BIOMASS VALORIZATION: ASSESSMENT AND CHARACTERIZATION OF BIOMASS WASTE FOR VALUABLE PRODUCTS

A DISSERTATION PRESENTED TO THE DEPARTMENT OF MATERIALS SCIENCE AND ENGINEERING (MSE)

AFRICAN UNIVERSITY OF SCIENCE AND TECHNOLOGY

BY

EZEALIGO UCHECHUKWU STELLA

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TITLE PAGE

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A Thesis Presented to the Department of Materials Science and Engineering

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In Partial Fulfilment of the Requirements for the Degree of

Doctor of Philosophy (Ph.D.) Degree

By

Ezealigo Uchechukwu Stella

Abuja, Nigeria

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CERTIFICATION

This is to certify that the thesis titled "**BIOMASS VALORIZATION: ASSESSMENT AND CHARACTERIZATION OF BIOMASS WASTE FOR VALUABLE PRODUCTS**" submitted to the school of postgraduate studies, African University of Science and Technology (AUST), Abuja, Nigeria for the award of the Doctoral degree is a record of original research carried out by Ezealigo Uchechukwu Stella in the Department of Materials Science and Engineering.

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ABSTRACT

Research in the use of biomass residues has a huge interest as their potentials span a wide range of applications. Processed residues are useful as green energy such as biofuel (pellets and briquettes), animal feed, antioxidants, and activated charcoal for filtration and even carbon capture. With this in mind, my doctoral research covers the assessment of biomass residues generated in Nigeria for bioenergy. Also, *Ficus benjamina* fruit, identified as a biomass waste, was characterized for its value addition in bioenergy application. The latter fruit was further characterized for its value as a potential feed substrate for animals as well as the chemical source for industrial applications. The results from the research within this framework include the following.

First, a proper bio-resource assessment, particularly, biomass residues availability and potential were investigated. This is a key requirement for an efficient and functional bioenergy sector in Nigeria, proposing to generate biofuel from agro-waste materials. In this study, computational and analytical approaches with mild assumptions were employed to evaluate the bioenergy potential in agricultural residues, including municipal solid and liquid waste. This assessment was performed using data from 2008 to 2018. The available technical potential of 84 Mt yielded cellulosic ethanol and biogas of 14,766 ML/yr (8 Mtoe) and 15,014 Mm^3/yr (13 Mtoe), respectively. The residues gave more biogas than cellulosic ethanol from the same amount of residue potential. The energy potential from residues in Nigeria may be tailored towards biogas production for diverse applications ranging from heat to electric power generation and therefore holds great potential in solving the current electricity crisis in Nigeria. It will also position the nation towards achieving the 7th sustainable development goal (SDG 7) on clean and affordable energy.

Secondly, having identified that some residues may be limited in supply due to seasonality and multiple applications for various purposes, there is a need to continue a search for more plant waste that is resourceful as a potential feedstock. *Ficus benjamina* (FB) is an ornamental plant that produces nonedible fruits considered as waste. These fruits have no defined application, hence, identifying the potential in these fruits for possible valorization is necessary. Detailed preliminary characterization was performed to determine its suitability as a biofuel feedstock. The whole fruit (pulverized) was characterized by scanning electron microscopy (SEM), energy dispersive X-ray (EDS), thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and bomb calorimeter. In addition, the physical, thermal, and chemical properties of FB fruits for potential biofuel application was determined using the proximate and ultimate analyses. Pulverized *Ficus benjamina* fruits (PFB) have a porous morphology that makes them less dense and a crystallinity index of 25.5%. The moisture, ash, volatile matter, and fixed carbon contents were 9.29, 6.26, 64.35, and 20.10%, respectively. The higher and lower heating values are 19.74 and 18.55 MJ/kg, respectively, and are comparable to other biomass feedstock. The results establish the possibility of using PFB as a solid biofuel.

Thirdly, another possible approach in valorizing FB fruit focuses on other value products and benefits for livelihood. On this basis, the nutritional analysis, as well as the identification and quantification of micro and macro-nutrients and amino acid profile, were performed. HPLC and GC-MS were used to investigate the sugar profile of the water extract and the chemical content on the extracts obtained with solvents (ethanol, n-hexane, and ethyl-acetate), respectively. Found in FB fruits were: eighteen (18) amino acids, diverse micro- and macro mineral content, metabolizable sugars (such as galactose and glucose), and other chemicals, including phytochemicals. In addition, these fruits showed low anti-nutritional factors such as

phytate and tannins. From these findings, FB fruits offer diverse biological potential and functions and may be a prospective bio-resource for animal feed. The high fiber content reveals rich lignocellulose for bowel bulkiness. This result indicates that the fruits of FB can offer health benefits and can serve as a biomaterial. Thus, FB fruits may possess the potential as an additive material for animal feed, and phytochemicals for industrial and pharmaceutical uses.

Keywords: Biomass residue, valorization, *Ficus benjamina* fruits, bioenergy potential, feedstock, waste.

DEDICATION

This doctoral research is dedicated to my Maker, and in gratitude for life and health. I am truly indebted to Him.

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LIST OF PUBLICATIONS

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ABBREVIATIONS

FB	*Ficus benjamina*
PFB	Pulverized *Ficus benjamina*
XRD	X-ray Diffraction
XRF	X-ray fluorescence
EAC	Ethyl acetate fraction
EAW	Ethanol extract after water
FEE	Ethanol extract
nHEX	n-Hexane Extract
TEA	Technical energy available
CO_2	Carbon dioxide

CHAPTER ONE

Introduction and Background

1.0 Introduction

From the scientific perspective, biomass is referred to the mass of living organisms that include plants, animals, and microorganisms [1]. In other words, any material with cellulose, lignin, sugars, fats, and proteins from the biochemical perspective can be considered as biomass [2]. The biomass from the plant is the weight of the dry matter produced after being dried to constant moisture content, and about 90% of biomass is derived from the fixing of carbon into organic compounds via photosynthesis.

The classification of plants biomass includes the above- and-below ground tissues such as leaves, twigs, roots, branches of plants, as well as the boles and rhizomes of grasses. Furthermore, such biomass can be categorized as wood and non-wood biomass. Although the former is abundant but has management restrictions that limit usage, owing to the potential harm of deforestation to the environment [3]. However, the valorization of non-wood biomass has become the interest of researchers in diverse scientific fields [4]. The non-wood biomass compared to its woody counterpart is available, cheaper, easier to process, and may require less energy during its transformation into relevant materials via diverse conversion techniques. This non-wood biomass is broadly classified into agricultural residues, native plants, and non-wood plant fibers.

The venture towards obtaining higher values from waste, even to making products for commercialization, is called biomass waste valorization [5]. However, for this research, we prefer the terminology biomass valorization. According to Tuck et al., the term “plant waste” describes any organic material that is separated from the primary material (that is, the initial purpose for its cultivation). For instance, maize is planted for food, but corn stover, stalk, corn cob, and sheaves are waste materials generated from maize plantations. Most times, the waste generated from these plant materials can be more than the much-needed food crop produced in terms of quantity per time. Nearly all wastes have some value. For example, soil nutrients are replenished with compost from stover, while lignin (a waste product) can serve a biofuel purpose to power paper mills. Beyond the traditional uses of waste, which are greatly limited in their optimal utilization in this dispensation, this waste can be processed into modern, eco-friendly, and valuable products. As a result, we concentrate on ways of getting a sophisticated value from wastes that meets the needs of this high-tech age. The latter is implicated in, first, quantifying the value of the various waste or residues generated by means of biomass assessment that also incorporates the conversion strategies required for optimized value from the waste.

Furthermore, there is a need to identify other biomass residues that are exclusively considered as waste for their compositional assessment and evaluation. The characterization of these residues considers their chemical contents such as polysaccharides, lignin, triglycerides (from fats and oils), and proteins. In addition, detailed pre-treatment methods are necessary also for the monomeric chemical compounds that are vital building blocks for valuable products. Exploiting waste for a profitable course, however, requires a positive multidisciplinary approach. Hence, it is pertinent to emphasize on the optimization of the conversion routes to ensure that valorization of the various matter in residual biomass contributes to profitable products.

1.1 Market opportunity

Agro wastes are organic materials and are generated yearly in huge quantities (i.e., more than hundreds of megatonnes (Mt)/year). Obi et al. (2016) reported that about 998 million tonnes of agricultural waste are produced each year, and 80% of this waste are organic. [6, 7]. These wastes are potential bio-resource materials that have valuable applications in various fields [2, 8]. Such enormous potential in biomass waste has contributed to a new economy called a bio-based economy and the marketable products include chemicals - e.g., lubricants, surfactants, monomers for plastics, fibers, and industrial solvents. These chemicals are raw materials for industries [9, 10, 11]. These valued products from agro waste although are economic and eco-friendly compared to the synthesized equivalent, may contribute slightly to the sustainability of chemical production because the demand for these chemicals is higher than what the available residues or feedstock can generate. However, these chemicals can serve its purpose in some small-scale industries. It is noteworthy that the gait of research in biomass valorization is revving, as chemical manufacturers indicate increased interest in renewable feedstock. It is noteworthy that the poor management of waste contributes to climate change and CO_2 emission. However, the latter challenges can be mitigated by substituting biomass-based resources for fossil carbon-based, which can lead to carbon-neutral products, if the entire process is sustainable using a life cycle assessment approach. Here in this research, we focused on agricultural and municipal wastes. With rapid urbanization and the increasing global population (about 9 billion), municipal waste is presumably an important source of biomass waste. Also, the demand for chemical products is high in economically developing economies, like Nigeria. Based on the aforementioned, biomass residues in Nigeria are reviewed. The assessment of biomass residues is a vital requirement towards establishing a biomass resource market and a bio-based economy.

1.2 Biomass residues

Plants are food sources and may be processed into various food products. Plant parts such as the leaves, stem, husk, and stalk, left after the harvest of crops are called crop residues. Most times, the latter is considered as waste and often burnt on the farm. These plant wastes are resource materials that are available for different purposes such as the replenishing and preserving of the soil nutrients through composting and mulching. This bulk waste from the harvest possesses lots of carbon amongst other nutrients like nitrogen (N), sulphur (S), phosphorus (P), and potassium (K). Season after season, a large amount of residue is produced in the northern, western, southern, and middle belt regions of Nigeria after the harvest period. These huge residues constitute a nuisance to the environment due to how poorly they are treated. The residues generated from crops are categorized into field-based and process-based [12]. The various sources of biomass residues are as follows:

• Agricultural residues: husks, pods, bagasse, leaves, corn stalks, straw, seed hulls, nutshells.

• Forest residues: tree branches, wood waste, bark, sawdust, timber wane, mill scrap, forest pruning.

• Municipal waste: municipal solid waste (MSW), sewage sludge, refuse-derived fuel, food waste, wastepaper, and yard clippings.

• Energy crops: *Jatropha curcas*, poplars, willows, switchgrass, miscanthus, canary grass.

• Biological: animal waste, aquatic species, and biological waste.

In contrast to the conventional wood-burning practice for heating and cooking in underdeveloped nations, modern bioenergy use biomass for contemporary heating, power production, and transport fuels. These residues can play vital roles in bioenergy production.

1.3 Current Status of Bioenergy

Here are the specific data regarding the status of bioenergy both worldwide and in Nigeria, and their prospects for the forthcoming years. Biomass has significant potential to boost energy supplies in populous nations with rising demand, like Nigeria.

Global primary demand for bioenergy was almost 65 EJ in 2020 (12% of total), of which about 90% was solid biomass. Some 40% of the solid biomass was used in traditional cooking methods which is unsustainable, inefficient, and polluting, and was linked to 2.5 million premature deaths in 2020. The use of solid biomass in this manner falls to zero by 2030 in the NZE, to achieve the UN Sustainable Development Goal 7. Modern bioenergy in 2020 <40 EJ (7% of total) but >100 EJ (20% of total) in 2050 according to NZE [13].

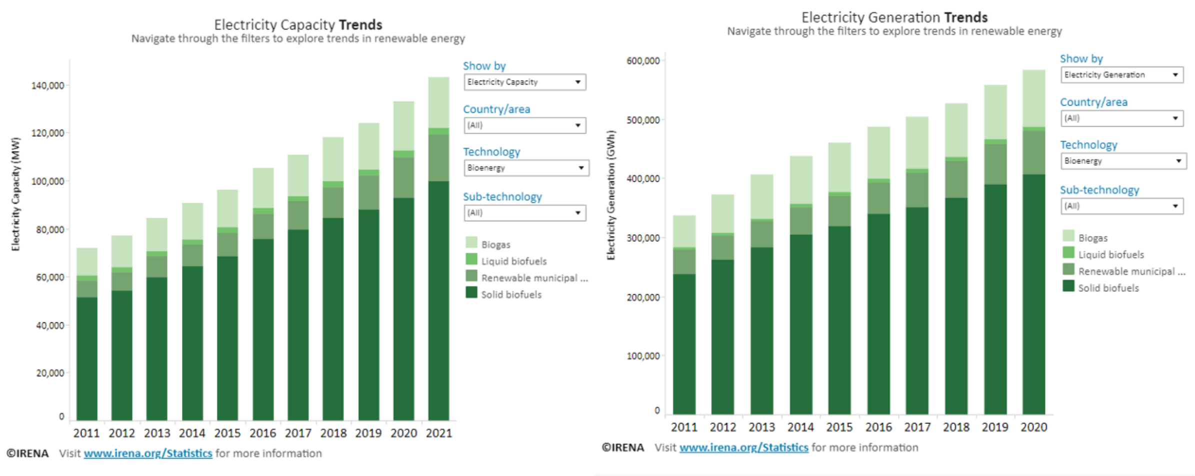

Fig. 1.1: Global installed bioenergy capacity and electricity generation in Nigeria

Installed bioenergy capacity worldwide in 2018: 118 MW, out of which 84 MW solid biofuels > 18 MW (70%) biogas > 12 MW (16%) renewable municipal waste (12%)> 2.4 MW (2%) liquid biofuels. Electricity generation: 445 GWh, out of which 366 GWh (70%) solid biofuels > 91 GWh (16%) biogas > 63 GWh (12%) renewable municipal waste > liquid biofuels 7 GWh (2%) [14].

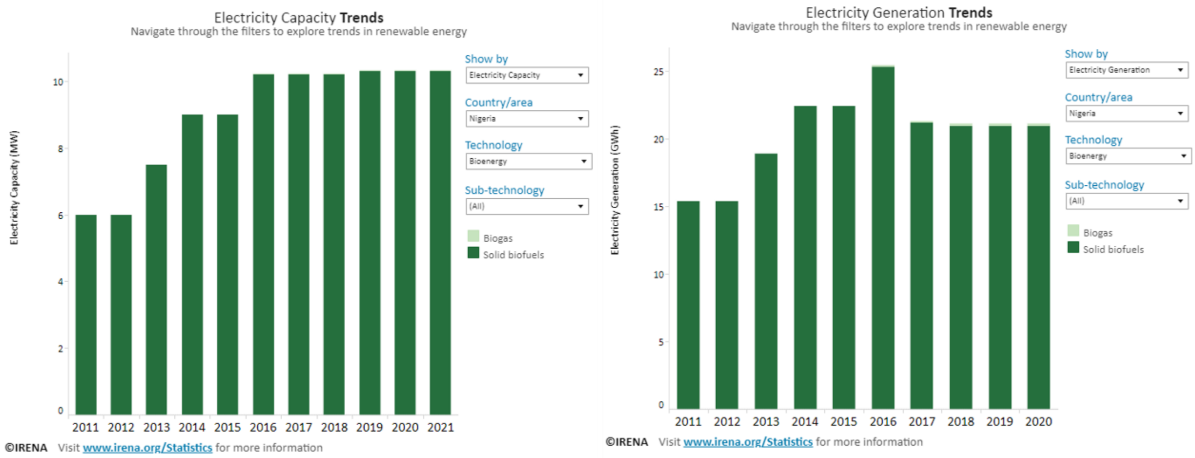

Fig. 1.2: Installed bioenergy capacity and Electricity generation in Nigeria

Installed bioenergy capacity in Nigeria in 2018: 10MW (10.20 MW solid biofuels and 0.03 MW biogas). Electricity generation from bioenergy in Nigeria in 2019: 21GWh, 20.96GWh from solid biofuels and 0.16 GWh from biogas. [14].

1.4 Overview and Motivation

The increasing global population, in Nigeria, has created a high demand for agricultural products to meet the increased need for food supply and security. The additional positive consequence of the latter is an increased vegetation that aids the mitigation of climate change for a sustainable ecosystem that tends towards CO_2 balance in the environment. This argument holds true after a proper life cycle assessment (LCA) is performed. However, there will also be lots of waste generated from farmland after harvest, crop process, horticulture, foods process, and municipal waste. These wastes are either incinerated or dumped into the water bodies or by the roadside. Such acts pollute the environment, generating offensive odor. In addition, the pollution of water, soil, and air cause various diseases. The poor waste management system prevalent in Nigeria coupled with the improving treatment techniques for biomass residues demand that these residues be valorized. These wastes based on their compositions are sources of raw materials of diverse forms for different purposes. For these reasons, this project focuses on applying the waste-to-wealth approach to these residues and bio-wastes to some human needs while curbing the challenges of pollution.

Research Objectives

The research documented in this thesis presents first, the assessment of biomass of residues available in Nigeria for their bioenergy potential, especially towards modern biofuels. However, there is a need to identify other biomass resources that can be included in the list of feedstocks for bioenergy or biomaterials for other applications such as animal feed and feed composition, thus reducing the competition for biomass residues for diverse usage. The latter concern was the second focus of the research work. Hence, we examined *Ficus benjamina* fruits for their potential use for bioenergy and potential substitute to feedstock.

The following are the main research objectives of this research:

1. Biomass assessments are available for residues generated in Nigeria. However, detailed computational analysis and estimation towards the conversion of these residues into modern bioenergy potential are yet to be done. This work seeks to evaluate the biomass residues generated for the past 11 years (2008-2018) to understand the amount of residue generated, consistency in residue generation, bioenergy (modern biofuel) that are obtainable from these wastes and their applicability to energy production in Nigeria.

2. After the estimation of the common residues from agro sources, we realized that the low technical potential of these residues; and their seasonality concerning residue generation is a challenge to sustainable biofuel production. This study seeks to identify

new biomass residue resources. Hence, *Ficus benjamina* fruits were characterized for their potential in biofuel applications.

3. Beyond their potential as biofuel, the fruits of *Ficus benjamina* can be used for other purposes. This study characterized the nutritional composition and the extracts to identify the chemical potentials for diverse benefits that can be derived from valorizing these fruits.

1.5 Scope and Organization of Thesis

The thesis consists of six chapters:

Chapter one provides background and introduction of biomass and biomass residues, highlight the types of residues and the concept of valorization of these residues into products of value. A brief insight was provided on the market as well as the economic benefits of the bio-products from biomass residues.

Additional motivation for this work as well as a detailed literature review that serves as the basis for the project was revised in chapter two. This chapter explained the various beneficial products that are obtainable when residues are transformed.

Chapter three considers biomass valorization to bioenergy: an assessment of biomass residues' availability and bioenergy potential in Nigeria; this has been published [15]. Analytical formulas were employed to calculate and estimate both the waste generated from agro-resources (directly or indirectly) and the biofuel potential inherent in these residues.

Here, we narrowed down to a newly identified biomass waste, called *Ficus benjamina* fruits. This study provides insight into the potentials in *Ficus benjamina* fruits. Hence, chapter four covers the preliminary characterization and valorization of *Ficus benjamina* fruits for biofuel application; this has also been published [16].

Still on the valorization of *Ficus benjamina* fruit, chapter five focuses on the nutritional and chemical composition of the fruit and extracts. This chapter covers the potential benefits of these fruits in other fields of applications that are not related to bioenergy.

Finally, Chapter six includes the implications, conclusions, challenges, and suggestions for future work.

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CHAPTER TWO

Literature Review and Fundamentals

2.0 Literature review

Nigeria is brand-named the 'giant of Africa' [1]. This country is fortunate to have the largest population on the African continent (about 206 million persons as shown in Fig. 2.1) with the median age of 18.1[1, 2].

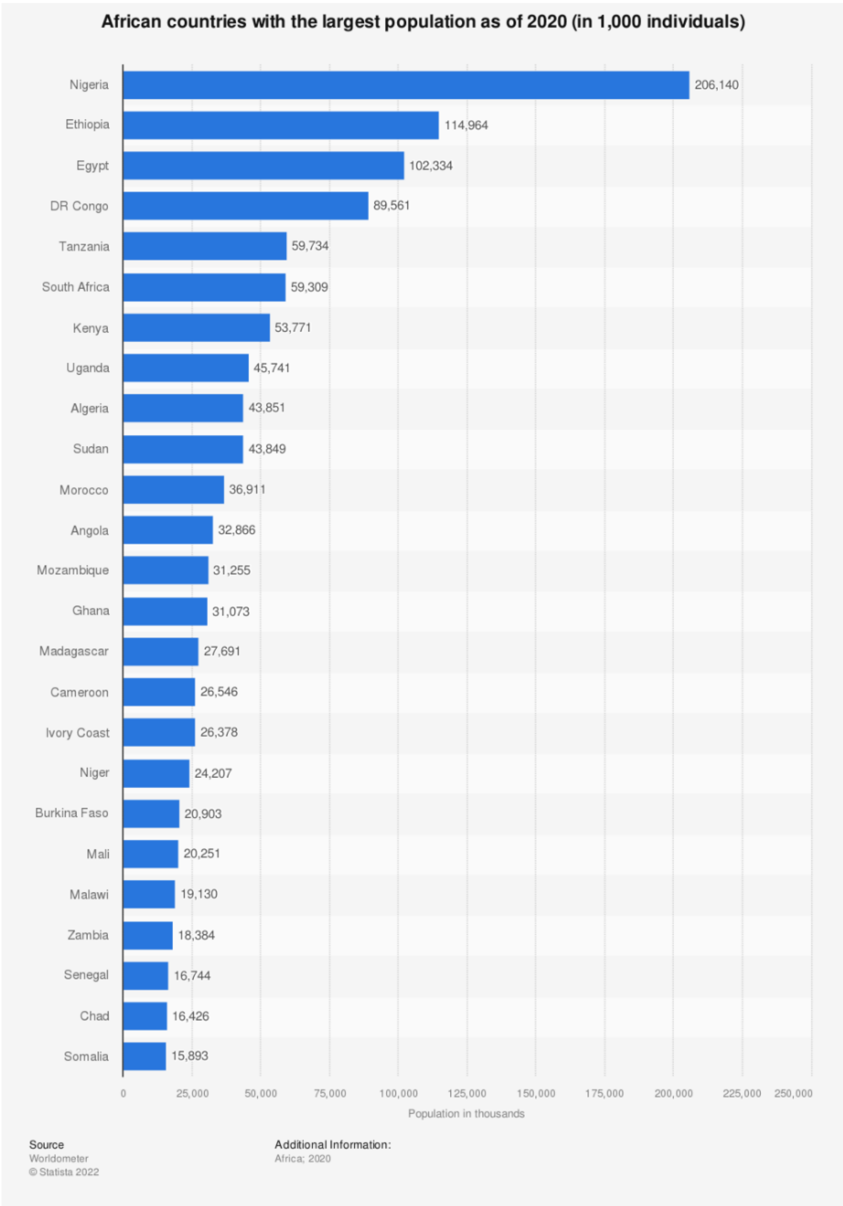

Fig. 2.1: The population of Nigeria compared to other African countries

(https://www.statista.com/statistics/1121246/population-in-africa-by-country/#:~:text=Nigeria%20has%20the%20largest%20population,Africa%2C%20reaching%20102%20million%20people)

The large landmass, climatic conditions, and geographical mapping contribute immensely to the brand name. With the great expanse of land for cultivation, forestry, and water bodies, agricultural business is hugely supported. Nigeria has fertile soil coupled with a suitable climate for crop production. In addition, the human resource (which is largely the youth) is great and can support the massive output of agricultural products. Besides the human capital, Nigeria has a massive solar energy supply that supports biomass resources available for renewable energy production [3]. The map shows the crop zones in Nigeria along with the variation in solar energy and rainfall across the country, categorizing the land into the major and minor cropland (Fig 2.2). Furthermore, Fig. 2.3 shows the relationship between solar radiations to the generation of agro residues.

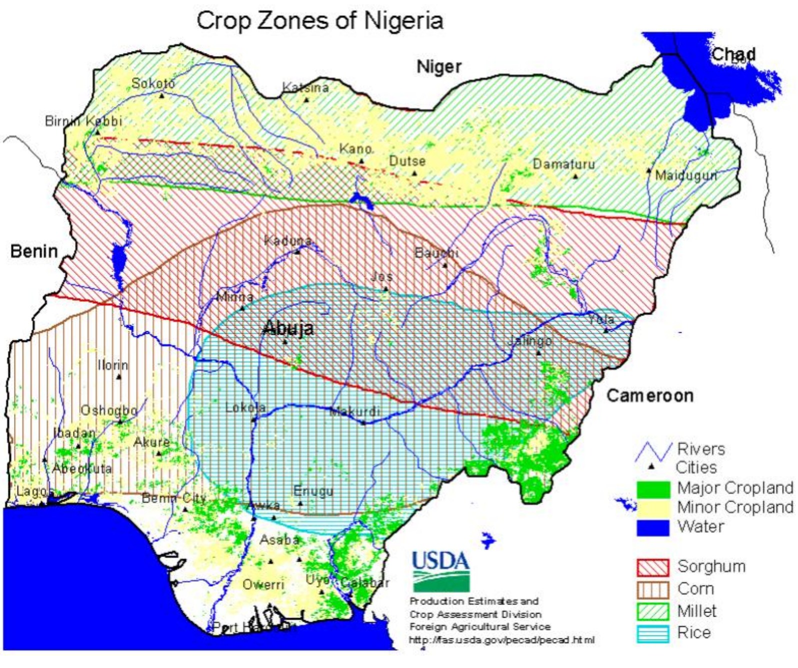

Fig. 2.2: The map of Nigeria displaying the Crop Zones in Nigeria [4]

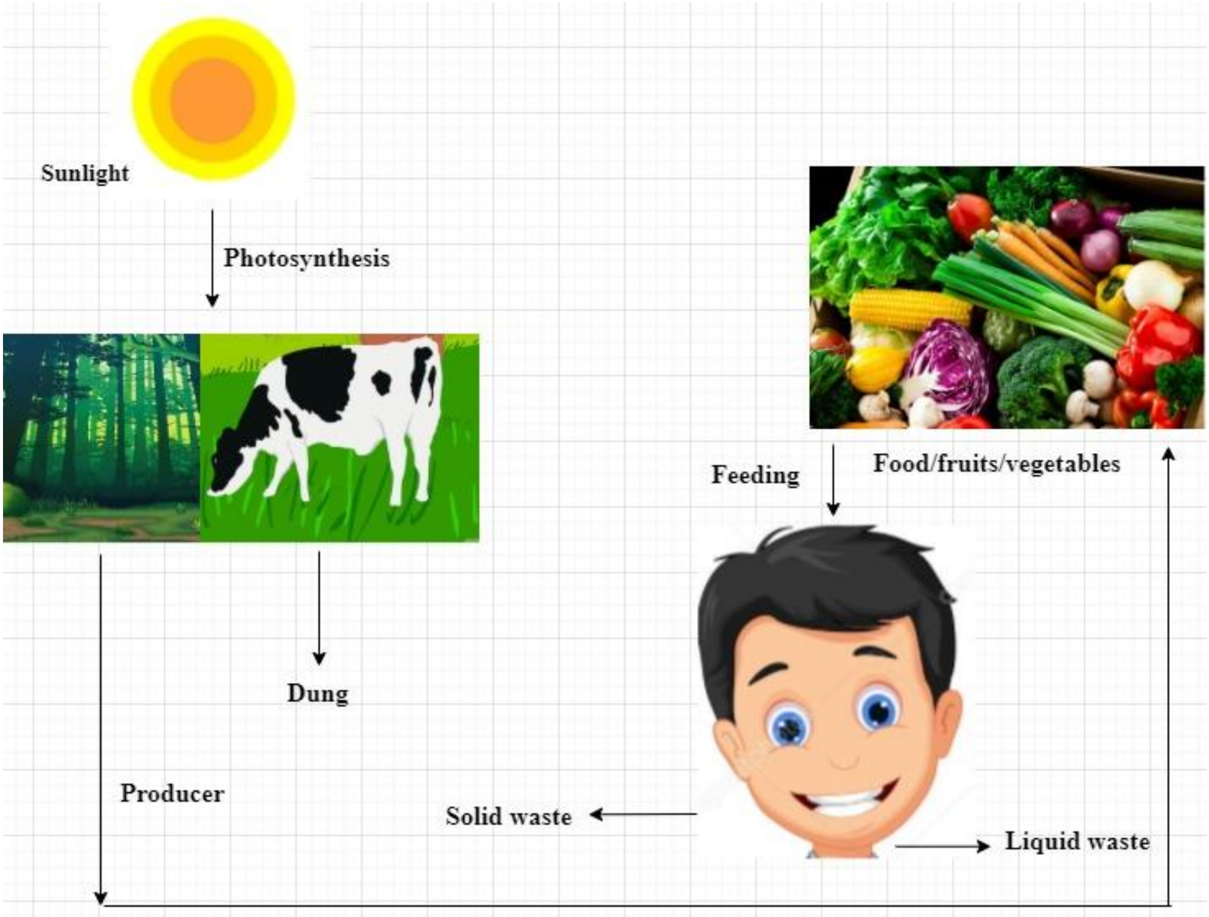

Fig. 2.3: The connection between solar and various sources of biomass waste

The agricultural sector in Nigeria used to be the mainstream economy before the era of crude oil [5]. Olukunle reported that the revolution in this sector holds the key to economic development for most Sub-Saharan countries, like Nigeria [6]. This sector is preferred for the growth in the nation's GDP because it is effective in reducing poverty, considering the large percentage of the population (about 70%) that lives in the rural areas [7]. Also, the functional agricultural sector favorably handles unemployment amongst youths. Beyond the crop production that solves the challenge with food security, the generation of residues for value products is an added advantage. Although, Nigeria currently operates a diversified economy, with individual freedoms that coexist with centralized economic planning and governmental control, she is also actively advancing towards circular economy efforts [8]. Hence, the appropriate transformation of these residues can facilitate the transition of Nigeria through circular economy into a circular bioeconomy. For this reason, biomass residues or agro waste should be valorized. Although such economic growth is possible, it requires optimal development and improvement in the agricultural sector.

Considering the sustainable development of Nigeria's economy and climate change challenge, biomass residues as a renewable energy resource holds great potential. These residues are basic raw materials for different energy carriers **[**9**]**. Several studies into the appropriate use of these residues have emerged and have become a new research field. It is worthy to note that both developing and developed countries deploy the use of biomass residues for some energy supply. However, some developing and developed countries aim to increase biofuel output and usage to cover a wide range of applications such as cooking, drying, electricity, and transportation.

2.1 Biomass Residue Availability

Crop production and its residual waste contribute immensely to sustainable biomass availability than forestry when climate change is considered. Scientific literatures report various ways, where these wastes are used to solve challenges in human needs that includes food security, manure, animal feed, etc. In addition, the benefits include the maintenance of the ecosystem but focuses primarily on climatic sustainability, human health concern, bioenergy preference, renewable raw materials, ecological stability, and the overall sustainable development.

The conventional waste management practice comprises burning, landfill, and incineration. These methods are also common waste treatment techniques, but unfortunately, they incur cumulative negative effects on both the soil, waterbody, air, and the ecosystem at large [10]. The deployment of modern and advanced techniques transform waste into products of value. This concept of valorization is beneficial. Such transformation converts the pollution prone waste residues, usually generated in large quantities, into a variety of well-needed products. Thus, solving the challenge of dealing with pollution. However, there must be a balance between the safe destruction of waste and waste transformation into beneficial products, especially from the perspective of the safety of the ecosystem. One of the crucial factors to consider is the amount of residue or waste generated, and from the previous discussion, there is the possibility of high residue generation in Nigeria. Besides, the landmass, agronomic climate for agriculture and a high population are favorable. However, there is a need to assess and evaluate these biomass residues for their availability towards valorization.

2.2 Crop residue Management

Crop residue management has become a challenge to sustainable environment and ecosystem with consequent effect on climate change in Nigeria. Most farmers in Nigeria still adopt the oldest and fastest way to manage crop residues by burning them. This method eliminates pests, unwanted weeds, and microbes by altering their natural habitat. In addition, burning also adds phosphorus (P) and potassium (K) to the soil from the ashes rich in minerals. The mineral elements reduce soil acidity, but the disadvantage of this waste management technique is that it contributes to the atmospheric pollution that negatively influence the environment, soil, human health, and economic conditions as pollutants are released into the atmosphere. Also, these pollutants contribute to greenhouse gases (GHG). For instance, a tonne stubble upon burning generates 199 kg ash, emits 3 kg of particulate matter, about 2, 60, and 1460 kg of Sulphur, carbon monoxide (CO), and carbon dioxide (CO_2), respectively [10]. Theoretically, that was the amount of CO_2 removed from the atmosphere via photosynthesis. Biomass decomposition releases CH_4 with a considerably more significant warming effect and may become more problematic. During the burning of residues on farmland, the heat kills soil microbes, destroys organic matter, reduces the nutrients on the surface soil, alter the pH and moisture content of the soil. Unfortunately, the latter does not favor crop yield and productivity. However, regardless of the problems of landfills and waste burning, these residues are used appreciably by the rural dwellers to their benefit.

2.3 Traditional application of the biomass residues

2.3.1 Soil enhancement

Crop residues have high and rich nutrient contents and retain soil moisture via composting or green manure. One tonne of paddy straw contains approximately 2.3, 5.5, 25, and 1.2 kg of P_2O_5, N, K_2O, and S, respectively, as well as about 50-70% micro-nutrients [11]. Waste residues prevent soil erosion and the washing away of surface soil nutrients.

The summary of the beneficial use of biomass residues on soil enhancement include the following:

- To increase soil productivity and yields.
- To improve soil structure.
- Cushion the effect of impact (force) on the soil surface by wind shear.
- Improve water infiltration rates and conserves soil moisture content.
- Maintains soil organic matter as plant nutrient is recycled. It provides a suitable environment and a nutrient source for soil organisms, including earthworms and microorganisms.

Also, the environmental benefits include:

- The mitigation of flood into streams as the soil retains water.
- The decrease surface runoff and sedimentation.
- The provision of clean and quality water: by filtering and detoxifying pollutants.

- Also, biomass residues reduce nonpoint source pollution (from pesticides and fertilizers) on land.

2.3.2 Mushroom and fodder cultivation

Crop residues such as straw from rice and wheat make an excellent substrate for growing mushrooms. These edible mushrooms possess nutritional values that are highly recommended for medicinal and therapeutic purposes [11]. In addition, agro waste is processed into freshly grown fodders for animal feed as an alternative to hay and dried grasses. These residues serve as a substrate on which these fodders grow, and the latter are rich in their nutritional content for a healthy animal. The new feeding technique preserves the grasses for other uses yet provide healthy food alternatives for both men and animals. Such application of residues towards cultivating healthy food under controlled conditions improve the dietary status of food as well as the economic standards of the masses. Besides, this application combats environmental pollution resulting from dumping, composting, and the burning of agro waste.

2.3.3 Animal feed

The high cost of animal feed is one of the major concerns in funding animal husbandry projects. An alternative is to source local feed for these animals. Preferably, crop residue may suffice for the commercial feed to reduce costs [12]. It is also a known fact that residues from cereal crops are about two-thirds of the bulk cereals. For example, barley and wheat straw are rich in fiber and are used as animal feeds for ruminants in the tropic and sub-tropic regions but they are low-quality roughages with poor nutrients values required for healthy animals. The use of these residues alone results in poor feeding methods for adult ruminants due to the poor digestibility, low metabolizable energy, crude protein, as well as unavailable minerals and vitamins. Contrastingly, agro-industrial by-products like pulp from citrus are digestible and are hydrolyzed by enzymes to release nutrients that nourish the animals. It, therefore, requires that straw should be appropriately pre-treated to improve the nutritional value of crop residues for animal feeds [13, 14].

2.3.4 Mulching

The technique of mulching is an ancient technology in farm practice. Crop residue used to cover the topsoil ensures that the surface soil is organically rich during the emergence of the shoot [15]. Mulching fulfils a multi-dimensional purpose that combines the preservation of soil content and soil ecology for the productivity of crops [16, 17, 18]. The protective layer provided by the mulch prevents soil erosion. The use of crop residues improves the soil quality by adding humus while ensuring soil nutrient stability. Mulching is necessary especially in the face of climate change, thus improving water infiltration and conserving soil water via increased soil porosity and reduced soil evaporation [19, 20]. The overall process protects against drought and food insecurity. Before the era of pesticides, mulching was one of the ways to control weeds via allelopathic effect [21]. It also regulates the temperature for both the growth of the seedling and enhanced soil microbial activity.

2.4 Biomass residues valorization beyond the traditional use

Agricultural residues are used traditionally for heating but generate smoke and other pollutants. The compost of crop residues releases methane into the atmosphere or is processed into ash for crop pest control. However, advancements in technology and urbanization condemn the traditional use of these residues. Alternatively, these residues are transformed via sophisticated techniques into pellets and briquettes, bioethanol, bio-lubricants, and bio-carbon amongst other materials for diverse applications (Table 2.1).

Valorization refers to the creation of value from any material that may or may not be valuable to man and his environment. This process involves the transformation of residues and waste into products of value. Table 2.1 shows the diverse methods for making beneficial products from biomass residues as biomass valorization of residues requires that crop and animal waste are appropriately processed into goods and services that serve the needs of mankind.

Table 2.1: Products from Biomass Residue and the Technology Used

S/N	Residues/waste	Technology/methods	Products	References
1	Sugar cane bagasse	Fermentation	Alcohol	[22, 23]
2	Conocarpus wastes and others	Pyrolysis	Bio-charcoal	[24, 25]
3	Coconut shell	Pyrolysis	Activated charcoal	[26]
4	Oil palm waste	Densification	Pellets	[27, 28]
5	Palmyra palm shell and red gram stalk	Compaction	Torrified pellets	[29]
6	Cashew nut waste	Pyrolysis	Bio-oil	[30, 31]
7	Sugarcane (back and pulp) and rice husks	Fermentation	Biogas (Syngas)	[32]
8	Agro-waste blend	Gasification	Biogas	[33]
9	Melon seed oil Insect	Mechanical extraction	Biodiesel	[34, 35]
10	Agro-industrial waste	Extraction and purification	Antioxidants	[36, 37]
11	Wheat straw	Densification	Briquettes	[38]
12	*Codonopsis pilosula* / cardanol	Maceration Organic synthesis	Lubricants	[39, 40]
13	Oil (Jatropha and karanj oil, and others)	Oil extractor	Transformer oil	[41, 42, 43]

S/N	Residues/waste	Technology/methods	Products	References
14	Rice husk, corncobs, and sawdust composite	Compaction	Particleboard	[44, 45]
15	Sugarcane bagasse fiber/ corn cob and cassava stalk	hydraulic press	Roofing sheet	[46]
16	Olive stones	Physical activation	Activated carbon	[47]
17	Cassava waste Cassava waste blend	Extraction and precipitation	Bioplastics	[48] [49]
18	Agro-food waste	Culture fermentation	Single-cell protein	[50]
19	Food waste Lignin biomass	Culture media	Bio-pigments Nano pigment	[51, 52]
20	Industrial agro waste (e.g., pectin)	Fermentation technique	Biocatalyst/ Enzyme	[53, 54]
21	Agro waste	Culture media	Biosurfactant	[55, 56]
22	Residual biomass waste	extraction	Biopolymer	[57, 58]
23	Agro waste	Extraction	Biofiber Cellulose fiber	[59, 60]
24	Agro waste	Extraction and precipitation	Nanoparticles	[61]

The valorization of residues generates products that are unique, diverse in usage, eco-friendly, and applicable in various fields: from home to the industry. A few of these by-products are enumerated below:

2.4.1 Feedstock for bioenergy

Biomass and agricultural residues are potential sources of sustainable and renewable energy. These residues such as corn stover, cassava peel and bagasse, cereal straw, sugarcane bagasse, potato peel, and oil palm have been explored for biofuel production capacity. For instance, barley straw and hulls are biomass resources for fuel pellets, which can be burned alone or co-fired with coal to produce heat and power. In addition, liquid biofuel is produced from these residues via diverse conversion techniques.

2.4.2 Animal Feed alternatives

With the increasing population and the rise in residue demand for bio-economy transition, there is a need for an alternative but high-nutrient feedstock for animal livestock [62]. Insects are functional foods with health benefits [63]. Microalgae have diverse applications as nutraceutical. They are used as a dietary ingredient for animal feed as a protein and vitamin supplement for aquaculture. Apart from the benefits (in nutrition), microalgae have disease-preventing molecules, including anti-oxidative and anti-microbial active agents that can preserve the health status of animals [64, 65]. Such food sources for animals are rich in proteins, carbohydrates, lipids, minerals, vitamins for both ruminant and non-ruminant animals because it is suitable for diverse digestive systems [66].

2.4.3 Biochar resource

Biochar is a product from plant or animal biomass after being heated in an oxygen-limited environment [67]. Biochar is applied to treat wastewater, sequester carbon, restore soil nutrients, or improve soil quality for enhanced plant growth. Unlike biochar, activated carbon is often used for adsorption [68, 69, 70, 71].

2.4.4 Activated carbon (AC)

The research interest in activated carbon synthesis, modifications and applications in diverse fields is rising significantly. The advantages of AC include simple synthesis routes, large surface area, high stability, availability of raw materials, and can be tailored to have desirable properties. Activated carbon can be produced from biochar that is further treated using physical or chemical methods. Such treatment enhances the features and properties of activated charcoal for a specific application. Biomass residues (and wastes) are carbonaceous precursors that can make AC suitable for adsorbing gases and water pollutants. They are also employed in making batteries for energy storage. The work of Srivastava et al. (2021) revealed that research in AC from agricultural waste and sludge shows increasing study interest for the next 50 and 25 years, respectively [72]. However, the effective adsorption of pollutants by any activated carbon is a function of several factors. The process of activation, modification, heating temperature, and hold time during AC synthesis determine the porosity and surface structure for pollutant adsorption [73].

2.4.4.1 Activated carbon as adsorbent material

2.4.4.1.1 Water treatment

Water pollutants are increasingly becoming a concern for human health and safety as well as those of marine animals [74, 75], and the use of AC as an adsorbent in water treatment process is now a research interest. The world of industrialization with urbanization, advanced technological processing, and population size has negatively contributed to this type of pollution [72]. Among the water treatment techniques, adsorption is widely desired due to the ease of operation, myriads of on-field applications, and high pollutant removal performance.

Activated carbon is well recognized as the most geriatric but widely used adsorbent for water and wastewater treatment, removing organic and inorganic pollutants [76]. The surface chemistry and the porous structure are the basic properties of AC when considered for some applications. This technique seems old, but research is still ongoing in water purification methods, especially in sub-Saharan Africa. Recent studies in AC focused on the strategy for the optimal removal of specific organic and inorganic pollutants which are increasingly found in water bodies due to industrialization. The review of Bhatnagar et al. [76] focused on the detailed information regarding activities towards the surface modification of activated carbon for water purification. Furthermore, Rigobello et al. [77] identified that granular activated carbon (GAC) was effective in removing diclofenac (99.7%), which is significantly more efficient than chlorine dioxide and chorine. The use of chlorine dioxide and chorine in oxidizing diclofenac indicated a poor elimination method with conventional water treatment for diclofenac because diclofenac and dissolved organic carbon were still detected in the treated water.

2.4.4.1.2 Carbon capture

The use of AC in gas phase adsorption is important to prevent the release of potential toxic gases and greenhouse gases (GHG) into the environment. The technologies for CO_2 capture and storage (CCS) were originally developed to reduce emissions at large stationary sources. Post-combustion CO_2 capture involves the separation of carbon dioxide from the released effluent gas obtained during the combustion of fossil fuels in power plants. This is commonly referred to as post-combustion CO_2 capture. Oxycombustion, pre-combustion, industrial CO_2 capture, and direct air capture are only a few of the additional technologies. Amine scrubbing, however, is the only approach for post-combustion collection that has been extensively demonstrated. This technology is regarded as being the most developed for many industrial applications [78]. Although, adsorption is still being developed for additional large-scale applications, it has been demonstrated at scale in pre-combustion CO_2 collection at the Valero Refinery in Port Arthur, Texas, in the United States (industrial CO_2 capture, DAC) [79]. Fig. 2.4 shows the desirable qualities for adsorbents for CO_2. The production of activated carbon is cost-effective and stable because the material, AC, does not age [80]. In literature, there are reports on the diverse waste materials used for making AC as shown in Fig. 2.4 [73]. Siti Noraishah et al. [81] made activated carbon from rice husks for CO_2 removal from industrial fumes. The adsorption of CO_2 is optimized when activated carbon is modified (or functionalized) with chemicals. The impregnation of AC with amine [82, 83], ionic liquids [84] and deep eutectic solvent (DES) [85] revealed an increase in CO_2 capture.

Zulkurnai et al. [85] illustrated that DES-functionalized AC adsorbed higher CO_2 than the non-functionalized AC due to the surface nitrogen that increased the active sites for CO_2 capture. This modification had effective CO_2 adsorption despite the reduced surface area of the DES-functionalized AC compared to the non-functionalized. However, the DES-functionalized AC adsorbed CO_2 through chemisorption [85]. In another study, Zhang et al. [86] confirmed that the use of nitrogen for surface modification increased CO_2 uptake.

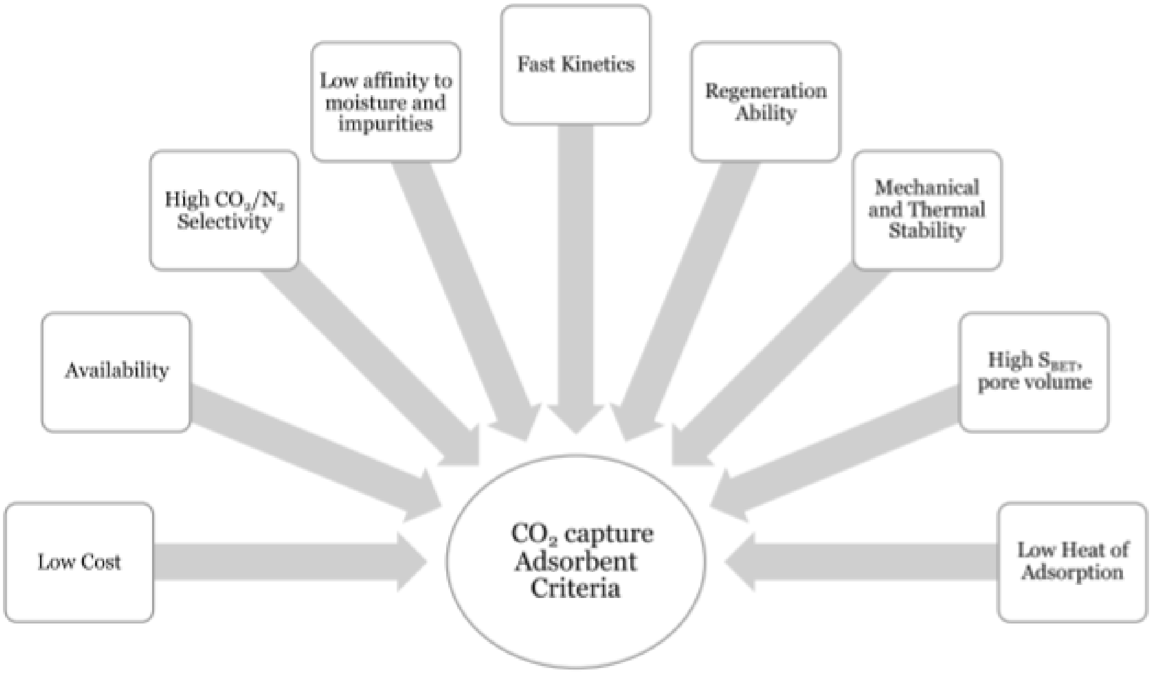

Fig. 2.4: Desirable qualities of a good adsorbent for CO2 Capture [87]

Fig. 2.5: Lignocellulose-based adsorbents for CO2 capture [73]

2.4.4.2 Activated carbon as catalyst

The optimal surface area, pore size, and pore volume of AC improve the catalytic performance and product selectivity for a reaction. The sites for adsorption in AC are pores of various sizes (micropores, mesopores, and macropores). Also, modification can add functional groups that facilitate adsorption for catalytic reaction to occur. AC is used as a catalyst to manufacture phenols and hydrocarbons. Interestingly, the lignocellulose-based AC exhibited more promising and satisfactory catalytic efficiency with an exceptional target in chemical selectivity than some commercial ACs and zeolite catalysts [88].

2.5 Biomass residue assessment

Biomass residue assessment is vital in describing and predicting the performance of the agricultural sector in Nigeria, especially for an overall economic potential in agricultural waste generation and conversion into valuable products. The assessment of biomass availability in Nigeria is necessary for providing vital information on the sustainable availability of residues for advanced biofuel/bioenergy potential. In addition to residue assessment, the modern biofuel potential from this estimated biomass residues are crucial in making important decisions towards investing in biofuel production in Nigeria, and by extension, transition to the bioeconomy. This assessment is a principal factor that confirms the quantity of residue generated yearly as well as its continuous availability, after taking into consideration the measure that is recoverable for energy purpose. It is also necessary to include the ideal conditions such as advanced conversion technologies in evaluating the potential biofuel estimate that can be produced from biomass residues, taking into cognizance detailed the technical potential of the residues.

The biomass residues considered covers a variety of biofuel feedstock of agricultural origin: crop waste (field and process-based residues as shown in Fig. 2.5), forest residues, and municipal waste (connected to the population in Nigeria). The information from this assessment indirectly provides the crop production record, thus justifying the need for improved crop and forest residues management to facilitate their continuous availability. In addition, vital review information on the chemical composition of these residues is essential. The latter seek innovative measures for the transformation of residues for the optimal delivery of potentials. However, the challenge is the competing interest for a given residue needed for making various valuable products. For instance, cassava peel is an example of biomass residues needed as pig feed, raw materials for bioethanol, biomethane, biochar, bioplastic, and other bio-products. Hence, the demand for this residue is high, but availability may be poor, thus negatively impacting industrial interest for such residue as compared to synthetic alternatives. The high demand for such a residue may result in inflation of residue, and consequently the bio-products. It is then obvious that such a challenge may jeopardize the transition to the use of bio-products due to their affordability. In this regard, there is a need for a deliberate search for new biomass residues that do not compete with food, although are suitable for other industrial purposes. The newly identified residue requires characterization and scientific evaluation. For instance, there are ornamental trees and fruits (as well as wild fruits) that are potential feedstock towards advanced and sustainable biofuels and resources for biomaterials. In summary, biomass residues (agro-wastes) contribute to various value chains for diverse applications considering their properties, composition, and component for bio-products which are beyond their traditional use as they resonate with advanced technology.

Fig. 2.6: Biomass residues used for assessment

2.6 Challenges with crop residues

The various traditional use of crop waste interferes with the availability of biomass residues for modern application in biofuel, wastewater treatment, and energy storage amongst other value-added products. Although the assessment of biomass residue availability is estimated using technical potential (i.e., the residue available after taking into cognizance the biomass-specific constraints that defined its use as a raw material or energy feedstock), rather than theoretical potential. It is noteworthy that this potential is lower than the latter. Hence, the available residues for bioenergy for industrial scale-up are smaller than imagined. The increasing research on biomass residue valorization and their applications may negatively influence the known traditional use of the same residues in maintaining soil nutrient as well as the preservation of the ecosystem. For this purpose, there is a need to identify other resources that may be considered as waste.

Trees produce fruits (or seeds) that may be considered ‘wild’, and such are less likely to be served as food or provide nutritional benefits for humans. An example of such is an ornamental tree called *Ficus benjamina*. Fruits from *Ficus benjamina* will first be assessed, then characterized for potential value-added products.

2.7 *Ficus benjamina* fruits: A biomass residue case study

The matured plant of *Ficus benjamina* is usually a huge tree planted for ornamental purposes. Such FB trees are usually 60-70 feet wide with a dense canopy and dropping branches [89], which prevents other plants to grow underneath them. These trees are herbaceous and may be referred to as evergreen tree, as it produces leaves and fruits all through the year. Their roots are adventitious and strong that looks dark grey, smooth with brown branchlets. The thick, shiny, and about 2-5-inch-long evergreen leaves cover the long branches. The leaf blade is oblong, elliptic, lanceolate, or ovate [89, 90, 91]. The nature of the FB tree ensures that it grow conveniently due to their versatility with soil type, drought, and salt tolerance. Also, the FB plant can produce a lot of residues (fruits and leaves) due to its great capacity for propagation. It can be propagated through its stem and roots, with the rapid growth of both trunk and branches after pruning. FB tree has no known disease to retard its growth [89, 92].

The fruits of FB trees drop like 'tears from the tree', hence, the name weeping fig. The fruit may appear twice or thrice on a matured FB tree per year. The fleshy FB fruits, with less than 5 inches, are oval (round) in shape and occur in different colors: yellow, orange, red and purple. Furthermore, FB is dependent on a mutualistic association with host-specific pollinating fig wasps for reproduction. Such pollination type and the nature of the fruit creates an avenue for more fruits [92]. Regarding the mode of propagation, rapid growth, and the type of reproduction, there are lots of residues: prune residues, fruits and leaves from FB trees that contribute to biomass waste. Environmentally, both the fruits and leaves cause litter problems. However, among these wastes, the fruits are potential feedstock that is of interest for valorization.

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CHAPTER THREE

Biomass Valorization to Bioenergy: Assessment of Biomass Residues' Availability and Bioenergy Potential in Nigeria

3.1. Introduction

Bioenergy is the energy sourced from biological wastes (often from plant and animal), as such, they are renewable energy. Modern solid biomass, liquid biofuels, and biogases obtained from sustainable sources, excluding the traditional biomass, are examples of bioenergy. This energy form is used in dedicated power plants to provide dispatchable power and minimal emissions, especially when co-fired with coal or natural gas, and its combination with carbon capture and storage (CCS) provides far fewer emissions.

In 2020, the biofuels used had a rate of about two mb/d as the expected volumes can double in the stated policies scenarios (STEPS) by 2030, quadruple in the announced pledges scenario (APS) by two-and-a-half times and triple in the Net Zero Emissions (NZE). The utilization of modern solid biofuel is foreseen to rise by 30-70% in all scenarios by 2030. By estimation, biogas will grant 400 million people access to clean cooking in the NZE by 2030, using 2.5 exa-joules (EJ) of biomethane with an overall biogas demand reaching 5.5 EJ. Although there are ambiguities on the exact level of the world's sustainable bioenergy supply potential, the estimate is at least 100 EJ to a 150-170 EJ. Energy crops, organic by-products, and leftovers from agriculture, forestry, municipal solid waste, and wastewater are possible sources of sustainable bioenergy. In 2050, the supply of modern solid bioenergy in the NZE will increase to 75 EJ, with roughly 45 percent from forestry by-products and residues, 25% from energy crops, and the rest from agricultural residues and urban solid waste [1, 2].

Biomass residue from agricultural products is abundant, and it has a strong potential for sustainable renewable energy generation [3]. As reported in 2005, biomass is responsible for about 14% of the primary energy consumed globally, but the global domestic biomass supply in 2018 was 55.6 EJ and 85 % of this supply came from traditional biomass sources like wood chips and pellets [4]. Solid biofuel contributes about 90% of the bioenergy utilized today. Some of this solid biomass is employed in the current technology to generate electricity and heat while reducing emissions [4, 5]. In all scenarios, modern forms of solid bioenergy are on the increase, and extends beyond power and heat generation to serve as feedstock to produce liquid and gaseous biofuels, and even consumed directly in end-use industries. The consumption of solid bioenergy today is almost 55 EJ globally. About half of it, solid biomass (wood fuel), is utilized in traditional cooking and the making of charcoal. Developing economies use higher portion of the latter for cooking and heating, but it is an unsustainable and inefficient form of energy, and a principal source of household air pollution and premature death. After fossil fuels, solid bioenergy is the most widely used fuel type today, as traditional cooking methods consume about 40% of the total. In the STEPS and APS, solid bioenergy is frequently less used, but in the NZE, it is abolished as part of the goal to attain universal access to clean cooking by 2030 [1, 2]. Agricultural residues from crops and forestry can be converted to energy carriers (solid biofuel, biogas, and cellulosic ethanol) through several techniques, and have found applications in transport fuels, electricity, and heat generation [6].

Nigeria depends principally on fossil fuels (about 86%) and hydropower plants for electricity generation [7]. The overdependence on fossil fuels has negative implications for environmental sustainability [8, 9]. The lack of diversity and high-power demand are factors leading to inconsistency in the electricity supply in the country. Therefore, there is a need to adopt green energy sources with less environmental impact that will complement the hydro-plants, thereby decreasing pollution arising from the combustion of fossil fuels. Although Nigeria has a high agricultural production and population, the economic problems and lack of proper assessment of available biomass residues [10] have hampered significant progress targeted at transitioning to bioenergy from biomass residues.

Jekayinfa and Scholz [11] estimated residues generated from nine crops in Nigeria for 2000–2004. Their findings were restricted to only crop residues and was for five years. In the same vein, Simonyan and Fasina [12] estimated the bioenergy potential of residues from crops, perennial plantation, forestry, animal waste, and urban municipal waste in Nigeria using data for 2010 only. However, their study did not relate the estimated energy potential to a specific energy carrier. Alhassan et al. [13] used five crop residues obtained in Kwara State, Nigeria, to estimate the energy potentials for power solutions. In their assessment, they used theoretical potential values rather than the technical potential for these residues. The challenge is the limitation imposed using the latter potential due to its unreliability for energy application [14]. Therefore, there exists a knowledge gap in adequately quantifying the bioenergy potential in biomass residues.

The present work aimed at estimating the total energy obtainable from agricultural residues (crops, forests, and livestock) and municipal waste for biofuel application. We investigated an 11-year (2008–2018) span to arrive at a holistic perspective and meaningful conclusions. Specifically, we adopted a computational/analytical approach to determine the bioenergy potential for cellulosic ethanol and biogas. In conclusion, we highlighted some possible challenges to the generation of bioenergy and implications on the bioeconomy of Nigeria, and we made recommendations. Our findings are relevant to stakeholders, investors, and organizations in the sustainable environment and renewable energy sector for the government to adopt best practices towards the diversification of electric power generation in Nigeria.

3.2. Materials and methods

3.2.1. Case study

In this study, biomass resources in Nigeria were evaluated. These resources include crop residues, forest residues, livestock dung, and municipal waste generated in the country. The residue availability and bioenergy potential were assessed based on a resource focused computational and analytical approach, using the technical potential generated from residue produced in the year 2008–2018. Data were sourced from the Food and Agriculture Organization of the United Nations statistics (FAOSTAT) database [15]. The bioenergy potential of residues was estimated statistically. Although this method is simple, reproducible, low cost, and transparent, it is deficient in accounting for the economic dimensions required for evaluating the availability of land for energy crop production, the impact of bioenergy production on the environment, as well as social constraints for some key factors that elucidate the influence on soil, biodiversity, climate, cost, and other macro-economic factors on bioenergy potential.

The conceptual framework for the research is shown in Figure 3.1. The biomass residues are classified as agricultural residues and municipal waste. The various agricultural residues considered include crops (soya beans, seed cotton, sugar cane, sorghum, plantain, groundnut, coconut, rice, cocoa, millet, cowpea, cassava, yam, sweet potatoes, cocoyam, maize, and oil palm), forests (round wood processing such as logging, sawing, and timber processing), and livestock (dung from cattle, chicken, goats, pigs, and sheep). Solid and liquid municipal waste was evaluated from the estimated population of 16 major cities that represents the four geographical regions in Nigeria. Suitable conversion technologies were computationally implemented to transform these residues and wastes into energy carriers, which include solid fuel (from crude crop residues), cellulosic ethanol (from forest and crop residues), and biogas (from the forest, crop residues, livestock, and municipal solid and liquid wastes). It is worth noting that in this work, primary biomass (wood fuel and staple crops) was not considered because their conversion to energy carriers is detrimental to the environment (soil status, biodiversity, climate change) and food security. Additionally, certain energy crops (such as *Jatropha curcas*), grasses (e.g., switchgrass and seaweeds), and microfauna (such as algae) were excluded due to the limitation of certified or reliable data. Table 3.1 shows the categories of residues considered in this assessment.

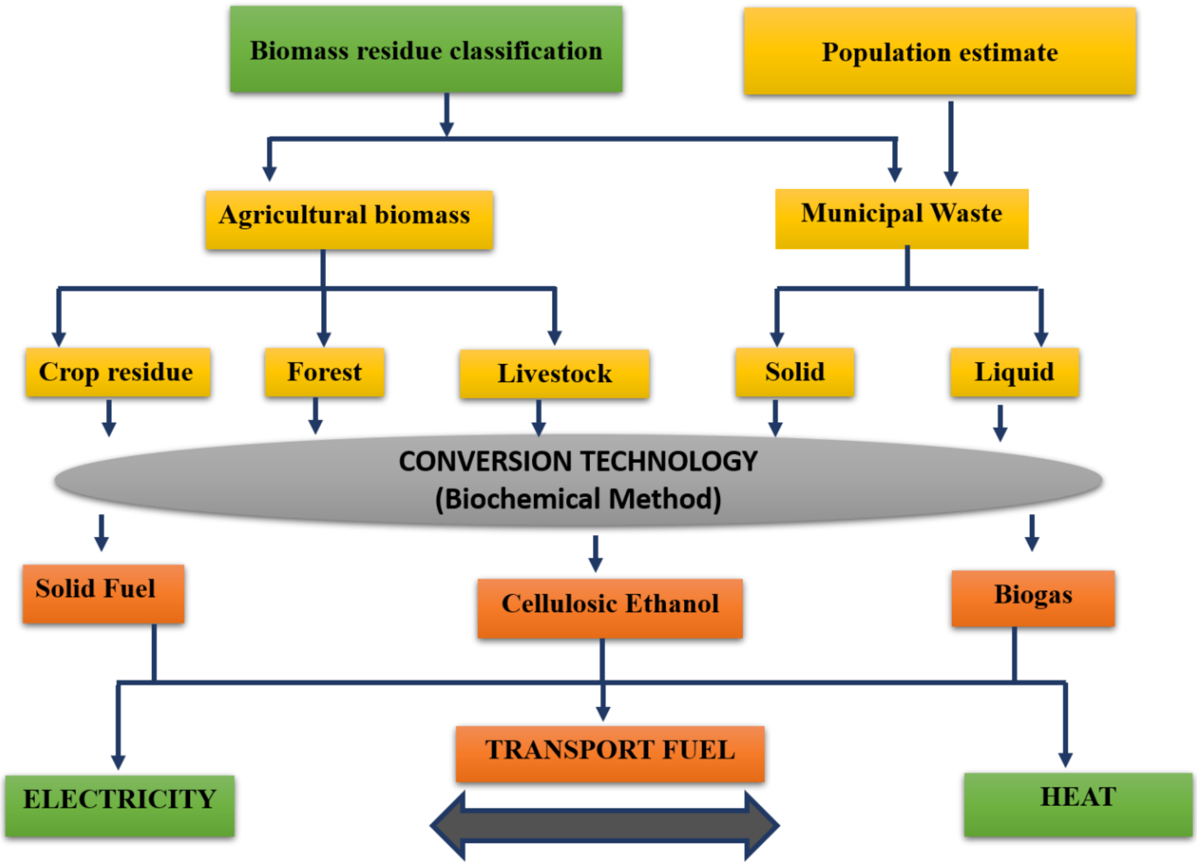

Fig. 3.1: Analytical framework for estimating solid biofuel, cellulosic ethanol, and biogas from residues

Table 3.1: Categories of biomass resources used for the bioenergy potential assessment

S/N	Class of residues	Category	Examples
1	Agricultural residues	Primary by-product	All residues from crops during harvesting (Table 3.2)
		Secondary residues	All crop residues during processing (Table 3.2)
		Tertiary residues	Municipal solid waste (MSW) and municipal liquid waste (MLW)
2	Forest residues	Primary by-product	Wood bark and wood slab
		Secondary residues	Sawdust
3	Livestock	Primary by-product	Manure

3.2.2. Crop residues

The crop residues investigated were resources from existing farmlands. However, some assumptions (section 3.2.2.1) were made to account for the key parameters for sustainability. **Table 3.2 shows** the annual crop production in Nigeria, and data were obtained from the **FAOSTAT** database [15]. The total crop production was highest in 2016, as 164.695, 158.807, and 159.947 million tonnes (Mt) were generated in 2016, 2017, and 2018, respectively (Table 3.2). Fluctuations were observed in the production of these crops across the 11 years. **Furthermore, a** total of 27 residues (Table 3.3) from 17 crops were considered.

3.2.2.1. Sustainability assumptions

Some assumptions that were considered are:

- **Land availability:** Primary energy crop (PEC) was not considered, hence, no land competition for animal husbandry or crop cultivation. There are no certified data regarding the annual production of PEC, yield, and cultivated land. Therefore, the land-use competition was not taken into account. Only cultivable land was used for the estimation, no expansion on arable land was included. There was no future projection on PEC.

- **Land use:** Since crops are given priority (more lands are allocated to food and fibers), the efficient use of land produces biomass that accounts for a large extent of the available residue for bioenergy assessment. In addition to land availability and use, farm management practices such as the use of improved seed, fertilizer, pest, and weed control with better technology (research and development, R&D) are the norm of farmers. It, therefore, supports residue availability. These agricultural practices ensure sustainable residue supply from existing farmlands.

- **Soil quality:** Soil quality is also an important factor. Lands with rich soil quality will yield more harvest (more residues) than those with poor soil nutrients. Hence, double cropping, alternate crop rotation, appropriate mineral fertilizer, and the use of compost on farmland may increase residue production [16, 17]

- **Biodiversity:** Biodiversity is limited as there is negligible forest encroachment since only farmlands already in use were considered in this assessment. Also, the use of technical residue potential preserves biodiversity because they are utilized for other purposes.

- **Climate change:** The right crop management system on farmlands can reduce climate change.

- **Water:** The rain-fed condition was assumed as Nigeria has suitable agro-climatic conditions.

- **Farm practice (Animal husbandry):** Regarding the livestock manure production, improved feeds with large pastureland support livestock production. With the use of technical residue potential, the pasture for livestock and manure for soil nutrient renewal is guaranteed.

Table 3.2: Crop production in Nigeria

Crop type	Crop Production (Mt)											
	2008	**2009**	**2010**	**2011**	**2012**	**2013**	**2014**	**2015**	**2016**	**2017**	**2018**	**Average**
Soya beans	0.591	0.427	0.365	0.493	0.65	0.518	0.624	0.589	0.615	0.73	0.758	0.578
Seed cotton	0.492	0.364	0.602	0.538	0.288	0.27	0.29	0.278	0.279	0.291	0.271	0.360
Sugar cane	1.41	1.4	0.85	0.756	1.09	1.27	1.41	1.45	1.49	1.49	1.42	1.276
Sorghum	9.32	5.28	7.14	5.69	5.84	5.3	6.88	7.01	7.56	6.94	6.86	6.711
Plantain	2.73	2.7	2.68	2.68	2.95	2.96	3.01	3.08	3.03	3.06	3.09	2.906
Groundnut	2.87	2.98	3.8	2.96	3.31	2.47	3.4	3.47	3.58	2.42	2.89	3.105
Coconut	0.234	0.243	0.264	0.265	0.265	0.266	0.268	0.269	0.283	0.282	0.285	0.266
Rice	4.18	3.55	4.47	4.61	5.43	4.82	6	6.26	7.56	6.61	6.81	5.482
Cocoa	0.367	0.364	0.399	0.391	0.383	0.367	0.33	0.302	0.298	0.324	0.333	0.351
Millet	9.06	4.93	5.17	1.27	1.28	0.91	1.4	1.49	1.55	1.5	2.24	2.800
Cowpea	2.92	2.37	3.37	1.64	5.15	4.63	2.14	2.31	3.02	2.49	2.61	2.968
Cassava	44.6	36.8	42.5	46.2	51	47.4	56.3	57.6	59.6	59.4	59.5	50.991
Yam	35	29.1	37.3	33.1	32.3	35.6	45.2	45.7	49.4	47.9	47.5	39.827
Sweet potatoes	3.32	3.3	3.47	3.52	3.59	3.68	3.67	3.82	3.89	3.96	4.03	3.659
Cocoyam	5.39	3.03	2.96	3.01	3.2	2.93	3.27	3.28	3.23	3.27	3.3	3.352
Maize	7.53	7.36	7.68	8.88	8.69	8.42	10.1	10.6	11.5	10.4	10.2	9.215
Oil palm	8.5	8.5	8	8	8.1	8	7.97	7.89	7.81	7.74	7.85	8.033
TOTAL	**138.514**	**112.698**	**131.02**	**124.003**	**133.516**	**129.811**	**152.262**	**155.398**	**164.695**	**158.807**	**159.947**	**141.879**
Average	**8.148**	**6.629**	**7.707**	**7.294**	**7.854**	**7.636**	**8.957**	**9.141**	**9.688**	**9.342**	**9.409**	**8.346**

Source: FAOSTAT [15]

3.2.2.2. Theoretical and technical crop residue potentials

The theoretical residue potential, for each crop, was obtained from the product of the total specific crop available for a given year, and the residue-to-product ratio (RPR). RPR is an index that indicates the weight of residue a particular crop generates, based on the produced amount [18]. Taking into account the variability of the RPR values due to several factors identified by Simonyan and Fasina [12], the mean RPR **was used.** The theoretical potential of the crop residues was estimated using **Eq. 1.**

$$P_{th} = P_{crop} \times RPR \tag{1}$$

where:

P_{th} = theoretical residue potential; P_{crop} = Crop production; RPR = residue to product ratio

The use of theoretical residue potential is not realistic because other forms of crop residue utilization may compete with its availability for bioenergy production. Hence, we considered only the recoverable residue fraction for each crop, referred to as the technical residue potential. The latter is defined as the surplus residue after considering the competition among other uses and spatial restrictions. It is estimated using Eq. 2. The obtained value gives the quantitative amount of the excess residues available for energy purposes.

$$P_{tech} = P_{th} \times Rf \tag{2}$$

P_{tech} = technical residue potential; Rf = recoverable fraction

The technical residue potential was used to estimate the energy potential of cellulosic ethanol and biogas.

3.2.2.3. Solid Fuel Energy Potential

The bioenergy potential in dried crop residues in their crude forms was calculated using **Eq. (3)**. The estimated solid fuel made from crop residues was obtained by multiplying the total annual technical crop residue potential and the lower heating values (Table 3.3).

$$P_{SFE} = P_{tech} \times LHV \tag{3}$$

P_{SFE} = Solid fuel energy potential; LHV = Lower heating value (MJ/kg)

Table 3.3: Parameters used in estimating bioenergy potentials from crop residues

Crop residues	RPR	Rf (%)	LHV (MJ/kg)
Soya beans straw	2.50[a]	100	12.38
Soya beans pods	1.00[a]	100	12.38
Seed cotton stalk	2.88	80	18.61
Sugar cane tops/leaves	0.11	80	15.81
Sugar cane bagasse	0.18	100	18.10
Sorghum straw	1.99	80	12.38
Plantain trunks & leaves	0.50	80	15.48[b]
Groundnut straw	1.25	100	17.58
Groundnut shell	0.37	100	15.66
Coconut Husk	0.42	100	18.63
Coconut Shell	0.25	100	18.09
Rice Husk	0.26	100	19.33
Rice Straw	1.66	80	16.02
cocoa bean Pods	0.93	80	15.12
Millet Straw	1.83	80	12.38

Crop residues	RPR	*Rf* (%)	LHV (MJ/kg)
Cowpea shell	1.75	100	19.44
Cassava stalk	0.06	80	17.50
Cassava peeling	0.25	20	10.61
Yam straw	0.50	80	14.24
Sweet potatoes straw	0.50	80	14.24
Cocoyam straw	0.50	80	14.24
Maize stalk	1.59	80	19.66
Maize husk	0.20	100	15.56
Maize cobs	0.29	100	16.28
Oil palm EFB	0.17	100	8.16
Oil palm kernel Shell	0.07	100	18.83
Oil palm fiber	0.14	100	11.34

Most mean values of RPR and *Rf* were obtained from Kemausuor et al. [19] and those of LHV from Simonyan and Fasina [12]. Other values with alphabetic superscripts were sourced as indicated [a][20]; [b][11].

3.2.2.4. Cellulosic Ethanol Potential

To estimate the bioenergy potential of the cellulosic ethanol upon the conversion of the crop residues by anaerobic digestion, some pre-treatment processes such as hydrolysis, enzymatic activities, and microbial fermentation were taken into account. The cellulosic ethanol production from crop residues was estimated using Eq. (4):

$$Y_{CE} = P_{tech} \times C_{glu} \times y_{hyd} \times y_{eth} \times \eta_{pre} \times \eta_{enz} \tag{4}$$

where:

Y_{CE} = yield of cellulosic ethanol.

P_{tech} = technical potential

C_{glu} = concentration of glucan.

y_{hyd} = yield of enzymatically hydrolyzed glucan.

y_{eth} = stoichiometric yield from glucose.

η_{pre} = Efficiency of pretreatment.

η_{enz} = Efficiency enzymatic cellulose conversion

In estimating the cellulosic ethanol production, we assumed fermentation and distillation processes to be 100%, as no loss was considered. The assumed values used for the estimation of cellulosic ethanol production are shown in Table 3.4.

Table 3.4: Summary of indices for cellulosic ethanol production from crop and forest residues

Conditions	$\boldsymbol{Y_{eth}}$	$\boldsymbol{Y_{hyd}}$	$\boldsymbol{\eta_{Pre}}$ **(%)**	$\boldsymbol{\eta_{enz}}$ (%)	$\boldsymbol{\rho_{Distil}}$ **(%)**	$\boldsymbol{\rho_{Ferm}}$ **(%)**	$\boldsymbol{\eta_{Scale}}$ **(%)**
No Pre-treatment	0.51	1.11	-	30	100	100	50
With Pre-treatment	0.51	1.11	80	90	100	100	80

Where; $\boldsymbol{\rho_{Distil}}$ = distillation efficiency; ρ_{Ferm}= fermentation efficiency. Values were sourced from Kemausuor et al [19].

During the hydrolysis of crop residues for cellulosic ethanol production, two scenarios were considered: no pre-treatment and pre-treatment. In the no pre-treatment case, the enzymatic activity is assumed to be minimal (about 30%) with a production of cellulosic ethanol scale-up (η_{Scale}) of about 50%. In the pre-treatment scheme, the enzymatic efficiency is assumed to be 90%, to yield cellulosic ethanol of 80%. The bioenergy potential of cellulosic ethanol was estimated from the lower heating value (LHV) of 28.9 MJ/kg, and ethanol density of 0.789 kg/L.

3.2.2.5. Biomethane potential

The estimation of biomethane was performed using the technical residue potential generated for the crop residues. To obtain the biomethane potential (BMP), the Buswell BMP equivalent (Eq. 5) was first determined.

$$Y_{BMP\ Buswell} = \left(Y_{Buswell,glu} \times C_{glu}\right) + \left(Y_{Buswell,hem} \times C_{hem}\right) \tag{5}$$

BMP is defined as the theoretical estimate based on the experimental evaluation of a given feedstock for the determination of the maximum volume of methane generated. It is the optimal methane volume per gram of volume solid (VS) of a substrate (i.e., the biodegradable fraction).

$Y_{BMP\ Buswell}$ = estimated biodegradable fraction in specific crop residue (feedstock) for biogas production using Buswell formula.

$Y_{Buswell.glu}$ = estimated glucan in specific residue using Buswell formula.

$Y_{Buswell.hem}$ = estimated hemicellulose using Buswell formula.

C_{glu} = concentration of glucan.

C_{hem} = concentration of hemicellulose

The maximum biogas estimate/potential was determined using Eq. (6):

$$Y_{Biogas} = P_{tech} \times Y_{BMP\ Buswell} \times \eta_{Scale} \tag{6}$$

Where, Y_{Biogas} = Biogas yield; η_{scale} = average efficiency for continuous biogas production, For the energy potential of biomethane, calculations were based on the following assumptions: 1 m^3 biomethane has a calorific value of 10 kWh STP; energy potential of CH_4 conversion and the conversion factor of TJ to Mtoe is 0.278 GWh/yr and 24, respectively.

3.2.3. Forest residues

From the FAOSTAT database [21], we obtained data on the average industrial round wood harvested yearly in Nigeria. The residues generated from the logging, sawing, and timber processing activities of round wood were determined using the assumption proposed by Koopmans and Koppejan [22]. These residues are classified into three: wood slab, wood bark, and sawdust. Wood slabs were taken to be 40% and 38% for logging and sawmilling processes, respectively while for sawdust 12% and 20%, in the same processes. In addition, the sawdust from particleboard was 10% while the residue from the wood bark during sawmilling was 12%. These values were adopted following Simonyan and Fasina [12] and Koopmans and Koppejan [22].

3.2.3.1. Cellulosic ethanol from forest residues

Similar to the ethanol estimation from crop residues, the cellulosic ethanol potential from wood residues was determined using Eq. (7).

$$Y_{CE}\ (forest\ residues) = P_{FR} \times C_{glu} \times Y_{hyd} \times Y_{eth} \times \eta_{Pre} \times \eta_{enz} \tag{7}$$

P_{FR} = annual production of forest residue

3.2.3.2. Biogas Potential from forest residues

The maximum biomethane production from forest residues was determined based on Buswell's formula using an expression similar to Eq. (5). However, an industrial-scale efficiency of 40% was assumed for methane production from forest residues. Hence, the biogas estimated at the industrial scale was obtained from Eq. (8).

$$Y_{Biogas}(Forest) = P_{FR} \times Rf \times \left[\left(Y_{Buswell,glu} \times C_{glu}\right) + \left(Y_{Buswell,hem} \times C_{hem}\right)\right] \times \eta_{Scale} \quad (8)$$

Recall that:

$$Y_{BMP} = \left(Y_{Buswell,glu} \times C_{glu}\right) + \left(Y_{Buswell,hem} \times C_{hem}\right)$$

3.2.4. Livestock residues

The data for the livestock population from 2008-2018 was obtained from FAOSTAT [23]. The residue considered was excreta (dung) estimated for each livestock following Eqs. (9) and (10).

$$Y_{man}(theoretical\ potential) = P_{livestock} \times EMP \quad (9)$$

Where, Y_{man} = Manure produced; EMP = estimated manure produced per day (kg/day)

$$Y_{man}(technical\ potential) = Y_{man}(theoretical\ potential) \times Rf \quad (10)$$

3.2.4.1. Biogas potential from livestock residue

The biomethane potential from manure was estimated from Eq. (11), with biomethane potential (Y_{BMP}) = 0.26111 m^3 CH_4/kg solid.

$$LMM = Y_{man}(technical\ potential) \times C_{TS} \times VS \times Y_{BMP} \quad (11)$$

LMM = Livestock manure methane (m^3/year)

VS = volatile solid fraction (kg solid/year)

C_{TS} = total solid (%)

3.2.5. Municipal waste

3.2.5.1. Municipal Solid Waste (MSW)

The quantity of municipal solid waste (MSW) was calculated from the population of major cities like Lagos [24] using Eq. (12). Sixteen (16) cities were considered. The organic fraction yield (C_{OF}) of the MSW was obtained from the literature on the various cities.

$$P_{MSW} = EP \times WG \times O_{wc} \tag{12}$$

where, P_{MSW} = total municipal solid waste production (kg/day); EP = estimated population per city (person); WG = waste generated (kg/person/day); O_{wc} = organic fraction

The estimate of biomethane potential from municipal solid waste was determined using Eq. (13).

$$Y_{biometane}(MSW) = P_{MSW} \times C_{OF} \times C_{TS} \times Y_{ABMP} \tag{13}$$

P_{MSW} = municipal solid waste (kg)

C_{OF} = Organic fraction yield

C_{TS} = Total solid yield

Y_{ABMP} = Average biomethane potential (m^3/kg) [19]

3.2.5.2. Municipal Liquid Waste (MLW)

Wastewater is an example of municipal liquid waste. The mixture of excreta, urine and water from the toilet make up wastewater for bioenergy generation. The potential biomethane from municipal liquid waste (MLW) is a function of the product of the quantity of liquid waste from the estimated population, the concentration of total solids, and biomethane potential, as shown in Eqs. (14) and (15):

$$P_{MLW} = EP \times AWE \tag{14}$$

EP = estimated population per city; AWE = average weight excreta per person per day (250 g) as derived by Feachem et al. [25].

$$Y_{biomethane}\ (MLW) = P_{MLW} \times C_{solid} \times VS \times Y_{CH4} \quad (15)$$

P_{MLW} = municipal liquid waste production (kg)

C_{solid} = solid yield (8.9275 g /100 g)

VS = volatile solid fraction (% VS)

Y_{CH4} = methane yield (m^3 CH_4/kg VS)

Note: The municipal liquid waste referred to in this study is made of liquid and dissolved human waste which form sewage sludge (a semi-solid waste) [26].

For municipal liquid waste, the volatile solid (VS) refers to the organic content/fraction of the waste, while the solids yield (C_{solid}) was assumed to be 8.9275 g /100 g [27]. Other factors used for the conversion are shown in Table 3.5.

Table 3.5: Indices for estimating the biogas potential of residues and wastes

Factors	Unit value	Reference
Volatile solid fraction	64.7% VS	[28]
Lower calorific value of CH_4	10 kWh/m^3 STP	[29]
Average methane yield VS reduction (MSW)	0.24 m^3 CH_4/kg VS	[30]
CH_4 yield (MLW)	0.525 m^3 CH_4/kg VS	[27]
Energy potential of CH_4 conversion to TJ/yr	0.278 GWh/yr	[19]
TJ to Mtoe conversion factor	24	

3.2.6. Data analysis

The data collected were analyzed using Microsoft Office Excel version 2016. Originlab 9 was used to plot the graphs.

3.3. Results

3.3.1. Crop Production and residue potentials

The residues from the crops considered include the straws, stalks, cobs, pods, shells, peels, and husks from the harvesting (field-based residues) and processing (process-based residues) activities.

The annual theoretical residues from a total of 27 sources (17 crops) showed a total value of 126, 116, and 119 Mt for 2016, 2017, and 2018, respectively (Table 3.6). The technical residues were also found to be 97, 89, 91 Mt for 2016, 2017, and 2018, respectively (Table 3.6). However, in 2009 both residue potentials (i.e., theoretical, and technical) had the least values.

The average crop production, theoretical and technical residues across the investigated period are 142 (Table 3.2), 109, and 84 Mt, respectively (Table 3.6). These values differ from the lowest and highest obtained data. Therefore, it is inferred that crop production and technical residues can sustain biofuel production.

Table 3.6: Estimated crop potential residues and bioenergy potentials

Year	Theoretical (Mt)	Technical (Mt)	Cellulosic ethanol		Biogas	
			ML/yr	Mtoe	Mm^3 CH_4/yr	Mtoe
2008	115.82	90.53	15578.77	8.52	15859.26	13.69
2009	90.45	71.10	12237.41	6.69	12405.56	10.71
2010	106.94	84.18	14429.09	7.89	14534.41	12.55
2011	93.88	72.37	13023.50	7.12	13198.36	11.39
2012	103.69	81.06	13835.72	7.56	14024.91	12.11
2013	97.14	75.60	12800.56	7.00	13095.00	11.31
2014	112.97	86.81	15529.04	8.49	15747.22	13.59
2015	115.95	89.13	15926.22	8.71	16173.43	13.96
2016	125.79	97.20	17226.24	9.42	17571.09	15.17
2017	116.14	88.66	15754.78	8.61	16144.15	13.94
2018	118.54	90.84	16088.55	8.79	16404.53	14.16
Average	**108.85**	**84.32**	**14766.35**	**8.07**	**15014.36**	**12.96**

Mt= million (Mega) tonnes; Mm^3 = Mega cubic meter (volume); Toe: tonne of oil equivalent is a unit of energy defined as the amount of energy released by burning one tonne of crude oil. Mtoe = one million toe.

3.3.2. Bioenergy potential from crop residues

3.3.2.1. Solid biofuel potential

Wood biomass is still used for energy purposes (in form of wood fuel) in Nigeria. The production of wood fuel shows an increasing trend from 2008-2018 (Fig. 3.2). This trend can escalate due to high demand with respect to the population. Further increase in the use of wood fuel contributes to climate change. However, maximizing the energy potential in crude crop residues can drastically reduce the direct combustion of wood. The solid fuel energy available in these crop residues was highest in 2016, followed by 2018 and 2017 (Fig. 3.3), with an average wood fuel production of 68.53×10^6 m^3.

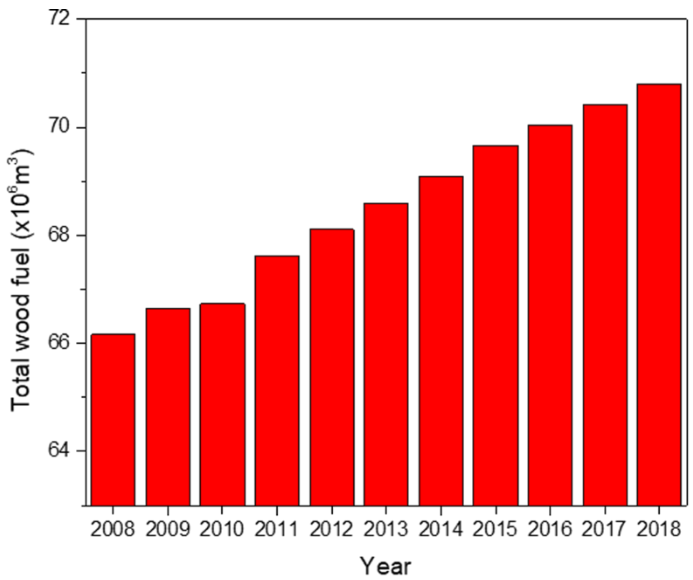

Fig. 3.2: Total annual wood fuel production. (Total wood fuel = Wood fuel + charcoal; Table S1, suppl. material)

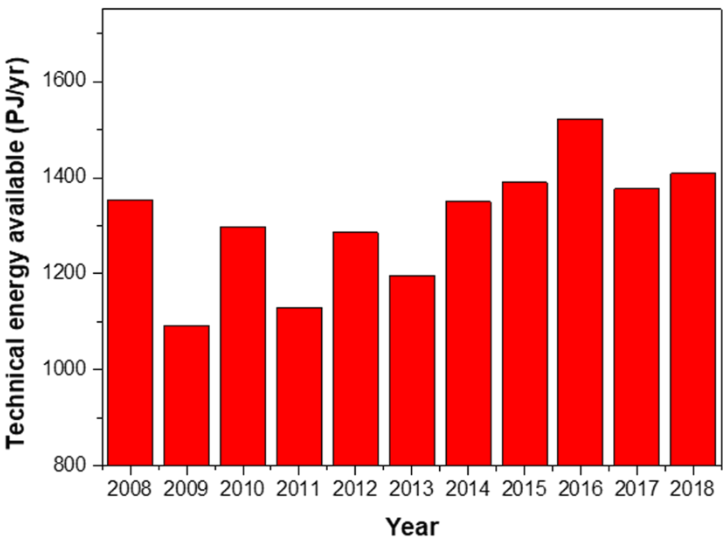

Fig. 3.3: Solid fuel potential showing the technical energy available (TEA) in crop residues generated annually in Nigeria.

3.3.2.2. Cellulosic ethanol and biogas production from crop residue

The estimated cellulosic ethanol production was highest in 2016-2018 (Table 3.6). Similarly, the energy from cellulosic ethanol followed the same trend. Since the volume of ethanol produced is greatly influenced by the quantity of residues, the particle size and enzymatic digestion are very important.

3.3.3. Residue and Bioenergy Potential from Forestry

3.3.3.1. Estimated Residue from Forestry

The estimated residues (i.e., the volume of sawdust, wood bark, and wood slab) generated during the harvest and processing of round wood, for industrial use, are given in Table 3.7. The variation in the generated residues from 2008-2013 and 2014-2018 was mainly due to the significant increase in the volume of industrial round wood harvested and processed in 2014. It is worth noting that the two groups (2008-2013 and 2014-2018) emerged due to a significant increase in wood production in 2014 (**Table S2, Suppl. Material**). Hence, we adopted such classification for better comparison and discussion.

Table 3.7: Estimated residues generated from forestry

Residues	Estimated average residues generated (m^3)	
	2008-2013	**2014-2018**
Saw dust	360,408	379,249
Wood Bark	71,621	75,424
Wood slab	1,352,490	1,422960
Total	1,784,519	1,877,633

3.3.3.2. Cellulosic Ethanol Production from Forest Residues

Cellulosic ethanol production from forest residues (i.e., wood slabs, wood bark, and sawdust) was also higher in 2014-2018 compared to 2008-2013 (Fig. 3.4). The treatment conditions were selected for estimating and assessing the maximum quantity of cellulosic ethanol, given the recalcitrant nature of the cell walls of forest trees. In both 2008-2013 and 2014-2018 groups, a higher cellulosic ethanol yield was obtained when compared with the no pre-treatment scenario (Fig. 3.4**)**. Pre-treatment condition is an important factor for maximum cellulosic ethanol yield from forest residues.

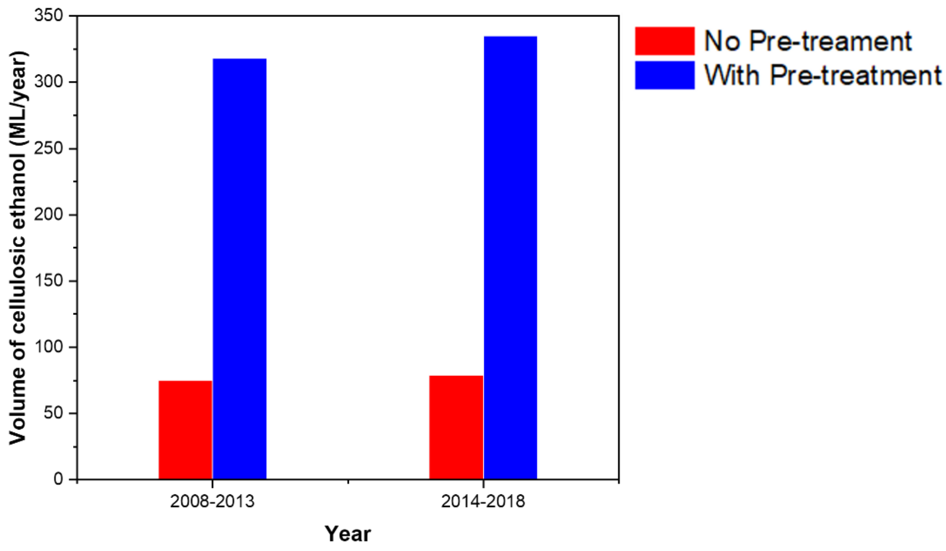

Fig. 3.4: Volume of cellulosic ethanol produced from forest residue with and without pre-treatment.

3.3.3.3. Biomethane Potential from Forest Residue

The biogas production from forest residue is relatively higher for the 2014-2018 period compared with that estimated for 2008-2013 (**Fig. 3.5; Table S3, Suppl. Material)**.

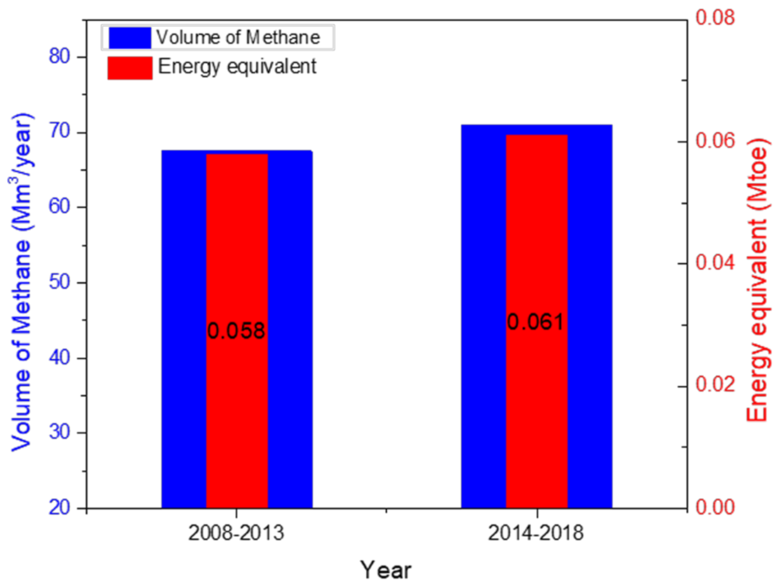

Fig. 3.5: Estimated volume of biomethane and the energy equivalent.

3.3.4. Livestock

3.3.4.1. Livestock Production

The total livestock production varied from 272 million (in 2014) to 308 million livestock (in 2011). Although, in 2011, individual livestock such as chicken and pigs experienced a significant drop in production. However, pig production, unlike chicken, showed a substantial increase and exceeded that of 2010. Despite these changes, the total annual livestock production showed a rising trend in the later years (i.e., 2014-2018). This can be attributed to the growing population (**Fig. 3.6; Table S4, Suppl. Material**).

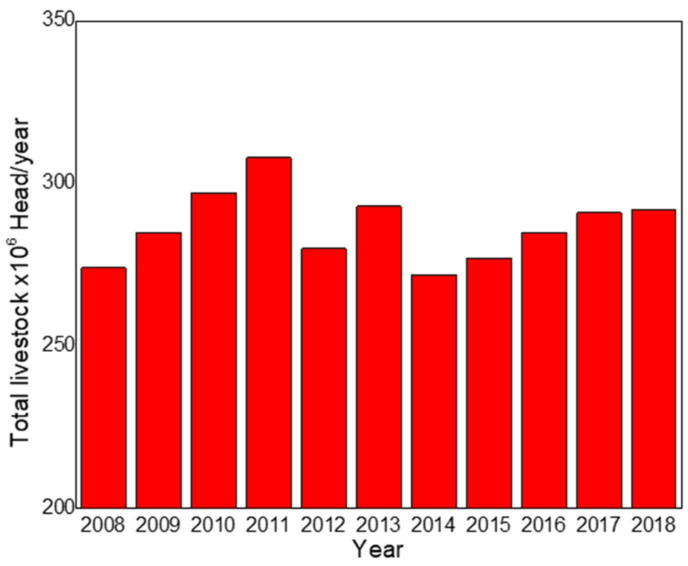

Fig. 3.6: Total annual livestock production

3.3.4.2. Biogas Potential from Livestock Manure

The bioenergy potential measured from recoverable livestock dung in the form of biogas was determined (Fig. 3.7). The result recorded the highest and least recoverable dung in 2018 and 2008, respectively (Table 3.8). Also, the estimated biomethane potential within the investigated 11-year period showed an increasing trend. A remarkable increase in bioenergy potential from livestock manure was observed in 2011, which may be due to the high production of cattle, goats, and sheep recorded in that year (**Table S4, Suppl. Material)**. From Fig. 3.7, a linear relationship was observed in the methane potential and the estimated energy equivalent.

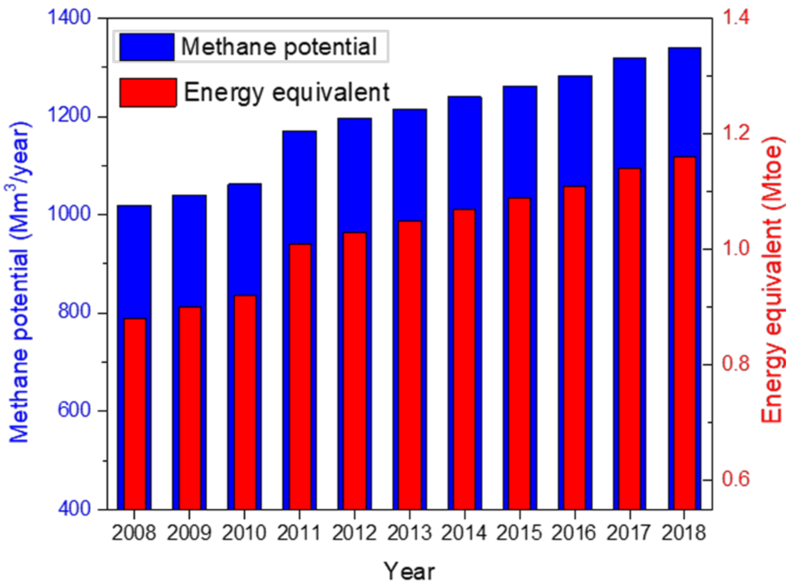

Fig. 3.7: Estimated energy potential of methane from livestock.

Table 3.8: Estimated livestock dung

Year	Dung Produced (million kg)	Recoverable Dung (million kg)	Dung VS per day (10^6)	Dung VS per yr (10^9)
2008	345	77.6	10.7	3.90
2009	351	79.1	10.9	3.98
2010	357	80.6	11.1	4.07
2011	401	87.9	12.3	4.49
2012	408	89.6	12.5	4.58
2013	414	91.0	12.7	4.65

Year	Dung Produced (million kg)	Recoverable Dung (million kg)	Dung VS per day (10^6)	Dung VS per yr (10^9)
2014	422	92.9	13.0	4.75
2015	430	94.9	13.2	4.83
2016	438	96.6	13.5	4.92
2017	449	98.8	13.8	5.05
2018	457	100	14.0	5.13

*VS= Volatile solid

3.3.5. Municipal Wastes

3.3.5.1. Municipal Solid Wastes

The waste generated by the population of 16 major cities (representing the four geographical regions in Nigeria) was evaluated for its biomethane potential. An increase in population gave a corresponding rise in the waste generated from food and other biodegradable materials (**Fig. 3.8**; **Table S5, Suppl. material**). These cities were: North (Abuja, Kano, Makurdi, Maiduguri, and Kaduna), South (Benin City, Port Harcourt); East (Onitsha, Enugu); West (Ife, Ilorin, Akure, Ado-Ekiti, Abeokuta, Lagos, and Ibadan).

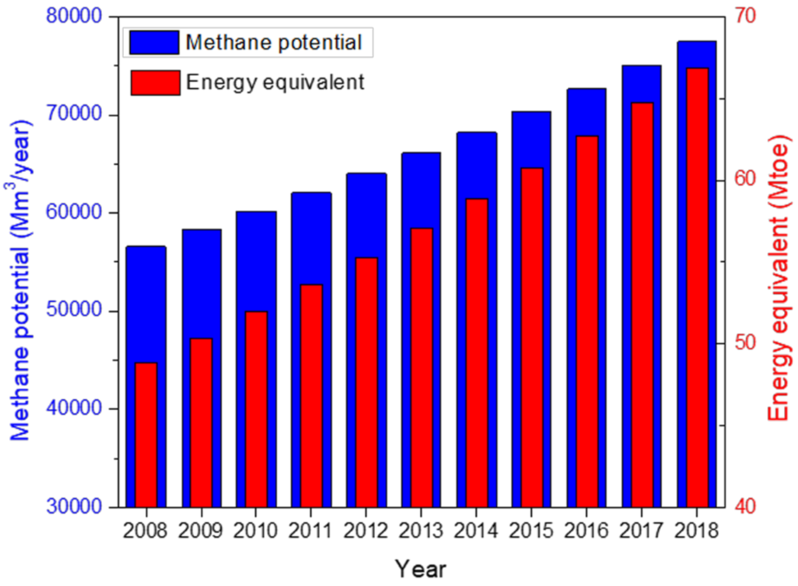

Fig. 3.8: Biomethane potential and energy equivalent estimate of municipal solid waste.

3.3.5.2. Energy Potential from Municipal Liquid Wastes (MLW)

The municipal liquid waste of the 16 major cities was estimated based on the assumption that a person produces an average of 250 g fecal waste daily [25, 31]. The estimated liquid waste increases per year with population growth, which subsequently leads to a rise in the biomethane potential (**Fig. 3.9; Table S6, Suppl. material**).

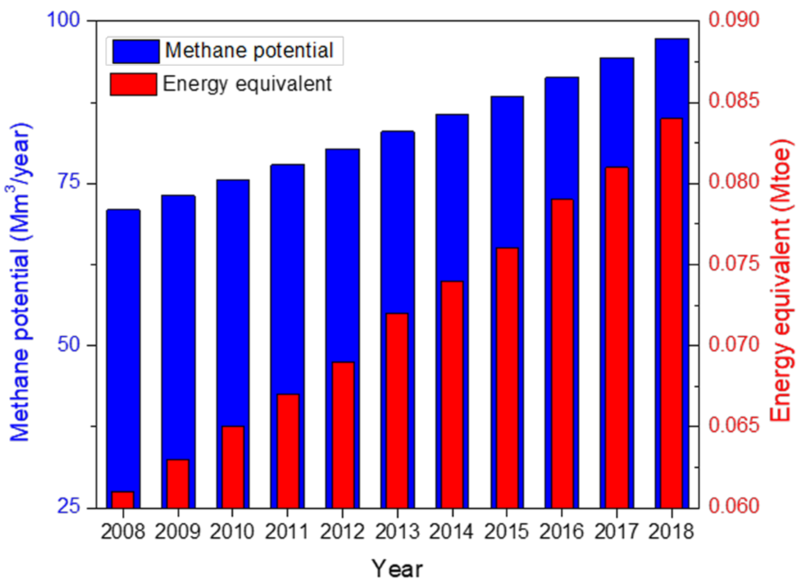

Fig. 3.9: Biomethane potential and energy equivalent of municipal liquid waste

3.4. Discussion

The present study on crop production in the last 11 years does not follow the increasing crop yield as reported by Jekayinfa and Schloz [11]. High quantities of technical potentials were recorded, and different forms of energy carriers with increased energy efficiency were estimated. However, when other potentials (such as environmental, socio-economic, and sustainable potentials) are taken into account, the overall generated residue potential may reduce. On the other hand, both the theoretical and technical residue potentials fluctuated within the investigated period. In the agricultural sector, farmers need to be enlightened on the importance of residues for energy generation. This will enable better collection and storage practices. Also, this awareness can potentially increase the number of agricultural residues. Crop residues can be processed by various techniques, which include gasification and pyrolysis (for biogas, bio-oil and biochar), fermentation (for cellulosic ethanol), and briquettes (as solid fuel) [32]. Solid biofuels (in the form of pellets and briquettes) made from residues of forest and crops are good alternatives to wood fuel and charcoal, as they potentially reduce the felling of trees and deforestation. Residual biomass from the enzymatic or fermentation process for cellulosic ethanol may further be processed into pellets [33] for combustion purposes. There is a market for pellets in Nigeria because the use of wood fuel is high [34]. The bioenergy produced from solid fuel depends on the generated technical crop residues. Similarly, the potential energy from crop residues follows the crop production trend. Briquettes and pellets made from crop residues can serve as wood fuel, thus, reducing the demand for conventional wood fuel and charcoal. Cellulosic ethanol is a liquid fuel obtained from the digestion of lignocellulose components of crop residues, which can be used in place of petrol [19]. On one hand, the quantity of cellulosic ethanol estimated from the residues' assessment was high from 2016 to 2018. However, the conversion processes of crop residues to biofuel, as well as the cost, must be considered. Also, the selection of suitable conversion technique is necessary for optimal ethanol yield. Although the estimated cellulosic ethanol has a huge potential as transport fuel with high-performance efficiency (in vehicles including racing cars), their optimal production is limited due to the recalcitrant structure of the cell wall [35, 36]. On the other hand, biogas production is more efficient compared to cellulosic ethanol, as indicated by the inherent potential energy measured in fossil fuel equivalent (Mtoe) in Table 3.6.

The increase in the use of wood fuel (Fig. 3.2) is primarily a result of the rise in population and poverty. Correspondingly, high wood fuel demand leads to deforestation. The felling of trees for energy purposes plagues Nigeria with the tragedy of climate change, soil infertility (due to erosion), and forest area depletion. Secondary biomass, which includes forest residues, serves as an alternative to wood fuel, for diverse energy forms. These residues are from fallen branches and wood barks during sawmilling and logging processes. Cellulosic ethanol and biogas can be obtained from forest residues. About 70% of the biogas produced now is used for power and heat, 20% is used for cooking, and the remaining 10% is converted to biomethane, according to WEO and the IEA [1, 2, 37]. The reduction of emissions from well-run biogas projects is accompanied by co-benefits like rural development and local job creation. The energy efficiency for biogas implies that biogas is suitable for electricity generation and can positively influence the power condition in Nigeria if properly appropriated. These power sources can serve the inhabitants of the rural areas where bioenergy plants are likely to be situated. Biofuel will not only reduce the adverse effect of smoke from the direct combustion of wood fuel during cooking on the health of the rural dwellers but also provide an alternative clean cooking energy source [38]. Besides cooking, modern forms of solid bioenergy electricity generation, as feedstock for produce liquid and gaseous biofuels [1,2]. However, regarding liquid biofuel, the degree of the recalcitrant varies with the age and maturity of forest residues, poses a challenge. For optimizing cellulosic ethanol production, the type of pre-treatment selected ensures a high estimate and resulting biofuel. Also, reducing the particle size of the residue enhances the surface area for effective hydrolysis. Moreover, smaller particle size promotes solubility and biodegradability of organic matter, leading to a significant increase in the cellulosic ethanol yield (Fig. 3.4).

The animals produced in large quantities in Nigeria include chicken, goats, sheep, cattle, and pigs (**Table S4, Suppl. material**). There is a direct relationship between the amount of manure generated and the quality of food intake when considering the weight of the animal. As shown in Table 3.8, the estimated dung generated, and the amount recovered for biogas production was rising monotonously per year (Fig. 3.7) despite the fluctuating livestock production (Fig. 3.6). This result agrees with the work of Suberu et al. [39] and confirms that Nigeria has a high potential of generating an enormous amount of biogas from animal dung. The present study does not include data from domestic livestock farmers from rural households in Nigeria due to the lack of certified data. The recoverability of the manure from livestock is quite a challenge except in the case of large and mechanized farms that utilize intensive farm practice for commercial purposes. Cattles have the potential of producing higher manure, but most farmers in Nigeria use the nomadic approach. The latter limits the amount of cattle dung for energy purposes. Hence, the quantity of manure recovered is about 50%. Better farm practices and management can enhance the recoverability of animal dung. Nigeria may have to impose mandatory intensive cattle rearing practices. Moreover, intensive farm practices are also economical in food management as the cattle eat more and burn fewer calories, as a result, a higher quantity of manure can be generated.

The high volume of biogas from both MSW and MLW (Fig. 3.8 and 3.9) may be ascribed to the high population, a consequence of migration to these major cities. This migration is mostly an indirect effect of social factors such as job search, a quest for improved living standards, industrialization, urbanization, and insurgency. The quantity of feces and urine excreted per day is a function of the climate, diet, volume of water consumed, and the occupation of an individual.

In our assessment, among the various energy carriers, biogas presents the highest potential and capacity for the development of both integrated and flexible bioenergy strategies in Nigeria. According to World bank data and world info, Nigeria consumed an average of about 2.2 Mtoe (24.72 bn KWh) of electric power per year [40, 41], of which the average estimated energy equivalent of biomethane? from crop residues and municipal solid waste combined can yield over 30% increase in energy for consumption. Therefore, biomass has a significantly high potential to improve the available electric energy supply, thereby providing a solution to the power outage problem currently experienced in the country. Our findings agree with Sobamowo and Ojolo [42]. Although there is a linear relationship between the methane potential and the energy equivalent of biogas, the estimated energy was lower than the volume of methane (Fig. 3.8 and Fig. 3.9). This result may be ascribed to the thermodynamic factors involved in the conversion of biogas to heat energy.

From an economic point of view, waste is a resource from production process, which reduces the extraction of fresh materials and the related energy consumption. The circular economy is a regenerative system that supports the optimal use of resources and waste, thus leading to an economic and ecological resource closed loop [43, 44, 45]. In the context of the present study, the circular economy approach prevents resource depletion (resulting from improper waste incineration or decomposition), high carbon footprint, and ensures production-consumption operations that promote sustainable growth along with the social well-being of Nigerians.

3.5. Biofuel Potentials and Challenges

3.5.1. Cellulosic Ethanol and Biogas Potentials

The potential for energy generation from waste, as well as its ability to control waste management, is of great benefit to the rapidly growing population. Nigeria can leverage the latter and the vast arable land to produce crops and residue generation for energy purposes.

Biomass gasification technology produces relatively clean energy that consists of methane and hydrogen gas from the carbon-based feedstock. The effluent from anaerobic digestion can be used as fertilizer to enhance the soil nutrients and maintain high crop production [27]. The lignocellulose nature of crop and forestry residues possess high biogas energy potential due to its rich methane content.

The conversion technology employed to transform biomass to biofuel depends on the quality of the feedstock. Poor feedstocks with 60-65% moisture content are preferably processed into other forms of biofuel. This diversification ensures an optimum biofuel recovery. The application of pre-treatment conditions (such as drying the biomass), improves its quality for gasification. Nigeria has high solar radiation capable of drying feedstock at a low cost. Besides, solar resources are abundant in regions where sufficient cereal residues are produced. The benefits and challenges of producing biogas or cellulosic ethanol from biomass residues are presented in Table 3.9.

Table 3.9: Potential benefits and challenges in cellulosic ethanol and biogas production in Nigeria.

	Factors	Biogas	Cellulosic ethanol
1	Bio-digester	Simple	Complex to handle due to multiple purification processes.
2	Feedstock type	Relatively dry and low moisture biomass are preferred for biogas production	All type of feedstock is suitable as water is required.
3	Energy cost	No drying is required.	High energy is needed for drying, grinding, and the purification of ethanol.
4	Technology	Low technical know-how is needed, at a low or medium biogas scale.	Advance technology is essential in both the design and installation of hardware for industrial ethanol production.
5	Research	Little research and development in the area of inoculation for constituent biogas production.	To overcome the recalcitrant nature of the biomass, constant R&D is necessary, even in the area of genetic modification of cellulose.
6	Products	Methane, CO_2, H_2, etc.	Cellulosic ethanol, water, fertilizer, and other recyclable products.
7	Cost	Relatively low-cost compared to ethanol production.	Enzymes, microbes for hydrolysis and fermentation, and equipment are capital intensive.
8	Engine modification	Needs regular adjustment	No intensive adjustment is required.

Source: [46, 47].

The comparison between biogas and cellulosic ethanol production (Table 3.9) has shown that the process of biogas production is simple, feasible, and less expensive [48]. Therefore, it is more appropriate to start with biogas production.

3.5.2. Challenges

The production of either biogas or cellulosic ethanol is feasible in principle, considering the availability of different types of residues and the high demand for a steady power supply. However, some challenges could potentially limit its viability in Nigeria, as discussed subsequently.

First, the assessment of biomass residues, as well as the estimation of total bioenergy potentials, involves many uncertainties. The latter can affect the available residue potential. Secondly, the technical residue potential is usually lower than the theoretical one. This reduction emanates from the various value chain of the residues. The competition makes it expedient to source biomass residues solely for energy production. In this regard, there is a need to identify other crop residues that have little or no competitive use. These crops include energy crops, grasses, algae, and other aquatic plants. Furthermore, poor mechanization may limit the collection as well as the conversion method involved in processing the residues [49]. The lack of data on some biomass (e.g., grass) with high bioenergy potential has contributed to insufficient information on the total residue estimate available in Nigeria. A more comprehensive residue valuation should include energy crops such as *Jatropha curcas* and aquatic weeds (water hyacinth, water lettuce, and bracken fern), which are abundant in swampy regions. There is also a need to regularly update the National biomass database.

The estimates for solid and liquid waste produced in Nigeria focused on the major cities, are shown in **Table S5 and S6 (Suppl. Material)**. Although these cities account for the large and diverse forms of waste estimated due to the high population, it represents only a fraction of the total population (16 major cities out of 36 States in Nigeria). Nonetheless, it is difficult to assess the data for major cities needless to consider those cities in rural areas. This barrier hinders the detailed assessment of municipal waste generated in Nigeria. Currently, only Abuja city practices a central sewage system while others practice a system where a few households are connected to a septic tank. Regarding MSW, the Nation needs to adopt a solid waste disposal practice, properly sorting waste into different categories. This will ensure better processing of MSW into energy carriers.

Another challenge in the realization of biofuel production hinges on infrastructure. This includes investment in bio-digesting systems, structural facilities, and technologies required for an efficient biofuel yield.

3.5.3. Implications on the bio-economy of Nigeria

An essential focus of the bioeconomy is the production and processing of biomass wastes into value-added products [50]. The valorization of biomass residue is connected to the sustainable utilization of renewable biological resources (which includes food, bio-based products, and bioenergy) leading to the restoration and preservation of biodiversity. Therefore, the bio-economic perspective provides a balance to the social, environmental, and economic benefits that promote the use of renewable resources, allowing an optimal trade-off between food and bioenergy production.

The implication of our assessment on the bioeconomy of Nigeria includes:

1. Prompts the implementation of good farm practices that will increase crop production, food security, residue generation, and consequently create jobs for the unemployed. Also, it leads to a sustainable ecosystem.
2. Provides business opportunities for innovative start-ups that will attract foreign investment in value-based products for the global market. This could position Nigeria at the forefront of the bioenergy market in Africa.
3. Diversification into bioenergy generation will enable a healthier environment and reduce greenhouse gas emissions from fossil fuels.
4. Decrease our overdependence on foreign nations, thereby making Nigeria's economy tend towards self-reliance (reducing external debits).

5. Enforce collaboration among researchers of various fields as well as the cooperation between Nigeria and other countries towards the establishment of functional bioenergy plants.
6. Facilitates the transition to a circular bioeconomy, as the information on the residues generated, their availability, and the bioenergy potential are valuable for policymaking.

3.5.4. Recommendations

The energy equivalent from crop residues is higher for biogas production than for cellulosic ethanol. Also, livestock manure, MSW, and MLW can be preferably processed into biogas. Hence, leading to a higher volume of biogas compared to cellulosic ethanol. Since biogas can easily be converted to electricity, Nigeria can partly deal with its electricity challenge by focusing on biogas production. Furthermore, the assessment and estimation of the bioenergy potential from biomass residues in Nigeria are but one side of the coin. A more holistic approach that accounts for the cost of establishing a functional biogas plant for residue conversion should also be taken into consideration. The concept of bioenergy from biomass resources involves a multi-dimensional study that includes raw material availability, assessment, and energy potential. It also covers various divisions from agriculture through the industrial, government, and power sectors. However, the socio-economic influence towards bioenergy establishment is another measure of its sustainability [51, 52, 53].

Finally, the implementation of proper biofuel policy is expedient, in this regard, the government plays a vital role in the exploitation of natural resources and the attainment of environmental sustainability [54]. However, sustained biofuel production requires the cooperation of other stakeholders [55, 56], as illustrated in Fig. 3.10. It is important to note that promoting the use of biogas in Nigeria may require the introduction of subsidies [57].

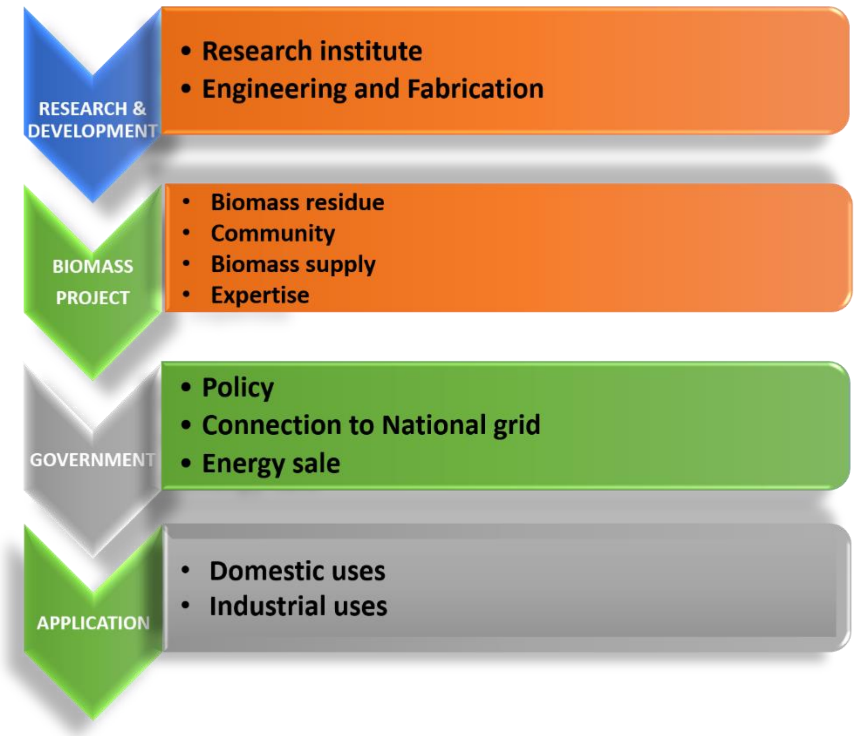

Fig. 3.10: Structural framework showing the stakeholders in bioenergy from biomass resources

3.6. Conclusions

The assessment of biomass residues and their bioenergy potential is often performed for either solid biofuel or biogas. However, in this work, we estimated the bioenergy potential from solid biofuel, liquid biofuel, and biogas perspectives. We discovered that 143 Mt of crop residues produces about 84 Mt of technical residue potential on average. Hence, only about 58% of the total residue is available for energy purposes. Our findings revealed that crop production is directly correlated with the quantity of biofuel produced. For the forest residues, enzyme pre-treatment led to higher cellulosic ethanol. Among the bioenergy carriers evaluated, biogas had the highest potential, with an average of 15014 Mm^3 from crop residues. Therefore, it is a more promising energy carrier to be adopted in Nigeria. Although biogas production is favored, there is a need to investigate its cost, feasibility, and the economic analysis of setting up the plant in Nigeria. Also, the pragmatic behavior of the biomass residues during anaerobic activity (i.e., the breakdown of lignocellulose content) needs to be experimentally validated. Finally, the policies that will facilitate the optimum collection of these biomass residues are expedient.

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CHAPTER FOUR

Preliminary characterization and valorization of *Ficus benjamina* fruits for biofuel application.

4.1 Introduction

Increasing global energy demand and climate goals make it necessary to seek sustainable and eco-friendly energy generation sources. Wood is currently the most common plant biomass used for heating purposes. However, it contributes to the health and environmental challenges [1]. Hence, the search for sustainable and efficient energy sources that make use of other plant biomass as an alternative to both fossil and wood fuel [2]. Among the various renewable energy sources, plant biomass holds prospective high potential due to advancements in conversion technologies such as gasification and pyrolysis. The conversion of lignocellulosic biomass can generate solid biofuel (e.g., pellet and briquettes that can substitute for wood fuel), bio-oil, bioethanol, and biogas.

First-generation biofuel requires food crops and oilseeds, raising food security issues [3]. Second generation biofuel relies on lignocellulose-based biomass, such as field- and process-based residues, which do not compete with food [4], though they contest with other applications such as livestock feed, mulching, and industrial purposes. Third-generation biofuel makes use of microalgae as feedstock. Although the latter has some prospects, it is not currently economical, and research is still at its relatively early stage. Fourth-generation fuel involves the genetic modification of plants and microalgae to reduce the lignin resistance during hydrolysis and increase CO_2 sequestration. This type of biomass possesses tremendous opportunities in contributing to the energy supply chain but pose various problems [5]. In this study, we investigate the potentials of non-edible whole fruits as biomass feedstock for energy production. Non-edible fruits from ornamental and forest trees are practically wasted. Generally, despite their rich lignocellulose contents, they are neither characterized nor included as possible biomass feedstock for biofuel production. It is important to note that the use of these fruits avoids the overdependence on crop residues. Also, the trees producing such fruits do not require re-planting, which makes them economical and sustainable. Weeping fig, botanically called *Ficus benjamina* (FB), is one of such trees. FB is native to Asia and Oceania, although it has adapted beyond its native range, spreading to other continents [6]. The wide range of adaptability allows it to flourish on fertile and moistened soils with sufficient sunlight [7]. It can tolerate drought, a wide range of soil types, and pH range from acidic to alkaline [8]. Due to its appealing properties as an easy-to-grow species and its high-density foliage and dimensions, FB has been introduced massively in urban areas all over the world, becoming a predominant species [9]. FB tree is generally used for ornamental and landscaping purposes [10], and not for their fruit. Nevertheless, they are capable of producing fruits more than two times a year [11]. FB fruits have no known application; hence they have no value and thus can be found as waste in parks, gardens, streets, highways, and riverbanks. In this regard, the novelty and priority of the present study are to understand the potential economic value that can be obtained from pulverized *Ficus benjamina* fruits (PFB). In order to achieve this, we performed fundamental physical, chemical, and thermal characterizations. From the preliminary results, it was discovered that PFB showed a very good prospect as a feedstock for biofuel application. This work intends to attract the attention of the scientific community and the government, such that appropriate programs for the collection of this fruit will be initiated.

To the authors' knowledge, this is probably the first paper that discusses the valorization of *Ficus benjamina* (FB) fruits. Detailed information is provided to access its potential application. For this purpose, the physical, chemical, and thermal properties must be evaluated [12, 13] to allow suitable processing and policy decisions towards biofuel application. As mentioned earlier, there are knowledge gaps with regard to the characterization of FB fruits, especially as feedstock for biofuel production.

Our motivation is to fill in some of the knowledge gaps by investigating the physicochemical and thermal properties. We also discuss the morphology, physical and chemical compositions.

4.2 Materials and methods

4.2.1 FB fruits collection and preparation

FB fruits were collected from the grounds of AUST (African University of Science and Technology, Abuja, Nigeria). Figure 4.1 shows the oval-shaped FB fruits with yellow orange color. The whole fruits collected were washed in clean water to remove impurities, sun-dried, and pulverized in a blender (BLG-403, China). The pulverized FB fruit was labelled as PFB. The latter was sieved using a mesh of 425 μm to obtain particles with size ≤ 425 μm, then stored in clean, air-tight Ziploc bags for characterization.

Fig. 4.1: An image of *Ficus benjamina* plant with fruits

Figure 4.2 illustrates the schematic experimental process and flow chart for valorizing FB fruits. The flow chart displays possible decisions that could be applied for further analysis.

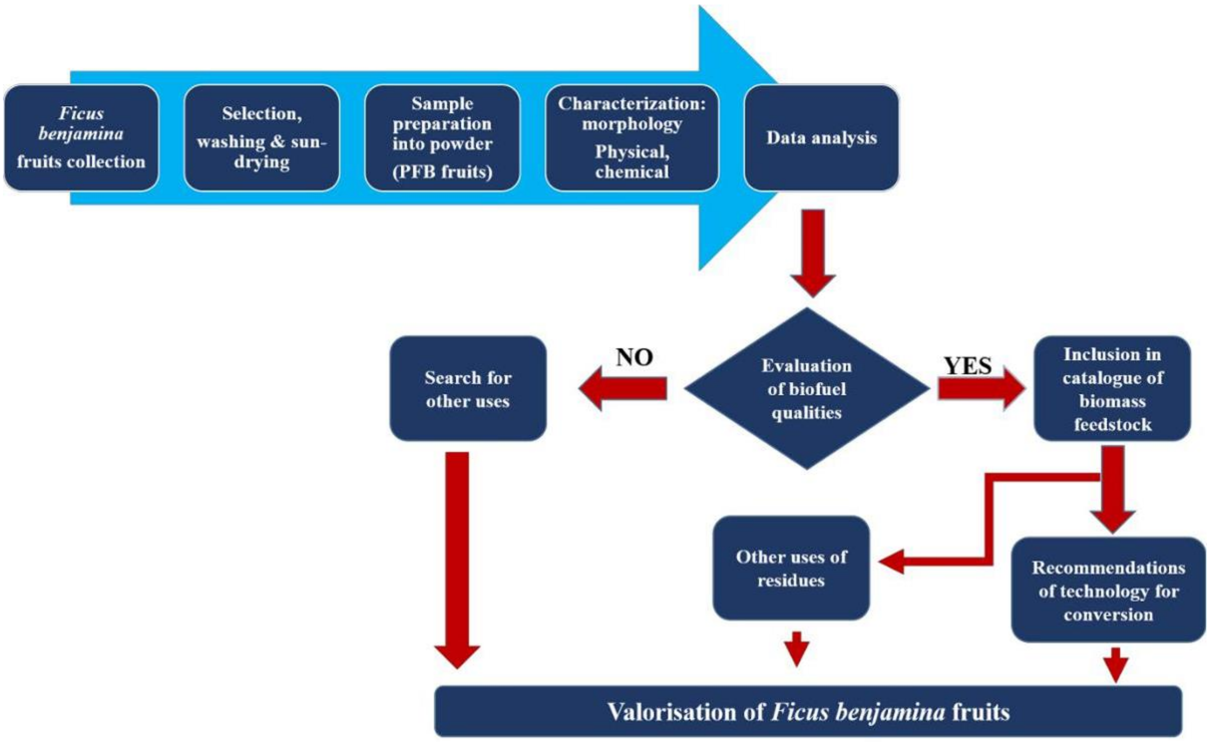

Fig. 4.2: Schematic showing the PFB valorization flow chart carried out in this work

4.2.2 Characterization of PFB

4.2.2.1 Morphological analysis

The morphology of the FB fruit (internal and external surface) and PFB was observed using a scanning electron microscope (ZEISS, EVO LS10, USA). The samples were mounted on conductive adhesive carbon tape then coated with a thin layer of gold to prevent surface charging. Energy dispersive X-ray (EDAX, USA) was used for the quantitative elemental analysis of the FB fruit.

The bulk density of PFB was determined using modifications of the method described by Stella Mary et al. [14]. An amount of PFB was added to a graduated glass cylinder (25 ml) and slightly tapped for 1–2 min to compact the content [15].

4.2.2.2 Proximate and ultimate analysis

The moisture and ash content of PFB was determined using the ASTM D7582 method [16]. The dry solid obtainable from PFB was determined following the approach by Singh et al. [17]. The volatile matter was calculated according to UNE 32,019 [18]. The fixed carbon content was estimated from the proximate analysis [17]. The carbon, hydrogen, and nitrogen contents of PFB were determined using a LECO CHN-2000 analyzer, while the sulphur content was determined in a LECO S-144DR analyser. The oxygen content was estimated according to [17, 19].

The ether extractives yield of PFB was obtained following the Randall method [20]. In a Soxhlet apparatus, PFB (1.0 g) was added in the sample chamber and 250 ml of petroleum ether in the receiver flask, then placed on a heating mantle. After 7 h of extraction, the petroleum ether was recovered using a rotary evaporator. The resulting extract was left in the oven at 70 ℃ until constant weight, then the ether extractive yield was calculated using Eq. 1.

$$Ether\ extractives\ (\%) = \frac{W_2 - W_1}{W} \times 100 \tag{1}$$

where W_1 = weight of empty oil flask; W_2 = weight of oil + flask after extraction; W = weight of PFB.

4.2.2.3 Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) analyses

The functional groups in the PFB were characterized by means of FTIR spectroscopy (Thermo Fisher Scientific, USA). First, PFB was mixed with KBr in a ratio of 1:10 then compressed into a pellet. All spectra were recorded in the absorbance mode at the wavenumber range of 4000–400 cm^{-1}. The XRD analysis of PFB was carried out with Cu-Kα radiation of wavelengths 1.540598 A generated at 40 mA and 45 kV (Empyrean).

The crystallinity index (CrI) of the sample was estimated using the Ruland–Vonk method [21, 22]. This method is based on the ratio of the area of the crystalline profile to the total area (Eq. 2).

$$Crystalinity\ index = [\frac{Area\ of\ crystalline\ peaks}{Area\ of\ all\ peaks\ (crystalline + amorphous)}] \times 100 \qquad (2)$$

4.2.2.4. Determination of the lignocellulose content

The lignocellulose content which comprises hemicellulose, lignin, and cellulose was determined gravimetrically [23]. The PFB (1.0 g) obtained after the ether extractives experiment (i.e., extractive-free PFB) was mixed with 10 ml of 0.5 mol/L of NaOH and heated at 80 ℃ for 4 h. The resulting mixture was washed till the pH became neutral then dried to a constant weight. The hemicellulose content is the difference between the initial (A) and final weights (B) (Eq. 3). For the lignin content determination, 1.0 g of extractive-free PFB was soaked overnight in 30 ml concentrated sulphuric acid (98%), after which, it was boiled at 100 ℃ for 1 h [24]. The resulting product was washed until no trace of sulphate ions was visible in the filtrate when tested with drops of barium chloride (10%). Afterwards, the residue was dried at 100 ℃ until a constant weight was attained, then the lignin content was estimated (Eq. 4).

Furthermore, the cellulose content was found by subtracting the ether extractives, hemicellulose, and lignin contents from 100 (Eq. 5).

$$Hemicellulose\ content\ (wt.\%) = \frac{A-B}{A} \times 100 \tag{3}$$

where A = weight of extractive-free sample; B = weight of dried hemicellulose; wt. = weight.

$$Lignin\ content\ (wt.\%) = \frac{A-C}{A} \times 100 \tag{4}$$

where A = weight of extractive-free sample; C = weight of dried lignin.

$$Cellulose\ content\ (wt.\%) = 100 - (Ether\ extractive\ (wt.\%) + Hemicellulose\ (wt.\%) + Lignin\ (wt.\%) \tag{5}$$

4.2.2.5. Thermal characterization

The calorific value of PFB was estimated as the higher heating value (HHV) and the lower heating value (LHV) using a bomb calorimeter (IKA C 4000). Moreover, the thermal behavior was characterized using thermogravimetric analysis (TGA; PerkinElmer 4000, USA). The de-volatilization was investigated in the temperature range of 30-900 ℃ (10 ℃/min) in a nitrogen atmosphere with a flow rate of 60 ml/min.

4.3 Results and Discussion

The biofuel potential of PFB is discussed based on their morphology, physical, chemical, and thermal properties.

4.3.1 Morphology

The morphology of the FB fruit revealed unique surface patterns, sizes, shapes, and orientations of the various parts (Fig. 4.3 (a)-(f)). As shown in Fig. 4.3(a)-4.3(d), the internal structure has irregular patterns with several cavities, making it less dense. The endocarp (Fig. 4.3(c)) revealed a planar sheet-like structure while the mesocarp (i.e., the region between the outer and the inner portion) showed pores (Fig. 4.3(d)). The lightweight and buoyancy of the dried FB fruit can be attributed to the less dense internal structure. In addition, the outer surface (epicarp) has a compact form with a uniformly distributed rough texture (Fig. 4.3(e)-(f)). For the PFB sample, the morphology (Fig. 4.3(g)) showed irregular shapes with the internal plant structure. This observation is different from that of pulverized unmodified Dikanut shell, which has a scattered orientation, few pores, and dense structure [25].

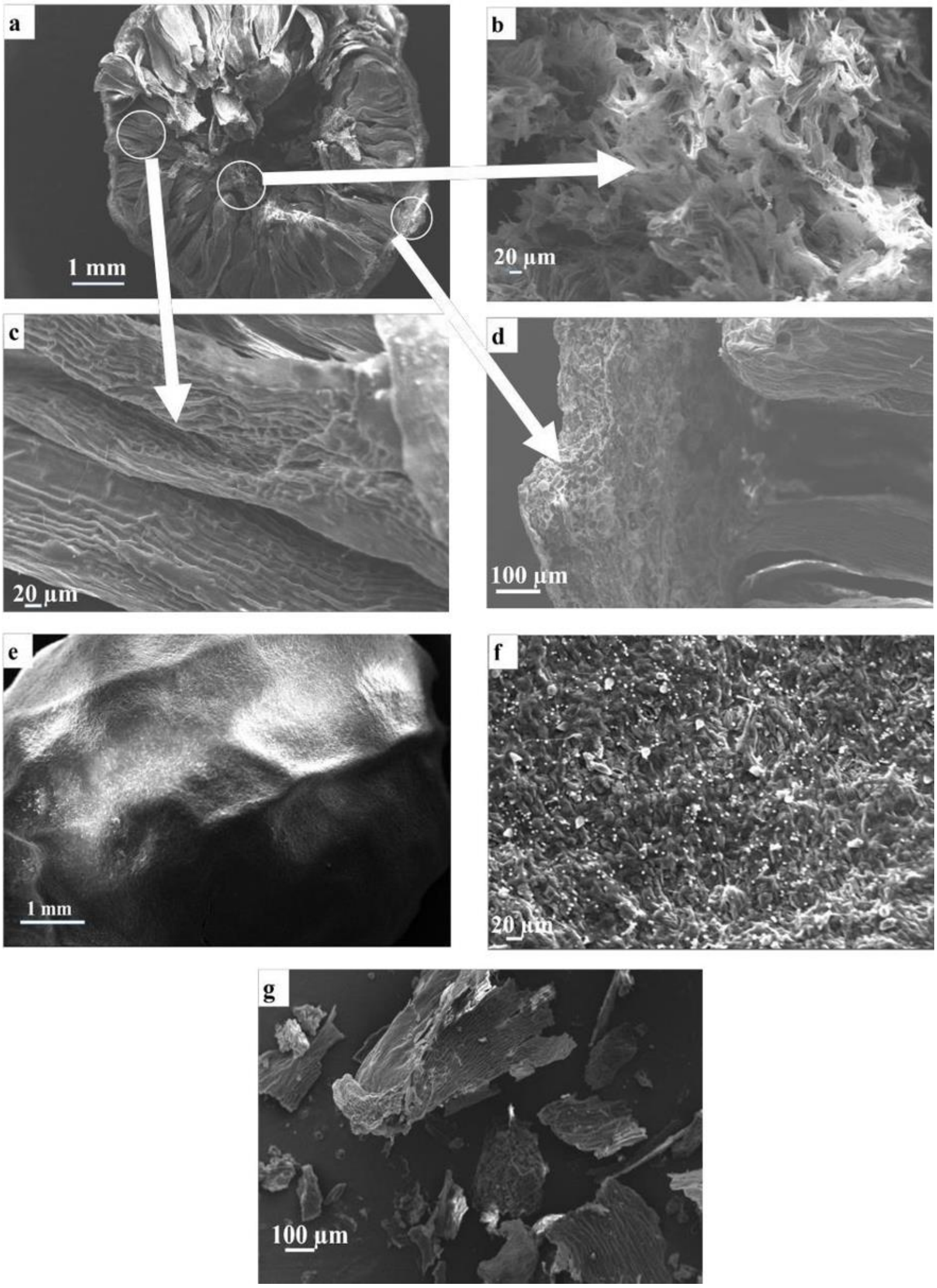

Fig. 4.3: Morphology of *Ficus benjamina* (FB) fruit: (a) the inner structure; (b, c, and d) are the magnified portion of (a); (e) the outer layer; (f) the magnified outer layer; (g) pulverized FB (PFB)

The bulk density of PFB is 0.32 g/ml. This can be related to the large granular particles from the epicarp (Fig. 4.3(e)-(f)) that create inter-particle voids, leading to the low value obtained. The bulk density of PFB is lower than the rice kernels of various rice cultivars (0.77-0.87 g/cm^3) [26]. The density of feedstocks has been reported to significantly influence their behavior during the thermochemical/biological conversion process [27].

The analysis of different parts (outer and inner portions) of the FB fruit by energy-dispersive X-ray spectroscopy (EDS) showed carbon, oxygen, and potassium elements in high concentrations (see Table 4.1). In principle, biomass with low metallic elements is more suitable for energy generation by combustion [28]. The alkali metals (calcium and potassium) identified by EDS can affect thermochemical conversion processes during biofuel production because they lead to unwanted by-products (such as slag, sinter, and foul formation) in the boiler. However, it must be noted that calcium is only present in the outer portion of the fruit and that potassium concentration is much higher in this part than in the inner portion of the fruit. Therefore, it may require the removal of the outer part of the fruit for its use as biofuel. The EDS analysis of other non-edible fruit waste [29] has been included in Table 4.2 for comparison purposes. PFB presents a similar carbon content to banana peel or orange bagasse but a lower oxygen content and a much higher potassium content (Table 4.2). It is worth noting that potassium may play a vital role as a catalyst, thus increasing the rate of conversion of biomass [13].

Table 4.1: Elemental composition of *F. benjamina* fruits by EDS

Sample	**Elements (%)**						
	C	**O**	**Ca**	**K**	**Zr**	**Mo**	**Al**
Outer portion of the fruit	0.03	31.95	36.83	38.54	ND	24.60	ND
Inner portion of the fruit	76.25	10.25	ND	8.85	4.66	ND	ND
PFB	53.41	15.65	ND	24.54	5.18	ND	1.19

ND = Not detected.

Table 4.2: Comparison of EDS elemental composition of FB fruit with other non-edible biomass

Element	PFB	Banana peel [29]	Orange bagasse [29]
C	53.41	52.32	61.94
O	15.65	39.67	37.12
Ca	-	-	0.70
K	24.54	5.93	0.24
Zr	5.18	-	-
Al	1.19	-	-
Si	-	0.49	-
Cl	-	1.59	-

4.3.2 Proximate and Ultimate Analysis

The moisture content of PFB, 9.29 wt.%, is lower than Eucalyptus wood sawdust and comparable to that of wheat straw [30], suggesting its suitability for energy purposes (such as pellets and briquettes) (see Table 4.3). Hence, it is suitable for application in thermal conversion systems for rapid heat transfer and storage. Furthermore, the low moisture content of PFB is beneficial as little energy input may be needed to remove moisture, thus reducing the cost of processing the feedstock. Compared to the other biomasses included in Table 4.3, PFB has the lowest volatile matter content, 64.35 wt.%, although this is just below that of pinewood and wheat straw. Volatile matter is combustible organic matter that may contribute to heat energy generation. Generally, plant biomass is known to have high volatile matter [31, 17], making it readily reactive to oxygen. During pyrolysis, some liquid products (such as bio-oil) can also be obtained, in which case less char will be generated from the feedstock. The fixed carbon of PFB is 20.10 wt.%, which is higher than that of eucalyptus sawdust [32], pinewood [33], grasses [31], seeds [13], and fruit peels [34], and slightly lower than that of wheat stalk.

The ash content of PFB is 6.26 wt.%. This is well below the high limit of 10%, which indicates that it possesses a good potential for thermal utilization [17, 35]. However, the ash content of PFB is higher than most of the biomass types considered in Table 4.3, except King grass. Moreso, the ash content is inversely proportional to the energy derived from the feedstock [36]. The formation of slag from the metallic elements in the ash presents operational challenges in boilers during thermal conversion at high temperatures. PFB may be suitable as briquettes and pellets for cooking and heating purposes in rural areas. Low ash content makes pyrolysis a suitable energy conversion route [37, 29]. The VM/FC ratio is an indicator of the quality of fuels. This ratio is lower for PFB than most of the biomass types included in Table 4.3, but comparable to pinewood and wheat straw.

The ultimate analysis showed a relatively high carbon content, of 50.56 wt.% (daf). Hydrogen, nitrogen, and sulfur contents are 5.82, 1.66, and 0.11 wt.% (daf), respectively, and the estimated oxygen content is 41.85 wt.% (daf). The comparison with other biomass feedstocks, presented in Table 4.3, shows the third-highest C content, the fifth-lowest H content, and the third-lowest O content for PFB. The sulfur content in PFB is low, as expected for biomass sources [19]. This finding is essential because a low oxide concentration of these elements will be formed during combustion. Therefore, PFB can be identified as eco-friendly during combustion, and suitable for energy production via gasification as pollutants are limited [1].

4.3.3 Ether Extractives

PFB produces low-yield ether extractives, 1.48 wt.% (Table 4.4). Since the latter is below 10%, its effect on the thermal conversion is negligible [37]. Therefore, it indicates that less liquid products (such as bio-oil) will be produced during pyrolysis. The extractives (majorly the oil content) are low and solidify even at room temperature, making it not appropriate for biodiesel production. This result agrees with the study on different walnut shells where the extractives were in the range of 1.4-1.7% [37].

4.3.4 van Krevelen Diagram and Biofuel Reactivity

The atomic ratio of H/C and O/C defines the reactivity of biofuels. The H/C ratio reflects the degree of condensation and aromaticity in the plant material. The lower the H/C (high aromaticity), the higher the energy content. On the other hand, oxygen does not make any useful contribution to the heating value but makes it difficult for the transformation of biomass into liquid fuels [19]. In this study, PFB showed the second-lowest O/C ratio compared to all the lignocellulosic materials reviewed in Table 4.5, including coconut shell. Fig. 4.4a shows the position of PFB in the van Krevelen diagram. The location of PFB is closer to fossil fuels than most of the biomass types included in this work, with the exception of that of pinewood and similar to that of coconut shell (Fig. 4.4a). It, therefore, implies that high energy density, stored as chemical energy, may be embedded in the C-C and C-O bonds [17]. The biofuel reactivity plot (Fig. 4.4b) revealed that the atomic ratios (H:C and O:C) of PFB are comparable to other biomass residues. The VM/FC ratio ranges from 3.20-6.64, and PFB was found in a similar position with the wheat straw and King grass. The VM/FC is higher than the atomic ratios, thus showing potential for biofuel, possibly solid biofuel [13].

Table 4.3: Proximate and Ultimate Analysis in comparison to other biomass feedstock

Biomass/ Residues		Proximate Analysis (wt. %)						Ultimate Analysis (wt.%, daf)					Reference(s)
		Mc	VM	Ac	FC	DS*	VM:FC*	C	H	N	S	O[a]	
Wood Sawdust	Eucalyptus	10.10	83.88	0.11	16.00	89.90	5.24	49.90	5.8	0.2	0.03	44.07	[32,37]
	Pine wood	14.00	67.72	0.4	17.88	86.00	3.79	54.30	5.20	0.40	0.00	40.00	[33,38]
Grasses	Switchgrass	6.01	73.32	4.01	16.66	93.99	4.40	49.33	7.31	0.52	0.08	42.58	[31,39]
	King grass	-	78.2	7.1	14.7	-	4.44	46.91	5.89	0.70	0.21	46.30	[40, 41]
Field-based	Corn stover	4.01	75.63	5.13	15.23	95.99	4.97	49.33	5.53	0.88	0.88	44.18	[31, 39]
	Wheat straw	8.45	65.59	4.99	20.97	91.55	3.13	43.20	5.00	0.60	0.01	39.41	[42, 30]
Processed-based	Orange peel	7.91	86.70	5.25	0.14	92.09	619.29	48.74	5.92	1.43	0.19	43.72	[34, 40, 43]
	Coffee husk	7.22	76.60	0.68	15.50	92.78	4.94	46.51	6.77	0.43	0.09	46.20	[44]
	Coconut shell	7.82	79.91	0.23	12.04	92.18	6.64	51.6	5.60	0.10	0.00	42.70	[45, 46]
Non-edible whole fruits	PFB	9.29	64.35	6.26	20.10	90.71	3.20	50.56	5.82	1.66	0.11	41.85	This study

*****: estimated by the current author; a: calculated by difference; daf: dry ash-free basis

Table 4.4: Summary of the Characterization of Biomass PFB

Characterization	Properties	Value
	Bulk density	0.32 g/ml
	Ether extractives	1.48 wt.%
Proximate analysis	Moisture content	9.29 wt.%
	Ash content	6.26 wt.%
	Volatile matter	64.35 wt.%
	Fixed carbon	20.10 wt.%
	Dry solid	90.71 wt.%
Ultimate analysis	Carbon (C)	50.56 wt.% (daf)
	Hydrogen (H)	5.82% wt.% (daf)
	Nitrogen (N)	1.66 wt.% (daf)
	Sulfur(S)	0.11 wt.% (daf)
	Oxygen (O)	41.85 wt.% (daf)
Biofuel reactivity	VM/FC	3.20
	H:C (daf)	1.37
	O:C (daf)	0.62
Lignocellulose composition	Cellulose	27.76 wt.%
	Hemicellulose	48.30 wt.%
	Lignin	21.30 wt.%
	Cellulose/lignin ratio	1.30
	Cellulose/hemicellulose ratio	0.57
Calorific value	Higher heating value (HHV)	19.743 MJ/kg (daf)
	Lower heating value (LHV)	18.549 MJ/kg (daf)

4.3.5. Cellulose/Hemicellulose Ratio

The cellulose/hemicellulose ratio is indispensable in estimating ethanol yield [27]. Biomass feedstock with a high cellulose/hemicellulose ratio yields high ethanol. However, this ratio is low in PFB (0.57) due to the high hemicellulose content (Table 4.5). Therefore, the production of ethanol from PFB will require pre-treatment and additional enzymes for hydrolysis.

Table 4.5: Biofuel reactivity of other biomass compared to PFB fruits

Biomass/ Residues	Name	Lignocellulose composition					Bioenergy activity				Reference (s)
		Cellulose (%)	Hemicellulose (%)	Lignin (%)	C:L*	C:H*	O/C daf	H/C daf	HHV (MJ/kg)	LHV (MJ/kg)	
Wood sawdust	Eucalyptus	43.80	20.70	27.10	1.62	2.11	0.66	1.39	20.00	-	[47, 37, 32]
	Pine wood	37.00	19.00	31.00	1.19	1.95	0.55	1.14	19.66	-	[48, 38, 49]
Grasses	Switchgrass	37.00	28.00	18.00	2.06	1.32	0.65	1.77	17.36		[50, 31,39]
	King grass	36.90	34.20	6.10	6.05	1.08	0.74	1.50	17.98		[51, 52, 41]
Field-based	Corn stover	37.72	20.62	30.50	1.24	1.83	0.67	1.34	17.31	-	[53, 31, 39]
	Wheat straw	45.12	9.16	37.41	1.21	4.93	0.68	1.38	17.25	-	[42]
Processed-based	Orange peel	11.93	14.46	2.17	5.50	0.83	0.67	1.45	18.92		[29, 41, 54]
	Coconut shell	36.13	20.36	32.33	1.12	1.77	0.62	1.29	17.35	-	[55, 46, 56]
	Coffee husk	43.18	10.20	17.42	2.48	4.23	0.75	1.73	15.20	-	[57, 44, 58]
Non-edible whole fruits	PFB	27.76	48.30	21.30	1.30	0.57	0.62	1.37	19.74	18.55	This study

*= estimated by current Authors; C:L = cellulose-lignin ratio; C:H = cellulose-hemicellulose ratio; H/C and O/C are atomic ratios determined using the formula reported by Pach et al. [59] and Ascough et al. [60], respectively.

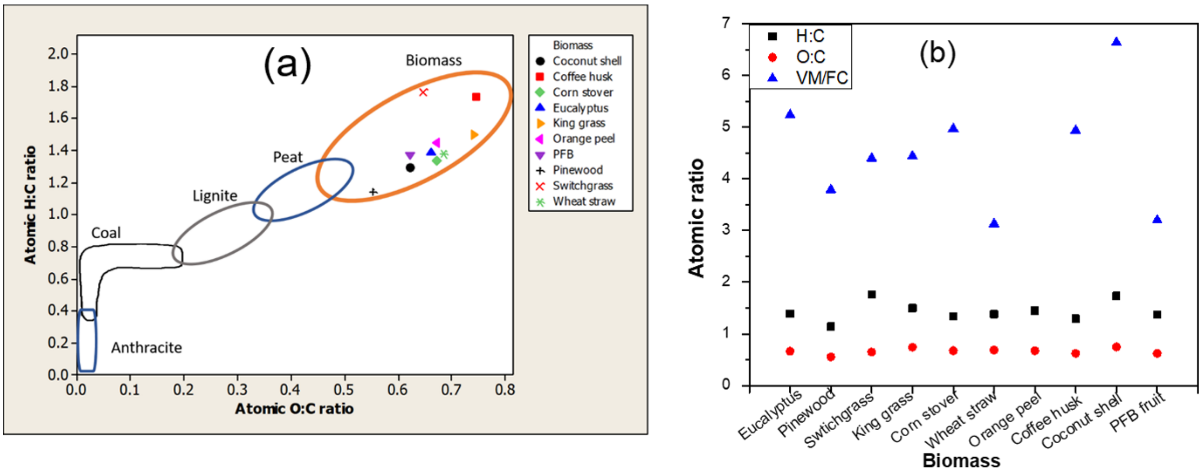

Fig. 4.4: (a) The van Krevelen diagram of biomass showing the position of PFB; (b) Biofuel reactivity plot, comparing PFB with other biomasses

4.3.6 X-ray Diffraction Analysis

In the XRD pattern of PFB (Fig. 4.5(a)), a broad peak was identified demonstrating its low crystallinity. The dominating amorphous nature may be a consequence of the rich carbon content. The latter could be linked to its lignocellulosic character, particularly its high hemicellulose content (48.30%), as shown in Table 5. The crystallinity index for PFB is 25.5%, which is similar to that of soy peels (25%) but lower than Açaí and coffee husk (30%) [32].

Generally, low crystallinity is associated with fast degradation. A narrower peak can also be identified in Fig. 4.5a. This corresponds to calcium oxalate hydrate oxide ($C_2Ca_5O_4.H_2O$), according to the International Centre for Diffraction Data (ICDD) reference card number 00-016-0379, which is identified as whewellite, with a monoclinic structure.

The XRD results are in good agreement with the EDS results, shown in Table 4.1, where Ca and C was identified. The presence of these elements makes PFB a suitable reinforcement agent in composites such as particleboards.

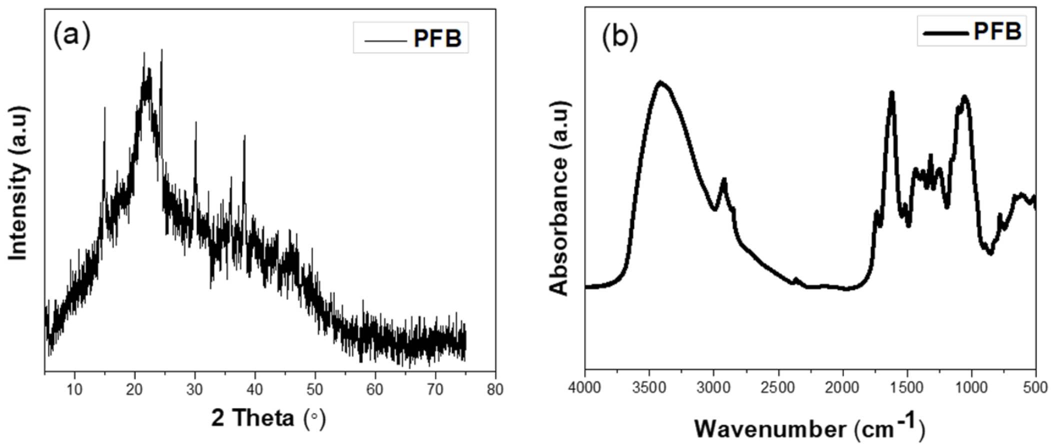

Fig. 4.5: (a) X-ray diffraction pattern; (b) Fourier transform infra-red spectroscopy spectra of PFB

4.3.7 Fourier Transform Infra-red spectroscopy (FTIR) Analysis

In the FTIR spectra of PFB, shown in Fig. 4.5b, alkanes, aliphatic-primary amines, and hydrocarbons are identified (see Table 4.6). These groups provide binding sites for other elements in the fuel matrix [29]. The fingerprint region exhibits peaks that can be attributed to C-H, C-N, and S=O, amongst others. In the diagnosis region, N-H, C=O, C=C, and N-O have strong stretching and bending vibration bonds. The broadband related to the OH bond confirms the presence of alcohols or phenols in the carbohydrate and lignin contents. During thermal hydrolysis, the OH groups in the lignocellulose and the C=O bond from the carboxylic ends may be released. The stretching vibration of C-H could be related to the hemicellulose or the alkyl chain of the lipid content. However, the lignin decomposition might further generate C=C bond. The existence of OH, C–O, C–H, and C=C functional groups in the structure of corn cob has been reported in the literature to facilitate the formation of condensable and non-condensable liquid with gaseous by-products [62].

Table 4.6: FTIR spectra band assignment of PFB

Band frequency(cm^{-1})	Bond	Functional group
3411.32	**OH** Stretching	Alcohol, phenol
3420.67	**N-H** Stretching	Aliphatic primary amines
2919.08	**C-H** Stretching	Alkane
2360.69	**O=C=O** Stretching	Carbondioxide
1734.75	**C=O** Stretching	Aldehyde
1617.78	**C=C** Stretching	α, β- unsaturated ketone
1521.97	**N-O** Stretching	Nitro compound
1382.81	**C-H** Bending	Alkane
1317.93	**C-N** Stretching	Aromatic amine
1250.96	**C-N** Stretching	Amine
1062.09	**S=O** Stretching	Aliphatic amine
781.07	**C-H** Bending	1,2,3-trisubstituted hydrocarbons
520.36	**C-Br** Stretching	Halo compound

4.3.8 Thermal Analysis

The calorific value of biomass is an important parameter to measure its biofuel potential. For PFB, the higher heating value (HHV) and lower heating value (LHV) are 19.743 and 18.549 MJ/kg, respectively (dry ash-free basis). The HHV value is higher than most of the biomass feedstocks included in Table 4.5 and similar to wood sawdust. This relatively high value is connected to the chemical composition, specifically the extractives and lignin. Furthermore, the HHV results from the energy density associated with the C-C chemical bond. The high calorific value of PFB implies that it is suitable for solid biofuel application. The thermochemical behavior of PFB was investigated using thermogravimetric analysis (TGA) to determine the thermal parameters that influence gasification. Results are shown in Fig. 4.6. The high cellulose and hemicellulose contents enhance the thermal degradation of PFB as it decomposes at 200-700 ℃. Three stages of degradation can be observed in Fig. 4.6: in the first stage, moisture removal occurred with 8.98% mass loss; a moderate mass loss (28.72%) was identified between 279 and 367 ℃, at the second stage, accompanied by the release of gases from the volatile and other organic matters. The decomposition of hemicellulose starts at a lower temperature than cellulose. See the agreement with the result provided in Table 4.5.

Lignin is the main constituent of the remaining char. Finally, at 367-535 °C, a high weight loss (42.39%) occurred due to the breakdown of cellulose, hemicellulose, and lignin with gaseous by-products, constituting the third stage of mass loss. The latter stage may be regarded as the active pyrolysis zone where the minor and major reactions take place. The thermal decomposition of PFB further affirms its chemical constituents. This result agrees with the lignocellulose components of biomass [32, 29].

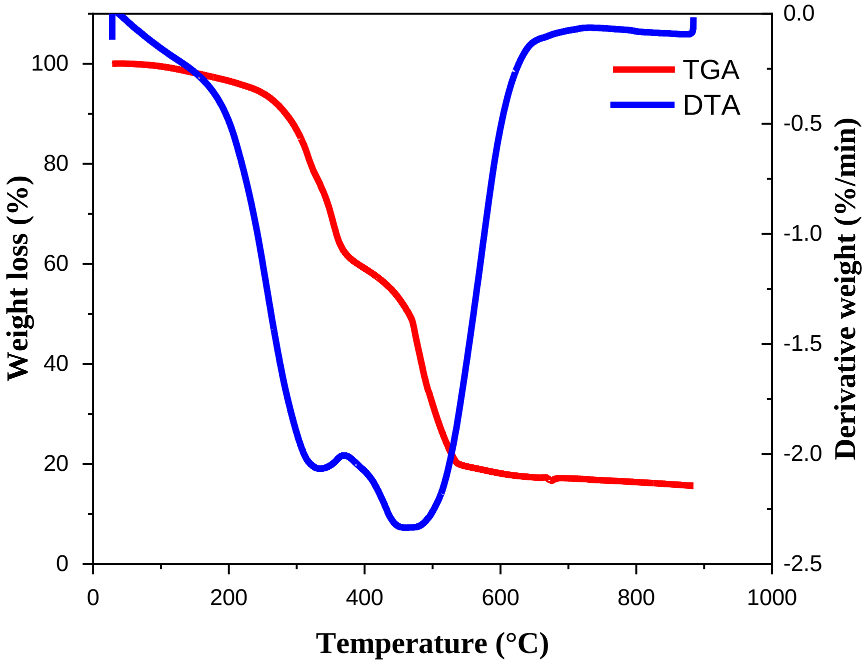

Fig. 4.6: Thermogravimetric analysis of PFB

4.3.9 Suitability of PFB as a biofuel feedstock and other applications

The moisture and ash contents of PFB are less than 10 wt.%, which makes it suitable for direct combustion or pyrolysis. In addition, the ether extractives of negligible value further support combustion. From our findings, PFB can favorably compete with the other documented biomass feedstock such as switchgrass and sawdust. Moreover, the rich calcium content makes it a suitable biomaterial for other applications, such as fillers in particleboard and biocomposite, while the high carbon content can be processed into bio-charcoal and activated carbon.

4.4 Conclusions

In this work, pulverized *Ficus benjamina* fruits (PFB) were extensively characterized for their potential biofuel application. The PFB are amorphous, rich in carbon, have negligible extractives, with low nitrogen and sulphur contents, which makes it eco-friendly as a solid biofuel. Also, the low bulk density and moisture content make PFB cost-effective to process into biofuel. The structural analysis by XRD showed a low crystallinity index value. Furthermore, PFB revealed a high heating value of 19.74 MJ/kg, thus having a high prospect as an alternative to sawdust and wood fuel. However, further research is needed prior to its application in local stoves and boilers, such as optimized pellet densification, analysis of the pellet's combustion properties, and storage.

From the EDS analysis results, the outer portion of the fruit holds all the calcium and most of the potassium present in the whole pulverized *Ficus benjamina* fruits. Considering the ether extractives content (1.48 wt.%) and the volatile matter (64.35 wt.%), these also have potential to generate biogas via decomposition; and should be further explored. Other envisaged applications of *Ficus benjamina* fruits that deserve attention, given their high carbon content and biogenic origin, may include biochar, activated carbon, bio-composite production for soil remediation and environmental sustainability.

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CHAPTER FIVE

Nutritional and chemical composition of *Ficus benjamina* fruit and extracts

5.1 Introduction

Fruits are good food sources. They are rich in organic compounds (such as phytochemicals and antioxidants) and minerals that play a vital role in maintaining a healthy status. Fig fruits from the Family of Moraceae have value for nutrition, health, and other benefits [1]. The fig family has species that serve as food and medicine that makes such plants useful. *Ficus carica* and *Ficus semicordata* have leaves and fruits that have several benefits, including generating income, in different cultural groups [2, 3]. However, the *Ficus benjamina* (FB) fruit has little published information regarding its economic value to the society.

F. benjamina, also referred to as the weeping fig, is a prominent, herbaceous, and perennial multipurpose tree, including beautification [4, 5]. The tree helps to purify the air, removing household air toxins such as formaldehyde and xylene [6]. Some literature reported the ethnobotanical benefits of the various parts of the FB tree with diverse medicinal potentials [7]. A review on Ficus species recounted that FB twigs and leaf juice are good insect repellent [8]. The leaves, bark and fruit have various bioactive constituents like cinnamic acid, lactose, quercetin, caffeic acid, naringenin and stigmasterol [7, 9]. The leaves of FB can be used as vegetables [10]. Phytochemicals, such as flavonoids, gallic acid, chlorogenic acid, rutin, and epicatechins, were reported to be higher in dark FB leaves than in the light-colored varieties [11, 12]. Essential oil from FB leaves was analyzed using gas chromatography (GC) with FID detector and gas chromatography-electron ionization mass spectrometry (GC-EIMS). The oil from the FB leaves at night has dominant active constituents that differ uniquely from the plant during the day, indicating the possible differences in the emissions of volatile compounds from FB plants during the day and at night [13].

FB fruits, small and oval, appear in various colors namely, yellow, red, and purple [14, 15]. FB trees produce fruits two to three times a year [16, 17]. This feature is connected to the parthenocarpic nature of FB (i.e., fruit formation without fertilization), and seeds are absent during fruit formation. However, these fruits have no established economic importance to the society, unlike *F. carica* and *F. semicordata*. FB fruits are not different from other figs, having similar characteristics of edible fruits of the same Genus, especially their aroma, which is a vital attribute of food [18]. Nevertheless, FB fruits are discarded as waste for lack of definite usefulness.

The work of Rahama and Mashi [19] determined the phytochemical and antibacterial properties of air-dried FB fruits. The phytochemical properties identified include saponins, flavonoids, alkaloids, tannins, amongst others. These phytochemical results showed that FB fruits are bioactive. Their report revealed the anti-microbial potentials on *Escherichia coli, Streptococcus pyrogens, Staphylococcus aureus,* and *Pseudomonas aeruginosa* with intense inhibition at higher concentrations.

Some birds and bats feed on FB fruits, as they also find shelter in the large canopy of FB trees, which presents these fruits as potential animal feed [14, 20, 21]. Alternatively, these fruits may contribute vital nutrients to the diet of many animals. Although many parthenocarpic fruits, such as tomatoes, summer squash, and some fruits from the Genus, Ficus (figs), are edible, the edibility of FB fruits is yet to be confirmed. There is no scientific proof that the FB fruit has the potential for food to both animals and humans, and consequently, there is no documented economic benefit of these fruits especially, towards livelihood. Hence, this study focuses on the proximate and nutritional contents of FB fruits. Also, the chemical profile of FB fruit extracts was performed to establish the suitability of FB fruits for food, medicine, and other purposes.

5.2 Materials and Methods

5.2.1 FB fruits Collection and Preparation

FB fruits collected from the African University of Science and Technology (AUST), Abuja, Nigeria, were washed in clean water to remove debris. They were sun-dried, pulverized in a blender (Binatone, China), and stored in clean, air-tight Ziploc bags for further analysis, as shown in Fig. 5.1.

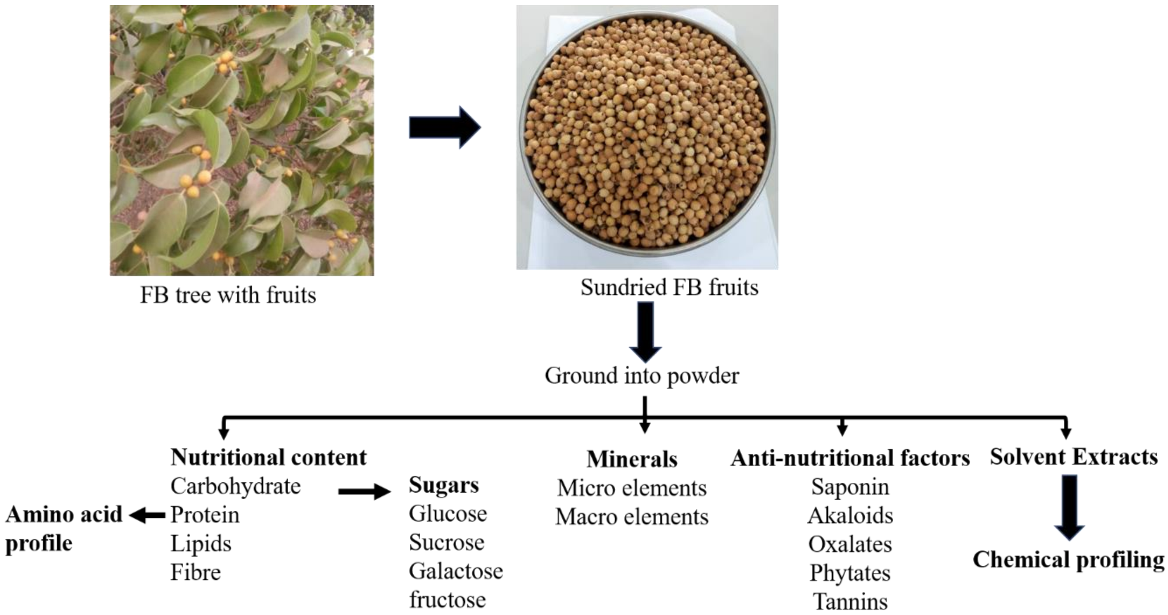

Fig. 5.1: Schematic diagram for assessing the food and chemical potentials in FB fruit.

5.2.2 Proximate and Nutritional Composition of FB Fruits

5.2.2.1 Moisture and ash contents

The moisture and ash contents were determined using the standard outlined by AOAC methods [22]. For the moisture content, crucibles having 2 g of ground sun-dried FB fruit were placed in a drying oven at 105 °C, removed after 4 h, cooled in the desiccator, and weighed. The ash content was evaluated with 2 g of the FB fruit in crucibles placed in a furnace at 900 °C for 7 min, then left to cool in a desiccator. The difference in the weight for both moisture and ash contents was recorded as a percentage, and the study was performed in triplicate.

5.2.2.2 Crude protein

A digestion tube containing a mixture of FB fruit (0.5 g), a Kjeldahl tablet, and 25 mL of concentrated H_2SO_4 was placed on a heating mantle until complete oxidation occurred as indicated by the observed green color. After cooling, the digest was diluted with distilled water to 100 mL. The digest of 10 mL was placed in the distillation apparatus with 25 mL of NaOH solution (40%). The mixture was placed on the heating mantle to evaporate the ammonia collected in a conical flask containing boric acid (4% w/v). The distillation was terminated after about twice the initial volume of boric acid was obtained. Then 25 mL of distillate was titrated against standard hydrochloric solution [0.02 M], and the corresponding titre values were recorded [23]. Following Eq.1, the percentage nitrogen was estimated.

$$\% \, Nitrogen = \frac{14.01 \times molarities \times extraction\ volume \times titre\ value \times 100}{Aliquot \times 1000 \times sample\ weight} \qquad (1)$$

Here, 14.01 = Relative molecular weight; Molarity = The concentration of HCl [0.0183 M]; Extraction volume = the total volume of the digest (100 mL); Titre value = the total volume of acid used on titration; Aliquot = the volume of digest that was distilled (10 mL); Sample weight = the weight in gram of the material used; 100 = percentage conversion factor.

The percentage crude protein was estimated using Eq. 2:

$$Crude\ protein\ (\%) = \% \, Nitrogen \ \times 6.25 \qquad (2)$$

Nitrogen conversion factor = 6.25.

5.2.2.3 Crude fiber

In a conical flask, 2 g of FB fruit was mixed with 200 mL of 0.128 M H_2SO_4 and placed on a hot plate for 30 min with a magnetic stirrer. Subsequently, the acid solution was drained using a cotton cloth. The solid matter collected was washed with hot water to remove acid residue, and further mixed with 200 mL of 0.313 M NaOH solution in a clean conical flask, then placed on the hot plate for 30 min with continuous agitation. The mixture was filtered, and the basic solid matter was washed with hot water. Fiber was collected in a crucible and placed in a hot air oven at 130℃ for 2 h, then cooled in a desiccator. The weight of the dried fiber was recorded and transferred into the furnace at 550℃ for 2 h [24]. The weight of the cooled ash obtained was recorded [25, 26], and crude fiber was estimated using the formula (Eq. 3).

$$\%\ Crude\ fiber = \frac{B-C}{A} \times 100 \tag{3}$$

A = Weight of FB fruit; B = Weight of crucible with fiber; C = weight of crucible with ash.

5.2.2.4 Lipid content

The FB fruits (0.5 g) was placed in a thimble, and thereafter in the sample chamber of the Soxhlet assembly with 250 mL of petroleum ether in the receiver flask. The extraction was allowed for 8 h, then left to cool. The petroleum ether was recovered using a rotary evaporator (Stuart, UK). To ensure that there is no solvent residue in the oil, the oil was placed in the oven for 30 min at 80 ℃. The lipid content was calculated (using Eq. 4), after the flask with the oil was cooled in the desiccator and weighed [27].

.

$$Lipid\ content\ (\%) = \frac{W_2 - W_1}{W} \times 100 \tag{4}$$

Where W_1 = Weight of empty oil flask; = W_2 = Weight of oil flask after extract; W = Weight of FB fruits.

5.2.2.5 Carbohydrate

The carbohydrate content was estimated as described by Adeniji et al. [28] using Eq.5.

$$\% \, Carbohydrate = 100 - (\% \; MC + \% \, Ash \; + \% \, CF + \% \, CP + \% \, LC) \tag{5}$$

Where, MC= moisture content; CF = Crude fiber; CP=Crude protein, and LC =Lipid content

5.2.3 Sugar Determination Using HPLC

The HPLC CECIL CE4200 Adept series with a dual-wavelength UV/Vis detector CECIL CE 4900D was used to identify and quantitatively determine the amount of the free sugars present in the aqueous extract of FB fruits. Fructose, galactose, glucose, and sucrose were detected. The Adept CE 4200 detector covers the range 190 to 700 nm, with an optical bandwidth of approximately 8 nm. A SphereClone ™ 5 µm ODS (2) 80Å with column size 150 mm x 4.6 mm (LC column) was used. The flow rate for acetonitrile: water (80:20) was set at 2.75 mL/min while methanol, as the mobile phase, wavelength of 283 nm for 5 min, was used to estimate lactose. For fructose and glucose assessments, the wavelengths of 250 and 200 nm, respectively for 10 min with acetonitrile: water (80:20) were used.

5.2.4 Amino Acid Profiling

Following the method derived from Edman degradation of proteins and peptides, the amino acid content of FB fruits was determined using the 120 A phenylthiohydantoin (PTH) amino acids analyzer (Applied Biosystems Inc., USA). The analyzer has a 2.1 ID x220 mm cartridge-style column packed with reverse-phase support (PTH-C18). The solvents, 5% aqueous tetrahydrofuran, acetonitrile, and buffers (sodium acetate buffer concentrates with pH 3.8 and pH 4.6) were the mobile phases for the gradient elution of PTH amino acids from the column.

5.2.5 Elemental Analysis by X-ray Fluorescence Spectrometer (XRF)

XRF was used to determine various elemental compositions in FB fruits. ARL QUANT'X EDXRF analyzer (Thermo-fisher Scientific, Switzerland). The tube voltage of 40kV, current of 1.24 mA and a copper film were used. The ground FB fruit (2.0 g) was placed in a sample holder and placed in an XRF Spectrometer under vacuum for 10 min.

5.2.6 Anti-nutritional factors

5.2.6.1 Oxalate

Using the titration method [29], oxalate content was estimated as 2 g of FB fruits was dispersed in 190 mL of distilled water. A volume of 10 mL of 6 M HCl was added, and digested at 100 °C for 1 h, cooled and made to the volume before filtration. The filtrate was precipitated with ammonium hydroxide, and the precipitate was dissolved in 10 mL of 20% sulphuric acid. The solution was titrated with 0.05 M potassium permanganate.

$$Oxalate = \frac{Titre\ value\ KMnO4 \times 0.00225 \times DF \times 105}{ME\ \times MF}$$

Where, DF = Dilution factor; ME= molar equivalent of $KMnO_4$ in oxalate; MF = mass of sample used.

5.3.6.2 Phytate

The Wheeler and Ferrel method was used to determine the phytate content [30]. This method relies on the solubilization of phytate by dilute acid and the subsequent precipitate of phytate as ferric salt. A mass of 4 g of the FB fruit was soaked in 100 mL of 2% HCl for 3 h and was filtered. A mixture containing 25 mL of filtrate and 5 mL of 0.3% of ammonium thiocyanate solution (an indicator) was added to 53.5 mL of water to increase its acidity for titration against iron (III) chloride-solution, containing about (0.00195 g of iron per mL), until a brownish yellow persists for 5 min [31].

$$Phytate\ content\ (g/100\ g) = Titre\ value \times 0.00195$$

5.2.6.3 Tannins

The tannin content in FB fruits was estimated using the method described by **Joslyn** [32]. FB fruit (2 g) mixed with 50 mL of distilled water was heated at 60 ℃. Then the hot filtrate was mixed with 10 mL of 4% copper acetate solution. This mixture was boiled for 10 mins, and the precipitate (residue) collected after filtration. The weight of dried residue was taken before and after incineration in a muffle furnace at 550 ℃, and the difference represents the tannin content.

5.2.6.4 Alkaloids

The alkaloid content was determined according to Habourne [33]. FB fruits (20 g) was soaked in a solvent mixture (10% acetic acid and ethanol) and kept standing for 4 h. The mixture was filtered, and filtrate dried over a steam bath to a volume of about a quarter of its original amount. Then a concentrated NH_4OH was added dropwise until a precipitate is formed, and the crude alkaloid was collected by centrifugation.

$$Alkaloid\ content\ (mg/100\ g)\ = \frac{Weight\ of\ residue}{Weight\ of\ powdered\ FB\ fruits}$$

5.2.6.5 Saponins

Using the AOAC method (2010), 2 g of FB fruits were placed in a Soxhlet extractor with a round bottom flask containing acetone for 3 h [22]. The crude lipid content of samples was extracted by refluxing with 150 mL acetone, followed by 100 mL methanol. The change in weight of the flask was recorded, expressing saponin content in mg/100g.

$$Saponin\ content\ (mg/100\ g)\ = \frac{A - B}{Sm}$$

Where A = mass of flask and extract; B = mass of empty flask; Sm = sample mass

5.2.7 Solvent extraction of FB fruits

The FB fruit was placed in a Soxhlet extractor fitted with a 0.5 L round-bottom flask with a condenser. A known weight of the FB fruit was placed in a filter paper and positioned in the sample chamber of the apparatus. Each extraction was performed for 6 h with 0.5 L n-hexane (nHEX), ethyl acetate (EAC), ethanol (FEE) and distilled water, respectively. The extract using water was preserved in the refrigerator for sugar content determination using HPLC. The chaff obtained was dried, and the content of the chaff was further extracted using ethanol, thus obtaining the ethanol after water extract (EAW). The extracts: n-Hex, EAC, FEE and EAW (Fig. 5.2) were characterized using GC-MS.

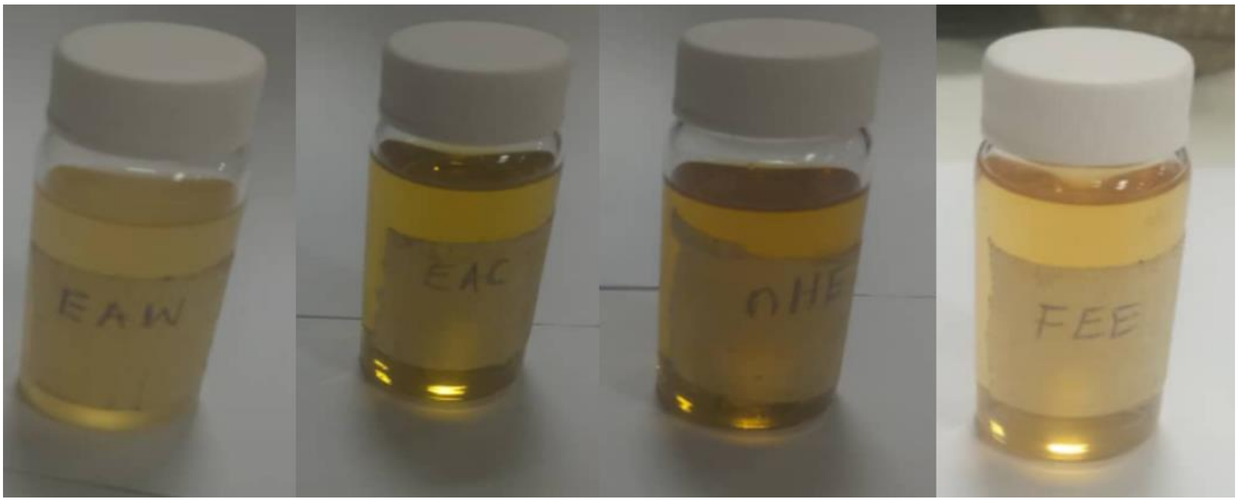

Fig. 5.2: Solvent extracts obtained from FB fruits

5.2.8 GC-MS Characterization of FB extracts

Qualitative characterization analysis of all the possible phytochemicals in the extracts [n-hexane (nHex), ethanol (FEE), ethyl acetate (EAC), and ethanol after water extraction (EAW)] was carried out using Gas Chromatography-Mass Spectrometry (GC-MS) using scan mode. This analysis was performed using a 7820A gas chromatogram coupled to a 5975C inert mass spectrometer (with triple-axis detector) and an electron-impact source (Agilent Technologies). The stationary phase of separation of the compounds was HP-5 capillary column coated with 5% Phenyl Methyl Siloxane (30 m length x 0.32 mm diameter x 0.25 µm film thickness) (Agilent Technologies). The carrier gas was Helium at a constant flow of 1.49 mL/min with an initial nominal pressure of 1.49 psi and an average velocity of 44.22 cm/s. 1 µL of the samples was injected at an injection temperature of 300 °C. Purge flow was 15 mL/min at 0.75 min with a total flow of 16.67 mL/min; gas saver mode was switched on. The oven was initially programmed at 40 °C (1 min) then ramped at 12 °C/min to 300 °C (10 min). Run time was 32.67 min with a 3 min solvent delay. The mass spectrometer was operated in electron-impact ionization mode at 70 eV with an ion source temperature of 230 °C, quadrupole temperature of 150 °C and transfer line temperature of 300 °C. The scanning of possible phytochemical compounds was from *m/z* 45 to 550 amu at 2.00 s/scan rate and was identified by comparing measured mass spectral data with those in *NIST 14 Mass Spectral Library* and literature. Before analysis, the MS was auto tuned to perfluorotributylamine (PFTBA) using already established criteria to check the abundance of *m/z* 69, 219, 502 and other instruments' optimal and sensitivity conditions. These abundances were outputs from the *NIST 14 Library search report* of the constituents of the extracts with each compound identified having a corresponding mass spectrum showing the abundance of the possible numerous *m/z* peaks per compound. The analysis was conducted in replicates as constituent compound name, respective retention time, molecular weight (amu), Quality ion (Q-Ion) and %Total.

$$\%\text{Total} = \frac{Abundance\ of\ individual\ constituents}{Total\ Abundance\ of\ all\ consituents\ in\ extract} \text{ x } 100$$

5.3 Results & Discussion

5.3.1 Proximate and Nutritional Composition

The results in Fig. 5.3 show that the moisture and ash content in FB fruits have low values compared to other fruits of the same Genus. The low moisture content of the sun-dried FB fruits is responsible for both their low density and high stability, amongst other features. In contrast, the ash content of FB fruits was higher than it is in the fruits of the Ficus species in Fig. 3 except for *F. hispidia* and *F. fistula*. The difference in the various species may be attributed to their macro and microelements present, hence, influencing the ash content in the fruit. It is worthy to note that ash content in plant is a function of the elemental composition [34].

The nutritional content (Fig. 5.3) revealed the presence of high carbohydrate content (25.77 %) and crude fiber (25.54 %) for FB fruits, but crude protein and lipid contents are low, having values of 5.94 % and 1.47 %, respectively. Generally, Ficus plants are rich in fiber but low in lipid and protein. The benefit of fiber, protein, and lipid is inestimable in human and animal diets [35]. The nutritional details of FB can account for its possible edibility, as found in other safe to eat Ficus fruits with complementary and healthy compositions. In line with the latter, FB fruit is a good food source for pigeons and other birds in New Guinea. The nutrients in FB fruit were compared to maize, a key element of animal feed. The work of Oladapo et al. [36] identified the nutritional composition of maize: yellow, white and popcorn with the moisture content of 9-11%, crude fat (12.9-14.20%) and crude protein (12.32-13.5%). Also, the nutrient value of cowpea husk (a crop residue) used as animal feed has a moisture content

(19.09%), crude protein 11.21%, while fat, carbohydrate, ash, and fiber are 0.81, 55.06, 11.25 and 22.12 %, respectively [37]. The nutritional contents of FB fruits were discussed and compared with maize (a premier cereal crop in the human population), yet a principal constituent of poultry feeds [38]. This competition for maize as food source is unhealthy because both man and animal depend directly or indirectly on maize grain. Consequently, the competition may grow worse with the rising global population. FB fruit which has similar nutrient values as maize, may serve as possible but partial substitute for animal feed.

The rich fiber in FB fruits reduces the absorption of cholesterol, prevents abdominal discomfort, and lowers the deposit of heavy metals in the colon, thus reducing the risk of health challenges from cholesterol and heavy metals [39]. This attribute of high fiber content is a property that is unique to most edible fruits such *as Ficus carica* and *F. palmate*. Baek et al. [40] used *F. carica* in the form of a paste supplement for the management of abdominal discomfort and constipation. Other nutritional benefits may be due to the presence of lignocellulose (a composition of carbohydrate and fiber). However, during digestion, enzymatic hydrolysis of lignocellulose yields simple sugars.

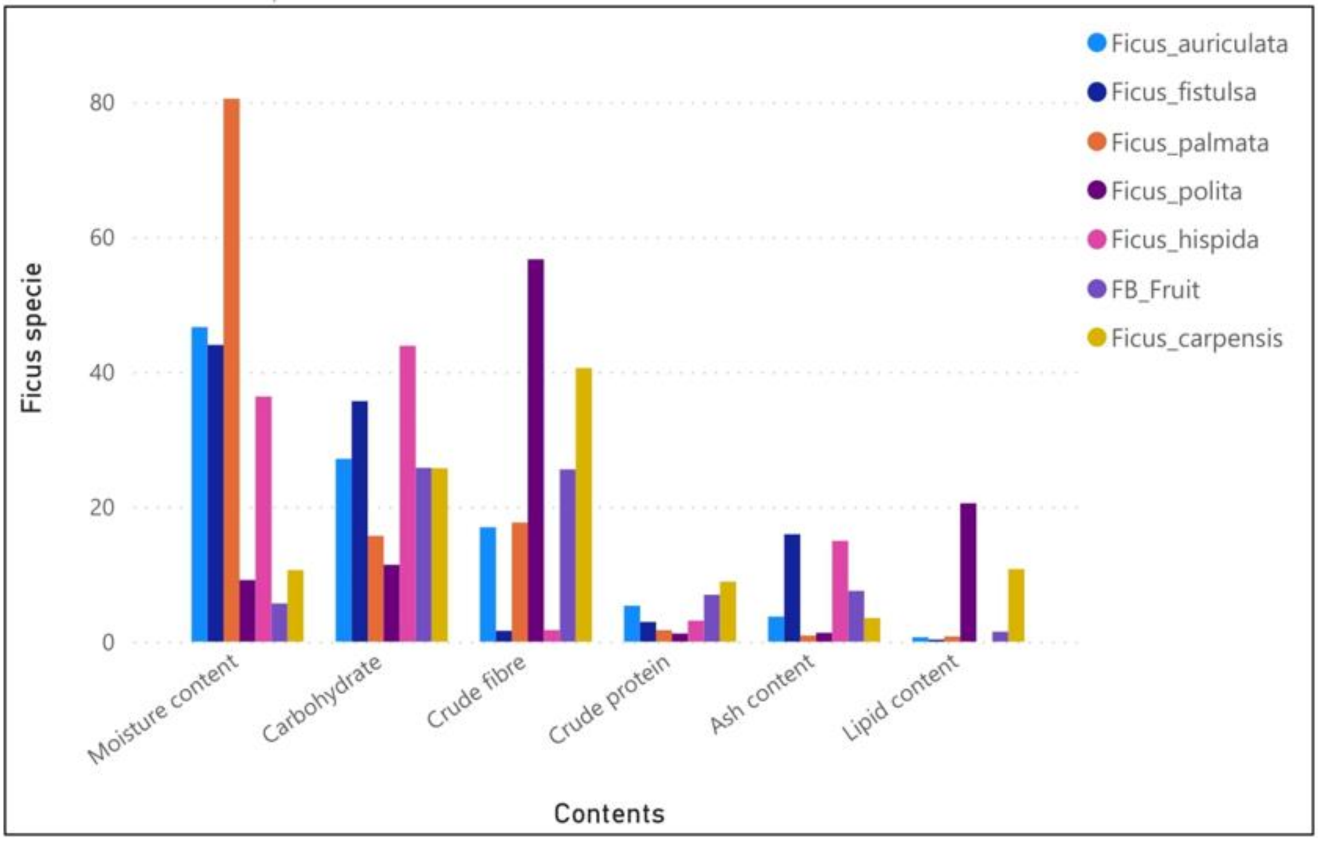

Fig. 5.3: Proximate and nutritional composition of FB fruits compared to fruits from other Ficus species

Sources of data in Fig. 5.3 used for the following Ficus species: *Ficus auriculata* [41]; *Ficus palmata* [42]; *Ficus hispida*[a] [43]; *Ficus fistuls*[a] [43], *Ficus polita*[b] [44]; *Ficus carpensis*[a] [45]: Where: [a]Oven-dried samples; [b] = % dry weight.

5.3.2 Sugar Content Analysis

As a parthenocarpic fruit, the fresh FB fruit suggests a slightly sweet taste due to natural sugars. The aqueous extract from FB fruits revealed high glucose (59.92 mg/g) and galactose (74.18 mg/g) contents compared to sucrose (10.20 mg/g) and fructose (2.20 mg/g). This result is comparable to the simple sugars obtained from maize grain hydrolysis. However, the variation between the latter and Ficus fruits can be attributed to the 850 Ficus species with diverse features. Some Ficus fruits have more sugar than others. Among the various sugars, sucrose (2.39 - 4.5%) was identified in some selected corn hybrids; some hybrids had glucose, xylose and fructose but lack maltose and arabinose [46]. The residues of corn stovers from sweet corn hybrids have a high concentration of soluble sugars such as xylose, arabinose, sucrose, fructose, glucose, mannose, and galactose, as well as the disaccharide, sucrose [47]. These simple sugars can efficiently provide energy roles in animals, besides their conversion into ethanol via fermentation [47]. However, the genetic variation influences both the biomass yield and soluble sugars content.

5.3.3 Amino Acid Content

A total of eighteen (18) amino acids were identified in FB fruits (Fig. 5.4). The presence of both essential amino acids (threonine, valine, leucine, and arginine) and non-essential amino acids (glutamic and aspartic acid, glycine, alanine, and tyrosine) were obvious. The protein profile reveals the concentration of glutamic acid, aspartic acid, glycine, leucine, alanine, and serine are 7.34, 5.71, 4.51, 4.09, 3.10 and 3.00, respectively. These amino acids play key roles in animals and humans, maintaining a healthy status [48]. About 25 free amino acids were identified in asparagus bean seeds and pods, with the total amino acid content in pods higher than those in seeds, but the total percentage of essential amino acids in seeds is higher than in the pods. However, the pods are rich in proline, glycine, glutamine, asparagine, along with the essential amino acids, arginine but methionine is found in pods only [49]. Several metabolic activities require the phosphorylation of serine, threonine, and tyrosine residues in proteins. The latter plays a predominant role in post-translational regulation in eukaryotes [50]. Islary et al. [51] obtained a total of 8 essential and 9 non-essential amino acids from 5 wild fruits: aspartic acid (1.151-3.837 %), glutamic acid (2.283-9.667 %), arginine (0.904-7.187 %), valine (0.142-1.029 %), leucine (1.849-19.665 %), and histidine (0.467-12.986%). Histidine and methionine have antioxidants potential for attenuating the damage of heavy metals on tissues. These amino acids work synergistically with vitamins A and E, thus protecting against lipid peroxidation and cell damage. Valine and leucine enhance functional muscles and bones. Arginine is critical to cell signalling for cellular functions such as hormonal release, cell division, blood clotting and wound healing, while glutamic acid serves as a neurotransmitter for physiological recovery [51].

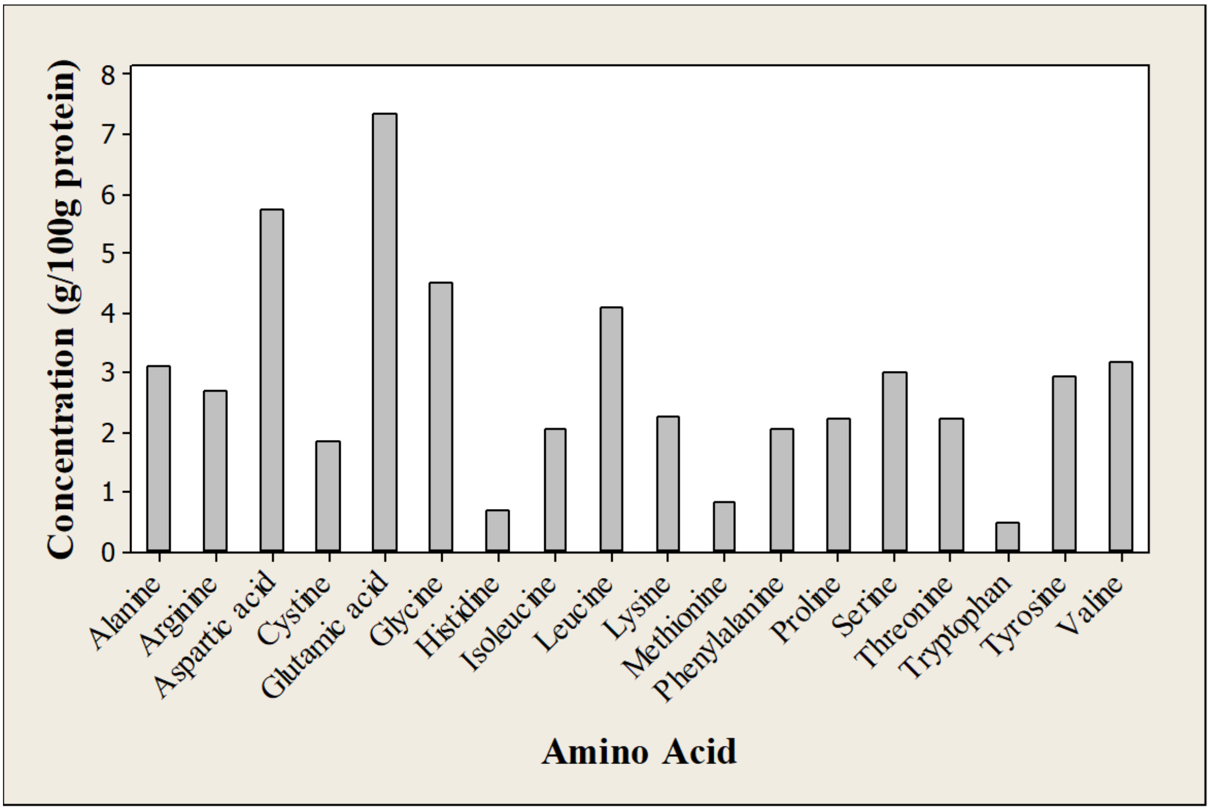

Fig. 5.4: Concentration of amino acids in FB fruits

5.3.4 Elemental analysis (Micro and macro-elements)

In FB fruit, as shown in Fig. 5.5a, there are several macronutrients: Sodium, phosphorus, potassium, calcium, magnesium, chlorine, and sulfur are needed in substantial amount in humans (and animals as well) for diverse metabolic activities. For micronutrients such as iron, copper, manganese, zinc, molybdenum, strontium, and aluminum, they are required in relatively small amounts. In addition, trace minerals like chromium and nickel were identified. The work of Ezealigo et al. [52] confirmed the rich calcium on the outer portion of FB fruits. Minerals affect the metabolic activities, growth, and reproductive wellbeing of animals and humans [53]. Specifically, minerals are involved in enzyme activation in metabolic pathways, catalysis, protein synthesis, osmotic pressure regulation, vascular transport, and cation/anion balancing, hence, they are needed for a robust healthy status. Also, the elements rubidium and strontium were found in FB fruit. The latter elements are a function of high calcium and potassium levels [54]. Qamar et al. [38] identified Na, Ca, Mg, K, and Fe in maize. Similarly, Amadioha et al. (2019) found Ca, P, Zn, Na, Mg, Fe, and K in healthy cowpea husk [37]. These elements are essential for humans, and consequently provide animal with healthy feeds.

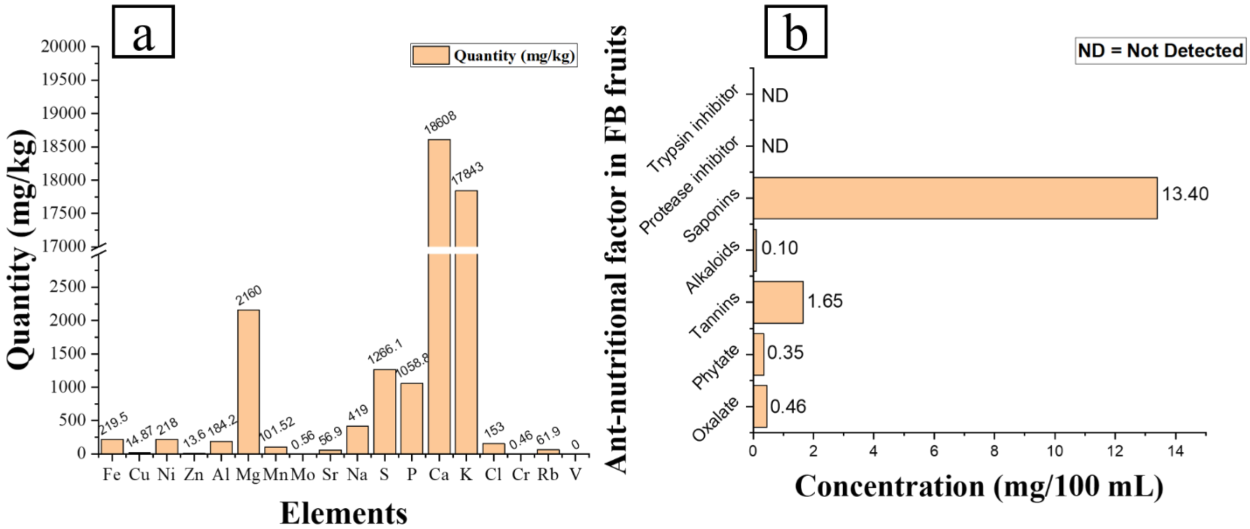

Fig. 5.5: (a) Elemental analysis of FB fruit by XRF; (b) Anti-nutritional content of FB fruits (mg/100 g)

5.3.5 Anti-nutritional factors

The anti-nutritional factors in FB fruits (Fig. 5.5b) are low compared to that in phytate, oxalate and tannin levels obtained by Kubmarawa [55] from *Hibiscus cannabinus* and *Haematostaphis* *barteri*. The values of anti-nutritional factors indicate the edibility of FB fruits. In addition, the low values shows that there will be no privative or interference with other nutrients/elements during absorption in the alimentary canal. Phytic acid chelate mineral elements, especially Ca, Mg, Fe, Zn, and Mo, thereby reducing their availability and absorption. Similarly, phytic acid forms complex products with proteins, thus inhibiting protein digestion and absorption [56]. The presence of high levels of tannins inhibits digestive enzymes. The formation of insoluble oxalate salts is detrimental, as high oxalates formed in the feeds may cause kidney damage and may also produce acute hypocalcaemia in animals. Although saponins are reported as anti-nutrient constituents; and in some cases, toxic due to some adverse effects in fish and cold-blooded animals as they possess hemolytic activity, it is only a few of the broadly classified saponins that are toxic. Contrastingly, saponin is also a natural antioxidant in the colon because it binds to cholesterol to prevent cholesterol oxidation; hence, it has a hypo-cholesterol effect [57]

5.3.6 Solvent extract analysis by GC-MS

The GC–MS analysis of FB fruits identified several compounds that are categorized into alcohols, aldehydes, esters, acids, terpenes, amongst other compounds. As shown in Tables 5.1-5.4, FB fruits solvent extracts showed a significant amount of Beta-Amyrone and 4,4,6a,6b,8a,11,12,14b-Octamethyl-1,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,14,14a,14b-octadecahydro-2H-picen-3-one in all extracts. Ethyl acetate (EAC) and n-Hexane extracts (nHEX) have squalene and share similar compounds (Tables 5.1 and 5.4). Alpha-Amyrin and Griseoviridin were identified in the nHEX extract only. All extracts except EAW have oleic acid and octacosane, while all extracts except FEE have a significant level of n-Hexadecanoic acid among other unique compounds (Table 5.3). FEE extract revealed the presence of 4H-1,2,4-triazole-3,5-diamine. Our findings agree with those found in the literature. Lazreg-Aref et al. [58] identified related compounds found in FB fruits in the n-hexane extract of *Ficus carica*. For *F. carica* extract, thirty-six chemical constituents with about 90.56% of the total peak area were phytochemicals belonging to the class coumarins. The ripe fruits of the wild fig, *F. palmate*, found in Mid-Himalayan region has rich taste, and contains anthocyanin, polyphenol, and Kaempferol as well as phenolics. Similarly, the antioxidant capacity of FB fruits is predicated on these phytochemicals. The latter chemicals have varying degree of influence on the wellbeing of animal health. Hence, fruits with potentials that are nutritionally and medicinally connected are good sources of food [42]. Mousa et al. [59] reported that the fruit extracts of *F. benjamina* L, *F. sycomorous* L, and *F. religiosa* L. exhibit anti-tumour and anti-bacterial activities, but no anti-fungal activity. The strong anti-bacterial activity justifies their use in folk medicine for skin disease [59, 60]. Moreover, these observations may be associated to the presence of high phytochemicals that demonstrates a strong bactericidal effect [61].

Rahama and Mashi [19] observed a remarkable inhibition against Streptococcus pyrogens using the ethanol fraction of FB fruit. In this present study, the chemical profiling of ethanol extract (FEE) provided some information on the inherent antimicrobial properties of FB fruit that may be responsible in the study made by Rahama and Mashi [19]. Table 5 shows diverse uses of the compounds and phytochemicals identified in the four FB fruit extracts, obtained from the GC-MS phytoscan. These identified compounds may play vital clinical roles, hence, relevant in chemical and pharmaceutical industries as some of these chemical constituents has been scientifically reported in medicinal leaves and roots of other plants.

Table 5.1: Ethyl acetate fraction (EAC)

SN	Retention Time (min)	Percentage Total of all compound (% Total)	Compound Name/Hit Name	Quality Ion (Relative Intensity, %)
1	24.917	12.160	.beta.-Amyrone	99
2	27.470	1.603	1,2-Benzisothiazol-3-amine, TBDMS derivative	40
3	23.479	2.582	13-Tetradecen-1-ol acetate	90
4	16.815	1.051	1-Docosene	95
5	27.337	1.286	1H-Indole, 5-methyl-2-phenyl-	25
6	21.377	1.218	1-Nonadecene	95
7	28.446	5.083	2(1H)Naphthalenone, 3,5,6,7,8,8a-hexahydro-4,8a-dimethyl-6-(1-methylethenyl)-	43
8	24.565	1.180	2,3-Nonadecanediol	84
9	25.472	12.733	4,4,6a,6b,8a,11,12,14b-Octamethyl-1,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,14,14a,14b-octadecahydro-2H-picen-3-one	99
10	28.198	1.286	5,7-Octadien-2-one, 3-acetyl-	64
11	26.177	2.007	6-Isopropenyl-4,8a-dimethyl-4a,5,6,7,8,8a-hexahydro-1H-naphthalen-2-one	83
12	26.748	1.173	6-Octadecenoic acid	70
13	17.086	2.626	9,12-Octadecadienoic acid (Z,Z)-	99
14	22.908	3.551	9-Nonadecene	97
15	26.881	1.330	benzenesulfonamide, N-(5-amino-2-hydroxyphenyl)-2,4,5-trichloro-	55
16	23.283	1.003	Cyclooctacosane	66
17	25.818	2.059	Eicosyl benzoate	97
18	23.751	1.065	Fumaric acid, 2,2-dichloroethyl tridecyl ester	35
19	22.521	13.335	Heptacos-1-ene	99
20	29.757	2.912	Hexadecanoic acid, 2-hydroxy-, methyl ester	90
21	15.700	1.756	n-Hexadecanoic acid	99
22	27.846	2.655	N-Methyl-1-adamantaneacetamide	35
23	23.023	2.973	Octacosane	95
24	26.032	4.653	Olean-12-en-3-ol, acetate, (3.beta.)-	92
25	21.285	0.643	Oleic Acid	74
26	23.624	1.149	Oleyl alcohol, trifluoroacetate	91
27	31.686	0.454	Sesquirosefuran	52
28	21.487	0.649	Squalene	81
29	26.581	5.099	Urs-12-en-3-ol, acetate, (3.beta.)-	99
30	30.375	1.090	Z-8-Pentadecen-1-ol acetate	25
	Total	**92.364**		

Table 5.2: Ethanol extract after water (EAW)

SN	Retention Time (min)	Percentage Total of all compound (% Total)	Compound Name/Hit Name	Quality Ion (Relative Intensity, %)
1	24.889	0.939	.beta.-Amyrone	97
2	27.459	3.082	[1,2,4]Triazolo[1,5-a]pyrimidine-6-carboxylic acid, 4,7-dihydro-7-imino-, ethyl ester	43
3	20.297	2.339	[1,2,4]-Triazolo[4,3-a][1,3,5]-triazine, 5,7-diacetylamino-3-methyl-	25
4	23.150	2.058	1-(4-Chlorophenoxy)-1-(1H-imidazol-1-yl)-3,3-dimethylbutan-2-one	41
5	28.186	1.714	1,2,5-Oxadiazol-3-amine, 4-(4-methoxyphenoxy)-	38
6	21.314	6.162	1-Benzazirene-1-carboxylic acid, 2,2,5a-trimethyl-1a-[3-oxo-1-butenyl] perhydro-, methyl ester	47
7	27.828	1.409	1-methyl-4-phenyl-5-thioxo-1,2,4-triazolidin-3-one	45
8	22.700	3.777	2-(Acetoxymethyl)-3-(methoxycarbonyl)biphenylene	35
9	25.651	2.482	2'-Hydroxypropiophenone, TMS derivative	38
10	25.755	3.070	3'-Chlorooxanilic acid N'-(3-ethoxy-4-hydroxybenzylidene)hydrazide	38
11	25.310	2.613	4,4,6a,6b,8a,11,12,14b-Octamethyl-1,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,14,14a,14b-octadecahydro-2H-picen-3-one	95
12	23.289	4.752	9-Octadecenoic acid (Z)-, 2,3-dihydroxypropyl ester	42
13	17.133	6.027	9-Octadecenoic acid, (E)-	99
14	26.870	2.512	Acetic acid, [4-(1,1-dimethylethyl)phenoxy]-, methyl ester	43
15	25.420	3.039	Benz[e]azulene-3,8-dione, 5-[(acetyloxy)methyl]-3a,4,6a,7,9,10,10a,10b-octahydro-3a,10a-dihydroxy-2,10-dimethyl-, (3a.alpha.,6a.alpha.,10.beta.,10a.beta.,10b.beta.)-(+)-	50
16	21.764	1.454	cis-Inositol tri-methylboronate	56
17	16.041	2.260	Cyclopentadecanone, 2-hydroxy-	94
18	24.346	1.389	Fumaric acid, 2-chloropropyl pentadecyl ester	50
19	21.684	1.991	Fumaric acid, 2-chloropropyl tridecyl ester	51
20	24.225	4.004	Heptacos-1-ene	99
21	15.723	1.975	n-Hexadecanoic acid	99
22	22.896	2.764	Nonacos-1-ene	99
23	23.012	1.932	Nonadecane, 1-chloro-	86
24	21.891	1.291	Octadecane	64
25	20.713	2.497	Octadecane, 1-(ethenyloxy)-	58
26	18.091	2.649	Oxacyclotetradecane-2,11-dione, 13-methyl-	95
27	24.588	16.582	Pyrido[2,3-d]pyrimidine, 4-phenyl-	42

Table 5.2: Ethanol extract after water (EAW) Continued

SN	Retention Time (min)	Percentage Total of all compound (% Total)	Compound Name/Hit Name	Quality Ion (Relative Intensity, %)
28	25.969	4.350	Sesquirosefuran	30
29	29.347	1.055	Tris(tert-butyldimethylsilyloxy)arsane	38
30	26.437	2.366	Urs-12-en-3-ol, acetate, (3.beta.)-	80
	Total	**94.534**		

Table 5.3: Ethanol extract (FEE)

SN	Retention Time (min)	Percentage Total of all compound (% Total)	Compound Name/Hit Name	Quality Ion (Relative Intensity, %)
1	24.831	10.476	.beta.-Amyrone	99
2	25.357	2.978	[1,2,4]Triazolo[1,5-a]pyrimidine-6-carboxylic acid, 4,7-dihydro-7-imino-, ethyl ester	46
3	21.741	1.535	1-Docosene	95
4	22.867	2.509	1-Heneicosanol	93
5	23.254	4.106	2-(Acetoxymethyl)-3-(methoxycarbonyl)biphenylene	47
6	25.253	15.163	4,4,6a,6b,8a,11,12,14b-Octamethyl-1,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,14,14a,14b-octadecahydro-2H-picen-3-one	95
7	26.824	6.106	4-Dehydroxy-N-(4,5-methylenedioxy-2-nitrobenzylidene)tyramine	46
8	27.245	5.308	4H-1,2,4-triazole-3,5-diamine, N3-(4-fluorophenyl)-N5-methyl-	38
9	28.585	12.496	Benz[c]acridine, 5,9-dimethyl-	38
10	23.474	6.767	Benzenamine, 4-(2-phenylethenyl)-N-(3,5-dimethyl-1-pyrazolylmethyl)-	70
11	26.061	3.526	Ethanone, 2-(2-benzothiazolylthio)-1-(3,5-dimethylpyrazolyl)-	38
12	23.705	5.166	Fumaric acid, 2-chloropropyl tridecyl ester	41
13	24.184	6.953	Nonacos-1-ene	99
14	22.983	2.519	Octacosane	95
15	15.810	1.593	Octadecanoic acid	95
16	17.213	2.048	Oleic Acid	97
17	26.373	3.153	Urs-12-en-24-oic acid, 3-oxo-, methyl ester, (+)-	97
	Total	**92.402**		

Table 5.4: n-Hexane Extract (nHEX)

S/N	Retention Time (min)	Percentage Total of all compound (% Total)	Compound Name/Hit Name	Quality Ion (Relative Intensity, %)
1	30.289	1.168	.alpha.-Amyrin	64
2	24.906	8.633	.beta.-Amyrone	99
3	26.084	3.879	12-Oleanen-3-yl acetate, (3.alpha.)-	90
4	17.306	1.618	1-Docosene	95
5	24.305	6.772	1-Heptacosanol	94
6	22.307	1.343	1-Nonadecene	90
7	26.246	1.342	2,2,6-Trimethyl-1-(2-methyl-cyclobut-2-enyl)-hepta-4,6-dien-3-one	49
8	26.875	1.272	2-Pyrimidinamine, 4,6-dimethyl-	52
9	25.587	11.765	4,4,6a,6b,8a,11,12,14b-Octamethyl-1,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,14,14a,14b-octadecahydro-2H-picen-3-one	99
10	29.746	1.440	6-Isopropenyl-4,8a-dimethyl-4a,5,6,7,8,8a-hexahydro-1H-naphthalen-2-one	66
11	29.162	1.121	7-Isopropenyl-1,4a-dimethyl-4,4a,5,6,7,8-hexahydro-3H-naphthalen-2-one	92
12	17.075	1.012	9,12-Octadecadienoic acid (Z,Z)-	98
13	24.710	1.167	9,19-Cycloergost-24(28)-en-3-ol, 4,14-dimethyl-, (3.beta.,4.alpha.,5.alpha.)-	45
14	26.742	1.047	9-Octadecenoic acid, (E)-	83
15	4.508	1.608	Cyclohexane, 1-ethyl-4-methyl-, trans-	91
16	4.080	1.342	Cyclopentane, 1-methyl-2-propyl-	58
17	19.812	1.183	Di-n-octyl phthalate	83
18	28.186	4.526	Eicosyl benzoate	64
19	27.586	1.044	Griseoviridin	91
20	21.880	2.166	Hexadecane	95
21	31.068	1.443	Hexadecanoic acid, 2-hydroxy-, methyl ester	44
22	28.879	2.369	Lanosterol	45
23	27.817	2.696	Methyl 2-hydroxydodecanoate	27
24	15.694	1.147	n-Hexadecanoic acid	99
25	24.444	8.299	Nonacos-1-ene	99
26	23.017	2.863	Octacosane	97
27	27.095	0.468	Oleic Acid	52
28	23.630	1.626	Triacontane	90
29	26.621	6.062	Urs-12-en-3-ol, acetate, (3.beta.)-	99
	Total	**82.421**		

Table 5.5: Phytochemical compounds identified in FB fruit extracts and their properties

S/N	Compounds	Class	Properties	Reference
1	4H-1,2,4-triazole-3,5-diamine		Antibacterial and antifungal	**[61]**
2	[1,2,4]Triazolo[1,5-a]pyrimidine-6-carboxylic acid, 4,7-dihydro-7-imino-, ethyl ester	Ester	Antioxidant	**[62]**
3	Griseoviridin	Peptide	Anti-mycobacterial, antibiotics	**[63, 64, 65]**
4	.beta.-Amyrone	Triterpenoids	Antifungal, anti-α-glucosidase, and moderate anti-acetylcholinesterase (AChE) activity	**[66, 67, 68]**
5	n-Hexadecanoic acid	Carboxylic acid	Antioxidant, Hypocholesterolemic	**[69, 70]**
6	Oleic Acid	Fatty acid	Antifungal, anti-inflammatory, antioxidants, antibacterial	**[69, 70]**
7	4,4,6a,6b,8a,11,12,14b-Octamethyl-1,4,4a,5,6,6a,6b,7,8,8a,9,10,11,12,12a,14,14a,14b-octadecahydro-2H-picen-3-one	Ketone	Anti-bacteria, antioxidant	**[69, 70]**
8	Squalene	Triterpenes	chemopreventive and chemotherapeutic agent	**[71]**
9	Alpha Amyrin	Pentacyclic triterpenoid	Anti-inflammatory	**[72]**
10	Octacosane	Alkanes	Mosquitocidal	**[73]**

5.4 Value chain for FB fruits

As shown in Fig. 5.6 and Table 5.5, there are various potentials in FB fruits. There are several ways FB fruits may serve the need of mankind. FB fruits can be processed into animal feed, chemical extracts (such as antioxidants and phytochemicals), activated charcoal for adsorption of impurities, and bioethanol, amongst others. Biochar and activated carbon are also obtainable from FB fruits and could be modified chemically for specific design and purposes (such as water and wastewater treatment, and as a detoxifying agent). All these possible potentials in FB fruit can ensure health safety, food security, and environment preservation. In summary, harnessing the potentials in FB fruit may hold holistic economic benefits to man.

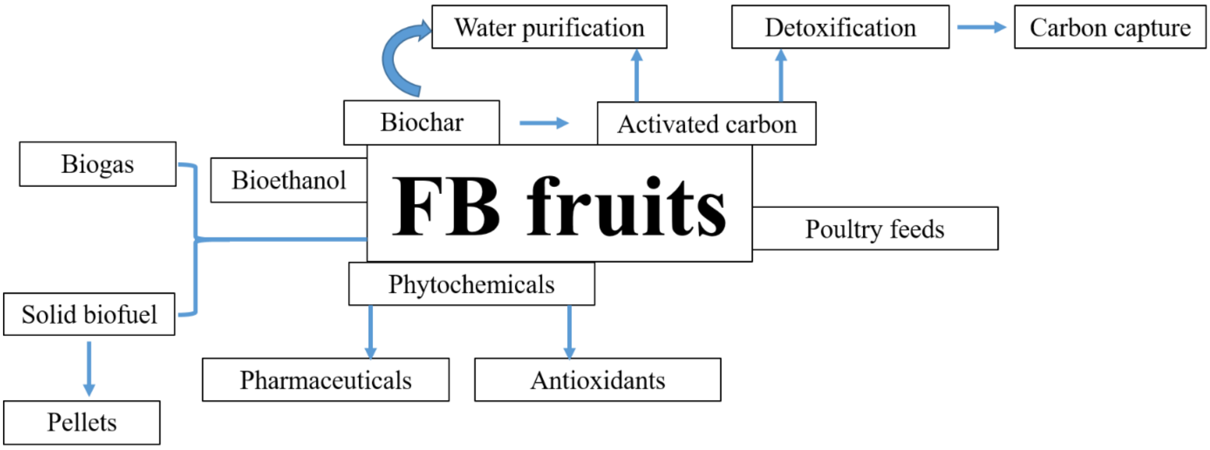

Fig. 5.6: Valorization of FB fruits, showing possible value chain with their products.

5.5 Conclusions

The rise in the global population has led to a commensurate increase in the demand for food, making it necessary to find other food sources or substitute for cereals, especially maize, used for poultry feed. FB fruits, just like other figs, is an excellent fruit with several bioactive compounds. In valorizing FB fruit, the nutritional values and other potential chemicals identified using the GC-MS reveal the possible application in the pharmaceutical and chemical industries.

The edibility of FB fruits defined by the micro and macro elements, carbohydrates, sugar compositions, crude lipid, fiber content, crude protein, amino acids, and anti-nutritional content supports the potential use of FB fruits as a partial substitute for animal feed, depending on the type of animal and feed. Nevertheless, the right combination of the ingredients for animal feed as well as the short- and long-term toxicology studies must be experimentally determined using animal models. Also, further study is needed for the optimization of extractives from FB fruits.

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CHAPTER SIX

Implications, conclusions, challenges, and suggestions for future work

6.1 Implications of Biomass Analysis

Firstly, the assessment of the available biomass residues in Nigeria towards modern bioenergy is necessary. This study is relevant in this era where the global population is increasing and the demand for energy is rising. The need for eco-friendly fuel is indispensable for the protection of the environment and climate from further deterioration. The latter preserves the cultivable land and forests from desertification, undue over-flooding, and irregular temperature of the earth. The assessment helps us identify what we have, and how best it can be processed into valuable products. This research further identifies the farm practices that are necessary to minimize waste with improved technology that offers optimized conversion processes for standard products comparable to fossil fuel. In addition to the transformation of residues, the role of the stakeholders cannot be over-emphasized as it spans from the function of research and development in biomass projects, through the government in policymaking for the good of the community to the regulation of functional industrial system for quality products from residues. The latter will also ensure that such products are available for the masses and at affordable cost, and then to the global market. Moreover, the implementation of the policies that facilitates the proper use of residues can further transform the economy of the country into a circular bioeconomy.

Secondly, it was observed that the quantity of the residue, particularly the technical potential, obtained from estimating the available residues is low. Also, the seasonality of crops is an additional problem, thus making the quantity of residue a challenge for sustainable biofuel production. There is the need to ensure consistent availability of feedstock (that meet the compositional requirements) for biofuel production that serve both households and industries. Beyond the identification of new potential feedstock, their detailed characterization is vital for biofuel purposes. *Ficus benjamina* was identified as one of the biomass wastes that is carbon-rich, but it is yet to be utilized and maximized. Since the propagation and reproduction of FB tree seems easy, there is a possibility that its frequent fruit production may ensure a consistent source of material for bioenergy. The characterization and analysis of FB fruit for bioenergy purpose have enlisted it for consideration for pellets, a safe alternative to the use of wood fuel.

Besides the possibilities of biofuel (for example, pellets), there are other products of value from the waste fruits of FB. This research further analyzed FB fruit for their nutritional and chemical potential for possible use as a substitute for animal feed among other valuable chemicals identified. FB fruits are, therefore, valuable materials for diverse applications rather than being disposed of or burnt.

6.2 Conclusion

In this work, we assessed the bioenergy potential of biomass residues of agricultural origin. This assessment was based on the residues referred to as potential residues (or technical potential) that may not necessarily compete for other use. Furthermore, the research on valorizing *Ficus benjamina* fruit for bioenergy purposes, or as animal feeding substitutes was performed. Although FB fruits cannot entirely be an alternative the conventional animal feed, it may be incorporated into the current feed to manage animal feed economically while maintaining the baseline nutritional standards for animals. Finally, FB fruits are potential biomaterial with sundry value chain for diverse applications.

6.3 Research Challenges and Future work

This research has provided knowledge on the bioenergy potential of biomass residues generated in Nigeria, including the fruits of *Ficus benjamina*, a popular ornamental and landscaping tree. The investigations on the fruits provided insights on various valorization paths towards making products of benefits to both animals and humans. However, further studies on the fruits to be performed are enumerated as follows:

1. In this study, it was shown that the ground FB fruit has high calorific value comparable to saw dust. The making of pellets from FB fruits may serve as an alternative to wood fuel. Hence, there is a need to make pellets from ground FB fruits and then characterize the same for their heat energy qualities. The automated pelletizing machine that records temperature, pressure and hold time for optimized pellet production was not available. Also, the cost for this experiment outside Nigeria was expensive. This limited the experimental procedures towards valorizing FB fruits into pellets.

2. Furthermore, the challenge encountered during this study is the availability of functional equipment needed for detailed characterization of the ground FB fruit. The fruit was then transformed into biochar and activated carbon, yet the equipment required to analyze the latter products made from FB for their possible applications in diverse research fields was another limitation. The proposed use of the biochar and activated carbon was for carbon capture or energy storage (or both), based on their properties. The basic understanding of the structure and function of the biochar and activated carbon will clearly define a specific value for FB fruits, thus creating another valorized product. Although this study was partly carried out, it was limited to activated carbons preparation by chemical activation. The preliminary study and characterization of the biochar and activated carbon was close to completion but the experiments on the physical adsorption of gases (N_2 and CO_2) for carbon capture was a work in progress. While efforts were made to ensure the completion of this project, the doctoral program time limit was another challenge.

3. In addition, this study identified the promising qualities of FB fruit in terms of its nutritional constituents, potential phytochemicals, and the low anti-nutritional factors, thus revealing the possibility of introducing FB fruit as part of animal feed. The limitation in this study is partly due to time that did not afford the opportunity to make animal feeds of various compositional proportions for evaluation. The proposed animal feed will include some portions of FB fruits in a formula to be fed to animals (chicks, in particular). Then, the varieties of feed compositions will further be evaluated for its palatability and nutritional composition. Also, toxicology study using chicks is necessary to verify the safety of animal feed formula made with FB fruits. Finally, this study was limited for the want of fund for the histology, hematology, biochemical, antioxidant, and enzyme assays.

Supplementary Materials

Biomass valorization to bioenergy: Assessment of biomass residues availability and bioenergy potential in Nigeria

Table S1- S5

Table S1: Annual wood production

Year	Round wood total (m^3) x 10^6	Wood fuel (m^3) x 10^6	wood charcoal (m^3) x 10^6
2008	71.8	62.4	3.76
2009	72.2	62.8	3.85
2010	72.6	62.8	3.94
2011	73.0	63.6	4.02
2012	73.4	64.0	4.11
2013	73.8	64.4	4.19
2014	74.9	64.8	4.28
2015	75.3	65.3	4.37
2016	75.6	65.6	4.44
2017	75.9	65.9	4.52
2018	76.2	66.2	4.60

Source: FAOSTAT (2020).

Table S2: Forestry Residue

Year	NO-PRETREATMENT				WITH PRE-TREATMENT			
	Cellulosic ethanol		Biogas (industrial scale)		Cellulosic ethanol		Biogas (industrial scale)	
	ML/yr	Mtoe	[Mm^3 CH_4/year]	Mtoe	ML/yr	Mtoe	[Mm^3 CH_4/year]	Mtoe
2008-2013	75.05	0.041	67.51	0.058	318	0.174	67.51	0.058
2014-2018	78.96	0.043	71.03	0.061	335	0.232	71.03	0.061

Table S3: Annual animal production

Animals	Population (Heads) x10^3 Tonnes										
	2008	2009	2010	2011	2012	2013	2014	2015	2016	2017	2018
Cattle	16.30	16.40	16.60	19.00	19.20	19.40	19.80	20.20	20.60	21.00	21.40
Chicken	174.0	183.0	192.0	149.00	159.00	135.0	137.0	143.00	147.0	143.0	139.0
Goats	53.80	55.10	56.50	67.30	69.00	70.70	72.00	72.50	73.80	78.00	79.30
Pigs	6.910	7.180	7.470	6.280	6.530	6.800	6.990	7.370	7.480	7.500	7.500
Sheep	33.90	34.70	35.50	38.40	39.30	40.30	41.30	41.60	42.10	42.50	43.00
TOTAL	274.0	285.0	297.0	308.0	280.0	293.0	272.0	277.0	285.0	291.0	292.0

Source: FAOSTAT (2020)

Table S4: Municipal solid waste generated and bioenergy potential

Year	Population 10^7	Waste/day 10^7	Waste/yr 10^9	Organic fraction 10^{11}	TS in Organic wasted generated 10^{11}	CH_4 potential Mm^3	Mtoe
2008	2.55	1.51	5.52	3.25	1.41	56545.56	48.82
2009	2.63	1.56	5.69	3.35	1.46	58320.71	50.35
2010	2.72	1.61	5.88	3.46	1.50	60159.28	51.94
2011	2.80	1.66	6.06	3.57	1.55	62064.37	53.58
2012	2.89	1.71	6.26	3.68	1.60	64044.29	55.29
2013	2.98	1.77	6.46	3.80	1.65	66084.90	57.05
2014	3.08	1.83	6.67	3.92	1.70	68197.63	58.88
2015	3.18	1.89	6.89	4.05	1.76	70388.54	60.77
2016	3.28	1.95	7.11	4.18	1.82	72657.90	62.73
2017	3.39	2.01	7.34	4.31	1.88	75011.05	64.76
2018	3.50	2.08	7.59	4.45	1.94	77455.19	66.87

Table S5: Municipal liquid waste generated and bioenergy potential

Year	Population 10^7	MLW generated/day (g) 10^9	MLW generated/yr (ton) 10^6	TS Organic waste generated 10^5	CH_4 potential Mm^3	Mtoe
2008	2.55	6.38	2.33	2.08	70.92	0.061
2009	2.63	6.58	2.40	2.14	73.16	0.063
2010	2.72	6.79	2.48	2.21	75.48	0.065
2011	2.80	7.00	2.56	2.28	77.88	0.067
2012	2.89	7.23	2.64	2.36	80.38	0.069
2013	2.98	7.46	2.72	2.43	82.96	0.072
2014	3.08	7.70	2.81	2.51	85.64	0.074
2015	3.18	7.95	2.90	2.59	88.41	0.076
2016	3.28	8.21	3.00	2.68	91.29	0.079
2017	3.39	8.48	3.09	2.76	94.28	0.081
2018	3.50	8.76	3.20	2.85	97.39	0.084