# ENHANCEMENT OF METHANE PRODUCTION FROM ANAEROBIC DIGESTION OF FOOD WASTE BY SPENT COFFEE GROUND DERIVED BIOCHAR

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Bachelor of Engineering Technology (Energy & Environmental)

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Bachelor of Engineering Technology (Energy & Environmental)

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# ABSTRAK

Biogas adalah sumber tenaga moden dan sesuai untuk keperluan masa hadapan dengan penerapan teknologi pencernaan yang tepat. Namun, teknologi pencernaan anaerobik (AD) masih menghadapi beberapa halangan termasuk produktiviti metana yang rendah, kecekapan operasi yang tidak stabil dan degradasi bahan refraktori yang tidak diingini. Oleh karena itu, untuk mengatasi kekurangan ini, biochar telah diakui sebagai alternatif tambahan yang menjanjikan dalam proses AD untuk meningkatkan produksi metana. Selain itu, biochar yang diperbuat dari biojisim merupakan sumber karbon yang sangat kaya yang dihasilkan dari biojisim yang memanfaatkan pembakaran terma. Kajian ini bertujuan untuk mencirikan biochar sebagai bahan tambahan dalam AD sisa makanan daripada biochar sisa kopi dan menganalisis lebih lanjut prestasinya dalam AD. Untuk meningkatkan kecekapan AD, keadaan optimum untuk dos biochar, jumlah bahan baku dan nilai pH dipelajari. Hasil dapatan eksperimen menunjukkan bahwa 13.528 ml/g hasil biogas dicapai pada keadaan optimum iaitu 500g bahan baku, 7g dos biochar dan nilai pH 7, dengan durasi proses selama 14 hari. Selain itu, analisis mencirikan sisa kopi biochar menunjukkan kandungan karbon meningkat dari 54.52% menjadi 87.76%, luas permukaan BET meningkat dari 9.2m$^2$/g menjadi 15.3 m$^2$/g dan mengandung fungsi OH, C-H, C-C dan C-O yang meningkatkan produksi biogas dalam AD. Dengan demikian, biochar SCG dapat meningkatkan produksi metana dan penghasilan biogas dalam AD sisa makanan.

# ABSTRACT

Biogas is a modern energy source and is suitable for necessities of the future with the appropriate application of digestion technology. However, anaerobic digestion (AD) technology still faces some challenges including low methane productivity, instable operation efficiency and undesired refractory substances degradation. Therefore, to overcome this shortcoming, biochar has been recognized as a promising alternative addition in AD process to enhance methane production. Furthermore, biochar derived from biomass is an exceptionally rich wellspring of carbon produced from biomass utilizing thermal combustion. This study aims to characterize biochar as an additive in AD of food waste from spent coffee ground (SCG) biochar and further analyze its performance in AD. To promote the efficiency of the AD, the optimum conditions for biochar dosage, the amount of feedstock and pH value were studied. The experimental findings revealed that 13.528ml/g of biogas yield was achieved under optimum conditions which were 500g of feedstock, 7g of biochar and pH 7, process duration of 14 days. Furthermore, the characterization analysis of SCG biochar showed carbon content was increasing from 54.52% to 87.76%, BET surface area was increasing from 9.2m$^2$/g to 15.3m$^2$/g and contains the -OH, C-H, C-C and C-O functions which enhanced the biogas production in AD. Thus, SCG biochar has potential to enhance methane production and biogas yield in AD of food waste.

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| | |
|---|---|
| & | And |
| Å | Angstrom |
| $cm^3/g$ | Cubic centimetres per gram |
| $m^3$ | Cubic meter |
| $m^3/g$ | Cubic metre per gram |
| °C | Degree Celsius |
| g | Gram |
| h | Hours |
| keV | Kilo electron volt |
| < | Less than |
| ml | Millilitres |
| ml/g | Millilitres per gram |
| mm | Millimetres |
| > | More than |
| nm | Nanometres |
| ppm | Parts per millions |
| % | Percentage |
| $cm^{-1}$ | Reciprocal centimetres |
| s | Seconds |
| $m^2/g$ | Square metre per gram |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| AD | Anaerobic Digestion |
| BET | Brunauer-Emmet-Teller |
| $CH_4$ | Methane |
| $CO_2$ | Carbon Dioxide |
| EDX | Energy Dispersive X-ray |
| FAO | Food and Agricultural Organization |
| FTIR | Fourier Transform Infrared |
| GHGs | Greenhouse Gases |
| $H_2$ | Hydrogen |
| $H_2O$ | Water |
| $H_2S$ | Hydrogen Sulphide |
| $N_2$ | Nitrogen |
| $NH_3$ | Ammonia |
| $O_2$ | Oxygen |
| SCG | Spent Coffee Ground |
| SEM | Scanning Electron Microscopy |
| VFAs | Volatile Fatty Acids |

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# CHAPTER 1

# INTRODUCTION

## 1.1 Background of Study

In this day and age, globalization has accelerated in recent years and has taken hold in a number of countries. As developed countries become more increase, the global energy demand is expected to rise by 48% in the next 20 years due to the increased in industrial activity and technological advancements (Moodley, 2021). Fossil fuel energy sources such as coal, natural gas, and oil is an important sources of energy production. Fossil fuel have been powering economies for over 150 years, and currently it supplies about 80% for the world's energy. However, due to the high energy demand, the use of fossil fuels leads to environmental emissions of greenhouse gases (GHGs) and pollutants (Asdrubali & Desideri, 2019).

On the other hand, increased food waste is seen as a major problem that might have negative consequences for the environment if it is not adequately treated. According to data from the Food and Agriculture Organization (FAO), nearly 1.3 billion tons of food waste are produced every year worldwide, which has not been effectively treated. Poor food waste management will have a negative influence not only on the environment, but also on the urban growth in developing countries. As a result, effective food waste treatment techniques must be established in order to overcome this type of waste by transforming it into another significant product. Food waste is the best candidate to convert to energy sources such as biogas. Thus, biogas can be regarded as a potential solution to address all of the issues about fossil fuels and the food waste problem as it can curb the GHGs by capturing these harmful gases and using it as fuel.

Biogas is an alternative to fossil fuels as it is less hazardous and naturally abundant and can be produced from organic waste decomposition (Anaya Menacho et al., 2022). Biogas consists of 45–60% methane ($CH_4$), 25–45% carbon dioxide ($CO_2$), 2–7% water ($H_2O$) at 20–40°C, 2–5% nitrogen ($N_2$), 0–2% oxygen ($O_2$), and less than 1% hydrogen ($H_2$), 0–1% ammonia ($NH_3$), and 0–6000 ppm hydrogen sulphide ($H_2S$) (Teymoori Hamzehkolaei &

Amjady, 2018). Biogas from waste and residues can play a critical role in the energy future. It is a multilateral renewable energy source that can replace conventional fuels to produce heat and power and also can be used as gaseous fuel in automotive applications. According to recent studies, biogas produced through anaerobic digestion (AD) has substantial advantages over other kinds of bioenergy because AD is a cost-effective and ecologically beneficial method.

AD is a process of metabolic degradation and stabilisation of organic materials under anaerobic conditions, such as hydrolysis, acidogenesis, and methanogenesis, that results in the creation of biogas. The conversion of organic waste to biogas takes place in phases, each involving interactions between different functional groups of bacteria. Therefore, a delicate balance between the microbes and environmental conditions is important for the stability and efficiency of AD systems. However, long-term operated AD can face a number of problems that may lead to anaerobic system failure (Zhang et al., 2018) such as decreases in biogas production. Therefore, adding biochar to AD systems enhances methane production and has proven to reduce the lag phase.

Biochar has a lot of advantages. It is environment-friendly, inexpensive and it can be produced from every source of biomass. In AD, biochar addition could mitigate toxins inhibitory, shorten methanogenic lag phase, immobilize functional microbes, and accelerate electron transferring between methanogenic and acetogenic microbes during AD process (Zhao et al., 2021). The starting biomass are the key factors for the composition of biochar. In this study, spent coffee ground (SCG) is used as sources of biochar. SCG is a material which is readily disposed but proves to be useful if properly treated (Andrade et al., 2020).

## 1.2 Problem Statement

Rapid urbanization and industrialization result in massive solid waste production. Food waste is one of the largest components of municipal solid waste in Asian countries, which is anticipated to reach 4.3 billion tonnes by 2025. (Arumdani et al., 2021). Generally, food waste has a high organic matter content and biodegradability, making it a good substrate for methane production during anaerobic digestion (AD). However, the characteristics results in the existence of inhibitory chemicals such as ammonia and volatile fatty acids (VFAs) which would results in low methane yield and instability of the AD system (Maurus et al., 2021). To counter these drawbacks, the additions of additives in the AD such as biochar can improve the conversion efficiency of substrates as well as enhancing its stability and methanogenesis rate. Nevertheless, the effects of biochar addition on AD could be due to the differences in biochar properties caused by a variety of factors such as feedstocks characteristics and operating conditions during biochar preparation. In reference, the researcher presented the findings of various biochar feedstocks characteristics that can be applied on AD of food waste for methane production. Previously, spent coffee ground modified biochar has shown a capability to improve the AD performance of methane yield by reducing the accumulation of VFAs and enhancing the activity of hydrogenotrophic methanogens (Kaur et al., 2020). However, up until now, no research focused specifically on addition of spent coffee ground biochar derived sludge to the AD of food waste for methane production. Therefore, this study is proposed to investigate the influence of spent coffee ground biochar derived sludge properties on the stability of the AD of food waste for methane yield. Overall, the goal of this study is to mitigate the mechanism of the properties of biochar towards the stability of AD of food waste by sequestering the product inhibition for methane production.

## 1.3 Objective

The objectives of this study include:

1. To characterize biochar as additive in anaerobic digestion of food waste from spent coffee ground.
2. To analyse the performance of biochar as additive in anaerobic digestion of food waste.

## 1.4 Scope of Study

The scope of the study focuses on the enhancement production of methane from food waste through anaerobic digestion (AD) by adding spent coffee ground (SCG) biochar. Firstly, the food waste will be collected from restaurants near Gambang. Type of food waste that will be used includes carbohydrates such as leftover rice and bread. The SCG will be dried and thermally treated before adding it to the anaerobic digester. Then, the SCG biochar will be further characterized by using Scanning Electron Microscopy (SEM), Brunauer-Emmett-Teller (BET) and Fourier Transform Infrared (FTIR).

Next, the performance of SCG biochar as additive will be analysed by comparing two anaerobic digesters, one is added with biochar while the other one is not. The parameter studied on the performance of biochar as additive in anaerobic digestion of food waste from SCG include the effect of pH (5, 6, 7, 8, 9), effect of the amount dosage of biochar ((1, 3, 5, 7, 9) g) and effect of the amount of feedstock ((100, 200, 300, 400, 500) g). Then, the biogas produced will be analysed by using online gas chromatography.

## 1.5 Significant of Study

Biogas is a renewable, as well as a clean source of energy. Gas generated through anaerobic digestion (AD) is non-polluting and it actually reduces greenhouse emissions. No combustion takes place during the AD process, meaning there is zero emission of greenhouse gasses into the atmosphere. Therefore, using gas from food waste as a form of energy is a great way to combat global warming. As clean fuel has been a concern to support local economic development in rural communities and to improve public health, the findings of this study will contribute to a promising alternative for biogas that can subdue various of energy and environmental issues through utilization of food waste for biogas production.

Food waste that is generated in excessive quantities has significant and negative environmental impacts. Using food waste can minimize the potential risk of environmental pollution and health from the conventional disposal method such as combustion and dumping in open area. Thus, using food waste can significantly reduce the risk of environmental contamination and health risks. As a result, this study depicts the future potential of renewable fuel that are beneficial in various environmental aspects.

Moreover, food waste contributes to the greenhouse gas (GHGs) emissions. Anaerobic digestion is a great way to prevent food waste from going to landfill and emitting GHGs there where the gas gives off contributes to global warming. Instead, the gas and other by-products are put to good use to power homes and fertilise crops whilst also providing an effective form of renewable energy.

# CHAPTER 2

# LITERATURE REVIEW

A literature review is an overview of a subject that includes applicable hypothesis, information and methods based on previous research. This chapter will review current studies on methane gas production from food waste in general.

## 2.1 Biogas Production

Biogas is a renewable energy source that can be harvested from organic waste, which received intensive research interested these decades. This biogas, which occurs naturally, spreads into the ambient, and its major component, methane, plays a serious role in global warming (Mar et al., 2022). Over the last few decades, methane has been used as a major fossil fuel and processed into electricity, transportation, and heating. Although natural gas resources account for the majority of methane consumption and utilisation today, bio-methane production from waste recovery methods has expanded significantly. In other words, infrastructures for biogas development extremely rely on specific equipment and the availability of control and management systems. Therefore, a sustainable industry can be installed and implemented to generate bioenergy from renewable and green natural resources.

Biogas produced from waste-derived biomass can be a promising alternative to natural gas (Ardolino & Arena, 2019). Biogas is the end product of the anaerobic digestion (AD) process consisting of $CO_2$, $CH_4$, and trace gases like $H_2S$, $N_2$ and $H_2$ (Teymoori Hamzehkolaei & Amjady, 2018). Organic waste, including livestock wastes and crop residues, are the primary substrates for biogas production in AD process. In addition to improve waste management, it helps to avoid potential environmental problems and to provide clean energy.

### 2.1.1 Sources of Biogas

Biogas production has usually been applied for waste treatment, mainly sewage sludge, agricultural waste (manure), and industrial organic waste streams (Kasinath et al., 2021). The primary source, which delivers the necessary microorganisms for biomass biodegradation and as well one of the largest single sources of biomass is from the food industry such as food waste and manure from animal production.

The mostly used sources for biogas production are the manure from cattle, pigs, and poultry litter (Damyanova & Beschkov, 2020). This application competes with the traditional use of manure for soil fertilization. When the amounts of manure prevail the demand for fertilization, biogas production is welcome because double problem is solved which on the one hand, the waste is destroyed and removed, and on the other hand, renewable energy is produced saving money and contributing for carbon cycle closing (Damyanova & Beschkov, 2020). That is why attention is paid to the utilization of cattle dung, lignocellulose waste, waste from food and beverage processing, activated sludge from wastewater treatment plants, and household solid waste with landfill gas use. The waste treatment is associated with energy production and reduction of the energy demand of the main enterprise.

Useful quantities of biogas can be generated from livestock waste, landfill gas, activated sludge from wastewater treatment plants and industrial, institutional, and commercial wastes. Livestock wastes constitute the most used substrate for biogas generation (If Solutions, 2020). Manure from poultry, cattle, and pigs are all good examples of this waste. This organic matter can be subjected to controlled decomposition to yield good quantities of biomethane. Landfill gas energy generation method uses accumulated wastes from domestic activities to develop biogas. Landfills are filled with suitable organic materials that decompose producing biogas that are modified and supplied to different processes. Next, sludge derived from wastewater purification is usually rich in organic materials thus forming a rich substrate for biogas generation. Once wastewater has been filtered out, activated sludge residue in the treatment plant can be channelled to specialized decomposition chambers where they generate methane. Other than that, many industrial manufacturing processes use organic raw materials in their production cycles. For example, food and beverage manufacturing companies use food materials that generate huge amounts of organic wastes. Biogas generation plants can harness these materials, allowing controlled decomposition to yield renewable energy. Table 2.1 shows different substrates and its biogas production.

Table 2.1 Studies on different feedstock and its operation condition for biogas production and its performance

| **Biomass** | **Inoculum** | **Operation Conditions** | **Methane yield** | **Reference** |
|---|---|---|---|---|
| Maize | Sludge | 36°C, 30 days | 379.0 mL $g^{-1}$ | (Martínez-Gutiérrez, 2018) |
| Sorghum | Digestates | 35°C, 30 days | 341.0 – 378.0 mL $g^{-1}$ | (Martínez-Gutiérrez, 2018) |
| Barley | Inoculum from anaerobic reactor | 37°C | 314.8 mL $g^{-1}$ | (Martínez-Gutiérrez, 2018) |
| Sugar beet | Digestate | 35°C, pH 8.1, 30 days | 350.4 – 399.4 mL $g^{-1}$ | (Martínez-Gutiérrez, 2018) |
| Sunflowers | Digestate | 35°C, pH 8.1, 30 days | 210 – 286.1 mL $g^{-1}$ | (Kulichkova et al., 2021) |
| Winter wheat | Digestate | 35°C, pH 8.1, 30 days | 269.2 – 327.6 mL $g^{-1}$ | (Kulichkova et al., 2021) |
| Rice straw | Sludge from manure compost (seed) | 55°C, pH 6.8 | 260 mL $g^{-1}$ | (Kulichkova et al., 2021) |
| Sunflower stalks | Granular sludge | 35°C, pH 7.0 | 302 mL $g^{-1}$ | (Kulichkova et al., 2021) |
| *Miscanthus sacchariflorus* | Anaerobic sludge | 39°C, 60 days HRT | 190 mL $g^{-1}$ | (Kulichkova et al., 2021) |
| Corn stover | Mixture from biogas plant | 37.5°C, 49 days | 585 mL $g^{-1}$ | (Yang et al., 2021) |
| Grass silage | Manure and crops | 55°C, 63 days | 405 mL $g^{-1}$ | (Anukam et al., 2019) |
| Wheat straw | Sludge wastewater | 35.1°C, pH 6.5-7.0, 30 days | 200-240 mL $g^{-1}$ | (Anukam et al., 2019) |

### 2.1.2 Properties of Biogas

Biogas is a satisfying alternative gaseous biofuel which can be produced from several organic resources and waste. Anaerobic digestion is a guaranteed alternative for treating biodegradable waste as it produces valuable fuel gas and subsequently a reduced volume of waste is disposed. Biogas production has a considerable role in waste management. It is not 100% greenhouse gas-free, nevertheless, it does not contribute to global warming. On the contrary, it helps to fight it. The methane can be combusted more cleanly than coal and can provide the desired energy with limited levels of carbon dioxide emission in the atmosphere (Bharathiraja et al., 2018). The carbon released from biogas can be absorbed by photosynthetic plants adding less total atmospheric carbon than the burning of fossil fuels.

The use of biomethane lowers water, soil, and air pollution not only because it eliminates fossil fuel related pollution, but the risk of potentially devastating accidents is also remarkably reduced. As an alternative source of both heat and electricity, biomethane helps preserve forests and biodiversity by providing reduced levels of harmful greenhouse gas. Additionally, the use of biomethane does not increase the concentration of greenhouse gases in the atmosphere because carbon dioxide and other gases that create the greenhouse effect are released into the atmosphere during the decomposition process of organic matter. In preference to living species including humans, biomethane is one of the best ways to satisfy the increased need for energy without contributing to warming of the planet which threatens their living (Bharathiraja et al., 2018). Biogas primarily consists of methane and carbon dioxide with small amounts of hydrogen, nitrogen, hydrogen sulphide, oxygen, water, and saturated hydrocarbons (i.e., ethane, propane). The detailed composition of biogas is discussed in Table 2.2.

Table 2.2 Composition of biogas

| **Constituent** | **Formula** | **Concentration (v/v)** | **Characteristic** | **Reference** |
|---|---|---|---|---|
| Methane | $CH_4$ | 40-75% | Combustible | (Benti & Asfaw, 2022) |
| Carbon dioxide | $CO_2$ | 15-60% | Non-combustible | (Bharathiraja et al., 2018) |
| Moisture | $H_2O$ | 1-5% | Non-combustible | (Bharathiraja et al., 2018) |

| | | | | |
|---|---|---|---|---|
| Nitrogen | $N_2$ | 0-5% | Non-combustible | (Bharathiraja et al., 2018) |
| Hydrogen | $H_2$ | Traces | Combustible | (Bharathiraja et al., 2018) |
| Hydrogen sulphide | $H_2S$ | 0-5000 ppm | Combustible | (Anukam et al., 2019) |
| Oxygen | $O_2$ | < 2% | Non-combustible | (Anukam et al., 2019) |
| Trace gases | - | < 2% | | (Anukam et al., 2019) |
| Ammonia | $NH_4$ | 0-500 ppm | | (Anukam et al., 2019) |

## 2.2 Anaerobic Digestion

Anaerobic digestion (AD) has been regarded as one of the most useful tools that can generate renewable energy. It is a low-energy process, making it more environmentally friendly and it has lower running costs as a result of the low energy inputs. AD has been used for many years for waste management such as wet biomass with high organic content to be used directly as a heating fuel or upgraded to serve as a replacement for natural gas (Abdel-Shafy & Mansour, 2018).

AD has been widely applied bioprocess to produce the biogas for fuels from organic waste degradation. AD has been integrated with other processes for increasing the digestion efficiency and waste valorisation. The integration of AD with other bioprocess optimizes the production of targeted product and reduces the waste.

One of the most obvious environmental benefits of anaerobic digestion is its function in reducing greenhouse gas emissions. By capturing methane gas that may have otherwise been lost to the atmosphere, AD operations displace fossil fuel energy use. This contributes to climate change mitigation and is notably beneficial in all AD technology use scenarios. The use of AD technology on farms provides a multitude of examples of how anaerobic digestion benefits the environment (My Technologies, 2020). As farmers work diligently to meet the increasing demand for food and remain viable and profitable in the current global marketplace, the efficient use of water and nutrients for crop and livestock needs can reduce costs and environmental impacts and contribute to safer, more productive farms.

Aside from numerous environmental benefits, there are several economic benefits associated with utilizing anaerobic digestion technologies. For wastewater treatment facilities that are incorporating food waste into anaerobic digesters, they experience two-fold savings through the reduction of energy costs via onsite power production, as well as through the receipt of tipping fees for accepting the food waste from food processing companies. Food and beverage processing facilities can leverage the same benefits on site, but the introduction of food waste into wastewater treatment facilities has become increasingly popular in recent years.

### 2.2.1 Process of Anaerobic Digestion

AD is the conversion of organic matter to methane-rich biogas through a series of interlinked processes such as hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In this process, biodegradation of organic materials occurs in the absence of oxygen resulting in the production of biogas which mainly consists of methane and carbon dioxide along with trace amounts of gases such as hydrogen sulphide and ammonia (Ahammed & Gadekar, 2021). Figure 2.2 shows the flow process of AD.

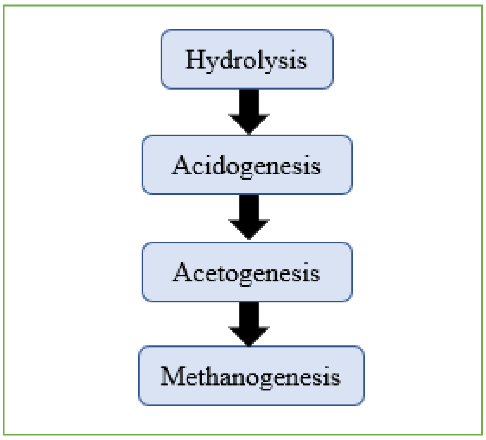

Figure 2.1 Flow process of anaerobic digestion

Anaerobic digestion for biogas production takes place in a sealed vessel called a reactor, which is designed and constructed in various shapes and sizes specific to the site and feedstock conditions. These reactors contain complex microbial communities that break down the waste and produce resultant biogas and digestate (the solid and liquid material end-products of the AD process) which is discharged from the digester (Harirchi et al., 2022).

Multiple organic materials can be combined in one digester, a practice called co-digestion. Co-digested materials include manure, food waste (i.e., processing, distribution and consumer generated materials), energy crops, crop residues, and fats, oils, and greases (FOG) from restaurant grease traps, and many other sources. Co-digestion can increase biogas production from low-yielding or difficult-to-digest organic waste. The figure below illustrates the flow of feedstocks through the AD system to produce biogas and digestate.

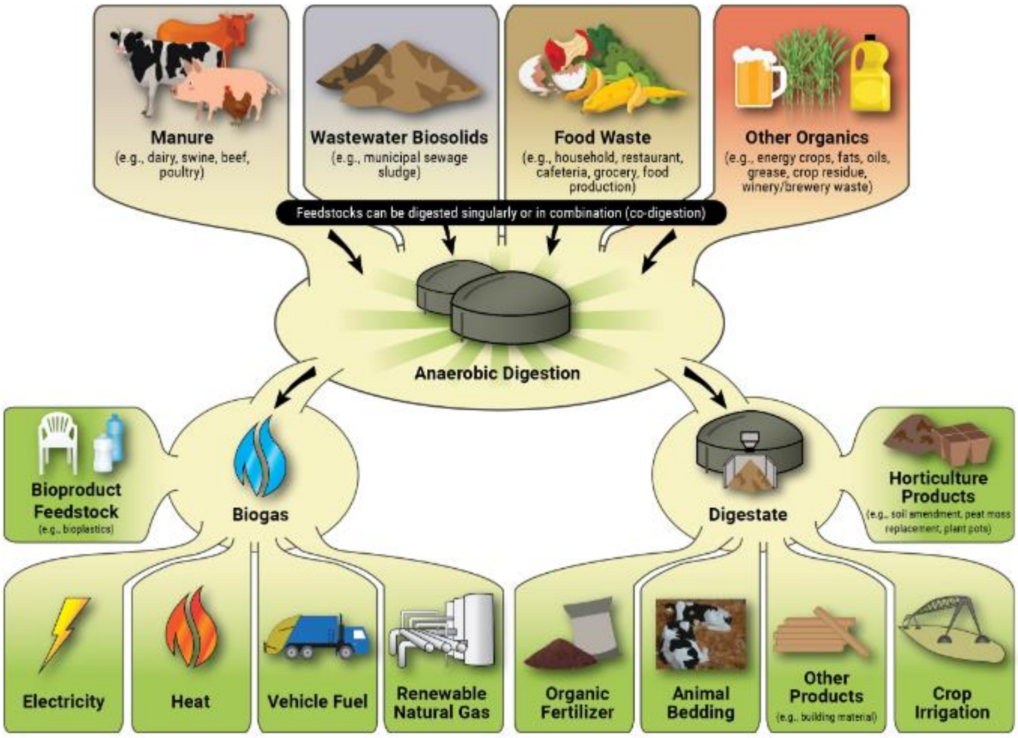

Figure 2.2 The flow of feedstock through the AD system to produce biogas and digestate

Source: Benti & Asfaw (2022)

#### 2.2.1.1 Hydrolysis

Organic material found in anaerobic digesters often comprises complex polymers that are inaccessible to microbes unless further broken down through hydrolysis or pre-treatments (Gujer & Zehnder, 2019). As a result, the process of hydrolysis serves the purpose of rendering organic molecules into its smaller components, which in turn can be utilized by acidogenic bacteria. A variety of waste pre-treatment options are being researched and utilized to optimize hydrolysis, especially for digesters that digest heavily lignocellulosic wastes (Meegoda et al., 2018). Hydrolysis has its own optimum temperature between 30-50°C with an optimum pH of 5-7.

Hydrolysis involves the conversion of polymeric organic matter (e.g., polysaccharides, lipids, proteins) to monomers (e.g., sugars, fatty acids, amino acids) by hydrolases secreted to the environment by microorganisms. Three key groups of hydrolases are involved in the process of anaerobic digestion: esterases, glycosidases and peptidases, which catalyse the cleavage of ester bonds, glycoside bonds and peptide bonds, respectively (Sikora et al., 2018). The bacteria most commonly associated with hydrolysis include representatives of the Firmicutes (Clostridia, Bacilli), Bacteroidetes and Gammaproteobacteria (Sikora et al., 2018). Usually, the same bacteria are also able to conduct acidogenesis, the second step in the decomposition of organic matter.

#### 2.2.1.2 Acidogenesis

Acidogenesis is the fermentation stage where the products of hydrolysis (soluble organic monomers of sugars and amino acids) are degraded by acidogenic bacteria to produce alcohols, aldehydes, and VFAs and acetate together with $H_2$ and $CO_2$ (Kamusoko et al., 2022). Acidogenic microorganisms are able to produce intermediate VFA and other products by absorbing the products of hydrolysis through their cell membranes. The specific concentrations of intermediates produced during the acidogenesis stage may be dependent on the digester's conditions.

The degradation of amino and acids also liberates ammonia gas. Acidogenic bacteria can be either facultative anaerobes or strict anaerobes. Those belonging to the family Enterobacteriaceae have been identified as active fermenters (Kamusoko et al., 2022). Species found in anaerobic digesters include Lactobacillus, Escherichia, Staphylococcus, Pseudomonas, Desulfovibrio, Selenomonas, Sarcina, Streptococcus, Desulfobacter, and Desulforomonas. These convert amino acids to fatty acids, acetate, and $NH_3$ (Hosen et al., 2019). Other species such as Clostridium, Eubacterium limosum, and Streptococcus, transform sugars into intermediary fermentation products (Hosen et al., 2019).

It has been reported that VFA concentrations can vary significantly for digesters operating at different pH levels, with seemingly contradictory results from different studies (Lukitawesa et al., 2019). Acidogenesis is thought to occur more quickly than the other stages of anaerobic digestion, with acidogenic bacteria having a regeneration time of less than 36 hours (Meegoda et al., 2018). The production of VFA gives impact for the final stage of methanogenesis, VFA acidification is widely reported to be a cause for digester failure (Al-Sulaimi et al., 2022).

### 2.2.1.3 Acetogenesis

The two final steps of anaerobic digestion, acetogenesis and methane formation, are tightly connected. Acetogenesis is the process by which these higher VFA and other intermediates are converted into acetate, with hydrogen also being produced. As a result of the generation of acetate during acidogenesis, a portion of the original substrate has already been converted into a substrate suitable for methanogenesis (Detman et al., 2021). $H_2$ concentration is a key factor regulating the metabolism of acetate and methane formation. The formation of biogas from fermentation products only occurs if the hydrogen concentration is less than the feasible thermodynamic concentration (A. Hassaan et al., 2022). Therefore, it is difficult to detect $H_2$ in the biogas.

Due to the limited number of substrates for methanogenesis, methanogens are strictly dependent on partner microbes with which they form syntrophic systems. Syntrophy is a special type of symbiotic cooperation between two metabolically different types of microorganisms, which depend on one another for the degradation of a certain substrate, typically through the transfer of one or more metabolic intermediate. In this case, the partner microbes oxidize non-gaseous products of acidogenesis to acetate, carbon dioxide, hydrogen and formate that are directly utilized by the methanogens, making the entire syntrophic metabolism efficient and thermodynamically favourable. This is the essence of acetogenesis. The process of hydrogen or formate transfer between acetogenic bacteria and methanogenic Archaea is an excellent example of syntrophy (Sikora et al., 2018).

### 2.2.1.4 Methanogenesis

Methanogenesis is the final stage of anaerobic digestion where accessible intermediates are consumed by methanogenic microorganisms to produce methane. Methanogenic microorganisms are limited to a small selection of substrates. With regards to the environmental needs of methanogenesis, methanogenic microorganisms tend to require a higher pH than previous stages of anaerobic digestion (Meegoda et al., 2018). At the same time, methanogens appear to have a substantially longer regeneration period in anaerobic digestion than other microorganisms, ranging from 5 to 16 days (Meegoda et al., 2018).

Methanogens carry out methanogenesis which produces methane ($CH_4$), a potent greenhouse gas. There are three pathways for methanogenesis to occur which are acetoclastic methanogenesis, methylotrophic methanogenesis, and hydrogenotrophic methanogenesis. Acetoclastic methanogens use acetate as an electron acceptor while methylotrophic methanogens use methanol or methylamines. Hydrogenotrophic methanogens use $H_2$ and $CO_2$ to carry out methanogenesis. Hydrogenotrophic and acetoclastic methanogenesis are the most common pathways in freshwater systems (Mobilian & Craft, 2021).

### 2.2.2 Parameter of Anaerobic Digestion Process

The performance of anaerobic digester can be controlled by studying and monitoring the variation in parameters like the effect of pH, effect of the amount of biochar dosage, effect of hydraulic retention time, effect of temperature, and types of feedstocks. Any drastic change in these can adversely affect the biogas production. Thus, these parameters should be varied within a desirable range to operate the AD efficiently (Nevzorova & Kutcherov, 2019).

#### 2.2.2.1 Effect of pH

pH is an expression of the intensity of the basic or acid condition of a liquid, a measure of the acidity of a solution. Commonly, methanogens in wastewater treatment systems are most active in the neutral pH range (Abdelgadir et al., 2019). pH is a major variable to be monitored and controlled. The range of acceptable pH in anaerobic digestion is theoretically from 5.5 to 8.5. Beyond this range, digestion can proceed, but with less efficiency. The biomass inhibited at pH 9 is able to regain activities after adjusting the pH to neutrality, but that inhibited at pH 5 is not (Allaart et al., 2021).

At acidic conditions produced can become quite toxic to the methane bacteria. For this reason, it is important that the pH is not allowed to drop below 6.2 for a significant period of time. Because this parameter is very important, thus the system needs to control the pH. A falling pH can point toward acid accumulation and digester instability. Gas production is the only parameter that shows digester instability faster than pH. For acidogenesis process to proceed normally, concentration of volatile fatty acids (VFAs) should be low.

#### 2.2.2.2 Effect of the Amount of Biochar Dosage

The addition of porous, carbon-based conductive materials is an emerging and effective method for achieving preferable performance during AD, which attracted increasing attentions (Qiu et al., 2019). Biochar as the precursor of activated carbon, is a preferred solid material produced by the thermochemical conversion of biomass or biodegradable wastes in anaerobic condition (Pytlak et al., 2020). A recent study showed that the methane yield increased by 25% during AD of animal manure after the optimal biochar dosage of 5–10% amended (Yang et al., 2021) and the positive roles of biochar in the AD of multiple organic wastes have been undoubtedly verified (Pan et al., 2019).

It has been proposed that biochar reinforces AD through providing a habitat for microbial colonization or constructing a conductive platform for interspecies electron transfer. Furthermore, biochar application has an extra important role on enhancing AD stability under inhibitory conditions. biochar could mitigate the bio-toxicity shock of oil-based compounds by adsorbing compounds and enriching functional microflora (Kong et al., 2018).

Biochar accelerated the biomethanization during AD under the double inhibition of ammonium and acids by enriching Methanosaeta and Methanosarcina (Li et al., 2021). Besides, the carbon-based material was known to stimulate microbial diversity and methanogenesis under anaerobic conditions in oil-polluted sediment (Bonaglia et al., 2020). Therefore, it can be hypothesized that the application of biochar will be a more efficient technology to enhance AD process under bio-toxicity suppression of oily sludge. However, some researchers have reported that biochar application at high loads could cause negative impacts on AD (Pytlak et al., 2020). It indicates that the dosage-effect of biochar on AD remains highly disputed for the existence of uncertain factors.

#### 2.2.2.3 Effect of the Hydraulic Retention Time

Hydraulic Retention Time (HRT) is an important operational parameter for the anaerobic reactors which can affect the conversion of volatile solids into biogas (Sarker et al., 2019). Generally, relatively long HRT is needed in anaerobic digestion of food wastes for certain type of substrates is persistent to anaerobic microbes. Some studies suggested that 60 – 90 days is required in order to achieve complete digestion of substrates. Shorter HRT is desirable as it is directly related to the reduction of capital cost and the increase of process efficiency.

Hydraulic retention time is the volume of the aeration tank divided by the influent flow rate:

$$\mathrm{HRT}=\frac{\text{Volume of aeration tank } (\mathrm{m}^3)}{\text{Influent flow rate } (\mathrm{m}^3/\mathrm{d})}=\frac{V}{Q} \qquad 2.1$$

Where HRT is hydraulic retention time (d) and usually expressed in hours (or sometimes days), the V is the volume of aeration tank or reactor volume ($m^3$), and Q is the influent flow rate ($m^3/d$).

Generally, HRT is a good operational parameter that is easy to control and also a macro conceptual time for the organic material to stay in the reactor. In bioreaction engineering studies, the reverse of HRT is defined as dilution rate, for which if it is bigger than the growth rate of microbial cells in the reactor, the microbe will be washed out, and otherwise the microbe will be accumulated in the reactor. Either of these situations may result in the breakdown of the biological process happening in the reactor.

#### 2.2.2.4 Effect of the Temperature

In anaerobic digestion process, temperature is not only important for microbial metabolic activities but also for the overall digestion rate, specifically the rates of hydrolysis and methane formation. In general, anaerobic digestion process can occur within a wide range of temperatures. This temperature range has been divided into three groups: psychrophilic, mesophilic and thermophilic. Anaerobic digestion can be developed for different temperature ranges including, mesophilic temperatures of approximately 35°C and thermophilic temperatures ranging from 55°C to 60°C (Schmidt, 2019). Conventional anaerobic digestion is carried out at mesophilic temperatures, that is, 35–37°C. However, the thermophilic temperature range is worth considering because it will lead to give faster reaction rates, higher gas production, and higher rates of the destruction of pathogens and weed seeds than the mesophilic temperature range. However, the thermophilic process is more sensitive to environmental changes than the mesophilic process (Fikri Hamzah et al., 2019).

Temperature of digester affects the activities of the anaerobic bacteria and waste decomposition. The rate of degradation and biogas production is enhanced at higher temperatures (Hossain et al., 2022). The optimum digester temperature, considering both the potential biogas yield and heat demand, is one of the most crucial factors for economical operation of digester (Babaei & Shayegan, 2019). Other researchers reported that the concentration of free ammonia (inhibitor) increased with the rise of temperature, leading to the decrease in biogas yield (Babaei & Shayegan, 2019).

#### 2.2.2.5 Effect of the Types of Feedstocks

A great variety of organic material can be used in anaerobic digesters as a feedstock for generating biogas. However, there are scientific, engineering, and legal limits to what can be added successfully to a digester. In addition, the feedstock needs to be a liquid mixture with an appropriate moisture content. Most easily biodegradable biomass materials are acceptable as feedstocks for anaerobic digestion. Common feedstocks include livestock manure, food-processing waste, and sewage sludge. The energy production potential of feedstocks varies depending on the type, level of processing and pre-treatment and concentration of biodegradable material (Farm Energy, 2019).

Livestock manure is one of the most common feedstocks employed in AD because it is readily available in agricultural farms. Despite containing many characteristics favorable for AD (neutral pH, different microbes, a wide variety of nutrients), they produce a lower amount of biogas than other feedstocks because they are already predigested by the animal intestine (Uddin & Wright, 2022). However, manure is often added as a base substrate and codigested with other feedstock because of its desirable characteristics.

Feedstocks such as sewage sludge and slaughterhouse waste may contain harmful toxic components for the AD bacteria (Lisowyj & Wright, 2020). These should be treated to avoid collapsing the AD bacteria. It is also essential to remove or minimize non-biodegradable components from the feed to use the digester volume effectively. Woody biomass or feedstock with higher lignin content are not easily digestible in the AD process and require specific pretreatments to break down the biomass structure. Codigestion of different feedstocks can improve the biogas production in AD, given that the resulting nutrients balance is optimum for the AD process (Lisowyj & Wright, 2020). The selection of proper feedstock depends on multiple factors such as availability, desired application of AD products, environmental conditions, digester technology, and economic benefits.

## 2.3 Biochar

Biochar is a carbon-enriched biomaterial generated in the combustion of biomass through a process called pyrolysis (Santos et al., 2019). In pyrolysis, biomass such as crop and forestry residues, manure, municipal and industrial wastes are decomposed at temperatures higher than 400°C, in the complete or near absence of oxygen. As a result, syngas, bio-oil, and biochar are produced. Biochar technology shows promise in mitigating climate change as well as reducing waste and producing energy as a by-product.

Although it looks a lot like common charcoal, biochar is produced using a specific process to reduce contamination and safely store carbon. During pyrolysis organic materials, such as spent coffee ground, wood chips, leaf litter or dead plants, are burned in a container with very little oxygen. As the materials burn, they release little to no contaminating fumes. During the pyrolysis process, the organic material is converted into biochar, a stable form of carbon that cannot easily escape into the atmosphere. The energy or heat created during pyrolysis can be captured and used as a form of clean energy. Biochar is by far more efficient at converting carbon into a stable form and is cleaner than other forms of charcoal (Spears, 2018).

Biochar addition could mitigate toxins inhibitory, shorten methanogenic lag phase, immobilize functional microbes and accelerate electron transferring between methanogenic and acetogenic microbes during AD process (Chiappero et al., 2020). Some researchers reported that the biochar addition reduced the lag time (28–64%) and improved the biogas production (22–40%), and correspondingly the relative abundance of electro-active microorganisms and Methanogens were enriched by 24.61% and 43.8% (Wang et al., 2018). Besides, high surface area, considerable porosity, abundant existence of functional groups and excellent electron transferring ability were also the key advantages of biochar, as compared with other materials, in enhancing anaerobic methane production (Kumar et al., 2021).

### 2.3.1 Sources of Biochar

Biochar is produced from biomass. The biomass and raw material from which biochar is produced mainly come from a variety of materials. Agricultural biomass is considered as the most abundant renewable resource. Main sources and feedstock of biochar include crop straw and residues, animal manures, fruit pits, twigs, and leaf litter forestry wastes as well as food leftover (Shaaban & Abid, 2021). The material for biochar production plays a pivotal role in the composition of the resulting biochar. Table 2.3 shows the different sources of biochar and its compositions.

Table 2.3 Biochar prepared from different biomass sources and its composition

| Sources | Dry mater (g/kg) | Surface area ($m^2/g$) | Volatile matter | References |
|---|---|---|---|---|
| Miscanthus straw pellets | 982 | 34 | 116 | (Cabeza et al., 2018) |
| Oil seed rape straw pellets | 974 | 16 | 164 | (Cabeza et al., 2018) |
| Rice husks | 985 | 20 | 75 | (Tomczyk et al., 2020) |
| Soft wood pellets | 985 | 26 | 142 | (Tomczyk et al., 2020) |
| Wheat straw pellets | 986 | 26 | 106 | (Ippolito et al., 2020) |

### 2.3.2 Properties of Biochar

Biochar properties are affected by several technological parameters, mainly pyrolysis temperature and feedstock kind, which differentiation can lead to products with a wide range of values of pH, specific surface area, pore volume, volatile matter, ash, and carbon content (Tomczyk et al., 2020). High pyrolysis temperature promotes the production of biochar with a strongly developed specific surface area, high porosity, pH as well as content of ash and carbon, but with low values of volatile matter content. Biochar produced from animal litter and solid waste feedstocks exhibit lower surface areas, carbon content, volatile matter compared to biochar produced from crop residue and wood biomass, even at higher pyrolysis temperatures (Tomczyk et al., 2020). The reason for this difference is considerable variation in lignin and cellulose content as well as in moisture content of biomass.

Important and physical properties of biochar include bulk density and particle density, particle size, macro and microporosity, surface area, water holding capacity. The physical properties of biochar depend mainly on the feedstock and the temperature at which the biochar is produced. As the temperature increases the persistence of the carbon matrix increases but there can be less available nitrogen and mineralizable organic compounds. In high mineral ash biochars, carbon content is reduced significantly. Other factors that can affect biochar properties are type of pyrolizer residence time and heating rate.

Diameter of biochar particles is typically measured by fraction passing through a series of sieves of different sizes. The particle size depends on feedstock and its pre-processing, and the production technique and temperature. It can be reduced by grinding biochar, and sieving can ensure a more uniform particle size. It also can be increased by pelletizing or granulating. Particle size also influences the pore volume between particles and the bulk density, the water holding capacity and hydraulic conductivity, the effectiveness in compaction remediation, the mobility and loss of biochar in wind and water, the speed of oxidation and aging and materials handling including dust production.

### 2.3.3 Biochar from Spent Coffee Ground

Coffee is a widely consumed drink with beans grown in more than 80 countries worldwide (Huong et al., 2020). There were large amounts of coffee residues produced from the manufacturing of coffee drinks and instant coffee preparation in the coffee industry worldwide over 6 million tons per year (Tangmankongworakoon, 2019). According to the International Coffee Organization, the global coffee yield has steadily increased during the last 150 years. The coffee residues have been able to be used for various purposes instead of throwing them as a waste. Coffee residues contain several organic compounds such as fatty acids, lignin, cellulose, hemicellulose, and other polysaccharides that justify its volatilization and had high levels of substances known to be toxic to many life processes, such as acids, free phenols, caffeine, and tannins (LIczbiński & Bukowska, 2022, Andlar et al., 2018). Coffee waste thus constitutes a source of serious environmental problems in coffee-using countries.

Some researchers had investigated the benefit of coffee residues as a biological resource for various valuable compounds. For example, there is study that addressed the use of biochar from coffee residues to help increase pH and absorb heavy metals in water and soil. Coffee residues along with biochar improved the quality of heavy metal-contaminated water and soil. It was indicated that biochar added to compost is advantageous as it helped in reducing ammonia emissions and increasing carbon capture moisture of the substance. Thus, biochar from spent coffee ground (SCG) has lots of advantages as it can be used for a variety of purpose.

Biochar has been used as a useful and economical material in many fields such as food additives, biogas, caffeine, pectin, feeds, protein, antioxidants, beverages, enzymes, compost, and the production of bioactive compounds (Jagdale et al., 2019). According to some previous studies, protein content in coffee grounds accounts for about 10%, pectin content for 52.62–55.14%, and cellulose for 15.29–17.04% of the mass of the grounds, which also have high carbon content (>50%) (Huong et al., 2020). Coffee grounds are a lignocellulosic material that can separate heavy metals and dye in water based on their porous structure and cellulose composition. The surface of carbonaceous materials contains many phenolic hydroxyl and carboxyl groups. In cellulose materials, these groups play an important role in ion exchange, and studies of treatment effectiveness indicated that the adsorption of pollutants depends upon the sur-face polar groups on the carbonaceous materials (Huong et al., 2020). Therefore, spent coffee grounds are an effective material in the synthesis of biochar.

# CHAPTER 3

# METHODOLOGY

Methodology is a way to find out the result of a given problem on a specific research. This chapter will describe the method to identify and justify the sampling method and demonstrate the techniques and instrument used for qualitative and quantitative of the expected result for given research problem.

## 3.1 Methodology Flowchart

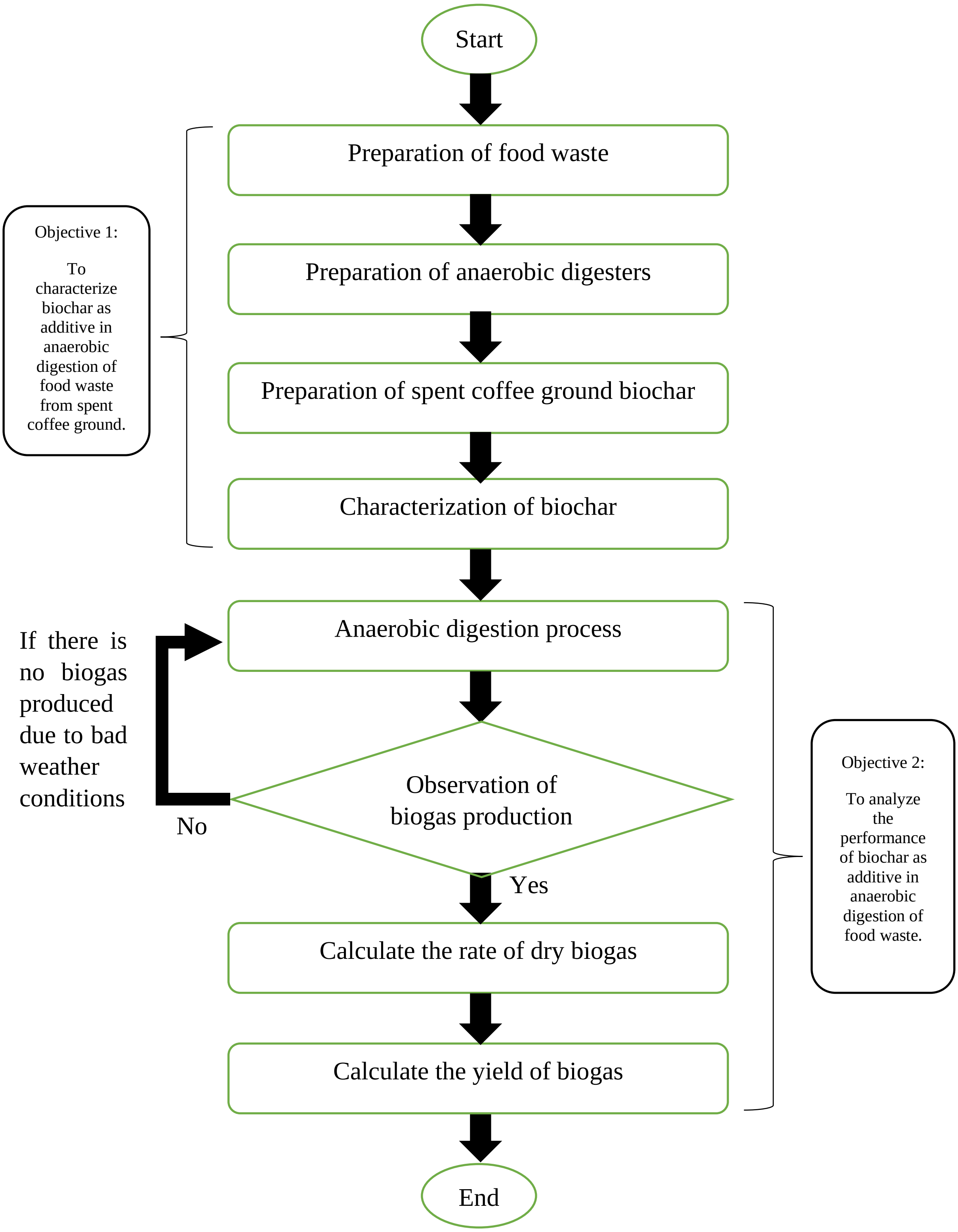

Figure 3.1 Methodology flowchart

## 3.2 Biochar from Spent Coffee Ground (SCG)

### 3.2.1 Material

The additive that was used in anaerobic digestion (AD) of food waste was biochar. The biochar was prepared from spent coffee ground (SCG) was collected from local coffee shops in Gambang. The collected SCG was cleaned to get rid of the mould.

### 3.2.2 Preparation of Spent Coffee Ground (SCG) Biochar

The biochar that was used in this study was prepared from SCG. Initially, the collected SCG was calcined in an oven at 105°C for 24 hours to remove the moisture content. Then, it was let to cool at ambient temperature. Subsequently, the biochar was placed in crucible and put into the furnace for the carbonization process at the temperature of 550°C for 3 hours. After cooling, the biochar obtained was kept in desiccator for further use.

### 3.2.3 Characterization of Spent Coffee Ground (SCG) Biochar

The morphology and surface structure of biochar was measured by Scanning Electron Microscopy (SEM). The Brunauer-Emmett-Teller (BET) specific surface area and textural analysis of biochar were determined by Nitrogen Adsorption-Desorption ($N_2$ Physisorption). Functional group identification of spent coffee ground (SCG) biochar was determined by Fourier Transform Infrared (FTIR).

#### 3.2.3.1 Surface Morphology Analysis by Scanning Electron Microscopy (SEM)

The SEM functions by projecting a beam of electrons through magnetic focusing lenses at a specimen and recording secondary electrons excited by the primary beam. The amount of secondary electron emission portrays the topography of the material since high points emit more secondary electrons than low points (Swapp, 2018). The morphology of the biochar was examined by setting up the electron beam energy at 20keV, working distance 10 – 10.5mm, and dead time of X-ray acquisition between 15 and 20%. Line-scan analysis were performed to analyse the morphology and content of the biochar. Dot maps of each element present in the samples were acquired through a series of scans lasting 30s, in order to acquire reliable maps.

#### 3.2.3.2 Textural Analysis by Nitrogen Adsorption-Desorption ($N_2$ Physisorption)

Brunauer-Emmett-Teller (BET) surface area analysis is a multipoint assessment of a material’s specific surface area using gas adsorption analysis, in which an inert gas such as nitrogen ($N_2$) is continually flowing over a solid sample, or the solid sample is suspended in a specified gaseous volume. The specific surface area, the micropores surface area, and the pore size distribution of the samples was determined from the nitrogen isotherms at −196°C using a Tristar 3000 Micromeritics instrument. The specific surface area was calculated using BET equation, the micropores surface area was determined using the t–plot method, and the pore size distribution with the Barrett–Joyner–Halenda (BJH) method. The samples were outgassed before measurement at 120°C under $N_2$ flow for over 1 hour.

#### 3.2.3.3 Functional Group Identification by Fourier Transform Infrared (FTIR)

Surface functional group on biochar surface were determined by Fourier Transformed Infrared (FTIR) using Thermo Fishcer Scientific Nicolet iS50 FTIR, where the spectrum for FTIR will be in the range of $400cm^{-1}$ to $4000cm^{-1}$ with a resolution factor of $4cm^{-1}$ (Rosi et al., 2022). Data were collected at room temperature by using Potassium Bromide (KBr) pellet technique.

## 3.3 Performance of Spent Coffee Ground (SCG) Biochar in Anaerobic Digestion (AD) of Food Waste

### 3.3.1 Preparation of Food Waste Inoculum

The feedstock that was used for the anaerobic digestion (AD) in this study is food waste that was collected from restaurants in Gambang. Solid food waste that was used consists of carbohydrates such as leftover rice and bread and fruits such as rotten banana and mango. The food waste was processed without any pre-treatment to remove physical impurities, except water addition to dilute and slurry the feedstock.

### 3.3.2 Anaerobic Digestion (AD) of Food Waste for Methane Generation

AD consists of four stages which are hydrolysis, acidogenesis, acetogenesis and methanogenesis, while each of which is performed by a unique set of functional microorganisms (Sun et al., 2022). Food waste will be used as feedstock of AD. The experiment was conducted using bottles as digester and were added with spent coffee ground (SCG) biochar. Subsequently, a silicone tube was connected to the bottle cap and a gas bag to observe the biogas production. Then, the bottles were placed at the same place and were observed for 14 days.

### 3.3.3 Analysis of Anaerobic Digestion (AD) Product Calculation

The performance of spent coffee ground (SCG) biochar was observed by the total amount of biogas produced in the anaerobic digestion (AD) of food waste. The total biogas production was measured by water displacement method. The volume of water displaced in the container is equal to the volume of the gas. One end of the silicone tube was connected to the digester and other end connected to invert measuring cylinder which contained water. The amount of gas calculated was equal to the volume of water displaced. The biogas was allowed to collect in the inverted measuring cylinder by displacing water.

The rate of wet and dry biogas production can be calculated as follows:

$$\text{Rate of wet/dry biogas production} = \frac{\text{Volume of wet biogas captured (mL)}}{\text{time taken (h)}} \qquad 3.1$$

Gases collected over water are saturated with water vapour. Therefore, $P_{total}=P_{biogas}+P_{water}$. The partial pressure of water in the mixture, $P_{water}$ is the equilibrium vapour pressure of water at the specified temperature. The combined Boyle's and Charles's Laws were used to calculate the volume of dry biogas using the equation below:

$$\frac{P_1V_1}{T_1}=\frac{P_2V_2}{T_2} \qquad 3.2$$

Lastly, the yield of biogas production was calculated using the following equation:

$$\text{Yield of biogas}=\frac{\text{Volume of dry biogas (ml)}}{\text{Mass of raw materials (g)}} \qquad 3.3$$

### 3.3.4 Analysis of Online Gas Chromatography (GC-TCD)

Biochar analysis through online gas chromatography or GC-TCD was performed in a GC-2014 by Shimadzu to analyse the composition of biogas produced from anaerobic digestion process. The operating condition for the GC-TCD 8ml/min carrier gas flow of Helium (He) with column held at 35°C at 5 minutes and ramped to 65°C at rate 20°C $\text{min}^{-1}$ (Sołowski, 2022). Then, the column was held at 65°C for 4 minutes. The injector was set to 150°C meanwhile DTCD1 and DTCD2 temperatures were set to 110°C and 60°C respectively.

# CHAPTER 4

# RESULTS AND DISCUSSION

This chapter covers the findings of the research as well as the discussion that sufficiently supports the findings achieved. Figures and tables are used to illustrate the key findings.

## 4.1 Biochar from Spent Coffee Ground (SCG)

Biochar with appropriate properties is an effective, inexpensive, and sustainable additive to enhance methane yield in anaerobic digestion. In principle, any biodegradable organic matter can be anaerobically digested to produce biogas, however the biogas produced can be enhanced by adding biochar as additive. In this study, biochar was prepared by spent coffee ground (SCG) and underwent calcination process. The SCG biochar was dried in an oven at 105°C for 24 hours to remove its moisture content, then it was calcined in a furnace at 550°C for 3 hours. Figure 4.1 shows the SCG before and after calcination process.

As shown in Figure 4.1 (a), the raw SCG that was collected from local coffee shops in Gambang was cleaned up to get rid of the mould. Figure 4.1 (b) and (c) show the SCG biochar after drying and calcining process. It shows that the SCG was decreasing after calcination as the heating of solids to a high temperature removes its volatile substance and oxidizes a portion of mass, thus reducing the amount of SCG.

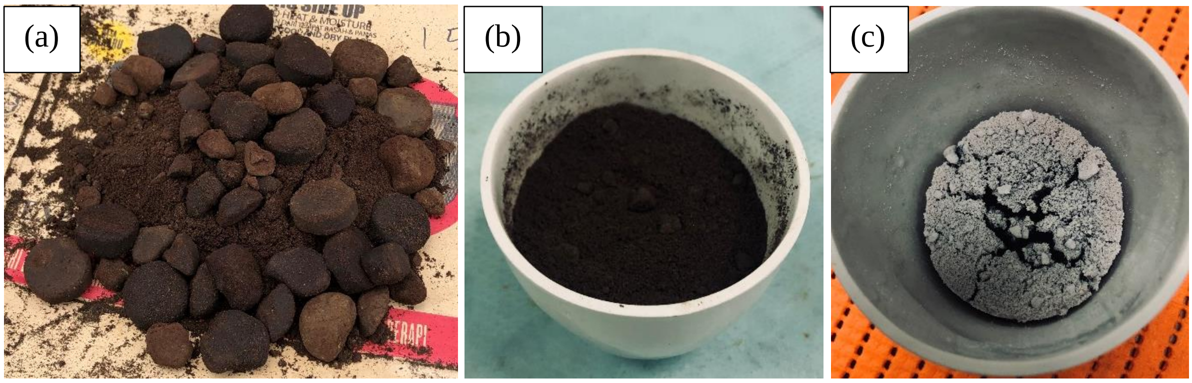

Figure 4.1 Spent coffee ground (a) raw, (b) after drying, and (c) after thermal treatment process

## 4.2 Characterization of Spent Coffee Ground Biochar

This study characterizes the biochar that was prepared by spent coffee ground (SCG). The raw SCG that was collected from local coffee shops and the one that had been calcined was sent to the laboratory to characterize the differences. Both samples were characterized to measure the morphology and surface structure by Scanning Electron Microscopy (SEM), while the specific surface area and textural analysis were determined by Brunauer-Emmett-Teller (BET) Nitrogen Adsorption-Desorption, and functional group identification was determined by Fourier Transform Infrared (FTIR). The results of these analysis will be presented and discussed in the following section.

### 4.2.1 Surface Morphology Analysis by Scanning Electron Microscopy (SEM)

SEM analysis is used to evaluate the change in the surface morphology of SCG before and after calcination process. The SEM images of raw SCG and SCG biochar are shown in Figure 4.2. Figure 4.2 (a) and Figure 4.2 (b) show the raw, untreated SCG at two magnification scales which are 200 and 1000. Meanwhile, Figure 4.2 (c) and Figure 4.2 (d) show the SEM images of biochar after calcination process at the same magnification scales. The corresponding EDX analysis is presented in Table 4.1.

SEM images of the SCG biochar in Figure 4.2 (c) and Figure 4.2 (d) show that the morphology of the pores is bigger than the raw, untreated SCG in Figure 4.2 (a) and Figure 4.2 (b). These pores are estimated about 5μm. Calcination process removes the volatile substances, oxidizing a portion of mass and rendering them friable. The calcination process enriches the SCG biochar with carbon as a significant amount of oxygen is released during calcination. The results shows that the raw, untreated SCG has 54.52% of carbon meanwhile the SCG that has been calcined has 87.76% of carbon. Based on Table 4.1, EDX analysis for the samples show that the main elements are C and O.

SCG biochar had a porous structure, and the surface morphology became gradually rougher than raw SCG, which may be because cellulose, hemicellulose and lignin in the straw were decomposed and carbonised (Wang et al., 2022). With the support destroyed, the surface began to decompose, which caused the biological structure of the biochar to collapse, thereby forming numerous mesopores (Wang et al., 2022). The pore diameters of mesopores gradually became larger. With its porosity and large specific surface area, biochar can provide a better habitat for microbial growth (Fagbohungbe et al., 2019).

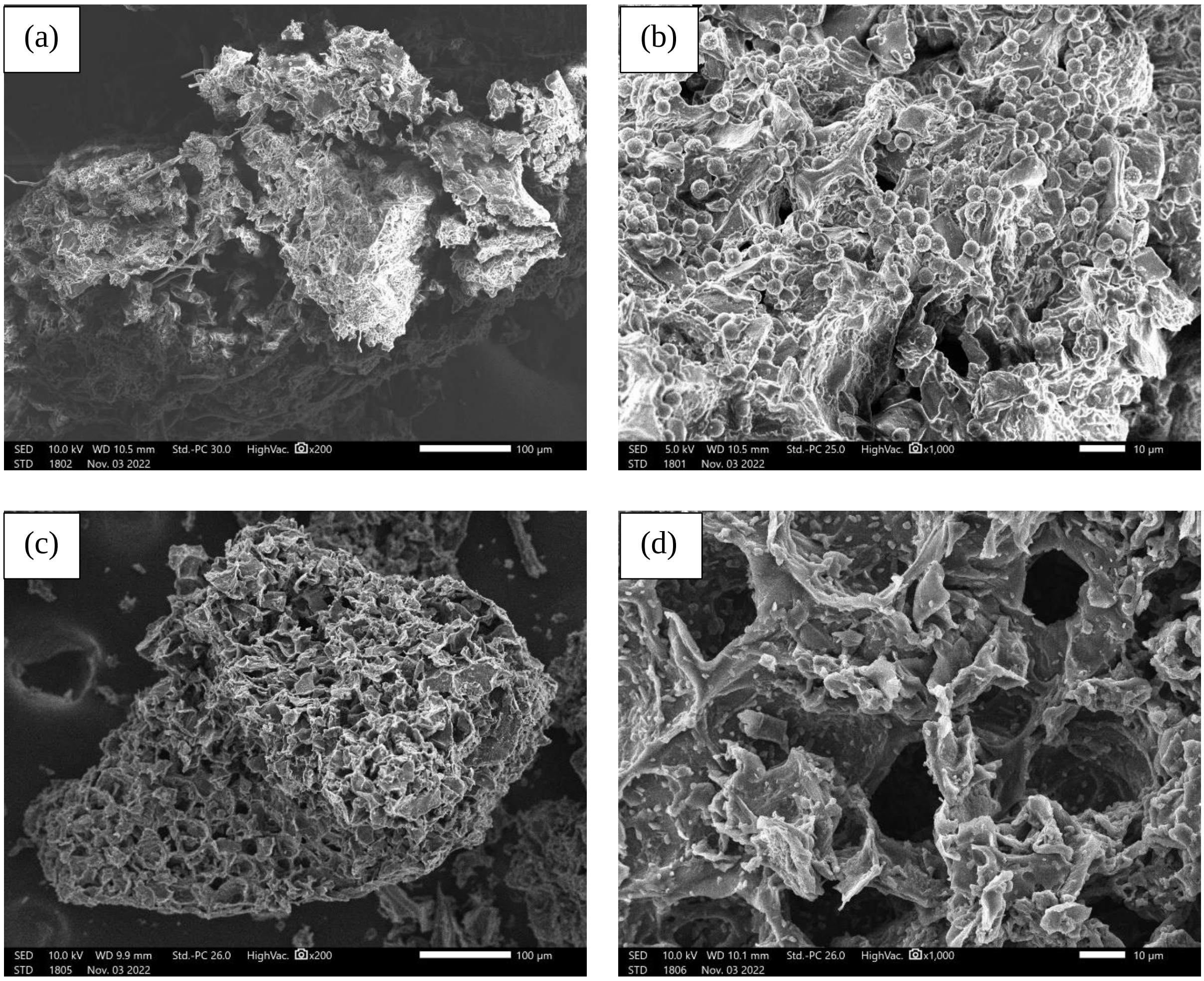

Figure 4.2 SEM images (a) raw SCG at 200 magnification scale, (b) raw SCG at 1000 magnification scale, (c) SCG biochar at 200 magnification scale, and (d) SCG biochar at 1000 magnification scale

Table 4.1 EDX analysis of raw SCG and SCG biochar

| **Samples** | **Elements** | **Mass (%)** | **Atomic (%)** |
|---|---|---|---|
| Raw SCG | C | 54.52 | 61.49 |
| | O | 45.48 | 38.51 |
| SCG biochar | C | 87.76 | 90.52 |
| | O | 12.24 | 9.48 |

### 4.2.2 Textural Analysis by Nitrogen Adsoption-Desorption ($N_2$ Physisorption)

Brunauer-Emmet-Teller (BET) aims to explain the physical adsorption of gas molecules on a solid surface and an important analysis for the measurement of the specific surface area of materials. BET analysis output of the raw SCG and SCG biochar are shown in Table 4.2 below. The obtained data show that the BET surface area of raw SCG was 9.1960 $m^2/g$, compared to 15.3282 $m^2/g$ in case of the SCG biochar. The surface area increases significantly following the calcination process of SCG. The obtained data revealed the presence of one type of pores in both samples which is mesopores (pore size is 2 – 50 nm). The average pore width of SCG biochar was a bit higher compared to the raw SCG. The average pore width of raw SCG and SCG biochar were 46.187 Å (4.6187 nm) and 47.796 Å (4.7796 nm) respectively. Thus, both samples were categorized as mesopores.

The surface area of the biochar is a vital parameter since its sorption and ion exchange properties are directly related to its surface area (Amalina et al., 2022). The increase in average pore width, specific surface area and total pore volume shown that the biochar has good adsorption performance and that the pores of biochar are very likely to become shelter places for microorganisms, which affects the abundance of microorganisms in the anaerobic digestion system, consequently affecting the anaerobic efficiency (Wang et al., 2022).

Table 4.2 Brunauer-Emmet-Teller analysis of the raw SCG and SCG biochar

| **Parameters** | **Raw SCG** | **SCG Biochar** |
|---|---|---|
| BET Surface Area ($m^2/g$) | 9.1960 | 15.3282 |
| Total pore volume ($cm^3/g$) | 0.010 | 0.017 |
| Average pore width (Å) | 46.187 | 47.796 |

### 4.2.3 Functional Group Identification by Fourier Transform Infrared (FTIR)

FTIR analysis was used to investigate the function group on the surface of spent coffee ground before and after the thermal treatment method. The KBR spectra of the raw SCG and SCG biochar are presented in Figure 4.3. The two main peaks are at 3400 and 1600$cm^{-1}$. The first peak is due to the -OH bond while the second is characteristics of C–C bond. Spectra of both samples revealed the absence of the peak at 3411cm$^{-1}$ SCG biochar which is associated with the hydrogen-bonded OH from water, implying the complete dehydration during thermal treatment method. Typical moieties present in the SCG were also identified which is the C–H stretching vibration that was detected at 2916 and 2847cm$^{-1}$. The peak at 900cm$^{-1}$ is associated with the C–O vibration (El-Azazy et al., 2021). The peak stayed the same in the spectrum of the SCG biochar at the same wavenumber.

The essential functional groups present at surface of biochar that increase its sorption properties include hydroxyl (−OH) group. The main factors that influence surface functional groups of biochar are biomass (Yaashikaa et al., 2020). In addition, when other properties such as pH, surface area and porosity are increased there is a chance of reduction in biochar functional groups (Yaashikaa et al., 2020).

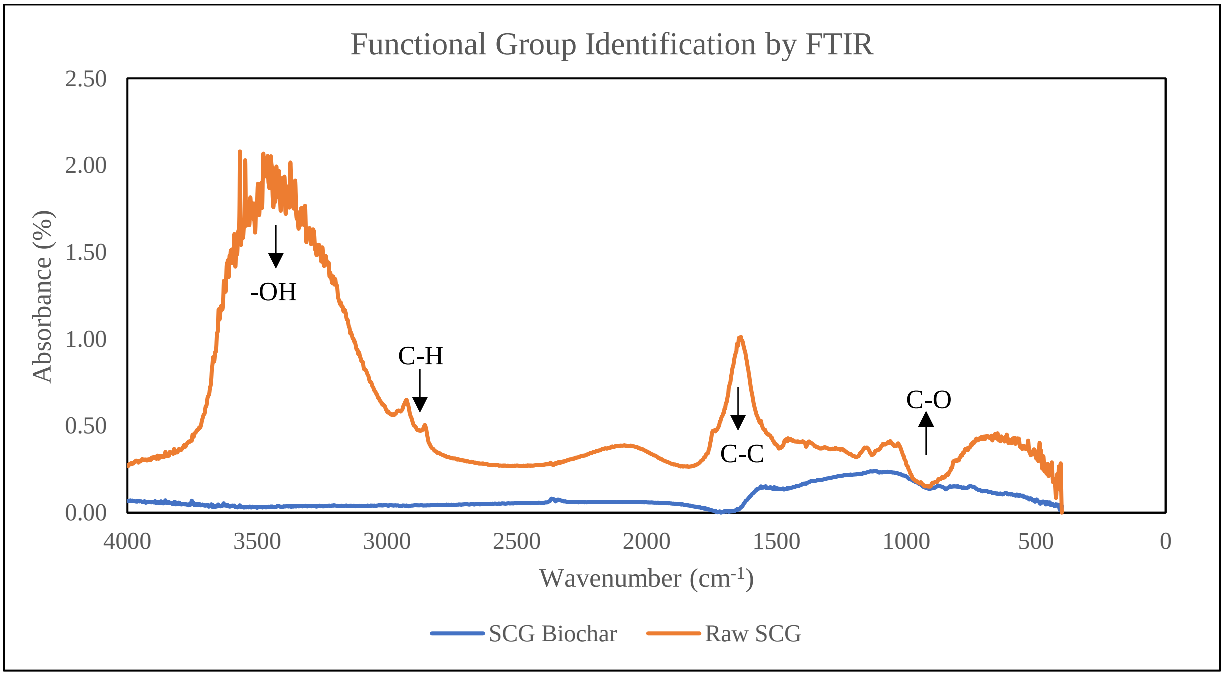

Figure 4.3 Fourier Transform Infrared analysis of raw SCG and SCG biochar

## 4.3 Parameters Affecting Anaerobic Digestion (AD) Process

This study demonstrates in enhancing the AD process of food waste by SCG biochar. The performance of anaerobic digesters can be controlled by studying and monitoring the variation in parameters of AD process. To promote the efficiency of AD, the study of optimum operating parameters is required. Hence, this study will evaluate three parametric effects of AD which are effect of the amount of biochar dosage, effect of the amount of feedstock and effect of pH. The results of these evaluation will be presented and discussed in the following section.

### 4.3.1 Effect of the Amount of Biochar Dosage

The parametric effect of the amount of biochar dosage on AD was analysed in five different amounts of biochar dosage which are 1g, 3g, 5g, 7g and 9g. In each anaerobic digester, 500g of food waste mixture was placed in a bottle (digester) then each digester was put at the same place that gets the most sunlight and all digesters will be observed for 14 days. Figure 4.3 shows the results of the methane yield at five different amounts of biochar dosage.

As depicted in Figure 4.3, the biogas yield increased from 0.594ml/g to 1.859ml/g when the dosage of biochar escalates from 1g to 3g. The yield of biogas keeps increasing from 5.652ml/g to 8.412ml/g as the dosage of biochar increases from 5g to 7g. However, the yield of biogas begins to decline from 8.412ml/g to 0.249ml/g when the dosage of biochar exceeds 7g. This was mainly due to the fact that moderate biochar addition could effectively alleviate VFA accumulation resulting in higher levels of methanogenic activity, while higher amount of biochar would lead to more propionic acid accumulated in the digester thereby reducing the AD process stability (Zhang et al., 2019). Thus, the effective amount of biochar dosage was noted as 7g. This amount of biochar dosage is then implemented in subsequent study.

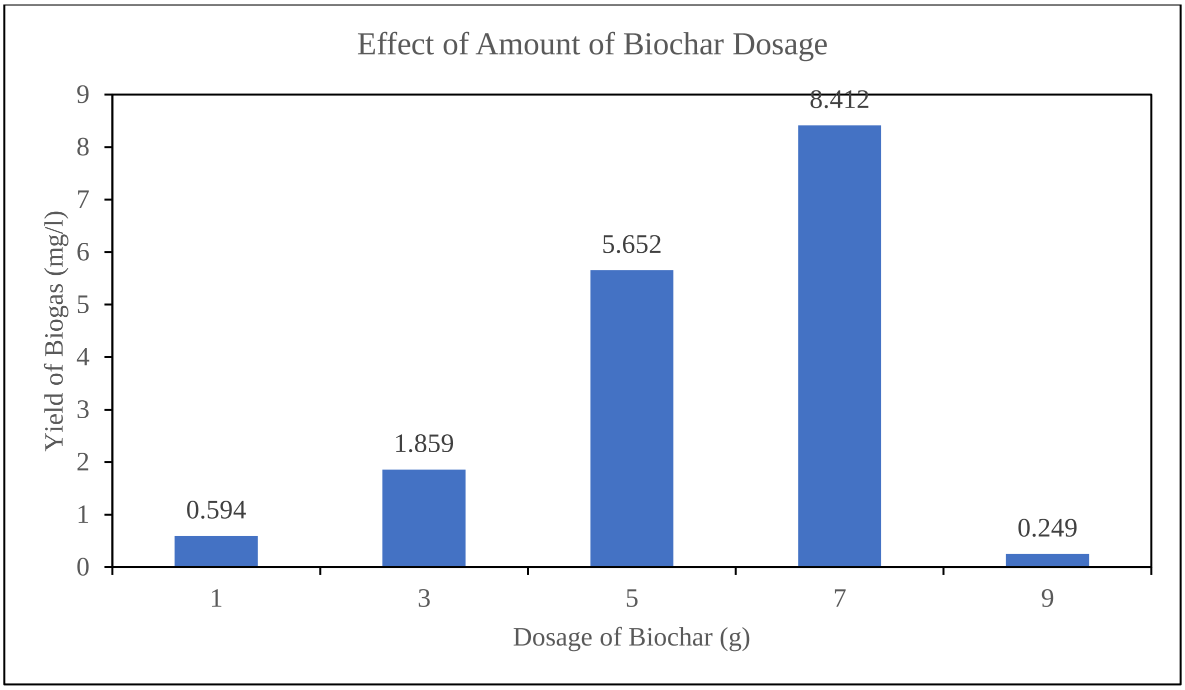

Figure 4.4 Yield of biogas by using different amount of biochar dosage in AD

### 4.3.2 Effect of the Amount of Feedstock

To maximize the operating conditions of the AD, another significant factor effecting the biogas yield was the amount of feedstock used in the anaerobic digesters. To assess this parameter, the biogas yield was observed in five different values of feedstock, ranging from 100g, 200g, 300g, 400g and 500g. This operation was done under fixed condition of biochar dosage (7g) and pH value (5). Figure 4.4 shows the effect of the amount of feedstock on yield of biogas.

As depicted in Figure 4.4, the yield of biogas increased from 0.479ml/g to 3.593ml/g when the amount of feedstock increased from 100g to 200g. Then, the yield of biogas decreased to 2.555ml/g when the amount of feedstock increased to 300g. However, the amount of water displaced during water displacement method for 300g of feedstock was higher, which was 800ml compared to 200g of feedstock which was 750ml. Nevertheless, the yield of biogas increased from 3.629ml/g to 6.131ml/g when the amount of feedstock used was 400g and 500g respectively. The findings demonstrate that the highest biogas achieved was at the highest amount of feedstock used in the AD. The amount of feedstock used significantly affected the AD process. The higher the feedstock used, resulted with maximum biogas production (Basumatary et al., 2021).

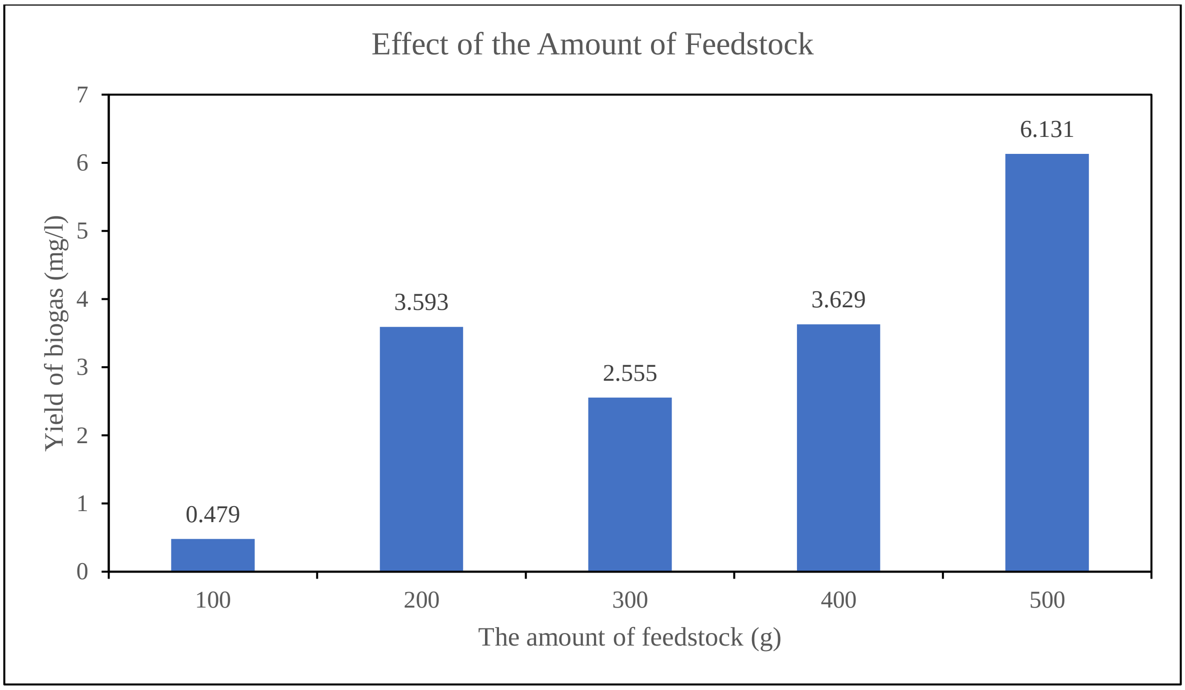

Figure 4.5 Yield of biogas using five different amounts of feedstocks in AD

### 4.3.3 Effect of pH Value

Other factors affecting the biogas yield to maximize the operating conditions of the AD was the pH value in anaerobic digesters which measures the feedstock concentrations. To assess this parameter, the biogas yield was observed in five different values of pH, ranging from 5, 6, 7, 8 and 9. This operation was done under fixed conditions of dosage of biochar (7g) and amount of feedstock (500g). Figure 4.5 shows the effect of the pH value on yield of biogas.

As depicted in Figure 4.5, the yield of biogas increased from 0.077ml/g to 0.364ml/g and further increased to 13.528ml/g when the pH value used were 5, 6 and 7 respectively. However, there are significant decrement of biogas yield which was 0.240ml/g and 0.144ml/g when the pH value used were 8 and 9 respectively. At the initial state of digestion, pH values for food waste were observed to be in the acidic range between 4.8 – 5.6. Previous study has reported that the optimum pH range for normal digestion is 6.6 – 7.6 in an AD system (Feng et al., 2020). Thus, the effective pH value for this study was noted as 7. This is because in the digesters with too low and too high pH values, the methanogenic activity was seriously inhibited, and then keeping the biogas yield at a low level (Zhang et al., 2019). This means that a more suitable range for microbial activity was obtained at neutral pH (7).

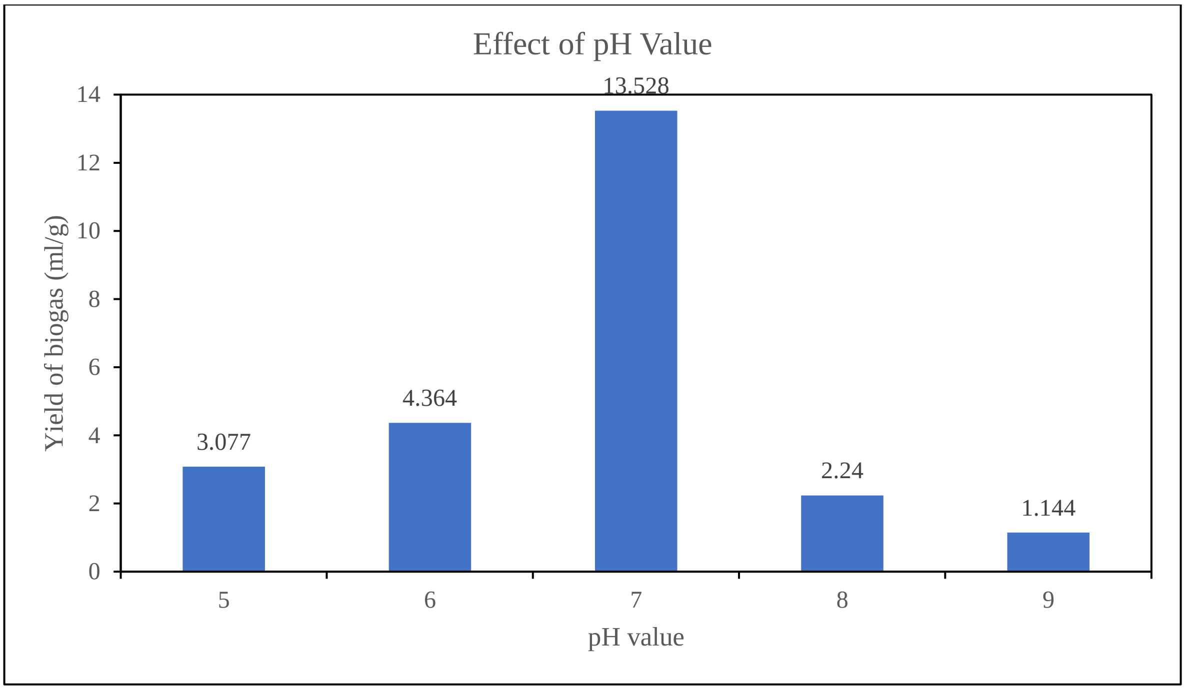

Figure 4.6 Yield of biogas using five different values of pH in AD

## 4.4 Identification of Biogas Composition through Online Gas Chromatography

Along with the biogas yield, the composition of the biogas produced also is crucial for a high-quality biogas. Therefore, the biogas produced from the AD was analysed to study the potential of food waste as a feedstock for biogas production. This process is done by employing online gas chromatography (GC-TCD) to identify and quantify the composition of biogas produced from two anaerobic digesters, one with added biochar and one without biochar, that were made from the optimized conditions (500g of feedstock at 7 pH value with added 7g of biochar) that resulting in the highest biogas yield. The result of these evaluation will be presented and discussed in the following section.

### 4.4.1 Analysis of Biogas Composition

It was revealed that the highest biogas yield achieved when the AD used 500g of feedstock at 7 pH value with 7g added biochar, resulting in 13.528ml/g of biogas. However, two anaerobic digesters were made, one with added biochar and one without biochar, to compare the biogas composition that were produced. The biogas contents of these samples were determined through GC-TCD. As characterized by GC-TCD, it can be seen that there were proportions of components produced from both AD which are tabulated in Table 4.3.

As tabulated in Table 4.3, there are four types of biogases present in both samples which include carbon dioxide ($CO_2$), oxygen ($O_2$), nitrogen ($N_2$), and methane ($CH_4$). For AD without added biochar, there were only two gases produced from the process which were $CO_2$ and $O_2$ which make up for 96.971% of total biogas which primarily dominated by $CO_2$ with 89.18%. Meanwhile, for AD with added 7g of biochar, there were four gases produced from the process which were $CO_2$, $O_2$, $N_2$, and $CH_4$ which make up for 95.343% of total biogas produced which also primarily dominated by $CO_2$ with 58.626%.

As shown in the Table 4.3, there were no methane gas produced from the AD without biochar meanwhile AD with added biochar contained 6.493% of methane gas. This may be caused by types of feedstocks used in the AD which was carbohydrate-rich food waste. Carbohydrates show the fastest conversion rate, but methane yield is not so elevated (Morales-Polo et al., 2018). Carbohydrate-rich substrates, in food waste terms, such as fruits, are enriched with simple sugars and disaccharides (Hagos et al., 2019). Anaerobic degradation of simple sugar may result in VFA formation and accumulation, leading to a decrease in methanogenesis inhibition (Morales-Polo et al., 2018). However, adding biochar in an AD prevents mild ammonia inhibition and increased methane yield (Pant & Rai, 2021). Biochar also helps in reducing the methanogenic lag phase by 30.3% and increases methane production in AD (Pant & Rai, 2021). Thus, AD with added biochar produced more methane than the AD without biochar.

Table 4.3 The composition of biogas produced from two ADs

| Samples | RT | Composition (%) | Name |
|---|---|---|---|
| AD without biochar | 1.413 | 0.000 | |
| | 2.643 | 89.180 | $CO_2$ |
| | 7.622 | 7.791 | $O_2$ |
| AD with added biochar | 1.413 | 0.000 | |
| | 2.640 | 58.626 | $CO_2$ |
| | 7.398 | 0.523 | $O_2$ |
| | 7.866 | 29.701 | $N_2$ |
| | 8.077 | 6.493 | $CH_4$ |

# CHAPTER 5

# CONCLUSION

## 5.1 Conclusion

The primary goal of this study is to produce methane gas from anaerobic digestion (AD) of food waste by adding an additive which is spent coffee ground (SCG) biochar. This study is carried out to identify the potential of SCG biochar as future additive for biogas production as well as to enhance the AD efficiency. To achieve the primary goal of this study, two objectives were established which includes the study of characterization analysis of biochar as additive in AD of food waste from SCG and to analyse the performance of biochar as additive by studying the optimum operating conditions of AD such as effect of biochar dosage, effect of the amount of feedstock and effect of pH value.

Based on previous studies, AD is said to produce around 45 – 60% of methane gas. However, the AD process requires a long operation time to obtain such amount which is around 41 days. To overcome this limitation, SCG biochar is used as additive to enhance biogas yield while minimizing the operation time. To ensure AD process can achieve maximum level of efficiency, the optimum operating conditions such as effect of biochar dosage, effect of the amount of feedstock, and effect of pH value were studied. Each optimum operating condition were investigated by varying the dosage of biochar ((1, 3, 5, 7 and 9) g), the amount of feedstock used ((100, 200, 300, 400 and 500) g), and pH value (5, 6, 7, 8, and 9). From this investigation, the highest biogas yield achieved was when the condition used 7g of biochar dosage with 500g of feedstock, and 7 pH value, resulting in 13.528ml/g of biogas yield.

For the determination of the methane gas, GC-TCD was employed on the two anaerobic digesters, one is added with biochar while the other one is not, to identify and quantify the components present. The identification depicts that AD with added biochar produced 6.493% of methane gas meanwhile, AD with no biochar only contained $CO_2$ and $O_2$ gases. Although the methane production was not as much as the $CO_2$ production, but the objective of this study is achieved. Biochar does enhance the methane production in AD.

## 5.2 Recommendation

Despite the potential of spent coffee ground (SCG) biochar to produce methane gas in the anaerobic digestion (AD) process, the total composition of methane gas produced is relatively low. Therefore, few improvements should be taken to make use this potential to the maximum efficiency. In this aspect, several recommendations that should be considered for future improvements includes:

1. Improve the condition of AD to improve the biogas yield.
2. Conduct an optimization study of feedstock types to find the most suitable feedstock types for enhancement of methane through AD.
3. Improve the performance of SCG biochar by treating it with other treatment methods.

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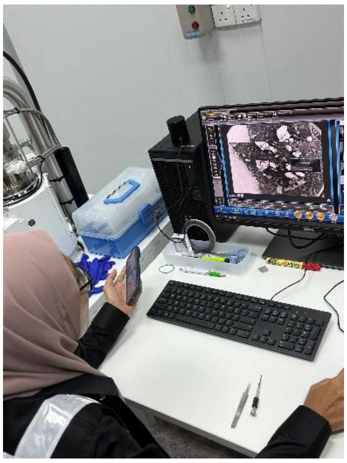
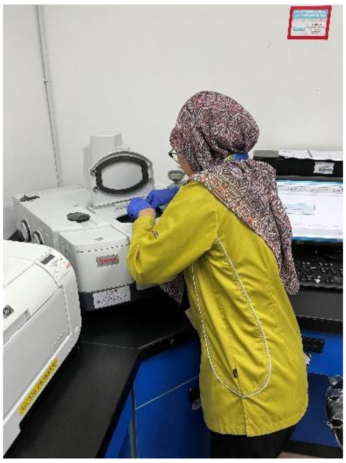
# APPENDICES

## Appendix A: Preparation of anaerobic digestion

# Appendix B: Characterization analysis of raw spent coffee ground and biochar





## Appendix C: Observation of biogas produced and water displacement method