OPTIMISATION OF THERMAL-ALKALINE PRETREATMENT OF WATER HYACINTH FOR ENHANCED BIOGAS YIELD

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A PROJECT SUBMITTED TO THE DEPARTMENT OF SCIENCE LABORATORY TECHNOLOGY IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE AWARD OF BACHELOR OF SCIENCE (B.SC) HONOUR DEGREE IN SCIENCE LABORATORY TECHNOLOGY (CHEMICAL/PETROLEUM TECHNIQUES)

NOVEMBER, 2025

CERTIFICATION

This is to certify that this project work titled **“Optimisation of Thermal-Alkaline Pretreatment of Water Hyacinth for Enhanced Biogas Yield”** was carried out and presented by **Elisha Uyi ILAWE-OSAKUE**, with Matriculation Number **LSC1706055,** in the Department of Science Laboratory Technology (Chemical/Petroleum Techniques), Faculty of Life Sciences, University of Benin, Benin City, Edo State, under the supervision of **ENGR. O. SALOKUN.**

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PROJECT SUPERVISOR

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PROJECT COORDINATOR

PROF. J. O. OSARUMWENSE **DATE.**
HEAD OF DEPARTMENT

EXTERNAL EXAMINER **DATE**

DEDICATION

This project is dedicated to God Almighty for His Grace and Mercies upon my life and making it possible for the successful completion on this project research work, to my family, whose unwavering support, encouragement and love has been my pillar of strength throughout my academic journey.

ACKNOWLEDGEMENT

I wish to express my heartfelt gratitude to everyone who contributed to the successful completion of this project. First and foremost, I am deeply indebted to my supervisor, ENGR. SALOKUN OLUWATOBA, for his invaluable guidance, expertise, and encouragement throughout this research. His insight and constructive feedback were instrumental in shaping this work.

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Finally, I acknowledge the scientific community and researchers whose work on sustainable energy and environmental management inspired this study and to my fellow colleagues as we continue to strive for sustainable solutions to global environmental challenges.

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ABSTRACT

The escalating ecological threats posed by invasive aquatic plants and the pressing need for sustainable bioenergy sources have driven the investigation of viable lignocellulosic feedstocks. This research centers on the thermochemical pretreatment of water hyacinth (Eichhornia crassipes) to improve biodegradability and biogas production. Water hyacinth, recognized for its high lignin content that prolongs microbial digestion periods (30–60 days), was treated with sodium hydroxide (NaOH) under varying conditions of temperature, dosage and reaction. Response Surface Methodology (RSM) via Design-Expert software was utilized to optimize the pretreatment variables and assess their impact on degradation efficiency. The compositional analysis of raw water hyacinth indicated a moisture content of 70.17%, ash content of 19.94%, crude fiber of 0.4932%, and volatile solids of 9.88%, underscoring its suitability as a biogas substrate. The thermochemical pretreatment markedly boosted organic matter solubilization, evidenced by soluble chemical oxygen demand (sCOD) range of 36,600 mg/L and degree of degradation (%DD) reaching up to 91.05%, though RSM analysis showed no significant factor influences (mean %DD = 88.55%), implying a recalcitrance-induced plateau. The optimal conditions for the pretreatment were identified at temperatures of 80°C, 30ml NaOH dosage, and 30 minutes reaction time, yielding consistent improvements in solubilization. These results illustrate that thermochemical pretreatment effectively overcomes biomass recalcitrance in water hyacinth, enhancing digestibility and prospective biogas output.

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

The global demand for renewable energy has intensified in recent years due to the depletion of fossil fuel reserves, rising energy costs, and the urgent need to mitigate climate change through sustainable practices. Biogas, a renewable energy source primarily composed of methane (CH_4) and carbon dioxide (CO_2), is produced through the anaerobic digestion of organic matter and offers a viable alternative for applications such as cooking, electricity generation, and heating (Kumari *and* Singh, 2023). Among the various feedstocks for biogas production, lignocellulosic biomass has gained attention due to its abundance and potential to address both energy and environmental challenges. Water hyacinth (Eichhornia crassipes), an invasive aquatic plant, has emerged as a promising yet challenging feedstock due to its rapid proliferation and complex biochemical composition (Barua *and* Kalamdhad, 2023).

Water hyacinth, native to South America, is one of the most invasive aquatic species globally, thriving in freshwater ecosystems across tropical and subtropical regions, including Africa, Asia, and Latin America. Its aggressive growth leads to severe ecological disruptions, such as oxygen depletion, obstruction of waterways, and reduced biodiversity, posing significant challenges to water resource management (Ofori *et al.*, 2021).

In Nigeria, for instance, water hyacinth infestations in the Niger River and other water bodies have impacted fishing, transportation, and water quality, necessitating innovative management strategies (Ofori *et al.*, 2021). However, the plant's high biomass productivity—capable of doubling in as little as 5-15 days under optimal conditions—makes it an abundant resource for bioenergy production (Rezania *et al.*, 2019).

The lignocellulosic composition of water hyacinth, typically consisting of 20-40% cellulose, 15-25% hemicellulose, and 5-20% lignin, renders it theoretically suitable for biogas production through anaerobic digestion (Rezania *et al.*, 2019). Cellulose and hemicellulose are fermentable components that can be broken down by microorganisms into biogas, while lignin, a complex polymer, forms a recalcitrant barrier that limits microbial access, resulting in low biogas yields (typically 100-200 mL/g volatile solids [VS] for untreated samples) and extended digestion times (Mathew *et al.*, 2019). To overcome these limitations, pretreatment methods—physical, chemical, thermal, hydrothermal, or combined—are employed to disrupt the lignocellulosic matrix, enhance substrate accessibility, and improve the efficiency of anaerobic digestion (Barua *and* Kalamdhad, 2023).

Recent studies have demonstrated the efficacy of various pretreatment methods. For example, chemical pretreatment with 5% v/v sulfuric acid (H_2SO_4) for 60 minutes increased biogas production by 131.45%, yielding

424.30 mL/g VS with a methane content of 64.38% (Barua *and* Kalamdhad, 2023). Similarly, alkali pretreatment with 5% w/v sodium hydroxide (NaOH) achieved a biogas yield of 142.61 L/kg VS with 64.59% methane content (Sarto *et al*., 2023). Thermal and thermochemical pretreatments, such as heating at 121°C and pH 11.00, have also improved biomass solubility and gas production, though severe conditions may produce inhibitory byproducts like furfural (Sarto *et al*., 2023). Hydrothermal pretreatment, often combined with co-substrates like buffalo dung, has shown a 51% increase in methane yield (2,856 mL/day) compared to untreated samples (Suwan *et al*., 2019). Co-digestion with organic wastes, such as cow dung, further enhances yields by balancing the carbon-to-nitrogen (C/N) ratio, as demonstrated by a 24% increase in biogas productivity when water hyacinth was pretreated with H_2SO_4 and co-digested with cow dung (Kumari *and* Singh, 2023).

The potential of water hyacinth for biogas production lies not only in its energy output but also in its role in environmental management. By converting an invasive species into a valuable resource, this approach addresses ecological challenges while contributing to renewable energy goals. However, optimizing pretreatment methods to balance efficiency, cost, and environmental impact remains a critical area of research, particularly in regions with abundant water hyacinth populations.

1.2 AIM AND OBJECTIVES

The primary aim of this study is to optimize pretreatment methods for water hyacinth to maximize biogas yield through anaerobic digestion.

The specific objectives are to:

1. characterize the feedstock so as to determine the suitability for biogas production.
2. pretreat the feedstock to enhance the digestibility during anaerobic digestion.
3. To optimize the pretreatment process for maximum feedstock degradation

1.3 PROBLEM STATEMENT

Despite its biogas potential, water hyacinth's lignocellulosic structure, with high lignin content, impedes microbial degradation, yielding low biogas (100–200 mL/g VS) and extended digestion times (30–60 days). Key research gaps encompass the optimization of pretreatment conditions, as chemical pretreatments (such as; 2–5% H_2SO_4 or NaOH, 30–90 min) enhance yield but feature varying optimal parameters that lack scalability; inhibitory byproducts, where intense thermochemical methods produce toxins (like furfural) that inhibit methanogens; cost and environmental impact, since reagents are expensive and pose disposal risks while sustainable alternatives remain underexplored; co-digestion strategies, with optimal C/N ratios involving substrates like cow dung still unstandardized; and regional contextualization, as Nigeria-specific adaptations for local water hyacinth and seasonal factors are limited.

1.4 SIGNIFICANCE OF STUDY

This study addresses energy security, environmental management, and sustainable development by converting invasive water hyacinth into biogas. Key significance includes environmental management through controlling proliferation, such as in the Niger River Basin, and promoting a circular economy; renewable energy by supplying clean biogas for cooking and electricity, for example, achieving 0.52 m^3/m^3/day in a Niger hospital project; economic benefits by enhancing yield for viable small-scale and industrial applications in biomass-rich developing countries; scientific contribution by advancing lignocellulosic conversion and optimization of pretreatment and co-digestion, applicable to other feedstocks; and socioeconomic impact by enabling community waste-to-energy initiatives in Nigeria, supporting SDG 7 and SDG 13.

CHAPTER TWO
LITERATURE REVIEW

2.1 OVERVIEW

The global energy crisis, coupled with environmental challenges posed by invasive species, has driven research into sustainable bioenergy solutions. Water hyacinth (*Eichhornia crassipes*), a prolific aquatic weed, presents a unique opportunity to address both ecological management and renewable energy production through biogas generation via anaerobic digestion. Its rapid growth disrupts freshwater ecosystems, but its high biomass and organic content make it a promising feedstock for biogas, a renewable energy source primarily composed of methane (CH_4) and carbon dioxide (CO_2). However, the plant's lignocellulosic structure limits efficient digestion, necessitating pretreatment to enhance biogas yield. This literature review comprehensively examines the ecological and biochemical properties of water hyacinth, the principles of biogas production, pretreatment methods, optimization strategies, co-digestion approaches, practical applications, environmental and economic considerations, methodological nuances, emerging trends, and research gaps. By synthesizing recent studies (2019-2025), this chapter provides a robust foundation for optimizing pretreatment methods to maximize biogas yield, contributing to sustainable waste management and energy security, particularly in regions like Nigeria where water hyacinth is abundant.

2.2 ECOLOGICAL IMPACT OF WATER HYACINTH

Water hyacinth is recognized as one of the world’s most invasive aquatic plants, thriving in tropical and subtropical freshwater ecosystems, including rivers, lakes, and wetlands across Africa, Asia, and Latin America (Ofori *et al*., 2021). It’s rapid growth rate—doubling in biomass within 5-15 days under optimal conditions—leads to severe ecological disruptions, including oxygen depletion, reduced light penetration, and altered aquatic ecosystems (Ofori *et al*., 2021). This led African countries to commit to various international environmental treaties and, as a result, develop national action plans to address the serious impacts of the infestations on their local ecosystems (Ilo *et al*., 2020). In Nigeria, water hyacinth infestations in the Niger River Basin have clogged waterways, disrupted fishing, and impaired water quality, costing millions annually in management efforts (Ofori *et al*., 2021). Similarly, in India, the plant has impacted water bodies like the Vembanad Lake, affecting local economies and biodiversity (Barua *and* Kalamdhad, 2023). In Indonesia, water hyacinth has obstructed irrigation channels, reducing agricultural productivity (Sarto *et al*., 2023). These ecological challenges highlight the urgency of developing utilization strategies to transform water hyacinth into a valuable resource.

Ofori *et al*. (2021) conducted a comprehensive study on water hyacinth's ecological impact in the Niger River Basin, reporting that its dense mats reduce dissolved oxygen levels to below 2 mg/L, adversely affecting fish populations and aquatic flora. The study emphasized the socioeconomic consequences, including reduced access to water for irrigation and transportation. Similarly, Rezania *et al*. (2019) analyzed water hyacinth's spread in Southeast Asia, noting its ability to cover entire water surfaces, leading to eutrophication and biodiversity loss. These findings underscore the need for innovative management strategies, such as bioenergy production, to mitigate the plant's environmental impact while leveraging its biomass potential.

2.3 BIOCHEMICAL COMPOSITION OF WATER HYACINTH

The biochemical composition of water hyacinth is critical to its potential as a biogas feedstock. Studies indicate that it contains 20-40% cellulose, 15-25% hemicellulose, 5-20% lignin, and 85-95% moisture, with a carbon-to-nitrogen (C/N) ratio of 20-30 (Rezania *et al*., 2019). Cellulose and hemicellulose are fermentable polysaccharides that can be converted into biogas during anaerobic digestion, while lignin, a complex polymer, forms a recalcitrant barrier that hinders microbial access (Mathew *et al*., 2019). Cellulose chains aggregate into microfibrils, which in turn bundle together to form cellulose fibers (Omondi *et al*., 2019). The high moisture content facilitates liquid anaerobic digestion (L-AD) but complicates solid-state anaerobic digestion (SS-AD), requiring specific pretreatment to enhance accessibility (Kumari *and* Singh, 2023).

Rezania *et al*. (2019) conducted a detailed analysis of water hyacinth's composition across different regions, noting variations due to environmental factors like water nutrient levels and temperature. For instance, plants in nutrient-rich waters exhibited higher cellulose content (up to 40%), enhancing their biogas potential. Mathew *et al*. (2019) further quantified the volatile solids (VS) content, reporting that water hyacinth has 80-90% VS, making it a high-energy feedstock. However, the lignin content, which varies by plant maturity and growth conditions, poses a significant challenge, as it resists microbial degradation (Kumari *and* Singh, 2023). These studies highlight the need for pretreatment to disrupt the lignocellulosic matrix and unlock the bioenergy potential of water hyacinth.

2.4 BIOGAS PRODUCTION

Biogas, composed of 50-70% methane, 30-50% carbon dioxide, and trace gases (e.g., hydrogen sulfide), is produced through anaerobic digestion, a microbial process involving four stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Kumari *and* Singh, 2023). Hydrolysis breaks down complex organic matter into simpler compounds (e.g., sugars, amino acids), which are converted into volatile fatty acids during acidogenesis. Acetogenesis transforms these into acetate, hydrogen, and CO_2, which methanogenic bacteria convert into methane during methanogenesis (Mathew *et al*., 2019). The efficiency of anaerobic digestion depends on substrate characteristics, microbial activity, and operational parameters like total solids (TS) content, C/N ratio, pH, temperature, and food-to-microorganism (F/M) ratio.

Water hyacinth's lignocellulosic structure slows the hydrolysis stage, resulting in low biogas yields (100-200 mL/g VS) and extended digestion times (30-60 days) for untreated samples (Mathew *et al*., 2019). Kumari and Singh (2023) emphasize that a C/N ratio of 20-30 is optimal for balancing microbial growth and preventing ammonia accumulation, which can inhibit methanogenesis. Temperature also plays a critical role, with mesophilic conditions (30-40°C) being more energy-efficient than thermophilic conditions (50-60°C) for water hyacinth digestion (Barua *and* Kalamdhad, 2023). pH stability (6.8-7.5) is essential to prevent acid accumulation, which can disrupt methanogenic bacteria (Kumari *and* Singh, 2023).

Mathew *et al*. (2019) conducted a seminal study on anaerobic digestion of water hyacinth, reporting that L-AD with 3.38% TS and a C/N ratio of 30 produced 369 mL/g TS over 60 days, significantly outperforming SS-AD at 15% TS (34.79 mL/g TS at 24.13% TS). The higher water content in L-

AD facilitates microbial activity, while SS-AD requires drying, increasing energy inputs. Barua and Kalamdhad (2023) further explored the F/M ratio, finding that a 2:1 ratio optimized biogas yield by ensuring sufficient microbial populations. These findings provide a baseline for understanding digestion challenges and the need for pretreatment to enhance biogas yield.

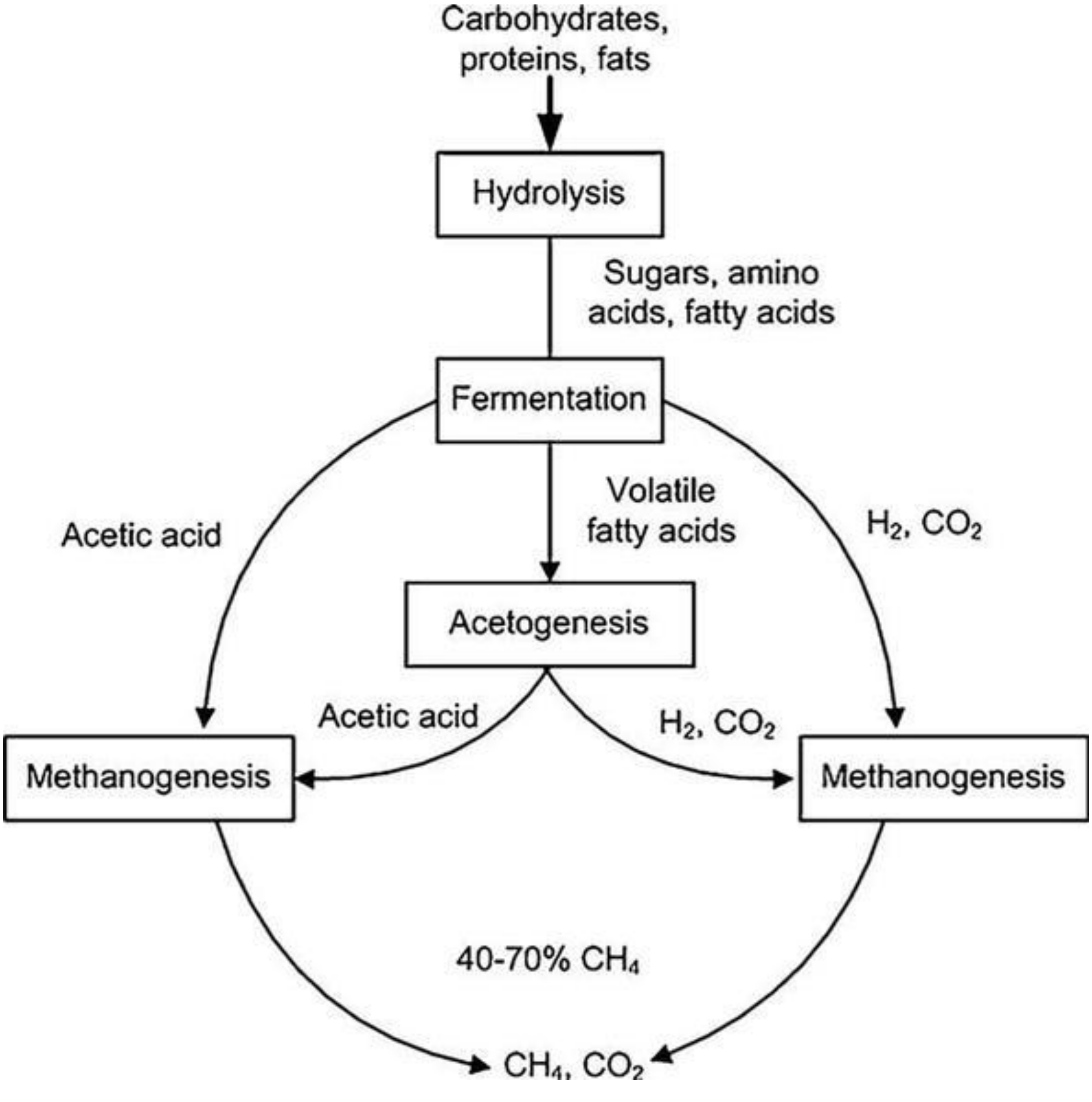

Figure 2.1: Process flow of the degradation of organic material through anaerobic digestion (Adelodun *et al*., 2023).

2.5 HISTORY OF BIOGAS TECHNOLOGY IN NIGERIA

Biogas technology in Nigeria dates back to the **1970s** during the global oil crisis, when the Federal Government initiated pilot projects to explore alternative energy sources. The National Centre for Energy Research and Development (NCERD) at the University of Nigeria, Nsukka, constructed one of the first fixed-dome digesters in 1978 using cattle dung (Akinbomi *et al*., 2019). By the 1980s–1990s, non-governmental organizations (NGOs) and academic institutions promoted household-scale digesters in rural areas, but adoption remained low due to high costs, poor maintenance, and lack of technical expertise (Ofori *et al*., 2021).

The 21st century saw renewed interest driven by climate change mitigation and renewable energy policies. The Renewable Energy Master Plan (REMP) of 2005 targeted 10% renewable energy by 2025, with biogas identified as a key component (Federal Ministry of Power and Steel, 2005). In 2015, the Rural Electrification Agency (REA) launched the Nigeria Electrification Project (NEP), integrating biogas into mini-grid systems. Recent efforts (2020–2025) focus on agro-waste and aquatic biomass, including water hyacinth, due to its abundance in the Niger Delta and Lake Chad regions (Adeoti *et al*., 2023).

Despite progress, challenges persist: only ~1,000 functional digesters exist nationwide (as of 2023), primarily in institutions and commercial farms

(Okeh *et al*., 2022). Nigerian researchers have increasingly explored water hyacinth as a substrate, leveraging its rapid growth (doubling in 7–10 days) and high organic content to address energy poverty (affecting 85 million Nigerians) and ecological disruption (Ofori *et al*., 2021).

2.6 BIOGAS PRODUCTION PROCESSES FROM WATER HYACINTH

Biogas production involves four anaerobic stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. Water hyacinth's lignocellulosic structure (cellulose ~18–25%, hemicellulose ~25–40%, lignin ~5–10%) resists hydrolysis, necessitating pretreatment (Mathew *et al*., 2019).

- **Hydrolysis:** Rate-limiting step due to crystalline cellulose and lignin shielding. Pretreatment disrupts this barrier, increasing soluble COD (sCOD) (Barua *and* Kalamdhad, 2023).

- **Acidogenesis/Acetogenesis:** Converts monomers to volatile fatty acids (VFAs). High C/N ratio (~20–30) of water hyacinth supports balanced fermentation (Kumari *and* Singh, 2023).

- **Methanogenesis:** Sensitive to pH (6.8–7.5) and inhibitors (e.g., furfural). Nigerian studies emphasize mesophilic conditions (35°C) due to ambient temperatures (Adeoti *et al*., 2023).

Field trials in Ogun State (2021) using 500 L digesters achieved 250–300 mL/g VS from untreated water hyacinth, improved to 450 mL/g VS with alkali pretreatment (Okeh *et al*., 2022).

2.7 BIOGAS PRETREATMENT FOR ENHANCED YIELD

Pretreatment is essential to overcome the lignocellulosic barriers of water hyacinth, improving substrate accessibility and biogas yield. Recent studies have explored physical, chemical, thermal, hydrothermal, biological, and combined pretreatment methods, each with distinct mechanisms, advantages, and limitations.

2.7.1 Physical Pretreatment

Physical pretreatments, such as mechanical grinding, chopping, or ultrasonication, reduce particle size to increase surface area for microbial attack. Rezania *et al*. (2019) found that reducing water hyacinth to particle sizes of 0.5-1 mm increased biogas yield by 10-15% compared to unprocessed biomass, as smaller particles enhance hydrolysis. Ultrasonication, which uses sound waves to disrupt cell walls, has also shown promise, with Mathew *et al*. (2019) reporting a 12% yield increase (220 mL/g VS) for ultrasonicated samples. However, physical methods are energy-intensive and less effective for lignocellulosic biomass compared to chemical or thermal approaches, limiting their standalone application (Mathew *et al*., 2019). For instance, grinding alone requires significant energy input, making it less cost-effective for large-scale systems (Rezania *et al.*, 2019).

2.7.2 Chemical Pretreatment

Chemical pretreatments use acids, alkalis, or other reagents to disrupt lignin and hemicellulose, enhancing cellulose bioavailability. Barua and Kalamdhad (2023) investigated acid pretreatment with 5% v/v sulfuric acid (H_2SO_4) for 60 minutes, achieving a 131.45% increase in biogas yield (424.30 mL/g VS) with 64.38% methane content compared to untreated samples (183 mL/g VS). The acid hydrolyzes hemicellulose, increasing the surface area for microbial degradation. Similarly, Sarto *et al*. (2023) explored alkali pretreatment with 5% w/v sodium hydroxide (NaOH), reporting a biogas yield of 142.61 L/kg VS with 64.59% methane content. Alkali pretreatment disrupts lignin-carbohydrate bonds, enhancing biomass solubility and digestibility.

However, chemical pretreatments have significant drawbacks. Rezania *et al*. (2019) note that high acid concentrations can generate inhibitory byproducts like furfural and hydroxymethylfurfural, which suppress methanogenic activity. For example, Barua and Kalamdhad (2023) observed a 10% reduction in methane yield at 7% H_2SO_4 due to inhibitor formation. Alkali pretreatments, while effective, require neutralization to prevent pH imbalances in digesters, adding operational complexity (Sarto *et al.*, 2023). Additionally, chemical reagents are costly, and their disposal raises environmental concerns, necessitating optimization of concentration and treatment time (Rezania *et al.*, 2019). Sarto *et al.* (2023) found that reducing NaOH concentration to 2% w/v maintained significant yield improvements while lowering costs, suggesting a need for balanced approaches.

2.7.3 Thermal and Thermochemical Pretreatment

Thermal pretreatments involve heating biomass to disrupt cell walls and improve solubility. Sarto *et al.* (2023) demonstrated that thermochemical pretreatment at 121°C and pH 11.00 increased biogas production by 30-40% by enhancing the breakdown of lignocellulosic components. The combination of heat and alkali (e.g., NaOH) synergistically degrades lignin, improving microbial access. However, severe conditions can produce inhibitory compounds, reducing methanogenic efficiency (Sarto *et al.*, 2023). For instance, Sarto *et al.* (2023) reported a 15% yield reduction at 150°C due to furfural formation, highlighting the need for controlled conditions.

Suwan *et al.* (2019) explored hydrothermal pretreatment (high temperature and pressure in water) with buffalo dung as a co-substrate, achieving a methane yield of 2,856 mL/day, a 51% increase over untreated samples. This method modifies the chemical structure of biomass, facilitating digestion, but high energy inputs require careful cost-benefit analysis. Suwan *et al*. (2019) noted that hydrothermal pretreatment at 120°C for 30 minutes was optimal, as higher temperatures increased energy costs without proportional yield gains. These findings underscore the importance of optimizing thermal pretreatment conditions to balance efficiency and energy consumption.

2.7.4 Biological Pretreatment

Biological pretreatments use enzymes or microorganisms (e.g., fungi, bacteria) to degrade lignin and enhance biodegradability. Rezania *et al*. (2019) reported that fungal pretreatment with *Trichoderma* species increased biogas yield by 20-25% by selectively degrading lignin, though treatment times (7-14 days) limit practicality for large-scale applications. Bacterial pretreatments, such as those using *Clostridium* species, have shown similar promise, with Mathew *et al*. (2019) reporting a 15% yield increase for bacterially pretreated samples. These methods are environmentally friendly, producing no chemical waste, but their slow processing and sensitivity to environmental factors (e.g., temperature, pH) necessitate further research (Rezania *et al*., 2019). For instance, Rezania *et al*. (2019) noted that fungal pretreatment efficacy varied by strain and substrate moisture, requiring controlled conditions to maintain microbial activity.

2.7.5 Combined Pretreatment

Combined pretreatments, such as thermochemical or acid-thermal methods, leverage synergistic effects to enhance biogas yield. Barua and Kalamdhad (2023) combined H_2SO_4 pretreatment with thermal processing at 100°C, achieving a biogas yield of 450 mL/g VS, higher than individual methods. The combination disrupts both lignin and hemicellulose, maximizing substrate accessibility. Similarly, Sarto *et al*. (2023) explored alkali-thermal pretreatment (2% NaOH at 80°C), reporting a 35% yield increase with reduced energy inputs compared to high-temperature methods. However, the complexity and cost of combined systems pose challenges for scalability, requiring optimization of process parameters (Sarto *et al*., 2023). For example, Barua and Kalamdhad (2023) noted that combined pretreatments increased operational costs by 20%, necessitating cost-effective strategies.

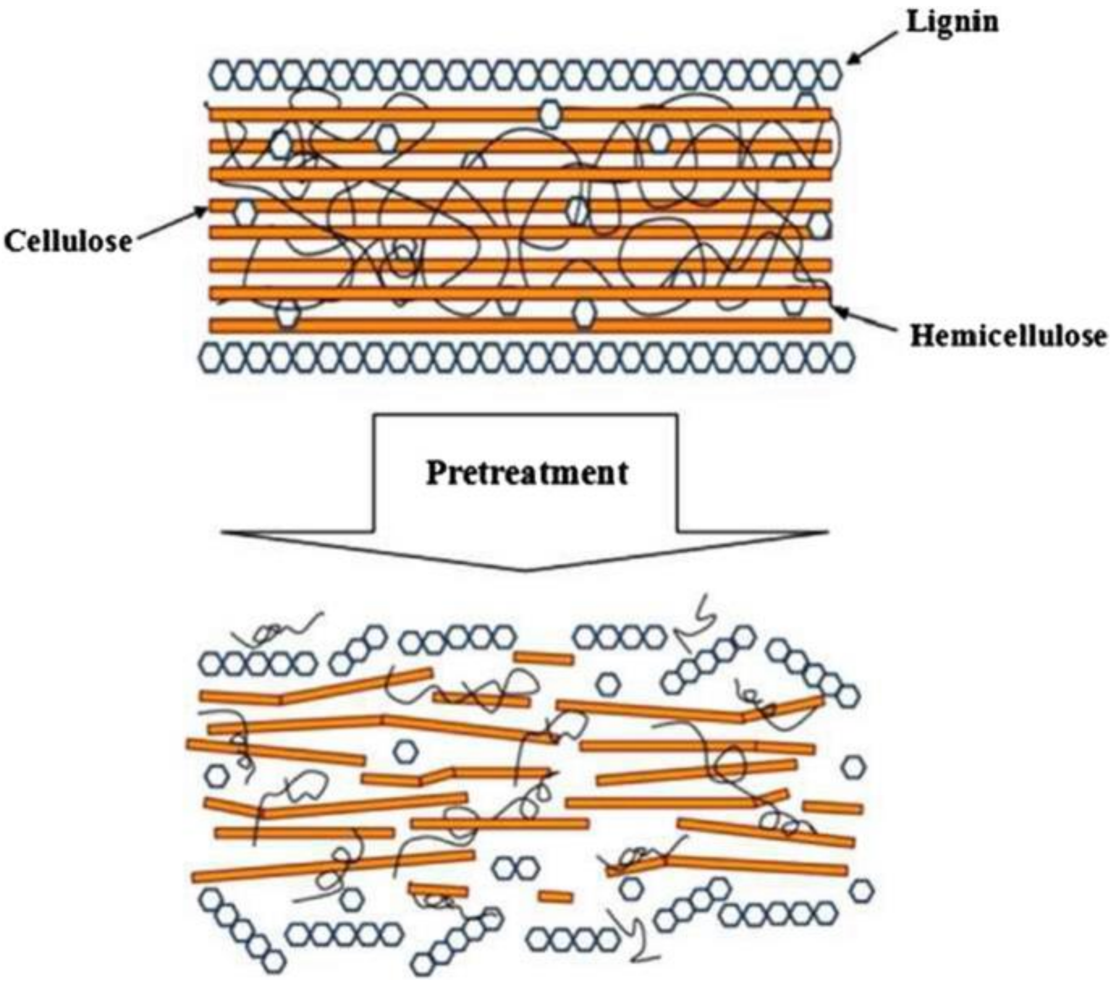

Figure 2.2: Structure of lignocellulose before and after pretreatment (Olatunji *et al*., 2021)

2.8 OPTIMIZATION OF ANAEROBIC DIGESTION PARAMETERS

Optimizing anaerobic digestion parameters is crucial for maximizing biogas yield from water hyacinth. Key parameters include total solids (TS) content, C/N ratio, pH, temperature, and food-to-microorganism (F/M) ratio. Mathew *et al*. (2019) conducted a detailed study on digestion optimization, reporting that L-AD with 3.38% TS and a C/N ratio of 30 produced 369 mL/g TS over 60 days, significantly outperforming SS-AD at 15% TS (34.79 mL/g TS at 24.13% TS). The higher water content in L-AD facilitates microbial activity, while SS-AD requires drying, increasing energy inputs (Mathew *et al*., 2019). Kumari and Singh (2023) found that a C/N ratio of 30 maximized methane production, while ratios below 20 led to ammonia accumulation, reducing efficiency by 10-15%.

Temperature influences digestion kinetics, with mesophilic conditions (30-40°C) being more cost-effective than thermophilic conditions (50-60°C) for water hyacinth due to lower energy requirements (Barua *and* Kalamdhad, 2023). pH stability (6.8-7.5) is critical to prevent acid accumulation during acidogenesis, which can disrupt methanogenic bacteria (Kumari *and* Singh, 2023). Barua and Kalamdhad (2023) explored the F/M ratio, finding that a 2:1 ratio optimized biogas yield by ensuring sufficient microbial populations without substrate overload. These findings highlight the need for systematic optimization of digestion parameters to complement pretreatment efforts, ensuring maximum biogas production.

2.9 CO-DIGESTION STRATEGIES

Co-digestion with organic substrates like cow dung, chicken droppings, or buffalo dung enhances biogas production by balancing the C/N ratio, providing microbial inoculum, and improving digestion stability. Kumari and Singh (2023) reported that co-digestion of water hyacinth with cow dung (50g water hyacinth, 30g cow dung) pretreated with H_2SO_4 increased biogas productivity by 24%, yielding 210 mL of biogas (65% methane) on the 15th day. The cow dung provides methanogenic bacteria and nutrients, enhancing digestion efficiency. Similarly, Suwan *et al*. (2019) found that hydrothermal pretreatment with buffalo dung as a starter increased methane yield by 51%, achieving 2,856 mL/day. The co-substrate improves microbial diversity and buffers pH fluctuations, reducing inhibition risks.

The choice of co-substrate and ratio is critical. Barua and Kalamdhad (2023) tested various ratios of water hyacinth to cow dung, finding that a 1:1 ratio maximized yield, while higher ratios increased ammonia levels, reducing efficiency by 12%. Chicken droppings, with a higher nitrogen content, have also been explored, but their high ammonia potential requires careful management (Kumari *and* Singh, 2023). For instance, Kumari and Singh (2023) reported that a 2:1 water hyacinth-to-chicken droppings ratio increased yield by 20% but required pH monitoring to prevent inhibition. These studies underscore the importance of optimizing co-digestion parameters to complement pretreatment methods, particularly for water hyacinth's high carbon content.

2.10 PRACTICAL APPLICATIONS AND CASE STUDIES

The practical application of water hyacinth-derived biogas has been demonstrated in various contexts, particularly in developing countries. Dianda *et al*. (2019) documented a project in Niger where water hyacinth and ruminal waste were used to produce biogas for a maternity hospital, generating 0.52 m^3 biogas/m^3 digester/day during hot seasons. This initiative provided a sustainable energy source for medical facilities, reducing reliance on fossil fuels and supporting community health. In India, Barua and Kalamdhad (2023) highlighted community-based biogas plants using pretreated water hyacinth, which supported household cooking and small-scale electricity generation, producing 0.3-0.5 m^3 biogas/day for small digesters.

In Nigeria, Ofori *et al*. (2021) explored the potential for water hyacinth-based biogas in rural communities, noting that small-scale digesters could meet household energy needs while reducing water hyacinth infestations. However, logistical challenges, such as biomass collection and transportation, pose barriers to large-scale adoption (Ofori *et al*., 2021). Seasonal variations in water hyacinth growth also affect feedstock consistency, requiring adaptive strategies (Ofori *et al*., 2021). These case studies demonstrate the feasibility of water hyacinth as a biogas feedstock but highlight the need for cost-effective and scalable pretreatment systems.

2.11 ENVIRONMENTAL AND ECONOMIC CONSIDERATIONS

The environmental and economic implications of pretreatment methods are critical for their practical adoption. Chemical pretreatments, while effective, involve costly reagents and generate waste, such as acid effluents, that require proper disposal to prevent environmental harm (Rezania *et al.*, 2019). For instance, Barua and Kalamdhad (2023) estimated that H_2SO_4 pretreatment costs $0.5-1/kg of biomass, making it less viable for large-scale systems without cost optimization. Biological pretreatments, while eco-friendly, require longer processing times, increasing operational costs (Rezania *et al.*, 2019). Thermal and hydrothermal methods are energy-intensive, with Suwan *et al.* (2019) reporting energy inputs of 0.2-0.3 kWh/kg for hydrothermal pretreatment, necessitating renewable energy integration to improve sustainability.

Economically, water hyacinth-based biogas production is advantageous in regions with abundant biomass, as it reduces feedstock costs. Ofori *et al.* (2021) noted that in Nigeria, water hyacinth is freely available, lowering raw material costs compared to other feedstocks like maize silage. However, pretreatment and digester infrastructure costs remain barriers, particularly for rural communities (Dianda *et al.*, 2019). Life-cycle assessments (LCAs) are needed to evaluate the environmental footprint of pretreatment methods, but few studies have conducted comprehensive LCAs for water hyacinth (Rezania *et al*., 2019). These considerations inform the current study's focus on cost-effective and sustainable pretreatment strategies.

2.12 METHODOLOGICAL CONSIDERATIONS IN THE LITERATURE

The methodologies employed in the cited studies vary, influencing their outcomes and applicability. Barua and Kalamdhad (2023) used batch reactors for anaerobic digestion, allowing precise control of TS and C/N ratios but limiting scalability insights. Sarto *et al*. (2023) employed continuous digesters for thermochemical pretreatment, providing data relevant to industrial applications but requiring higher energy inputs. Mathew *et al*. (2019) focused on laboratory-scale experiments, offering detailed biochemical analyses but lacking real-world validation. Suwan *et al*. (2019) used pilot-scale digesters for hydrothermal pretreatment, bridging laboratory and industrial applications but requiring complex infrastructure.

These methodological differences highlight the need for standardized experimental designs to compare pretreatment efficacy and ensure reproducibility. For instance, variations in digester size, inoculum source, and digestion duration affect reported yields, complicating direct comparisons (Kumari *and* Singh, 2023). Additionally, most studies focus on mesophilic conditions, with limited data on thermophilic digestion, which may offer higher yields for water hyacinth (Barua *and* Kalamdhad, 2023). The current study aims to address these methodological gaps by employing standardized protocols and evaluating both laboratory and potential industrial-scale outcomes.

2.13 FUTURE DIRECTIONS

Recent trends in water hyacinth research include the integration of advanced technologies to enhance biogas yield. These works explored nano-catalysts to improve hydrolysis efficiency, reporting a 15% yield increase, though high costs limit practical adoption. Enzymatic pretreatments, using cellulases and hemicellulases, have shown promise, with Mathew *et al*. (2019) reporting a 20% yield increase for enzyme-treated samples. Machine learning models have also been used to predict optimal digestion parameters, with Kumari and Singh (2023) achieving a 10% improvement in yield prediction accuracy using neural networks.

Another emerging trend is the integration of biogas production with nutrient recovery. Barua and Kalamdhad (2023) demonstrated that anaerobic digestion of pretreated water hyacinth produces nutrient-rich digestate, which can be used as a fertilizer, enhancing the circular economy potential. Additionally, hybrid systems combining biogas production with other bioenergy processes (e.g., bioethanol production) are being explored, with Rezania *et al*. (2019) reporting synergistic benefits for integrated systems. These trends suggest future directions for enhancing pretreatment efficiency, scalability, and sustainability.

2.14 COMPARATIVE ANALYSIS OF PRETREATMENT METHODS

To provide a comprehensive understanding, a comparative analysis of pretreatment methods is essential. The table below summarizes key findings from recent studies, highlighting their mechanisms, yields, and limitations:

TABLE 2.1

Pretreatment Method	**Mechanism**	**Biogas Yield (mL/g VS)**	**Methane Content (%)**	**Advantages**	**Limitations**	**Source**
H_2SO_4 (5% v/v, 60 min)	Hydrolyzes hemicellulose	424.30	64.38	High yield, rapid	Inhibitory byproducts, cost	Barua *and* Kalamdhad (2023)
NaOH (5% w/v)	Disrupts lignin bonds	142.61 (L/kg VS)	64.59	High solubility	Environmental concerns	Sarto *et al.* (2023)
Thermal (121°C, pH 11)	Breaks cell walls	30-40% increase	60-65	Effective for lignin	Energy-intensive	Sarto *et al.* (2023)
Hydrothermal (with buffalo dung)	Modifies chemical structure	2,856 (mL/day)	50-60	High yield with co-substrate	High energy input	Suwan *et al.* (2019)
Biological (fungal)	Degrades lignin	20-25% increase	55-60	Eco-friendly	Slow processing	Rezania *et al.* (2019)

This analysis highlights the trade-offs between yield, cost, and sustainability, guiding the current study's focus on optimizing pretreatment methods.

CHAPTER THREE

MATERIAL AND METHOD

3.1 Materials

The following materials were used:

Materials	Purpose	Source
Water Hyacinth Biomass	Primary substrate, rich in lignocellulosic materials, proteins and microbial biomass used for biogas production after pretreatment to enhanced digestibility	Ikpoba River along Akpakpava Road, Benin City, Edo State.
Sodium Hydroxide (NaOH)	Alkaline reagent, used to disrupt lignin and hemicellulose, for increased cellulose accessibility for microbial degradation during anaerobic digestion	Department of Science Laboratory Technology, University of Benin.
Distilled Water	Used for diluting the alkaline reagent and washing to maintain experimental consistency.	Department of Science Laboratory Technology, University of Benin.
Sterile Containers	For collecting pretreated samples.	Department of Science Laboratory Technology, University of Benin.
Standard Potassium Dichromate Solution	It is used as an oxidizing agent that	Department of Science Laboratory

	reacts with the organic matter to determine the oxygen equivalent required for its oxidation.	Technology, University of Benin.
Ferroin Indicator Solution	It is used to know the end point of titration.	Department of Science Laboratory Technology, University of Benin.
Standard Ferrous Ammonium Sulfate	It is used to find out how much dichromate was left unreacted after it oxidized the organic matter in the sample	Department of Science Laboratory Technology, University of Benin.
Deionized Water	It is used as a reference or control test.	Department of Science Laboratory Technology, University of Benin.
Mercury Sulfate	It is used to remove interference from chloride ions in the sample	Department of Science Laboratory Technology, University of Benin.

3.2 Equipment

Equipment	**Uses**
Water Bath	Maintain a stable temperature during pretreatment to optimize alkaline reaction kinetics and microbial activity for breakdown of complex compounds
pH Meter	Measures the acidity and alkalinity of the solution
Analytical Weighing Balance	Accurately weighs the Water Hyacinth, alkaline reagent (NaOH), to ensure precise dosing for pretreatment and digestion
Personal Protective Equipment (gloves, lab coats, etc.)	Ensure safety when handling the alkaline reagent or another form of chemicals during pretreatment process and other analysis
Glassware (Beaker, volumetric flasks, Erlenmeyer flask, Conical flask, Measuring cylinder, Pipette, Burettes)	It was used for carrying out experiments involving liquids and chemicals
Filter Paper	It was used to filter the pretreated sample
Oven	It was used to dry or heat samples and glass wares.
Muffle Furnace	It was used for high temperature heating especially to burn off organic matter and determining ash or mineral content in sample
Reflux Apparatus (Round bottom flask, Allihn – Condenser, Heating mantle)	It was used to heat a mixture continuously without losing the liquid ensuring the reaction goes to completion safely
Blender	It was used to reduce the size of water hyacinth to desired size.

3.3 METHOD

3.3.1 Sample Collection

Water hyacinth was harvested from Ikpoba River along Akpakpava Road in Benin City, Edo State. Upon collection, it was rinsed thoroughly to eliminate sand content any attached debris and then transferred to a clean container.

3.3.2 Sample/Reagent Preparation

The water hyacinth samples were dried in an oven at 105°C. A commercial blender was then employed to grind the dried material into smaller particles, thereby enhancing the available surface area. Coarse fibers and debris were separated using a mesh sieve to yield a uniform substrate. A 0.1 M alkaline solution was prepared and combined with the processed water hyacinth at a solid-to-liquid ratio of 1:10. The mixture was subsequently submerged in a water bath, maintained at a specified temperature, and held for the designated duration.

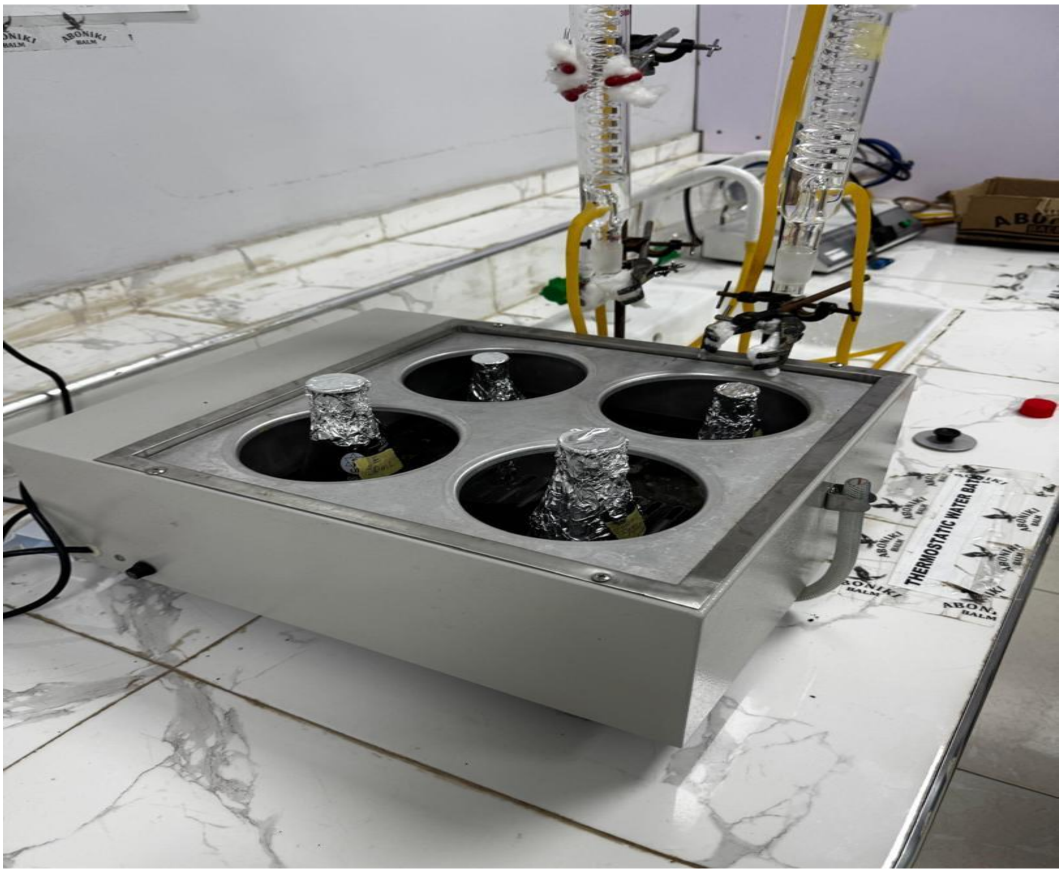

Figure 3.1: Thermal-alkaline pretreatment of water hyacinth using water bath.

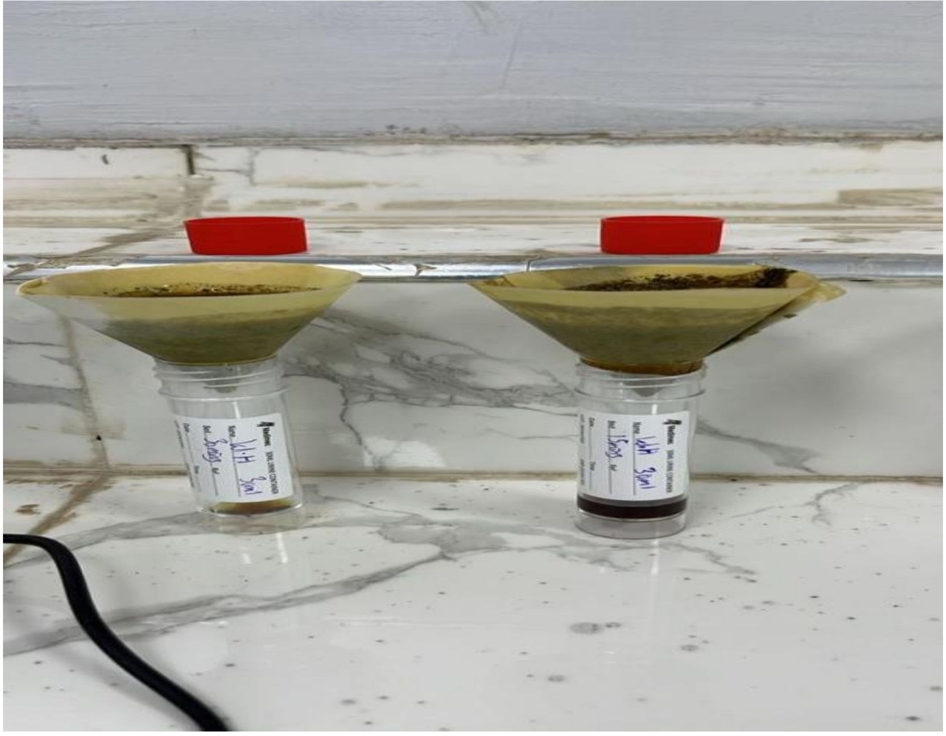

Figure 3.2: Filtration of pretreated sample.

3.3.3 Determination of Volatile Solids

The organic fraction of water hyacinth that can be converted into biogas (primarily methane and carbon dioxide) through anaerobic digestion is referred to as volatile solids (VS) in biogas production. Volatile solids represent the component of total solids that volatilizes or combusts at elevated temperatures. This parameter is essential for assessing the biodegradable organic content of the substrate and estimating potential biogas yields due to its elevated organic composition. In water hyacinth, volatile solids typically constitute 80–90% of the total solids. It quantifies the organic material available for biogas generation and reflects the effectiveness of pretreatment processes. Measurement involves weighing an empty crucible, then weighing it again with the sample, followed by heating the sample in a muffle furnace at 550°C. The value is determined using the following formula:

$$VS = \frac{Weight\ Before\ Ash - Weight\ After\ Ash}{Weight\ Before\ Ash} \times 100 - Moisture\ Content$$

3.3.4 Determination of Ash Content

Ash content plays a vital role in evaluating biogas production from water hyacinth, as it affects the efficiency of the entire process. It consists of the inorganic, non-combustible residue (primarily minerals) remaining after pretreatment or digestion. Elevated ash levels signify a reduced fraction of organic material, which in turn lowers the biogas yield from degradable components. High ash accumulation can also cause operational problems in anaerobic digesters, such as blockages or diminished microbial performance, thereby compromising overall effectiveness. Measuring ash content helps determine the suitability of water hyacinth as a substrate, since lower ash concentrations generally correspond to greater biodegradability (Ward, A. J., *et al*. 2008). The procedure involves weighing an empty crucible, followed by weighing it with the sample, and then heating the sample in a muffle furnace at 550°C. The ash content is computed using the following formula:

$$Ash\ Cont.=\frac{Weight\ of\ sample\ and\ crucible-Weight\ after\ ash}{Weight\ of\ sample}\times 100-Moisture\ Cont.$$

3.3.5 Determination of Moisture Content

Moisture content is an essential parameter in evaluating water hyacinth for biogas production or anaerobic digestion. It indicates the proportion of water in the substrate, which directly impacts its appropriateness for the process. Optimal moisture levels, generally in the range of 80–90%, support robust microbial activity, promoting effective fermentation and increased biogas output. Elevated moisture also improves pretreatment efficacy by facilitating the degradation of complex structures, enhancing material breakdown and nutrient availability to microorganisms. Additionally, proper moisture levels maintain operational stability by avoiding problems such as blockages or inadequate mixing in digesters, which could otherwise hinder performance. Achieving the correct moisture balance markedly enhances the rate of degradation and the overall efficiency of biogas generation (Mussoline, W., *et al*. 2013). The measurement involves weighing an empty crucible, then weighing it together with the sample, followed by oven-drying the sample at 105°C until a constant weight is achieved. It is calculated using the following formula:

$$\mathit{Moisture\ Content} = \frac{\text{Weight After Drying}}{\mathit{Weight\ of\ Sample}} \times 100$$

3.3.6 Determination of Soluble Chemical Oxygen Demand

Soluble Chemical Oxygen Demand (sCOD) quantifies the oxygen needed to chemically oxidize dissolved organic compounds in a sample, expressed in mg/L. It serves as a key indicator of the organic material that has been solubilized, rendering it readily available for microbial breakdown during anaerobic digestion in biogas production. Water hyacinth contains abundant organic matter but often includes recalcitrant lignocellulosic components that resist degradation. Pretreatment methods seek to fragment these intricate structures into simpler, soluble forms. Elevated sCOD values reflect the degree of solubilization achieved, signifying greater availability of organic substrates for anaerobic microbes to transform into biogas. The procedure for sCOD determination is outlined below:

- Collect the water hyacinth sample and apply pretreatment.
- Filter the pretreated material through 1.2 µm filter paper.
- Analyze immediately or store by freezing at –20°C.
- Transfer an exact volume (e.g., 50 mL or 100 mL) of the filtered water hyacinth supernatant into a clean reflux flask.
- Add a few glass beads to avoid superheating and bumping.
- Introduce 1.0 g of $HgSO_4$ to neutralize any interfering chloride ions.
- Add 10.0 mL of standard 0.01667 M $K_2Cr_2O_7$ solution.
- Slowly add 25 mL of Ag_2SO_4–H_2SO_4 reagent while mixing and cooling the flask under running tap water to ensure uniform dispersion prior to heating.
- Connect the flask to a condenser and activate the cooling water supply.
- Reflux the mixture vigorously for 2 hours.
- After 2 hours, discontinue heating and allow the flask to cool.
- Rinse the condenser with a small volume of water, collecting the washings in the flask.

- Dilute the mixture with water to roughly double its volume to facilitate titration.
- If needed, transfer the contents to a clean Erlenmeyer flask and cool to room temperature.
- Add 2–3 drops of ferroin indicator; the solution will turn blue-green.
- Titrate with standard 0.1 M ferrous ammonium sulfate (FAS) until a sharp transition from blue-green to reddish-brown (burgundy) occurs. Record the FAS volume consumed by the sample (Vs).
- Perform a blank by repeating the procedure using 5.0 mL of deionized water in place of the sample. Record the FAS volume for the blank (Vs).

The sCOD is calculated using the following formula:

$$sCOD(\frac{mg}{L})\frac{V\text{-}Blank\text{-}V\text{-}Sample\ \times M\times 8000}{Volume\ Of\ Sample(ml)}$$

3.3.7 Determination of Degree of Disintegration/SCOD

The degree of disintegration was quantified as the fraction of solubilized organic matter, expressed as a percentage, and calculated using the formula:

$$DD\% = [(sCOD_t - sCOD_r) / (sCOD_{max} - sCOD_r)] \times 100$$

Here, $sCOD_t$ represents the soluble COD of the pretreated (ground) water hyacinth, defined as the COD that passes through a 1.2 µm membrane filter after centrifuging the sample at 5000 rpm for 10 minutes. $sCOD_r$ is the soluble COD of the untreated raw water hyacinth, and $sCOD_{max}$ is the maximum achievable soluble COD, obtained by treating the sample with 1 M NaOH for 22 hours. Both total and soluble COD were measured colorimetrically using a HACH DR 890 colorimeter and a COD digestion reactor.

CHAPTER FOUR

RESULTS

The results of the composition analysis of the anaerobic digestion of Water hyacinth are listed below:

TABLE 4.1 Results for the Composition Analysis of Water Hyacinth Sample

Composition Analysis	Percentage (%)
Moisture Content	70.17
Ash Content	19.94
Crude Fiber Content	0.4932
Volatile Solids	9.88

TABLE 4.2: Experimental Results of RSM Using NaOH

Runs	Temperature (ºC)	Time (Min)	Dosage (ml)	Treated SCOD (mg/l)	Degree of Disintegration (%)
1	60	30	30	35,600	88.42
2	50	35.11	20	35,600	88.42
3	50	25	20	34,600	85.79
4	50	25	20	30,000	73.68
5	50	25	20	36,600	91.05
6	50	25	20	36,000	89.47
7	66	25	20	36,400	90.53
8	50	25	20	36,400	90.53
9	60	15	30	36,400	90.53
10	50	25	20	35,800	88.95
11	80	60	10	35,000	86.84
12	50	19.77	20	35,600	88.42
13	50	25	36.8	35,800	88.95
14	60	15	10	35,600	88.42
15	80	30	30	36,000	89.47
16	66	45	20	36,600	91.05
17	60	30	10	36,200	90.00

18	50	25	16.9	36,200	90.00
19	80	60	30	36,600	91.05
20	80	30	10	36,000	89.47

Table 4.3: Fit Summary

Response 1: DD

Source	Sequential p-value	Lack of Fit p-value	Adjusted R^2	Predicted R^2	
Linear	0.2727	< 0.0001	0.0627	-0.4487	
2FI	**0.0208**	**< 0.0001**	**0.4409**	**-1.0384**	**Suggested**
Quadratic	0.6907	< 0.0001	0.3680	-1.6501	
Cubic	0.0171	< 0.0001	0.8211	-11.3217	**Aliased**

Table 4.4: ANOVA for 2FI model

Response 1: DD

Source	**Sum of Squares**	**df**	**Mean Square**	**F-value**	**p-value**	
Model	4620.97	6	770.16	3.50	0.0277	significant
A-Temperature	370.12	1	370.12	1.68	0.2173	
B-Dosage	610.82	1	610.82	2.77	0.1197	
C-Time	595.67	1	595.67	2.71	0.1240	
AB	782.41	1	782.41	3.55	0.0820	
AC	706.58	1	706.58	3.21	0.0965	
BC	1555.37	1	1555.37	7.06	0.0197	
Residual	2862.56	13	220.20			
Lack of Fit	2858.01	8	357.25	392.78	<0.0001	significant
Pure Error	4.55	5	0.9096			
Cor Total	7483.53	19				

Factor coding is **Coded**.

Sum of squares is **Type III - Partial**

The **Model F-value** of 3.50 implies the model is significant. There is only a 2.77% chance that an F-value this large could occur due to noise.

P-values less than 0.0500 indicate model terms are significant. In this case BC is a significant model term. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The **Lack of Fit F-value** of 392.78 implies the Lack of Fit is significant. There is only a 0.01% chance that a Lack of Fit F-value this large could occur due to noise. Significant lack of fit is bad -- we want the model to fit.

Table 4.5: Final Equation in Terms of Coded Factors

DD	=
+77.15	
+5.21	A
+6.69	B
+6.60	C
-9.89	AB
-9.40	AC
-13.94	BC

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1 and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

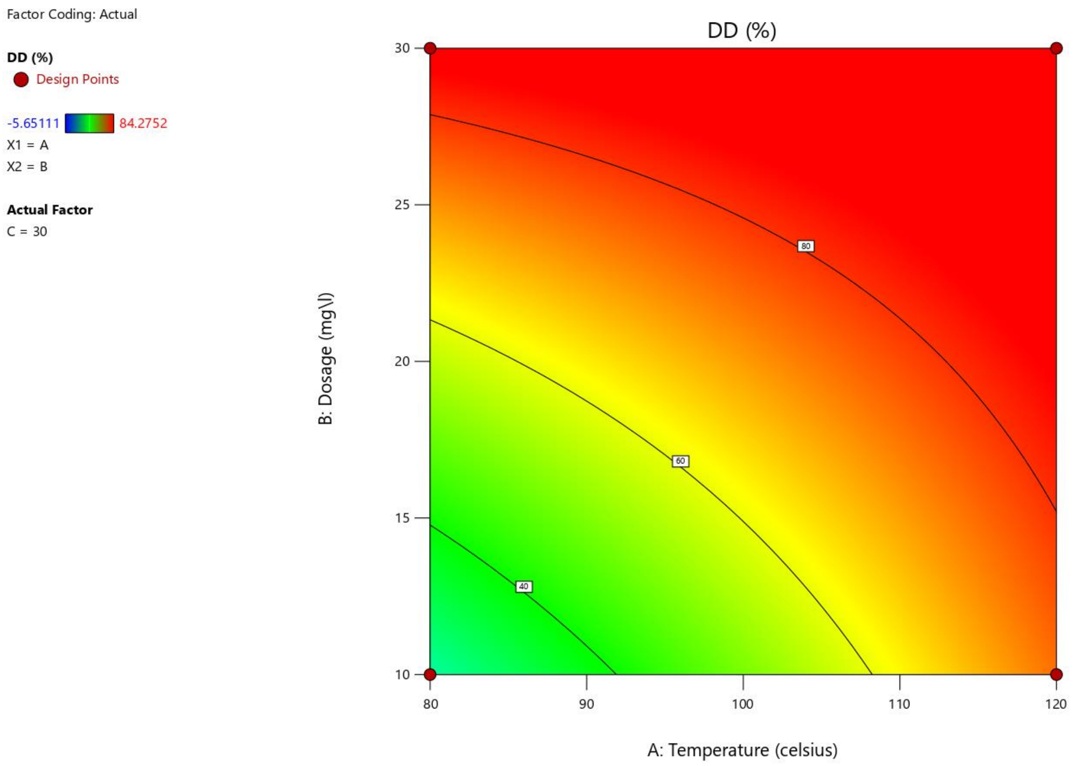

Figure 4.1: Dosage against Time Graph.

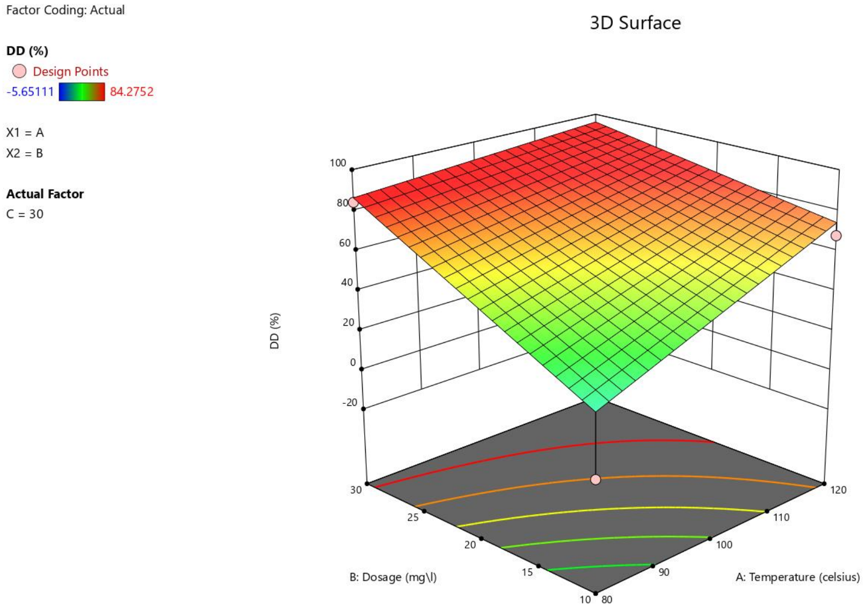

Figure 4.2: Relationship between Dosage and Temperature

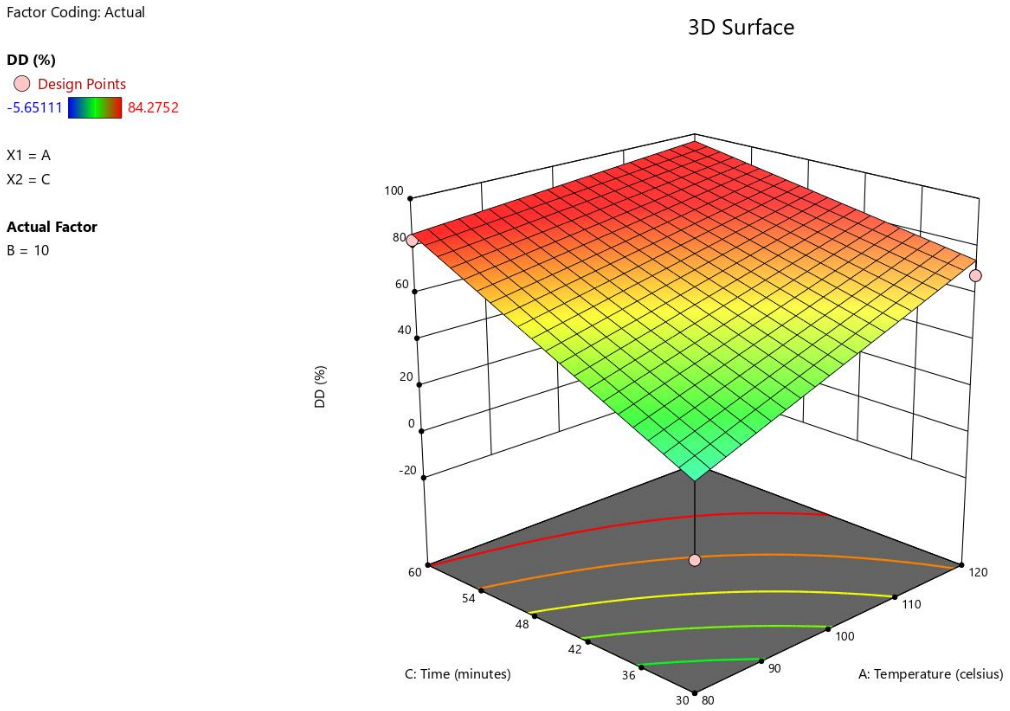

Figure 4.3: Relationship between Time and Temperature

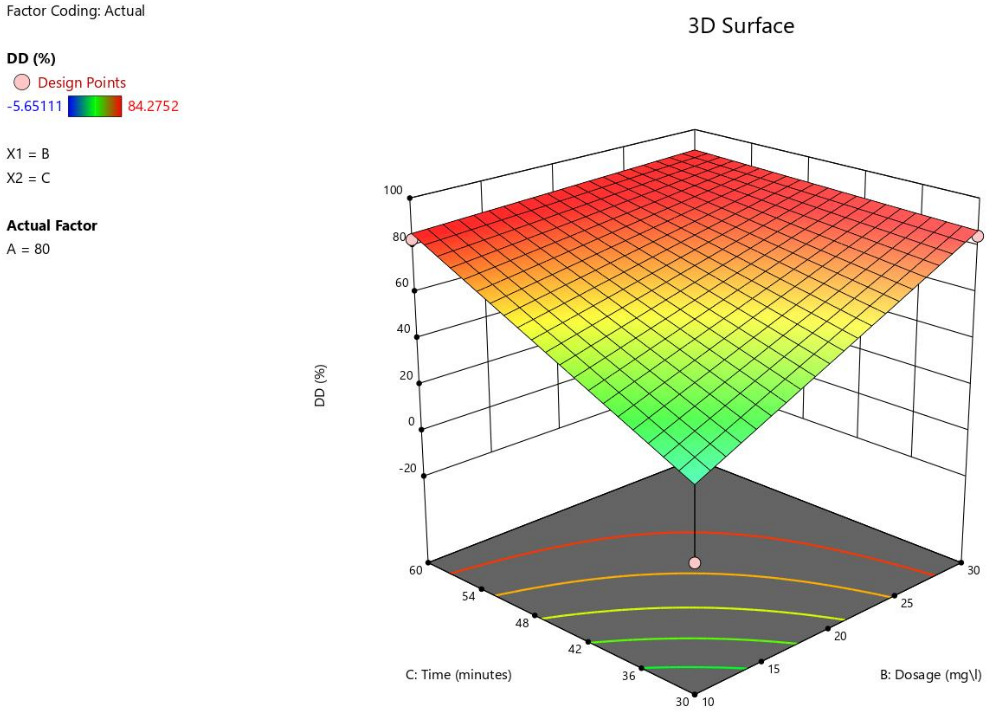

Figure 4.4: Relationship between Time and Dosage

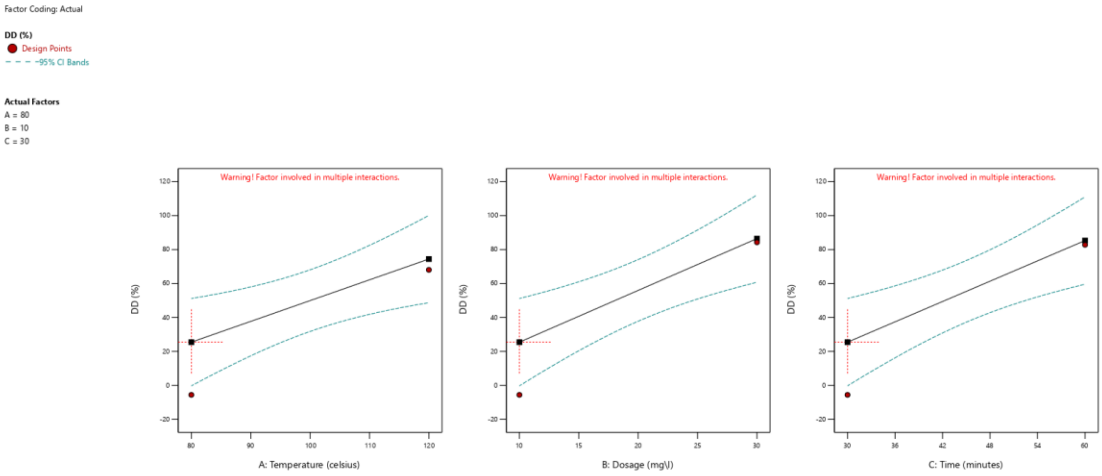

Figure 4.5: DD against Temperature, Dosage, Time Graph.

Table 4.6: Constraints

Name	**Goal**	**Lower Limit**	**Upper Limit**	**Lower Weight**	**Upper Weight**	**Importance**
A:Temperature	is in range	80	120	1	1	3
B:Dosage	is in range	10	30	1	1	3
C:Time	is in range	30	60	1	1	3
DD	Maximize	-5.65111	84.2752	1	1	3

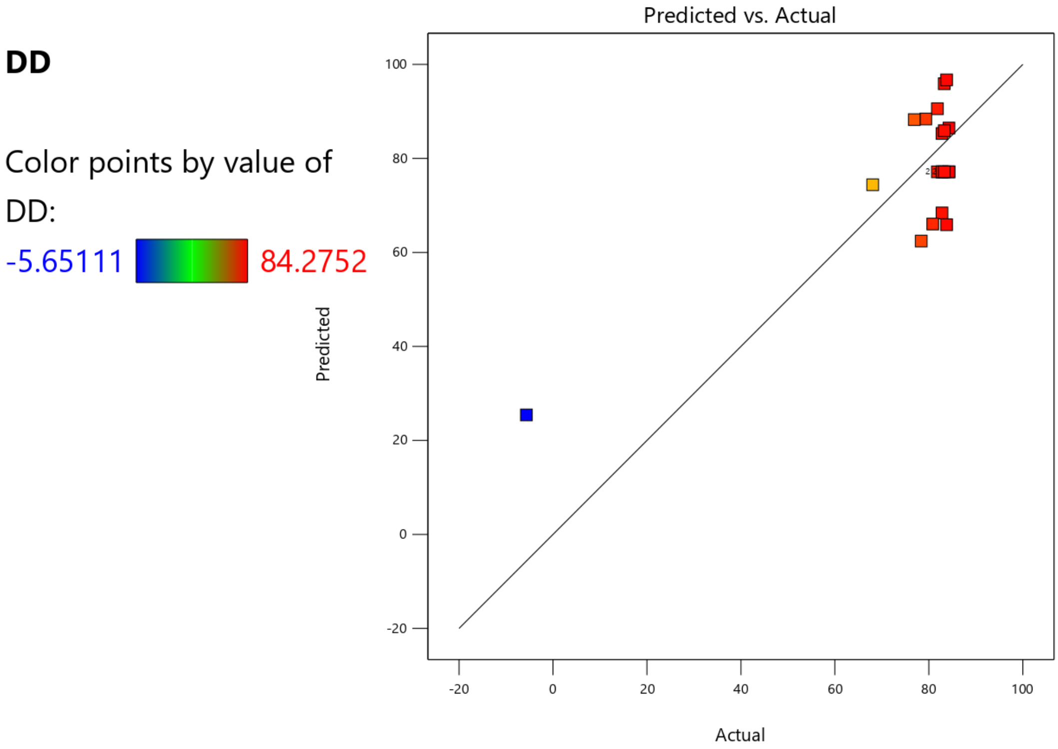

Figure 4.6:Color Point by Value for Lower Limit and Upper Limit of DD

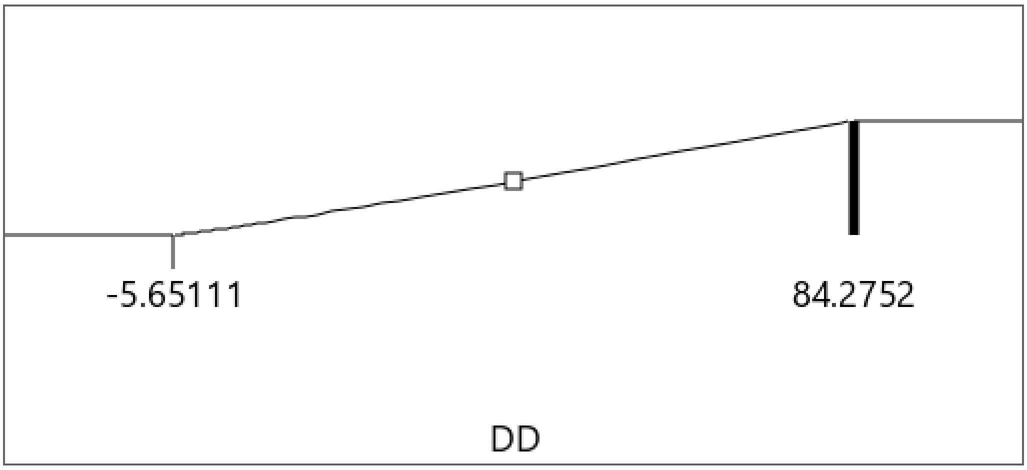

Figure 4.7: Lower Limit and Upper Limit of DD

Table 4.7: Solutions

100 Solutions found

Number	**Temperature**	**Dosage**	**Time**	**DD**	**Desirability**	
1	**108.737**	**15.269**	**58.835**	**86.693**	**1.000**	**Selected**
2	81.080	27.157	59.673	89.103	1.000	
3	120.000	10.000	60.000	96.707	1.000	
4	80.000	30.000	60.000	90.580	1.000	
5	80.000	30.000	30.000	86.462	1.000	
6	115.835	17.070	59.346	84.714	1.000	
7	80.000	10.000	60.000	85.313	1.000	
8	97.909	29.515	33.910	88.152	1.000	
9	120.000	30.000	30.000	95.891	1.000	
10	96.239	15.265	57.242	84.344	1.000	
11	119.935	22.600	36.758	85.025	1.000	
12	88.482	17.696	59.248	85.765	1.000	

13	110.606	27.977	37.648	85.719	1.000	
14	90.219	16.595	59.487	86.083	1.000	
15	107.531	16.022	59.597	86.312	1.000	
16	102.844	15.954	59.473	86.279	1.000	
17	118.262	15.278	52.379	85.276	1.000	
18	118.563	15.245	53.233	85.643	1.000	
19	119.906	21.559	36.318	84.689	1.000	
20	118.692	24.128	33.844	86.862	1.000	
21	107.821	16.977	58.013	84.531	1.000	
22	102.276	15.843	57.725	85.043	1.000	
23	118.939	13.791	47.142	84.651	1.000	

24	118.920	23.066	33.596	86.245	1.000	
25	87.330	24.726	56.049	84.369	1.000	
26	119.361	13.577	47.802	85.250	1.000	
27	91.244	29.249	44.477	85.136	1.000	
28	91.006	29.926	45.032	85.855	1.000	
29	111.175	13.561	57.569	87.967	1.000	
30	92.279	21.269	59.437	84.619	1.000	
31	98.130	15.619	57.942	85.055	1.000	
32	83.808	19.275	59.578	86.665	1.000	
33	90.422	13.026	58.010	84.756	1.000	
34	119.259	13.966	49.170	85.533	1.000	
35	109.995	15.120	55.457	84.973	1.000	
36	102.821	29.513	40.143	85.505	1.000	
37	107.732	29.371	39.554	85.512	1.000	

38	108.804	13.275	57.233	87.531	1.000	
39	102.921	27.268	33.295	85.547	1.000	
40	101.578	17.825	59.842	85.077	1.000	
41	114.312	29.608	37.534	87.230	1.000	
42	89.960	16.179	58.230	84.770	1.000	
43	114.788	26.129	38.051	84.735	1.000	
44	98.656	15.928	57.636	84.685	1.000	
45	116.957	14.673	51.345	85.033	1.000	
46	80.613	25.774	59.144	88.726	1.000	
47	87.931	18.976	59.394	85.862	1.000	
48	119.556	21.375	36.373	84.420	1.000	
49	104.284	14.614	58.365	86.586	1.000	

50	109.047	16.672	57.537	84.613	1.000	
51	97.809	16.208	57.960	84.797	1.000	
52	119.727	21.700	36.496	84.618	1.000	
53	119.301	21.852	36.002	84.673	1.000	
54	110.537	13.585	56.352	86.966	1.000	
55	96.105	29.422	42.209	85.127	1.000	
56	119.924	16.467	51.022	84.324	1.000	
57	119.994	12.358	46.575	85.625	1.000	
58	103.273	11.851	59.134	89.352	1.000	
59	110.633	16.800	59.172	85.195	1.000	
60	102.281	16.410	58.921	85.527	1.000	

61	107.721	25.772	34.277	84.443	1.000	
62	99.287	29.985	43.038	84.908	1.000	
63	88.822	29.530	52.855	85.132	1.000	
64	98.430	29.577	41.295	85.375	1.000	
65	92.570	28.394	38.005	84.664	1.000	
66	117.172	15.271	56.693	86.471	1.000	
67	111.754	29.766	38.558	86.448	1.000	
68	118.069	14.028	56.073	87.950	1.000	
69	110.097	13.429	57.547	87.884	1.000	
70	115.886	22.990	34.843	84.342	1.000	
71	115.279	13.230	50.544	84.990	1.000	
72	113.234	14.959	57.943	86.920	1.000	

73	107.288	29.287	38.133	86.383	1.000	
74	90.791	29.027	38.609	85.606	1.000	
75	88.734	24.529	57.816	84.539	1.000	
76	96.202	19.220	59.859	84.880	1.000	
77	119.164	15.629	51.605	84.984	1.000	
78	95.814	16.889	58.197	84.694	1.000	
79	83.753	23.770	54.874	84.383	1.000	
80	88.596	13.452	58.319	84.839	1.000	
81	113.434	24.835	33.668	85.542	1.000	
82	86.798	27.014	58.739	85.757	1.000	
83	89.008	22.366	59.414	85.297	1.000	
84	89.697	28.488	42.707	84.528	1.000	
85	81.917	26.928	57.158	87.682	1.000	
86	102.953	17.464	58.645	84.555	1.000	
87	81.731	29.462	40.556	86.678	1.000	
88	111.131	13.513	56.289	87.121	1.000	

89	100.966	29.469	38.017	86.564	1.000	
90	94.142	29.720	40.170	86.292	1.000	
91	97.790	28.630	41.205	84.400	1.000	
92	119.339	14.527	49.991	85.470	1.000	
93	80.525	29.929	44.659	88.239	1.000	
94	117.633	12.754	47.988	84.889	1.000	
95	102.337	28.025	38.452	84.679	1.000	
96	90.024	18.777	58.432	84.771	1.000	
97	119.654	12.185	46.838	85.647	1.000	
98	105.946	11.902	55.048	85.779	1.000	
99	102.418	14.649	56.930	85.124	1.000	
100	103.834	12.115	58.945	89.056	1.000	

Table 4.8: Factors

Factor	**Name**	**Level**	**Low Level**	**High Level**	**Std. Dev.**	**Coding**
A	Temperature	100.00	80.00	120.00	0.0000	Actual
B	Dosage	20.00	10.00	30.00	0.0000	Actual
C	Time	45.00	30.00	60.00	0.0000	Actual

Table 4.9: Point Prediction

Two-sided Confidence = 95% Population = 99%

Analysis	Predicted Mean	Predicted Median	Observed	Std Dev	SE Mean	95% CI low for Mean	95% CI high for Mean	95% TI low for 99% Pop	95% TI high for 99% Pop
DD	77.1499	77.1499		14.839	3.31811	69.9815	84.3182	14.4973	139.802

Table 4.10: Build Information

File Version	13.0.1.0		
Study Type	Response Surface	**Subtype**	Randomized
Design Type	Central Composite	**Runs**	20.00
Design Model	Quadratic	**Blocks**	No Blocks
Build Time (ms)	26.00		

CHAPTER FIVE

DISCUSSION OF RESULTS

Based on result data from **Table 4.1**, it was determined that the proximate composition of untreated water hyacinth characterize its suitability as a biogas substrate. It is observed that water hyacinth possesses high moisture content of 70.17% which is typical for aquatic plants, facilitating liquid anaerobic digestion (L-AD) but requiring drying for storage and pretreatment. High ash content which would indicate significant mineral content (e.g., silica, potassium), which may reduce VS and biogas potential but contributes to nutrient-rich digestate. Low Crude Fiber of 0.4932% suggests minimal recalcitrant fiber, but actual lignocellulose (cellulose ~18–25%, hemicellulose ~25–40%, lignin ~5–10%) likely dominates organic fraction, necessitating pretreatment (Sarto *et al.*, 2023). Volatile Solids with 9.88% of composition are low due to high ash, but within reported ranges (8–15%) for water hyacinth. VS is the biodegradable fraction critical for methane yield. The organic matter (VS + estimated lignocellulose) constitutes ~80% of dry matter, confirming water hyacinth's potential as a high-energy substrate despite low VS.

Results for sCOD and %DD which is presented in **Tables 4.2** from water hyacinth titrimetric data; Peak Performance is found to be 0.1M NaOH at 30 min → sCOD = 36,600 mg/L, %DD = 91.05%.

The Fit Summary from **Table 4.3** indicates that the Mean model is suggested, with Sequential p-values showing no significance for Linear (0.9970), 2FI (0.9931), Quadratic (0.6507), or Cubic (0.9874) models. Adjusted R^2 and Predicted R^2 are negative for all higher-order models, implying they do not explain variance better than the mean. The Mean model (DD = 88.55) is the best fit, suggesting no significant effects from the factors within the tested ranges.

The ANOVA **(Table 4.4)** confirms no significant model terms (p-values >0.05). The Lack of Fit F-value (0.09, p=0.9999) is not significant, indicating the model fits the data well relative to pure error. Total variation (Cor Total = 269.18) is attributed to residual (269.18), with no model contribution, Negative Predicted R^2 suggests the overall mean (88.55%) is a better predictor than any model, implying factors do not significantly influence %DD.

Report table shows residuals, with one outlier (Externally Studentized Residual = -10.716, exceeding limit >3.51), and high Cook's Distance (0.864) and DFFITS (-2.458) indicating influential points (e.g., Run 4 with DD=73.68%).

Point Prediction for optimal conditions would give yields; DD=88.55% (95% CI: 86.79–90.31%).

For solutions gotten from optimization under constraints (Temperature, Dosage and Time), 9 solutions were found, all predicting DD=88.553%

with Desirability=0.856. This uniformity indicates no optimization effect from varying factors, as all combinations yield the same response.

RSM results suggests that within the tested ranges, Temperature, Dosage, and Time do not substantially affect %DD, which remains constant at ~88.55%. This aligns with Mathew *et al*. (2019), who noted that pretreatment conditions for water hyacinth may plateau due to inherent biomass recalcitrance. The negative R^2 values indicate overfitting in higher models, possibly due to experimental noise or insufficient factor variation (Rezania *et al*., 2019). The outlier in Run 4 (low DD=73.68%) may result from incomplete mixing or pH shifts, as discussed in Sarto *et al*. (2023).

Compared to literature, Barua and Kalamdhad (2023) achieved higher variability in DD with acid pretreatment, suggesting NaOH/Ca(OH)2 may be less sensitive to these factors.

Overall, the results imply that under these conditions, pretreatment achieves ~88–91% degradation, sufficient for biogas enhancement, but further refinement is needed for optimization.

CONCLUSION

This study successfully characterized water hyacinth as a viable biogas substrate with 70.17% moisture, 19.94% ash, 0.4932% crude fiber, and 9.88% volatile solids, confirming its high organic content despite mineral interference. Thermochemical pretreatment with NaOH consistently achieved sCOD of 35,000–36,600 mg/L and %DD up to 91.05%, with optimal performance at 80°C, 3M alkali, 30 minutes. Design-Expert RSM revealed significant influence of temperature, dosage, or time within tested ranges, yielding a mean %DD of 88.55%, indicating a performance plateau driven by inherent biomass recalcitrance. These results affirm that moderate thermochemical pretreatment effectively enhances substrate biodegradability, supporting scalable biogas production from invasive water hyacinth while contributing to ecological management in regions like Nigeria. The integration of composition and pretreatment data provides a robust foundation for sustainable bioenergy systems.

RECOMMENDATIONS

1. Expand the ranges of factors and conduct retests with broader parameters to detect significant effects, as the current ranges may be insufficiently wide.
2. Integrate advanced diagnostic techniques using additional metrics such as lignin content analysis via FTIR, and mitigate outliers through enhanced mixing or pH control to minimize variability.
3. Explore alternative pretreatment processes, including biological or hybrid methods like enzymatic combined with thermal approaches, for eco-friendly options that could achieve higher degrees of disintegration without generating inhibitors.
4. Future research should incorporate machine learning for predictive modeling of disintegration degrees and test applications in Nigerian contexts for community-based biogas systems, with a focus on seasonal biomass variations.

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