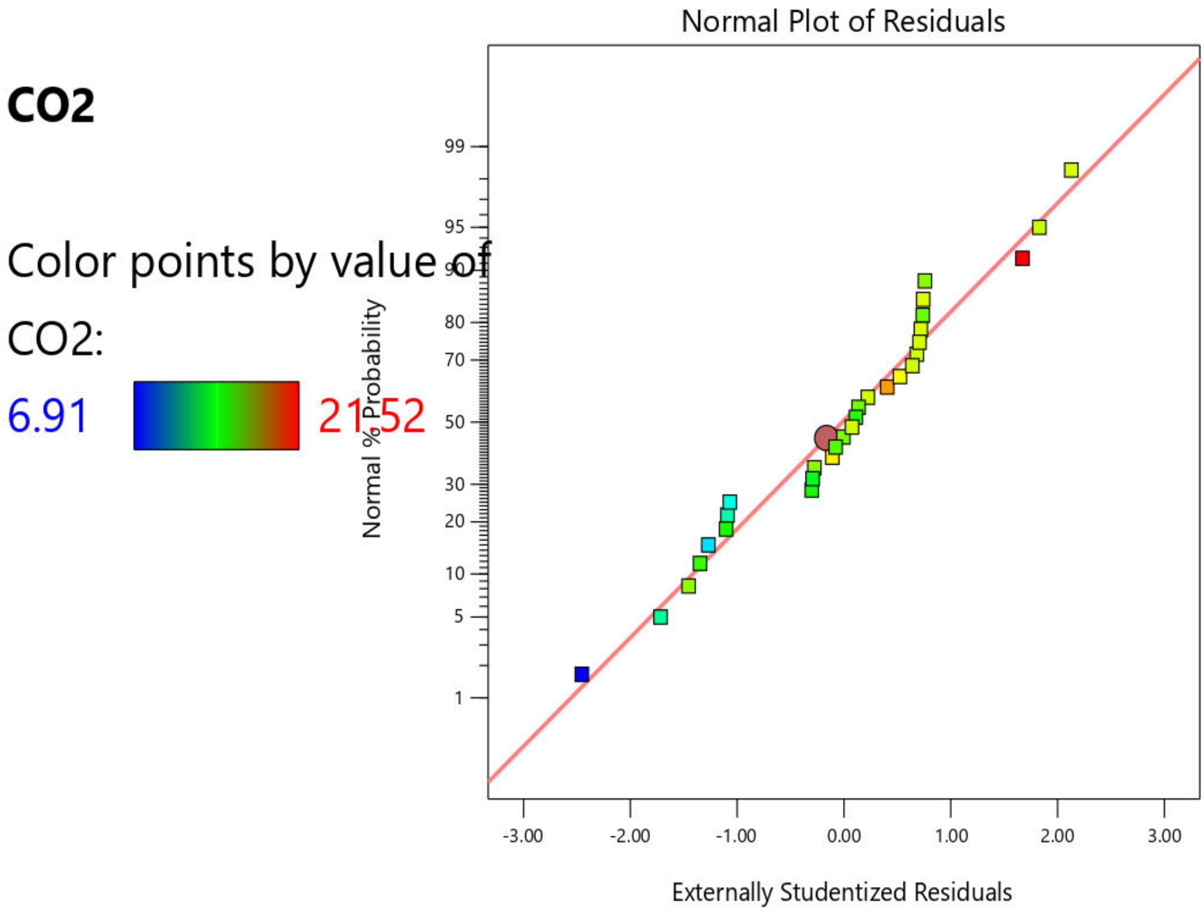

Figure A2.1.1: Normal plot of CO_2

b) Predicted vs Actual

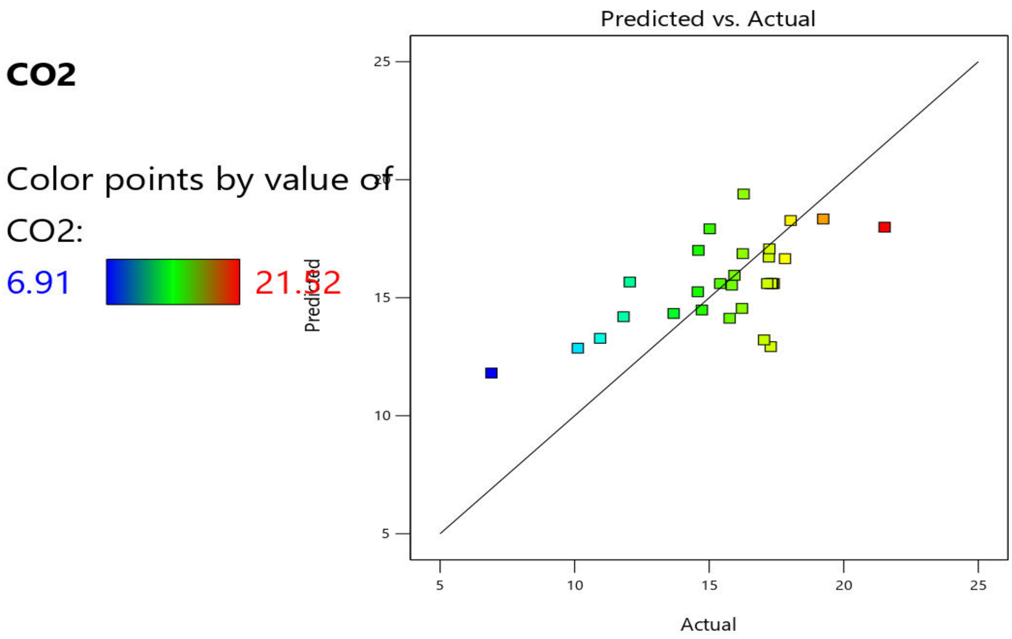

Figure A2.1.2: Predicted vs Actual CO_2

APPENDIX 3: Hydrogen Sulphide

A3.1: Diagnostics

a) **Normal Plot**

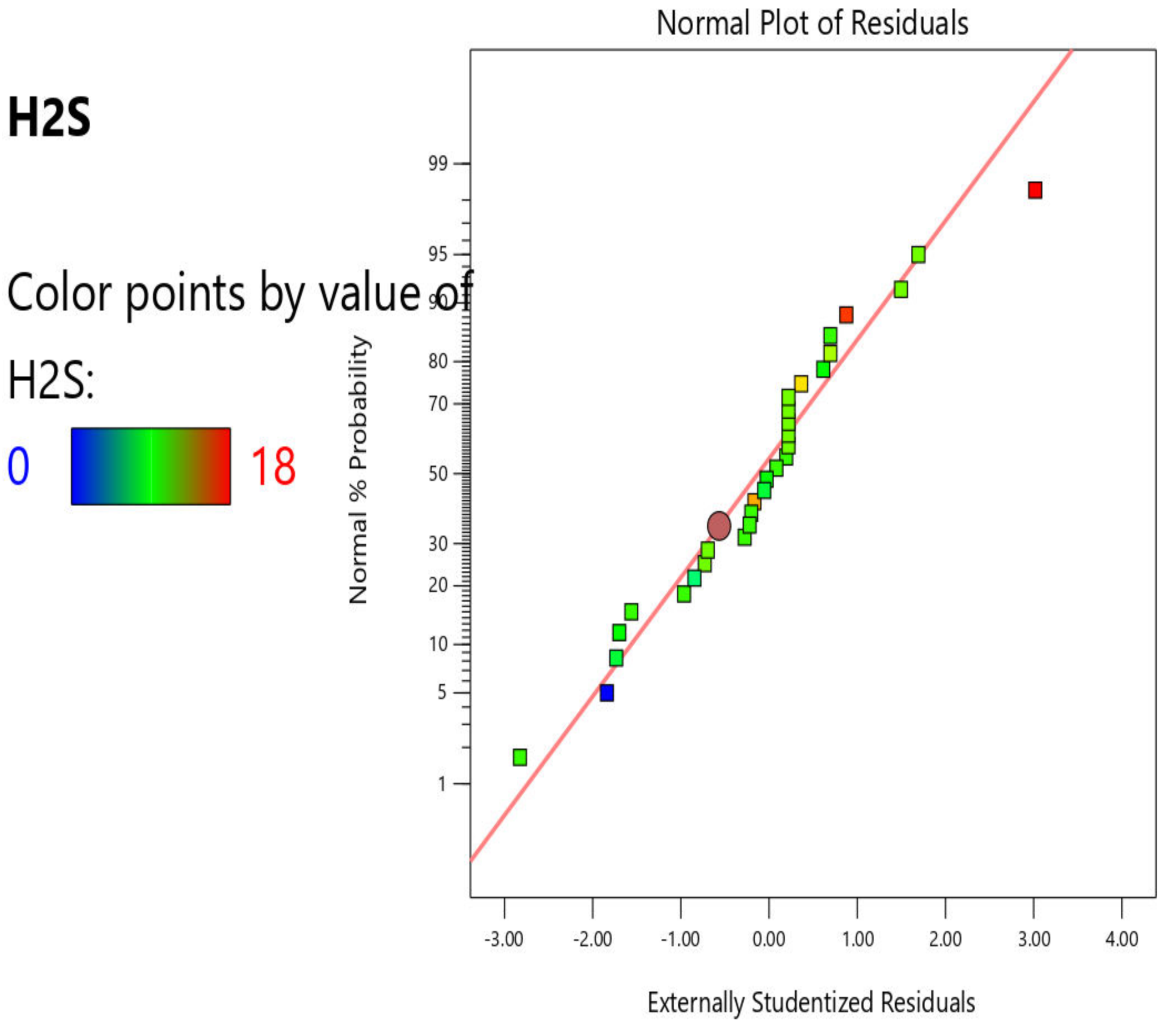

Figure A3.1.1: Normal plot for H_2S

b) Predicted vs Actual

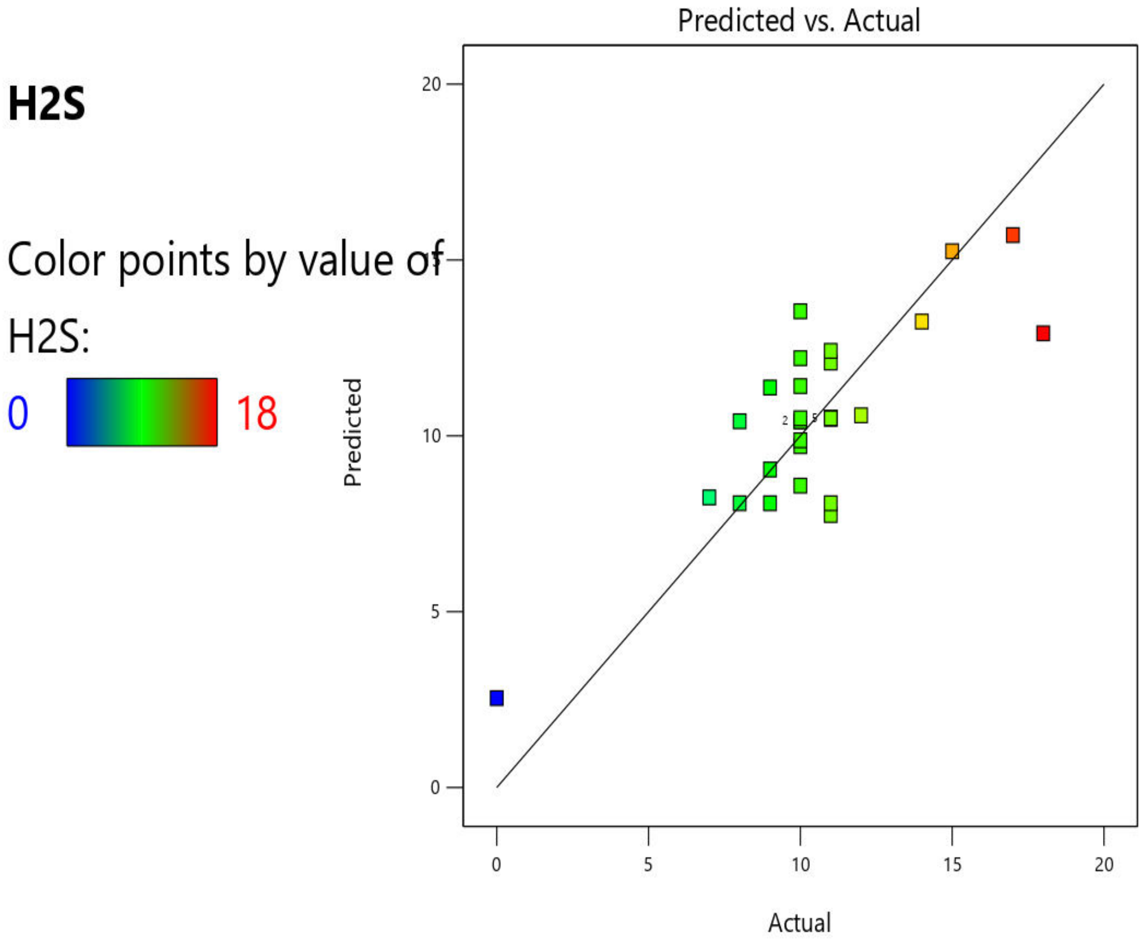

Figure A3.1.2: Plot of Predicted vs Actual H_2S

APPENDIX 4: Research Licenses

A4.1: NACOSTI Research Permit

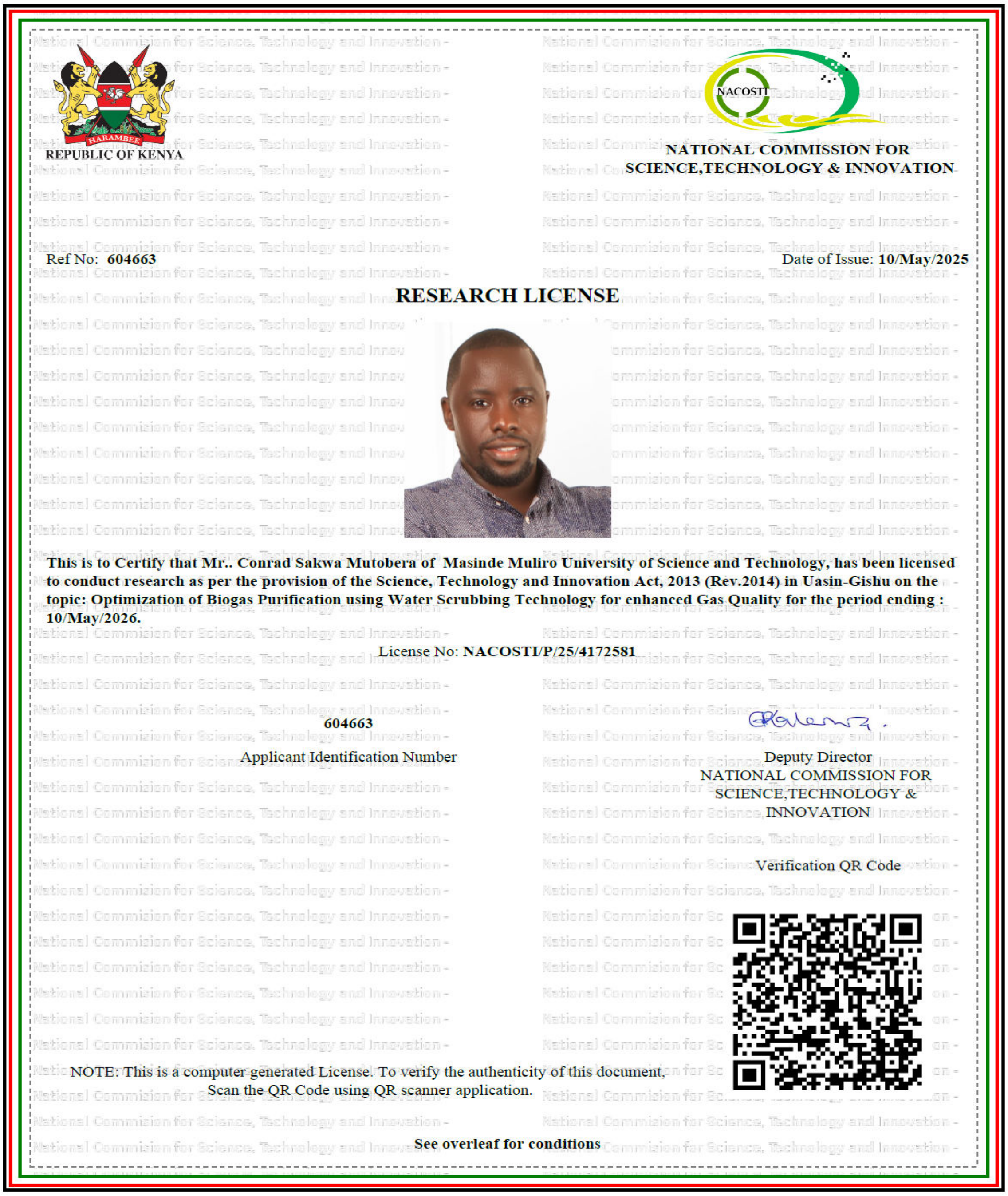

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Figure A4.1.1: NACOSTI research permit