

DEVELOPMENT OF A NOVEL STRATEGY FOR CLEAN BIOGAS GENERATION FROM ORGANIC WASTES THROUGH ANAEROBIC DIGESTION PROCESS

A Thesis

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BY

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October 2024

DECLARATION



I hereby certify that the information presented in this thesis entitled **“Development of a Novel Strategy for Clean Biogas Generation from Organic Wastes through Anaerobic Digestion Process”** is entirely my own account of the research performed under the supervision of Dr. Pankaj Kalita and Prof. Pranab Goswami. Any part of this work has not been submitted earlier for the award of any degree, diploma, fellowship or its equivalent to any University or Institution.

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CERTIFICATE

This is to certify that the work presented in the thesis entitled "**Development of a Novel Strategy for Clean Biogas Generation from Organic Wastes through Anaerobic Digestion Process**", submitted by **Shayaram Basumatary**, a student in the School of Energy Science and Engineering, Indian Institute of Technology Guwahati, India for the award of the degree of Doctor of Philosophy has been carried out under our supervision. He has fulfilled all the requirements according to the institute's rules, and the investigations embodied in his thesis have not been submitted elsewhere before for the award of any other degree or diploma.

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ABSTRACT

The global energy demand is gradually rising due to urbanisation, industrialisation and human population growth. However, burning fossil fuels and the organic waste materials produced by the industries, municipal, and agricultural sectors contribute to tremendous environmental impacts like air, soil and water pollution. Likewise, livestock manure and food waste are also responsible for contributing to greenhouse gas (GHG) emissions. Biological waste, such as cattle dung and food waste, are the most abundant waste resources and contribute significantly to producing low-cost and sustainable forms of energy via anaerobic digestion. Anaerobic digestion is a realistic approach for managing waste materials and harnessing renewable energy concurrently. However, various operating parameters like substrate mixing ratio, pH value and temperature affect the anaerobic digestion. Optimising these parameters is crucial to obtain a higher biogas generation rate from the substrates. These parameters validate several biochemical and microbial processes and enhance methane yield. To achieve the appropriate substrate-water (S/W) mixing ratio for cattle dung (CD), vegetable waste (VW) and cooked kitchen waste (CKW), experiments were performed individually at different S/W mixing ratios for each substrate under controlled and uncontrolled temperatures. The ideal S/W ratio was achieved at 50:50 for cattle dung and cooked kitchen waste, whereas it was 60:40 for vegetable waste. Further, controlled digesters ($35\pm 2^\circ\text{C}$) exhibited higher biogas generation rates than uncontrolled digesters (ambient temperature). Co-digestions of vegetable waste and cooked kitchen waste with cattle dung were also studied by varying the co-substrate mixing ratios (SMR) without and adding biochar under controlled and uncontrolled temperatures, considering the ideal S/W ratio 50:50. Biochar from agricultural waste was prepared through pyrolysis process. The primary goal of the investigation is to focus on the effect of SMR, biochar addition, and temperature on AcoD. Considerably, the biochar-added and mesophilic digesters achieved a higher cumulative methane yield (CMY) than the respective digesters

without biochar. This improvement can be attributed to the porous structure of biochar, as well as the presence of macronutrients and metal ions such as Na, Ca, K, and Mg within it. In the case of anaerobic co-digestion (AcoD) of cattle dung and vegetable waste, the maximum cumulative methane yield was detected for the SMR of 60:40 (CD60%: VW40%) for all sets of biochemical methane potential (BMP) tests, where the biochar-added mesophilic digester (SMR 60:40) achieved 13.19% and 39.26% higher than the respective uncontrolled digester with and without biochar for retention period of 45 days. On the other hand, the maximum CMY was achieved for the SMR of 40:60 (CD40%:CKW60%) for all sets of BMP tests in the case of anaerobic co-digestion of cattle dung and cooked kitchen waste. The mesophilic biochar added digester (SMR 40:60) was 14.68% and 32.47% higher than the respective uncontrolled digesters with and without biochar. Consequently, considering the SMR of 60:40 (for AcoD of CD and VW) and SMR of 40:60 (for AcoD of CD and CKW), another set of BMP experiments were performed by adding varying quantities of biochar: 5, 10, 15, 20 and 25 g/L, to identify the most feasible needed amount of biochar to be added for maximum CH₄ generation. However, the digester containing 15 g/L biochar exhibited the highest CMY for both cases, confirming that 15 g/L biochar addition can be recognised as the most effective for AcoD of cattle dung and food waste.

Furthermore, in order to check the feasibility of the optimum S/W 50:50 ratio obtained from a batch study on a lab scale, a field-scale experiment was performed in a 1 m³ capacity anaerobic digestion plant at three different phases. The first phase of experiments was carried out in the summer season without using a solar energy-assisted water heating (SAH) system. Subsequent phases were conducted both without and with the use of a solar energy-assisted water heating system during the winter. An average volume of 6.26 m³/week of biogas was generated during the summer season (first phase) against the loading of 50.00±1.50 kg cattle dung slurry/day. Subsequently, in the third phase of the experiment, the integration of the solar

energy-assisted heating system improved the cumulative biogas yield by an average of 9.93% over the digester without solar energy-assisted heating (second phase) during the winter season. This study confirms that adopting a passive solar energy-assisted heating system for a field-scale anaerobic digestion plant is a novel approach towards enhancing the biogas yield.

The previous study confirmed that biogas production depends on the feedstock used, climatic conditions, and technology adapted for production, which affect the composition of generated biogas. Carbon dioxide and other trace gases, such as hydrogen sulphide, water vapour, etc., must be removed to augment the quality of biogas. In this regard, a purification system was developed by incorporating an adsorbent column. Three different biochars generated from coconut husk, sugar cane bagasse and water hyacinth biochars were pyrolysed at three different pyrolysis temperatures of 550, 450, and 350 °C and were used as adsorbents to remove CO₂ and H₂S and other impurities from the raw biogas. The CO₂ removal efficiency has been found to be 55.02%, 49.65% and 45.22%, respectively, when coconut husk, sugarcane bagasse and water hyacinth-based biochar were used as adsorbent. Amongst several factors affecting CO₂ and H₂S adsorption, the surface area played an important role in adsorption along with the associated factors like higher alkalinity, carbonization temperature and mineral elements on the biochar surface.

Keywords: Anaerobic digestion; Co-substrates mixing ratio; Biochemical methane potential; Solar energy-assisted heating system; Biochar; Biogas purification.

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NOMENCLATURE

Abbreviations

AAE	Alkali and alkaline earth
AC	Ash content
AcoD	Anaerobic co-digestion
AD	Anaerobic digestion
ASTM	American Society for Testing and Materials
BC	Biochar
BET	Brunauer–Emmet–Teller
BMP	Biochemical methane potential
CBY	Cumulative biogas yield
CD	Cattle dung
CH	Coconut husk
CKW	Cooked kitchen waste
CMR	Co-substrates mixing ratio
CMY	Cumulative methane yield
C/N	Carbon/Nitrogen
CSTR	Continuously stirred tank reactor
DBY	Daily biogas yield
DMY	Daily methane yield
EDS	Energy dispersive X-ray spectroscopy
ET	Evacuated tube
FC	Fixed carbon
FT-IR	Fourier transform infrared
FW	Food waste
F/W	Feedstock/water
GC	Gas chromatograph
GHG	Greenhouse gas
HRT	Hydraulic retention time
LPG	Liquid petroleum gas
MC	Moisture content
MGM	Modified Gompertz model
OLR	Organic loading rate

PV	Photovoltaic
SAH	Solar-assisted heating
SB	Sugarcane baggage
SCB	Specific cumulative biogas
SDG	Sustainable development goal
SEM	Scanning electron microscope
SMR	Substrate mixing ratio
SMY	Specific methane yield
S/W	Substrate/water
TS	Total solid
UN	United Nations
USA	United States of America
VFA	Volatile fatty acids
VM	Volatile matter
VS	Volatile solid
VSR	Volatile solid reduction
VW	Vegetable waste
WH	Water hyacinth
WHBC	Water hyacinth biochar
XRD	X-ray diffractometer

Symbols

Y_P	Maximum methane yield potential
Y_R	Highest methane yield rate
Y_C	Accumulated cumulative methane yield
R^2	Coefficient of determination
λ	Lag phase time
t	Time
d	Day
C_{p1}	Specific heat of water
C_{p2}	Specific heat of slurry
m_h	Mass flow rate of hot water
ε	Effectiveness of heat exchanger

t_{h1}	Hot water inlet temperature
t_{h2}	Hot water outlet temperature
t_{c1}	Initial temperature of the slurry
t_{c2}	Final temperature of the slurry
S_{BET}	BET surface area
V_p	Pore volume
d_p	Pore diameter



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CHAPTER 1

INTRODUCTION

This chapter briefly discusses the origin and the problems associated with biological waste, such as cattle dung, food waste (cooked and uncooked), and waste management through anaerobic digestion. The essential parameters necessary for effective anaerobic digestion are also briefly discussed, along with the objectives, and the scope of the study has also been included.

1.1 MOTIVATION

The rapid depletion of the world's energy reserves and environmental pollution are humankind's most significant challenges in this century. The mitigation of carbon dioxide (CO₂) emissions and related global warming demands the search for alternative sources of energy in order to reduce the dependency on fossil fuels. Further, the organic waste materials produced by the industries, municipal, and agricultural sectors contribute to tremendous environmental impacts like air, soil, and water pollution (Abdel-Shafy en Mansour, 2018). Likewise, food waste and livestock manure are also responsible for contributing to greenhouse gas (GHG) emissions. One-third of global food production for consumption gets wasted due to human activities and storage problems, which is about 1.3 billion tons per year (Dhamodharan et al., 2015; FAO, 2011), generating tremendous social, economic and environmental issues (Xu et al., 2018). According to the Food Wastage Footprint and Climate Change, FAO (2014), around 25% of vegetables are wasted globally at the bottom of the production chain (Edwiges et al., 2018). It is also estimated that food waste will sharply increase from 2.78 billion tons to 4.16 billion tons in Asian countries by 2025 (Ren et al., 2018). For a developing country like India, several routes like weddings, canteens, hotels, institutional hostels, social and family

functions, and households contribute to large food waste production. India is the second largest producer of fruits and vegetables in the world (Chakraborty en Venkata Mohan, 2019) which is around 314.5 million tonnes in the year 2018-2019 (The Hindu, 2019) but wasted around 30–40% of its total market production annually. On the other hand, India is also a tremendous source of animal manure. The total livestock population in India is estimated to be about 512.05 million in 2012, out of which bovine and cattle populations are 299.9 million and 190.90 million, respectively (Livestock Census, 2012). Gupta et al. (2016) reported that the majority of cattle in India are cows, which can generate 9–15 kg of dung per day (Gupta et al., 2016). Moreover, around 300 million cows are estimated to produce 3 million tonnes of cattle dung daily in India (The Hindu, 2018).

The landfill of cattle dung or food waste (FW) creates adverse environmental impacts such as the production of leachates, soil and groundwater contamination, the release of toxic gases, etc. (Ren et al., 2018). The primary greenhouse gas (GHG) emissions generated from untreated CD and FW are CH₄, nitrous oxide (N₂O), carbon dioxide (CO₂), and fluorinated compounds, which contribute to climate change (U.S. EPA, 2021). Global food loss and waste contribute approximately 8% of anthropogenic GHGs (4.4 gigatons of CO₂ equivalent annually) (U.S. EPA, 2021). Additionally, the emissions from livestock contribute about 18% (7.1 billion tons CO₂ equivalent) of global GHG releases (Sejian et al., 2016). Investigators conveyed that volatilised ammonia (NH₃) from manure deposits into the aquatic and global ecosystems or transforms into N₂O emissions (Aguirre-Villegas en Larson, 2017). Approximately 80% of agricultural methane and 35% of all anthropogenic methane releases were generated from livestock, equivalent to 2.2 billion tons of CO₂ (Sejian et al., 2016). Therefore, promoting and implementing policies and technologies to reduce and reuse such waste products have become essential. In this regard, the United Nations (UN) adopted the 2030 Agenda for Sustainable Development in 2015. Among the various goals/agenda, UN

Sustainable Development Goal (SDG) 12.3 targeted to halve per capita global food waste at the retail and consumer levels and to reduce food losses along production and supply chains, including post-harvest losses by 2030 (United Nations, 2015). The UN SDG 12.5 also targets to decrease waste generation by 2030, focusing on waste prohibition, minimisation, recycling, and promoting reuse (United Nations, 2015). The European Commission has defined a standard EU methodology for food waste accounting and proposed relevant indicators as a commitment towards SDG 12.3 (Corrado en Sala, 2018). The government of India is also committed to the 2030 agenda, which includes SDGs. NITI Aayog is responsible for monitoring the progress of the SDGs and coordinating them with central and state ministries (NITI Aayog, 2019). Though India has no dedicated policy or framework towards SDG 12, its elements are fused in several policies in different fields (RIS, 2016).

Among the various strategies for reducing emissions, anaerobic digestion (AD) is a highly appropriate and adaptable technology for managing biodegradable waste materials, which converts organic waste into biogas (Appels et al., 2011; Hagos et al., 2017). Production of biogas from waste through AD is one of the most significant challenges of this century. Biogas is an essential renewable fuel for remote locations in developing nations. India, which has a huge generation of food waste and cattle dung, could emphasise biogas production, which could be utilised in different sectors to fulfil the country's increasing energy demand. Anaerobic digestion is a biological treatment process that involves four stages, viz. hydrolysis, acidogenesis, acetogenesis and methanogenesis, in the depletion of oxygen in a digester or reactor (Pramanik et al., 2019). The biogas generated from AD can be used as a fuel for heating, transportation, or co-generating electricity and heat (Cioablă et al., 2017). The International Renewable Energy Agency (IRENA) reported that India's biogas production potential was 14 MW in 2023 (IRENA, 2024). The government of India has also targeted the production of 48.55 MW of energy from biogas plants by 2022 (Kadam en Panwar, 2017). Moreover, the

National Biogas and Manure Management Programme (NBMMP) has reported that about 49,66,628 biogas plants have already been installed in India up to 31st March 2017 against the estimated 1,23,39,300 number of potential for family-type biogas plants (MNRE, 2018). Considering the present scenario, anaerobic digestion for biogas production has gained immense attention worldwide.

Microorganisms play significant roles during the AD process. The growth of microorganisms depends on many factors: temperature, pH levels, degradability of feedstocks, hydraulic retention time (HRT), and the mineral composition within the substrates. The microorganisms grow best at mesophilic and thermophilic temperatures (Mahmudul et al., 2021) and in the pH range of 6.8–7.2 (S. Basumatary et al., 2021). The carbon-nitrogen (C/N) ratio is also an essential factor, signifying the nutrient levels of feedstock for microorganisms. The inhibition of NH_3 increases for the substrate with a low C/N ratio, which is toxic to microorganisms (Mao et al., 2015). Instead, when the C/N ratio is excessively high, the anaerobic bacteria quickly consume nitrogen, causing a lower biogas yield (Kothari et al., 2014). The feedstock's optimum C/N ratio of 20–30 [10] can be maintained by blending the high and low C/N ratio feedstock (Kothari et al., 2014). In addition, CH_4 yield is influenced by the total solid (TS) and volatile solid (VS) present in the slurry. Hence, it is crucial to maintain the ideal ratio of substrate and water during slurry preparation to maximise methane generation. Apart from optimising the various operating parameters, adding some additive materials is essential to increase AD performance, thereby enhancing waste recycling processes. In recent years, the usage of biochar (BC) in the AD process has been reported by various researchers in scientific publications to improve overall AD performance (Chen et al., 2023). The BCs are carbon-rich compounds obtained with pyrolysis at 300–900°C or carbonisation at 170–250°C, respectively (Cha et al., 2016). This pyrolysis process involves biomass's thermochemical decomposition in oxygen depletion. Adding BC to AD's slurry enhances CH_4 yield, improves

operational stability, shortens the microbial growth period, immobilises microbes and upgrades the biogas quality (Chen et al., 2023; Zhao et al., 2021). Several investigators have reported that the high porosity, high surface area, alkalinity nature, existence of functional groups and high electron transfer capability of BC enhances the CH₄ yield in the AD process (Kumar et al., 2021).

1.2 NEED OF THE RESEARCH WORK

The increasing energy demands, rapid depletion of non-renewable fossil fuels, and the necessity to manage organic waste emphasise the importance of developing sustainable alternative energy sources. Organic wastes like cattle dung or food waste contain high moisture and volatile organic solids. The landfill of such organic waste creates adverse environmental impacts due to the formation of leachates, contamination of soil and groundwater, and the emission of harmful gases (Ren et al., 2018). The anaerobic digestion is an alternative route for transforming waste into bioenergy. Utilizing biogas for energy has the potential to substitute fossil fuels, lower GHG emissions, mitigate resource depletion, lessen dependence on external energy supplies, and result in cost savings. This study explores the biogas production potential from waste feedstocks, like vegetable waste (uncooked), kitchen waste (cooked), and cattle dung. Although various studies have reported the anaerobic co-digestion (AcoD) of CD and FW, no previous literature has documented the feasibility of incorporating biochar in the AcoD process, especially at different co-substrate mixing ratios of CD and FW. The biochar addition to AD's slurry enhances CH₄ yield, shortens the microbial growth period, immobilises microbes and upgrades the biogas quality. Investigating and identifying the optimum co-substrate mixing ratio (SMR) and the required amount of biochar to be mixed for maximum methane yield is essential. Further, low-cost purifying biogas is necessary for efficient utilisation. These challenges can be overcome using biochar (prepared from available waste biomass) with a

highly porous structure with a large surface area, enabling it to adsorb impurities such as H₂S, CO₂ and volatile organic compounds from biogas.

1.3 SCOPE OF THE PRESENT RESEARCH WORK

The scope of the present study is to identify the optimum substrate-water (F/W) mixing ratio and ideal co-substrate mixing ratio (SMR) for the anaerobic co-digestion of cattle dung and food waste. In the first attempt, the anaerobic digestion of cattle dung (CD), untreated vegetable waste (VW) and cooked kitchen waste (CKW) was performed individually at different F/W ratios under controlled and uncontrolled temperatures. Before the experiments, initial characterisations of feedstocks were performed to know the feasibility of substrates for the anaerobic digestion process. The untreated VW and CKW were collected from the Indian Institute of Technology (IIT) Guwahati Hostel mess, whereas cow dung was collected from a farm near the IIT Guwahati campus. Secondly, the biochemical methane potential (BMP) tests for anaerobic co-digestion of food waste (untreated VW or CKW) with fresh CD were studied for different SMRs, maintaining 1:1 F/W and optimizing the best result from the study. The BMP tests were conducted on a laboratory scale batch-type under uncontrolled (ambient temperature) and controlled (mesophilic temperature) conditions, considering 1L capacity glass reagent bottles as digesters without and with the addition of biochar (BC). The impact of BC addition on AcoD concerning CH₄ yield, pH value, VS reduction, etc., under ambient and mesophilic temperatures and its characterisation using Field Emission Scanning Electron Microscopy (FESEM), Brunauer–Emmet–Teller (BET), X-ray diffractometer (XRD), CHNSO analyser, energy-dispersive X-ray spectroscopy (EDS), Fourier Transform Infrared spectroscopy (FTIR), were studied to determine the evidence of the effect of BC addition. Considering the ideal SMR ratio, further AcoD experiments were performed to identify the suitable amount of BC to be added to the AcoD of CD and FW for higher biogas production. Thirdly, considering the best F/W ratio, the biogas generation potential experiment was

conducted in the field scale AD digester (1 m³ capacity) using CD as a substrate by maintaining the digester at mesophilic conditions with the help of a solar-assisted water heating system during winter seasons. Further, a novel biogas purification test set-up was designed, fabricated and operated to remove impurities such as CO₂, H₂S, and volatile organic compounds from the biogas using coconut husk (CH), sugarcane baggage (SB), and water hyacinth (WH) biochars as adsorbents which were prepared at the pyrolysis temperatures of 550, 450 and 350 °C.

1.4 OBJECTIVES OF THE RESEARCH WORK

Based on the current challenges and research gaps, the present study is to assess the effect of different operating parameters like slurry mixing ratio, temperature, pH value, etc., for biogas production that can be executed to design and set up large-scale biogas plants in several remote locations in the country. This present investigation aims to provide a perfect competitive promotion of biogas as the alternative clean fuel for household applications in rural areas. Whereas biogas mainly comprises methane (CH₄) and carbon dioxide (CO₂) along with some traces of gases such as water vapour, hydrogen sulphide (H₂S), nitrogen, hydrogen and oxygen. The CO₂ and trace gases must be removed because the hydrogen gas is corrosive, and water vapour may cause corrosion when combined with H₂S on metal surfaces and reduce the heating value. The following objectives have been addressed in the present investigation:

1. To investigate the effect of operating parameters on the biogas production rate in batch-type lab-scale anaerobic digestion with cattle dung and food waste as substrate
2. To analyse the effect of the co-digestion on biogas production rate both with food waste and cattle dung with biochar addition followed by biochemical methane potential.
3. To investigate the performance of field-scale 1 cum anaerobic digestion plant with and without a controlled environment using cattle dung as a feedstock.
4. To develop a biogas purification system for the removal of CO₂ and H₂S.

1.5 THESIS ORGANISATION

The present thesis covers the seventh chapter with appropriate sections and subsections and contains references and visible research outputs. A brief description of these chapters is mentioned as follows:

- **Chapter 1** consists of a brief discussion about the source of cattle dung and food waste and their issues related to improper management, the proposed anaerobic digestion technique, objectives, the need for the study, and the scope of the thesis.
- **Chapter 2** presents a detailed literature review on the anaerobic digestion of cattle dung and food waste, operating parameters for the anaerobic digestion process, enhancement of biogas generation using biochar, and biogas purification techniques.
- **Chapter 3** consists of a description of the experimental setup and different phases of experiments and methodologies. The detailed procedures for physiochemical, biochemical, and analytical analyses are provided.
- **Chapter 4** presents the laboratory-scale anaerobic digestion of fresh cattle dung, vegetable waste and kitchen waste to identify the optimum feedstock-water mixing ratio and the effect of various operating parameters during anaerobic digestion. Also, it consists of the anaerobic co-digestion of cattle dung, vegetable waste and cooked kitchen waste to identify the effective co-substrate mixing ratio without and with the addition of biochar performed under ambient and mesophilic temperatures.
- **Chapter 5** deals with the field scale anaerobic digestion of cattle dung using optimum substrate-water mixing ratio and the effect of various operating parameters during anaerobic digestion.
- **Chapter 6** discusses biogas purification using biochar (water hyacinth, coconut husk and sugarcane baggage) as the adsorbents produced at different pyrolysis temperatures.
- **Chapter 7** summarises the key findings of the experimental investigation and the scope for future work.

CHAPTER 2

LITERATURE REVIEW

This chapter covers the detailed literature review on the anaerobic digestion of cattle dung, cooked kitchen waste, vegetable waste and co-digestion of different co-substrates with the addition of biochar to enhance biogas generation. This chapter also includes a literature review on biogas purification using different bio-waste materials techniques.

2.1 BIOCHEMICAL PROCESS OF ANAEROBIC DIGESTION

The production of biogas from biological waste via anaerobic digestion is one of the most significant challenges of this century. Biogas is a vital renewable fuel for remote locations in developing nations. Anaerobic digestion (AD) is a multistep biological route based on a reduction process where a community of robust microorganisms executes in a stable, self-regulating steady-state in the absence of oxygen, transforming complex organic matter into a mixture of methane, carbon dioxide and other gases (Mehariya et al., 2018). Biogas is a colourless gas that smoulders with a clear blue flame similar to liquid petroleum gas. The biogas produced during AD is a mixture comprising mainly of methane ($\text{CH}_4 \approx 50\text{--}75\%$), carbon dioxide ($\text{CO}_2 \approx 25\text{--}50\%$) and small traces of hydrogen sulphide (H_2S), hydrogen (H_2), nitrogen (N_2), carbon monoxide (CO), oxygen (O_2), water vapour (H_2O) or other gases and vapours of various organic compounds. The process of biogas production from a different organic material mainly depends on the types of substrates that can be converted into biogas, while their chemical compositions and biodegradability are the key factors for biogas and methane production.

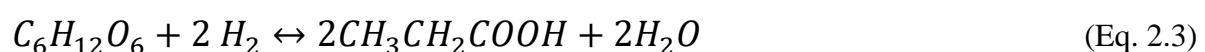
Anaerobic digestion of biodegradable organic matter occurs in four biochemical steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. Several microorganisms, such as

acidogenic, acetogenic, and methanogen bacteria, are responsible for the production of biogas (Appels et al., 2008; Kothari et al., 2014). Organic matter in any waste is found in the form of polymers such as carbohydrates (cellulose, hemicelluloses, starch, etc.), oils, fats and proteins. In general, microorganisms cannot utilise these polymers because of the large size of the molecule, which cannot penetrate the cell wall of the micro-organisms (Kothari et al., 2014). Therefore, acidogenic bacteria initially hydrolyse these polymers by producing extracellular enzymes (cellulose, xylanase, amylase, lipase, proteolytic enzymes, etc.). The carbohydrates, proteins, oils, and fats are hydrolysed into simple monomeric sugars, amino acids, and fatty acids, respectively (in the first step). The hydrolysed organic compounds (monomeric sugars, amino acids and fatty acids) are utilised by the acidogenic or acid-forming bacteria for their growth and accumulate volatile fatty acids (VFA) such as acetic acid, propionic acid, butyric acid and valeric acid along with carbon dioxide, water and hydrogen, called acidogenesis (in the second step). The VFA, except acetic acid, such as propionic acid, butyric acid and valeric acid, are again utilised by acetogenic bacteria for their growth and form acetic acid and hydrogen, called acetogenesis (third step). Finally, methanogens use acetic acid, hydrogen and carbon dioxide to form methane gas, called methanogenesis. A simplified flow of the four digestion stages is shown in Fig. 2.1.

Considering $C_6H_{10}O_4$ as an approximate chemical formula for biological waste, the reactions taking place during the AD process are described in the following. The hydrolysis reaction can be written as (Kothari et al., 2014)



The accumulation of the VFA reactions (Kothari et al., 2014) are



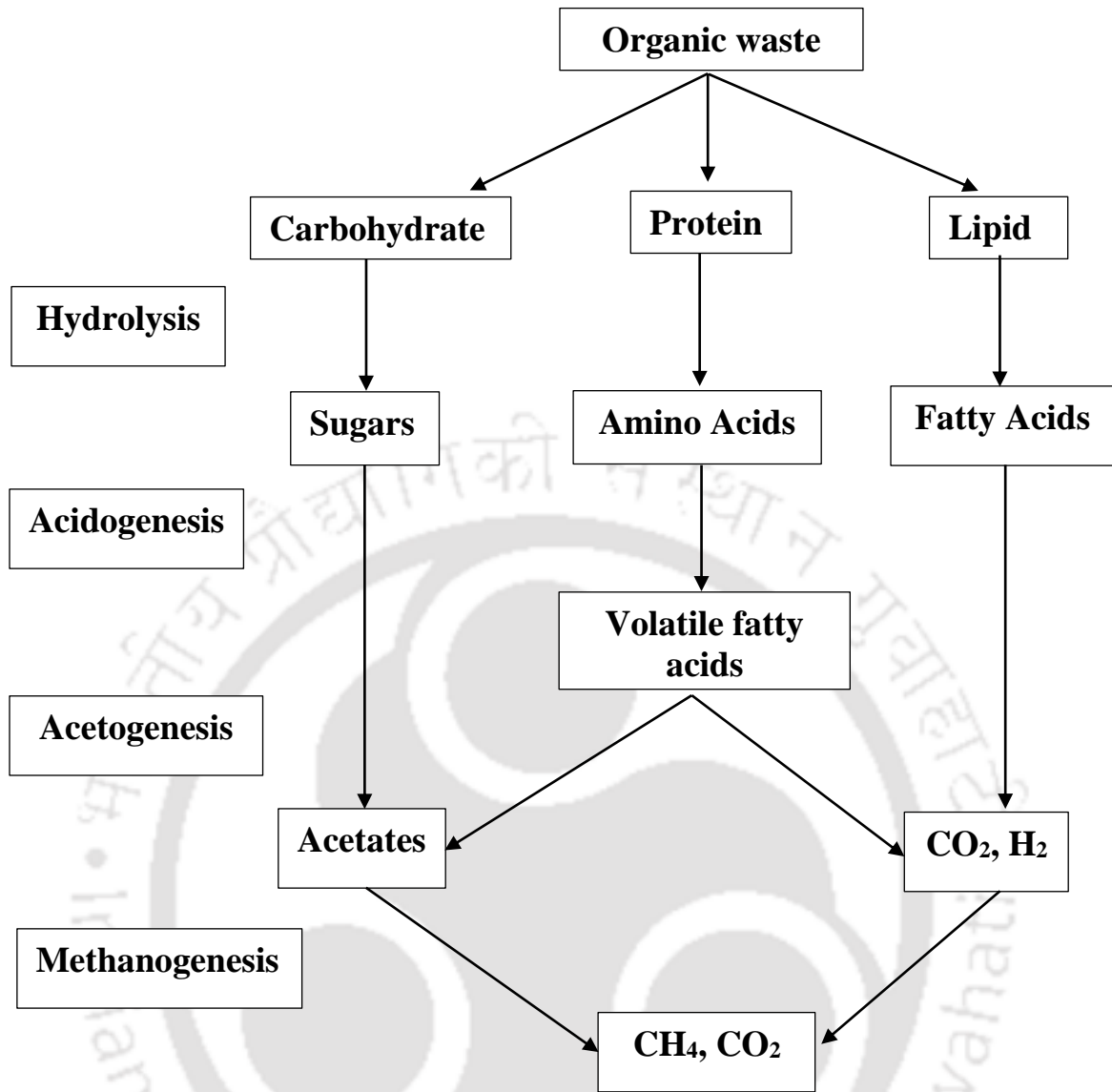
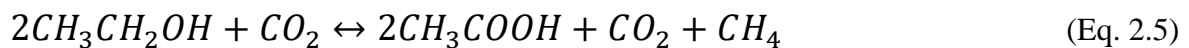


Fig. 2.1 Stages of anaerobic digestion (Kothari et al., 2014; Pramanik et al., 2019)

The acetogenesis reaction (Kothari et al., 2014) can be written as



The CH_4 is generated from a number of substances such as ethanol, acetic acid, methanol, carbon dioxide and hydrogen. The methanogenesis reactions (Kothari et al., 2014) can be written as

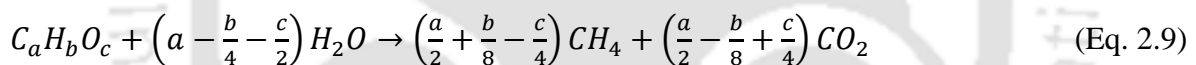




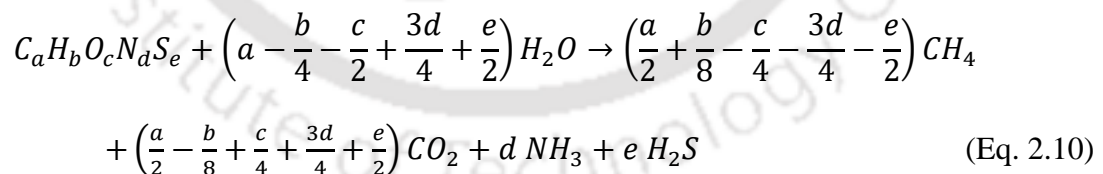
Approximately 70% of the methane is produced through the acetate pathway.

2.2 THEORETICAL BIOGAS POTENTIAL

The potential of biogas yield of the anaerobic digestion of a particular type of organic waste and the gas composition can be determined by the chemical composition of a feedstock. Assuming all substrate is converted to either carbon dioxide or methane and that the substrate's carbon, hydrogen, and oxygen composition are known, the following general gas Eq. 2.9 can be used to find a theoretical molar and volumetric methane output (Buswell en Mueller, 1952). The authors reported that it is possible to get 95 –100% yields calculated from this equation with a bit of care.



Boyle (1977) modified the chemical reaction of Buswell and Mueller (1952) and included nitrogen and sulfur to obtain the fraction of ammonia and hydrogen sulfide in the produced biogas, as shown in Eq. 2.10.



2.3 PARAMETERS INFLUENCING ANAEROBIC DIGESTION

The various operational parameters like temperature, pH, C/N ratio, organic loading rate (OLR), hydraulic retention time (HRT), total solids (TS) and volatile fatty acids (VFAs)

concentration affect biogas production (Kainthola et al., 2019). The main parameters influencing anaerobic digestion are described in detail in the paragraphs below.

2.3.1 Temperature

Temperature is one of the most critical parameters for the AD process. In the AD process, three temperature ranges viz. psychrophilic (25 °C), mesophilic (around 35 °C) and thermophilic (around 55 °C) are used (Hagos et al., 2017; Kothari et al., 2014; Kwietniewska en Tys, 2014). Various researchers reported that the microorganisms grow best at temperature ranges of mesophilic and thermophilic (Hagos et al., 2017), but the thermophilic process is harder to control and needs more energy to keep the fixed temperature of the reactor (Chen et al., 2008; Divya et al., 2015; Kwietniewska en Tys, 2014). Therefore, it is essential to maintain a stable operating temperature in the digester to improve biogas production since frequent fluctuations in temperature affect the growth of bacteria inside the digester.

2.3.2 pH value

Multiple organisms require different optimal pH growth in the biogas production process. The balance between ionised and non-ionised forms of sulphide and ammonia in the digester is regulated by pH. In lower pH ranges, non-ionised sulphide is prevalent, while in higher pH ranges, non-ionised ammonia dominates, which can inhibit microbial activity. pH fluctuations affect the concentration of H⁺ ions, directly influencing microbial growth and, consequently, the AD process. Therefore, correcting imbalanced and low pH conditions in the digester is essential. The most favourable range of pH to obtain maximal biogas production in anaerobic digestion is 6.8–7.2 (Appels et al., 2008; Kwietniewska en Tys, 2014; Mehariya et al., 2018; Ward et al., 2008). In AD, methanogenic bacteria are extremely sensitive to pH variations and prefer a pH of around 7.0 (Hagos et al., 2017; Kothari et al., 2014; Ward et al., 2008). Acidogenesis microorganisms (acid-forming bacteria) are relatively less sensitive to pH

and are tolerable in the range of 4.0–8.5. However, the optimal pH for hydrolysis and acidogenesis is between 5.5 and 6.5 (Appels et al., 2008; Kwietniewska en Tys, 2014). At the beginning of the fermentation, acidogens and acetogens produce acids and CO₂; consequently, pH decreases. Afterwards, the methane-producing bacteria consume the acids, and the pH of the digester increases and then stabilises (Kwietniewska en Tys, 2014). This factor is significant because it influences the proportion of ionised and non-ionised forms of inhibitors of methanogenesis.

2.3.3 Organic loading rate

Organic loading rate (OLR) is a critical operational parameter affecting biogas yield. It is the capacity of the AD system for the biological conversion or the feeding amount of organic material (expressed as COD or volatile solids (VS)) to the system daily per m³ of digester volume (Kothari et al., 2014). As the organic loading rate increases, the biogas yield increases to some extent, but above the optimal OLR, the biogas yield decreases due to overloading (Babaee en Shayegan, 2011; Mao et al., 2015). This may be due to the accumulation of inhibitory substrates, such as fatty acids, in the digester (Kothari et al., 2014). Various researchers reported that an extremely high OLR leads to higher hydrolysis/ acidogenesis bacterial activity than methanogenesis bacterial activity and thus increases fatty acids from excessive substrate in a short time. After that, the pH of the digester decreases, and methane-forming bacteria are not able to convert so many acids to methane (Kwietniewska en Tys, 2014). Thus, it is very important to control the OLR in continuous systems.

2.3.4 Carbon/ Nitrogen ratio

AD systems are sensitive to the C/N ratio, which reflects the nutrient levels of a digestion substrate. In the case of the feedstocks with a high C/N ratio, the methanogens consume nitrogen rapidly, which results in lower gas yield (Kothari et al., 2014). On the other

hand, the feedstocks with an excessively low C/N ratio increase the risk of ammonia inhibition, which is toxic to methanogens and causes insufficient utilisation of carbon sources (Kothari et al., 2014; Mao et al., 2015). The optimal C/N ratio for anaerobic digestion has been found to be between 20 and 30 (Hagos et al., 2017; Naik et al., 2014), 25 and 35 (Kondusamy et al., 2014) or between 25 and 30 (Y. Li et al., 2017a) with a ratio of 25 being the most commonly used (Pang et al., 2008). However, the optimal C/N ratio is a function of the feedstock type and varies with the feedstock type. The optimum C/N ratio of the materials can be maintained by mixing materials with high and low C/N ratio (Kothari et al., 2014).

2.3.5 Hydraulic retention time

The retention time is also an important parameter that needs to be observed during the AD process. It can be defined as the time necessary for the complete degradation of organic matter or the average time organic matter remains in a digester (Kothari et al., 2014; Pramanik et al., 2019). It is related to the microbial growth rate and depends on the process temperature, OLR and substrate composition (Mao et al., 2015). Kothari et al. (2014) reported that a retention time of 10–40 days is necessary for biomass digestion in mesophilic temperature, while it could be lower in thermophilic temperature (Kothari et al., 2014).

2.4 CATTLE DUNG AS A SUBSTRATE FOR ANAEROBIC DIGESTION

Cow dung has a wide variety of microorganisms varying in individual properties. Several authors have acknowledged the biogas production from manure as a cost-effective mitigation technology for greenhouse gas (GHG) emissions (Hamelin et al., 2014). One kg CD has the potential to produce 35–40 L of biogas at the substrate-waster (S/W) mixing ratio of 1:1 in the temperature range of 24–26 °C for HRT of 55–60 days (Gupta et al., 2016). Kalia and Singh (2004) reported that one m³ biogas plant (Deenbandhu) can produce 0.028 m³ biogas for a daily feed rate of 22 kg CD (TS content of 9–10%) at an S/W ratio of 1:1. Werner et al.

(1989) also reported that CD generated from 3–5 cattle can run a simple 8–10 m³ biogas plant which can produce 1.5–2 m³ biogas/day which is sufficient for the family 6–8 persons, can cook meal for 2 or 3 times or may light two lamps for 3 h or run a refrigerator for all day and can also operate a 3 KW motor generator for 1 h. However, various operating parameters affect the rate of biogas production from CD. The digester's temperature affects the AD of CD. Mackie and Bryant (1995) mentioned that thermophilic (60°C) AD of CD is more stable than mesophilic (40°C) at different HRTs (viz. 10, 15, and 20 days). (Kumar Jha et al., 2013) reported that about 10% relatively higher biogas was generated in the thermophilic reactors than in mesophilic reactors. El-Mashad et al. (2004) investigated the influence of daily upward and downward functions temperature on the performance of AD of CD in completely stirred tank reactors (CSTRs). Their experiment results proposed the possibility of using available solar energy in the daytime to heat the reactor(s) without needing heat storage during the night. The biogas production also depends on the organic loading rate (OLR). (Li et al., 2009) reported CD can produce 313.4 mL/gVS CH₄ at 35±1 °C and a loading rate of 10gVS/L for HRT of 45 days. Massé and Saady (2015) studied the impact of OLR on AD of solid CD (TS of 11%–16%). The authors revealed that the quality of CD rather than the OLR directly influenced the specific methane yield (SMY). An SMY of 184.9±24.0, 189.9±27.3, and 222±27.7 NLCH₄/kgVS has been achieved at an OLR of 3.0, 4.0, and 5.0 g TCOD/kg inoculum for HRT of 21 days respectively. Kumar Jha et al. (2013) reported that biogas and methane yields increased as the substrate concentration decreased from 15.18% to 7.68% TS. From the literature study, it is confirmed that cattle dung has biogas production potential. However, various operational parameters must be maintained in the desired range, implementing the various enchantment methods of biogas generation.

2.5 VEGETABLE WASTE AS A FEEDSTOCK FOR ANAEROBIC DIGESTION

Several researchers have performed experiments on AD of fruits and vegetable waste (FVV) (Sitorus en Panjaitan, 2013; Viswanath et al., 1992; Zhao et al., 2016) and also their feasibility assessment (Masebinu et al., 2018) for biogas production. However, the efficiency of biogas production depends on the activity of microorganisms. Therefore, it is necessary to study the factors that affect microbial activity. The authors (Mao et al., 2015; Mehariya et al., 2018; Zhang et al., 2014) reviewed the findings by several researchers on the various operating parameters like temperature, pH, retention time, C/N ratio etc., which affect the biogas production rate. The quantity and quality of biogas yield also depend on the mixing ratio or concentration with water or various substrates. Jiang et al. (2018) studied the effects of source, concentration, and temperature on the AD of VW and reported that thermophilic conditions were more suitable for AD than mesophilic conditions. Bouallagui et al. (2003) experimented with feed concentrations and HRT's effect on biogas production from fruit and vegetable waste at mesophilic conditions. Some authors studied the characteristics of VW and reported the feasibility of anaerobic digestion for energy recovery. Since VWs are easily biodegradable and composed of high moisture (>80%) and high organic matter (>95% dry basis), these wastes can be used as a substrate for anaerobic digestion, as demonstrated by different authors (Jiang et al., 2012; D. Li et al., 2017). The types of VW also affect the biogas production rate. Gunaseelan (2004) studied around 54 kinds of fruits and VW and reported that the methane yield ranged from 0.180 to 0.732 L/gVS for fruits and 0.19 to 0.41 L/gVS for vegetables. Bong et al. (2018) reported that VW has low lipid but relatively high cellulosic content. In contrast, FW and KW have high lipid content because of animal fat and oil. Studies have reported that VW had a lipid content of 11.8%, whereas kitchen waste was reported to have a 21.6% lipid content, respectively (Pramanik et al., 2019). Based on a review of various literature, it can be inferred that food waste can be considered as a favourable feedstock for anaerobic digestion.

2.6 FOOD WASTE AS A SUBSTRATE FOR ANAEROBIC DIGESTION

Anaerobic digestion is one of the most effective approaches for food waste (FW) management and environmental benefits (Li et al., 2018). Various researchers investigated the potential for biogas production from food waste. Its theoretical methane production rate typically ranges from 0.4 to 0.5 L CH₄/gVS (Li et al., 2018; Nagao et al., 2012). However, FW characteristics have been varied from place to place (Dhamodharan et al., 2015). Food waste comprises mainly three organic components: carbohydrates, proteins and lipids, which have different theoretical methane yields and bioconversion rates (Esposito en Frunzo, 2012). Y. Li et al. (2017a) investigated the influence of carbohydrates, proteins and lipids on the AD of FW and the relationship between the parameters that characterise digestion. The authors identified that FW, rich in lipids, could produce more methane than carbohydrates and proteins. However, high lipid content can cause system failure due to the formation of long-chain fatty acids. Xu et al. (2018) reported that carbohydrates and proteins have a higher hydrolysis rate due to their rapid degradability compared to lipids. Cho et al. (1995) reported a 472 ml/g VS methane yield with 86% anaerobic biodegradability of the Korean food waste. However, the methane production of every category of FW falls in the range of 346–551.4 mL/ gVS (Pramanik et al., 2019). Moreover, the effect of various operational parameters is the most crucial factor that needs to be studied for efficient biogas production. Park et al. (2008) examined the impact of pH value for AD of FW. They showed that the appropriate pH range for thermophilic acidogens for batch tests was around 6–7, and the optimum pH condition was 6. However, Pramanik et al. (2019) reviewed and reported that the pH values of most FW are in the acidic range of 4.1–6.5. Kim et al. (2006) studied the effect of temperature and HRT on AD of food waste. The authors reported that the rates of biogas and methane production by thermophilic digesters were higher than those by mesophilic digesters, regardless of HRT. Liu et al. (2017) found that the optimal OLR on AD of FW under thermophilic and mesophilic conditions were

2.5 and 1.5 g-VS/L/day, respectively. The authors also reported at the same OLR, the methane yield under thermophilic conditions was 33–49% higher than that under mesophilic conditions. Li et al. (2017) also compared the thermophilic and mesophilic digestion of FW and waste-activated sludge mixture. The thermophilic system (407 mL/g VS) yielded better CH₄ production than the mesophilic system (350 mL/g VS). Various researchers have characterised food wastes to understand the physical, chemical, and biological behaviours of food wastes and to predict the potential for biogas production. Zhang et al. (2014) reported that FW's TS and VS contents are 18.1–30.9 and 17.1–26.35 %, respectively, indicating that water accounts for 70–80% of FW. Zhang et al. (2007) also reported that FW has a moisture content (MC) of 74–90%, VS/TS of 80–97%, and C/N of 14.7–36.4. Using FW with high VS content, the quantity and quality of biogas can improve the (C. Zhang et al., 2014). Also, due to the high MC, FW is an easily biodegradable organic substrate (Dhamodharan et al., 2015; C. Zhang et al., 2014). Zhang et al.(2007) reported that the nutrient contents of FW indicate that food waste contains the required nutrients for anaerobic microorganisms. Hence, from the study of various literature, it can be concluded that food waste is the more favourable feedstock for anaerobic digestion. Table 2.1 shows the significant findings from anaerobic digestion of food waste as a substrate conducted under various operating conditions.

Table 2.1 Biogas production using food waste as substrate

Reference	Reactor	Temperature/ HRT	Description/methodology	Major findings
Dompara et al. (2023)	Batch	37°C, 22 days	TiO ₂ and ZnO/Ag powders were added to improve the efficiency of biogas yield.	<ul style="list-style-type: none"> The addition of ZnO/Ag and TiO₂ improved the biogas cumulative yield by 12 and 44%, respectively.

Mohamed Ali et al. (2023)	Semi-continuous	37°C, 15 and 20 days	Hydrolysis of FW was done by the immobilized biofilm with inoculum concentrations of 10%, 30%, and 50%, respectively. The inoculum used was mesophilic anaerobic sludge from a sewage treatment plant.	<ul style="list-style-type: none"> • 50% inoculum-to-feed ratio showed the highest biogas volume around 2 L • The digester with 20 HRT has shown a high production of biogas compared with a digester operated at HRT 15.
Singh et al. (2023)	Batch	35°C, 45°C and 55°C, operated for different cycles 1 (0–30 days), 2 (31–60 days) and 3 (61–90 days)	Both pre-processed and post-processed discarded FW residue was used as a substrate, with anaerobic sludge as a buffer substrate.	<ul style="list-style-type: none"> • CH₄ yield was maximum at the thermophilic condition • A temperature range of 38°C to 50°C was suitable for steady biogas production
Alam et al. (2022)	Batch	22–32 °C and 32–37 °C, 14–45 days	University mess and cafeteria FW were used in three different dry anaerobic digesters and executed in 2 phases.	<ul style="list-style-type: none"> • Approximately 2079 ml of biogas was produced on the 37th day • The biogas produced a blue flame, indicating strong energy potency.
Wang et al. (2021)	Batch	35°C and 55°C	FW and anaerobic granular sludge were added in a mass ratio of 2:1; Bentonite was also added in six different concentrations of 0, 0.5, 1, 2, 3 and 5 g/L	<ul style="list-style-type: none"> • CH₄ production increased by 68.52% in mesophilic digestion and 56.79% in thermophilic digestion when 3 g/L and 5 g/L of bentonite were added, respectively.
Xue et al. (2020)	Batch	37 ± 1 °C, 40 days	FW rich in lipids (L), carbohydrates (C), and proteins (P) were used as a substrate to investigate the interaction between C/N and LCP ratios.	<ul style="list-style-type: none"> • Results reflect the interaction between C/N ratio and LCP composition • The highest 595 mL CH₄ /gVS at a C/N ratio of 25 and LCP ratio of 63.25:22.62:14.13.
Muñoz et al. (2020)	Batch	Between 15 and 21 °C, 15 days	Inoculum was collected from an AD plant treating piggery waste; FW was characterized, and BMP was determined. Post-digestate was used as a cultivation medium for microalgae.	<ul style="list-style-type: none"> • Psychrophilic AD of FW results in lower biogas yields • After 15 days, microalgae were successfully harvested in all post-digestate dilutions.

Martí-Herrero et al. (2019)	Continuous plug flow digester	Psychrophilic conditions	The inoculum source was cow rumen, and after eight weeks, the digester was fed exclusively with fruit and vegetable waste, operating at an organic loading rate of 1 kgVS/(m ³ d)	<ul style="list-style-type: none"> • Biogas production increased to 0.34 m³/kgVS when the organic loading rate was reduced by half to 0.57 kgVS/(m³d) • Starting the digester with cow rumen allows for a smooth transition to FVW while maintaining stable biogas production. • The passive solar design raises the slurry temperature by 3–6°C above the ambient temperature throughout the year
Y. Li et al. (2017b)	Batch	37°C, 30 days	Chinese food, Seed sludge was used as inoculum	<ul style="list-style-type: none"> • 385 mL/g VS CH₄ yield
Kumar et al. (2016)	Continuous stirred tank reactors	Mesophilic & thermophilic, 18 to 30 days (18, 20, 24 and 30 days)	The organic load ratio was from 1.0 to 6.1 kg COD/m ³ -d. Feedstock concentration was adjusted to 30–110 gCOD/L using distilled water. No additional nutrients were supplied during the operation period	<ul style="list-style-type: none"> • Thermophilic conditions showed better CH₄ production and organic removal due to enhanced hydrolysis and microbial activity at higher temperatures • Peak methane production: 1.30 m³/m³-d under thermophilic conditions
Tampio et al. (2014)	Semi-continuous	37°C, 35 days	Domestic food waste, United Kingdom	<ul style="list-style-type: none"> • CH₄ yields at 3–6 kgVS/m³day OLR obtained 5–10% higher for untreated FW (0.483±0.013 m³CH₄/kgVS than autoclaved treated FW (0.439±0.020 m³ CH₄/kg VS)
Li et al. (2009)	Batch	35±1 °C, 45 days	Used Chinese food, Inoculum: Sludge from a swine waste treatment plant	<ul style="list-style-type: none"> • 35.7 mL/g VS CH₄ yield
Kim et al. (2006)	Batch	55°C, 30 days	Korean food waste, substrate to inoculum ratios of 1:1.	<ul style="list-style-type: none"> • A higher and faster rate of CH₄ was obtained at thermophilic conditions. • 223 ml CH₄/gCOD was detected.
Cho et al. (1995)	Batch	35°C, 28 days	A BMP test was performed using cabbage as a feedstock.	<ul style="list-style-type: none"> • 277 mL/g VS CH₄ was found.

2.7 CO-DIGESTION OF FOOD WASTE AND CATTLE DUNG WITH OTHER CO-SUBSTRATES

Anaerobic co-digestion (AcoD) of various raw materials may be a cost-effective way to balance macro- and micronutrients and decrease the formation of inhibitors or accumulation of toxic compounds, thereby improving biogas production (Barua et al., 2018a). Cattle dung is a well-studied co-substrate for AcoD of FW for its high buffering capacity and high nitrogen content, which could enhance the digestion efficiency and rich in micronutrients necessary for optimum bacterial growth (Dhamodharan et al., 2015). The high biodegradability of FW makes it a promising organic substrate for AD. However, mono-digestion of FW often leads to digester instability and even failure at higher organic loading rates, especially under thermophilic conditions, due to the accumulation of VFAs and ammonia inhibition (Nagao et al., 2012; Xu et al., 2018). The co-substrates can supply micro-nutrients and alkalinity, overcome the disadvantages of single digestion of FW, efficiently use equipment, and share costs by processing multiple waste streams in a single facility. Zhou et al. (2015) observed that AcoD of food waste and corn stalk enhanced biomethane production by 22.48% and 41.55% than mono-digestion of FW and corn stalk, respectively. Yong et al. (2015) also reported that co-digestion increased methane production yield by 39.5% and 149.7% compared to mono-digestion of FW and straw, respectively (Yong et al., 2015). Besides, the anaerobic co-digestion process necessitates the utilisation of a suitable mixing ratio to maintain steady conditions within the reactor. Several studies have addressed methane production and ammonia inhibition in AcoD of FW and CD at different substrate combination ratios and OLRs (Agyeman en Tao, 2014; El-Mashad et al., 2004). In the present study, substrates such as cattle dung, cooked kitchen waste, and untreated vegetable waste were selected to investigate the effect of the mixing ratio of feedstocks. Several researchers studied the AcoD of CD and FW, and their significant findings on AcoD of CD and FW are given below in Table 2.2.

Table 2.2. Performance in AcoD of cattle dung and food waste

Reference	Substrate	Reactor	Temperature/HRT	Description/methodology	Major findings
Yu et al. (2024)	Cabbage waste and cattle manure (CM)	Both batch and continuous	37 °C, six different HRTs of 25, 20, 15, 10, 8, & 5 days	Five mixing ratios of 1:0, 3:1, 1:1, 1:3, and 0:1 were tested in batch experiments	<ul style="list-style-type: none"> • The optimal mixing ratio of cabbage waste to CM was 3:1, yielding a maximum CH₄ yield of 440 mL/gVS • 10 days HRT was best for CH₄ production, and production decreased with decreasing HRT and stopped at HRT of 4 days
Mirabi et al. (2024)	Lignocellulosic/lipidic wastes with CM	Batch	35 °C, 120 days	The three tested ratios are (0.5, 1, and 2) for inoculum to substrate (ISR) and (20:80, 50:50, 80:20) for substrate composition (SCR)	<ul style="list-style-type: none"> • The highest efficiency was observed with ISR = 0.5 and SCR = 4, where BG and CH₄ production were 36% and 45% of the control (CM alone)
Luo and Pradhan (2024)	FW and waste sewage sludge (WAS)	Batch	Ambient temperature, 30 days	FW/WAS ratio was 60:40%. Inoculum: Digested sludge	<ul style="list-style-type: none"> • 321.91±8.24mL/gVS was achieved. • CH₄ yield was 34% higher than the WAS mono-digestion

Thakur et al. (2024)	FW and bio-flocculated sewage sludge (BFS)	Continuous stirred tank reactors (CSTR)	37 ± 2°C	Daily effluent added: 30 mL FW + 1600 mL BFS Inoculum: Digested sludge from a paper mill	• 233 mL mL/g VS _{added}
Abbas et al. (2023)	Canteen FW and CM	Batch	37 ± 1°C	Tested with seven different CFW/CM ratios (0:1, 1:4, 2:3, 1:1, 3:2, 4:1, and 1:0) based on wet weight, and compared with 0:1 and 1:0. 100 g of intestinal fluid from the cow was used as an inoculum	• The 4:1 CFW/CM ratio resulted in the best C/N ratio of 17.95 ± 0.05, the highest biogas yield
Khanthong et al. (2023)	FW and algae (AL)	Semi-CSTR	35°C, 30 days	AL and FW mixing ratios of 0:100, 20:80, 40:60, 60:40, 80:20, and 100:0 were considered	• AL/FW 20:80 ratio resulted in the highest CH ₄ with an accumulation of 404 mLCH ₄ /gVS
Oduor et al. (2022)	FW and water hyacinth	Batch	37°C, 81 days	The mixing ratio was WH: FW (v/v) and was 100:0, 85:15, 70:30, 55:45, 30:70, 15:85, and 0:100. The inoculum was collected from an active FW anaerobic reactor	• The WH: FW of 70:30 detected the highest maximum SMY of 616.01 mLCH ₄ /gVS

Kaushal et al. (2022)	FW (Chicken and fish), algae, and CM	Batch	35– 42 °C	Experiments were designed using Stat-Ease Design-Expert Software-13, considering the optimization of HRT, OLR, temperature, and ISR ratio. Cow urine was used as an inoculum	<ul style="list-style-type: none"> • Chicken waste produced the highest biogas yield due to its higher biogas potential • The optimal values for CBY and CH₄ generation were 2.245 L and 1.346 L, respectively
Rahmani et al. (2022)	Wheat straw with FW and CM	Batch	35 °C	Study on optimizing process parameters, ISR, C/N, and TS content	<ul style="list-style-type: none"> • Co-digestion of the three substrates resulted in a 20% higher biogas yield compared to mono-digestion, with 21% VS removal • ISR 2 and C/N 35 improved biogas yield by 24% and 17.4%, respectively than mono-digestion
Xu et al. (2022)	FW, sugarcane leaves (SLs), and CD	Batch	37 ± 1 °C 21 days	The FW, SL, CD, FW + SL, FW + CD, and FW + SL + CD were used as the feedstocks (mono and mixed) in ten different ratios, keeping the inoculum constant	<ul style="list-style-type: none"> • The optimum mixing ratio of FW:SL: CD (VS basis) was 85:11.25:3.75 • The ratio resulted in a 444.72% improvement in CH₄ production, which was higher than the mono-digestion of SL and CD

Dhungana et al. (2022)	FW, CM, poultry litter (PL) and goat manure (GM)	Semi-continuous	Average temperature (10–21°C) in two phases (October to February and February to April), HRT 60 days	FW, CM, PL and GM were co-digested at mixing ratios (FW:PL: CM) of 2:1:1, 2:2:1, 1:1:2, 1:1:1 (wt/wt) and FW:PL: GM at mixing ratios of 2:1:1 and 1:1:2. The process was conducted at an OLR of 1 gVS/L/day with a TS content of 8%	<ul style="list-style-type: none"> • Maximum CH₄ yield was achieved from the co-digestion of FW, PL, and GM at a mixing ratio of 2:1:1. • Co-digestion of FW, PL and CM at a mixing ratio of 2:2:1 produced negligible CH₄ under the same temperature conditions.
Zhang et al. (2022)	FW, CM and corn straw (CS)	Semi-continuous	38 °C, 2 phases (40 days with OLR at 2gVS/L.day and 26 days (4gVS/L.day)	Monodigestion and co-digestion with two and three substrates with different ratios were tested. The inoculum was collected from a sewage digester.	<ul style="list-style-type: none"> • Digester with 75% FW + 25% CS had most Syntrophomonadaceae (26.7%) and CH₄ yield (467.3–507.6 mL/g VS) among co-digestion trials

Hamzah et al. (2022)	Pineapple waste (PW) with CD	Batch	37 ± 1 °C, 40 days	AcoD was performed for five different CD to pineapple waste ratios (1:1, 1:2, 1:3, 2:1 and 3:1) based on VS contents; also, mono-digestion for PW was done	<ul style="list-style-type: none"> • The highest CMY was gained at a ratio of 1:3 • The average CH₄ content was elevated by 23% in the AcoD process, compared to mono-digestion of PW, with improving C/N ratio, pH value, total ammonia nitrogen, and VS removal efficiency
Wang et al. (2020)	KW and PM	Batch	37 °C, 120 days	TS content varies from 5% to 15%. Seed sludge was used as an inoculum	<ul style="list-style-type: none"> • Specific CH₄ yield was 278.8–291.7NmL/gVS when TS contents varied from 5% to 15% and decreased at 20% TS content (259.8 NmL/gVS)
Dennehy et al. (2016)	KW and PM	Batch	37 °C, 32 days	The highest CH ₄ was achieved at a PM/FW mixing ratio of 40:60% (wet mass basis). The inoculum was from a semi-continuous digester operating in the laboratory	<ul style="list-style-type: none"> • 521±29mL/gVS CH₄

Zhai et al. (2015)	KW and CW	Batch	35°C, 45 days	Mixing 1:1 on the wet weight basis maintained the pH value at 7.5	• 179.8 mL/gVS methane
Agyeman and Tao (2014)	FW & CM	Semi-continuous	36 °C, 178 days	Mixing ratio 1: 1 on VS basis. OLR: 2 gVS/L/d for fine, medium and coarse FW	• 0.4–0.64 m ³ CH ₄ /kg VS
Zhang et al. (2013)	FW & CM	Batch type	35±1 °C, 30 days	FW and manure ratio of 2	• 388 mL/g VS CH ₄ yield
El-Mashad and Zhang (2010)	FW & CM	Batch type	35 °C, 30 days	CM and FW mixing ratio was 52: 48 %	• 311 mL/g VS CH ₄ yield
Li et al. (2009)	KW & CM	Batch type	35±1 °C, 45 days	KW and CM ratio 1: 1 (on VS basis). Sludge from a swine waste treatment plant was used as inoculum	• 298.6 mL/gVS CH ₄ yield

2.8 ANAEROBIC DIGESTION FED WITH BIOCHAR

In recent years, the usage of biochar (BC) in the AD process has been reported by various researchers in scientific publications to improve overall AD performance (Chen et al., 2023). Biochar is a carbon-rich compound produced through biomass thermochemical decomposition in the absence of oxygen. The BCs are obtained with pyrolysis at 300–900°C or hydrothermal carbonisation at 170–250°C, respectively (Cha et al., 2016). This pyrolysis process involves biomass's thermochemical decomposition in oxygen depletion. Adding BC to AD's slurry enhances CH₄ yield, improves operational stability, shortens the microbial growth period, immobilises microbes and upgrades the biogas quality (Chen et al., 2023; Zhao et al., 2021). Several investigators have reported that the high porosity, high surface area, alkalinity nature, existence of functional groups and high electron transfer capability of BC enhances the CH₄ yield in the AD process (Kumar et al., 2021).

Recent studies have shown that the addition of biochar in AD of food waste increased the biogas yield (Wambugu et al., 2019; Wang et al., 2017). Sunyoto et al. (2017) added pine sawdust biochar (produced at 650 °C) to the AD of aqueous carbohydrate food waste made from white bread and observed increased CH₄ production by 41.6%. Adding 8.3 g/L biochar to the food waste produced higher methane (from 55 to 78%), while 33.3 g/L biochar addition resulted in the lowest yield (Sunyoto et al., 2017). Wang et al. (2017) added vermi-compost-based biochar (500 °C) to mixed kitchen waste and observed that the biochar acted as a buffer and increased CH₄ production due to 15–20% (w/w) biochar addition. It is hypothesised that the biochar creates a surface area for colonisation by the microbial flora in the AD and acts as an adsorbent for compounds such as limonene and ammonia that would otherwise inhibit the performance of the AD (Wambugu et al., 2019). Moreover, the alkaline nature of biochar was regarded to increase CH₄ content by reacting CO₂ and H₂S with alkaline substances in ash and upgrading the biogas in situ (Wang et al., 2017). In the present study, food waste and cattle

dung manure were selected for anaerobic co-digestion to understand the possible role of biochar. Table 2.3 describes the impact of BC addition on the anaerobic digestion of organic waste conducted at different operating conditions.

Table 2.3 Impact of biochar addition on anaerobic digestion

Reference	Substrate	Reactor	Temperature/ HRT	Preparation of biochar	Major findings
Hu et al. (2024)	Waste-activated sludge (W)	Batch type	35 °C, 30 days	Three BC, namely, raw sludge W-BC, alkali-treated sludge residue BC, and alkali-treated fermented sludge residue BC were produced at a pyrolysis temperature of 400°C. The additive dosage of BC was 8 g/L.	• CH ₄ yield improved by 22.1% by adding BC.
Xu et al. (2024)	AcoD of FW with shell waste	Batch type	37 °C, 30 days	Bagasse biomass following torrefaction at 450°C with N ₂ .	• BC increased CH ₄ yields by 18.45% in the AD of SW.
Jin et al. (2024)	Waste-activated sludge	Batch type	35 °C, 30 days	Pyrolysis of Iron-rich fermented sludge residue at 400°C for 2 hours. The additive dosage of BC was 1 g biochar/g volatile suspended solids.	• Improvement of methane yield by 23.4%.
Ngo et al. (2024)	Chicken manure	Batch & semi-continuous	37 °C, 84 days	Wood BC was produced at 550°C for 2 hours.	• BC integration exhibited an 8.4-fold increase in CMY.
Wang et al. (2024)	Kitchen waste	Batch type	37 °C, 90 days	Straw digestate was pyrolyzed at 500°C, and then two kinds of Fe ₂ O ₃ -modified BC were prepared after treatment, and 15g/L BC was added.	• Methane yield increased by 20.8%.
Ding et al. (2024)	Swine manure	Batch type	37 °C, 30 days	Coconut shell BC was produced at 450°C. BC was added at a TS concentration of 3%.	• The addition of BC enhances the stability and efficiency of AD.
Liu et al. (2023)	CM	Batch type	38±1 °C, 25 days	Rape, potato, and wheat straw-based biochar at 600 °C. 10 g/L of was added.	• CMY was increased up to 35.45–52.66%.

Chen et al. (2023)	Glucose as a food substrate	Batch type	37 °C, 35 days	BC was produced from peanut shells at 500 °C. 0.5 g/L, 1.0 g/L, 5.0 g/L, and 10.0 g/L BC was added.	<ul style="list-style-type: none"> • Food to micro-organism ratio of 2.0, BC shortened the entire AD duration for 13 days.
Suthar et al. (2022)	CD	Batch type	35 °C, 35 days	BC was prepared from water hyacinth at 350 °C. 0.5, 1.0, and 1.5% (v/v) BC was added.	<ul style="list-style-type: none"> • Methane content in biogas improved by 9.33– 19.8%.
Sun et al. (2022)	FW	Batch type	37 °C, 30 days	Corn straw BC was prepared at 300, 600 and 900 °C. 10 g/L of biochar was added.	<ul style="list-style-type: none"> • CMY increased 42.07%.
Quintana-Najera et al. (2022)	Microalgae and cellulose	Batch type	37 °C, 30 days	Oakwood BC was produced at 450 °C.	<ul style="list-style-type: none"> • Biogas yielded 1.8– 4.6 times more than without BC.
Liu et al. (2022)	Sewage sludge and FW (1:1)		55 °C, 25 days	BC was prepared biogas residue from the FW treatment plant at 500 °C.	<ul style="list-style-type: none"> • Enhances the DIET of microbes and promotes the growth of bacteria, which improves biogas yield.
Kizito et al. (2022)	Chicken manure	Batch type	36°C, 55 days	Slow pyrolysis of hardwood pruning, rice husks and bamboo at a temperature of 600 °C.	<ul style="list-style-type: none"> • CMY enhanced from 277 to 438 mL/gVS with the addition of 10% BC.
Sun et al. (2022b)	Glucose and food waste	Batch type	37°C	Pyrolysis of corn straws at 300, 600 and 900 °C. 10 g/L of biochar was added.	<ul style="list-style-type: none"> • CMY of FW increased by 42.07% with the addition of BC pyrolysed at 900°C.
Sugiarto et al. (2021)	FW	Batch type	40 days	Pine sawdust was pyrolysis at 650 °C.	<ul style="list-style-type: none"> • CMY increased by 46.9%.
Zhou et al. (2020)	Municipal sludge		35 days	Corn Stover-based BC was fast pyrolysed at 500°C after pre-treatment with sulfuric acid. 66.6 g/L BC was added.	<ul style="list-style-type: none"> • Methane production enhanced by 26.2%.

2.9 PURIFICATION OF BIOGAS

The raw biogas can be used to generate electricity and heat directly. However, biogas production depends on the types of feedstock used, climatic conditions, production site, various operating parameters and technology adapted for production. All these parameters affect the composition of generated biogas. The main constituents of biogas are methane (CH_4) and carbon dioxide (CO_2), along with some traces of gases such as hydrogen sulphide (H_2S), nitrogen (N_2) and hydrogen (H_2). In general, biogas is a mixture of gases consisting of 50–75 % CH_4 , 25–50 % CO_2 , 0–10 % N_2 , 0–3 % H_2S , 0–1 % H_2 and a minimal concentration of other gases (Wu et al., 2024). The CO_2 and trace gases must be removed to improve the quality of biogas for suitable applications. The presence of CO_2 (up to 47 volume%) reduces its calorific value, burning velocity, and flammability limit, and hence, cost-effective and environment-friendly technologies for biogas upgrading, such as CO_2 removal, must be developed (Chen et al., 2024). Natural gas contains 98–75 % of CH_4 , promoting higher burning energy with higher burning velocity and flammability than biogas due to the lack of impurities like CO_2 (Aghel et al., 2022). Also, when biogas is used as vehicle fuel, CO_2 gas occupies more space in the cylinder tanks, requires additional energy to compress the biogas, and indirectly increases operating costs (Aghel et al., 2022; Wu et al., 2024). Further, the H_2 gas is also corrosive, and water vapour may cause corrosion when combined with H_2S on metal surfaces, reducing the heating value (Kulkarni et al., 2019). Therefore, other impurities, like CO_2 and H_2S , must be removed to achieve high-quality biogas, such as natural gas.

Various biogas purification methods, such as adsorption, absorption, membrane, and cryogenic separation, are available. Several researchers performed experiments to remove CO_2 and H_2S from the biogas. Zeolites or porous alumino-silicates have a high affinity towards CO_2 ; however, their relatively high adsorption strengths for gas hamper desorption and increase the cost of production for an industry (Pramanik et al., 2021). Aghel et al. (2022) reported that

high-pressure water scrubbing is the most simple and reliable biogas absorption technology, which promotes large-volume biogas processing. However, it has a higher electricity consumption (0.30–0.34 kWh/m³). Due to the low energy requirement, scrubbing technology using chemical adsorption is one of the most attractive methods since the process works at ambient pressure, high loading of scrubbing liquid, high purity CH₄, and low CH₄ loss. However, there are disadvantages if alkylamine compounds, such as monoethanolamine and diethanolamine, are used. In the reduction step, the degradation of amines results in raw material loss and harmful chemical species formation. Maile et al. (2017) used NaOH at different concentrations, such as 1 M, 2 M and 3 M, for CO₂ removal from biogas. The authors found that higher concentrations increased removal efficiency and absorption rates. The maximum CO₂ removal efficiency achieved in their study was 66%. The major disadvantage is that the toxic solvents are needed in this process.

Cryogenic processes are based on temperature differences for the liquefaction of biogas compounds. Three main systems were identified in a study focusing on cryogenic technologies for biogas upgrading and CO₂ separation. The first system involves cooling the biogas to remove water before further cooling and compression to produce liquid CO₂. The second system utilizes a distillation column to extract and liquefy CO₂. Lastly, the desublimation process removes CO₂ during its phase transition from vapour to solid within a heat exchanger (Tan et al., 2017; Wu et al., 2024). In this method, the separation depends on different sublimation points, where CH₄ (–161.5 °C) has a lower sublimation point than CO₂ (–78.5°C). While this method of separation is appealing due to its ability to produce very high-purity gas, it is associated with significant electricity consumption and necessitates cryogenic production technology capable of achieving temperatures as low as -125 °C under pressures of 20–50 bar (Mehrpooya et al., 2020). Therefore, the investment cost in this process is high.

Biological biogas upgrading utilizes microorganisms to convert CO₂ and H₂ into methane. In these methods, microorganisms act as catalysts, facilitating the conversion of CO₂ and H₂ into CH₄. Biological processes offer several advantages over chemical processes, including operation at moderate temperatures and atmospheric pressure, making them more energy-efficient and environmentally friendly (Wu et al., 2024). However, more energy is required, and investment costs are high. Further, there is a high risk of biological contamination in this method.

The physical adsorption of H₂S from biogas is a common and effective method employed in its removal, as evident from the literature (Mrosso et al., 2020), like activated carbon, zeolites, iron sponge (basically iron oxide on wood chips), etc. Moreover, activated carbon is one of the most widely used adsorbents utilised for H₂S removal. However, high temperature and pressure requirements for obtaining activated carbon, an activation process and low selectivity at elevated temperatures are some of the challenges in this area (Boehm, 1994; Shang et al., 2012). Kulkarni et al. (2019) conducted experiments using NaOH, Ca(OH)₂, mono-ethanolamine, activated carbon and steel wool under single and multiple purification columns to remove H₂S in biogas generated from the floral waste feedstock. The authors reported that the H₂S removal efficiency of 88.61–92.41% and 94.94–96.84% can be achieved using a single purification column and multiple purification columns. Table 2.4 presents a comparative analysis of various biogas purification methods, highlighting their key features such as efficiency, operational conditions, energy consumption, and the quality of the upgraded gas. This comparison provides valuable insights into the advantages and limitations of each method, allowing for a better understanding of their applicability in different biogas upgrading scenarios.

Table 2.4 Characteristics of different biogas purification technologies (Shah et al., 2021; Wu et al., 2024)

Technology	Type of process	Pressure (bar)	Energy consumption (kWh/m ³)	Methane purity (%)	Advantage	Disadvantage
Water scrubbing	Absorption	6–10	0.30–0.33	> 97	<ul style="list-style-type: none"> • Handling and operation are easy • Large volume gas can be processed • High CH₄ purity can be achieved 	<ul style="list-style-type: none"> • High cost of investment • High pressure and energy required • A huge amount of water required • High risk of biological contamination
Chemical scrubbing	Absorption	1–1.5	< 0.15	> 99	<ul style="list-style-type: none"> • Easy to operate • High efficiency • High CH₄ purity can be achieved • CO₂ and H₂S can remove easily 	<ul style="list-style-type: none"> • Investment in amine solvents is costly • Toxic solvents are required • Solutions are required for reasonable disposal
Solvent scrubbing	Absorption	4–7	< 0.25–0.33	> 96	<ul style="list-style-type: none"> • Simple process and easy operation • Fewer liquid inputs required • Smaller upgrading unit • High CH₄ purity • CO₂ and H₂S can be removed simultaneously 	<ul style="list-style-type: none"> • Toxic organic solvents • Difficult regeneration of organic solvents • High temperatures are required for H₂S separation.
Membrane separation	Permeation	20–36	< 0.18–0.35	> 90–99	<ul style="list-style-type: none"> • No usage of chemicals • High selectivity to impurities • Removes H₂O vapour • Less mechanical deterioration 	<ul style="list-style-type: none"> • Cost of membranes is high • Multiple purification steps are needed • Low membrane selectivity • Pretreatment is needed

Cryogenic separation	Compression & condensation	40	< 0.18–0.66	> 99	<ul style="list-style-type: none"> • High CH₄ purity can be achieved • No use of chemicals and water • Reuse of CO₂ is possible 	<ul style="list-style-type: none"> • Investment and operation costs are high • High energy is required
Pressure swing adsorption	Adsorption/desorption	4–8	< 0.25	> 96	<ul style="list-style-type: none"> • Dry process • No solvent and water needed • No microbial impurity 	<ul style="list-style-type: none"> • Higher CH₄ loss • Pre-purification equipment is required to remove H₂S • Pre-drying is needed to remove water from biogas
Biological methods	Photosynthetic/reaction	Ambient pressure	–	> 99	<ul style="list-style-type: none"> • High CH₄ recovery • Transforms CO₂ into other products • Production of active biomass • Low requirements for land and H₂O 	<ul style="list-style-type: none"> • High investment cost and energy; • Low photosynthetic CO₂ uptake; • High risk of biological contamination.
	Chemoautotrophic reaction	Ambient pressure	–	> 99	<ul style="list-style-type: none"> • High selectivity, process efficiency, and CH₄ purity • Can be integrated with the AD process 	<ul style="list-style-type: none"> • A high amount of reductant is needed • Still under development

2.9.1 Biochar as a potential CO₂ and H₂S adsorbent

Biochar produced from readily available biomass can be a promising adsorbent for CO₂ and H₂S adsorption from biogas. Biochar is a solid carbonaceous adsorbent that can be obtained via pyrolysis, gasification, torrefaction, or hydrothermal processing of various biomass such as woody biomass, animal manures, food wastes, seaweed, etc. BC is approximately ten times cheaper than other CO₂ adsorbents because of the wide availability of biomass (Dissanayake et al., 2020). The adsorption capacity of BC, which is the amount of gas adsorbed per unit weight of BC, mainly depends on the physico-chemical properties of the BC, such as the surface area, pore size, pore volume, basicity of BC surface, presence of surface functional groups, presence of alkali and alkali earth metals, hydrophobicity, polarity, and aromaticity (Chiang en Juang, 2017; Dissanayake et al., 2020).

The adsorption occurs via van der Waals forces between gas molecules and BC and is influenced by its physical properties, including specific surface area, pore diameter, and pore volume of BC (Zhang et al., 2014). Creamer and Gao (2016) highlighted that a larger surface area of BC offers more active sites for CO₂ adsorption via physical processes. As a result, BC, with a greater surface area, exhibits a more capacity for CO₂ adsorption. The surface area of BC increases with higher pyrolysis temperatures and longer residence times, likely due to the release of volatile matter, which develops the pore volume. Further, the pore volume and pore diameter also play an essential role in CO₂ adsorption. The release of volatile organic matter from the polymeric backbone of the feedstock causes the formation of porous structures in BC, and a larger total pore volume provides more active sites for interaction between CO₂, H₂S and BC (Dissanayake et al., 2020). Several studies have reported that micro-pores with a diameter of less than 1 nm are more significant to CO₂ adsorption (Chiang and Juang, 2017; Zhang et al., 2013).

The adsorption of CO₂ and H₂S onto the BC surface is also affected by the chemical properties of the BC. The surface functional groups of BC play an important role in CO₂ adsorption because of their contribution to surface basicity, which enhances the affinity of the BC for CO₂ (Dissanayake et al., 2020). N-containing functional groups like amide, imide, pyridinic, etc., as well as O-containing functional groups such as ketones, pyrones, and chromenes, enhance surface basicity (Xing et al., 2014). In contrast to the acid-base interaction between CO₂ and BC surface, several researchers indicate that O-containing acidic functional groups, like hydroxyl, carboxyl, and carbonyl groups, enhance CO₂ adsorption on carbonaceous surfaces by promoting H-bonding between CO₂ molecules and the carbon surface (Dissanayake et al., 2020; Xing et al., 2014). Further, the presence of alkaline metals and alkaline earth metals such as Na, K, Ca, and Mg may increase the CO₂ and H₂S adsorption capacity of BC through chemisorption, as the presence of such metals can improve the basicity of BC (Ma et al., 2022; Xu et al., 2016). Several investigators reported that the hydrophobicity and polarity of BC affect the CO₂ capture performance of BC. The BC with hydrophobic and non-polar properties can enhance CO₂ adsorption by reducing the competition from water molecules. Low H/C and O/C ratios (<0.2) signify a high degree of aromaticity and fixed carbon, which are chemically stable (Dissanayake et al., 2020). As the pyrolysis temperature increases, the molar ratios of O/C and H/C decrease, likely due to the loss of volatile organic compounds and the enhancement of dehydrogenation and deoxygenation reactions. These processes lead to the formation of more aromatic structures, reducing the BC's polarity and increasing its hydrophobicity (Huang et al., 2017; Shaaban et al., 2014).

Moreover, the adsorbent does not require regeneration after saturation with sulfurous compounds, and it can be directly used as a soil amendment product and as a beneficial fertiliser (Creamer et al., 2014; Zhang et al., 2017). The various studies reported in scientific publications on biochar's CO₂ and H₂S adsorption capacity are illustrated in Table 2.4.

However, very little literature on biogas purification using BC produced from waste biomass is available. In the present study, adsorbent materials, like biochar, are considered to purify biogas.

Table 2.5 Biochar utilisation for adoption of CO₂ and H₂S

Reference	Biomass	Biochar preparation	Findings/Results
Bora et al. (2024)	Bamboo and banana peel	Biochar was prepared at 500 °C for 3h	<ul style="list-style-type: none"> Approximately 89.2% and 87.7% of H₂S were removed from the raw biogas using banana peel and bamboo BC while utilizing the packed bed system
Zhang et al. (2024)	Pine and corn straw	BCs were prepared after hydrothermal pre-treatment using FeCl ₃ as a medium at pyrolysis temperatures of 700, 800, and 900°C	<ul style="list-style-type: none"> The CO₂ adsorption capacity of corn straw BC was increased from 3.07 wt% to 4.42 wt% when the pyrolysis temperature increased from 700 to 900 °C The CO₂ adsorption capacity of pine straw BC gradually decreased from 5.35 wt% to 3.73 wt% with increasing pyrolysis temperature
Zhang et al. (2023)	Bamboo charcoal, wood pellet, and coconut shell	Each BC was modified through physical (H ₂ O, CO ₂) and chemical (ZnCl ₂ , KOH, H ₃ PO ₄) activation to improve carbon capture performance	<ul style="list-style-type: none"> The results show that a more than 2.35 mmol·g⁻¹ (1 bar, 298 K) CO₂ capture capacity was achieved using the activated biochar samples
Mukherjee et al.(2021)	Spent soffee ground (SCG)	BC were prepared at 400, 500 and 600°C	The maximum CO ₂ removal capacity of SCG 600 obtained was 2.8 mmol/g under the investigated adsorption temperature at 30 °C
Choudhury and Lansing (2021)	Corn Stover and maple wood	The BC was prepared in an inert N ₂ atmosphere for 10 min at 500 °C. Modified biochar was obtained by impregnating Iron	<ul style="list-style-type: none"> Iron impregnation led to an increase in H₂S adsorption capacity by up to 3.9 times; Fe₃O₄ in the impregnated biochar was converted to FeSO₄ following its reaction with H₂S
Wuri et al. (2021)	Sugarcane Bagasse (SB)	BC was made at 255°C for 3 hours.	<ul style="list-style-type: none"> The highest CO₂ reduction was achieved with 50% SB-based BC and 50% natural zeolite

Sawalha et al. (2020)	Almond shells, eucalyptus and coffee grains	Biomass was pyrolyzed at 500°C for 1 h inside a muffle furnace; after that, activated carbon was prepared with reagents: KOH and ZnCl ₂	<ul style="list-style-type: none"> Eucalyptus demonstrated an adsorption capacity of approximately 690 mgH₂S/g BC, followed by almond at 230 mg/g and coffee grounds at 22 mg/g. BC activated with KOH exhibited higher adsorption efficiency and capacity compared to ZnCl₂
Pelaez-Samaniego et al. (2020)	Wood chips of pinus patula (PP) and eucalyptus grandis (EG)	Gasification chars were generated using a downdraft gasifier. The EG char was produced by gasifying EG with an airflow of 30 L/min, while the PP chars were obtained from PP using airflows of 20 L/min and 40 L/min	<ul style="list-style-type: none"> PP chars produced at higher airflow rates are more effective (81%) in biogas purification compared to activated carbon. Gasification temperature and airflow, along with surface area, affect the most
Promraksa and Rakmak(2020)	Palm oil mill residues	Pyrolysed at 500°C	<ul style="list-style-type: none"> Adsorption capacity for CO₂ was found to be 0.46 mmol/g
Zhu et al. (2020)	Wood	Wood BC was obtained by pyrolysis at 450 °C and then activated	<ul style="list-style-type: none"> Wood BC showed higher H₂S removal capability (2.2 mgH₂S/g) compared with sludge-activated carbon (0.1 mgH₂S/g) because of the high mesoporous structure
Igalavithana et al. (2020)	Pine sawdust and paper mill sludge	BC were prepared at 550°C and steam activated for 45 min at the same temperature. Paper mill sludge BCs were produced at 300 and 600°C	Due to high surface area and microporosity, pine sawdust BC showed significantly higher CO ₂ adsorption capacity than paper mill sludge BC.
Sahota et al. (2018)	Tree leaf waste	BC was prepared at 200°C, 300°C and 400°C through slow pyrolysis for a period of 3h using a traditional kiln	<ul style="list-style-type: none"> BC prepared at 400°C removes 84.2% H₂S (from 1254 ppm to 201 ppm) from raw biogas for 25 min in a continuous adsorption tower
Sethupathi et al.(2017)	Perilla leaf, soybean stover, Korean oak and Japanese oak	Prepared at 700°C	<ul style="list-style-type: none"> They reported that H₂S adsorption was preferred more than CO₂ due to the physical and chemical properties of the BCs
Zulkurnai et al. (2017)	Sea mango	Activated BC prepared at 500°C	BC derived from sea mango with 9.851 mg/g CO ₂ adsorption capacity

Madzaki et al. (2016)	Sawdust biochar	BC were prepared at 450, 750 and 850°C	<ul style="list-style-type: none"> The amount CO₂ of adsorption were 0.23, 0.43, and 0.47 kg CO₂/kg BC at a temperature of 30°C.
Creamer et al. (2014)	Sugarcane bagasse (SB) and hickory wood	BC was prepared at 300, 450, and 600°C	<ul style="list-style-type: none"> SB biochar produced at 600°C showed the most adsorption of CO₂ (73.55 mg /g at 25°C)

2.10 RESEARCH GAPS OF THE LITERATURE REVIEW

Countries like India, which boasts the largest rural population and vast agricultural land, have massive biogas production and consumption potential. Despite several advantages, bio-energy is not yet widely used in rural regions. The Indian rural area, which is located outside the townships with limited access to domestic fuel and energy but with a greater focus on agriculture and landscapes, generates a variety of biogas feedstock substrates such as animal manure, agricultural residues, and energy crops.

Anaerobic digestion is considered one of the most environment-friendly alternatives for effectively managing biodegradable waste, such as cattle dung and food waste. This review analysed the operating parameters of anaerobic digestion, co-digestion studies, the impact of biochar addition in anaerobic digestion, and the adsorption capacity of biochar for CO₂ and H₂S from biogas during purification. Anaerobic digestion of cattle dung and food waste is often claimed to have a higher biogas potential. Still, assessing which technology holds the most significant potential for cattle and food waste and its economic feasibility is difficult. Co-digestion of cattle dung and food waste has rarely been conducted with biochar addition besides other additives. Biochemical methane potential assays were seldom performed to determine the optimum substrate-water mixing ratio and co-substrate mixing ratio with the optimum quantity of biochar addition. Optimising an ideal mixing ratio and the optimum amount of biochar that needs to be added are essential for improving the overall anaerobic digestion process.

Further, controlling temperature in field-scale anaerobic digestion plants is crucial in rural areas of India. A solar-assisted water heating system is the only solution due to rural areas' lack of electrical connectivity. Further purification of biogas is essential. Only a few studies have found the use of biochar, especially in the adsorption of CO₂ and H₂S from biogas. Therefore, applying biochar prepared from waste biomass for biogas purification is crucial, as it reduces waste biomass and unwanted environmental impact.

2.11 THESIS OBJECTIVES

Based on the research gaps and availability of the resources, the following objectives have been formulated for the present investigation:

1. To investigate the effect of operating parameters on the biogas production rate in batch-type lab-scale anaerobic digestion with cattle dung and food waste as substrate
2. To analyse the effect of the co-digestion on biogas production rate both with food waste and cattle dung with biochar addition followed by biochemical methane potential.
3. To investigate the performance of field-scale one cum anaerobic digestion plant with and without a controlled environment using cattle dung as a feedstock.
4. To develop a biogas purification system to remove CO₂ and H₂S.

The next chapter discusses the methods and materials designed to address the above objectives.

CHAPTER 3

MATERIALS AND METHODS

Various experimental investigations were conducted to accomplish the objectives of the present research work. The anaerobic digestion experiments were performed in different phases utilising cattle dung and cooked and uncooked food wastes as feedstocks. The enhancement of biogas generation and purification using various biochars was also conducted. The detailed methodology is summarised below.

3.1 EXPERIMENTAL FLOW CHART

In order to accomplish the objectives of the research, experiments were conducted in various phases, as illustrated in Fig. 3.1. In phase I, the anaerobic digestion experiments were performed on the laboratory scale using cattle dung, vegetable waste and cooked kitchen waste individually, as the substrate to identify the optimum substrate-water (S/W) ratio as well as the effect of various operating parameters such as temperature, pH value, and volatile solid (VS) degradation were also studied. Further, considering the ideal S/W ratio, biochemical methane potential (BMP) tests were performed by varying the co-substrate mixing ratio (SMR) to detect the best SMR for AcoD without and with biochar addition under uncontrolled and controlled temperatures. In phase II, field-scale anaerobic digestion experiments were performed considering the best F/W ratio detected in phase I experiments for cattle dung under controlled and uncontrolled temperatures. The controlled experiments were performed using a solar-assisted water heating (SAH) system. Biogas purification experiments were conducted in phase III using the novel biochar produced from different bio-waste.

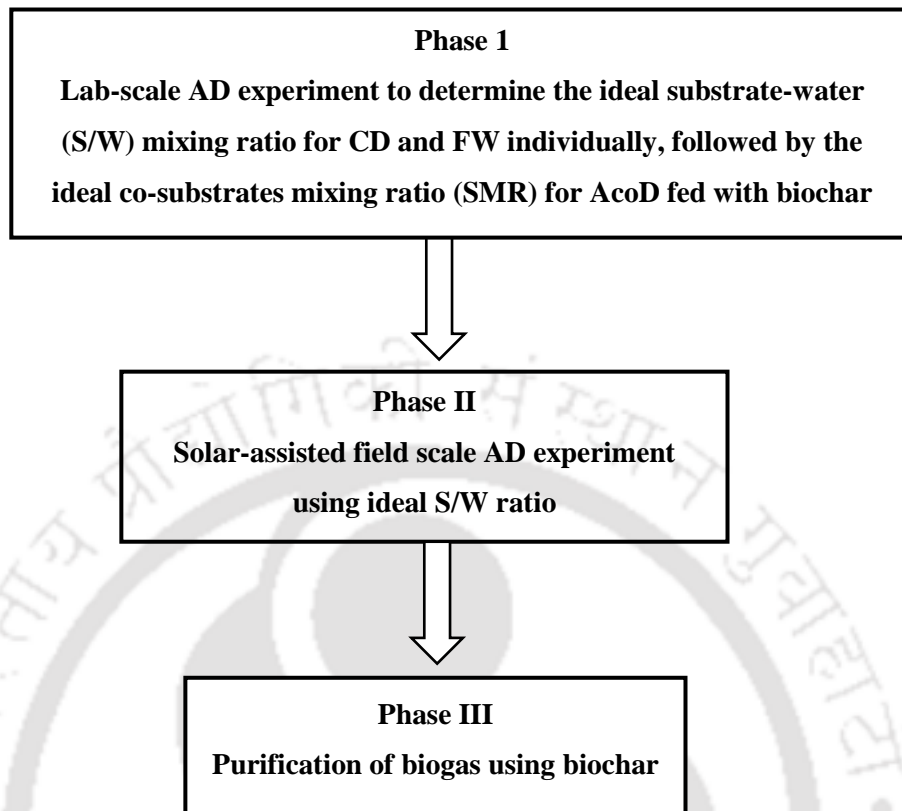


Fig. 3.1 Workflow diagram of the present work

3.2 ANAEROBIC DIGESTION STUDY

This study carried out anaerobic digestion experiments for CD and VW individually on a laboratory scale using the water-displacement method under controlled and uncontrolled temperatures. However, BMP tests were performed for CKW. The details of the collection and characterisation of substrates and experimental set-up are illustrated in the following section.

3.2.1 Substrates collection and preparation

Uncooked vegetable waste (VW) and cooked kitchen waste (CKW) were collected from the Indian Institute of Technology Guwahati (IITG) hostel mess, Assam, India (latitude 26.1879° N, longitude 91.6916° E). Cattle dung (CD) was collected from a farm near the IITG campus (Fig. 3.2). The CKW mainly comprised brinjals, potatoes, cabbages, steamed rice,

beans, red lentils, and spices. In contrast, uncooked VW was a mixture of green mustard leaves, cabbages, potatoes, long gourds, eggplants, radish leaves and other vegetables. VW and CKW were ground individually using a high-speed mixer grinder before being introduced into the digester. The substrates were dried in the hot air oven at 105 °C for 24 h for moisture removal, then powdered and sieved using a 0.2 mm sieve (Dhamodharan et al., 2015). The sieved powder sample was used for characterisation.



Fig. 3.2 (a) Chopped vegetable waste, (b) cooked kitchen waste, and (c) cattle dung

3.2.2 Characterisation of substrates

Moisture content (MC), total solids (TS) and volatile solids (VS) contents in the substrates were determined as per the guidelines of the American Public Health Association (APHA, 2017). Substrates were kept in a hot air oven (Optics Technology, India) at 105 °C for 24 h to determine the MC, whereas VS was determined using a muffle furnace (Optics Technology, India) at $550 \pm 2^\circ\text{C}$ for 2 h. Carbon (C), Hydrogen (H) and Nitrogen (N) contents in both the substrates were measured using the elemental analyser (Euro EA, Model: Eurovector EA 3000, Italy), and Oxygen (O) content was deduced by difference as per ASTM standards D5373-16. All the parameters were measured thrice, and average values were considered.

3.2.3 Lab scale experimental setup for biogas production

Laboratory scale experiments were carried out to identify the best feedstock-water (F/W) mixing ratio for anaerobic digestion of cattle dung and vegetable waste. Glass reagent

bottles with a capacity of 1000 mL were used as digesters. The feedstock-water mixtures were fed into the digesters, and all the fittings and joints were sealed tightly to prevent leakage of gas. One end of a flexible pipe was inserted into the digester while the other end was connected to the gas washing bottle filled with water. Nitrogen gas was purged into the digester for 2–3 minutes at the beginning of the experiments to create anaerobic conditions. Temperature-controlled experiments were performed under mesophilic ($35\pm 2^\circ\text{C}$) and thermophilic ($55\pm 2^\circ\text{C}$) conditions. Hot water baths (Equitron Medica Pvt. Ltd., Model: #8428, India) were used to maintain the digesters at mesophilic and thermophilic conditions. The daily gas production was measured using the water displacement method and recorded for the HRT of 55 days. The digesters' temperature was measured every four hours using thermocouple sensors (Make: Digi-Sense, Model 20250-03). The set-up was comprised of a provision to collect the sample from the digester for weekly measurement of pH value throughout the retention time using a pH meter (Make: Eutech, Model: 700).

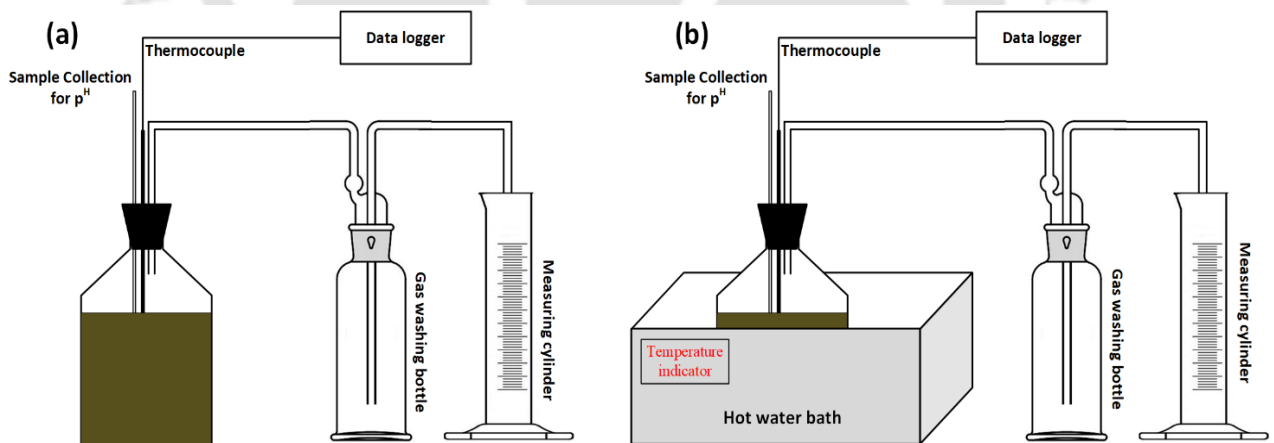


Fig. 3.3. Schematic diagram (a) Uncontrolled experimental setup (b) Controlled experimental setup

3.2.4 Biochemical methane potential (BMP) experimental setup

BMP tests were conducted to investigate the methane generation potential from the bio-waste using 1000 mL reagent glass bottles as the digesters. The total slurry volume of each digester was set to 700 mL, and subsequently, all fittings and joints were sealed to avoid any potential leakages. A silicon pipe was connected to the digester, and the aspirator bottle was filled with a 1.5N NaOH solution (Barua et al., 2018b). Alkali indicator thymol blue was also added to 1.5N NaOH solution. The daily methane yield (DMY) was measured using the liquid-displacement method. Researchers reported that 1.5N NaOH reacts with CO₂ and produces carbonate compounds by absorbing CO₂ gas (Barua et al., 2018b). Henceforth, the amount of liquid displaced from the aspirator can be considered as the amount of CH₄. A provision was made for purging nitrogen gas. Nitrogen gas was purged inside the digester for 2–3 minutes to make it anaerobic at the start of experiments. The pH value was recorded weekly using a pH meter (Mettler Toledo, Model: FEP20-Plus). Experiments were conducted at ambient (uncontrolled) and controlled temperatures. The temperature was measured with the help of thermocouples (T-type) sensors (Digi-Sense, Model 20250-03) at an interval of 3h for uncontrolled digesters. A hot water bath (Equitron Medica Pvt. Ltd., Model: #8428, India) was used to maintain the digesters at mesophilic (35±2°C) conditions. The schematic diagrams of the BMP test setup are depicted in Fig. 3.4.

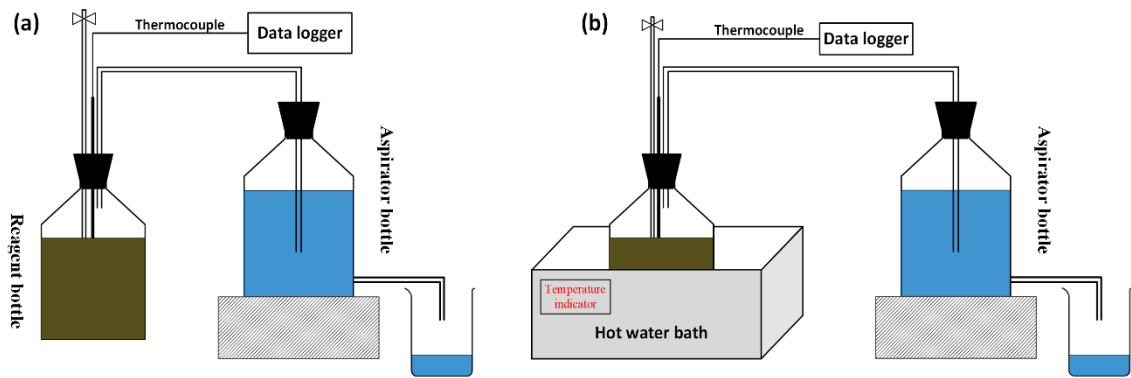


Fig. 3.4. Schematic diagrams of the BMP test setup at (a) ambient, (b) mesophilic conditions, and (c) Photograph of BMP set-up

3.2.5 Experimental matrix for anaerobic digestion

The experimental design for anaerobic digestion is illustrated in the following sections.

3.2.5.1 Anaerobic digestion of cattle dung and vegetable waste

Two sets of laboratory-scale experiments for biogas production were conducted using batch-type anaerobic digesters. In the first set, the biogas production rate for cattle dung and vegetable waste was investigated at different feedstock-water (F/W) ratios under uncontrolled conditions. Four different F/W ratios were maintained for the substrates while conducting the experiments, as revealed in Table 3.1. The second set of experiments dealt with the production

of biogas for both CD and VW under mesophilic (35 ± 2 °C) and thermophilic (55 ± 2 °C) conditions. Here, the digesters were prepared considering the best F/W ratio for both the substrates and the digesters were placed in a stirred hot water bath (Make: Equitron Medica Pvt. Ltd., Model: #8428, India) for maintaining controlled condition throughout the experiment. The schematic diagrams of both the experimental setups are depicted in Fig. 3.2.

Table 3.1 Experimental matrix for AD of CD and VW

Cattle dung (CD)				
F/W ratio	Digester	Feedstock (g)	Water (g)	Total quantity (g)
20:80	CD1	140	560	700
40:60	CD2	280	420	700
60:40	CD3	420	280	700
80:20	CD4	560	140	700
Vegetable waste (VW)				
F/W ratio	Digester	Feedstock (g)	Water (g)	Total quantity (g)
20:80	VW1	140	560	700
40:60	VW2	280	420	700
60:40	VW3	420	280	700
80:20	VW4	560	140	700

3.2.5.2 Biochemical methane potential test for cooked kitchen waste

BMP tests for AD of CKW were conducted for 45 days. Five different F/W ratios, such as 20:80, 40:60, 50:50, 60:40, and 80:20, were maintained for the BMP test (Table 3.2). Experiments were performed at ambient temperature using 1000 mL glass reagent bottles as the reactors. The total working volume of each reactor was 700 mL. The reactors were sealed with rubber corks and connected to aspirator bottles containing 1.5 N NaOH. The detailed experiment procedure was summarised in Section 3.2.4 (BMP test set-up).

Table 3.2 Experimental matrix for AD of CKW

Digester	Mixing ratio (CKW: W%)	CKW (g)	Water (g)
CKW20:W80	20:80	140	560
CKW40:W60	40:60	280	420
CKW50:W50	50:50	350	350
CKW60:W40	60:40	420	280
CKW80:W20	80:20	560	140

3.3 ANAEROBIC CO-DIGESTION STUDY

In this study, BMP experiments were carried out for AcoD of CD with VW and CD with CKW in a laboratory scale under controlled and uncontrolled temperatures with and without the addition of biochar. The details of the materials collection, characterisations, and experimental set-up are illustrated in the following section.

3.3.1 Substrates collection

The details of substrate collection for AcoD are already mentioned in section 3.2.1.

3.3.2 Materials collection and preparation of biochar

Biochars derived from water hyacinth (WH) and coconut husk (CH) were incorporated into the anaerobic co-digestion study. Water hyacinth (WH) was collected from a lake on the IIT Guwahati campus. Initially, WH was washed with water to remove the soil and dust particles, followed by cutting into small pieces (around 3 mm). The water hyacinth underwent sun-drying for 5–6 days. Dry CH was obtained from a market near the campus. The CH was also cut into small pieces (around 3 mm). Subsequently, WH and CH were dried in a hot air oven (Hamco, India, Model:105 series) for 24 h at 105 °C to remove moisture. Consequently, the pyrolysis was performed individually for WH and CH by utilising a pyrolyser (Das and Co, India, Model: DC/RTF/8) at 550 °C, employing a heating rate of 10 °C per minute, and maintaining a residence time of 1 h. Argon gas was supplied to maintain oxygen-free conditions

inside the pyrolyser. The BC was ground in a high-speed mixer grinder followed by sieving using a mesh size 100 μm sieve and then added to anaerobic digesters. For analytical characterisation, sieved powder samples of BC were dried in the oven for 24 h at 105 $^{\circ}\text{C}$ before the analysis. A schematic diagram and photograph of the pyrolyser are depicted in Fig. 3.5.

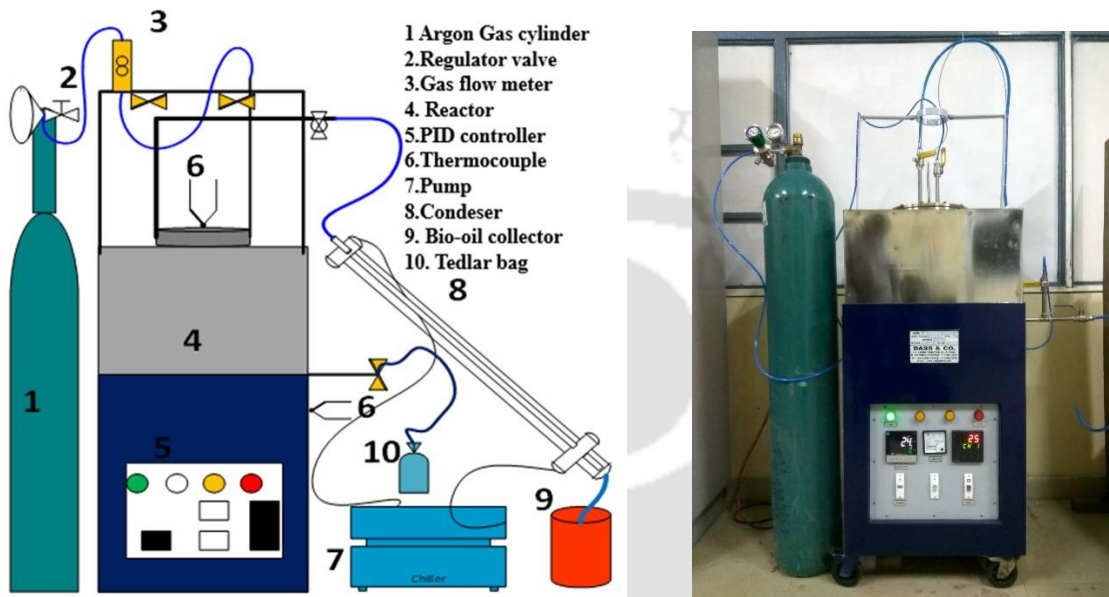


Fig. 3.5. Pyrolyser (a) Schematic diagram (b) Photograph

3.3.3 Characterisation of biochars

The MC, ash content (AC), and volatile matter (VM) content were analysed as per the protocols outlined in ASTM E871-82, ASTM D1102-84, and ASTM E872-82, respectively, and fixed carbon (FC) content was determined by mass difference. Ultimate analysis was conducted to find C, H, N, S, and O composition using a CHNS analyser. The pH value of BC samples was measured by mixing the BC and distilled water in the ratio of 1:10 (w/v), as reported by (Sunyoto et al., 2017) using a pH meter (Mettler Toledo, Model: FEP20-Plus). The surface morphology of BC was examined by a field emission scanning electron microscope (FESEM) (Zeiss, Germany, Model: Sigma 300). The samples were put on a stainless-steel stub using carbon tape followed by a double gold coating to defer degradation and to develop a

charge. The samples were then analysed using SEM imaging. The surface elemental composition was also analysed to estimate the presence of mineral matter in BC using energy-dispersive X-ray spectroscopy (EDS) (Zeiss, Germany, Model: Sigma). The crystalline constituents of BC were detected using a powder X-ray diffractometer (XRD) with $\text{CuK}\alpha$ radiation at $\lambda=1.54 \text{ \AA}$ (Rigaku Technologies, Japan, Model: Smart Lab). The diffraction pattern was recorded in the scanning range (2θ) of $10^\circ-80^\circ$ at a scanning speed of $4^\circ/\text{min}$. Brunauer–Emmet–Teller (BET) pore size, surface area, and pore volume were investigated using a BET analyser (Quantachrome Instruments, Model: Autosorb, IQ MP). Vacuum degassing of BC was performed before the analysis for 5 h at 200°C . Fourier transform infrared (FTIR) spectra were recorded to detect the surface functional groups using an FTIR spectrometer (PerkinElmer, Singapore, Model: Spectrum two) at wave numbers from 4000 to 400 cm^{-1} with 4 cm^{-1} resolutions.

3.3.4 Experimental matrix for anaerobic co-digestion

The experimental design for anaerobic co-digestion of cattle dung with food waste is described in the following sections.

3.3.4.1 Anaerobic co-digestion of cattle dung and vegetable waste

Three sets of BMP experiments were initially performed at varying percentages of CD: VW mixing ratio such as 70:30, 60:40, 50:50, 40:60 and 30:70 on a wet mass basis simultaneously in triplicate with and without BC blending using 1000 mL glass reagent bottles as a batch-type digester, as revealed in Table 3.3. The feedstock-distilled water was maintained at a ratio of 1:1. Entire experiments were performed under uncontrolled and controlled environments for 45 days. Sets I represent the experiments without BC blending, whereas 10 g of water hyacinth BC was added randomly in each of the digesters represented by sets II and III. Moreover, set III was maintained at mesophilic ($35\pm 2^\circ\text{C}$) conditions using a hot water bath, whereas sets I and II were conducted under uncontrolled temperature. The highest

methane generation potential was detected for the SMR of 60:40 in all three sets of experiments. Henceforth, considering the SMR 60:40, set IV experiments were carried out by adding different amounts of BC (in g/L): 5, 10, 15, 20, and 25 to identify the most feasible quantity of BC needed to be added for maximum CH₄ generation.

Table 3.3 Experimental matrix for anaerobic co-digestion of CD and VW

CD:VW(w/w #)	CD, g	VW, g	Water, g	Total
(a)	(b)	(c)	(d)	(e)=(b)+(c)+(d)
70:30	245	105	350	700
60:40	210	140	350	700
50:50	175	175	350	700
40:60	140	210	350	700
30:70	105	245	350	700

w/w weight by weight basis

3.3.4.2 Anaerobic co-digestion of cattle dung and kitchen waste

Four sets of BMP tests were initially performed at varying percentages of CD: CKW mixing ratio viz. 100:0, 0:100, 70:30, 60:40, 50:50, 40:60 and 30:70 on a wet mass basis simultaneously in triplicate with and without BC blending using 1000 mL glass reagent bottles as a batch-type digester, as revealed in Table 3.4. The CD: CKW of 100:0 and 0:100 were considered as the control digesters for the BMP test. The present study added no inoculum, considering CD as the active substrate. The feedstock-distilled water was maintained at a ratio of 1:1. Entire experiments were performed in uncontrolled and controlled temperatures for HRT of 55 days. Sets I and II represent the experiments without BC blending, whereas 15 g/L of coconut husk BC was added randomly in each of the digesters represented by sets III and IV. Moreover, sets II and IV were maintained at mesophilic (35±2 °C) conditions (Ferrer et al., 2010) using hot water baths (Equitron Medica Pvt. Ltd., Model: #8428, India), whereas sets I

and III were conducted under uncontrolled temperature. The schematic diagrams of the BMP test setup are shown in Fig 3.4. The highest methane generation potential was detected for the SMR of 40:60 in all four sets of experiments. Henceforth, considering the SMR 40:60, set V experiments were carried out under ambient temperature by adding different amounts of BC (in g/L): 5, 10, 15, 20, and 25 to identify the most feasible quantity of BC needed to be added for maximum CH₄ generation.

Table 3.4 Experimental matrix for anaerobic co-digestion of CD and CKW

CD:CKW(m/m #)	CD, g	CKW, g	Water, g	Total
(a)	(b)	(c)	(d)	(e)=(b)+(c)+(d)
100:0	350	0	350	700
70:30	245	105	350	700
60:40	210	140	350	700
50:50	175	175	350	700
40:60	140	210	350	700
30:70	105	245	350	700
0:100	0	350	350	700

m/m mass by mass basis

3.4 FIELD-SCALE EXPERIMENT STUDY

For the field-scale study, a Deenbandhu model anaerobic digester of 1 m³/day capacity was installed at the Technology Complex, IIT Guwahati, following all the design and construction protocols prescribed by (IRENA, 2016; Kudravelli, 2013). A helical type of heat exchanger made of copper was installed at the digester's bottom to maintain the digester's temperature in the mesophilic range (33.5±0.11°C). Two solar Photovoltaic (PV) panels (Vikram Solar, Model: Eldora VSP.72.315.03.04), each with rated peak power of 315 W, were used to run a DC pump (0.5 HP) to circulate the hot water from the evacuated tube (ET) solar

collector insulated storage tank (Nuotech Solar System Pvt. Ltd, India) of 500 litres capacity to the heat exchanger. Both solar PV and ET collectors were mounted on the ground at a 26.20° tilt angle facing the south direction. The tilt angles were fixed considering the latitude angle of Guwahati, India. The water circulating pump was run with a flow rate of 0.02 ± 0.004 kg/s at night (from 18.00 h) for 6–7 h. The flow rate was recorded with a water flow meter. A total of five thermocouples were fixed inside the digester to record the temperature of the slurry at different locations. The logarithmic mean temperature difference and effectiveness of the heat exchange were calculated, which are presented in Table 3.5. A stirrer was also installed inside the digester to break the scum formed during digestion. A provision was also made for sample collection at the top of the digester. The gas produced was collected in a biogas balloon (Siya Instruments). Experiments were conducted considering CD as substrate with the S/W mixing ratio of 1:1 (TS content: $7.70 \pm 0.02\%$). At the start of the investigation, 1375 ± 2.50 kg of CD was appropriately mixed with an equal amount of water and fed to the digester. The daily average feed of CD slurry (CD+W) was 50 ± 1.50 kg/day. The weekly digesters' temperature and weekly pH values were recorded using a data acquisition unit (Keysight, Model: 34972A) and a pH meter (Eutech, Model: 700). The schematic diagram and photograph of the experimental setup of the AD plant are depicted in Fig. 3.6 (a) and (b), respectively. The studies were carried out in three separate periods. The first and second periods of experiments were conducted in the year 2018–2019 without a solar-assisted heating (SAH) system for 28 weeks in the summer (April–October) and 16 weeks in the winter (November–February), respectively. However, the SAH system was implemented for the third experiment period in the following year, 2019–2020, during the winter season (November–February). The temperature was recorded at different coordinates of the AD plant every 3 h interval between 9.00 h Indian Standard Time (IST) and 1.00 h (IST) weekly.

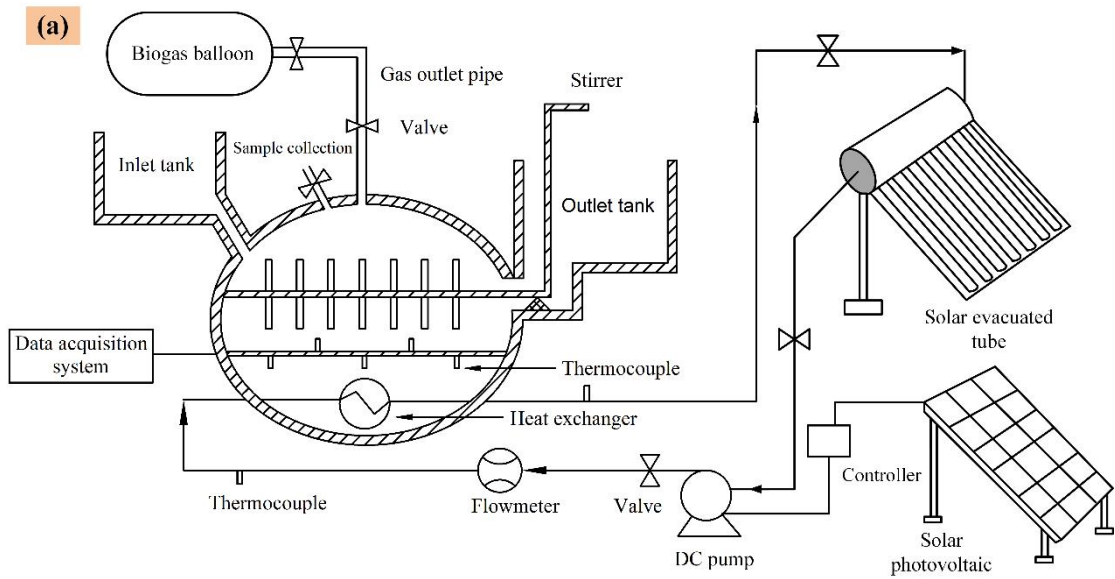


Fig. 3.6. Field-scale AD plant setup (a) schematic diagram (b) photograph.

Table 3.5 Parameters related to heat exchanger

Parameters	Value
Hot water inlet temperature (t_{h1}), °C	61.9±1.90
Hot water outlet temperature (t_{h2}), °C	36.6±2.04
Initial temperature of slurry (t_{c1}), °C	21.6±0.23
Final temperature of slurry (t_{c2}), °C	33.5±0.11
Logarithmic mean temperature difference, °C	21.0±0.04
Specific heat of water (C_{p1}), kJ/kg K	4.18
Specific heat of slurry (C_{p2}), kJ/kg K	4.16
Mass flow rate of water (m_h), kg/s	0.02±0.003
Effectiveness (ϵ)	0.63

3.5 KINETIC STUDY

The estimation of maximum CH₄ yield potential (Y_P), highest CH₄ yield rate (Y_R), and lag phase time (λ) were performed by simulating accumulated cumulative CH₄ yield (Y_C) value at a specified time (t) using the modified Gompertz equation (Zhang en Wang, 2021) illustrated in the Eq. (1). This model is widely acknowledged as a reliable method for predicting cumulative CH₄ generation potential (Barua et al., 2018b). The kinetic parameters Y_P , Y_R and λ were determined using Matlab R2021b software. Simultaneously, the coefficient of determination (R^2) was also calculated to assess the correlation between the model and the experimental data.

$$Y_C = Y_P \exp \left[- \exp \left\{ \frac{Y_R \times e}{Y_P} (\lambda - t) + 1 \right\} \right] \quad (3.1)$$

where e signifies Euler's number (equal to 2.71828).

3.6 STATISTICAL ANALYSIS

The statistical analysis was performed using the statistical product and service solutions (SPSS) 29 software (International Business Machines Corporation, USA), considering $p < 0.05$ as statistically significant.

3.7 PURIFICATION OF BIOGAS

3.7.1 Materials collection and preparation of biochar

For the study of purification, biochars were produced from coconut husk (CH), sugarcane baggage (SB), and water hyacinth (WH) using a pyrolyser (Das and Co, India, Model: DC/RTF/8). Dry CH and SB were collected from the local market near the campus. Meanwhile, WH was collected from a lake on the IIT Guwahati campus. After cleaning, it was cut into small pieces (around 3 mm) and sun-dried for 6–7 days. Before pyrolysis, CH and SB were also cut into 2–3 mm pieces' size. Before introducing the pyrolyser to remove moisture, each biomass was individually subjected to 24 hours of drying in a hot air oven (Hamco, India,

Model: 105 series) at 105°C. Consequently, the pyrolysis was performed individually at the carbonisation temperatures of 550°C, 450°C, and 350°C with a heating rate of 10°C/min, while maintaining a residence time of 1 h. Argon gas was supplied to maintain oxygen-free conditions inside the pyrolyser. The biochars prepared at 550°C represented CH 550, SB 550, and WH 550 for CH, SB, and WH, respectively. Likewise, CH 450, SB 450, WH 450, and CH 350, SB 350, and WH 350 represent 450 and 350°C, respectively.

3.7.2 Characterisation of biochars

The methodology for biochar characterisation before and after the purification was executed as outlined in section 3.3.3. However, only EDX, FTIR, and XRD analyses were conducted after being used to purify biogas.

3.7.3 Biogas purification experimental setup and procedure

Two types of biogas purification setups were considered in this study. Fig. 3.7 shows the schematic diagram of the biochar-based biogas purification set-up. The biogas scrubbing unit mainly consists of a compressor, biogas flowmeter, moisture separator, adsorbent column and pressure gauge. The adsorbents used for purification were CH, SB and WH biochars prepared at 550, 450 and 350 °C pyrolysis temperatures. Firstly, the biochar adsorbent was packed in an acrylic tube of 300 mm in length and 50 mm in diameter. Prior to that, a wire mesh screen (0.25 mm) was fitted at the bottom of the tube to support biochar (BC). The biogas was collected from the biogas plant (Deenbandhu model) installed at the Technology Complex (TC), IIT Guwahati, using a biogas balloon (Siya Instruments). The biogas was compressed using a compressor and allowed to pass through a moisture separator containing silica gel with a pressure of 1.2 ± 0.2 bar and, subsequently, through an acrylic tube packed with biochar. The column consists of three parts; the topmost (50 mm) and bottommost (50 mm) parts are outlets and inlets. Tedlar bags were used to collect the outlet gas; consequently, sample testing was analysed using a gas chromatograph (Nucon Engineers, Model: Series 576). The gas

chromatograph (GC) was operated under a thermal conductivity detector (TCD) with argon (Ar) gas as a carrier gas. The experiments were performed three times in order to establish repeatability.

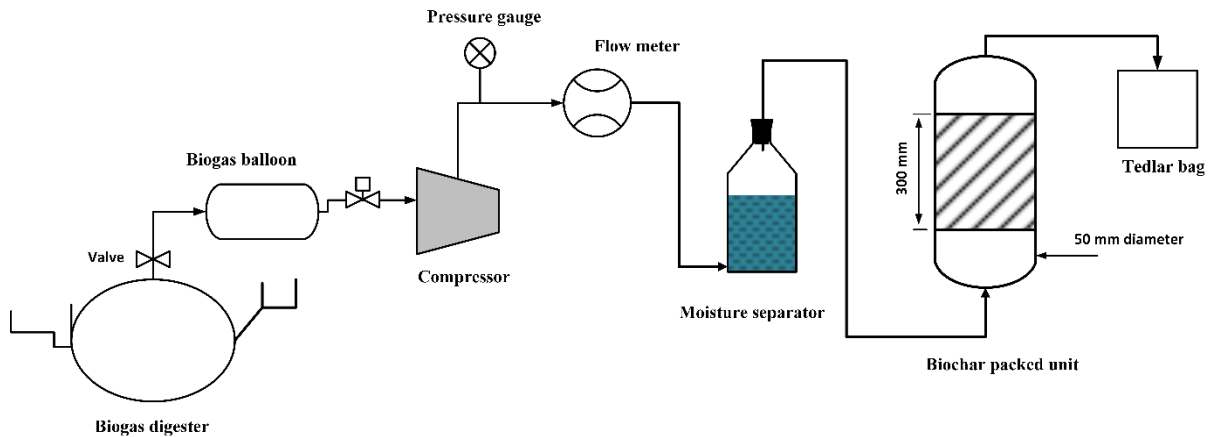


Fig. 3.7 Biochar based biogas purification setup

Fig. 3.8 shows the schematic diagram of a biochar-based biogas purification set-up with 1.5 N NaOH solution treatment. The description of set up 2 is almost similar to set up 1, as mentioned above. However, a glass reagent bottle containing 1.5 N NaOH was introduced between the biochar-packed column and the moisture separator. In this setup, the biogas was compressed using a compressor and allowed to pass through the acrylic tube packed containing biochar and then, gas was allowed to pass through the glass reagent bottle filled with 1.5 N NaOH (500 ml) to remove CO_2 , H_2S and other gases. Moreover, the gas from the glass bottle (1.5 N NaOH) was then allowed to pass through another silica gel washing bottle to absorb the moisture in the biogas. The gas was collected using a tedlar bag for sample testing. The composition of biogas was analysed using a gas chromatograph (Nucon Engineers, Model: Series 576). The gas chromatograph (GC) was operated under a thermal conductivity detector (TCD) with argon (Ar) gas as a carrier gas.

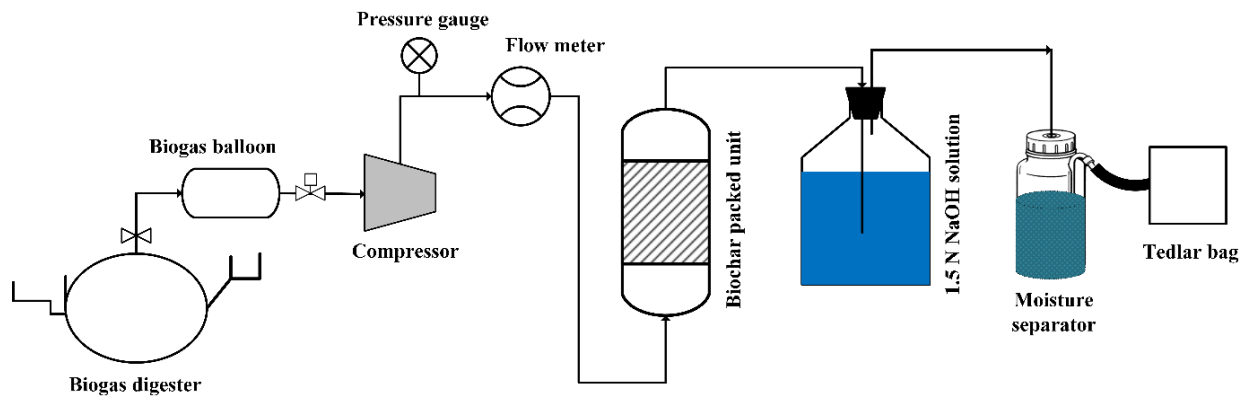


Fig. 3.8 Biochar-based biogas purification setup with 1.5 N NaOH treatment

3.8 SUMMARY OF METHODOLOGY

In Chapter 3, the methodology of the thesis is presented. The initial characterisation of substrates, the lab- and field-scale AD process methodology, and biogas purification have been discussed. In the next Chapter, the results of the batch-type lab-scale AD under controlled and uncontrolled environments were studied.

CHAPTER 4

PERFORMANCE INVESTIGATION OF BATCH TYPE

ANAEROBIC DIGESTION

This chapter includes the anaerobic digestion of cattle dung, vegetable waste and cooked food waste, focused mainly on the effect of various parameters like substrate-water (S/W) mixing ratio, co-substrate mixing ratio (SMR), pH value, and temperature on biogas production.

4.1 ANAEROBIC DIGESTION OF CATTLE DUNG AND VEGETABLE WASTE

4.1.1 Characterisation of substrates

The characterisation of feedstocks provides useful information about its suitability for AD potential for energy production. The MC, TS and VS contents in CD and VW were estimated from the proximate analysis and presented in Table 4.1. The TS contents for CD digesters with different F/W ratios were found to be in the range of 3–13%, while for VS digesters, it varied from 1.5–6.5%. TS content for 100% CD and 100% VW were observed to be $15.91 \pm 0.17\%$ and $8.37 \pm 0.02\%$ respectively. It can be inferred that the TS contents of VW digesters were almost 50% less than CD digesters, considering the same F/W ratio. Jain et al. (2015) reported that the TS content of cattle dung and pig manure slurries falls in the range of 3–12%. Meanwhile, the TS content of fresh cattle dung (100% CD) usually falls in the ranges of 15–19% (Salam et al., 2015; Shyam, 2001). According to Salam et al. (2015), several researchers considered the optimum value of TS content for cattle dung slurry in the range of 7–9% for maximum gas production. In the present study, VS contents of CD and VW digesters were found to be in the range of 80.5–83.5% and 93.0–94.5%, respectively. The results for CD are revealed to be consistent with the findings reported on cattle dung by Veluchamy and Kalamdhad (2017). Likewise, various researchers have reported the TS and VS contents in fruit

and vegetable waste for AD process as 8.9% and 77.2% (Gulhane et al., 2017), 8–18% and 86–92% (Bouallagui et al., 2005), 8.3% and 93% (Bouallagui et al., 2009), 9.5% and 92% (Edwiges et al., 2018), 11% and 87% (Bouallagui et al., 2004) respectively. The moisture content of CD and VW digesters were also determined and found to be in the range of 87–97% and 93–98.5%, respectively, whereas 100% CD and 100% VW samples showed 84.08 ± 0.26 % and 91.65 ± 0.02 % respectively. Zhang et al. (2007) characterised several food wastes and reported that the MC and (VS/TS) ratios were 74–90% and 80–97%, respectively, with a C/N ratio of 14.7–36.4. Hence, it is evident that the TS, VS, and MC contents for the substrates and slurries closely agreed with the literature and were within the acceptable range for biogas production.

Table 4.1 Initial characterisation of CD and VW

Parameters	Cattle dung digesters					Vegetable waste digesters				
	100% CD	CD1	CD2	CD3	CD4	100% VW	VW1	VW2	VW3	VW4
MC (%)	84.08 ± 0.26	96.88 ± 0.02	93.91 ± 0.04	90.82 ± 0.05	87.36 ± 0.07	91.65 ± 0.02	98.47 ± 0.03	96.83 ± 0.03	95.33 ± 0.05	93.66 ± 0.05
TS (%)	15.91 ± 0.17	3.09 ± 0.02	6.15 ± 0.04	9.16 ± 0.05	12.57 ± 0.07	8.37 ± 0.02	1.55 ± 0.03	3.22 ± 0.03	4.56 ± 0.08	6.34 ± 0.05
VS (% TS)	83.50 ± 0.10	80.75 ± 0.23	81.61 ± 0.09	82.19 ± 0.18	83.14 ± 0.10	94.25 ± 0.14	93.13 ± 0.17	93.46 ± 0.29	93.77 ± 0.07	94.08 ± 0.06

The elemental composition of the substrates was investigated and presented in Table 4.2. The carbon (C) content in VW was higher than in CD, while nitrogen (N) content was lower in VW than in CD, resulting in a higher C/N ratio for VW. The C/N ratio of substrates is essential for the AD system as it primarily influences the biogas yield. The optimal C/N ratio for anaerobic digestion has been found to be in the range of 20–30 (Hagos et al., 2017), 25–35 (Kondusamy en Kalamdhad, 2014) or between 25–30 (Y. Li et al., 2017a). Again, some researchers prefer a C/N ratio of 25 for the maximum production of biogas (Pang et al., 2008).

Substrates with lower C/N ratio increase ammonia accumulation, which is toxic to methanogenic bacteria and causes insufficient utilisation of carbon sources (Kothari et al., 2014; Mao et al., 2015; Sitorus et al., 2013). Conversely, with a higher C/N ratio of the substrates, the biogas production rate decreases because of the rapid nitrogen consumption by methanogenic bacteria (Kothari et al., 2014). The present investigation showed a C/N ratio of 25.82 for CD, which falls within the optimal range of the C/N ratio. At the same time, the C/N ratio of VW was 37.13, which is comparable, as reported by Sitorus and Panjaitan (2013), but slightly higher than the optimal value. Therefore, VW's higher C/N ratio may adversely affect the biogas yield. A detailed study on biogas production from VW has been provided in section 4.1.3.3.

Table 4.2 Chemical compositions of CD and VW

Feedstock	C (%)	N (%)	H (%)	O (%)	C/N
CD	36.69	1.42	3.51	58.39	25.82
VW	37.88	1.02	4.43	56.67	37.13

4.1.2 FTIR analysis

The raw cattle manure (100% CD) and vegetable waste (100% VW) were characterised using FTIR spectroscopy, and the ensuing spectrum with corresponding wave numbers is given in Fig.4.1. The wide peaks at 3281.63 cm^{-1} and 3286.64 cm^{-1} for CD and VW represent the O-H stretching vibration of the phenol and carboxylic group (Lv et al., 2013). The peaks observed at 2917.40 and 2840.23 cm^{-1} for CD were assigned to the aliphatic methylene group's C-H (CH_3 and CH_2) stretching vibration (Ali et al., 2012). Further, for CD, the peak at 1736.76 cm^{-1} ascribed to the C=C, C-O group and 1634.76 cm^{-1} assigned to the C=O group suggested the presence of different groups of lignin and amide I or carboxylates groups, respectively (Ali et al., 2012; Lv et al., 2013; Yadav et al., 2017). These peaks were absent in the case of VW. On

the other hand, the peak showed for VW at 2359.98 cm^{-1} was due to C-O asymmetrical stretching vibration (Whitely et al., 2006), and 1594.73 cm^{-1} characterizes the N-H deformation and C=N stretching of amide II group. These two peaks were unidentified in CD. Peaks obtained at 1412-1420 cm^{-1} illustrated the COO^- and C-N stretching due to carboxylic acid and amide III groups, respectively. Then again, peaks attained at 1012–1032 cm^{-1} corresponded to the stretching vibration of C-O of polysaccharides or polysaccharide-like substances and also established the presence of hemicellulose, cellulose and lignin, while 515-530 cm^{-1} ascribed the C-H bond due to the aromatic structures (Hihu Muigai et al., 2021). It was inferred from the study that both feedstocks possessed a decomposable composition that was helpful for the anaerobic digestion process.

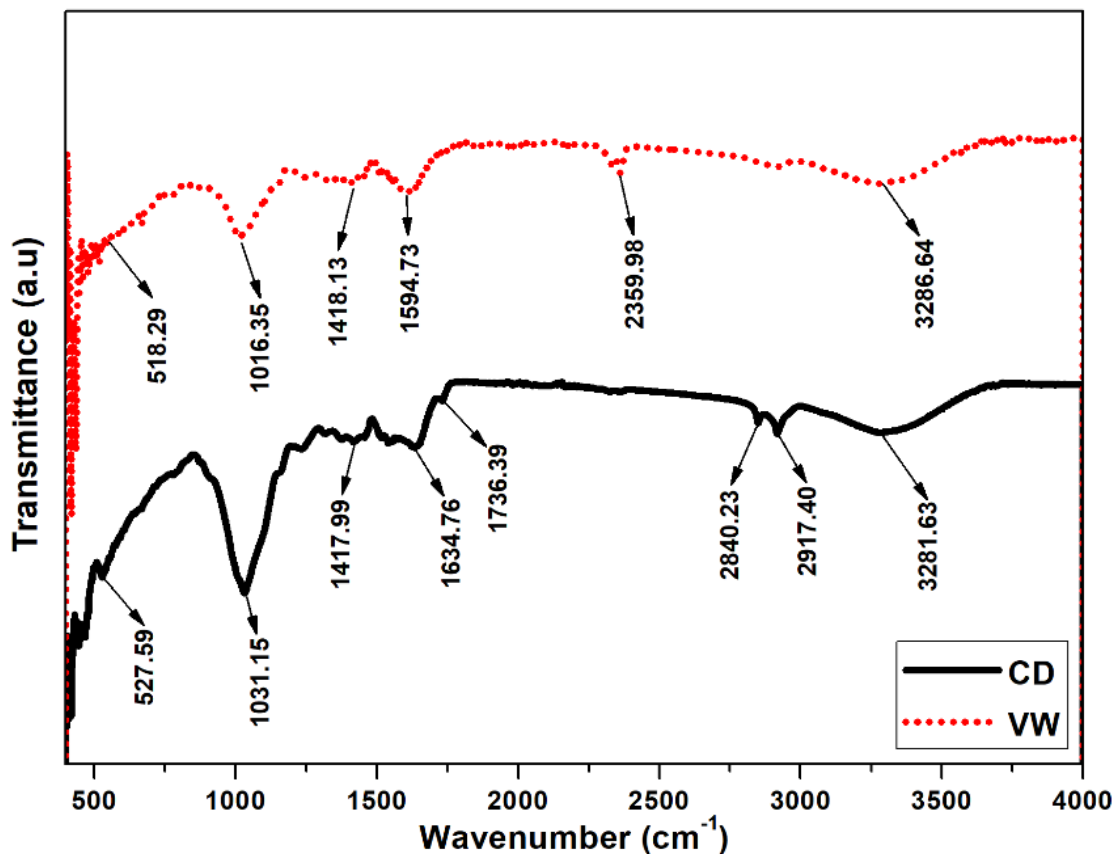


Fig. 4 1. FTIR spectra for cattle dung and vegetable waste

4.1.3 Experimental analysis of uncontrolled digesters

Anaerobic digestions carried out without controlling the temperature (ambient condition) for both CD and VW are presented in this section. Four different F/W ratios were maintained for CD and VW individually, and variations of pH, temperature and daily biogas yield were recorded and compared.

4.1.3.1 Variation of pH value

The pH value plays an essential role in the AD process, which measures the substrates' acid concentration. Many investigators analysed the pH value during the AD process, and the most favourable range was reported to be between 6.8 and 7.2 (Ali et al., 2012; Hagos et al., 2017; Hamelin et al., 2014). The comparison of pH values for cattle dung (digesters CD1, CD2, CD3 and CD4) and vegetable waste (digesters VW1, VW2, VW3 and VW4) measured weekly are presented in Fig. 4.2. At the initial stage of digestion, pH values for cattle dung digesters were observed to be in the acidic range, i.e. between 5.6–6.8. Then it increased gradually up to 7.0 in the fourth week, where the gas production also reached the maximum value. The measured value obtained can be considered the most acceptable condition for maximum gas production because of the higher growth of methanogens. On the other hand, the pH value for vegetable waste was in a highly acidic range of 3.2–4.0 throughout the entire retention time. Bouallagui et al. (2004) reported that the pH value for vegetable waste (VW) was in the range of 3.5–5.0, with an average value of 4.2 throughout the digestion process. As vegetable waste is highly biodegradable, it leads to rapid acidification in the AD process and, hence, inhibits the rate of biogas production (Gulhane et al., 2017). From the present study, it can be concluded that additional adjustment techniques are necessary for vegetable waste to control the pH value within the desired range.

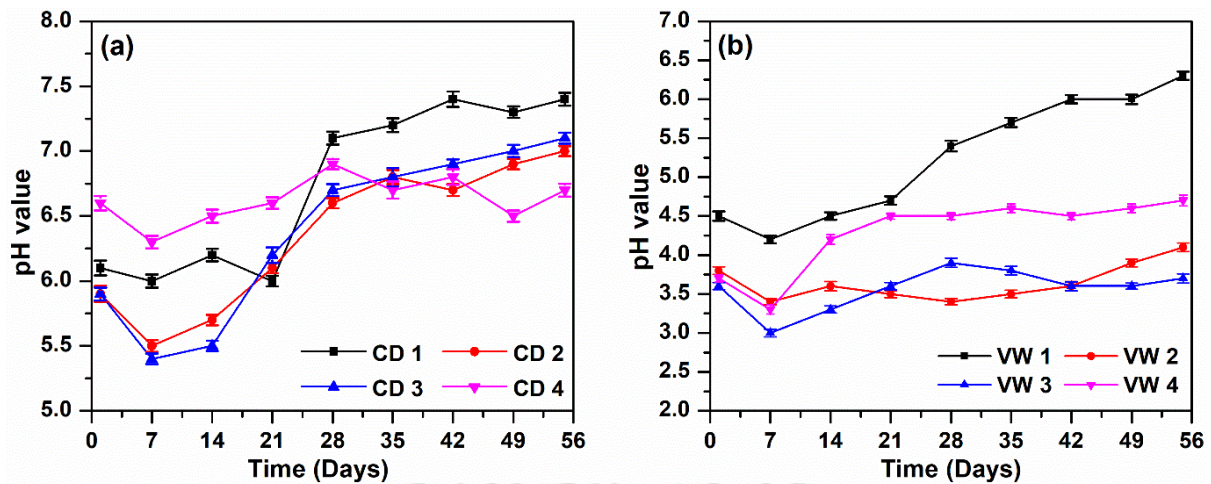


Fig. 4.2. Variation of pH in the digester for (a) cattle dung and (b) vegetable waste

4.1.3.2 Variation of temperature

Temperature is one of the main factors for the survival of microorganisms during the anaerobic digestion process (Hagos et al., 2017). The daily temperature variations of the digesters have been illustrated in Fig. 4.3. The comparative analysis of the effect of temperature on biogas production is presented in Fig. 4.5, with a thorough discussion provided in Section 4.1.4.1. It was observed that the decrease in the digester temperature with retention time resulted in the reduction of biogas production for both substrates. From the analysis, it can be interpreted that the effect of temperature is significant on gas production. In Fig. 4.3, the temperature fluctuations were recorded to be minimal for all the digesters throughout the retention time. The average temperatures for all the digesters were recorded to be in the range of 24–28 °C. The minimum average temperature was recorded to be 25.2 °C for CD4 with an F/W ratio of 80:20, while VW4 with the same F/W ratio showed a minimum average temperature of 24.6 °C. The temperature variations affect microbial growth, reducing the production rate due to the small temperature changes (Hagos et al., 2017). Controlling and maintaining the digester temperature at the optimum range can result in a higher gas production rate.

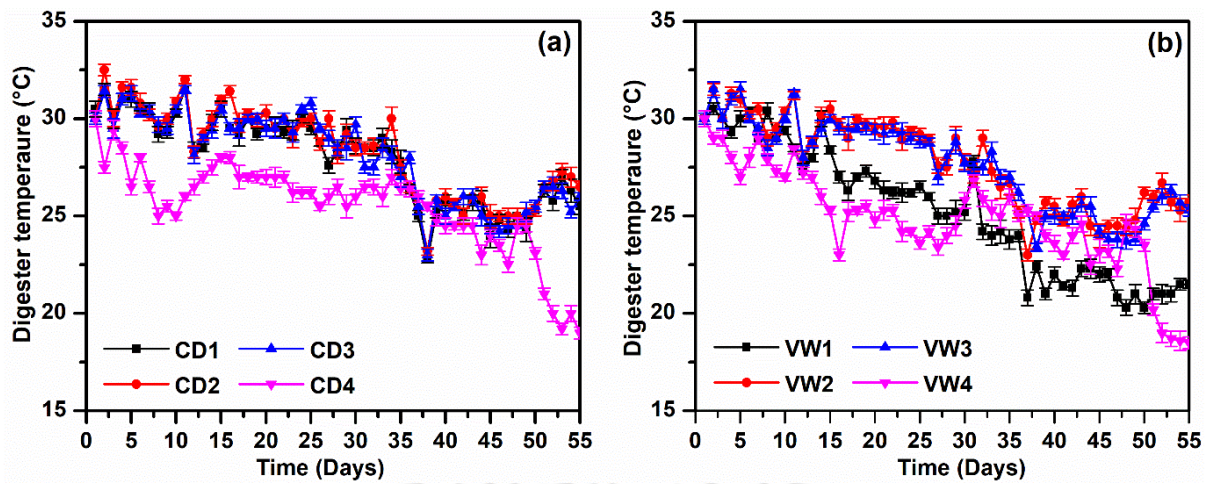


Fig. 4.3. Variation of digester temperature for (a) cattle dung and (b) vegetable waste

4.1.3.3 Biogas production

The daily biogas production plots under uncontrolled conditions from the AD of cattle dung and vegetable waste are presented in Figs. 4.4 (a) and (b), respectively. It was observed that, at the initial stage of anaerobic digestion, biogas production was found to be very low for CD1, CD2 and CD4, while for CD3, the biogas production started from the first week. Okonkwo et al. (2018) explained that the lower production of biogas at the early stage is due to the active nature of acid-forming bacteria. They also clarified that biogas production begins only after all oxygen present in the digester has been used up by aerobic bacteria at the early stage of the digestion process (Okonkwo et al., 2018). For digester CD1, biogas production was zero in the first week, which started to increase from the second week onwards, attained a maximum value of 312 ± 4.5 ml in the fourth week and then decreased from the fifth week onwards. For digester CD2, the trend was similar to CD1, with a maximum value of 447 ± 5.7 ml. The highest daily biogas production was recorded for CD3, with a peak value of 503 ± 4.9 ml at the beginning of the fourth week (23rd day). Many researchers reported that the maximum biogas production depends on the TS content in the slurry. In CD3, TS content was found to be $9.16 \pm 0.05\%$, which falls in the range of optimal value (7–9%) as reported by Salam et al.(2015). The trend of daily biogas production was slightly different for CD4. Here, the biogas

production gradually started increasing from the third week and fourth week onwards; it showed a zigzag pattern with a minimum and maximum value of 54 ± 3.3 and 260 ± 6.0 ml, respectively. Due to the high TS content ($12.57\pm 0.07\%$) in the CD4 and low TS content ($3.09\pm 0.02\%$) in CD1, unstable bacteria growth might occur. Further, at very high VS, hydrolysis/acidogenesis bacteria are more active than methanogenesis bacteria, which increases fatty acids (Kwietniewska and Tys, 2014); thereby, the biogas production may decrease. Unlike cattle dung, vegetable waste digesters followed a completely different pattern. VW1 digester resulted in very low biogas production, with nearly zero values at frequent intervals. However, digester VW2 showed a significant increase in biogas production with a maximum value of 103 ± 3.7 ml (fourth week) compared to 19 ± 2.1 ml of maximum production for VW1. In the case of VW3, the biogas production started from the first week onwards with the highest daily biogas production compared to the other three VW digesters. The maximum value recorded for VW3 was 159 ± 4.3 ml during the second week. The plot of digester VW4 showed a sudden increase in biogas production from the third day of the first week, and a maximum peak value of 218 ± 3.2 ml was achieved in the second week. However, there was a sudden decrease in biogas production, and it remained at almost zero value in the third and fourth weeks, and short peaks were observed in the remaining periods.

The cumulative biogas production of all the digesters for the entire digestion period are depicted in Figs. 4. 4 (c) and (d). The trends of cumulative biogas production of CD looked alike for all F/W ratios, whereas, for VW digesters, different cumulative trends were noticed at different F/W ratios. The maximum cumulative biogas production for 55 days was recorded to be 11.25 litres (L) for CD3, having an F/W ratio of 60:40, which was 153%, 55% and 101% more compared to CD1 (4.43 litres), CD2 (7.26 litres) and CD4 (5.60 litres). The cumulative biogas production obtained from the anaerobic digestion of vegetable waste was very low compared to cattle dung. At an F/W ratio of 60:40, the maximum cumulative biogas production

was found to be 4.08 litres for VW3. Other digesters, i.e. VW1, VW2 and VW4, produced 94% (0.24 litres), 39% (2.49 litres), and 35% (2.67 litres) lower cumulative biogas, respectively, compared to VW3. It can be inferred that biogas production highly depends on the TS and VS content of the substrates. Digesters of CD having 50% more TS than VW digesters are attributed to have approximately more biogas potential. Plazzotta et al. (2017) reported that low TS and high VS fractions promote rapid hydrolysis for vegetable waste during the AD process, which results in the accumulation of rapid acidification and prevents the potential for biogas production. Both CD and VW substrates with the same F/W ratio of 60:40 having TS of 9.16 ± 0.05 and $4.56 \pm 0.08\%$ were found to be the best from this investigation, which has been carried forward temperature-controlled experiments in the following sections.

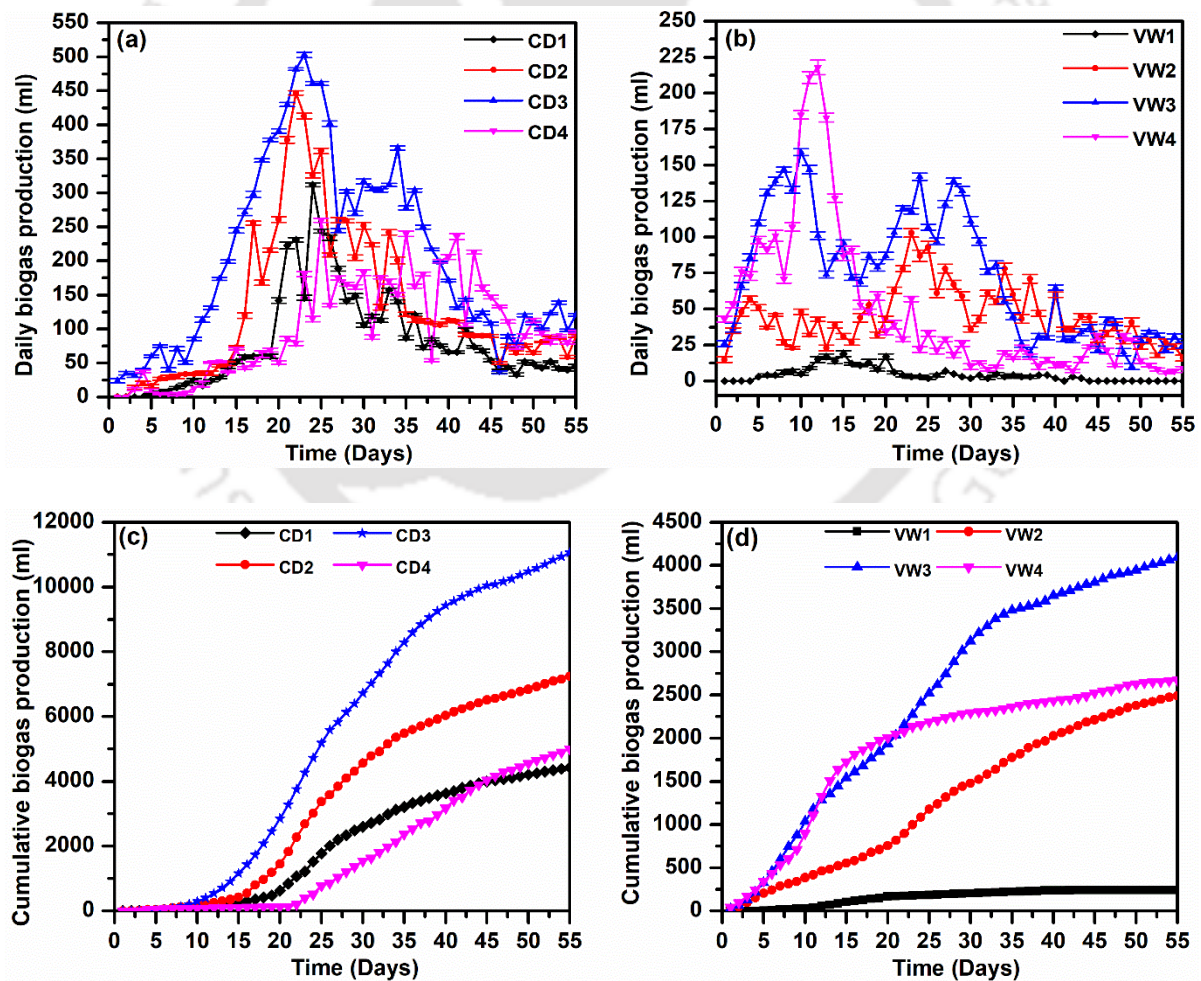


Fig. 4.4. Biogas generation under uncontrolled conditions (a) daily production of CD digesters, (b) daily production of VW digesters, (c) cumulative production of CD digesters and (d) cumulative production of VW digesters

4.1.4 Experimental analysis of controlled digesters

4.1.4.1 Biogas production

To understand the effect of temperature on biogas production rate, experiments were carried out under mesophilic ($35\pm 2^\circ\text{C}$) and thermophilic ($55\pm 2^\circ\text{C}$) conditions considering an F/W ratio of 60:40, and the comparative results were analysed. The daily variation in biogas generation at different conditions for both CD and VW is illustrated in Figs. 4. 5 (a) and (b). Daily biogas production trends for both CD and VW at uncontrolled and mesophilic conditions were observed to be alike. The daily biogas production was recorded to be higher at the mesophilic condition with a peak value of 523 ± 5.7 ml on the 24th day for CD3_35 °C and 189 ± 4.8 ml on the 12th day for VW3_35 °C compared to the respective digesters at the uncontrolled condition for both feedstocks. On the other hand, the biogas generation trends in thermophilic conditions were completely different from those in digesters in uncontrolled and mesophilic conditions. The biogas generation rate was boosted up at the early stage of the AD process for both substrates, yet a rapid decline was noted from 3rd week onwards. The maximum daily biogas production of 654 ± 5.4 ml was achieved on the 13th day of retention time for CD3_55°C, whereas 234 ± 6.3 ml was attained on the 16th day for VW3_55°C at the thermophilic condition. Various researchers reported that the biogas production rate at thermophilic conditions is higher due to higher metabolic rates, specific growth rates, and higher destruction rates of pathogens than in the mesophilic range (Kim et al., 2006; C. Zhang et al., 2014). The cumulative biogas yields for controlled and uncontrolled digesters are compared and depicted in Figs. 4. 5 (c) and (d). For mesophilic and thermophilic conditions, cumulative biogas yields were 21.72 and 22.75% higher for CD and 15.20 and 23.25% higher for VW compared to uncontrolled digesters. Bouallagui et al.(2004) mentioned a higher percentage of biogas yields in thermophilic digesters than in psychrophilic and mesophilic digesters. Chae et al.(2008) reported that the biogas yield from swine manure at 30 and 35 °C

is 3.0 and 17.4% higher than at 25 °C. The authors also noted that the methane content is higher at a higher temperature. The retention time was even lower at thermophilic conditions due to thermophiles' high catalytic activity (Kothari et al., 2014). From the present investigation, it can be concluded that the biogas production rate at thermophilic conditions is faster than in mesophilic and ambient conditions at similar concentrations and volumes. However, more energy is needed to maintain the thermophilic temperature.

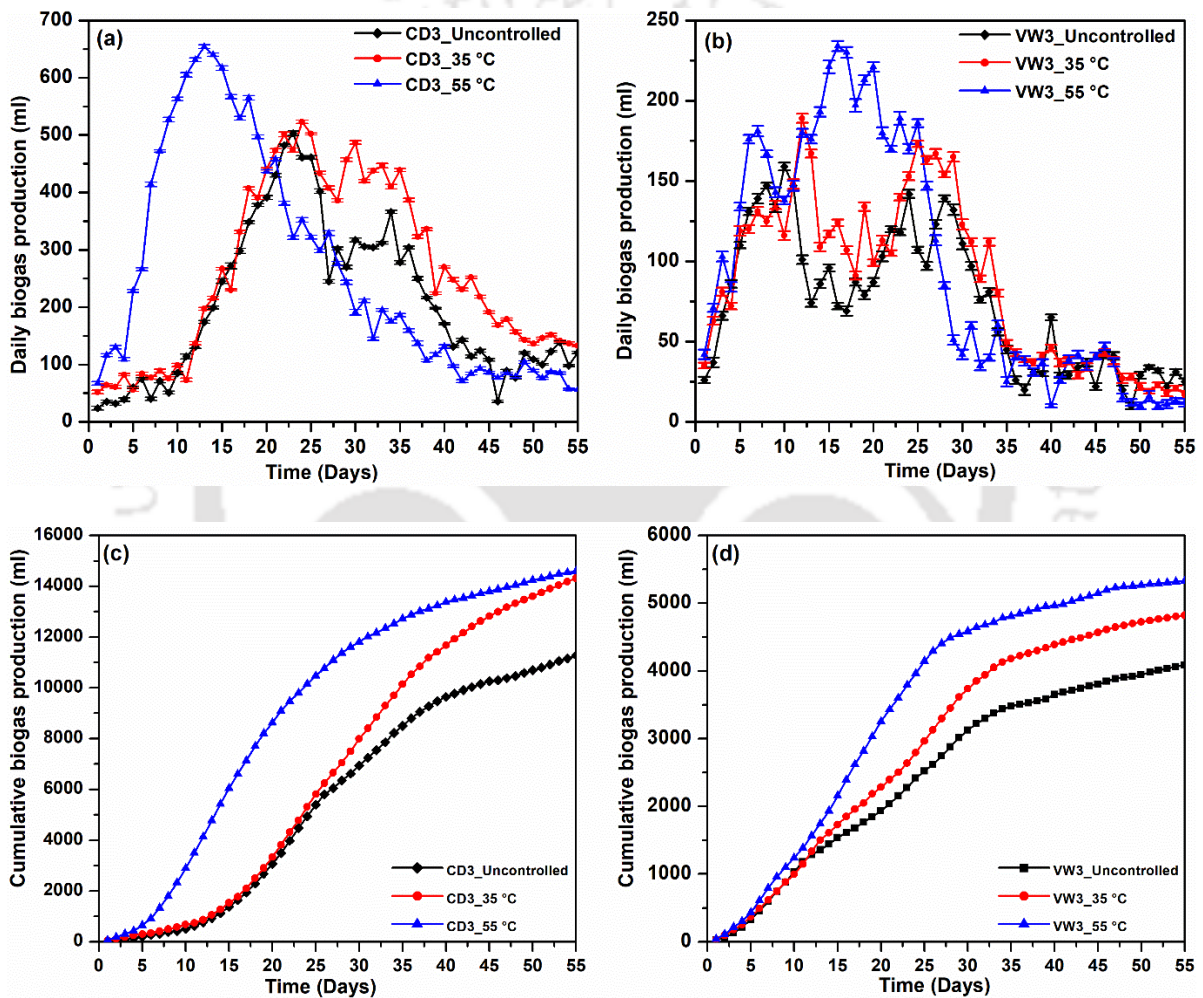


Fig. 4. 5. Biogas generation under temperature-controlled conditions (a) daily production of CD digesters, (b) daily production of VW digesters, (c) cumulative production of CD digesters and (d) cumulative production of VW digesters.

4.1.4.2 Kinetic study

A kinetic study for the cumulative biogas produced from all the digesters was carried out using the modified Gompertz model equation according to Zhai et al.(2015) to identify the ideal F/W ratio for maximum biogas yield potential on the anaerobic digestion of CD and VW at uncontrolled temperature. The maximum biogas production potential was detected in the digesters CD3 (11.58 L) and VW3 (4.26 L), with an F/W ratio of 60:40 for CD and VW, respectively. Moreover, lower lag phase duration was also observed for the digesters CD3 (12.08 d) of cattle dung and VW3 (2.60 d) of vegetable waste at uncontrolled temperature. Therefore, CD3 and VW3 were found to be the optimum F/W (60:40) ratio in the kinetic study as well. Like the experimental study, CD3 and VW3 were also studied using the kinetic model under mesophilic and thermophilic conditions to get a proper insight into the effect of temperature in the AD process. The biogas yield potential was found to be highest at the thermophilic condition for both the digesters CD3_55°C (15.64 L) and VW3_55°C (5.33 L). Moreover, the R² value was found to be greater than 0.98 for all the digesters, demonstrating that biogas production was well simulated. The results of the kinetic studies are presented in Table 4.3.

Table 4.3 Kinetic values of anaerobic digestion of CD and VW

Digesters	Y_p (L)	Y_R (mL/day)	λ (day)	Y_C (L)	R ²
Uncontrolled temperature					
CD 1	4.44	186.53	16.10	4.43	0.99
CD 2	7.16	302.92	14.61	7.26	0.99
CD 3	11.58	406.07	12.08	11.25	0.99
CD 4	7.07	175.63	18.04	5.60	0.99
VW 1	0.24	12.19	7.10	0.24	0.99
VW 2	3.01	62.43	6.44	2.49	0.99
VW 3	4.26	121.34	2.60	4.09	0.99
VW 4	2.51	134.65	2.81	2.67	0.98

Controlled temperature					
CD3_35±2 °C	14.32	471.00	13.01	14.40	1.00
CD3_55 ±2 °C	15.64	575.10	5.02	14.60	0.98
VW3_35±2 °C	5.06	151.80	4.60	4.82	0.99
VW3_55 ±2 °C	5.33	217.32	4.10	5.33	0.99

4.1.5 Summary of anaerobic digestion of CD and VW

The investigation enabled to establish relationships among the parameters affecting biogas yield. Highly acidic VW digesters prevented the biogas production rate. The optimum TS and VS contents observed for a 60:40 F/W ratio favoured the AD process and delivered maximum cumulative biogas for both CD and VW. Compared to uncontrolled and mesophilic conditions, the highest biogas production potential was observed for a 60:40 F/W ratio under thermophilic conditions. This is also confirmed by the kinetic study carried out using the modified Gompertz model. Hence, the study provided some fundamental understandings that could be implemented to install large-scale pilot plants in various rural areas.

4.2 MONO-DIGESTION OF CATTLE DUNG

This section illustrates the comparative analysis of pH value, temperature, and biogas production variations in lab-scale experiments.

4.2.1 Variations of pH and temperature

The pH value of slurry plays a vital role in affecting microorganisms' growth rate at various stages of the AD process. The comparison of pH values for uncontrolled (digesters D1, D2, D3) and controlled (digesters R1, R2, R3) conditions measured weekly are depicted in Fig. 4.6 (a). Variation of pH values with retention time was observed for each digester. The pH value was in the acidic range (5.5–6.5) at the initial stages of the AD process (up to the third week). However, it gradually increases and reaches more than 7.0 after the seventh week onwards. Several studies reported that methanogenesis's most appropriate pH range is between

6.5 and 8.2, and its best pH value is 7.0 (Kothari et al., 2014; Mao et al., 2015). Furthermore, the most favourable pH value for hydrolysis and acidogenic lies between 5.5 and 6.5 (Hagos et al., 2017; Kothari et al., 2014). From the fourth week onwards, pH values for Set-I and Set-II were found to be in the desired range (6.8–7.2), which were consistent and comparable with the outcomes reported by Basumatary et al. (2021) and Hagos et al. (2017).

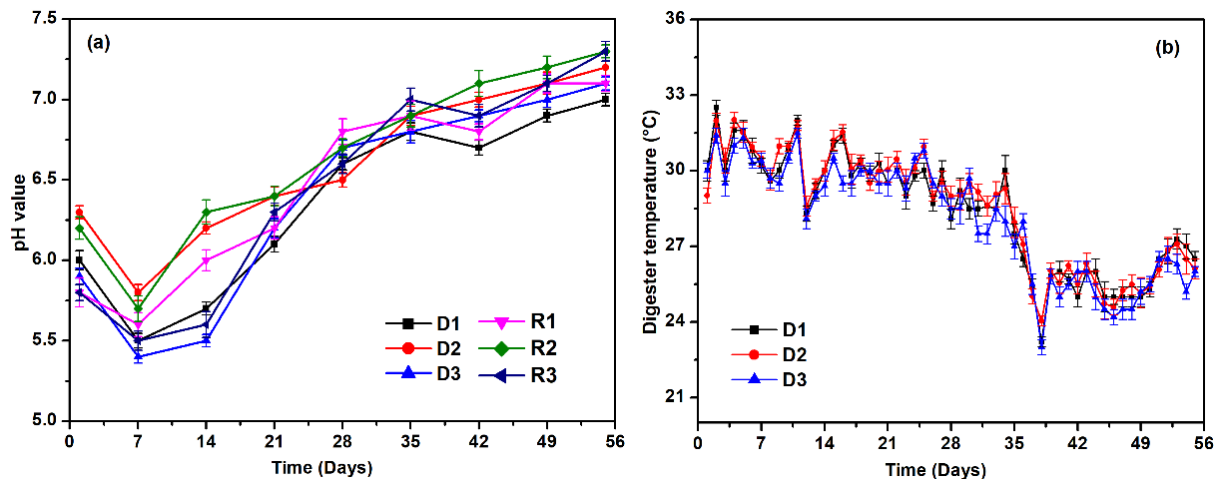


Fig. 4.6 Variation of (a) pH value and (b) temperature in the lab-scale experiment

Besides the pH value, the temperature is an essential operational parameter for developing microorganisms in the AD process. The daily variations of the uncontrolled digester temperature have been depicted in Fig. 4.6 (b). As the digesters R1, R2 and R3 were maintained at a controlled temperature ($35 \pm 2^\circ\text{C}$), their temperature plots were not presented in Fig. 4.6. For uncontrolled digesters, it was seen that the temperature decreased with retention time. The daily average temperature was recorded between 27°C and 32°C up to the fifth week and 23.0 – 27.5°C afterwards. The reduction in temperature was due to the change in weather conditions. However, fluctuations in temperature were detected at a minimum for all uncontrolled digesters throughout the digestion period. The biogas production rate declined from the third week onwards for uncontrolled digesters and the fourth week onwards for controlled digesters. The biogas yield for the controlled digester was found to be higher than the uncontrolled digesters.

It may be inferred that the pH and temperature change greatly influence the AD process's biological activity. Henceforth, biogas production through the AD process can be enhanced significantly by maintaining a digester's pH value and temperature at an optimum range.

4.2.2 Biogas production from lab-scale experiment

The graphs of daily and cumulative biogas production under uncontrolled (Set-I) and controlled (Set-II) conditions are presented in Fig. 4.7. A lower quantity of biogas production was obtained at the early stage of anaerobic digestion for each digester. It might be because the methanogens population were initially low and took some time to grow. The biogas generation trends of each uncontrolled digester were observed to be alike (Fig. 4.7 (a)). For digester D1, a low amount of biogas production was identified up to the 2nd week; however, it increased from the 2nd week afterwards. The highest peak value of 447 ± 7.30 ml was found for D1 on the 22nd day and started to decrease from the 5th week afterwards. The declination rate of biogas production was also found to be faster than digesters D2 and D3. In the case of D2, the biogas generation commenced from the 1st day of the anaerobic digestion with the highest value of 513 ± 7.10 ml at the end of the 3rd week (20th day). Whereas for digester D3, the peak value of 503 ± 8.00 ml was attained on the 23rd day of the retention period. Moreover, temperature variations also affect biogas production. The evidence of fluctuations in biogas generation was detected during the hydraulic retention time of 25–35 as the fluctuations in temperature occurred. The maximum specific cumulative biogas (SCB) generation was 247.75 ml/gVS for digester D2, which was 28.41% and 26.12% more than D1 (176.64 ml/gVS) and D3 (182.29 ml/gVS), respectively, for a retention period of 55 days (Fig.4.7(b)). The SCB generation of digester D2 was found to be consistent with the result reported by (KeChrist et al., 2017). The TS contents in reactors D2 ($7.70 \pm 0.02\%$) and D3 ($9.10 \pm 0.05\%$) were within the range of 7–9%, which is considered to be ideal and is comparable to the findings of Salam et al.(2015).

Consequently, it can be said that the feedstocks' TS and VS contents significantly impact the biogas yield.

The AD process was also performed under mesophilic conditions to understand the behaviour of biogas generation potential with the variation of the S/W ratio under the controlled environment. The daily and specific cumulative biogas generation for the S/W mixing ratio of 1:1.5, 1:1 and 1.5:1 at mesophilic temperature ($35\pm 2^\circ\text{C}$) are illustrated in Figs. 4.7 (c) and (d). A similar daily biogas yield (DBY) trend was observed for the mesophilic digesters R2 and R3. The biogas generation was found to be commenced immediately from the 1st day of investigations for all controlled digesters. The DBY was identified to be higher for the controlled digesters R1, R2 and R3, with a maximum value of 487 ± 6.50 ml on the 29th day, 571 ± 8.61 ml on the 27th day and 567 ± 5.72 ml on the 24th day of retention time compared to uncontrolled digesters D1, D2 and D3 respectively. Several studies reported that the faster growth of microorganisms at mesophilic conditions accelerates biogas production than in psychrophilic conditions (Hagos et al., 2017). Kothari et al. (2014) conveyed that more than 22% higher amounts of biogas could be achieved at the mesophilic condition for sewage sludge than in the psychrophilic state. In the present investigation, specific cumulative biogas (SCB) yields from the mesophilic digesters R1, R2 and R3 were 243.37 ml/gVS, 311.14 ml/gVS and 250.98 ml/gVS. The SCB yields of the digesters R1, R2 and R3 were identified to be 27.42%, 20.70% and 27.37% higher than the yields of digesters D1, D2 and D3, respectively, for a 55-day digestion period. Further, SCB yield in the digester R2 was achieved by 19.34% and 21.78% higher than R3 and R1, respectively. However, a higher and better rate of biogas yield was found for the S/W ratio of 1:1 under controlled and uncontrolled conditions.

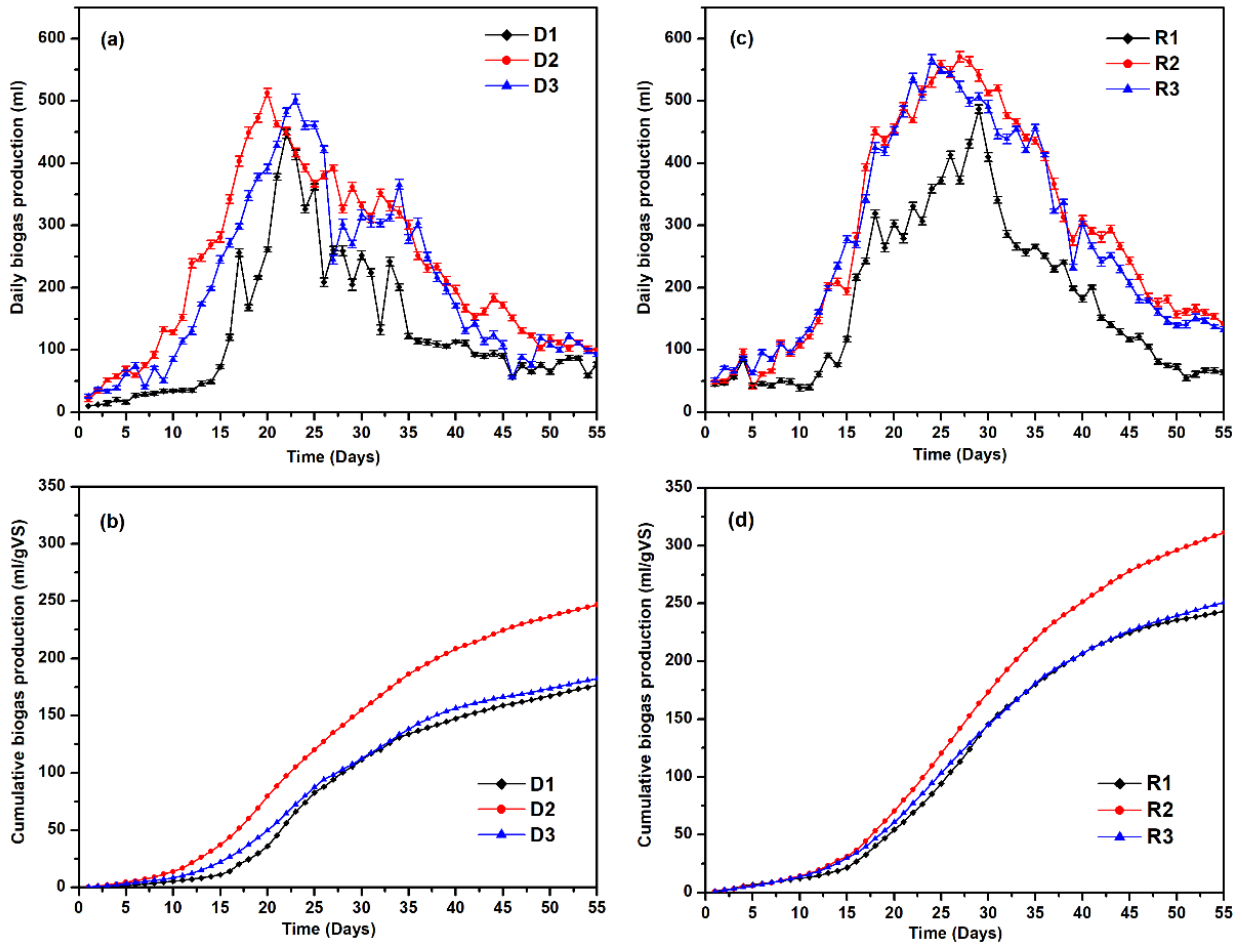


Fig. 4.7. (a) Daily (b) cumulative biogas production at uncontrolled conditions, and (c) daily (d) cumulative biogas production at controlled conditions.

4.2.3 Kinetic analysis

Table 4.4 illustrates the kinetic analysis performed using the modified Gompertz model. The highest biogas generation potential (Y_P) value was achieved in the digesters D2 (258.30 ml/gVS) and R2 (337.42 ml/gVS). The controlled digesters R1, R2 and R3 were found to be 31.38%, 23.45% and 30.46% higher specific Y_P than respective uncontrolled digesters D1, D2 and D3. Further, the Y_R value was also noticed to be higher for R2 (10.32 ml/gVS-day) in controlled digesters and D2 (8.26 ml/gVS-day) in the case of uncontrolled digesters. The correlation coefficient (R^2) value was found to be approximately 0.99 for the lab-scale experiment, representing that the predicted results perfectly fit experimental values.

Table 4.4 Kinetic analysis for mono-digestion of CD

Digesters	Y_P (ml/gVS)	Y_R (ml/gVS-day)	λ (day)	Y_C (ml/gVS)	R^2
Uncontrolled batch-type digesters					
D1	177.95	7.33	14.46	176.64	0.99
D2	258.30	8.26	10.66	246.75	0.99
D3	188.55	6.58	12.20	182.29	0.99
Controlled batch-type digesters					
R1	259.32	9.03	14.05	243.37	0.99
R2	337.42	10.32	10.19	311.14	1.00
R3	271.16	8.33	12.49	250.98	1.00

4.2.4 Summary of mono-digestion of cattle dung

The present investigation assisted in identifying the best optimum parameters for maximum biogas production. The highest biogas generation potential of CD was achieved for the 1:1 S/W ratio conducted under controlled ($35\pm 2^\circ\text{C}$) and uncontrolled environments in the batch-type lab-scale experiments. The SCB yields of 1:1 S/W ratio were 247.75 ml/gVS and 311.14 ml/gVS for uncontrolled and controlled digesters, respectively, for HRT of 55 days. The experimental results were confirmed with kinetic analysis performed using the modified Gompertz kinetic model.

4.3 ANAEROBIC DIGESTION OF COOKED KITCHEN WASTE

4.3.1 Characterisation of cooked kitchen waste

The initial characteristics of CKW are presented in Table 4.5. The food waste was mainly cooked rice, pumpkins, roti, dal, shambar, and potatoes. Based on the proximate analysis, MC, TS, and the VS/TS ratio were $82.79\pm 0.23\%$, $17.21\pm 0.41\%$, and $93.16\pm 0.79\%$, respectively. These compositions depend on the type of food waste collection. A high VS/TS

ratio indicates a high organic natured biomass and is considered ideal for generating biogas and methane. The C/N ratio of KW was 15.86. The optimal C/N range suggested in the literature was 14–25 for anaerobic biodegradation (Varsha et al., 2022), representing the nutrients required for microbial growth. The pH value was detected to be 4.83. The low pH value is due to the content of spices in Indian food. The pH value was found to be lower than the optimum value. It confirms that pH value adjustment is required for micro-organisms' growth. However, in this study, adjustment of pH value was not conducted.

Table 4.5 Initial characterisation of cooked kitchen waste

Parameters	Cooked kitchen waste
Moisture content (%)	82.79 ±0.23
Total Solid (%)	17.21 ±0.41
Volatile Solid (%TS)	93.16 ±0.79
Carbon (%)	51.23
Hydrogen (%)	6.22
Nitrogen (%)	3.23
Sulphur (%)	0.38
Oxygen	38.94
C/N ratio	15.86
pH value	4.83±0.07

4.3.2 Daily and cumulative methane yield

The biochemical methane potential (BMP) from cooked kitchen waste was performed at substrate water mixing (S/W) of 80:20, 60:40, 50:50, 40:60 and 20:80 % on a wet mass basis. The BMP tests were conducted at ambient temperature. The daily and cumulative methane yields are depicted in Fig. 4.8. The methane generation commenced on the first day for all the mixing ratios but in varying proportions. Higher methane yield was achieved between the 2nd

and 3rd week of the BMP test and decreased drastically from the 4th week onward. Lesser methane generation was detected for S/W 20:80 (CKW 20:W80), whereas higher was achieved for S/W 50:50 (CKW50: KW50) with a peak value of 142 ml CH₄ on the 17th day of hydraulic retention time. The digester S/W 50:50 achieved 69.55%, 16.03%, 12.46% and 27.11% higher amount of cumulative methane yield (CMY) than S/W 20:80, S/W 40:60, S/W 60:40 and S/W 80:20, respectively. Comparing all the mixing ratios, the digester with S/W50:50 was the best mixing ratio among the other digesters.

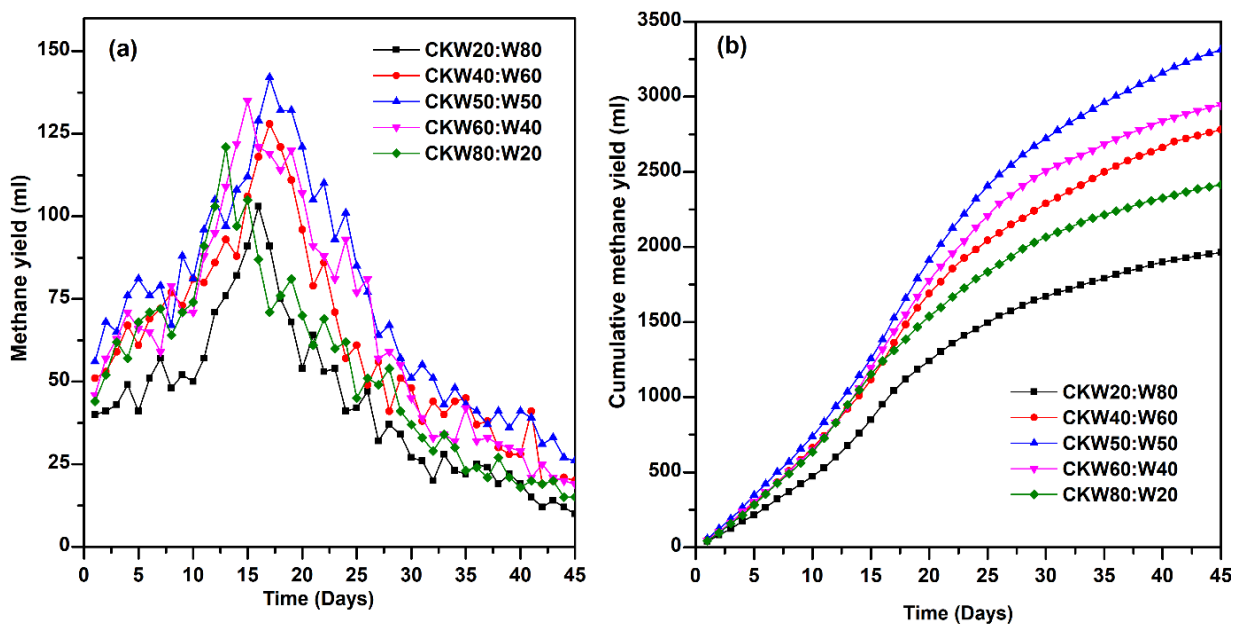


Fig. 4.8 Methane yield (a) daily and (b) cumulative for mono-digestion of CKW

4.3.3 Kinetic analysis

A kinetic study for the cumulative methane yield was carried out using the modified Gompertz model equation according to Zhai et al.(2015) to identify the ideal S/W ratio for maximum CH₄ yield potential. The maximum biogas production potential was detected in the digesters CKW50:W50 (3541.5 mL), whereas the lowest was for CKW20:W80 (1971.90 mL). Moreover, the digesters CKW50:W50 (3.2 days) had a lower lag phase period. Therefore, CKW50:W50 was also found to be the optimum S/W ratio in the kinetic study. The R² value

was found to be greater than 0.98 for all the digesters, demonstrating that biogas production was well simulated. The results of the kinetic studies are presented in Table 4.6.

Table 4.6 Kinetic parameters for mono-digestion of CKW

Digesters	Y_P (mL)	Y_R (mL/day)	λ (day)	Y_C (mL)	R^2
CKW20:W80	1971.90	75.20	3.6	1962.00	0.99
CKW40:W60	2897.20	100.80	3.5	2782.00	0.99
CKW50:W50	3541.50	119.00	3.2	3313.00	0.99
CKW60:W20	3066.40	112.80	4.2	2946.00	0.99
CKW80:W20	2476.70	91.90	3.4	2415.00	0.99

4.3.4 Summary of anaerobic digestion of cooked kitchen waste

This study investigated the effect of the substrate-water ratio on the anaerobic digestion of cooked kitchen waste. A favourable substrate mixing ratio is required for more methane generation. The substrate ratio KW50:W50 achieved the highest methane generation potential in the mono-digestion of cooked kitchen waste for a retention time of 45 days. Further, the lag phase period was found to be lower compared to other mixing ratios. The study provided some fundamental understandings that could be implemented to install large-scale pilot plants in various rural areas.

4.4 ANAEROBIC CO-DIGESTION OF CATTLE DUNG AND VEGETABLE WASTE

The biochemical methane potential (BMP) from the anaerobic co-digestions of cattle dung and vegetable waste carried out at different co-substrate mixing ratios (SMR) under ambient and mesophilic temperatures are presented in this section. The BMP tests were conducted for different SMRs to optimise the ideal SMR ratio. The effect of biochar addition

on methane yield, temperature, pH value and degradation of volatile solids during the anaerobic digestion were recorded and compared.

4.4.1 Characterisation of co-digestion of cattle dung and vegetable waste

The MC, TS, VS, and initial pH values of CD and VW at various SMRs, viz. 70:30, 60:40, 50:50, 40:60, and 30:70, are presented in Table 4.7. Examining these parameters is crucial for assessing the suitability of a substrate for AD since they significantly influence the stability of the AD process and, consequently, CH₄ generation. In the current study, a significant quantity of MC was detected in the substrates and the slurry of CD and VW. The appropriate range of MC assists the anaerobic bacteria in the hydrolysis and acidogenesis phases of AD, whereas severely low moisture levels impede the AD process due to acid build-up (Odejebi et al., 2021; Osei-Owusu et al., 2023). In contrast, a high MC in the substrate promotes microbial activity in AD (Lay et al., 1997). Several research studies have described the significant role of TS content in influencing the rates of hydrolysis and methanogenesis, consequently impacting methane generation in the AcoD of organic wastes (Indren et al., 2020; Johnravindar et al., 2022). Abbassi-Guendouz et al. (2012) observed the highest methane generation rate at a TS concentration of 10%, which decreased if the TS value increased from 10% to 30% (Abbassi-Guendouz et al., 2012). High TS contents can impede mass transfer between microorganisms and substrate and lead to a decline in methane yield (Johnravindar et al., 2022). In the present study, TS values of the slurries at different percentages of CD and VW mixing ratio were found in the range of 5.79–8.23%, whereas its value was 15.92% and 8.37% for 100% CD and 100% VW, respectively. The TS value of CD was detected to be aligned with the literature reported by Wang et al. (2022) and Salam et al. (2015). On the other hand, the TS and VS (% TS) content of VW were $8.37 \pm 0.02\%$ and $94.25 \pm 0.14\%$, which were detected to be consistent with the result reported by (Bouallagui et al., 2009). The high values of VS (%TS) confirm that the CD and VW are easily degradable organic materials necessary

for the AD process. Table 4.7 demonstrates that the slurry's VS (%TS) content increased from 74.70% to 83.32% as the VW percentage increased. The pH level of the slurry was perceived in the acidic range (4.66–5.98). The C/N ratios of CD and VW were 25.82 ± 1.10 and 18.75 ± 0.29 , whereas their values were found in the range of 19–23 for different mixing ratios. The C/N is an essential factor as it measures the nutrient level of microorganisms.

Table 4.7 Initial characterisation of CD, VW and their slurries

Parameters	CD	VW	Substrate mixing ratio				
			SMR	SMR	SMR	SMR	SMR
			70:30	60:40	50:50	40:60	30:70
MC (%)	84.08 ± 0.26	91.65 ± 0.02	91.77 ± 0.09	92.14 ± 0.07	92.86 ± 0.04	93.31 ± 0.10	94.21 ± 0.15
TS (%)	15.92 ± 0.17	8.37 ± 0.02	8.23 ± 0.09	7.86 ± 0.06	7.14 ± 0.04	6.69 ± 0.09	5.79 ± 0.15
VS (%TS)	83.50 ± 0.10	94.25 ± 0.14	74.70 ± 0.17	75.93 ± 0.11	77.00 ± 0.03	81.06 ± 0.05	83.32 ± 0.06
C/N ratio	25.82 ± 1.10	18.75 ± 0.29	22.81 ± 0.13	21.92 ± 1.02	21.26 ± 0.54	20.52 ± 1.06	19.14 ± 0.25
pH	6.39 ± 0.09	4.31 ± 0.05	5.98 ± 0.09	5.76 ± 0.10	5.21 ± 0.08	5.03 ± 0.06	4.66 ± 0.05

4.4.2 Characterisation of water hyacinth biochar

The physicochemical properties and structures of BC effect the AcoD process (Ovi et al., 2022). Table 4.8 illustrates the characterisation of BC. The pH level of WHBC was in the alkaline range (10.08 ± 0.08) due to the existence of AC and volatilisation of acidic functional groups (Kumar et al., 2021). The AAE (alkali and alkaline earth) metals (K, Ca, Mg and Na)

enhance the alkalinity of co-digestion (Basumatary et al., 2024). Further, some elements like C, N and P assist the growth and metabolism of microbes (Liu et al., 2023). The H/C and O/C values were obtained to be 0.04 ± 0.00 and 1.28 ± 0.03 , respectively. These low values of H/C and O/C signify the high hydrophobicity and adsorption capacity of organic pollutants like ammonium (Johnravindar et al., 2022; Xie et al., 2022). The surface textural properties of BC were also analysed. The S_{BET} , V_p and d_p were detected as 14.10 ± 0.11 m^2g^{-1} , 0.05 ± 0.01 cm^3g^{-1} , and 11.94 ± 0.05 nm, respectively. The porosity nature of biochar offers a surface area for the adsorption of carbon dioxide in the AD process (Aramrueang et al., 2022).

Table 4.8 Initial characterisation of water hyacinth biochar

Analysis	Parameter	Unit	Value
Proximate analysis			
	MC	%	3.82 ± 0.43
	AC	%	19.59 ± 0.27
	VM	%	13.64 ± 0.14
	FC	%	62.96 ± 0.05
CHNSO study			
	C	wt %	41.35 ± 0.11
	H	wt %	1.67 ± 0.08
	N	wt %	3.34 ± 0.06
	S	wt %	0.52 ± 0.03
	O	wt%	53.11 ± 0.10
	H/C	–	0.04 ± 0.00
	O/C	–	1.28 ± 0.03
AAE metals			
	K	wt %	9.47 ± 0.02
	Ca	wt %	5.73 ± 0.09
	Na	wt %	1.41 ± 0.07
	Mg	wt %	1.11 ± 0.08

Others elements			
P	wt %		3.12±0.09
Cl	wt %		5.23±0.10
Physisorption isotherm			
S _{BET}	m ² /g		14.10±0.11
V _p	cm ³ /g		0.05±0.01
d _p	nm		11.94±0.05
Others analysis			
pH	–		10.07±0.06
EC	dS/m		33.28±0.85

Further, the SEM image of WHBC represented in Fig. 4.9 (a) signifies biochar's porous structure that provides areas for the colonisation of anaerobic bacteria. The SEM image of BC shows a knaggy surface with scattered micropores and mesopores. Micropores are observed between the particles. The wavelength of FTIR spectra detected (Fig. 4.9 (b)) are presented in Table 4.9. These functional groups provide direct interspecies electron transfer (DIET) and raise the progress of microbes in the AcoD system (Shen et al., 2020). The WHBC displays the functional groups, including C–H vibrational groups (aromatic and aliphatic) at 874 and 562 cm⁻¹, stretching vibrations of saturated and unsaturated alcohols C–O at 1037cm⁻¹ and aromatic C=C stretching at 1409 cm⁻¹. Additionally, a low peak at 3610 cm⁻¹ in FTIR spectra represents O–H stretching. Besides, XRD spectra (Fig. 4.9 (c)) detected at various 2-theta (2θ) values confirmed the presence of CaCO₃ (29.36°, calcite), CaSO₄ (40.98°, 66.76°), KOH (30.86°, 50.37°) and K₂SO₄ (58.76°) which provide buffering effect in AcoD process (Jiang et al., 2020). The evidence of the presence of these compounds was verified with JCPDS (Joint Committee on Powder Diffraction Standards) cards. The diffraction peaks in the obtained patterns matched well with JCPDS #74-1639 for CaSO₄, JCPDS #72-1937 for CaCO₃ (calcite), and JCPDS #78-0190) for KOH.

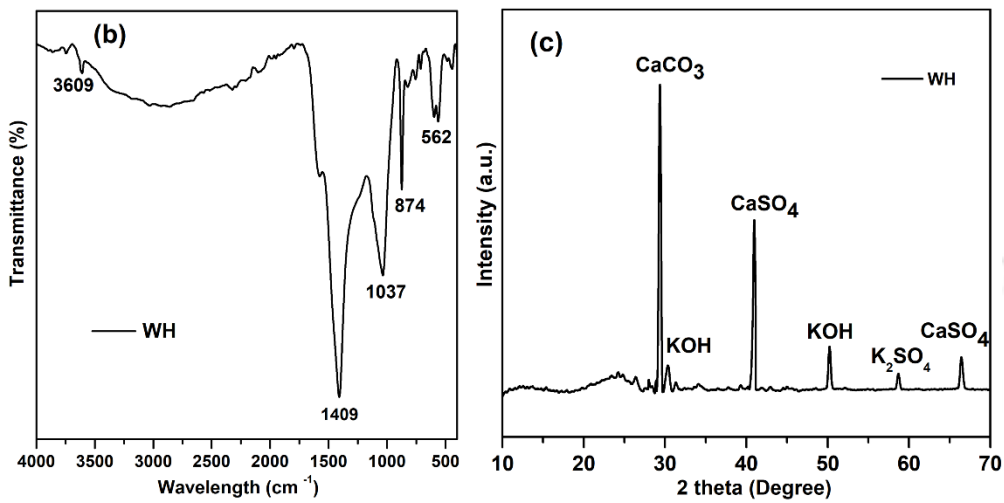
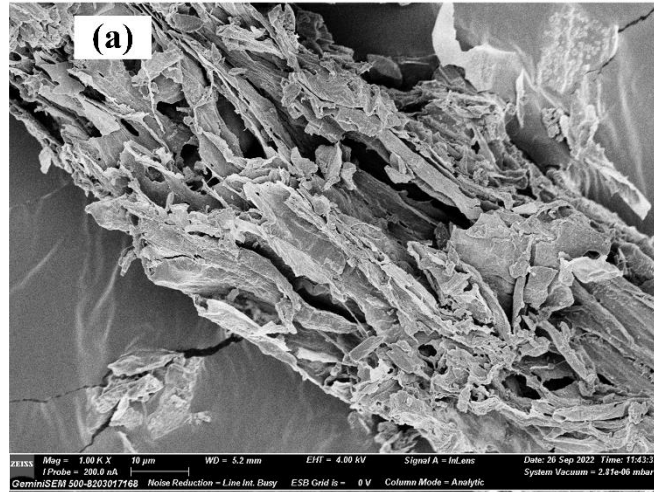


Fig. 4.9 (a) FESEM image (b) FTIR spectra (c) XRD spectra of biochar

Table 4.9 Visible wavelength and functional groups of the WH biochar

Wavelength from literature (cm ⁻¹)	Wavelength from this study (cm ⁻¹)	Bond	Functional group	Reference
3650–3600	3610	O–H	Alcohol, phenol	(NIU, 2023)
1700–1400	1409	C=C	Alkene, aromatic	(Shen et al., 2015)
		C=O	carbonyl	(Bordoloi et al., 2022)
1200–1000	1037	C–O	alcohol	(Domingues et al., 2017)
900–690	874	C–H	aromatic	(Shen et al., 2015)
	562	C–H	Aliphatic, aromatic	(Shen et al., 2015)

4.4.3 Result of anaerobic co-digestion of cattle dung and vegetable waste

4.4.3.1 Daily and cumulative methane yield

The daily and cumulative CH₄ yield for AcoD of CD and VW at various SMRs viz. 70:30, 60:40, 50:50, 40:60, and 30:70 without and with BC addition under ambient and mesophilic temperatures are presented in Fig. 4.10 and Fig. 4.11. From Fig. 4.10, it was evident that CH₄ production was initiated from the very first day for all SMRs. The CH₄ production rate was low at the initial phase of the AcoD process. This could be because the oxygen in the reactors restricts the growth of microbes and CH₄ yield at the initial phase of the AD operation (Deepanraj et al., 2017). Subsequently, a high rate of CH₄ yield was observed, likely due to the speedy growth of microbes. Afterwards peak period, CH₄ generation declined. The high peak methane generation was attained between the end of the 2nd and 4th weeks of the AcoD period.

Biochar can enhance biogas generation by improving the microbial environment in the digester. The BC-added digesters conducted at ambient and mesophilic temperatures achieved a higher DMY rate compared to the corresponding ambient digesters without BC. In comparison to the uncontrolled digesters UR1, UR2, UR3, UR4 and UR5 without BC, the BC-added digesters UD1, UD2, UD3, UD4, and UD5 resulted in higher DMY rate with peak values of 164 mL on the 21st day, 189 mL on the 22nd day, 153 mL on the 20th day, 137 mL on the 20th day, and 119 mL on the 17th day, respectively. Likewise, the maximum peak values of DMY for the mesophilic digesters MD1, UD2, MD3, MD4 and MD5 with added BC were obtained on the 20th (196 mL), 18th (209 mL), 17th (179 mL), 18th (161 mL) and 17th (149 mL) days of the retention time, respectively. The BC-added digesters with SMRs of 70:30, 60:40, 50:50, 40:60, and 30:70 was found to be 26.53, 30.04, 22.62, 20.95, 18.91 % higher volume of CMY than respective digesters without BC respectively, conducted at ambient temperature, for the HRT of 45 days. Likewise, the BC-added mesophilic digesters generated 36.42, 39.26, 33.47, 32.90 and 31.47% higher amounts of CMY than the uncontrolled digesters. This study confirms that the AD system can be improved by adding BC. This investigation also concludes that maintaining the reactor temperature at mesophilic conditions can increase CH₄ generation.

Previous studies revealed that BC provides the colonisation areas for anaerobic bacteria and serves as the adsorbent for substances, such as ammonia and limonene, which may hinder co-digestion (Wambugu et al., 2019). The alkaline nature of BC improves methane concentration via a reaction of carbon dioxide and H₂S with alkaline materials available in ash, consequently enhancing the biogas quality (Wang et al., 2017). Various elements and properties of BC, which influence the improvement of the CH₄ production rate, are demonstrated in Section 4.4.2. Additionally, BC is a thermal insulator that primarily benefits mesophilic environments, where steady temperatures are necessary for efficient methanogenesis. In this study, the highest CH₄ yield was achieved at SMR 60:40 for all groups of BMP experiments. The SMY obtained for CMR of 60:40 at HRT of 45 days were 158.98, 227.24 and 261.75 mLg⁻¹VS⁻¹ for groups I, II and III, respectively. The current investigation demonstrated that the SMR of 60:40 can be recommended as the most likely SMR for the co-digestion of CD and untreated VW.

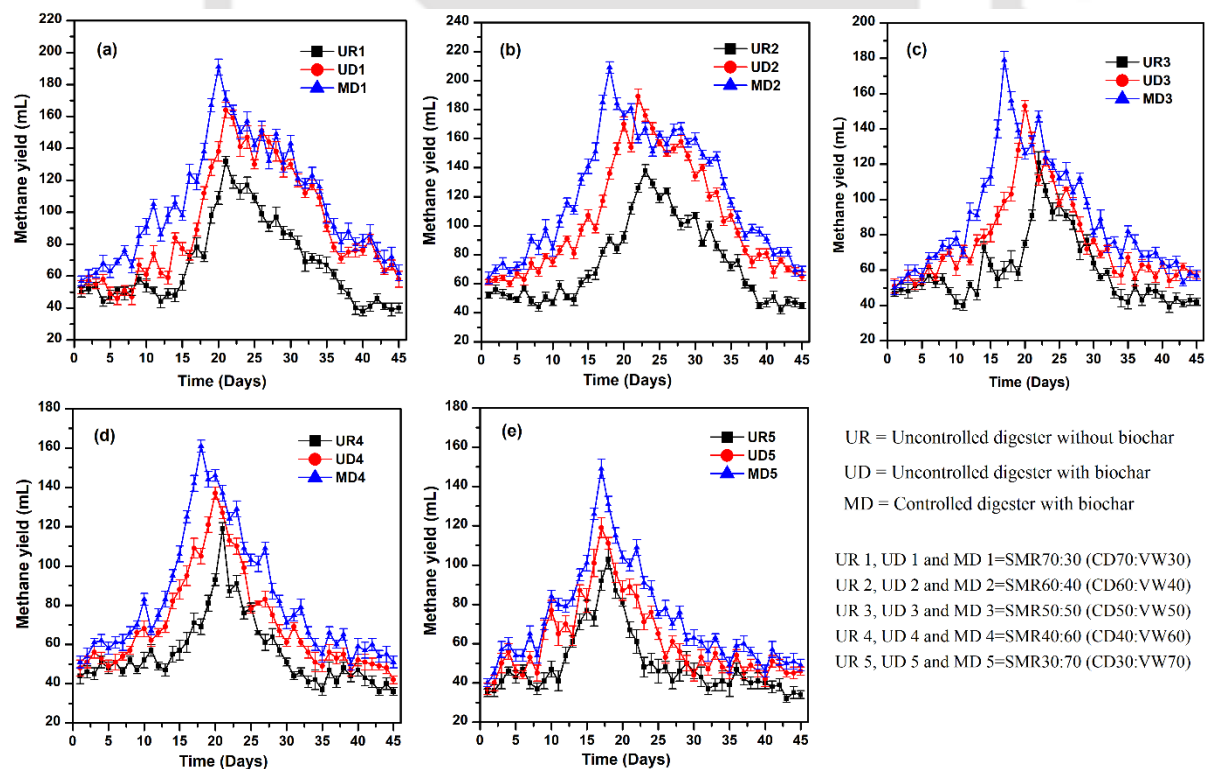


Fig. 4.10. Daily methane yield from the digesters (a) SMR 70:30, (b) SMR 60:40, (c) SMR 50:50, (d) SMR 40:60, and (e) SMR 30:70

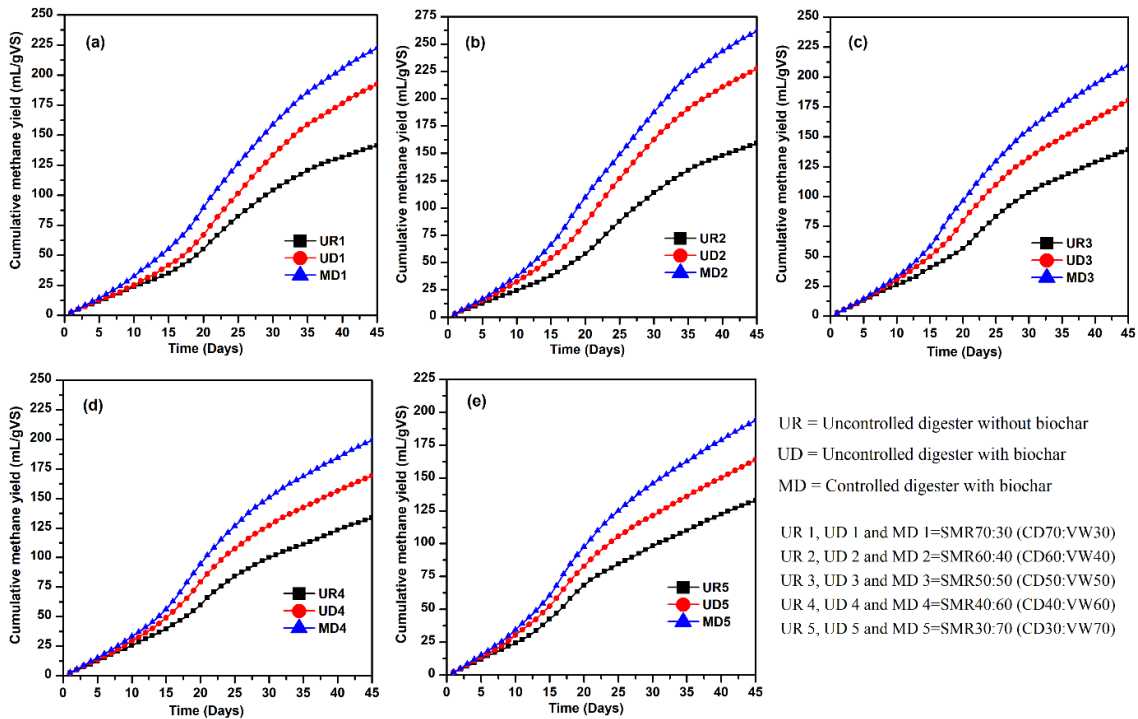


Fig. 4.11. Cumulative methane yield from the digesters (a) SMR 70:30, (b) SMR 60:40, (c) SMR 50:50, (d) SMR 40:60, and (e) SMR 30:70

In addition, it is desirable to incorporate a suitable dose of BC into the digester to achieve a higher methane yield. In this regard, another group of BMP experiments (set IV) was conducted at ambient conditions by adding varying quantities of water hyacinth BC, such as 5, 10, 15, 20, and 25 gL⁻¹. These digesters were designated as BCD 05, BCD 10, BCD 15, BCD 20, and BCD 25, respectively. The daily and cumulative CH₄ yield for set IV are presented in Fig. 5. The SMY for digesters BCD 05, BCD 10, BCD 15, BCD 20, and BCD 25 were found to be 199.28, 213.40, 240.07, 225.51, and 208.62 mLCH₄/gVS for HRT of 45 days. Moreover, BCD 15 exhibited 16.99%, 11.11%, 6.06%, and 13.10% higher CMY than BCD 05, BCD 10, BCD 20, and BCD 25, respectively. This study reveals that blending 15 gL⁻¹ water hyacinth-based BC in the CMR of 60:40 can be realised as the most significant CMR for the AcoD of CD and VW. Optimisation of SMR for AcoD provides numerous advantages, including boosting the methane yield rate, enhancing process stability, mitigating inhibition, utilising

various substrates, nutrient recycling, improving system performance, and supporting environmental sustainability.

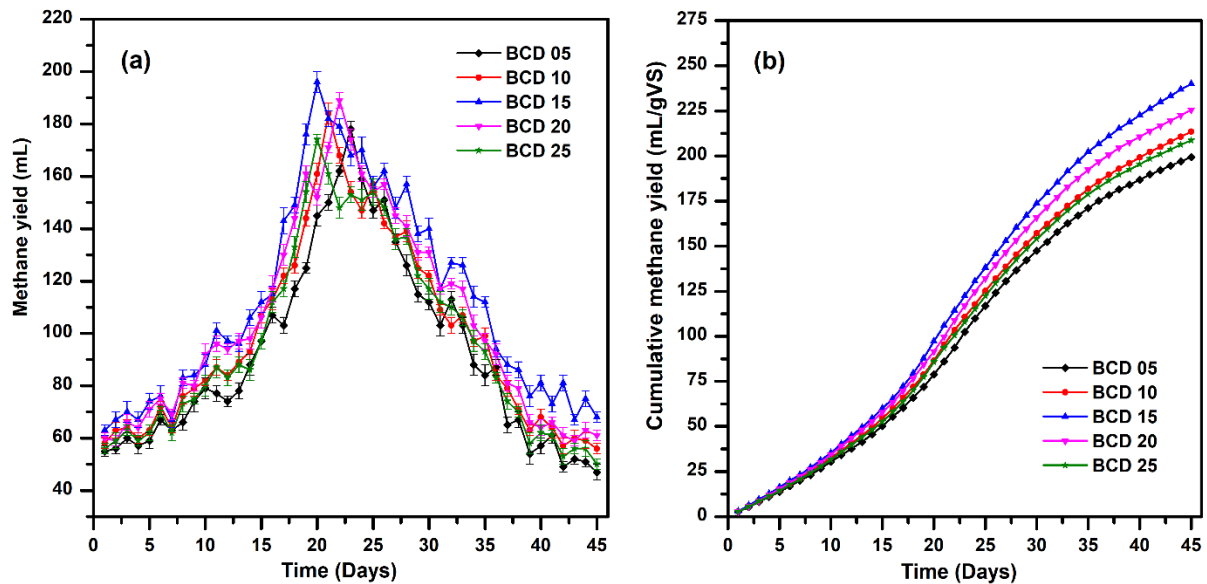


Fig. 4.12. (a) Daily and (b) cumulative methane yield for the digesters with varying amounts of biochar addition

4.4.3.2 pH value and temperature

Fig. 4.13 depicts the pH value recorded at ten-day intervals for all the groups of the BMP test. The decrease in pH value was observed during the first week; however, it was detected to be increased in the second week afterwards. This phenomenon occurs because acidogenic and acetogenic bacteria produce acids and CO_2 during the initial phase of co-digestion. Afterwards, the pH value rises due to the consumption of acids by methane-generating bacteria (Kwietniewska et al., 2014). The pH range for set I was obtained to be 4.6 to 6.5, while the incorporation of WHBC into sets II and III increased the pH levels to a range of 5.4 to 6.8. In set IV, the pH value was observed to rise as the quantity of WHBC increased. Adding 25 g/L WHBC resulted in a higher pH value of 6.3–6.9, whereas 5 g/L WHBC addition led to a lower pH range of 5.8–6.4. Several AAE metals (Na, K, Ca and Mg), which were detected in the EDX analysis of the BC, improve the alkalinity level of the reactors

(Aramrueang et al., 2022). Additionally, it is necessary to optimise the quantity of BC required to maintain the suitable pH range for different substrates. The daily temperature variations recorded every 4 hours for uncontrolled digesters ranged from 19.7 to 26.4 °C throughout the experimental period.

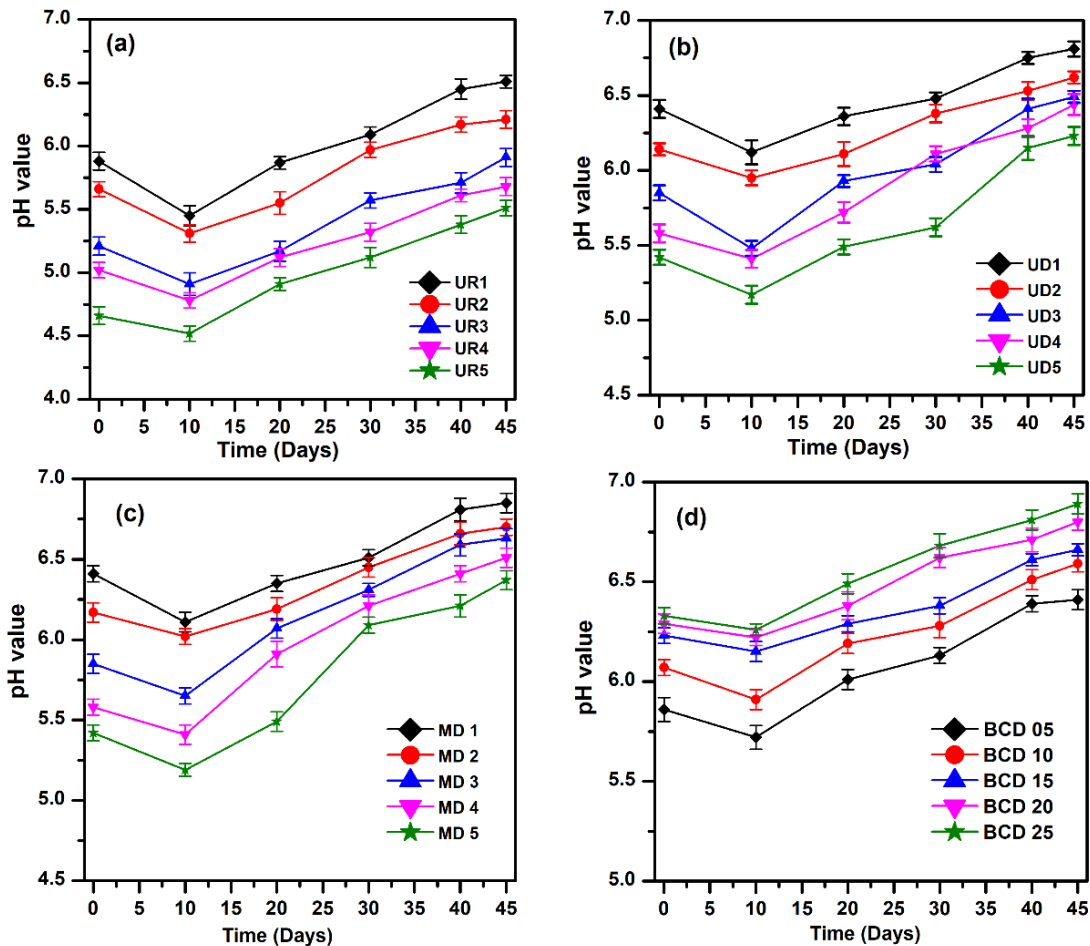


Fig. 4.13. Variation of pH value at (a) ambient temperature without biochar, (b) ambient, (c) mesophilic temperatures with biochar, and (d) ambient with different amounts of biochar for SMR 60:40.

4.4.3.3 VS degradation

Reduction in VS value helps to estimate the performance of the AD process. Fig. 4.14 illustrates the volatile solid reduction (VSR) analysis conducted at ten-day intervals for all BMP test sets. The VSR was evaluated using the Van Kleeck equation (Switzenbaum et al., 2003). The VSR in sets I, II and III were obtained to be 53.00–60.10%, 57.00–63.70%, and

59.30–65.15%, respectively. The SMR 60:40 resulted in the highest VSR for all groups of experiments. Further, the mesophilic digesters were found to have a higher VSR value. The VSR value for SMR 60:40 of sets III was 65.14%, higher than SMR 60:40 of sets I (60.10%) and II (63.71%). Similarly, the volatile solid reduction of the reactors BCD 05, BCD 10, BCD 15, BCD 20, and BCD 25 in set IV on the HRT of 45 days were calculated as 61.26, 62.20, 64.16, 63.77 and 62.05%, respectively, exceeding the VSR for CMR 60:40 of set I. A decrease in VS favours the AcoD process, indicating increased biogas yield (Barua and Kalamdhad, 2017). The SMR with the highest VSR exhibited a higher CH₄ generation rate and vice versa.

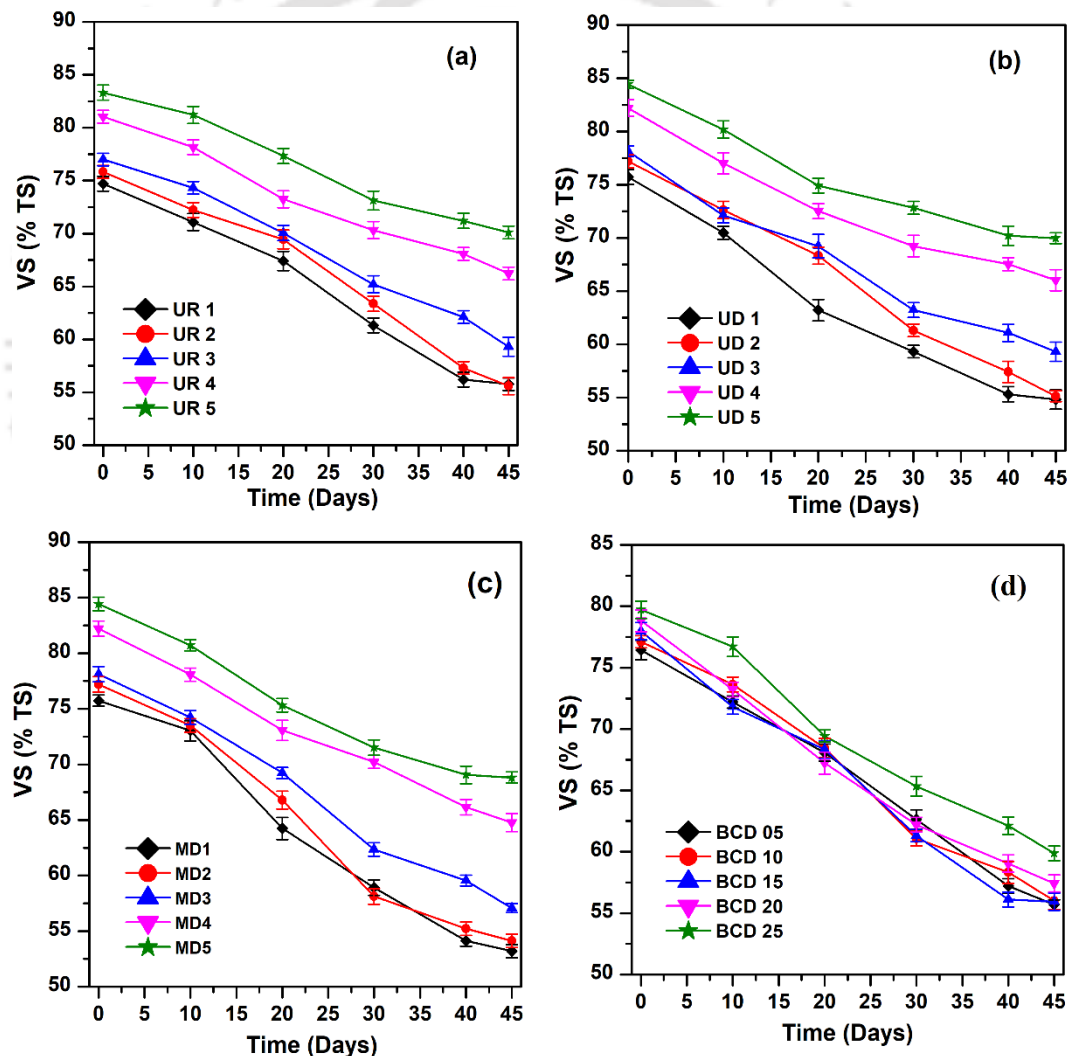


Fig.4.14. Degradation of VS (% TS) content under (a) ambient temperature without biochar, (b) ambient, (c) mesophilic temperatures with biochar, and (d) ambient with different amounts of biochar for SMR 60:40.

4.4.3.4 Kinetics analysis

The results of the kinetic study are incorporated in Table 4.10. The kinetic parameters for all sets of experiments were tested. The SMR of 60:40 resulted in a higher specific CH₄ production potential than other SMRs within their corresponding experimental groups, whereas the SMR of 30:70 exhibited a lower BMP value. Higher Y_P and Y_R values were obtained for the biochar-added mesophilic digester with an SMR of 60:40 compared to respective digesters with and without incorporation of BC performed at ambient temperature. Similarly, in the case of set IV, which was conducted by adding varying quantities of BC, 15 g/L biochar-added digester resulted in a higher CH₄ generation potential. The specific CMY potential for the digester with 15 g/L BC-added was 268.95 mL/gVS, whereas the 15 g/L BC-added digester exhibited a lesser potential (229.22 mL/gVS). Furthermore, BC 15 showed a higher value of Y_R (7.58 mL/gVS-day) compared to other reactors of set IV. This study confirms that biochar can enhance methane generation rate in the co-digestion of cattle dung and untreated vegetable waste, where 15 gL⁻¹ BC added may be considered the optimum amount of biochar required to be mixed. The R^2 value was above 0.98, demonstrating the predicted and experimental results fit well.

Table 4.10. Results of kinetic analysis for AcoD of CD and VW

Digesters	Y_P (ml CH ₄ /gVS)	Y_R (ml/gVS-day)	λ (day)	Y_C (ml/gVS)	R^2
Set I (Without biochar at ambient temperature)					
UR1	154.67	4.39	6.47	142.31	0.99
UR2	174.82	4.89	6.11	158.98	0.99
UR3	150.42	4.11	5.05	139.39	0.99
UR4	144.84	4.02	4.57	133.65	0.99
UR5	145.91	4.11	4.29	132.88	0.99
Set II (With biochar at ambient temperature)					
UD1	226.28	6.03	6.14	192.33	0.99
UD2	240.47	7.17	5.36	227.24	0.99

UD3	206.77	5.58	5.59	180.12	0.99
UD4	188.84	5.41	5.36	169.07	0.99
UD5	177.15	5.16	4.49	163.86	0.99
Set III (With biochar at mesophilic temperature)					
MD1	250.00	6.93	6.86	222.26	0.99
MD2	275.23	8.23	6.77	261.75	0.99
MD3	224.99	6.79	5.93	209.52	0.99
MD4	220.28	6.54	5.72	199.20	0.99
MD5	209.66	6.27	4.89	193.90	0.98
Set IV (With varying biochar at ambient temperature)					
BCD 05	239.52	6.45	6.97	199.28	0.99
BCD 10	245.05	6.81	6.72	213.40	0.99
BCD 15	268.95	7.58	6.65	240.07	0.99
BCD 20	239.26	6.22	6.75	225.51	0.99
BCD 25	229.22	6.13	6.85	208.62	0.99

4.4.4 Summary of AcoD of CD and VW

Incorporating water hyacinth biochar into the AcoD of untreated vegetable waste with cattle dung has significantly enhanced the methane generation rate. This enhancement can be attributed to the porous nature of BC, which provides a colonial space for microbes. The macronutrients, alkali and alkaline earth metals of biochar enhance the slurry's alkalinity in the AD. Further, the functional groups (O–H, C–H, C=C, and C–O) increase the electron exchange among the electron donor and acceptor microorganisms. The most favourable SMR of CD and VW was identified at 60:40, as assessed by the BMP tests. Additionally, biochar-added mesophilic digesters with a 60:40 co-substrates ratio resulted in 39.26% and 13.19% higher CH₄ yield than those without and with biochar at ambient temperature for the BMP test period of 45 days. The addition of 15 gL⁻¹ BC to AcoD of CD and VW exposed the significant quantity of BC necessary for effective blending. This study determines that blending a suitable amount of BC into AcoD can boost CH₄ production.

4.5 ANAEROBIC CO-DIGESTION OF CATTLE DUNG AND COOKED KITCHEN WASTE

This section discusses the anaerobic co-digestions of cattle dung and cooked kitchen waste at five different SMRs under ambient and mesophilic temperatures. The effect of biochar addition on methane yield, temperature, pH value, and degradation of volatile solids during the anaerobic digestion were recorded and compared.

4.5.1 Characterisation of co-digestion of cattle dung and cooked kitchen waste

The MC, TS, VS, and initial pH values of CD and CKW at various SMRs viz. 100:0, 0:100, 70:30, 60:40, 50:50, 40:60, and 30:70 are presented in Table 4.11. Examining these parameters is crucial for assessing the suitability of a substrate for AD since they significantly influence the stability of the AD process and, consequently, CH₄ generation. In the current study, a significant quantity of MC was detected in the substrates and the slurry of CD and CKW. The appropriate range of MC assists the anaerobic bacteria in the hydrolysis and acidogenesis phases of AD, whereas severely low moisture levels impede the anaerobic digestion process due to acid build-up (Odejobi et al., 2021; Osei-Owusu et al., 2023). In contrast, a high MC in the substrate promotes microbial activity in AD (Lay et al., 1997). Several research studies have described the significant role of TS content in influencing the rates of hydrolysis and methanogenesis, consequently impacting methane generation in the AcoD of organic wastes (Indren et al., 2020; Johnravindar et al., 2022). Abbassi-Guendouz et al. (2012) observed the highest methane generation rate at a TS concentration of 10%, which decreased if the TS value increased from 10% to 30%. High TS contents can impede mass transfer between microorganisms and substrate and lead to a decline in methane yield (Johnravindar et al., 2022). In the present study, TS values of the slurries at different percentages of CD and CKW mixing ratio were found in the range of 8.70–9.70%, whereas its value was 15.90% and 18.58% for 100% CD and 100% CKW, respectively. The TS value of

100% CD was detected to be aligned with the literature reported by Wang et al. (2022). The role of VS content is also significant because it represents the organic matter within a substrate that can be transformed into biogas through the AD process. Table 4.11 demonstrates that the slurry's VS content increased from 76.75% to 82.15% as the CKW percentage increased. The pH level of the slurry was perceived in the acidic range (5.0–5.4). The detection of a low pH value can be attributed to the oiliness and spiciness commonly found in Indian cooked foods. The C/N ratio is also an essential factor as it measures the nutrient level of microorganisms. The C/N ratio of 100% CD and 100% CKW were found to be 25.82 ± 1.10 and 13.33 ± 1.07 , whereas, for slurries at different SMRs, its values ranged from 14.30–17.60. The C/N ratio of the present study was detected to be lower than the optimum C/N ratio (20–30), as its value was low for CKW. Studies have reported that CKW can be effectively digested to produce higher methane yield, even at lower C/N ratios (Salangsang et al., 2022; Wang et al., 2023). In this regard, Salangsang et al. (2022) found higher methane yields at a C/N ratio of 13–18 compared to a higher C/N ratio of 23–31.

Table 4.11 Initial characterisation of CD and CKW and their slurries

Parameters	100%	100%	Substrate mixing ratio of CD and CKW				
	CD	CKW	70:30	60:40	50:50	40:60	30:70
MC (%)	84.08 ± 0.26	81.42 ± 0.12	91.29 ± 0.45	90.44 ± 0.10	90.30 ± 0.30	90.55 ± 0.03	90.56 ± 0.15
TS (%)	15.90 ± 0.17	18.58 ± 0.12	8.71 ± 0.45	9.56 ± 0.10	9.70 ± 0.30	9.45 ± 0.03	9.44 ± 0.15
VS (% TS)	83.50 ± 0.10	92.35 ± 0.07	76.78 ± 0.78	77.94 ± 0.45	79.38 ± 0.42	81.90 ± 0.47	82.14 ± 0.35
pH value	6.37	5.05	5.41	5.37	5.21	5.17	5.09

	±0.32	±0.12	±0.06	±0.03	±0.05	±0.03	±0.03
C (%)	36.69	39.45	40.66	40.94	41.29	41.89	42.02
	±0.12	±0.11	±0.09	±0.12	±0.09	±0.10	±0.13
N (%)	1.42	2.96	2.32	2.44	2.55	2.63	2.92
	±0.05	±0.04	±0.05	±0.05	±0.03	±0.11	±0.08
C/N ratio	25.82	13.33	17.53	16.78	16.19	15.93	14.39
	±1.10	±1.07	±0.91	±1.30	±0.72	±0.81	±1.02

4.5.2 Characterisation of coconut husk biochar

The initial characterisations of BC are shown in Table 4.12. The BC was found to have a high C and N content, providing nutrients to microorganisms (Pan et al., 2019). The EDX analysis confirmed the existence of inorganic materials like K, Ca, Mg, Na, and P in BC. Alkali and alkaline earth metals in BC increase the AD system's alkalinity, consequently enhancing methane production (Chiappero et al., 2020). Further, BC's pH value was 9.58 ± 0.08 , signifying a high alkaline nature. The BET surface area, pores volume, and diameter were measured as $50.03 \text{ m}^2/\text{g}$, $0.05 \text{ cm}^3/\text{g}$, and 3.23 nm , respectively, indicating the textural properties of BC. Aramrueang et al. (2022) reported that the high porosity of BC provides a large surface area for carbon dioxide removal during carbonation reaction in the AD system, leading to higher methane production (Aramrueang et al., 2022).

Table 4.12 Initial characterisation of coconut husk biochar

Analysis	Parameter	Unit	Value
Proximate analysis			
	Moisture content	%	3.28 ± 0.06
	Ash content	%	10.43 ± 0.23
	Volatile matter	%	15.51 ± 0.18
	Fixed carbon	%	70.78 ± 0.07

Ultimate analysis			
	C	wt %	76.83±0.19
	H	wt %	3.65±0.07
	N	wt %	1.16±0.12
	S	wt %	0.23±0.09
	O	wt%	18.36±0.11
Alkali and Alkaline earth elements			
	K	wt %	5.67±0.45
	Ca	wt %	0.27±0.03
	Mg	wt %	0.20±0.03
	Na	wt %	0.50±0.02
Others elements			
	P	wt %	0.18±0.03
	Cl	wt %	2.03±0.32
	Si	wt %	0.53±0.03
BET analysis			
	BET surface area	m ² /g	50.03
	Pore volume	cm ³ /g	0.05
	Pore diameter	nm	3.23
Other properties			
	pH		9.58±0.08
	EC.	dS/m	35.29±0.21

The visual inspection of BC's FESEM microphotographs illustrates its microstructure. The SEM image (Fig. 4.15 (a)) of BC reveals a highly porous structure with oval-shaped pores arranged in a tubular pattern, which can provide colonial areas for microorganisms. The results of the FTIR spectra wavelength shown in Fig. 4.15 (b) were compared with some reported literature, as shown in Table 4.13. The functional groups like O–H, C–H, N–H, C=C, and C–O, which were detected at different wavelengths, have the potential to enhance direct interspecies electron transfer and increase the microbial activity of anaerobic bacteria (Shen et

al., 2020). The XRD spectra of the BC are shown in the Fig. 4.15 (c). The peak points demonstrate the existence of CaCO_3 (calcite, $2\theta=29.36^\circ$), CaSO_4 ($2\theta=40.99^\circ$, 66.75°), K_2SO_4 ($2\theta=58.76^\circ$), and KOH ($2\theta=50.37^\circ$) crystals in BC. Due to the presence of substantial surface area and active sites in BC, it has the potential to provide buffering capacity during decomposition in the AD process (Jiang et al., 2020).

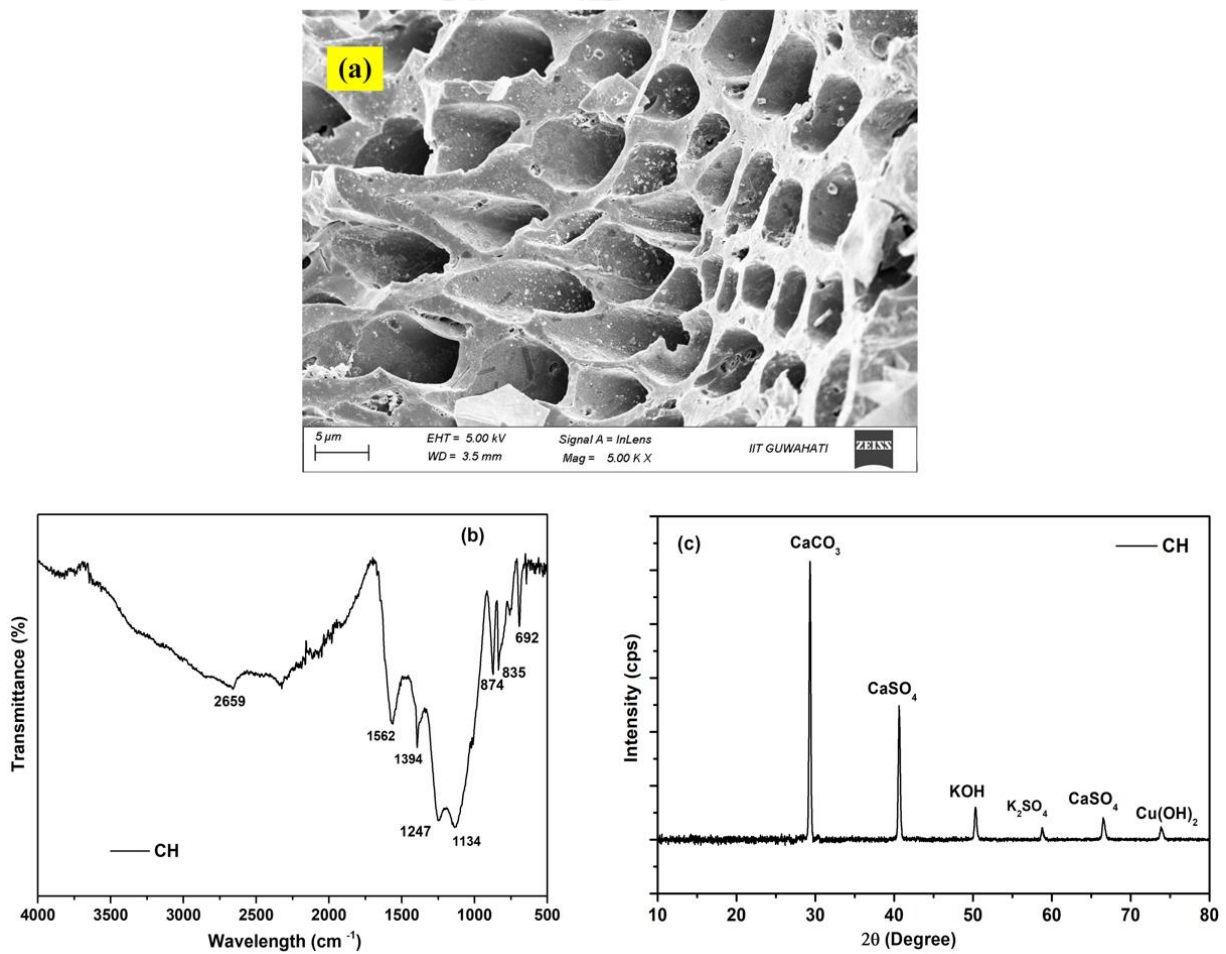


Fig. 4.15. (a) FESEM image, (b) FTIR spectra, and (c) XRD spectra of biochar

Table 4.13 Visible wavelength and functional groups of the CH biochar

Wavelength (cm ⁻¹)	Wavelength from this study (cm ⁻¹)	Bond	Functional group	Reference
3300–2500	2659	O–H stretching	Carboxylic acid	(Merck KGaA, 2023)
1640–1550	1562	N–H	Primary and secondary amine and amide	(NIU, 2023)
1420–1330	1394	O–H bending	Alcohol	(Merck KGaA, 2023)
1275–1200	1247	C–O stretching	Alcohol, ether	(NIU, 2023) (Merck KGaA, 2023)
1205–1124	1134	C–O stretching	Alcohol	(Merck KGaA, 2023)
900–700	874	C–H bending	Alkene, aromatic	(Bordoloi et al., 2022)
840–790	835	C=C bending	Alkene	(Merck KGaA, 2023)
730–665	692	C=C bending	Alkene	(Merck KGaA, 2023)

4.5.3 Results of anaerobic co-digestion of cattle dung and cooked kitchen waste

4.5.3.1 Daily and cumulative methane yield

The daily and cumulative CH₄ yield for AcoD of CD and CKW at various SMRs viz. 100:0, 0:100, 70:30, 60:40, 50:50, 40:60, and 30:70 under ambient and mesophilic temperatures are presented in Fig. 4.16 and Fig. 4.17. From Fig. 4.16, it was evident that CH₄ production was initiated from the very first day for all SMRs. In the first week, the DMY was less; however, the gas generation increased from the 2nd week onwards. A higher rate of DMY was achieved between the 2nd and 4th weeks of AD. The temperature-controlled and BC-added digesters detected a higher DMY rate than the respective uncontrolled digesters. In comparison to the reactors R1, R2, R3, R4 and R5 maintained at ambient temperature, the mesophilic reactors RC1, RC2, RC3, RC4, and RC5 resulted in higher DMY with peak values of 128 mL on the 20th day, 144 mL on the 17th day, 158 mL on the 16th day, 165 mL on the 19th day, and 148 mL on the 18th day, respectively. Likewise, the maximum peak values of DMY for the

controlled digesters DC1, DC2, DC3, DC4 and DC5 with added BC were obtained on the 18th (151 mL), 17th (185 mL), 17th (224 mL), 20th (227 mL) and 17th (174 mL) days of the retention time, respectively. The declination of DMY was found to be started at the end of the 3rd week for all reactors. The cumulative methane yield at 55 days (CMY55) of the mesophilic digesters without and with biochar addition was higher than that of the respective digesters at ambient temperature. The ambient temperature digesters C5, C6, D1, D2, D3, D4, and D5 of set III with BC added generated 15.97, 16.26, 18.07, 22.06, 21.68, 20.85 and 17.91% higher volumes of CMY55 than the respective digesters of set I without BC, respectively, whereas 13.59, 13.14, 15.19, 20.59, 25.43, 24.63 and 22.89% higher amount of CMY55 were obtained for the mesophilic digesters C7, C8, DC1, DC2, DC3, DC4 and DC5 with BC added than the respective digesters of set II without BC, respectively. Compared with the mono-digestion of CD and CKW, the AcoD digesters achieved higher CMY yield in all sets of experiments. This analysis confirms that AcoD enhances the methane production rate. Numerous researchers have reported that BC plays a vital role by providing a surface area for microbial colonisation and serving as an adsorbent for compounds like limonene and NH₃, which might otherwise impede the performance of the AD system (Wambugu et al., 2019). Biochar has adsorption properties that can mitigate the negative effects of inhibitory compounds present in the feedstock or produced during digestion. By adsorbing these compounds, biochar prevents their interference with microbial activity, thereby enhancing methane production. Additionally, the alkaline properties of BC enhance CH₄ content through a reaction of CO₂ and H₂S with alkaline elements present in ash, thereby improving the biogas quality (Wang et al., 2017). The evidence of various factors and properties found in BC that contributed to the enhancement of methane yield was illustrated in Section 4.5.2. Further, BC can act as a thermal insulator, helping to maintain stable temperatures within the digester. This is particularly beneficial in mesophilic conditions, where optimal temperatures are required for efficient methanogenesis. In the current study, comparing all the mixing ratios, the SMR 40:60 of CD and CKW was detected to be the best SMR for all sets of experiments. The specific methane yield (SMY) obtained for

SMR 40:60 at HRT of 55 days were 168.81, 188.41, 213.29, and 249.98 mL/gVS for sets I, II, III, and IV, respectively. This study confirms that the SMR 40:60 can be considered the most effective feedstock mixing ratio for the AcoD of CD and CKW. Further, methane production for the mesophilic digesters with BC added was detected to be higher than that of the ambient temperature digesters with BC added. This study demonstrates the feasibility of conducting the AD process under mesophilic conditions with the incorporation of BC to achieve methane generation.

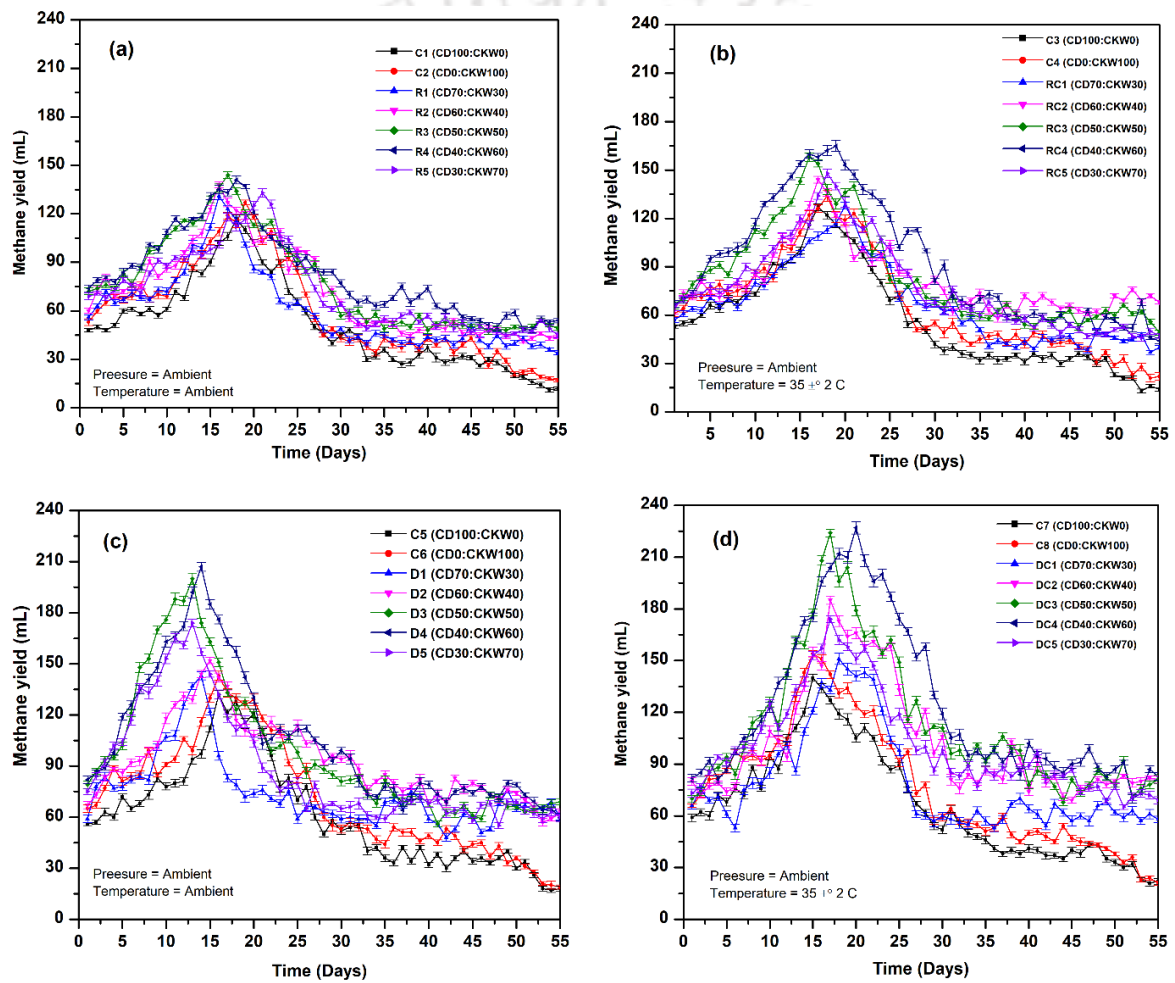


Fig. 4.16. Daily methane yield at (a) ambient, (b) mesophilic temperatures without biochar addition, and (c) ambient, (d) mesophilic temperatures with biochar addition

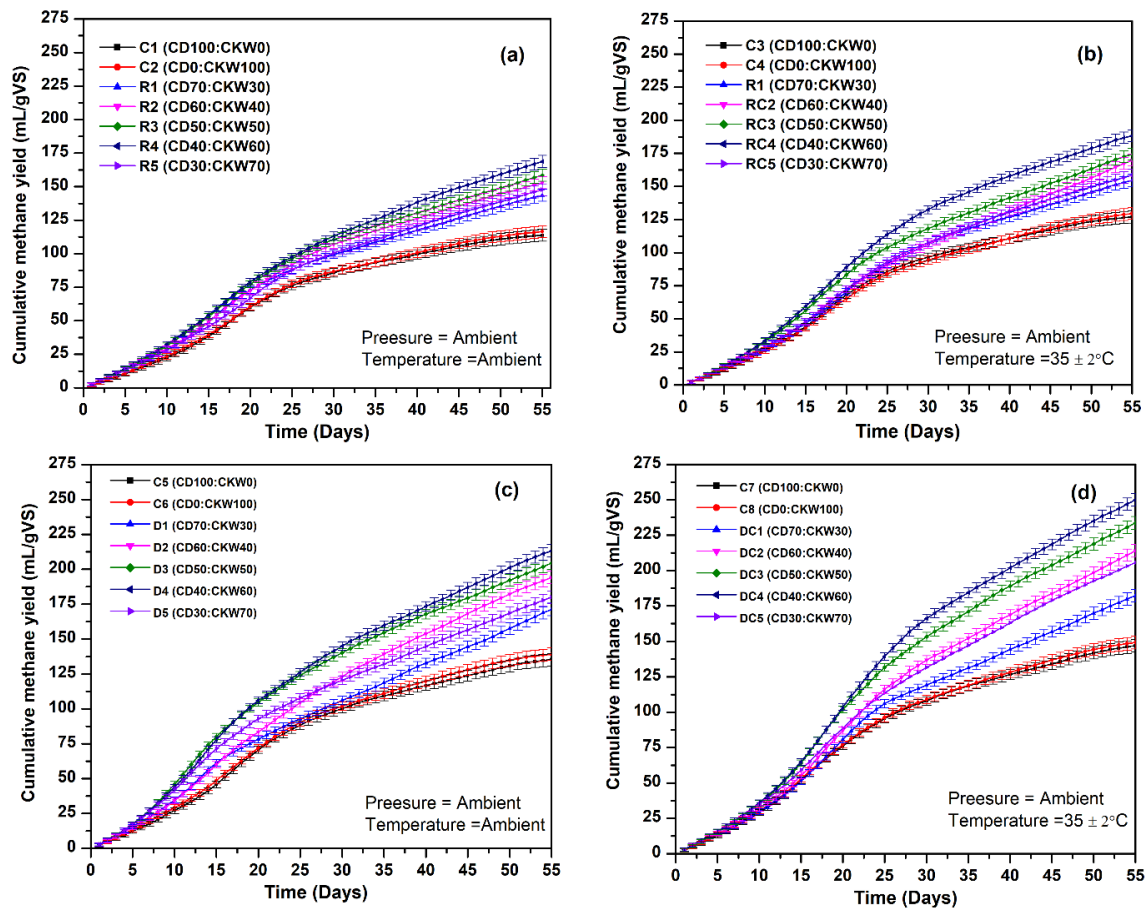


Fig. 4.17. Cumulative methane yield at (a) ambient, (b) mesophilic temperatures without biochar addition, and (c) ambient, (d) mesophilic temperatures with biochar addition

Additionally, determining an appropriate quantity of BC to be added to the anaerobic digester for maximum methane generation is desirable. In line with the ideal SMR 40:60, another set of BMP tests (set V) was carried out at ambient temperature by adding varying amounts of biochar: 5 g/L, 10 g/L, 15 g/L, 20 g/L, and 25 g/L, representing the digesters by BC 05, BC 10, BC 15, BC 20, and BC 25 respectively. The results of DMY and CMY for set V are depicted in Fig. 4.18. The SMY for digesters BC 05, BC 10, BC 15, BC 20, and BC 25 were found to be 184.04, 193.35, 214.56, 208.13, and 197.60 mLCH₄/gVS for HRT of 55 days. Moreover, BC 15 exhibited 14.23%, 9.89%, 3.00%, and 7.91% higher CMY than BC 05, BC 10, BC 20, and BC 25, respectively. This study demonstrates that 15 g/L BC addition in the slurry of SMR 40:60 can be recognised as the most effective substrate mixing ratio for the

AcoD of CD and CKW. Optimising SMR in AcoD processes provides various benefits, including enhanced CH₄ production, improved process stability, reduction of process inhibition, utilisation of diverse feedstocks, nutrient recycling, increased process efficiency, and environmental sustainability.

Furthermore, a one-way analysis of variance (ANOVA) was also performed for all sets of experiments to determine whether the CMY differences were statistically significant. The detailed results of ANOVA have been provided in the supplementary material. The analysis reported that the CMY was statistically significant ($p < 0.001$) with high F-values ($F > 4$) for each set of experiments. The CMY was also compared within the individual set of experiments with controlled digesters. The CMY of the control digesters were not significantly different with SMR 70:30 ($p > 0.05$) and SMR 30:70 ($p > 0.05$) but were significantly different with SMR 60:40 ($p < 0.05$), SMR 50:50 ($p < 0.05$), and SMR 40:60 ($p < 0.05$) for each set of experiments. On the other hand, the p-values of set V were found to be $p > 0.05$, which indicates that CMY differences are not significant. The CMY of each digester of set V was detected as statistically significant to the SMR 40:60 of set I. This analysis demonstrates that the addition of the optimum quantity of biochar significantly promotes the CMY.

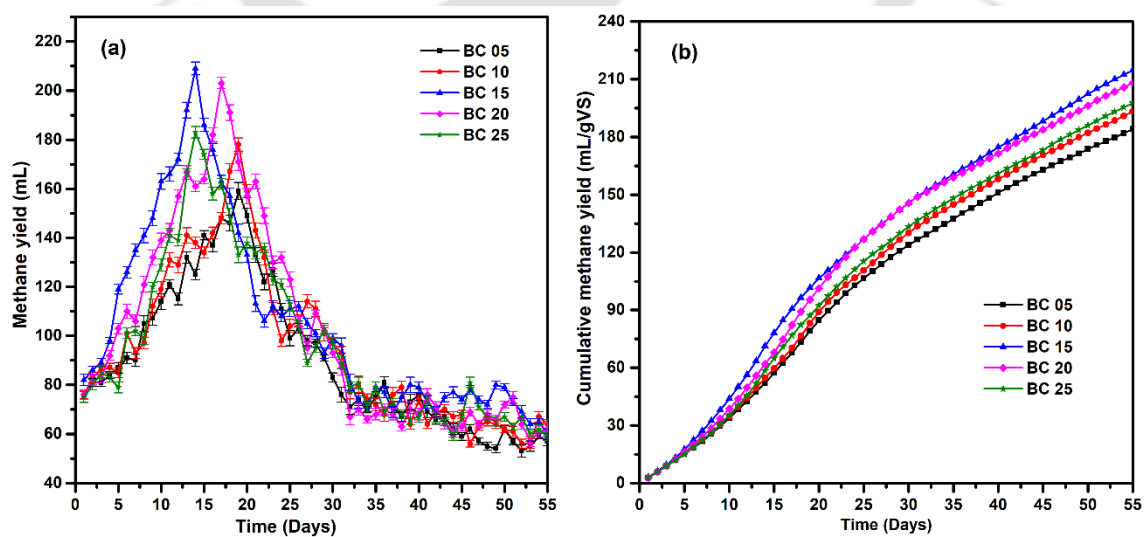


Fig. 4.18. (a) Daily and (b) cumulative methane yield for the digesters with varying amounts of biochar addition

4.5.3.2 Temperature variation

Temperature is the most crucial parameter for the evolution of microbes (S. Basumatary et al., 2021). In the present study, the temperature variations were recorded in intervals of 3h for the uncontrolled digesters. However, the temperature of controlled digesters was not monitored since they were consistently maintained at a mesophilic temperature (35 ± 2 °C). The daily maximum, minimum, and average temperature values were documented by considering the average temperature values for all the digesters, as the temperature in the uncontrolled digesters remained relatively constant during the recorded time intervals. The plot of temperature variations is shown in Fig. 4.19. The daily average temperature was detected in the 20.6–30.6 °C range with an average value of 26.7 °C for HRT of 55 days. The maximum and minimum temperatures were recorded to be 34.5 °C and 19 °C, respectively, throughout the experiments. The uncontrolled digesters' average temperature was below the mesophilic temperature range, resulting in a lower methane generation rate in these digesters. Thus, this study demonstrates that methane production can be improved by maintaining the reactor's temperature in the mesophilic condition.

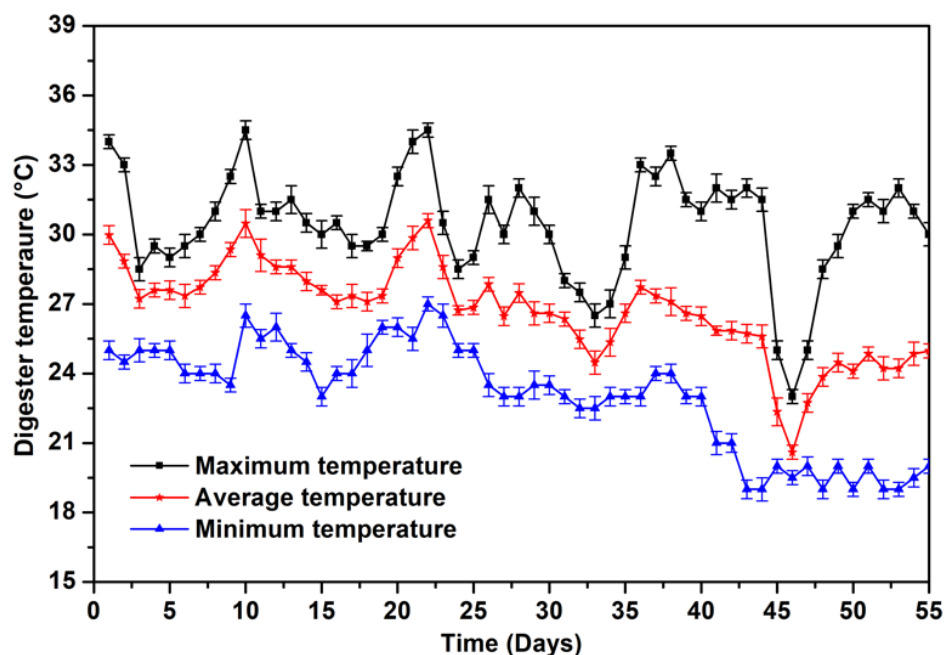


Fig. 4.19. Variation of digesters' temperature

4.5.3.3 pH value and VS degradation

The variations of pH value and VS degradation are illustrated in Fig. 4.20 and Fig. 4.21, respectively. The pH value and VS analysis were initially performed on the 7th day of the retention time. Afterwards, the variations of pH value and VS analysis were conducted at intervals of ten (10) days for all the sets of experiments. The pH and VS analyses indicate the changes occurring in the BMP test due to the AcoD of CD and CKW. The pH value decreases in the first week for all digesters but increases from the second week onwards. In the early phase of the AD process, microorganisms produce acids and CO₂, but later, pH increases as CH₄-producing bacteria consume the acids (Kwietniewska et al., 2014). The pH values remained consistently within a highly acidic range of 4.9–6.1 throughout the entire HRT for sets I and II. Improvement in pH value was obtained due to the BC addition in sets III, IV and V, which were 5.8–6.8. Several ions like Na, K, Ca and Mg in the BC increase the slurry's alkalinity level (Aramrueang et al., 2022). These metal ions were found to be present in the outcomes of the EDX analysis in this study. Further, optimising the required quantity of BC to be added is needed to maintain the desired pH range for various feedstocks.

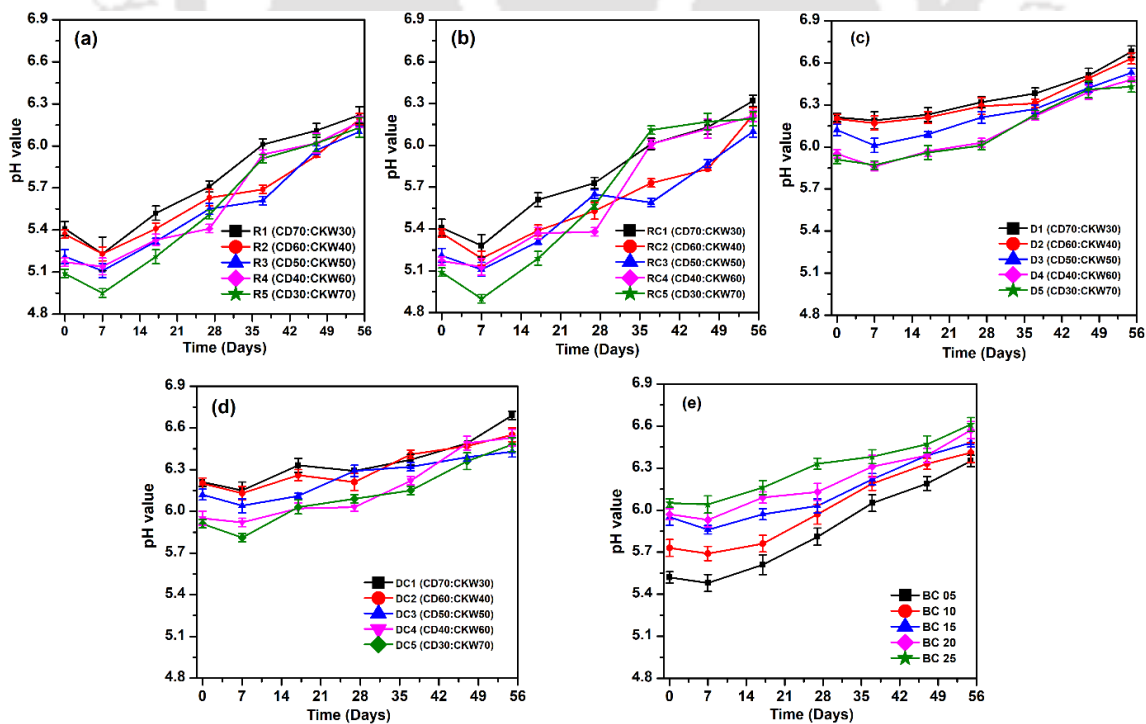


Fig. 4.20. Variation of pH value at (a) ambient, (b) mesophilic temperatures without biochar addition; and (c) ambient, (d) mesophilic temperatures with biochar addition; and (e) different amounts of biochar addition for SMR 40:60.

A decrease in VS content was detected as the analysis of the samples progressed over time, as depicted in Fig. 4.21. The volatile solid reduction (VSR) was determined following the Van Kleeck equation, as outlined in the literature by Switzenbaum et al. (2003) (Switzenbaum et al., 2003). The VSR in sets III and IV were in the range of 52.30–64.50% and 56.50–67.00%, whereas 48–62% and 52.5–63.90% for sets I and II, respectively. The highest VSR was achieved for SMR 40:60 for all sets of experiments. The VSR in the SMR 40:60 of sets III and IV were 64.36% and 66.85%, noticed to be higher than SMR 40:60 of sets I (61.84%) and II (63.60%), respectively. Likewise, the VSR for the digesters BC 05, BC 10, BC 15, BC 20, and BC 25 in set V were calculated as 62.22, 62.83, 64.41, 63.17 and 63.10% respectively, surpassing the VSR of the digester with SMR 40:60 in set I. A reduction in VS is a positive outcome of the AcoD process, which signifies an enhancement in biogas production (Barua en Kalamdhad, 2017). The mixing ratios that depicted the highest methane yield showed maximum VSR and vice versa.

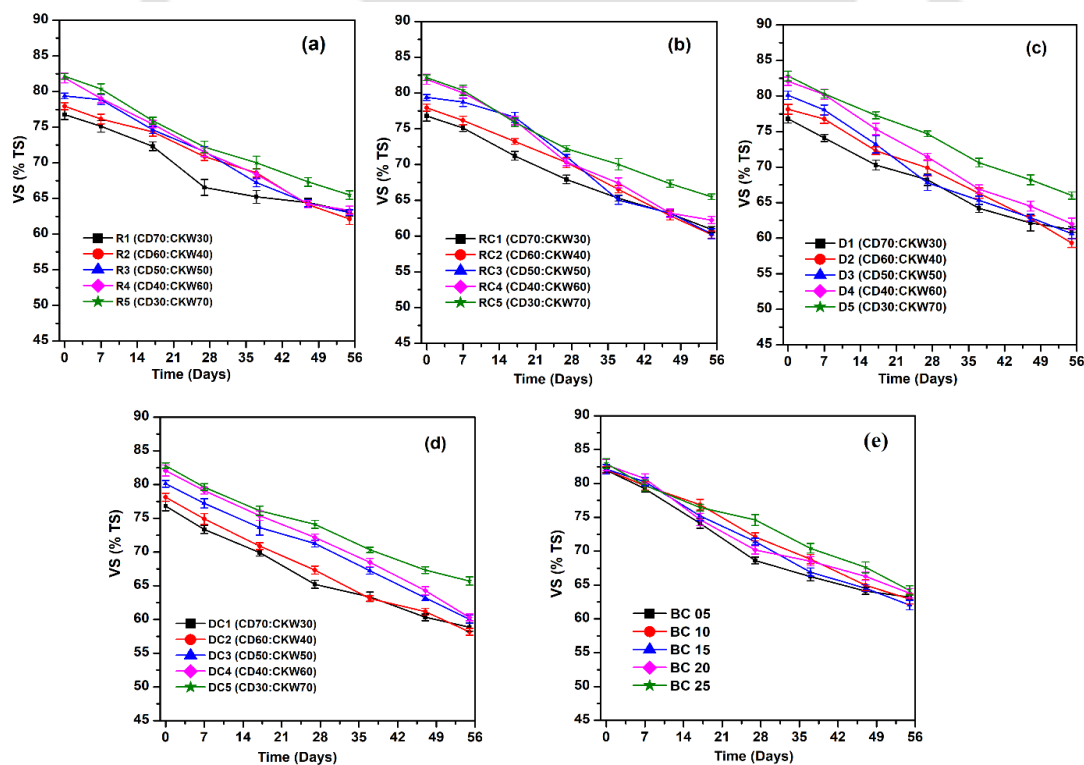


Fig. 4.21. Degradation of VS (% TS) content at (a) ambient, (b) mesophilic temperatures without biochar addition; (c) ambient, (d) mesophilic temperatures with biochar addition; and (e) different amounts of biochar addition for SMR 40:60.

4.5.3.4 Kinetic study

The outcomes of the Gompertz model analysis are depicted in Tables 4.14 and 4.15. The kinetic parameters Y_P , Y_R and λ for all sets of experiments were verified. The SMR 40:60 for sets I, II, III and IV yielded higher specific methane generation potential than other SMRs in the respective groups of experiments. In contrast, a lower value of methane generation potential was detected for the SMR 70:30 for AcoD of CD and CKW. The specific methane generation potential of mesophilic digesters RC4 (192.23 mL/gVS) and DC4 (260.37 mL/gVS) exceeded that of their corresponding ambient temperature digesters R4 (176.86 mL/gVS) and D4 (217.47 mL/gVS), respectively. Additionally, the Y_R value for RC4 (5.39 mL/gVS-day) and DC4 (6.85 mL/gVS-day) was also higher than that of R4 (4.39 mL/gVS-day) and D4 (5.57 mL/gVS-day), respectively. Likewise, in comparing the kinetics results between set V and R4 (SMR 40:60) of set I, BC 15 demonstrated a higher potential for CH₄ production rate. The specific CH₄ generation potential for BC 15 was 218.93 mL/gVS, whereas BC 05 achieved a lower potential (192.37 mL/gVS). The Y_R value for BC 15 (5.61 mL/gVS-day) was also higher than the other digesters of set V. The lag phase of all digesters was also determined using the Gompertz model. The lag phase represents the early period in the bacterial growth curve, which depends on factors such as the bacterial species, the composition of the growth medium, and environmental conditions. The exponential growth begins once bacteria adapt to their surroundings. In this study, the initial lag phase of all the digesters was found to be not too much difference. Only the biochar-added mesophilic digesters were detected slightly higher, possibly due to adjustments in environmental conditions. However, for SMR 40:60 with varying BC added, the lag phase time of BC 15 was determined to be lower. This investigation confirms that BC can improve the CH₄ generation in the AcoD of CD and CKW, where 15 g/L BC addition was detected as the ideal quantity of BC needed to be blended. The R^2 value for all sets of experiments was approximately 0.99, representing a close match between the predicted and actual experimental results.

Table 4.14 Results of kinetic analysis for AcoD of CD and CKW

<i>Mixing ratio</i>	Y_P (mL/gVS)				Y_R (mL/gVS. day)				λ (day)				Y_C (mL/gVS)				R^2
	Without BC		With BC		Without BC		With BC		Without BC		With BC		Without BC		With BC		
	Set I	Set II	Set III	Set IV	Set I	Set II	Set III	Set IV	Set I	Set II	Set III	Set IV	Set I	Set II	Set III	Set IV	
100:0	115.31	127.66	136.76	148.25	3.90	4.07	4.15	4.48	3.62	3.70	3.58	3.47	113.75	126.97	135.37	146.95	0.99
0:100	118.31	132.28	142.16	151.81	3.57	3.83	4.11	4.43	3.46	3.32	3.03	3.13	116.48	129.53	139.10	149.13	0.99
70:30	141.79	159.23	185.32	188.25	3.79	4.22	3.81	4.72	2.37	3.71	0.93	3.76	140.31	154.68	171.26	182.37	0.99
60:40	155.01	183.09	211.47	226.88	4.14	4.05	4.69	5.47	3.17	3.28	3.03	4.65	151.35	169.91	194.19	213.97	0.99
50:50	162.15	177.35	205.43	242.93	4.36	4.65	5.41	6.27	2.86	2.97	1.59	4.61	160.10	174.32	204.42	233.75	0.99
40:60	176.86	192.23	217.47	260.37	4.39	5.39	5.57	6.85	2.76	2.96	2.12	3.23	168.81	188.41	213.29	249.98	0.99
30:70	153.31	164.57	182.55	220.13	3.89	4.28	4.49	5.12	3.08	3.64	0.74	3.56	147.81	158.58	180.04	205.64	0.99

Set I and set II – Ambient temperature digesters, Set II, Set IV – Mesophilic temperature digesters and BC – Biochar

Table 4.15 Results of kinetic analysis for SMR 40:60 with varying amounts of BC addition on AcoD of CD and CKW

Digesters	Y_P (mL)	Y_R (mL/gVS-day)	λ (day)	Y_C (mL/gVS)	R^2
BC 05	192.37	4.85	3.31	184.04	0.99
BC 10	201.34	5.11	3.44	193.35	0.99
BC 15	218.93	5.61	2.12	214.56	0.99
BC 20	209.58	5.56	3.46	208.13	0.99
BC 25	203.26	5.27	3.28	197.60	0.99

4.5.4 Summary of AcoD of CD and CKW

Adding coconut husk biochar into the anaerobic co-digestion of cattle dung and cooked kitchen waste has proven to enhance methane production significantly. This improvement can be attributed to the porous structure of biochar, as well as the presence of macronutrients and metal ions such as Na, Ca, K, and Mg within it. The most favourable substrate mixing ratio of cattle dung and cooked kitchen waste was detected at 40:60 as evaluated by the BMP tests. Further, higher CMY was achieved at mesophilic temperature. The SMY of 188.41 mLCH₄/gVS and 249.98 mLCH₄/gVS were obtained without and with biochar addition at mesophilic conditions for a substrate mixing ratio of 40:60. The addition of 15 g/L biochar to AcoD of cattle dung and cooked kitchen waste revealed the ideal quantity of biochar needed to be blended. This study demonstrates that incorporating a suitable amount of biochar into anaerobic co-digestion can enhance methane generation efficiency.

4.6 SUMMARY OF THE CHAPTER

In the initial phase, the anaerobic digestion study for the substrates viz. cattle dung, cooked kitchen waste, and untreated vegetable waste was performed individually at different substrate water (S/W) mixing ratio under uncontrolled (ambient temperature) and controlled ($35\pm 2^\circ\text{C}$) conditions. The maximum cumulative biogas yield was detected at an S/W ratio of 50:50 for the cattle dung and cooked waste, whereas for vegetables, it was achieved at an S/W of 60:40. Further, controlled digesters exhibited higher biogas generation rates than uncontrolled digesters. The parameters other than TS and VS contents, the C/N ratio and the pH concentration of the substrates were also studied. The pH value for vegetable waste and cooked kitchen waste was detected in a highly acidic range that falls below the optimum range (6.8–7.2) throughout the entire retention time. Likewise, the C/N ratio of food waste was also not in the desired range. From the present study, it can be concluded that additional adjustment techniques are necessary for food waste to control the pH value within the desired range. Therefore, anaerobic co-digestion experiments were adopted to maintain these parameters to a nearly desirable range. Considering the ideal S/W ratio of 50:50, anaerobic co-digestions of VW and CKW with CD were studied by varying the co-substrate mixing ratios (SMR) of 70:30, 60:40, 50:50, 40:60 and 30:70 without and with biochar (BC) addition under controlled and uncontrolled temperatures. In the study of AcoD of CD and VW, the CMY of the BC-added digesters with SMR of 70:30, SMR 60:40, SMR 50:50, SMR 40:60, SMR 30:70 was found to be 26.53, 30.04, 22.62, 20.95, 18.91% higher volume than those respective digesters without BC, respectively, conducted at ambient temperature. Further, the mesophilic digesters with BC added generated 36.42, 39.26, 33.47, 32.90 and 31.47% higher volumes of CMY than the respective uncontrolled digesters without BC, respectively, for the HRT of 45 days. Likewise, in the study of AcoD of CD and CKW, the digesters with BC added generated 18.07, 22.06, 21.68, 20.85 and 17.91% higher volumes of CMY than the digesters without BC, respectively,

in the case of ambient temperature, whereas, 23.06, 29.27, 31.50, 32.47 and 28.13% higher amount of CMY were achieved for the mesophilic digesters compared to uncontrolled without BC digesters. This investigation concludes that maintaining the reactor temperature at mesophilic conditions can increase CH₄ generation. This study also confirms that BC can improve the AD process. Therefore, it can be inferred that the BC plays a vital role in the AD process by providing a surface area for microbial colonisation and serving as an adsorbent for compounds. Also, the alkaline properties of BC enhance CH₄ content through a reaction of CO₂ and H₂S with alkaline elements present in ash, thereby improving the biogas quality. The optimum SMR was obtained at SMR 60:40 for the AcoD of CD and VW but at SMR 40:60 for CD and CKW, as evaluated by the biochemical methane potential (BMP) tests. Considering the ideal SMR, another set of BMP tests was carried out at ambient temperature by adding varying amounts of biochar: 5g/L, 10 g/L, 15 g/L, 20 g/L, and 25 g/L to determine the appropriate quantity of BC to be added. The digester containing 15 g/L biochar exhibited the highest CMY for both cases, confirming that 15 g/L biochar addition can be recognised as the most effective for AcoD of cattle dung and food waste. This study demonstrates that incorporating a suitable amount of biochar into anaerobic co-digestion can enhance methane generation efficiency. In the next chapter, Chapter 5 presents and discusses the results of the field digester based on the methodology explained in Chapter 3.

CHAPTER 5

PERFORMANCE INVESTIGATION OF FIELD SCALE ANAEROBIC DIGESTION PLANT

This chapter deals with the anaerobic digestion of cattle dung on the field scale of a digester and focuses mainly on the effect of various parameters like pH and temperature on biogas yield from the anaerobic digestion.

5.1 CHARACTERISATION OF SUBSTRATE

The proximate and elemental analyses of CD are presented in Table 5.1. Substrate characterisation was carried out to understand the feasibility of biogas potential. The TS contents of CD slurry at various mixing ratios were found to be in the range of 6.00–9.20%. Salam et al. (2015) reported that various investigators recognised that the optimal value of TS contents falls between 7% and 9% for CD slurry for maximum biogas yield. For fresh CD, TS content was observed to be 15.90%, consistent with the results mentioned by (Salam et al. 2015) and (Wang et al., 2022). The VS content of the CD was found to be $14.71 \pm 0.10\%$, which falls in the range of the results described by Dhamodharan et al. (2015). Further, the MC at different S/W ratios of 1:1.5, 1:1, and 1.5:1 were found to be in the range of 90.50–94.00%, whereas, for the 100% CD, MC's value was $84.08 \pm 0.26\%$. A similar range of MC was reported in past literature (Xin et al. 2018; Venkateshkumar et al. 2023). The MC should be maintained in the desired range, as it helps the microorganisms in the hydrolysis and acidogenesis stages of AD process, while extremely low MC inhibits the AD process due to the accumulation of acids (Odejobi et al. 2021; Osei-Owusu et al. 2023). In contrast, high MC content in the feedstock increases the methanogenic activity during the AD process (Lay et al., 1997). Maintaining the C/N ratio in the desired range is also essential as it measures microorganisms'

nutrients (Mao et al., 2015). Several studies conveyed that the favourable value of the C/N ratio for the AD process falls in the range of 20–30 (Hagos et al., 2017), 25–30 (Y. Li et al., 2017a) or between 25 and 35 (Kondusamy en Kalamdhad, 2014). The deviation of the C/N value for the optimum condition causes a negative impact on biogas production (Hagos et al., 2017). Various investigations described that at the higher value of the C/N ratio, methanogenic bacteria consume nitrogen quickly, and in the case of the lower value, it causes accumulation of ammonia, which is toxic for microorganisms, thereby reducing biogas yield (Kothari et al., 2014; Mao et al., 2015). The C/N ratio value of 25.82 found in the present investigation falls in the optimum range for biogas production. Accordingly, the CD can be recommended as an appropriate substrate for the AD process compared with the literature.

Table 5.1. Initial characterisation of cattle dung

Parameters	Value (%)
<u>Proximate analysis (as received basis)</u>	
Moisture	84.08 ± 0.26
Total solid	15.90 ± 0.17
Volatile solid	14.71 ± 0.10
<u>Ultimate analysis (dry basis)</u>	
Carbon	36.69
Nitrogen	1.42
Hydrogen	3.51
Oxygen	58.39
C/N ratio	25.82
H/C ratio	0.10

5.2 RESULTS OF EXPERIMENTAL ANALYSIS OF FIELD-SCALE AD PLANT

5.2.1 Variation of pH value

Fig. 5.1 depicts weekly variations of pH value recorded from the 2nd week of April to October. The fluctuations of pH value were observed to be less in the field-scale plant digester. The pH values were 6.9–7.3, with an average of 7.2 for the entire experiment period. The study suggests that additional pH value adjustment methods are not required for biogas production through the AD process of CD with a 1:1 S/W ratio.

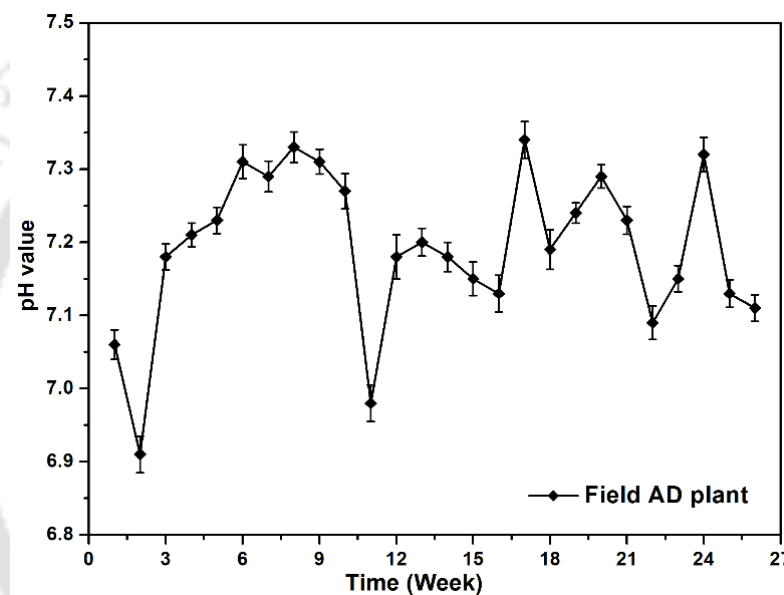


Fig. 5.1. Weekly pH value for field AD plant

5.2.2 Variation of temperature

The field AD plant's weekly average temperature variation plot is presented in Fig.5.2 (a). The digester's weekly average temperature for 28 weeks of the experimental period was found to be in the range of 26.3–34.8°C during the summer season. Experiments were conducted without a solar-assisted heating (SAH) system during this period. However, temperature fluctuations were observed in some weeks. There was a significant reduction in biogas production due to the temperature fluctuations that happened throughout the wet season. El-Mashad et al. (2003) demonstrated that the biogas generation rate declined due to

fluctuations in digester temperature and also mentioned the possibility of using solar energy in the AD process. Hence, maintaining digester temperature at the desired condition is essential for maximum biogas generation. During the winter season, the ambient average temperature of Guwahati, India, falls in the range of 19–24°C with a minimum average temperature of 14–18°C (Worldweatheronline, 2020). Therefore, an SAH was integrated with the digester to maintain the temperature at mesophilic conditions. The weekly average digester's temperature variations during the winter season under uncontrolled and controlled conditions are presented in Fig. 5.3 (a). The average temperature of the digester at ambient conditions, i.e. without the SAH system, was found in the range of 18–26°C during the winter (November–February). However, temperature improvement was noticed by applying an SAH system during the winter of the following year. The current investigation showed that it is possible to increase the digester's average temperature in the range of 31–34°C with the addition of the solar-assisted heat exchanger, which is sustainable for biogas production. Gaballah et al. (2020) reported a similar range of temperature increments.

5.2.3 Biogas production

The weekly and cumulative biogas generation from the field-scale AD plant under uncontrolled temperature for 28 weeks are presented in Fig. 5.2 (a) and 5.2 (b), respectively. The weekly biogas production rate in the 1st and 2nd weeks was relatively less but increased from the 3rd week onwards, and the highest peak value of 6.53 m³ was obtained in the 6th, 9th and 16th weeks. The fluctuations of biogas generation were detected between the 13th and 20th weeks of investigation. However, a relative reduction in biogas generation was also observed from the 25th week onwards. The reduction may be due to the decrease in temperature as the growth of microorganisms depends on the weather conditions. An average volume of 6.26 m³ biogas has been collected weekly, equivalent to 0.89 m³/day for the 50±1.50 kg CD slurry loading rate. Thus, the daily average biogas production of 0.04 m³/kg/day was recorded, which

was found to be in the range reported by (Ilbas et al., 2022). The cumulative biogas yield (CBY) was also calculated for the period of 28 weeks. The total CBY was found to be 175.40 m³ for 28 weeks. The specific biogas generation was achieved to be 243.37 ml/gVS, which was determined by dividing the daily average biogas yield with total VS (g) fed daily into the field AD digester. The biogas compositions produced in the field-scale AD plant were analysed with a GC. The compositions of methane, carbon dioxide and nitrogen were found to be in the range of 53–57 % (v/v), 39–42 % (v/v) and 4–5 % (v/v), respectively.

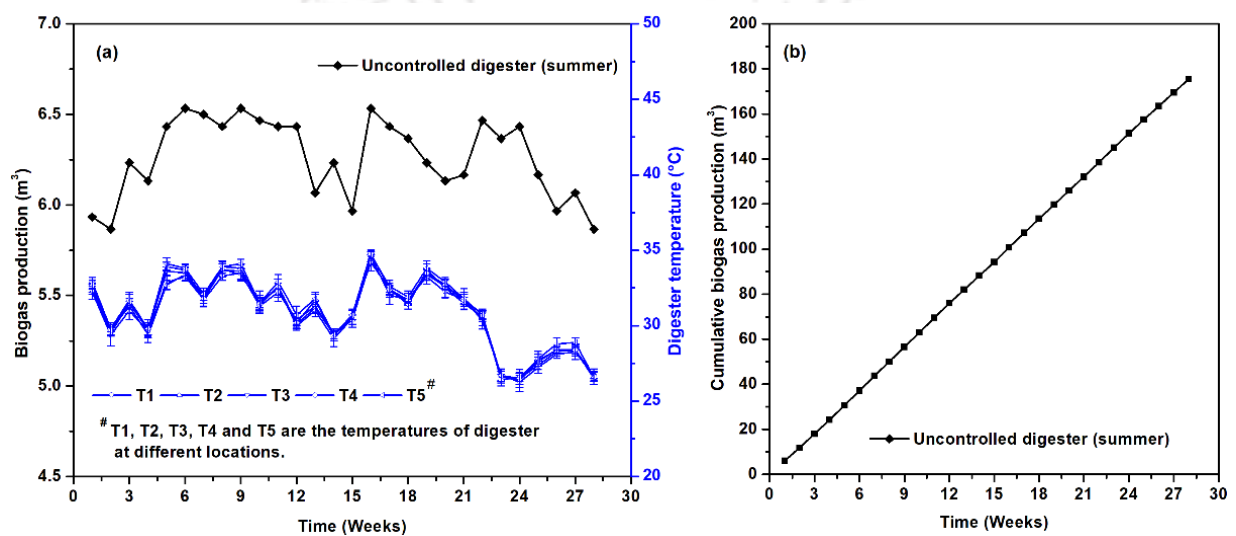


Fig. 5.2. (a) Weekly averages of biogas production and temperature (b) cumulative biogas production during the summer season for the field-scale AD plant.

The weekly and cumulative biogas generations during the winter season (November–February) were also recorded, analysed and compared for the period of 16 weeks without the SAH system and with SAH in the following year. The comparison of biogas production and temperature variations during the winter, both with and without the implementation of the SAH system, are illustrated in Fig. 5.3. The production of biogas under uncontrolled conditions was found to be decreasing as temperature decreased and then started increasing with the increase in ambient temperature. The lowest average biogas production was detected to be 5.50 m³ on the 12th week of investigation as the average temperature was found to be decreasing from

22±1°C to 20±1°C. The maximum biogas yield without the SAH system was found to be 6.07 m³ in the 2nd week at the beginning of the winter season. However, less fluctuations in biogas yield under controlled conditions was observed compared to uncontrolled condition, with an average biogas yield of 6.40 m³/week for 16 weeks of investigations against the feeding of 25 kg CD/day (S/W 1:1). The average specific biogas generations were 248.19 ml/gVS and 224.00 ml/gVS under the controlled and uncontrolled conditions respectively during the winter season, whereas, the specific biogas generation during the summer season (uncontrolled) was 243.37 ml/gVS. Moreover, the trends of biogas generation with SAH systems were found to be more stable compared to biogas generation without implementing the SAH system. The CBY of the controlled AD plant was found to be 9.93 % higher, with a peak value of 6.57 m³ on the 3rd day, compared to the uncontrolled AD plant during the winter. From the present investigations, it can be concluded the SAH system could be implemented to generate a higher amount of biogas in cold climate regions.

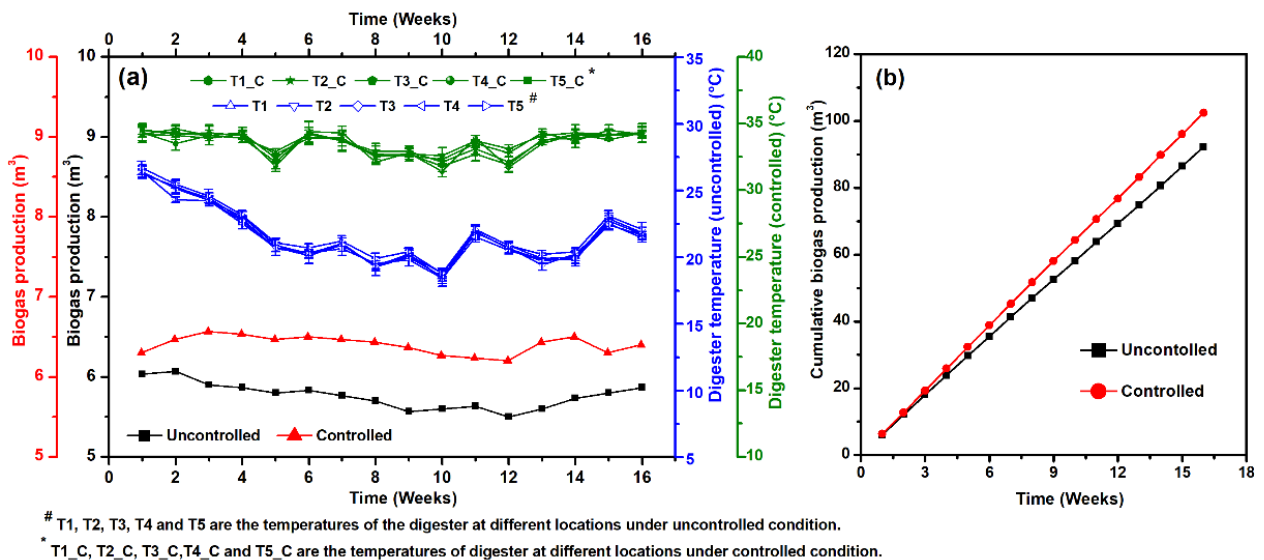


Fig. 5.3 (a) Weekly averages of biogas production and temperature (b) cumulative biogas production under uncontrolled and controlled conditions during the winter season for the field-scale AD plant.

5.2.4 Results of kinetic analysis

The kinetic analysis for field-scale experiments conducted during the winter seasons under controlled and uncontrolled conditions was also performed. The results of kinetic model parameters are summarised in Table 5.2. The Y_P value of the AD plant under controlled conditions (141.49 m^3) was found to be higher than the AD plant's digester when it was under uncontrolled conditions (127.59 m^3). The weekly biogas production rates were $6.29 \text{ m}^3/\text{week}$ and $7.08 \text{ m}^3/\text{week}$ for uncontrolled and controlled digesters. The R-squared value was around 0.99, indicating a well-simulated correlation between the predicted and actual experimental results.

Table 5.2. Kinetic analysis for field-scale AD plant

Digester	Y_P (m^3)	Y_R (m^3/week)	λ (week)	Y_C (m^3)	R^2
Uncontrolled	127.59	6.29	0.62	92.27	0.99
Controlled	141.49	7.08	0.76	102.43	0.99

5.2.5 Summary of field scale AD plant

Considering the S/W ratio of 1:1 as the optimum ratio, experiments were conducted in a field-scale AD plant. The weekly measured pH values range was detected to be 6.9–7.3 throughout the digestion period. The weekly average biogas yield of $6.26 \text{ m}^3/\text{week}$ was obtained for 28 weeks, with a maximum peak value of $6.53 \text{ m}^3/\text{week}$ during the summer season for a feeding rate of $50 \pm 1.50 \text{ kg CD slurry/day}$. The installation of the solar-assisted water system improves the biogas production rate by 9.93 % during the winter season compared to the digester without SAH conducted in the previous year. The study indicated that the SAH system can be used as an active heating source for on-field AD plants with promising potential for maximizing biogas production. Experimental investigations are recommended for future work, considering large-capacity AD plants and assessing economic feasibility so that installations of such plants can be integrated into rural development strategies that contribute to the overall progress and well-being of rural communities.

5.3 ECONOMIC ANALYSIS OF AD PLANT

The following sections deal with the financial analysis of a 1 m³ and 3 m³ capacity Deenbandhu model biogas plant for household cooking. The details of the factors considered and the analysis are discussed below.

5.3.1 Selection of biogas plant

The capacity of a biogas plant to be installed mainly depends on the number of persons to be served and the amount of cattle dung available. The amount of biogas (m³) generated was considered for one day. In this study, biogas required for cooking was considered 0.34–0.43 m³ (Singh and Sooch, 2004), whereas gas generation potential per kg CD was considered 0.04 m³. In the analysis, the amount of CD required per 1 m³ is 25 kg. Table 5.3 shows the amount of CD and the number of animals needed for a family for a 1 m³ and 3 m³ capacity biogas plant.

Table 5.3 Number of persons, animals, and quantity of dung (Singh and Sooch, 2004)

Sl. No.	Particulars	Biogas plant	
		1 m ³	3 m ³
1.	Number of persons required	2–3	7–9
2.	Quantity of cattle dung (kg) required	25	75
3.	Number of animals required	2–3	6–7

5.3.2 Cost of installation

Table 5.4 shows the cost of constructing and installing the 1 m³ and 3 m³ biogas plants. The current market prices of major construction materials in Guwahati, Assam were considered while conducting the cost analysis for civil works. The total installation cost was calculated considering the government subsidy of 30% of the net price. The installation cost was approximately Rs. 15155.00 and Rs. 26880.00 for 1m³ and 3m³, respectively.

Table 5.4 Cost of construction materials and installations (Civil Work)

Sl. No.	Significant components of civil works	Cost (Rs.) for biogas plant	
		1 m ³	3 m ³
1.	Brick	2500.00	8700.00
2.	Sand	1600.00	3200.00
3.	Stone	1150.00	2300.00
4.	Cement	2550.00	5500.00
5.	G.I. pipe, 2.5 cm	850.00	850.00
6.	Steel rod	1250.00	2100.00
7.	Plastic pipes	550.00	550.00
	Total construction cost (a)	10450.00	23200.00
8.	Labour cost	7500.00	11000.00
9.	Others cost	1000.00	1200.00
	Total cost (b)	8500.00	12200.00
10.	Supply line (c)	2700.00	3000.00
	Total cost (a + b + c)	21650.00	38400.00
11.	Subsidy (considering 30%)	6495.00	11520.00
	Total cost of installation	15155.00	26880.00

5.3.3 Operational cost

Considering annual depreciation on civil work, installations, cost of CD, and annual maintenance charges, the annual operation costs were calculated. The life span of the plant was taken for 15 years. Interest on the initial installation cost was not considered when calculating the biogas plant's annual operational cost as most people in rural India do not take financial assistance from the bank. Table 5.5 shows the cost of cattle dung in terms of liquid petroleum

gas (LPG). The calculations were carried out using Eq. 5.1 (Singh and Sooch, 2004). During the analysis, fresh CD and LPG cost Rs. 0.5/kg and Rs. 65/kg in Guwahati, India.

$$\text{LPG equivalent of CD} = \frac{\text{Weight of CD} \times 0.3 \times 0.43}{12.3} \quad (\text{Eq. 5.1})$$

where

The weight of dry CD cake is 30%,

1 m³ of biogas = 0.43 kg of LPG,

1 m³ of biogas = 12.3 kg of CD cake

Table 5.5 Cost of cattle dung in terms of LPG

Sl. No.	Particulars	Unit	Quantity/Price	
			1 m ³	3 m ³
1.	Annual CD requirement	kg	9125.00	27375.00
2.	Cost of CD	Rs.	4562.50	13687.50
3.	LPG equivalent to CD	kg	95.70	287.10
	Cost of CD in terms of LPG	Rs.	6220.58	18661.74

The cost of cattle dung in terms of LPG was determined to be Rs. 6220.58 and Rs.18661.74 per year for 1m³ and 3m³ capacity AD plants, respectively, while the amount of CD required per day was considered as 25 kg/day per m³ of AD plant. Further, annual depreciation rates of 4%, 5%, and 2% of the total installation cost were applied for construction works, the gas supply line, and maintenance charges, respectively. The amount of total annual operational cost was achieved by Rs. 5548.50 and Rs. Rs.15347.50/year for 1m³ and 3m³ capacity AD plant respectively. Table 5.6 shows the annual operational cost of the 1m³ and 3m³ capacity Deenbandhu biogas plant.

Table 5.6 Annual operational cost of biogas plant

Particulars	Annual depreciation rate	Cost (Rs.)	
		1 m ³	3 m ³
Construction works	4%	418.00	928.00
Gas supply line	5%	135.00	150.00
Maintenance charge	2%	433.00	768.00
Cost of CD		4562.50	13687.50
Total annual operational cost		5548.50	15347.50

5.3.4 Annual income

The digested slurry of the biogas plant can be used as fertiliser or making cow cake. Therefore, the cost of the digested slurry was considered for cost analysis while considering the cost of digested dry slurry as Rs.0.75/kg. As mentioned earlier, the dry-dried weight was considered 30% of the total residual. Further, 80% of the biogas generated was presumed to be used to calculate the annual income from the biogas as the analysis was considered for cooking applications in rural areas. The details of the calculation of yearly income are depicted in Table 5.7. For the calculation of LPG equivalent to biogas, the factor 0.43 was multiplied by the amount of biogas available for cooking, as 1 m³ of biogas was considered 0.43 kg of LPG. Total income was achieved to be Rs. 10214.53 and Rs. 30643.58 annually for 1m³ and 3m³ capacity AD plants.

Table 5.7. Calculation of annual income from biogas plant

Particulars	Units	Quantity/Cost	
		1 m ³	3 m ³
Annual biogas production	m ³	365.00	1095.00
Amount gas available for cooking	m ³	292.00	876.00
LPG equivalent of biogas	kg	125.56	376.68
Income from biogas #	Rs.	8161.40	24484.20
Quantity of digested slurry	kg	9125.00	27375.00
Quantity of digested after drying	kg	2737.5	8212.50
Income from dried slurry	Rs.	2053.13	6159.38
Total annual income	Rs.	10214.53	30643.58

Income from biogas = Cost of LPG X LPG equivalent of biogas

5.3.5 Payback period

The payback period is the years a project needs to recover its initial investment cost through annual net profit. Therefore, the payback period was determined by dividing the cost of installation of the biogas plant by the annual profit. In contrast, yearly profit is the difference between the annual income from the biogas plant and the annual operational cost of it. A short payback period is more desirable for economic activity. The annual profit from the 1m³ and 3m³ capacity biogas plant was obtained to be Rs. 4666.03 and Rs. 15110.08, respectively. The details of the results are depicted in Table 5.8. The payback for the present study was found to be 3.25 years and 1.78 years respectively for 1m³ and 3m³ capacity AD plant.

Table 5.8. Calculation payback period

Particulars	Unit	Amount/Year	
		1 m ³	3 m ³
a. Total cost of installation	Rs.	15155.00	26880.00
b. Total operational cost	Rs.	5548.50	15533.50
c. Total annual income	Rs.	8161.40	30643.58
d. Annual profit (c-b)	Rs.	4666.03	15110.08
e. Payback period (a/d)	Year	3.25	1.78

5.3.6 Summary of economic analysis

An economic analysis was performed for the 1m³ and 3m³ capacity biogas plants. The study was conducted for the application of cooking while considering the feed rate of 25 kg CD/day per 1 m³ capacity, maintaining the substrate-water ratio of 1:1. Further, 1m³ biogas was taken equivalent to 0.43 kg of LPG and 80% of the biogas produced was accounted for in calculating the annual income from the biogas plant. The yearly profit obtained amounted to around Rs. 4666.03 (equivalent to 44 USD) and Rs. 15110.08 (equivalent to 143 USD) for 1m³ and 3m³ capacity Deenbandhu model biogas plants, while spending an installation expenditure of Rs. 15155.00 (143 USD) and 26880.00 (about 253 USD) and taking into account the prices of fresh CD, digested dry CD, and LPG at Rs. 0.50/kg, Rs. 0.75/kg, and Rs. 65/kg, respectively. The biogas plant yielded a short payback period of 3.25 and 1.78 years for 1m³ and 3m³ capacity AD plants.

CHAPTER 6

PURIFICATION OF BIOGAS

This chapter comprises the development, analysis, and discussion related to the purification of raw biogas through novel biochar prepared from waste biomass, namely coconut husk, sugarcane bagasse, and water hyacinth, using the pyrolyser.

6.1 CHARACTERISATION OF BIOCHAR

6.1.1 Proximate and Ultimate Analysis

The proximate and ultimate analysis of the biochars are shown in Table 6.1. The volatile matter was found to be in the range of 26.2–15.5%, 33.5–19.6% and 23.7–22.1% for CH, WH and SB, respectively, when pyrolysis temperature increased from 350 to 550 °C, whereas the fixed carbon was 62.10–70.80%, 52.40–63.30% and 74.20–77.45%. The C content in all biochar increased with temperature. This is attributed to the release of hydroxyl functional groups and the evaporation of VM. Higher pyrolysis temperature caused thermal degradation reactions, dehydration, decarboxylation and aromatization, which enriched the C content in the resulting biochar. These results are further confirmed by the significant decline of the VM in biochar as the temperature rose from 350° to 550°C. The higher amount of VM at lower temperatures is attributed to incomplete carbonization, which can be further correlated to the high atomic ratios. The higher amount of VM in biochar obtained at low temperatures could be attributed to the functional groups of C=O and C–H, identified in the FTIR analysis illustrated in section 6.14. The amount of AC in the biochar increased with the rise in temperature. The increase was due to the mineralization of the mineral matter, which is formed into ash during carbonization. The AC in CH, WH and SB for carbonisation temperature of 350–550°C was 9.20–10.50 %, 10.40–13.60% and 0.89–1.16% respectively. The AC of biochar produced from CH and WH was relatively high, while SB had the lowest quantities. The difference in AC is attributed to the varying amounts of alkali and alkali earth metals in the feedstock, which essentially appear in ash. Hence, the biochar produced at higher

temperatures would be preferred for applications. The high H/C and O/C ratios indicate a converse degree of aromaticity and carbon fixation (Guo et al., 2022), which are favourable for CO₂ capture (Zhang et al., 2023). Therefore, CH 550, SB 550 and WH 550 have higher absorption capacity of CO₂ and H₂S, respectively.

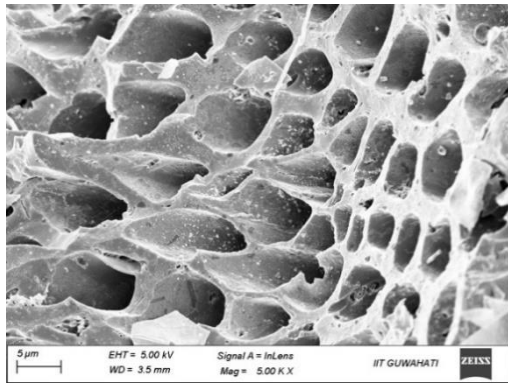
Table 6.1 Proximate and ultimate analysis of biochar

Analysis	CH 550	CH 450	CH 350	WH 550	WH 450	WH 350	SB 550	SB 450	SB 350
Proximate analysis (%)									
MC	3.28 ±0.06	3.07 ±0.10	2.46 ±0.21	3.81 ±0.42	3.73 ±0.12	3.65 ±0.10	2.03 ±0.33	1.81 ±0.21	1.18 ±0.29
AC	10.43± 0.23	10.11 ±0.16	9.23 ±0.18	13.60 ±0.18	12.3 ±0.15	10.42 ±0.30	1.16 ±0.11	1.09 ±0.10	0.89 ±0.05
VM	15.51± 0.18	21.05 ±0.11	26.17 ±0.14	19.61 ±0.29	28.41 ±0.10	33.47 ±0.20	19.39 ±0.22	22.14 ±0.20	23.72 ±0.12
FC	70.78± 0.07	65.77 ±0.21	62.14 ±0.15	63.28 ±0.07	55.55 ±0.17	52.46 ±0.30	77.42 ±0.09	74.96 ±0.15	74.21 ±0.20
Ultimate analysis (%)									
C	76.83± 0.19	71.45 ±0.33	68.37 ±0.17	40.32 ±0.70	40.12 ±0.13	39.13 ±0.30	74.72 ±0.10	67.21 ±0.12	61.49± 0.10
H	3.65 ±0.07	3.89 ±0.21	4.01 ±0.29	1.60 ±0.20	2.59 ±0.20	3.74 ±0.30	2.94 ±0.20	3.15 ±0.47	4.08± 0.30
N	1.16 ±0.12	1.23 ±0.10	1.31 ±0.09	3.30 ±0.20	3.41 ±0.12	4.18 ±0.20	0.83 ±0.15	0.89 ±0.11	0.92 ±0.40
S	0.23 ±0.09	0.19 ±0.05	0.09 ±0.05	0.14 ±0.08	0.11 ±0.06	0.10 ±0.09	0.12 ±0.07	0.11± 0.09	0.09 ±0.05
O	18.13	23.24	26.22	54.64	53.77	52.85	21.31	28.64	33.42

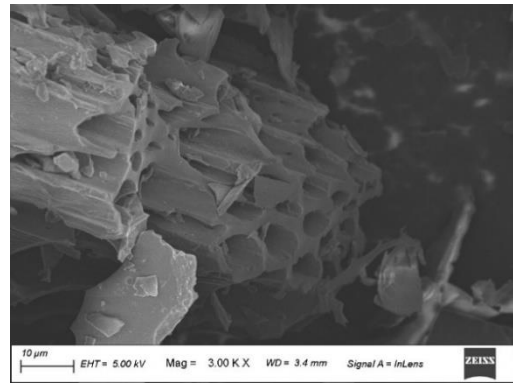
	±0.11	±0.14	±0.15	±0.40	±0.18	±0.20	±0.17	±0.23	±0.12
C/N	66.23	58.09	52.19	12.21	11.77	9.49	90.02	75.52	66.84
H/C	0.047	0.054	0.058	0.039	0.065	0.096	0.039	0.047	0.066
O/C	0.24	0.33	0.38	1.35	1.34	1.35	0.29	0.43	0.54
Others									
pH value	9.58	8.87	7.91	10.05	8.89	7.66	10.06	9.21	7.45
	±0.08	±0.09	±0.11	±0.08	±0.12	±0.05	±0.15	±0.36	±0.11

6.1.2 FESEM analysis

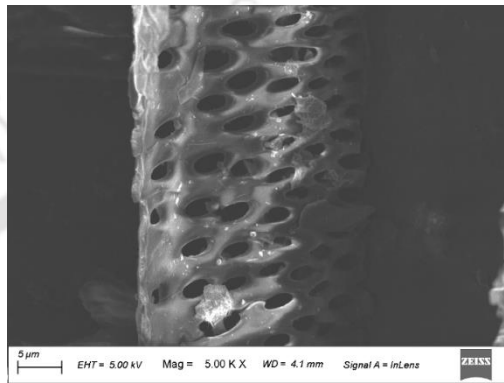
The visual inspection of FESEM microphotographs of different biochars illustrated the differences in microstructure among the biochars. The SEM images of all biochars are depicted in Figs. 6.1–6.3 demonstrate that their surfaces exhibit a rough, irregular texture characterised by dispersed micropores and mesopores. The porous structure developed in the biochar due to the thermal decomposition of cellulose and hemicellulose and the release of volatile matter at high temperatures. The SEM images of CH and SB biochars look like honeycomb structures, whereas the SEM images of WH show ununiform pores and irregular structures. The micropores were detected to increase with the increase in pyrolysis temperature. This phenomenon likely contributes to the increase in surface area with temperature, subsequently resulting in enhanced adsorption of CO₂ and H₂S. These pores serve as trap sites for CO₂ and H₂S molecules, where they undergo various physicochemical reactions (Sahota et al., 2018). The H₂S converts to S⁰ and S-related compounds due to the adsorption of H₂S molecules.



(a)

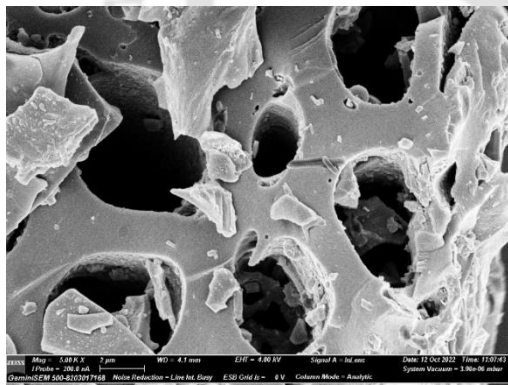


(b)



(c)

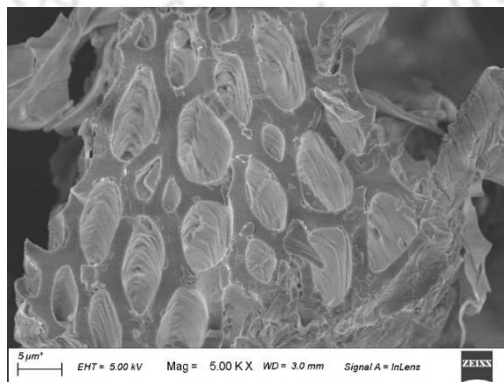
Fig. 6.1. FESEM microphotographs of biochar (a) CH 550 (b) CH 450 (c) CH 350



(a)



(b)



(c)

Fig. 6.2. FESEM microphotographs of biochar (a) SB 550 (b) SB 450 (c) SB 350

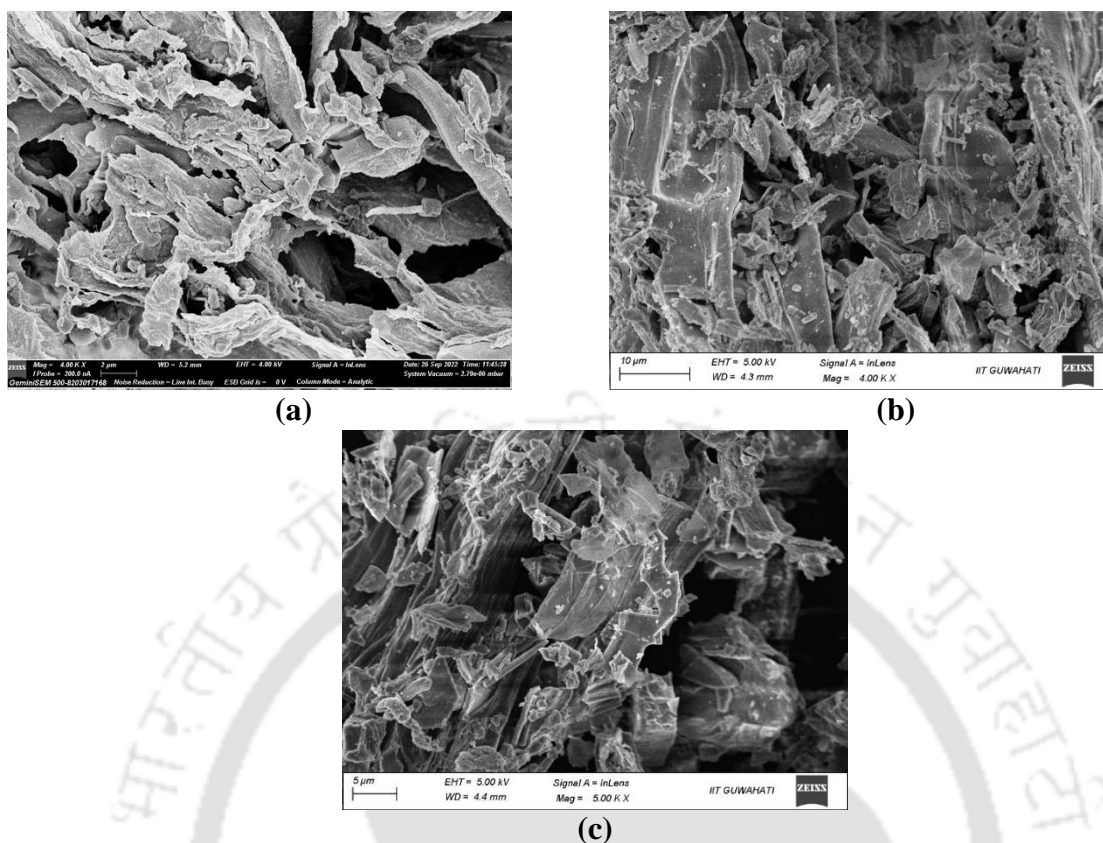


Fig. 6.3. FESEM microphotographs of biochar (a) SB 550 (b) SB 450 (c) SB 350

6.1.3 BET analysis

The results of the BET analysis of biochars are shown in Table 6.2. This analysis was carried out to identify the textural properties of the biochars. The CH, SB and WH biochars were produced at different pyrolysis temperatures of 550, 450 and 350 °C. Higher textural properties were detected at higher carbonization temperatures. These outcomes were attributed to eliminating volatile matter through dehydration, softening, melting and carbonization, resulting in high biochar pore volume and surface area. Researchers reported that the higher the BET surface area (S_{BET}) and pore volume, the higher the adsorption capacity (Sahota et al., 2018). The S_{BET} of the biochar at higher pyrolysis temperatures was found to be higher for the respective biochar produced at lower temperatures. The S_{BET} of CH 550, SB 550 and WH 550 were 50.025 m²/g, 17.780 m²/g and 14.000 m²/g, respectively, which are higher than CH 350 (14.466 m²/g), SB 350 (7.55 m²/g), and WH 350 (8.455 m²/g) respectively. Moreover, a higher

value of S_{BET} and pore volume was detected for CH than SB and WH biochar. The biochars may not be an adsorbent of CH_4 because of their pore sizes. CH_4 managed to escape without being adsorbed by the biochars, whereas the molecular-sized carbons in the range of 0.33 to 0.40 nm are capable of separating CH_4 from CO_2 (Sethupathi et al., 2017). However, H_2S and CO_2 have smaller molecular sizes than CH_4 . In the present investigation, all the biochar pores were more than 3 nm. Therefore, these biochars have the capability to absorb CO_2 and H_2S . The pore volume of CH 550 was $0.044 \text{ cm}^3/\text{g}$, which is higher than SB 550 ($0.042 \text{ cm}^3/\text{g}$) and WH 550 ($0.025 \text{ cm}^3/\text{g}$), respectively. Higher BET surface area and pore volume indicate a higher CO_2 and H_2S adsorption capacity for CH 550. The analysis concluded that raising the pyrolysis temperature enhances the aromaticity of biochar, consequently generating micro- and mesopores.

Table 6.2 BET properties of biochar

Biochar sample	CH 550	CH 450	CH 350	SB 550	SB 450	SB 350	WH 550	WH 450	WH 350
BET surface area (m^2/g)	50.025	24.978	14.466	17.780	13.323	7.55	14.000	11.044	8.455
Pore Volume (cm^3/g)	0.044	0.035	0.010	0.042	0.031	0.006	0.025	0.022	0.021
Pore size (nm)	3.328	3.311	3.057	12.862	9.637	3.791	11.923	7.651	3.317

6.1.4 FTIR analysis

The FTIR analysis was implemented to study the functional groups on biochar surfaces. Fig. 6.4 displays the FTIR spectra of CH, SB and WH biochars prepared at 550, 450 and 350 °C pyrolysis temperatures. In the present study, stretching hydroxyl (-OH) functional group

was found at $3800\text{--}3300\text{ cm}^{-1}$, stretching and vibration of C=C, C=O at $1640\text{--}1570\text{ cm}^{-1}$, bending of O-H at $1400\text{--}1200\text{ cm}^{-1}$, stretching C-O at $1200\text{--}1000$, C=C bending at ($900\text{--}690\text{ cm}^{-1}$) and the presence of C-H ($900\text{--}690\text{ cm}^{-1}$) in the aromatic ring, were also determined respectively. The wavelength and their respective explanation are depicted in Table 6.3. Ma et al. (2022) reported that the functional groups containing -O viz. -OH, C-O or C=O) over the biochar surfaces participates in the adsorbing H_2S . The strength of -OH, C-O and C=O functional groups over the biochar surface declines after adsorbing H_2S .

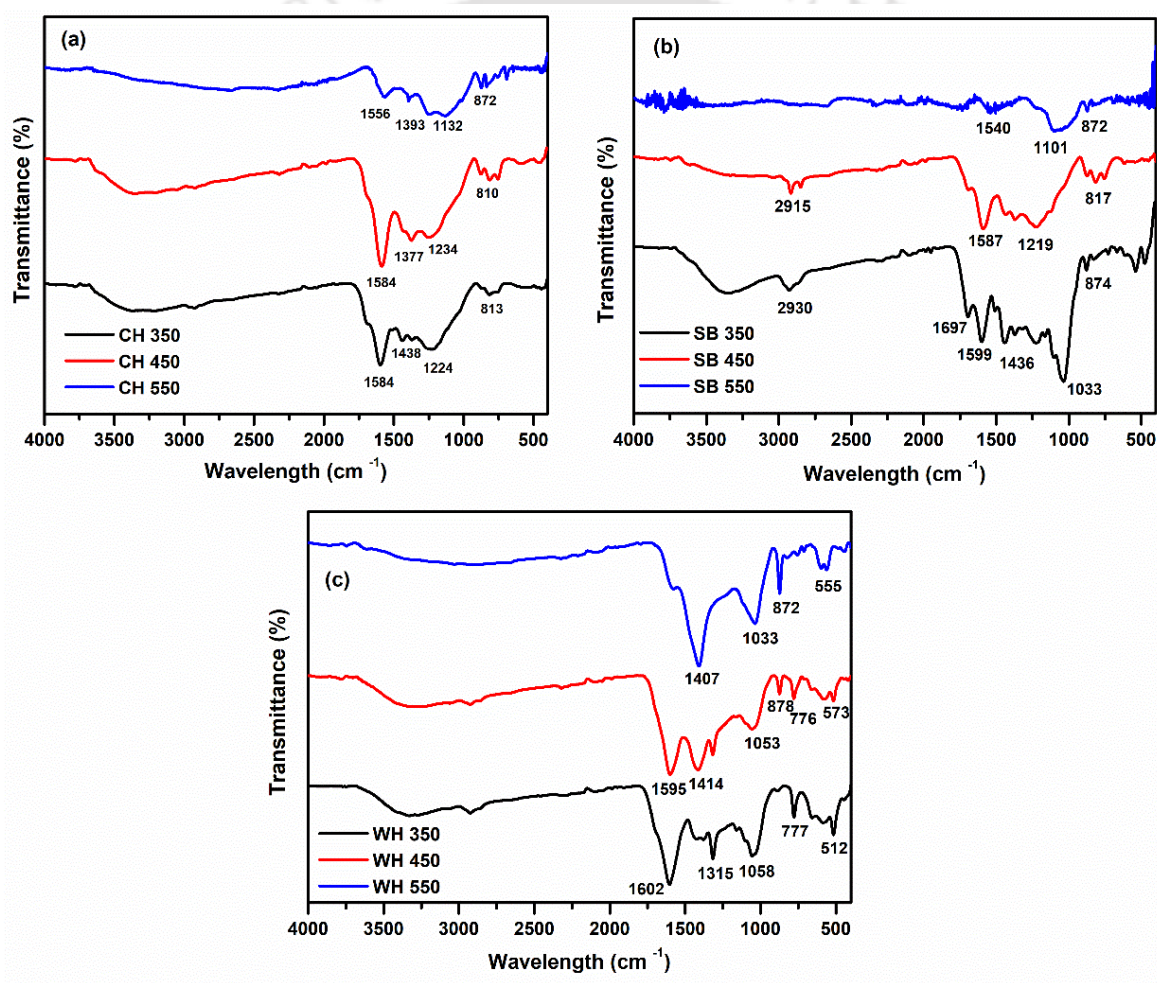


Fig. 6.4. FTIR spectra of biochar (a) CH, (b) SB, and (c) WH

Table 6.3 Visible wavelength and functional groups of the biochar

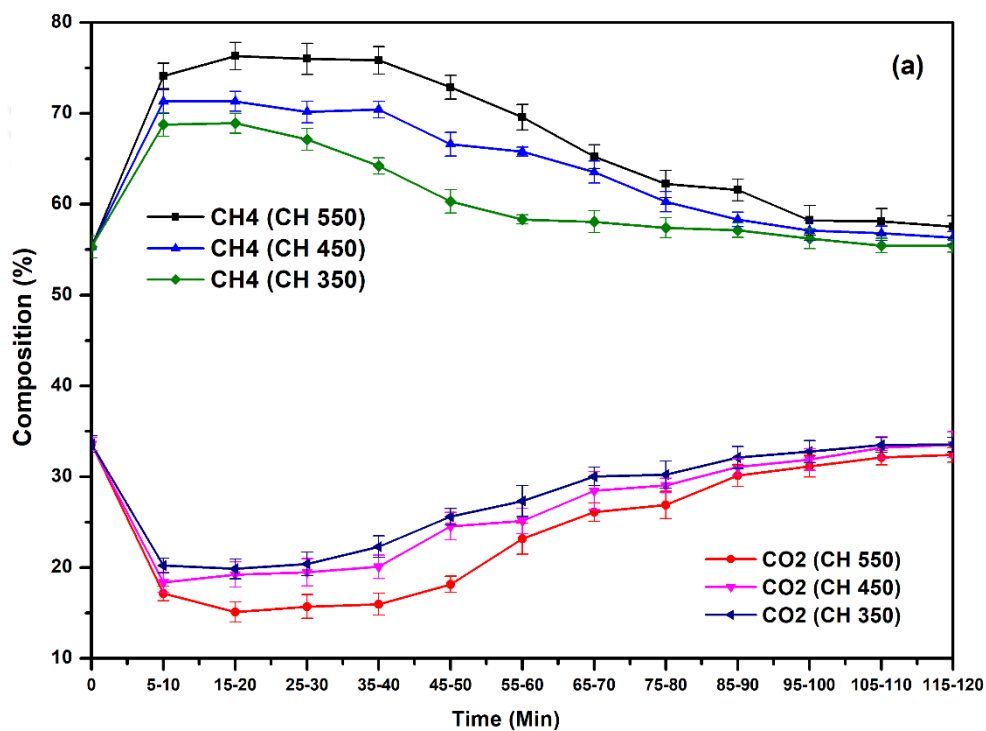
Wavelength (cm ⁻¹)	Wavelength from this study (cm ⁻¹)	Bond	Functional group	Reference
3800-3200	3800-3300	O-H stretching	Hydroxyl	(Bordoloi et al., 2022)
1700-1400	1640-1570	C=C stretching, C=O vibration	Alkene, aromatic carbonyl	(Shen et al., 2015) (Bordoloi et al., 2022)
1400-1300	1400-1330	O-H bending	Alcohol	(Shen et al., 2015)
1300-1200	1302-1250	O-H bending	Phenol	(Domingues et al., 2017)
1200-1000	1135-1030	C-O stretching	Alcohol	(Domingues et al., 2017)
900-690	956-690	C=C bending C-H bending	Alkene, pyridine, aromatic	(Bordoloi et al., 2022), (Shen et al., 2015)
	687-555	C-H vibration	Aliphatic, aromatic	(Shen et al., 2015)

6.2 OUTCOME OF BIOGAS PURIFICATION

6.2.1 Biogas purification using biochar

In this study, CH, SB, and WH biochars, pyrolysed at three different temperatures of 550, 450, and 350°C with a heating rate of 10 °C/min for the residence time of 1 h were used as adsorbent to remove CO₂, H₂S and other impurities from the biogas. Fig. 6.5 shows the initial composition and changes in CH₄ and CO₂ composition of biogas after treatment over different time ranges (0–120 minutes). In all the cases, it was found that CO₂ composition at the biogas outlet from the packed bed column reduced drastically within 5 minutes, whereas the composition of CH₄ was increased. The CO₂ composition remained almost constant for 40 minutes for biochars CH 550, SB 550, and WH 550, and then, it increased and attained its initial value after 100 minutes. On the other hand, CO₂ concentration was found to be increasing at 25 minutes and 10–15 minutes for the biochars prepared at 450 and 350 °C,

respectively. Therefore, higher CO₂ removal efficiency was detected for the biochars prepared at higher pyrolysis temperatures. The CO₂ removal efficiency of CH 550, SB 550 and WH 550 were 55.02%, 49.65% and 45.22%, respectively, whereas the lowest value was detected for CH350 (40.88%), SB 350 (37.90%), and WH 350 (36.41%) respectively. The higher CO₂ adsorption by CH 550, SB 550, and WH 550 might be due to the higher surface area than the biochars produced at lower temperatures. Hence, physisorption might be the principal mechanism of CO₂ adsorption on biochars. Further, CO₂ removal efficiency was detected higher for CH, followed by SB and WH. Moreover, micropore volume size is the most effective for CO₂ adsorption, and those pores were the highest in CH 550, SB 550 and WH 550, as detected by BET analysis. This analysis confirms that biochar can remove CO₂ and other biogas impurities.



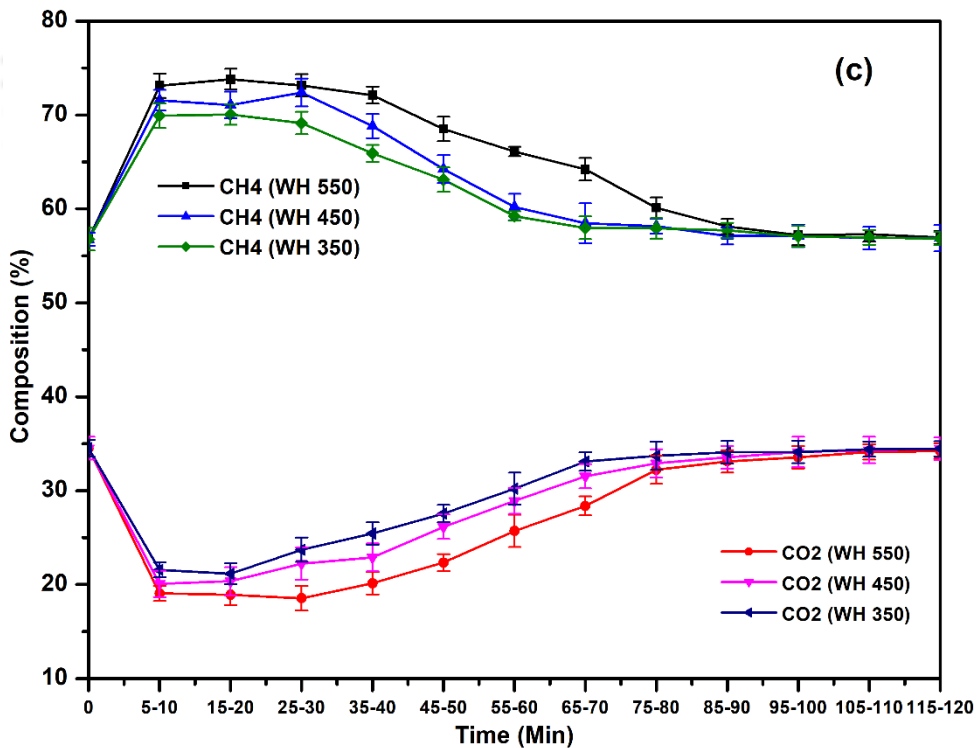
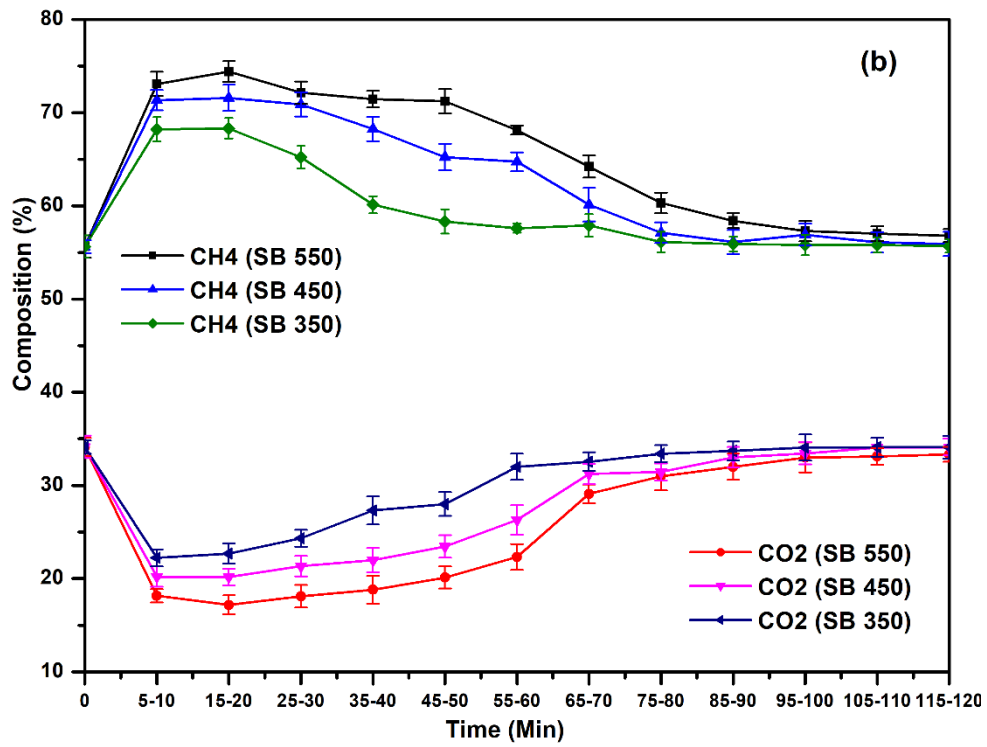


Fig. 6.5. CO₂ removal by biochars (a) CH, (b) SB, and (c) WH

6.2.2 EDX analysis

The results of EDX analyses for the biochars prepared from the CH, SB and WH at different carbonisation temperatures are shown in Table 6.4. The significant elements detected in biochars included C and O and small amounts of K, Ca, Mg, S, Si and Cl. The EDX analysis showed a higher amount of carbon content for CH 550 (85.3 wt%), SB 550 (89.4 wt%), WH 550 (71.0 wt%), which were prepared at higher pyrolysis temperatures (550 °C), followed by CH 450 (81.9 wt%), SB 450 (87.4 wt%), WH 450 (69.6 wt%) and CH 350 (75.6 wt%), WH 350 (77.2 wt%), WH 350 (64.2 wt%) prepared at lower pyrolysis temperatures (450 °C, 350 °C), respectively. On the other hand, O-content increased with the decrease in pyrolysis temperature. The decline in O-content with rising temperature can be attributed to the release of various oxygen functional groups as volatiles during pyrolysis, as illustrated by (Wu et al., 2016). The oxygen content ranged from 7 to 21.6 wt% in freshly prepared biochar samples, whereas S-content was around 0.0–0.6 wt%, which increased significantly after removing H₂S during biogas purification. The Ca, Mg and K on biochars provide the conditions for sulfate formation in the biogas purification process (Ma et al., 2022).

Table 6.4 EDX analysis of the biochars

Biochar	Composition of elements (wt.%)							
	C	O	K	Ca	Mg	S	Si	Cl
Coconut husk								
CH 550	85.3	7.3	3.8	0.5	0.2	0.2	0.3	2.1
CH 450	81.9	15.0	1.70	0.3	0.1	0.1	0.2	0.8
CH 350	75.6	20.4	2.4	0.1	0.1	0.0	0.1	1.3
Sugar cane baggage								
SB 550	89.4	7.7	1.7	0.3	0.2	0.1	0.2	0.4
SB 450	87.4	9.3	1.8	0.6	0.1	0.0	0.1	0.7
SB 350	77.2	20.7	0.6	0.3	0.6	0.0	0.6	0.1

Water hyacinth								
WH 550	71.0	8.8	7.9	3.7	0.8	0.0	0.4	7.0
WH 450	69.6	13.6	6.2	2.8	0.4	0.6	0.6	6.5
WH 350	64.2	21.6	5.3	3.9	1.1	0.0	0.0	3.7

6.2.3 XRD analysis

The XRD spectra of the biochars CH, SB and WH before and after H₂S sorption are shown in Figs.6.6–6.8. Freshly prepared biochars exhibited low-intensity peaks at small angles, primarily attributable to the amorphous phase present in the biochars. However, sharp peaks in the biochar after H₂S sorption suggest the formation of crystalline (inorganic) minerals. In the CH, SB, and WH biochar samples collected after H₂S sorption, strong peaks at 2-theta (2θ) = 23.08°, 27.76° were detected, indicating the formation of elemental S (S⁰). Sahota et al. (2018) reported that sulphate and S⁰ are produced on the surface and pores of the biochar, respectively, on exposure to trace amounts of O₂. The observed results revealed the likely formation of S⁰ (based on XRD analysis) in the pores. Following H₂S sorption, the biochars exhibit solely new diffraction peaks, signifying the presence of elemental sulphur and other sulphur-related compounds. The diffraction data of biochars after H₂S sorption also show strong peaks, which indicate the presence of various compounds like CaCO₃ ($2\theta=29.36^\circ, 43.26^\circ$), CaSO₄ ($2\theta=40.99^\circ, 56.03^\circ, 66.76^\circ$), KOH ($2\theta=26.66^\circ, 30.86^\circ, 50.36^\circ$), SO₂ ($2\theta=25.73^\circ$) and K₂SO₄ ($2\theta=48.93^\circ, 58.76^\circ$). From this analysis, it can be inferred that the prepared biochar is a good candidate for the absorption of H₂S, suggesting its potential utilization for removing H₂S from biogas.

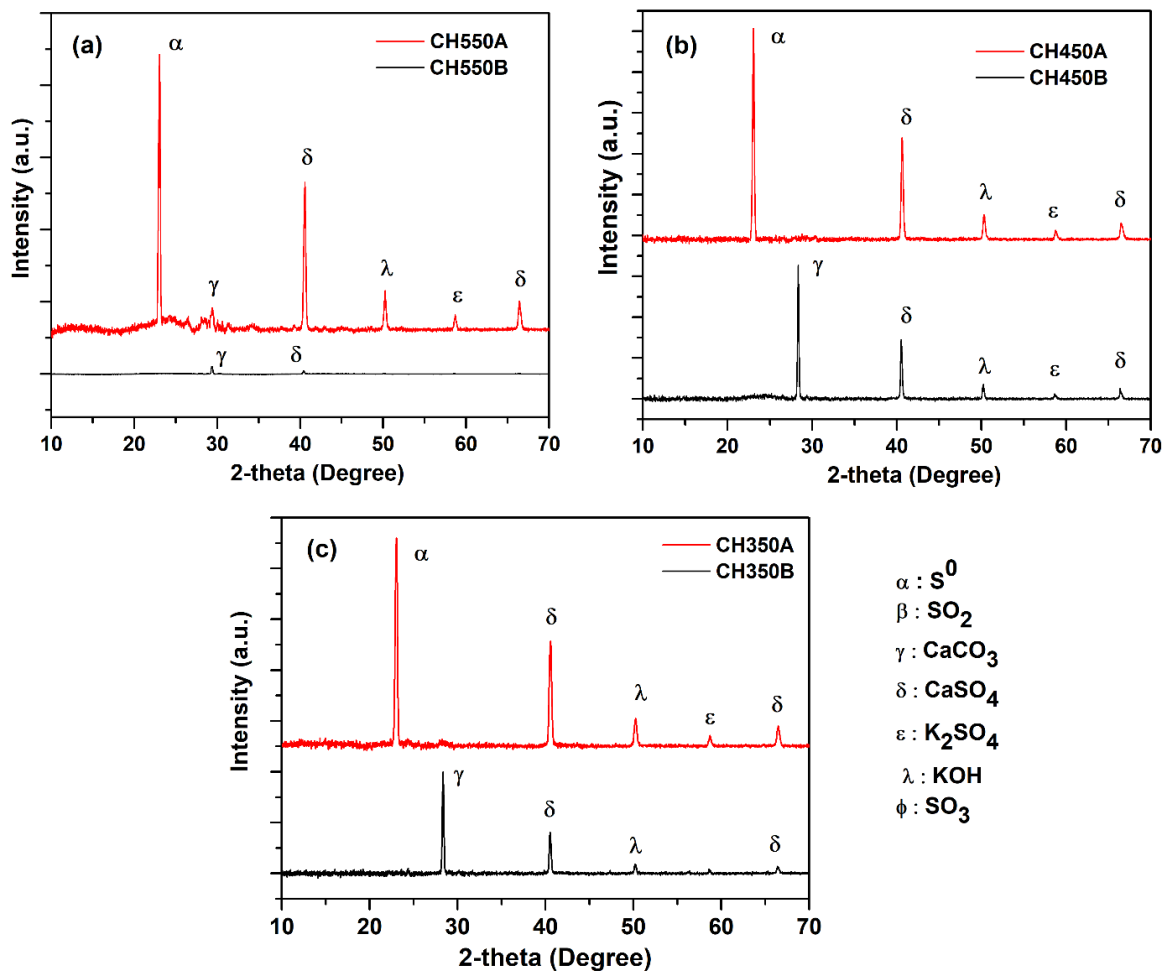


Fig.6.6. XRD patterns of biochar before and after H_2S sorption (a) CH 550 (b) CH 450 and (c) CH 350 where A stands for after, B stands for before.

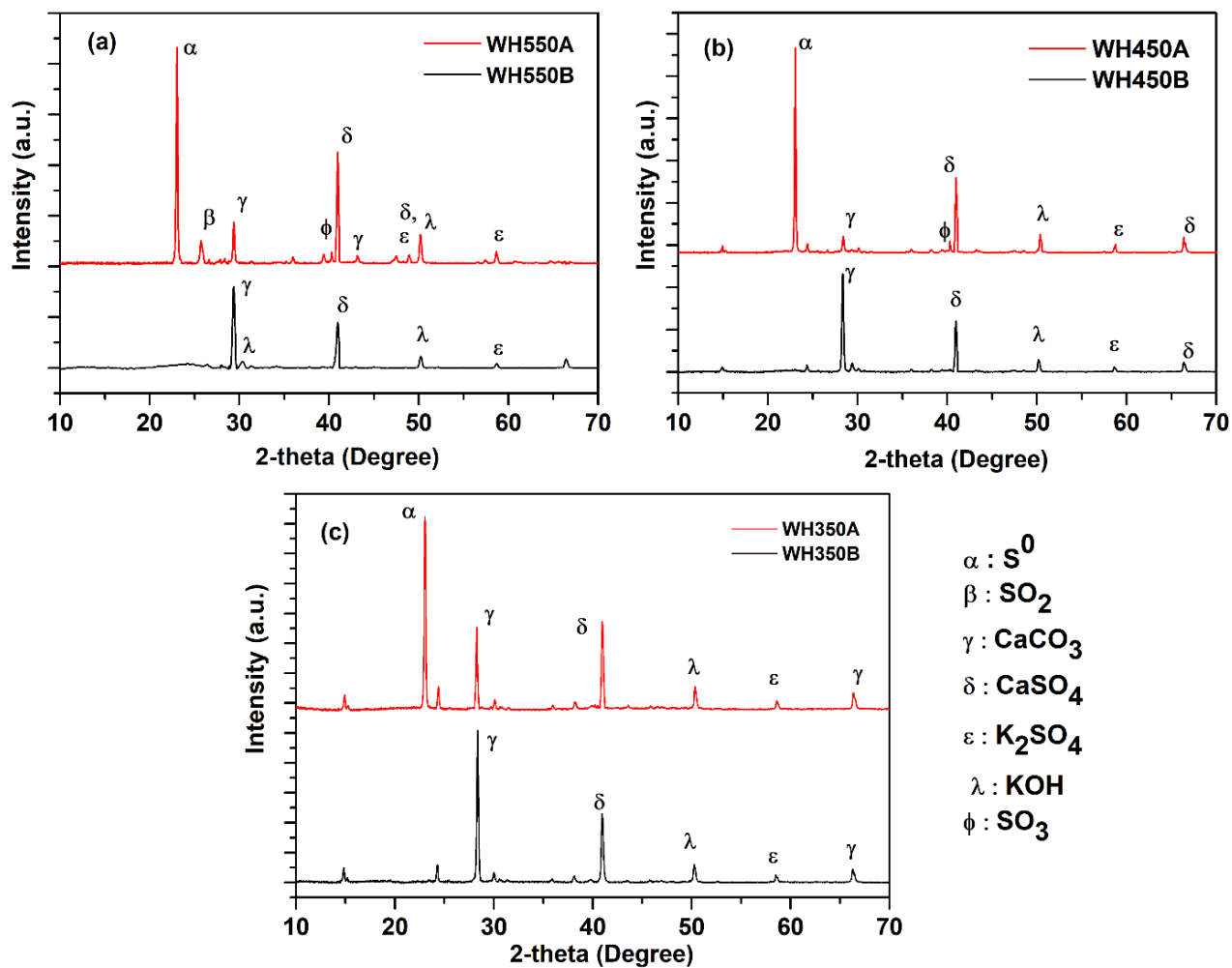


Fig.6.7. XRD patterns of biochar before and after H_2S sorption (a) WH 550 (b) WH 450 and (c) WH 350 where A stands for after, B stands for before.

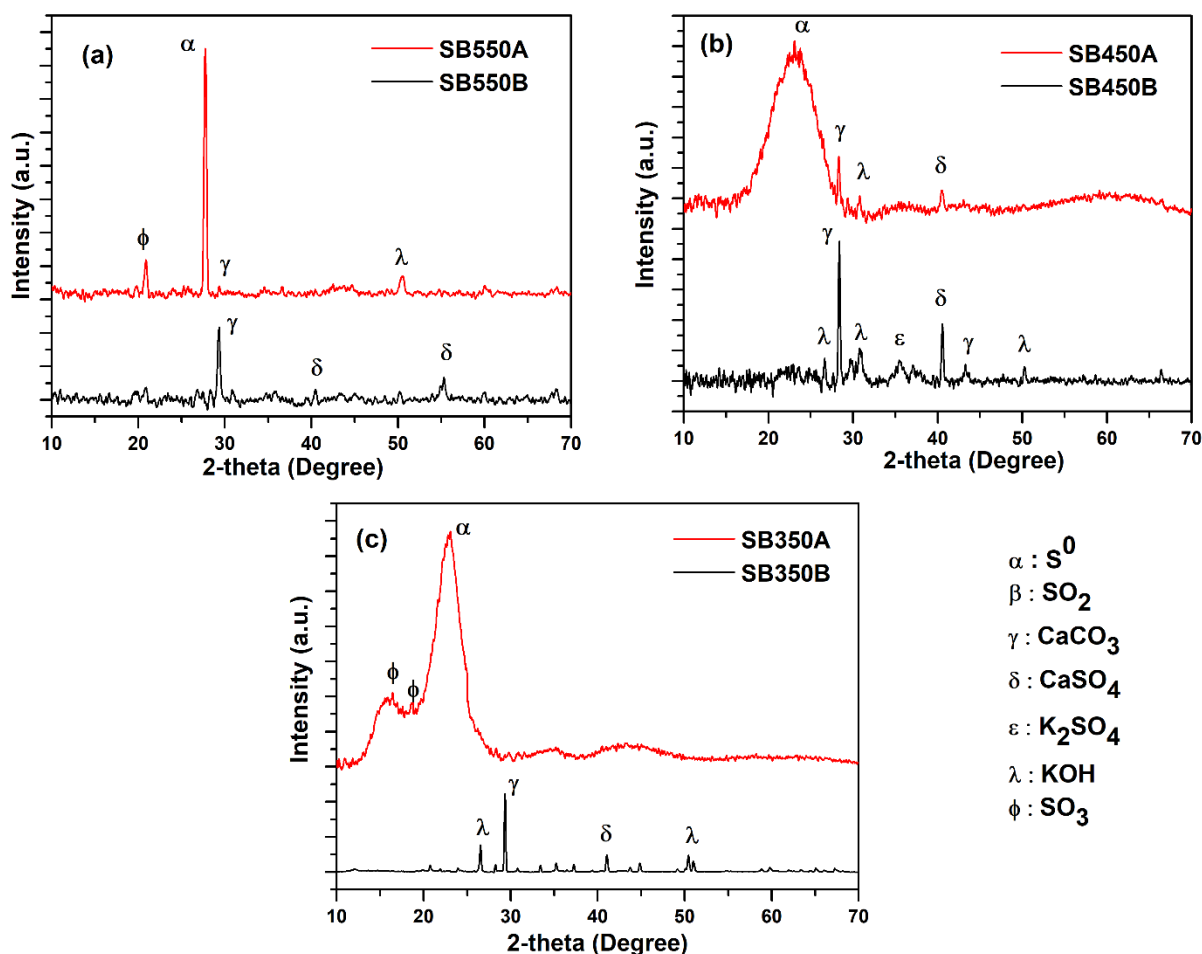


Fig.6.8. XRD patterns of biochar before and after H₂S sorption (a) SB 550 (b) SB 450 and (c) SB 350 where A stands for after, B stands for before.

6.2.4 Biogas purification using 1.5 N NaOH solution

The present study used 1.5 NaOH and biochars WH 550, SB 550 and CH 550 to remove CO₂, H₂S and other impurities from biogas. Fig.6.9 shows the initial composition and changes in CH₄ and CO₂ composition after treatment over different time ranges for all three types of biochars. In all the cases, it was found that CO₂ composition at the biogas outlet from the packed bed column reduced drastically within 5 minutes, whereas the composition of CH₄ was increased. This behaviour indicates that CO₂ and H₂S adsorption is favoured in 1.5 NaOH solution and biochar, respectively, and hence, a significant reduction of CO₂ and H₂S composition in outlet gas can be achieved quickly. The CO₂ composition remained

significantly low (0%) within 5 min and remained constant for the next 90 min. The CO₂ composition at the biogas outlet after passing through 1.5 N NaOH was found to be constant (0%) due to the absorption of CO₂ by 1.5 N NaOH solution forming carbonate compounds. In contrast, the composition of CH₄ was increased within 10 minutes, remained almost steady up to 40 minutes, and then started decreasing. The decrease in CH₄ composition might be due to a reduction in the sorption capacity of biochar for CO₂ and H₂S. The highest percentage of CH₄ composition was found to be 79.5%, 81.6% and 83.5% for WH 550, SB 550 and CH 550 at 20 minutes, respectively. This test confirms that CO₂ absorption capacity of coconut husk biochar higher is higher than water hyacinth and sugar cane baggage biochars.

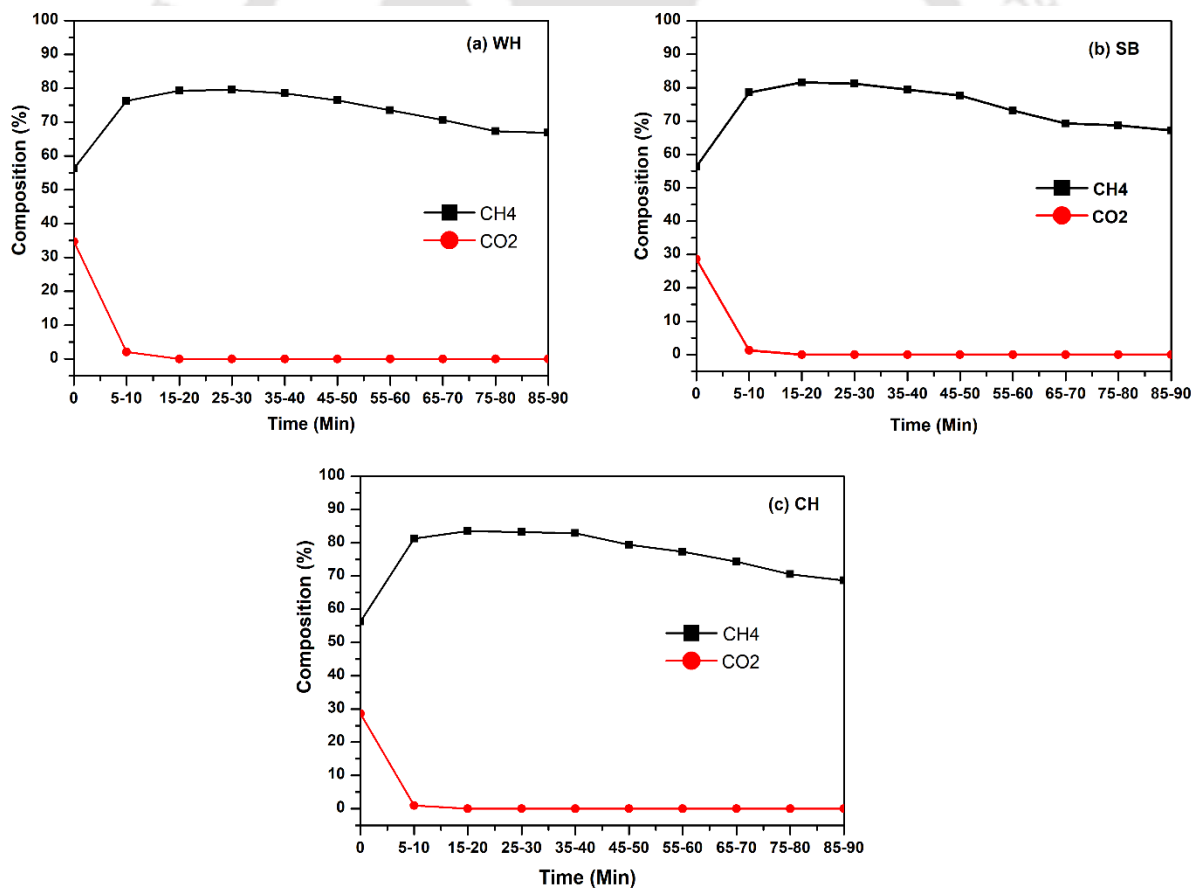


Fig. 6.9. CO₂ removal by biochars (a) WH 550 (b) SB 550 (c) CH 550

6.3 SUMMARY OF BIOGAS PURIFICATION

In this study, coconut husk, sugar cane bagasse and water hyacinth biochars, pyrolysed at three different temperatures of 550, 450, and 350°C with a heating rate of 10 °C/min for the residence time of 1 h were used as adsorbent to remove CO₂ and H₂S and other impurities from the biogas. A higher amount of CO₂ removal was achieved for the biochars prepared at higher pyrolysis temperatures due to the higher surface area than those produced at lower temperatures. The CO₂ removal efficiency of CH 550, SB 550 and WH 550 were 55.02%, 49.65% and 45.22%, respectively, whereas the lowest value was detected for CH350 (40.88%), SB 350 (37.90%), and WH 350 (36.41%) respectively. Further, evidence of H₂S removal was found in the analysis of XRD and EDX for saturated biochar. This was attributed to higher pH, which led to an increased rate of dissociation of H₂S and higher elemental sulphur conversion rates. Amongst several factors affecting CO₂ and H₂S adsorption, the surface area played an important role in adsorption along with the associated factors like higher alkaline pH, pyrolysis temperature and mineral elements on the biochar surface. In the next chapter, the conclusions and future works of the thesis are summarised.

CHAPTER 7

CONCLUSIONS AND RECOMMENDATION

This chapter deals with the overall conclusion attained after accomplishing all the objectives of the research work.

7.1 CONCLUSIONS

The initial characterisation of cattle dung and food waste demonstrates itself as a potential feedstock for biogas production through anaerobic digestion as it has a good amount of moisture content and is a highly volatile solid. Anaerobic digestion of cattle dung (CD), cooked kitchen waste (CKW), and vegetable waste (VW) was performed to identify the ideal substrate-water (S/W) mixing ratio at different S/W mixing ratios for each substrate under controlled ($35\pm 2^\circ\text{C}$) and uncontrolled temperatures. The optimum TS and VS contents observed for an S/W 50:50 ratio favoured the AD process and delivered maximum cumulative biogas for CD and CKW. The ideal S/W ratio was achieved at 50:50 for CD and CKW, whereas it was 60:40 for vegetable waste. Further, controlled digesters ($35\pm 2^\circ\text{C}$) exhibited higher biogas generation rates than uncontrolled digesters (ambient temperature). Considering the ideal S/W ratio of 50:50, anaerobic co-digestions (AcoD) of VW and CKW with CD were also studied by varying the co-substrate mixing ratios (SMR) of 70:30, 60:40, 50:50, 40:60 and 30:70 without and with biochar (BC) addition under controlled and uncontrolled temperatures. In the study of AcoD of CD and VW, the CMY of the BC-added digesters with SMR of 70:30, SMR 60:40, SMR 50:50, SMR 40:60, SMR 30:70 was found to be 26.53, 30.04, 22.62, 20.95, 18.91 % higher volume than those respective digesters without BC respectively, conducted at ambient temperature. Further, the mesophilic digesters with BC added generated 36.42, 39.26, 33.47, 32.90 and 31.47% higher volumes of CMY than the respective uncontrolled digesters without BC, respectively, for the HRT of 45 days. Likewise, in the study of AcoD of CD and

CKW, the digesters with BC added generated 18.07, 22.06, 21.68, 20.85 and 17.91% higher volumes of CMY than the digesters without BC, respectively, in the case of ambient temperature, whereas, 23.06, 29.27, 31.50, 32.47 and 28.13% higher amount of CMY were achieved for the mesophilic digesters compared to uncontrolled without BC digesters. This investigation concludes that maintaining the reactor temperature at mesophilic conditions can increase CH₄ generation. This study also confirms that BC can improve the AD process. Therefore, it can be inferred that the BC plays a vital role in the AD process by providing a surface area for microbial colonisation and serving as an adsorbent for compounds. Also, the alkaline properties of BC enhance CH₄ content through a reaction of CO₂ and H₂S with alkaline elements present in ash, thereby improving the biogas quality. The optimum SMR was obtained at SMR 60:40 for the AcoD of CD and VW but at SMR 40:60 for CD and CKW, as evaluated by the biochemical methane potential (BMP) tests. Considering the ideal SMR, another set of BMP tests was carried out at ambient temperature by adding varying amounts of biochar: 5g/L, 10 g/L, 15 g/L, 20 g/L, and 25 g/L to determine the appropriate quantity of BC to be added. The digester containing 15 g/L biochar exhibited the highest CMY for both cases, confirming that 15 g/L biochar addition can be recognised as the most effective for AcoD of cattle dung and food waste.

Further, a field-scale experiment was performed in a 1 m³ capacity anaerobic digestion plant at three phases, considering the ideal S/W 50:50 ratio detected in the lab scale study. The experiment's first phase was carried out in the summer season without using a solar energy-assisted water heating (SAH) system. Subsequent phases were conducted both without and with the use of a solar energy-assisted water heating system during the winter. The weekly measured pH values range was detected to be 6.9–7.3 throughout the digestion period. The weekly average biogas yield of 6.26 m³/week was obtained for 28 weeks, with a maximum peak value of 6.53 m³/week during the summer season for a feeding rate of 50±1.50 kg CD

slurry/day. The solar energy-assisted water system improves the biogas production rate by 9.93% during winter compared to the digester without SAH conducted in the previous year. The study indicated that the SAH system can be used as an active heating source for on-field AD plants with promising potential for maximizing biogas production. Experimental investigations are recommended for future work, considering large-capacity AD plants and assessing economic feasibility so that installations of such plants can be integrated into rural development strategies that contribute to the overall progress and well-being of rural communities.

The purification of biogas is needed for applications in various areas like cooking, running an engine, etc., to obtain high-quality gas fuel. In this study, a purification system was developed by incorporating an adsorbent column. Three different biochars originated from coconut husk, sugar cane bagasse and water hyacinth prepared at three different pyrolysis temperatures of 550, 450, and 350°C were used as adsorbent to remove CO₂ and H₂S and other impurities from the biogas. The CO₂ removal efficiency of CH 550, SB 550 and WH 550 were 55.02%, 49.65% and 45.22%, respectively, whereas the lowest value was detected for CH350 (40.88%), SB 350 (37.90%), and WH 350 (36.41%) respectively. The BC prepared at higher carbonisation temperature was found to have higher adsorption capacity for CO₂ and H₂S. The high H/C and O/C ratios of BC at high temperatures decreased, indicating a converse degree of aromaticity and carbon fixation, which are favourable for CO₂ capture. Higher BET surface area and pore volume were also found at high pyrolysis temperatures, indicating a higher CO₂ and H₂S adsorption capacity of CO₂ and H₂S, respectively. Moreover, the Ca, Mg and K on biochars provide the conditions for sulfate formation in the biogas purification process.

The present findings will be very much helpful for the effective utilization of wastes for clean and high-density fuel for cooking and decentralised power generation, which finally leads to the good health of the people and sustainability.

7.2 SCOPE FOR FUTURE WORK

The following work packages may be considered for the scope of future research

- Utilisation of food waste for large-scale biogas production, along with the integration of a biogas purification system and bottling facility for Bio-CNG generation and production of bio-fertilizer from the digested slurry.
- Studies on the reduction of hydraulic retention time of anaerobic digestion by enhancing microbial activities with suitable additives.
- Development of a control strategy and passive compression system for controlled consumption of biogas generation from an anaerobic digestion system.



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LIST OF PUBLICATIONS

Journal publications

1. **Basumatary, S.**, Das, S., Kalita, P., Goswami, P., 2021. Effect of feedstock/water ratio on anaerobic digestion of cattle dung and vegetable waste under mesophilic and thermophilic conditions. *Bioresource Technology Reports* 14.
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2. **Basumatary, S.**, Das, S., Goswami, P., Kalita, P., 2023. Investigation of the effect of slurry mixing ratio and temperature on biogas production from cattle dung in a field-scale anaerobic digestion plant. *Int. J. Ambient Energy* 45.
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3. **Basumatary, S.**, Goswami, P., Kalita, P., 2024. Impact of coconut husk biochar on methane production rate in batch type anaerobic digester fed with cattle dung and cooked kitchen waste. *Biomass and Bioenergy* 187.
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4. **Basumatary, S.**, Muigai, H.H., Goswami, P., Kalita, P., 2024. Enhancement of biomethane yield rate in anaerobic co-digestion of cattle dung and untreated vegetable waste through the amendment of water-hyacinth biochar. *Bioresource Technology Reports*. (Revision)
5. **Basumatary, S.**, Goswami, P., Kalita, P. Biogas purification employing adsorption on coconut husk, sugarcane baggage and water hyacinth-based biochar (Under review)

Conference publications

1. **Basumatary S.**, Goswami P., Kalita P., 2018. Food waste utilisation for anaerobic digestion: A sustainable energy conversion technique, In: National conference on Waste to Energy, National Institute of Technology Mizoram, India
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