PREPARATION AND CHARACTERISATION OF RICE AND WHEAT HUSKS COMPOSITE BRIQUETTE USE AS ALTERNATIVE FUEL FOR DOMESTIC COOKING

BY

KAYODE, Busola Isaac
MTech/SPS/2017/6911

DEPARTMENT OF PHYSICS,

SCHOOL OF PHYSICAL SCIENCES

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

NIGERIA.

OCTOBER, 2021

ABSTRACT

In this work a composite briquette formed from agricultural waste (rice and wheat husks) with the manual hydraulic press which was fabricated for this kind of purpose. The briquettes was prepared and characterised as an effort for alternative fuel to the rural and urban households in Nigeria. The samples of rice and wheat husks were grinded and sieved with a sieve mesh. Thereafter the samples were properly mixed with a binder made from gum Arabic and obtained seven different ratios of rice and wheat husks RH:WH (100:0, 90:10, 80:20, 70:30, 60:40, 50:50, 0:100). The ash content, moisture content, bulk density, volatile matter, fixed carbon and calorific values were determined. The experimental results show that the ash content ranged from 8.60 % to 12.01 %, moisture content ranged from 28.01 % to 42.97 %, bulk density ranged from 0.30 g/cm^3 to 0.33 g/cm^3, respectively after briquetting, FTIR spectra of the composite briquettes revealed the participation and presence of –NH_2, -NH, -CO, -COO, -OH, -CH_3, SO_2. The results obtained for the calculated calorific values of the composite briquette revealed that the calculated values are 17.54 MJ/kg and 17.05 MJ/kg which is in accordance with American Standard of Testing Materials (ASTM) and also in accordance with the reported values in the acceptable range of 17 MJ/kg to 21 MJ/kg. It was discovered that the briquette at 60:40 and 90:10 of rice and wheat husks have the highest calorific values and implies that they have more heating advantage and are therefore recommended as an alternative fuel to content with the conventional fuel (charcoal and firewood).

TABLE OF CONTENT

Content	page
Title page	i
Declaration	ii
Certification	iii
Dedication	iv
Acknowledgement	v
Abstract	vi
Table of content	vii
List of table	x
List of figure	x
Abbreviations	xii
CHAPTER ONE	
INTRODUCTION	
1.1 Background of the study	1
1.2 Statement of the problem	5
1.3 Aim and objectives of the research	6
1.4 Justification	7
1.5 Scope	8
CHAPTER TWO	
LITERATURE REVIEW	

2.1 Introduction 9

2.2 Previous research on farm waste briquette 11

2.3 Principle of experimental instrument 16

2.3.1 Hydraulic press machine (manual press) 16

2.3.2 Fourier-Transform Infrared spectroscopy (FTIR) 17

2.3.3 Thermogravimetric Analyzer (TGA) 18

2.3.4 Bomb calorimeter 19

2.4 Thermal properties of biomass 20

2.4.1 Moisture content 20

2.4.2 Ash content 21

2.4.3 Volatile matter 21

2.4.4 Calorific value 21

2.4.5 Bulk density 23

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials 24

3.2 Collection of rice and wheat husks 24

3.2.1 Preparation of composite briquette 25

3.2.2 Determination of composite of fuel briquette 27

3.3 The specific measure for characterizing composite briquette 27

3.3.1 Determination of moisture content 27

3.3.2 Determination of Ash content 28

3.3.3 Determination of calorific value 28

3.3.4 Density measurement 30

3.3.5 Determination of fixed carbon 30

3.3.6 Determination of volatile matter 30

3.3.7 Fourier-transform infrared spectroscopy (FTIR) 31

CHAPTER FOUR

RESULTS AND DISSCUSSION

4.1 Density measurement 33

4.1 Fourier-transform infrared spectroscopy of the samples 38

4.3 Relationship between calorific value, moisture content and ash content 43

CHAPTER FIVE

SUMMARY, CONCLUSION AND RECOMMENDATION

5.1 Summary 46

5.1 Conclusion 46

5.2 Recommendation 47

REFERENCES 48

LIST OF TABLES

Table	Title	Page
2.1	Global emission co_2 saving with different fuel options over wood fuel	10
2.2	Chemical compositions of wheat husk, coconut shell and rice husk	16
2.3	List of the calorific value of a range of biomass materials	23
4.1	Calculated densities for the briquette	35
4.2	Results obtained for moisture content of the samples	35
4.3	Results obtained for ash content of the samples	36
4.4	Calculated results obtained for volatile matter	36
4.5	Calculated results obtained for calorific value	37
4.6	Estimated wave number parameter from FTIR spectra	39
4.7	Estimated wave number parameter from FTIR spectra	40
4.8	FTIR spectra wave number with functional group	42
4.9	Calculated results for fixed carbon of the briquette	42
4.10	Comparison between the calorific value, moisture and ash content	45

LIST OF FIGURES

Figure	Title	Page
1.1	Rice husk	2
1.2	Wheat husk	4
2.1	Manual hydraulic press	17
2.2	Fourier Transform Infrared spectroscopy	18
1.5	Thermogravimetric analyzer	19
3.1	Flow chart for the preparation and characterization of composite briquette	25
4.1	Relationship between transmittance and wave number of the transmitted radiation	39
4.2	Relationship between transmittance and wave number of the transmitted radiation	40

ABBREVIATIONS

ASTM - American Standard of Testing Materials

CO - Carbon monoxide

CO_2 - Carbon dioxide

CV - Calorific Value

C_w - Specific Heat Capacity of Water

DIR - Dispersive Infrared

FC - Fixed Carbon

FTIR - Fourier Transform Infrared Spectroscopy

HHV - High Heating Value

IEA - International Energy Agency

LHV - Low Heating Value

LPG - Liquefied Petroleum Gas

ID - Identification

MT - Metric Ton

AT - Agilent Technologies

NO_2 - Nitrogen IV Oxide

PWH - Peta Watt Hour

SEM - Scanning Electron Microscope

SO_2 - Sulfur IV Oxide

V_1, V_2 - Volume (1,2)

XRD - X-ray Diffraction

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

The availability of petroleum products (fuel) such as diesel, kerosene and liquefied natural gas are sometime scarce and costly in Nigeria. Consequently, charcoal mostly gotten from wood is sometimes the available alternative. However, the production of charcoal is not environmentally friendly and cutting of trees lead to deforestation that often results to soil erosion, flood and global warming. Biomass(agricultural wastes) are found abundantly in many parts of Nigeria which can serve as an alternative source of energy for domestic cooking and small scale industries both in rural and urban areas in Nigeria when properly and adequately prepared and characterised (Garrido *et al.*, 2017).

Biomass has been used as a source of energy all over the world, which provides basic energy requirement for cooking and heating for rural households and in a variety of traditional industries in developing countries. In general, the energy used in such cases is characterized by low efficiency. Agricultural residue could potentially provide a much more extensive energy service than at present if they are used efficiently (Bhattacharya and Abdul- Salam, 2002).

The energy comes from both primary and secondary sources, primary sources are those that have energy production as their sole purpose, widely called 'energy crops'. Such crops include wood and woody plants and sugar beet as a source of bio-diesel. Secondary sources of biomass energy are those where fuel for energy production is a by-product of another process. Examples of secondary sources of biomass energy are sugar cane fiber, rice husk, wheat husk, barley husk, coconut shell, groundnut shell, rice straw, wheat straw and wood waste from horticulture, waste liquors from the pulp and paper industry, and manure from animal farming (Hughes, 2000).Biomass is as a sustainable energy source with carbon(iv)oxide (CO_2) neutralization potential, and only organic petroleum substitute, which is renewable. It also helps the atmospheric (CO_2) recycling and does not contribute to the greenhouse effect (Demirbas, 2010).

Rice is the second most consuming food item worldwide, with annual global demand of approximately 471 million and means per capital consumption around 57 kg per year (FAO, 2012). Accounting for roughly 20% of the bulk grain, rice husk (RH) is the most representative by-product of industrial processing of rice. Formed during grain growth, this protecting coat presents low-density and large volume. Rice husk (RH) layers are of four types: structural, fibrous, sponge-like, and cellule. Main constituents of RH include cellulose (50 %), lignin (30 %), and organic compounds (20 %). The organic fraction contains between 95 % and 98 % on average of amorphous hydrated silica by weight the rice husk has very poor nutritional value and high silica content, which discourages its reuse in the rice production sector (Lim *et al.*, 2012).

Figure 1.1 Rice Husk (source: www.indiamart.com)

Wheat is a major stable food that provides about 20 % of food energy and protein worldwide and also an excellent energy source for farm animal, in 2017 world production of wheat was 772 million tons which making it the second most-produced cereal, 42 % of wheat production was used as feed (FAO, 2012). It is also one of the common cereal grains, which serves as a major health food (source of carbohydrates), animal feed crop and recently small amount are being used as bedding for animal. Wheat husk is lignocellulosic agro waste, which is about 20 % of wheat, a small extent of wheat husk uses as cattle food and as fertilizer (Colin and Wilson, 2004). Cereal waste or agro waste product is an annually renewable fibre and is available in abundant volume throughout the world. The traditional use of these husk and straw includes bedding for animals and livestock feeding. The use of the cereal residues or by-product as a source of alternative energy in the production of renewable energy alleviate the shortage of wood resources and can have the potential to start a natural industry in countries where there are little wood resources left or because of environmental hazard. The composite industries are looking into alternative low cost lignocellulosic sources, which can decrease overall manufacturing costs and increase properties off the materials. Agro husk raw material could be a potential alternative replacing wood for making composites material particularly for domestic and small scale application. In the couple of years, cereal lignocellulosic raw material (straw, cornstalk, bagasse) has been used for making composites with polypropylene, polyethylene, polyester, polyvinyl acetate, polyurethane, poly (3-hydroxybutyrate-co-3-hydroxyvalearate), polylactic acid and Novolac resin (Bledzki *et al.,* 2009). The main factor that restrict the use of the cereal by-product, straw, shell, husk and other agricultural residues in composite have problems associated with the collection, storage, transportation of these materials and economics for the overall production of composites.

The wheat husk contains mainly carbon, oxygen, and small amount of silicon, potassium, sulphur, phosphorous, magnesium and chlorine. Wheat husk and rice husk show a higher proportion of carbon atom as compared to soft wood fibre. On the other hand wheat husk has higher proportion of carbon compared to coconut shell (Andrzej *et al.,* 2010). The higher proportion of carbon in fibre can be attributed to the presence of hydrocarbon rich waxy coating on the cuticle of fibre and lignin present on the surface. Wheat husk contain a little proportion of silicon with compared to soft wood. The tensile strength of wheat husk is higher than soft wood; the impact strength of a composite is influenced by many factors, including the toughness properties of the samples, the nature of interfacial region and frictional work involved in pulling out the fibre from the matrix (Andrzej *et al.,* 2010).

Figure 1.2 Wheat Husk (source: www.indiamart.com)

Rice husk and wheat husk are low cost attractive to current cooking methods, which substitute convectional cooking fuel. By utilizing our abundant agricultural residues, families can reduce the amount of firewood, charcoal and liquefied natural gas burn, which subsequently increases household income and improves household air quality. Rice husk and wheat husk are most low cost and low density materials available in large quantities as solid bye product. Both rice husk and wheat husk exhibit superior physical and mechanical properties that can be utilized more effectively in the development of composite materials for various applications.

1.2 Statement of the Problem

Energy sources for domestic and industrial use in Nigeria is rarely sufficient, the available sources are not cheap, un-accessible, not clean and insufficient for the economic demand with increase in population. To solve these problems, the citizens of Nigeria and other developing countries around the world have resorted to the use of firewood and charcoal for domestic cooking. However, these sources are not efficient and are not environmentally friendly. The fallen of trees for firewood or charcoal making leads to other environmental problems such as desertification, deforestation, erosion and flood. The cause of global warming has been attributed to these primary problems arising from tree falling. The annual rate of deforestation in Nigeria is 3.5 % approximately 350,000-400,000 hectares per year (FAO, 2018).

Furthermore, flood and erosion lead to loss of lives, properties and farmland thereby leading to insufficient food security for the country, breakout of diseases (epidemic) and invaluable economic losses. All these derived problems associated with the use of wood and charcoal for domestic cooking and small cottage industries lead to unquantifiable economic and ecological damages.

Another major problem bedeviling developing countries such as Nigeria is waste disposal and management. Farm by-products also account for a significant part of these wastes. Although most farm wastes are biodegradable, improper disposal can create conducive atmosphere for micro organisms such as fungi, bacteria which could be the pathogen for many diseases.

A good source of energy for either industrial or domestic purpose should be clean, that is environmental friendly, renewable, accessible, cheap and relatively efficient. Although no major source of energy satisfies these characteristics. Consequently, research into energy sources with relatively better characteristics is very active.

The fabrication of farm waste into briquette for energy source will thus reduce the amount of farm waste in the environment and provide alternative to sources of energy which would adequately reduce the environmental problems associated with uncontrolled tree felling for energy source.

The use of briquette from farm waste for energy source requires some preparation and characterisation before they can be considered as good alternatives for the source of energy. Among other properties, such as moisture content, ash content, combustion rate, ignition time, have direct impact on the calorific value and thermal fuel efficiency of the briquette. These properties should reach a threshold level before they can be considered as a good source of energy.

The investigation of several agricultural wastes for their thermal fuel capacity is therefore very important as it will reveal which material is a good biofuel and how their fuel capacity can be improved. Rice husk and wheat husk will be investigated in form of composite briquette to determine their thermal fuel capacity. This automatically will reduce the problem of waste management and energy crisis in Nigeria.

1.3 Aim and Objectives of the Research

The aim of this research work is to prepare and characterize the composite briquettes of rice to wheat husks as an alternative source of energy for domestic cooking.

The objectives are to:

i. prepare composite briquette from rice husk (RH) and wheat husk (WH) in mass ratios of (100:0, 90:10, 80:20,70:30, 60:40, 50:50, 0:100), i.e., RH:WH, respectively.
ii. determine the physical thermal properties(density, calorific value, moisture content, ash content and volatile matter) of the prepared briquette.
iii. determine the functional groups and fixed carbon of the prepared briquettes.

1.4 Justification

In Nigeria, especially in the rural areas charcoal is widely used as alternative source of energy for domestic cooking due to the scarcity and cost of convectional energy source such as diesel, kerosene and liquefied natural gas. In view of the environmental, ecological and economic losses associated with the use of firewood and charcoal as fuel for domestic and industrial purposes, clean, cheap and accessible alternative is therefore necessary. The production of briquette from farm waste with the good calorific value will thus address the problems associated with the use of firewood and charcoal for energy source. Firstly, the use of farm wastes for briquette and subsequently as a source of energy will reduce the problem associated with indiscriminate dumping of refuse of organic origin in Nigerian communities. This implies that the economic losses associated with refuse management and environmental health would be reduced considerably when farm wastes are used as biomass fuel. Secondly, the use of agricultural residue for energy source has a potential to diversify sources of financial income to the farmers. This would also encourage farmers to cultivate more of the crops whose residue can be converted efficiently to biomass fuel, thereby, addressing the problem of food security in the country. Furthermore, the environmental and ecological challenges (such as; desertification, deforestation, erosion and flooding) linked to the use of charcoal for domestic cooking will be reduced to the barest minimum. Consequently, this research when concluded will address the problems of diseases/epidemic, desertification, deforestation, erosion, flooding and the resulting losses to individuals and corporate properties.

1.5 Scope

Determination of heat value of agricultural waste (rice and wheat husks) will be our major concern in order to serve as an alternative energy source for the domestic and small scale industrial cooking with the use of some measure such as moisture content, ash content, volatile matter, calorific value, bulk density, fixed carbon and functional group.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Introduction

All over the world today many researchers are focusing on means of utilizing either industrial or agricultural wastes as a source of alternative fuel for the industry. This waste utilization would not only be economical, but also result in foreign exchange earnings and environmental pollution control.

The increase in population resulted to a significant increase in energy consumption, since the demand of electricity and primary energy has augmented from 71,071 peta watt hour (pwh) in 1973 to 141,304 pwh in 2009, likewise electricity generated boosted up to 227 %, 6,115 pwh in 1973 and 20,055 pwh in 2009 (IEA, 2011). The large scale hydropower system and fossil fuel combustion power system cover this necessity, causing a lot of havoc in environmental, economic, political and social impact worldwide. Despite the advantages that these fossil resources have, such as high energy density, stability during combustion process and relative ease for extraction and transportation, there are some serious effects that could not be neglected. Some of them are energy dependency, health hazards on the users, historical wars and social displacement, global warming caused by greenhouse gases.

As a result of the continuous used of fossil fuels for energy production, carbon(iv)oxide (CO_2) emissions have gone high. In 1971, global emission was about 15,000 MT, in 2009 it was increased to 30,000 MT.As at 2009 coal produced 43% of CO_2emission while oil produced 37 % and gas produced 20 % (IEA, 2012). In 2014 global emission was about 96,280 MT, in 2016 it was decreased to 82,634 MT (IEA, 2019). In view of this, Biomass such as agricultural wastes (Rice husk and wheat husk, coconut shell, rice straw, snail shell, maize stalk, wheat straw, sawdust, waste paper) was introduced in a way of reducing the emission of CO_2.

In respect of this, table 2.1 shows the four different options that have been carried out by the GEMIS (Global Emission Model for integrated System) (UNDP, 2004).

The four options are shown below:

Option 1: 100 % Fuel wood stove

Option 2: Briquette (80 %) – wood system (20 %)

Option 3: 100 % Briquette stove system

Option 4: 100 % Natural gas burner

Table 2.1 Global emission CO_2 saving with different fuel options over wood fuel (UNDP, 2004).

Options	Annual demand $\times 10^6$(kg)	CO_2equivalent $\times 10^3$ ton/annum	Net CO_2 saving over100% wood system,$\times 10^3$ton/annum
Option-1	14.244	17.370	-
Option-2	10.13	4.390	12.98
Option-3	9.039	0.954	16.42
Option-4	2.367	5.312	12.06

(CO_2) equivalent emission results for different options are obtained. Among the four options the highest (CO_2) emission amounting to 17370 tons per year was obtained from option- 1 (100% wood fuel used) and the lowest (CO_2) emission amounting to 954 tons was obtained from option- 3 (100% briquette fuel). The briquette fuel option is the most environmental friendly in the study area among possible other fuel options including natural gas.

Cost of (CO_2) reduction was calculated on the basis of (CO_2) emission data obtained from results analyzed. For calculating the cost of (CO_2) reduction from the replacement of wood fuel with briquette fuel was considered. The lowest cost for (CO_2) reduction means that the briquette fuel option is economically viable than wood option. A sensitivity analysis was done by varying the performance ratio of wood fuel and briquette fuel, then the cost of (CO_2) reduction was found to be ratio of 1.47 and 1.15 respectively (Ahiduzzaman *et al.*,2012).

Concerning the global issues, rice husk briquette fuel can meet the millennium development goal (MDG) to ensure environmental sustainability, improved energy efficiency and use of cleaner alternatives can help to achieve the sustainable use of natural resources and reduce emission to protect the global environment (UNDP project, 2004).

2.2 Previous Research on Farm Waste Briquette

Biomass is commonly used for feed fiber and energy production such as electric power, heat and liquid biodiesel. Among non-hydro renewable resources, biomass is the second most significant source for power production. Wind power existing capacity at the end of 2010 was 198GW followed by 52 GW from biomass (Vassilev *et al.,* 2013). Some of the most attractive characteristics of biomass are huge variety of species with adequate characteristics for producing energy (Vassilev *et al.,* 2013), decentralized location of the resources, existing technologies for direct biomass utilization such as bio-digestors, gasifiers or stove. Moreover, these characteristics generate opportunities for promoting employment creation for technicians and farmer (Gabriele and Luca, 2013). Low density values, non-uniform shapes, sizes and high moisture content associated with raw biomass are some of the most serious problems regarding biomass utilization, especially when solid wastes are used. As such, biomass densification is ideal to curb this problem of moisture retention, as this produces solid biomass from the powdery form. Therefore biomass densification might be applied. This process produces bigger structures from biomass powders. Typically, the products are densified solid in pellets or briquettes with standardize dimension and high mechanical resistance (Tesxeria *et al.,* 2012).

Tanko *et al.,* 2020, studied the characterized composite of agricultural waste (rice husk and coconut shell) as briquette. He determined the calorific value composite briquette of rice husk and coconut shell in different ratios (50:50, 60:40, 70:30, 80:20, 90:10). The experimental result shows that the calorific value of briquette ranged from 14.7715 MJ/kg to 17.8543 MJ/kg. It was observed that composite briquette at ratio 90:10 and 60:40 has the highest calorific value. He also established how particle size can affect the calorific value of the biomass.

Daian (2015) carried out a research on rice husk ash (RHA) base on physical, chemical, thermal, mineralogical and morphologiucal. The characterization of the RHA focus on the use of three distinct methods (grates furnace, fluidized bed and suspension/entrained combustion).

The result confirmed that combustion conditions define the characteristics of each RHA, even though the raw material used was essentially the same.

Research was carried out by Andrzej *et al.,* (2010) on potential of grain by-product (barley husk and coconut shell) as alternative filler for soft wood fibre as reinforcement for thermoplastic. The particle morphology and particle size was investigated by scanning electron microscopy. Chemical composition and surface chemistry of the fibres was also determined. It was found out that barley husk and coconut shell composite are 35% and 20% better than soft wood composite respectively, also confirmed that particle size, shape and fibre surface properties have greater influence.

Nichola and Folorunsho (2012) have established that the briquette formed from rice husk only can be used to fire crucible furnace to melt low temperature melting metals such as Lead, Zinc and Aluminum which probably may be due to the restriction to only one biomass.

Combustible gases were produce from biomass materials such as coconut shell, groundnut shell and rice husk and this was investigated by Sivakumar *et al.,* (2012) under high temperature of 800^0C using gasification technique. For the coconut shell, it was found that the calorific value is 23.01% higher than ground nut shell and 45.45% higher than rice husk.

Muhammad *et al.,* (2013) studied the characteristic composite of rice straw and coconut shell as briquette. Their study showed that rice straws when burns directly produces large amount of smoke and high volatile matter, but reverse is the case for coconut characteristics. Also, carbonization process was used on the biomass which increased the total Sulphur content of the biomass materials, which eventually affect the environment through pollution.

Muthukumar and Lingadurai (2014) developed a polymer matrix composite using coconut shell powder and groundnut shell powder in different volume and evaluate its tensile strength, flexural property and hydraulic behavior. However the plate specimens were characterized using electron microscope, the resulting composite are more environmentally friendly.

Also at Muara- Talang village in Indonesia, a research was carried out by Muhammad *et al.*, (2013) found out that rice straw and coconut shell as solid residues are biomass materials that are not optimally used by farmers and potentially become environmental pollutants. These biomass materials can be used as alternative energy source. Investigation shows that 114 tons of rice straw and 80 tons of coconut shell produced regularly. Most of these residues emit CO, CO_2 and NO_2 as gas when burned which resulted to environmental gas pollutants.

It was also found that, rice straw has 15.61 % of fixed carbon, coconut shell has 78.32 % of fixed carbon and rice straw has fuel value of 1525.5 Cal/g while that of coconut shell has the fuel value of 7283.5 Cal/g.

In order to meet rapidly increasing energy demand, alternative energy sources must be utilized effectively. Of the alternative energy sources, biomass has a great potential today and in the future, since it is renewable, in contrast to the nature of the fossil fuels. Solar energy is indispensable for the plant life and by means of photosynthesis, plants deposit some of the solar energy as a mass in their body (Schiffman and D' Alessio 2003).

When biomass is burnt, this energy is revealed. Biomass has either been burned directly or processed to take advantage of its energy content. In the former case, since biomass usually contains a high content of moisture, it is necessary to remove the moisture content before combustion. Furthermore, the densities of almost all of the biomass samples are very low and therefore some problems occur during their transportation. For these reasons, direct combustion of biomass is not practical. In the latter case, some process such as pyrolysis or gastification had been widely applied to biomass to gain its energy content (Ravidranath and Hall 1995). However, this process led to new problems. Some alternative studies have been conducted to use the biomass as a binder in the production of coal briquettes (Gurubuz-Beker *et al.*, 1998). Moreover, some biomass samples such as cotton stalks, tea waste, waste paper and wheat straw, pine cones, barley husk and sawdust were used to obtain bio-briquettes.

The samples size and shapes are one of the most important factors for composite materials. The effective surface area which may have influence on mechanical properties inversely depends on particle size and shape. Most of the particles are round and regular shape and small amount of particle are rod-type, it was observed that 75-80 % of all types of fibres were distributed in the range of 50-300μm but the distribution frequency is not same for all types of fibres.

Fibres surface layer is relatively minor portion of fibre but it plays an important role in wettability and surface tension. It has long been postulated that the cereal fibres surface contents extractive, lipid and proteinaceous compound and the lipid molecule is usually bonded to the protein molecule by ester orthioester bond (Stevens, 2012). The amount of lipid on the fibre surface has influence on hydrophobicity and on surface tension. The more amount of lipid on the fibre surface means the more hydrophobic and more surface tension as well as smoother the fibre surface forming a thin film. Wheat husk fibre surface is relatively rough and surface is covered by extractive fat and proteinious molecules. It is also observed fibre defects, damage and internal cracks on the fibre surface. Table 2.3 shows the chemical composition of barley husk, coconut shell and rice husk. It can be seen that the cellulose contents in wheat husk is 41.74 %, in coconut shell is 34 % and in rice husk is 50 %. Another structural materialist starch, whose content is 11% in wheat husk and less than 1 % in rice husk, Coconut shell does not contain starch. It is also observed that wheat husk and coconut shell contain 4 % and 2 % of protein and 4 % and 5 % of fat respectively. On the other hand fat and protein content by rice husks are less that 0.5 %. The rest of the compositions of fibres are inorganic, extractive and ash contents.

Table 2.2: Chemical compositions of wheat husk, coconut shell and rice husk(Bledzki, 2010).

Composition(%)	Wheat husk	Coconut shell	Rice husk
Cellulose	36	34	50
Hemicellulose	18	21	20
Lignin	16	27	30
Starch	9	0	0.45
Protein	6	2	0.45
Fat	5	5	0.45

2.3Principle of experimental instrument

2.3.1 Hydraulic press

The hydraulic press machine works on the principle of pascal's law, which states that when pressure is applied to a confined fluid, the pressure change occurs throughout the entire fluid. Within the hydraulic press, there is a piston that works as a pump that provides a modest mechanical force to a small area of the sample. The manual hydraulic press machine consists of four major parts: housing frame, the mould unit, jack and ram and pressure cap. This machine was designed with a strong and compact housing frame which is made up of a 4mm thick angle bar of a total height of 49cm, two angle bar were welded to the top of the frame, two holes were drilled so as to drive a bolt and nut to the frame this bolt and nut will hold the mould to the frame so as to prevent the mould from shaking during the briquette processing. The mould is the part of the press that holds the samples for compaction; it was made up of a pair cylindrical pipe of 120mm with a diameter of 50mm. The pressure cap achieves proper compaction during processing.

Figure 2.1 Manual hydraulic press machine

2.3.2 Fourier-Transform Infrared Spectroscopy

The physical principles underlying infrared spectroscopy have been appreciated for more than a century. As one of the far techniques that can provide information about the chemical bonding in a material, it is particularly use for the nondestructive analysis of solids and thin films, for which there are few alternative methods. Liquids and gases are also commonly studied, more often in conjunction with other techniques. Chemical bonds vary widely in their sensitivity to probing by infrared techniques. For example, carbon-sulfur bonds often give no infrared signal, and so cannot be detected at any concentration, while silicon-oxygen bonds can produce signals intense enough to be detected when probing sub-monolayer quantities, or on the order of 10^{13} bond cc (MEE, 2020).

Figure 2.2 Fourier-Transform infrared spectroscopy (source: MEE 2020)

2.2.3 Thermogravimetric analyzer (TGA)

The principle of TGA analyzer is base on the simple fact that the sample is weighed continuously as it is being heated to elevated temperature and changes in the mass of a sample. Changes in temperature affect the sample. It is used in analysis of volatile products, gaseous products lost during the reaction in thermoplastics, thermosets, composites, films, fibers, coating and paints. In TGA analysis, the sample is heated in a given environment (air, N_2, CO_2, He, Ar) at controlled rate. The change in the weight of the substance is recorded as a function of temperature or time. The temperature is increased at a constant rate for a known initial weight of the substance and the changes in weights are recorded as a function of temperature at different time interval. This plot of weight change against temperature is called TGA curve or thermogram. The wide ranges of application of TGA are composition of multi-component system, thermal stability of materials, oxidation stability of materials, estimated lifetime of a product, decomposition kinetic of materials, the effect of reactive or corrosive atmosphere on materials, moisture and volatile content on materials.

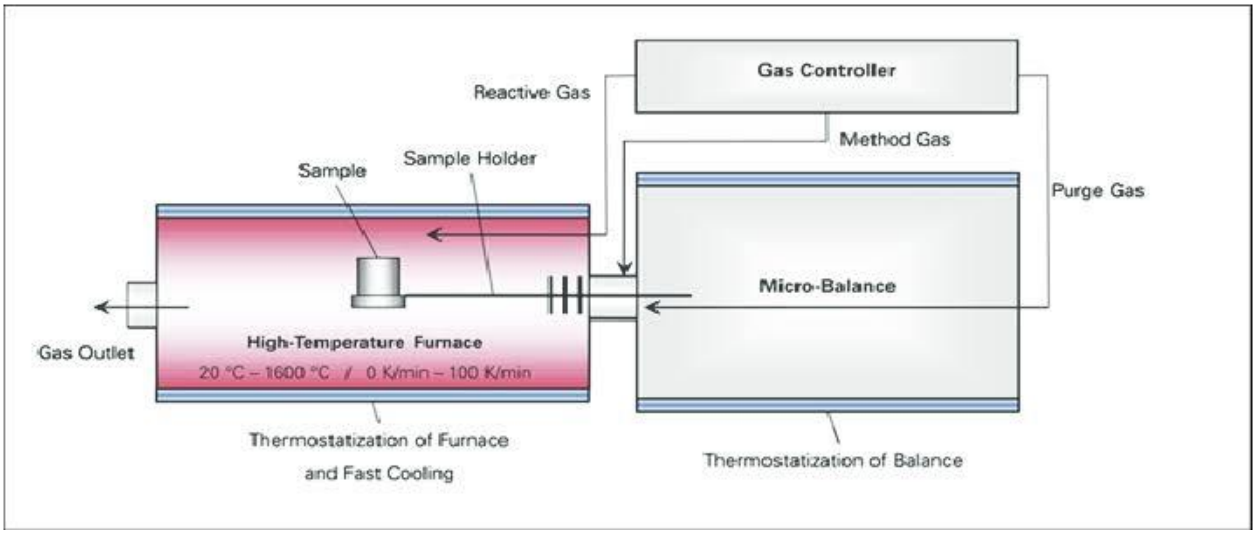

Figure 2.3 Schematic diagram of the TGA- thermogravimetric analyzer(source: MEE, 2020)

2.3.4 Bomb calorimeter

Bomb calorimeter is a machine used to measure the calorific value (high heat value) of a material either in MJ/kg or Cal/g. The calorific value from this machine is the high heat value (HHV), which includes the latent heat of the vapor emitted from the specimen. This machine consists of a small cup to contain the sample, an 1108 oxygen bomb with an oval bucket which fits into the insulting water jacket, a built-in semi-automatic system for charging the bomb with oxygen, high precision electronic thermometer a bright color, touch screen display for data entry and operation control, special communication port for printer, computer and network(LAN) connections, the dewar(to prevent heat flow from the calorimeter to the surroundings), ignition circuit connected to the bomb and a removable compact flash memory card slot for simple program updates and test report archiving.

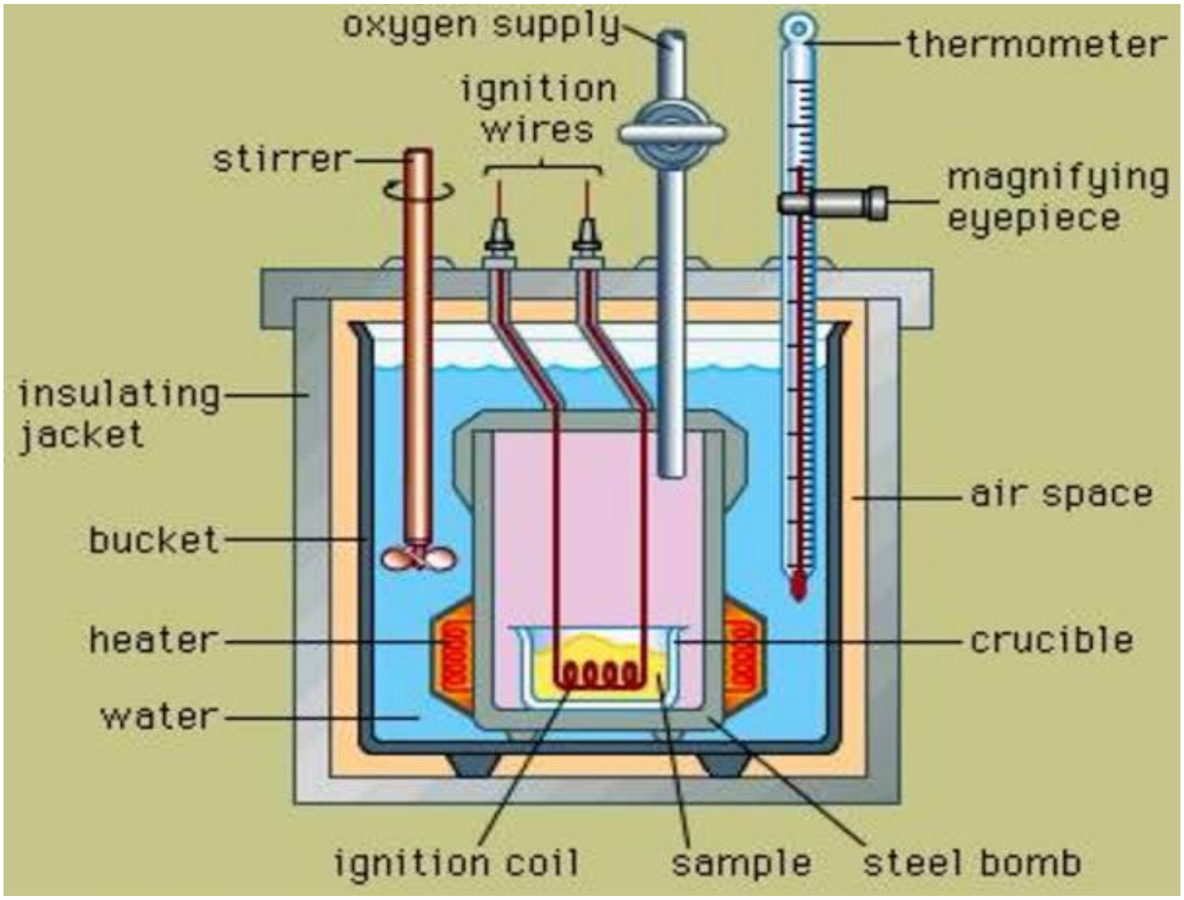

Figure 2.4: Bomb calorimeter (source: www.shutterstock.com)

2.4 Thermal Properties of Biomass

Each type of biomass has its specific properties which determine its performance as a fuel in combustion. Most important properties regarding thermal conversion of fuel is as follows:

2.4.1. Moisture Content

The moisture content of biomass (agricultural waste) is the amount of water in material expressed as a percentage of the material's weight. The weight can be referred to on wet basis. The moisture content has great value on the biomass as fuel base on which the moisture content is measured. The water's weight is expressed as a percentage of the sum of the water, ash, and dry-and –ash-free matter. Similarly, when calculating the moisture content on a dry basis, the water's weight will be expressed as a percentage of the weight of the ash dry and ash free matter. Oven with crucible machine is used for its characterization (Tanko *et al.,* 2020).

2.4.2. Ash Content

It takes the same process with moisture content on wet, dry and ash free basis, it is known as inorganic component. It is generally expressed on dry basis. It is the inorganic matter left out after complete combustion of the biomass, which contain calcium, potassium, magnesium and phosphorus element (Imeh *et al.,* 2011). Hot plate thermometer with crucible was be used for the characterization.

2.4.3. Volatile Matter

Volatile matter refers to the heat that is released when the biomass is heated up to around 400 to 600 °C, during the heating process the biomass converts into volatile gases and solid char. The biomass has a high volatile matter content (up to 80 %) while coal has low volatile matter content (20 %). Volatile matter reckon on the loss of weight in relation with the moisture determined at heating rate, it composed of hydrocarbons and gases such as CO and CO_2. Volatile matter of a fuel is the condensable and non-condensable vapor released when the fuel is heated. Its amount depends on the rate of heating and temperature to which it is heated (Imeh *et al.*, 2017).

2.4.4. Calorific Value

It can be used as a way of identify the amount of heat energy released during the complete combustion of unit mass of biomass. The calorific value is one of the most important characteristics of a fuel, and it is useful for planning and controlling of the combustion plants. The calorific value is measured in a standard test of a bomb calorimeter (expressed in Cal/g or MJ/kg). Bomb calorimeter will be used for the characterization (Mc-kendry, 2002).

. There are two types of calorific value:

1. Higher Heating Value (HHV): it is use to measure the amount of heat released by a complete combustion of a unit of a sample at constant volume in an oxygen atmosphere and at the standard condition (101.3Kpa, 25C). The HHV takes into account the latent heat vaporization of water.

2. Lower Heating Value (LHV): it is use to measures the maximum amount of sensible heat which can be extracted from the combustion of fuel it water in the fuel and water generated during combustion exit as steady in gaseous state. (Mc-kendry, 2002). It is assumed that the latent heat of vaporization of the water cannot be recovered.

The higher calorific value of fuel = $(M_1/M_2) \times (T_c + T_1 - T_2) \times C_w / M_f$ 2.1

Where M_1 and M_2 are the masses of water in copper calorimeter and water equivalent of bomb calorimeter respectively. M_f is mass of fuel sample whose calorific value is to be determined. T_1 and T_2 are the final and initial temperature of water sample. T_c is the temperature correction for radiation losses. C_w is the specific heat capacity of water. When quoting a calorific value, the moisture content needs to be stated as this reduces the available energy from the biomass.

Table 2.3: List of the calorific values of a range of biomass materials (Mckendry, 2002).

Biomass	Moisture%	Volatile matter (VM)%	Fixed carbon (FC)%	Ash content (%)	Lowerheating value (MJ/kg)
Coconut shell	10	50	12	1	18.6
Wheat husk	10	59	21	4	17.3
Barley husk	10	55	18	3	16.1
Lignite	34	29	31	6	26.8
Rice husk	16	40	30	2	16.5

2.4.5 Bulk Density

Bulk density refers to the weight of material per unit volume. The density of the processed product impacts on fuel storage requirement, the sizing of the materials handling system and how the material is likely to behave during subsequent thermo-chemical/biological processing as a fuel/feed stock. Graduated cylinder machine will be used for its characterisation. An important characteristic of biomass material is their bulk density.

$$\rho = m/v \qquad 2.2$$

Where m = weight (mass) of the materials

v = volume (size) of the materials

ρ = bulk density

CHAPTER THREE

3.1 MATERIALS AND METHOD

The following materials were used for the preparation of rice and wheat husks as briquette

i. Rice and Wheat husks (samples)
ii. Binder (Arabic gum)
iii. Bomb calorimeter to determine the calorific value
iv. Calibrated cylinder to determine the density of the materials
v. Hot plate (Gallenkamp, CAT No.7/HL-037) thermometer with crucible to determine the ash content of the sample
vi. Balancing scale(Digital scale AND GF 2000)
vii. Hydraulic press (manual press)
viii. Ethanol and Acetone
ix. Sieve (710µm)

3.2 Collection of rice and wheat husks

Two selected samples from agricultural waste (rice husk and wheat husk) that are locally produced in Nigeria were obtained. Rice husk was obtained from rice processing mill industry at arugungu area in kontagora Niger state and wheat husk was obtained fromwheat depot at new market in kontagora.

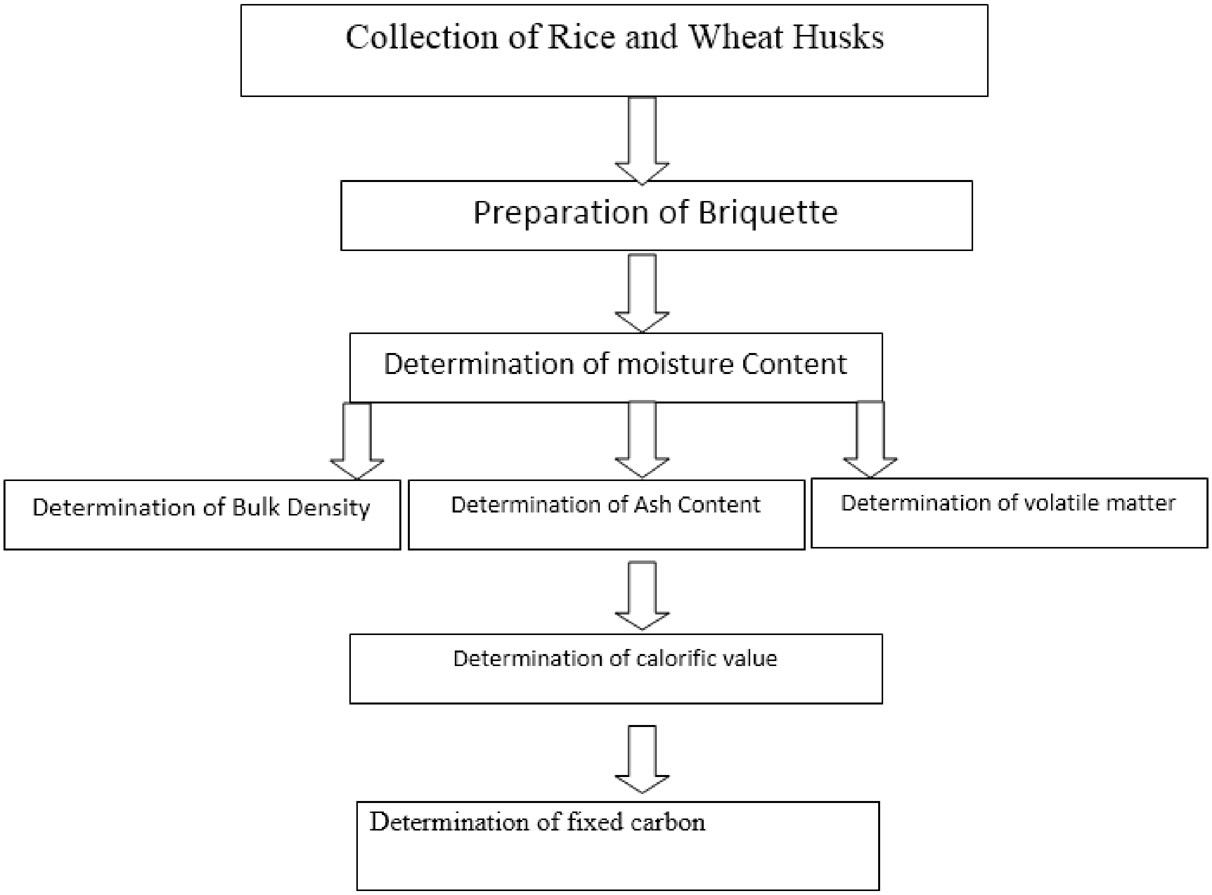

Figure 3.1: Block diagram for the preparation and characterisation of composite briquette**.**

3.2.1. Preparation of Composite briquette

Samples (rice husk and wheat husk) were dried separately in a gravity oven with temperature of about 115 °C for 2 hours. Then the sample was grinded into a powder form by multifunctional food processing machine. Both samples (rice and wheat husks) were sieved under the same condition with the same sieve mesh size of 710 µm in order to obtain uniform sizes, because the calorific value of a biomass depends in its particle size. The smaller the particles size the stronger the density of the briquette and the higher the calorific value. Arabic gum was used as binder for the composite briquette in different ratios. Binder was prepared using 250 ml of warm water; rice husk was thoroughly mixed together with wheat husk after grinded them into a powder form in the ratio RH:WH (100:0,90:10, 80:20, 70:30, 60:40, 50:50, 0:100) and the composite briquette was compacted together to form cylindrical shape by using hydraulic press machine.

The Mettler TG-50 was used to dry about 10 g of the sample; the sample was dried slowly by heating of (115 $^{\circ}$C) and was also held isothermally for 10 minutes depending on the moisture content of the sample. If the thermocurve indicated insufficient drying, the isothermal drying will be repeated.

Thermal gravimetric analysis (TGA) was conducted by thermal gravimetric analyzer (TGA Q500). The sample was placed on small metal plate and on smooth surface by glass needle so that the surface will be perfectly regular and has no holes or lump or areas.

TGA was used to measure change in weight in relation to change in temperature. The measured weight loss curve gives information on change in sample composition, thermal stability and kinetic parameters for chemical reaction in the sample. TGA causing mass change both in physical and chemical changes. The physical change involves gas adsorption, gases desorption and phase transitions (vaporization and sublimation) and chemical change involves decomposition, break down reactions, gas reactions and chemisorptions.TGA are used for characterization of thermal stability, material purity and determination of humidity.

3.2.2 Determination of Composite of fuel briquette

The homogenous and composite of the fuel briquette can be achieved through grinding of the samples into powder. The sample was sieved under the same condition because calorific value of a biomass depends on the particle size (710µm). The samples were properly mixed together with the mass ratio of the sample (100:0, 90:10, 80:20, 70:30, 60:40, 50:50 and 0:100) rice husk(RH) to wheat husk(WH) respectively for the characterization in order to determine which ratio has the heating value (calorific value). The binder was made from Arabic gum to be used as binding agent of the mixture.

3.3 The specific measure for characterizing composite briquette

3.3.1 Determination of Moisture Content

The moisture content analysis was done in the Centre for Genetic Engineering and Biotechnology at Federal University of Technology Minna, Niger state. The sample was determined the water content of the material by heating the sample to a constant mass at a specific temperature. However, a balance or scale sensitive to 0.1% of the mass of the sample having a capacity equal to, greater than, the wet mass of the sample was tested. The specific quantity of the moist sample based on the maximum particle size was weighed and recorded immediately as wet weight of the sample, the quantity of sample was then placed in a crucible of known mass. The sample in a crucible was transferred to thermogravimetric analyzer (TGA) for drying. The drying process of the wet sample to a constant weight was maintained at temperature of 115±5°C, then after some time, the sample was brought out and allowed to cool, and there after the weight of the cooled sample was measured and recorded immediately as dry weight sample. The moisture content was calculated using the following relation (Rbeck, 1999).

$$\% \, moisture = \frac{W_{m-}D_m}{D_m} \times 100 \qquad 3.1$$

Where w_m is mass of water in the sample

D_m is dry mass of the sample

3.3.2 Determination of Ash Content

This process was carried out in the Centre for Genetic Engineering and Biotechnology Federal University of Technology Minna, Niger state. 0.5g of sample was weighed in a ceramic crucible and then placed on an electric muffle furnace at constant temperature (105°C). This process was allowed to take place for three hours. After three hours, the furnace was off and the ceramic crucible containing the ash was also allowed to cool down. The crucible containing the ash was weighed and then, ash content of each of the samples was determined by the difference between the crucible before and after heating. The percentage of Ash content (%AC) was calculated as follow:-

Weight of ash before heating (W_1)/ weight of ash after heating (W_2) × 100

$$\% \text{ Ash Content} = \frac{W1}{W2} \times 100 \qquad 3.2$$

3.3.3 Determination of calorific value

The bomb measurement was based on the oxygen depletion calorimetry which relies on the 1108 oxygen through the system. In the process the mass loss rate is converted into energy. The direct determination of calorific values was made by using bomb calorimeter. The experiment was carried out at centre research laboratory, university of Ilorin, Kwara state, Nigeria.

The determination of calorific value was arranged and selected in the display unit of the bomb calorimeter,0.5 g of the sample was weighed using digital balance, the sample must not more than 1 g for the correct value otherwise the value of the sample might not be accurate during the process. The sample was charged into oxygen bomb vessel in which the combustible charges can be burnt, and the oxygen bomb vessel was filled up with oxygen for the combustion to take place. The connecting tube that supplies the oxygen was then removed and bomb containing the oxygen with the sample was connected with an electrode to provide ignition current to a fuse wire which was then submerged into the bucket containing water for about 2 litres in the calorimeter. The electrode was then connected directly onto the vessel while the bomb and the bucket were held into the calorimeter jacket to serve as a thermal shield. The sample holder with the sample weigh was inserted in the machine, and then the machine was on for the process to take place. The machine was allowed for a time period of 6 to 7 minutes for the combustion to take place, and then the result was displayed in the display unit in cal/g which later converted to MJ/kg by method applied. The procedure was repeated for the whole samples.

The direct determinations of calorific value of the samples are the techniques used to measure the heat flow as a function of temperature with the total area under the curve being proportional to the heat of combustion. A typical thermocurve shows the two-steps decomposition with the first peak relating to the combustion of volatiles and the second to the fixed carbon in the sample. The calorific values of the sample (rice husk and wheat husk) was determined from the percentage of volatile matter and fixed carbon using a thermo-gravimetric technique.

Conversion method from cal/g to MJ/kg: 1cal/g = 0.0041868MJ/kg

3.3.4 Density Measurement

The density test was done in the laboratory Centre for Genetic Engineering and Biotechnology Federal University of Technology Minna Niger state. The actual mass of the sample was measured and recorded with the balancing scale (digital balance). To obtain the actual density (ρ) of the sample, the measured mass of the sample was divided by the volume difference.

The actual mass of the sample was measured and recorded with digital balance. 6cm^3 water was added into a calibrated cylinder with a volume marking recorded as V_1 (initial volume). The fine powder of the sample was then submerged into the water in the cylinder and the new volume was also recorded as V_2(final volume). The difference between the initial volume (V_1) and final volume (V_2) was given the actual density (ρ) of the sample. Density value was estimated using equation (2.2)

3.3.5 Fixed carbon

Fixed carbon is the solid combustible residue that remains after a coal particle is heated and the volatile matter is expelled. The fixed carbon content of the sample was determined by subtracting the percentages of moisture content, ash content and volatile matter. The percentage of fixed carbon (FC) was calculated as follows:-

$$\% FC = 100\,\% - (\% AC + \% MC + \% VM) \qquad (3.3)$$

3.3.6 Volatile matter

Volatile matter was the amount of heat released when the sample of 7.5 g was heated up to 550 °C in crucible with oven dry weight in the furnace for 10 mins. Volatile matter depend on the loss of weight in relation with the moisture content, it composed of hydrocarbons and gases. The percentage of volatile matter was calculated as:

$$\% \text{ VM} = \frac{W1 - W2}{W1} \times 100 \qquad 3.4$$

Where w_1 is weight of the sample before heating

w_2 is weight of the sample after heating

3.3.7Fourier-Transform Infrared Spectroscopy (FTIR)

The Fourier Transform Infrared spectroscopy (FTIR) analysis was carried out at Agilent technology, 288B corporation driver, Dolphin Estate, Ikoyi. Lagos, Nigeria. Fourier Transform Infrared (FTIR) spectroscopy is base on the idea of the interference of radiation between two beams to yield an *interferogram.* The latter is a signal produced as a function of the change of path length between the two beams. The two domains of distance and frequency are inter-convertible by the mathematical method of Fourier- transformation. The radiation emerging from the source passed through interferometer and the sample before reaching the detector. The output signal is amplified in which high-frequency contributions are eliminated by filter; the data are converted to digital form by an analog-to-digital converter and transferred to the computer for Fourier transformation (Barbara, 2004). FTIR spectra were measured using Happ-Genzel and samples were obtained in the range of 4000cm^{-1} to 600cm^{-1} as shown in fig 4.1 and 4.2. The sample holder was cleaned with acetone thoroughly in order to avoid sample contamination and then quantity of the sample was placed on the measurement cell of the spectrometer. The sample device called an interferometer was used to identify samples by producing an optical signal with all the IR frequencies encoded into it. Each spike (absorption band) in the Infrared radiation spectrum represents absorption of energy.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

The experimental results obtained in this project are hereby shown in Table 4.1 to 4.10. The Graduated cylinder, thermogravimetric analyzer, Bomb calorimeter and Hot plate was used to obtained the density, moisture content, calorific value and ash content of the samples (wheat and rice husks) as indicated in the Tables 4.1 to 4.5. The Fourier Transform Infrared(FTIR) spectrometry was done for the data value and the atomic compounds of the samples are shown in the Table 4.6, 4.7 and 4.8.The relationship between the transmittance and wave number of the transmitted radiation are also shown in Figures. 4.1 and 4.2. The comparison between the calorific value, moisture content and ash content of the rice husk with wheat husk samples was presented in Table 4.10.

4.1 Density Measurements

The actual and measured densities of the sample are presented in Table 4.1. The measured density for the briquettes ranged between 0.26 g/cm^3 to 0.33 g/cm^3. Briquette at 60:40 and 90:10 has the density of 0.30 g/cm^3 and 0.32 g/cm^3 respectively. The density of 60:40 has the highest fixed carbon, followed by 90:10 with higher in moisture and ash contents. It appears from the result that composite briquette at density of 60:40 and 90:10 has high mass per unit volume and also has high energy content or heating value (calorific value), these results were similar to the work carried out by Imeh *et al*., (2017). To simplify comparisons of density across different system of units, it is sometimes replaced by the dimensionless quantity relative density or specific gravity which is the ratio of the density of the material to that of a standard material, usually water. Thus, for a relative density less than one means that the substance float in water. The density of the material varies with temperature and pressure. This variation is typically small for solids and liquids but much greater for gases. Increasing the pressure on an object decreases the volume of the object and thus decreases the density. Increasing the temperature of the substance will increase its density by increasing its volume. These accounts for the fact that low density biomass briquette usually have low energy density. However, the low energy density of a rice husk is equally enhanced by mixing it with wheat husk which has a bit had high density than the rice husk.

Table 4.1: Calculated density for the sample composites

S/NO	Sample ID	Sample weight (g)	Sample volume (cm^3)	Density (g/cm^3)
1	Wheat husk	3.00	9.77	0.30
2	Rice husk	3.00	11.72	0.26
3	50:50	3.00	10.79	0.28
4	60:40	3.00	10.68	0.30
5	70:30	3.00	9.46	0.30
6	80:20	3.00	8.96	0.33
7	90:10	3.00	9.29	0.32

Table 4.2: Results obtained for moisture content of the samples

S/NO	Sample ID	Wet weight (g)	Dry weight (g)	Moisture content (%)
1	Wheat husk	0.5	0.36	28.11
2	Rice husk	0.5	0.39	28.01
3	50:50	0.5	0.35	41.81
4	60:40	0.5	0.35	42.15
5	70;30	0.5	0.35	41.40
6	80:20	0.5	0.35	42.85
7	90:10	0.5	0.36	39.10

Table 4.3: Results obtained for Ash content of the sampless

S/NO	Sample ID	Sample weight before heating (g)	Sample weight after heating (g)	Ash content (%)
1	Wheat husk	0.5	0.38	11.63
2	Rice husk	0.5	0.38	12.01
3	50:50	0.5	0.41	8.60
4	60:40	0.5	0.38	12.04
5	70:30	0.5	0.40	10.00
6	80:20	0.5	0.41	8.66
7	90:10	0.5	0.38	11.83

Table 4.4: Calculated results obtained for volatile matter of the samples

Sample ID	Weight before (g)	Weight after (g)	Volatile matter (%)
Wheat husk (100)	7.5	6.73	11.01
Rice husk (100)	7.5	6.66	10.92
50:50	7.5	6.65	11.52
60:40	7.5	6.86	5.53
70:30	7.5	6.49	13.41
80:20	7.5	6.45	13.81
90:10	7.5	6.97	9.52

Table 4.5: Results obtained for calorific value test of the samples

S/NO	Sample code	Mass (g)	Mesh size (μm)	H(Energy in Cal/g)	H(Energy in MJ/kg)
1	RH(100:0)	0.5	710	3944.4	16.51
2	WH(100:0)	0.5	710	4357.3	18.24
3	R:W (90:10)	0.5	710	4071.3	17.05
4	R:W (80:20)	0.5	710	3471.0	14.53
5	R:W (70:30)	0.5	710	3805.8	15.93
6	R:W (60:40)	0.5	710	4190.2	17.54
7	R:W (50:50)	0.5	710	4042.8	16.93

4.2 Fourier Transform Infrared Spectrometry of the Samples

The FTIR spectrometer engages the use of an interferometer, interference of radiation between two beams to yield an interferogram, produced as a function of the change in path-length between the two beams. The output signal is amplified in which high-frequency contributions are eliminated by filter; the data are converted to digital form by an analogue-to-digital converter and transferred to the computer for Fourier-transformation. The wave numbers and mode of vibrations that resulted in the peaks are shown in the Tables 4.6, 4.7 and 4.8, Figure 4.1 and 4.2 for the two samples.

Table 4.6: Estimated wave number parameter from FTIR spectra for composite briquette

Peak number	Wave number (cm^{-1})	Intensity (cm)
1	685.83016	74.76313
2	1017.56323	67.36633
3	1237.47616	85.20252
4	1319.47759	86.29566
5	1371.66032	84.76660
6	1539.39053	82.79035
7	1632.57397	79.45292
8	2922.23286	85.56268
9	3257.69327	78.96579

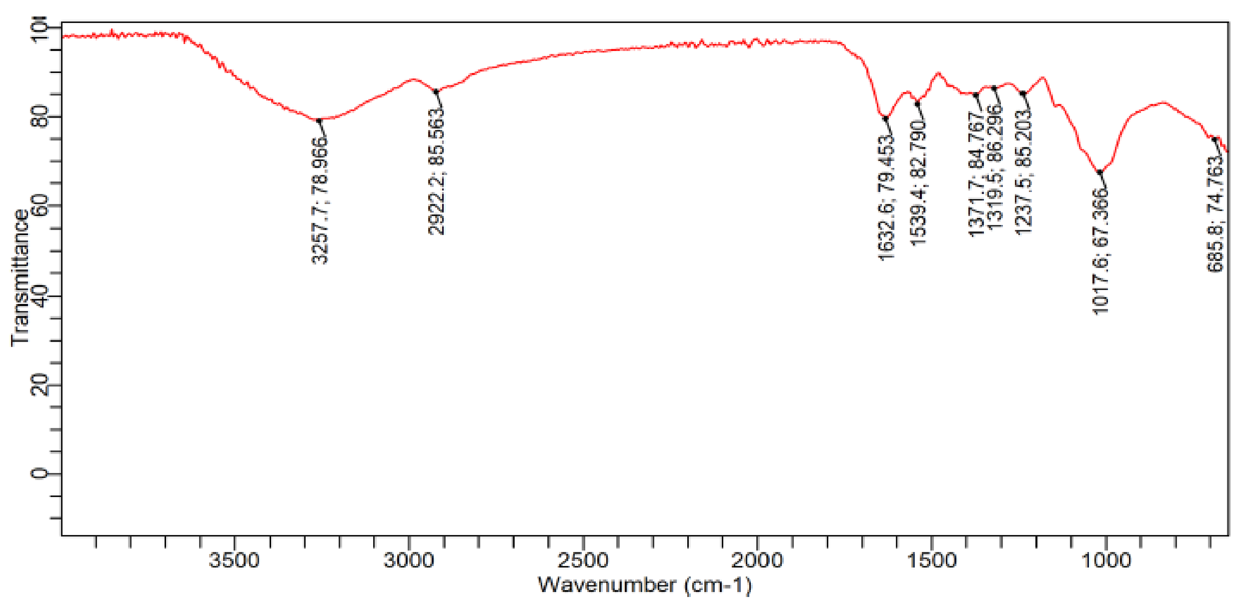

Figure 4.1 Relationship between transmittance and wave number of the transmitted radiation for the composite briquette

Table 4.7: Estimated wave number parameter from FTIR spectra for composite briquette

Peak number	Wave number (cm^{-1})	Intensity (cm)
1	861.01504	80.18406
2	1013.83589	62.62978
3	1237.47616	82.89866
4	1319.47759	84.15317
5	1375.38766	83.29664
6	1539.39053	82.29142
7	1632.57397	77.74172
8	2925.96020	83.55958
9	3253.96593	80.03662

s

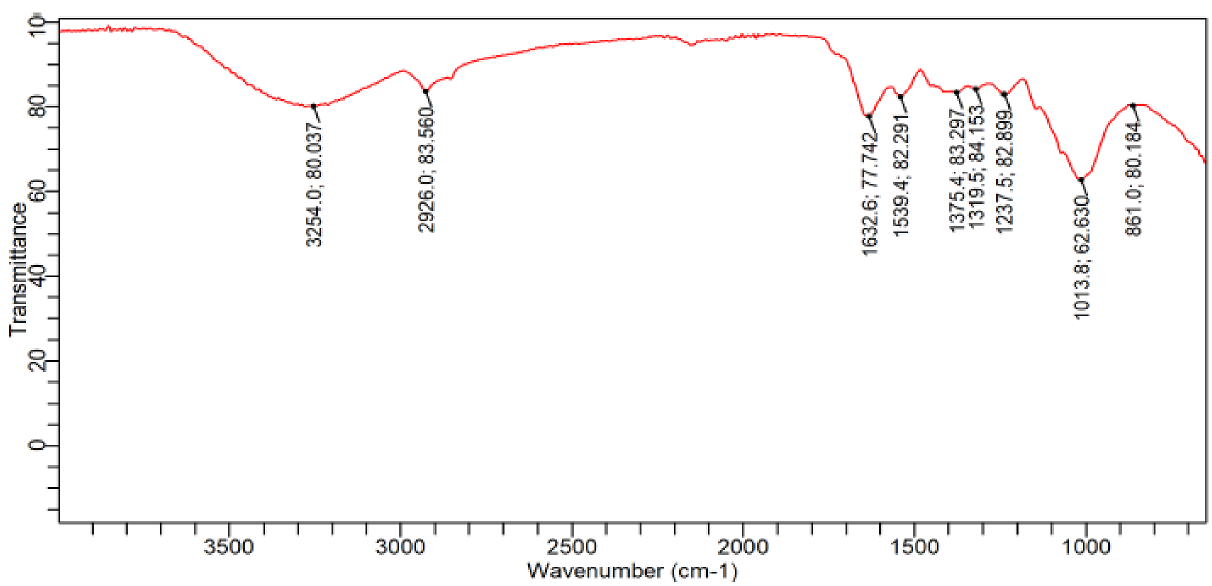

Figure 4.2 Relationship between transmittance and wave number of the transmitted radiation for the composite briquette

The main composition of wheat husk consists of Alcohols, Amides, Carboxylic acid, Alkenes, Alkyl halides, silicon and Ethers. These organic materials consisted functional group of O-H, N-H, C=O. C-F, C-O,=C-O-C and C-I. The classes of compounds contained in rice husk are Alcohols, Alkyl halides, Amides, Alkyls, Alkenes, Alkynes and Alkanes. The main functional groups present are O-H, C-H, C-C, C-O, ≡C-H and C-I (Tanko *et al.*, 2017 & Kumar *et al.*, 2014). The relationship between transmittance and wave number of the transmitted radiation for the composite samples was shown in Figure 4.1 and Figure 4.2. It was observed that the bands 685.8cm^{-1} to 861.0cm^{-1} belong to Si- H stretching vibration (Javed *et al.*, 2011). Bands between 1013.8cm^{-1} and 1017.6cm^{-1} indicated the present of Si- O- Si bending vibration. Bands at 1371.7 cm^{-1} to 1375.4 cm^{-1} indicated the presence of CH_3/COO shifting vibration (JiZang *et al.*, 2019). Band 1237 cm^{-1}, 1319.5 cm^{-1}and 1632.6 cm^{-1}belongs to CO and OH group stretching vibration and also indicated the presence of Alcohols and Carboxylic acid respectively (JiZang *et al.*, 2019). Band at1539.4 cm^{-1} belongs to amino group (presence of amides) (Huihui song *et al.*, 2019). Band 2922.2 cm^{-1} indicated the presence C-H group bending vibration (Sarma *et al.*, 2020). Bands from 3253.9 cm^{-1}to 3257.7 cm^{-1}belong to O-H shifting vibration (Tanko *et al.*, 2020) as indicated in table 4.8.In respect to this explanation it shown that the values gotten from FTIR were correct.

Table 4.8: Wave number (cm^{-1}) with respect to presence of functional group from FTIR spectra for composite briquette

Wave number (cm^{-1})	Functional group	Mode of vibration
3253.96593	Free and intermolecular –OH, Alcohols	OH stretch
2925.96020	CH_2And CH_3In Aliphatic Compound, Alkens,Alkynes	CH antisym and asym stretch
1632.57397	CO and NH2 in primary amides	CO stretch
1375.38766	COO-group in carboxylic acid	Antisym stretch
1237.47616	Hydrated sulphonic acid	SO_3 stretch
1013.83589	Si-O-Si	Si stretch
861. 01504	Si-H in vinyl compounds	CH_2 bending

Table 4.9 Calculated results for fixed carbon of the composite sample

Sample ID	%VM	%AC	%MC	%FC
Wheathusk(100)	11.01	11.63	38.11	49.26
Rice husk (100)	10.92	12.01	28.01	49.08
50:50	11.52	8.60	41.89	38.01
60:40	5.53	12.04	42.15	40.31
70:30	13.41	10.00	41.40	35.23
80:20	13.81	8.66	42.97	34.70
90:10	9.52	11.83	39.10	39.57

4.3 Relationship between calorific value, moisture content and ash content of the samples

The effect of particles size on the measured heating values of biomasses was investigated by Ismail (2012) using different mesh size. The investigation shows that the smaller the size of the particles the stronger the density and the higher the calorific values of the samples.

The results obtained for the calorific value, moisture and ash contents of the samples are presented in Tables 4.4 to 4.6. The high heating value (calorific value) is measured directly with bomb calorimeter. It was found that rice and wheat husks have calorific value of 16.51 MJ/kg to 18.24 MJ/kg, moisture content of 28.01% to 38.11% and ash content of 12.01 % to 11.63% respectively before briquetting. The analysis results show that the calorific value ranges from 14.53 MJ/kg to 17.54 MJ/kg, moisture content ranges from 39.10 % to 42.85 % and ash content ranges from 8.60 % to 12.04 % respectively after briquetting. The result obtained from this analysis and calculated calorific value before and after briquetting of the samples show good agreement with that of the American Standard of Testing Materials (ASTM) of the calorific value range from 17 to 21 MJ/kg(Ismail 2013). It was observed that the composite briquette at 60:40 and 90:10 of rice and wheat husks has the better calorific values when compared with the American Standard of Testing Materials range 17-21MJ/kg for biomass. This shows that 60:40 and 90:10 composite briquettes have high heating advantage and is therefore recommended as an alternative fuel for domestic use to compete with the conventional fuel. Comparison between the calorific value, moisture and ash contents of the rice and wheat husks samples were made as presented in Table 4.7. From the table, the calorific value of the composite briquette formed at 60:40 is greater than the composite briquette formed at 90:10, while moisture content and ash content at 60:40 is greater than that at 90:10. In the way the calorific value obtained from composite briquette at 50:50 is less than that obtained at 90:10 but their moisture and ash contents differs, also the calorific value obtained from composite briquette at 70:30 is less than that at 50:50 while the moisture content and the ash content differs too. This is an evidence that the calorific value of the sample do not dependent on their moisture and ash contents but directly related to their total fixed carbon content, which means the higher the fixed carbon content of plant biomass the higher the energy (Imeh *et al.*, 2017).

Table 4.10: Comparison between the calorific value, moisture and ash contents of the samples

S/NO	Sample ID	Sample weight (g)	Moisture content (%)	Ash content (%)	Calorific value (MJ/kg)
1	Rice husk	0.5	28.01	12.01	16.51
2	Wheat husk	0.5	28.11	11.63	18.24
3	R/W (50:50)	0.5	41.81	8.60	16.93
4	R/W (60:40)	0.5	42.15	12.04	17.54
5	R/W (70:30)	0.5	41.40	10.00	15.93
6	R/W (80:20)	0.5	42.85	8.66	14.53
7	R/W (90:10)	0.5	39.10	11.83	17.05

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Summary

The relationship between calorific value, moisture content and ash content of the composite briquette (rice with wheat husks) in control parameters of constant particle size of 710µm and Arabic gum as binder were investigated. The analysis of the data obtained from the experiment proved that the calorific value of the biomass does not depend on moisture and ash contents but the density of the sample. Likewise it is a proved from experiment above that the density of a biomass is a function of calorific value of the biomass. From the result shown, wheat husk before briquetting and after briquetting formed at 60:40 and 90:10 has higher mass per unit volume (density) 0.3g/cm^{3}and also the higher calorific values. Fourier Transform Infrared spectroscopy (FTIR) was used to determine the main constituent functional groups of rice husk and wheat husk. For the wheat husk, the functional groups were Alcohol, Amides, Carboxylic acid, Alkenes, Alkyl halides and Ethers respectively, while for the rice husk, and their functional groups were Alcohol, Amides, Alkanes Alkyl, Alkenes, Alkyne and Alkyl halide respectively.

5.2 Conclusion

The suggested methodology to analyze the composite briquette (rice husk with wheat husk) was the effort to provide an affordable alternative fuel for domestic use to rural and urban household in Nigeria. It was discovered that rice husk and wheat husk have calorific value of 16.51 MJ/kg and 18.24 MJ/kg respectively, density of 0.26 g/cm^{3}and 0.31 g/cm^3, moisture content of 28.01% and 38.11% and ash content of 12.01 % and 11.63 % respectively before briquetting. The experimental results prove that the calorific values ranged from 17 MJ/kg to 21 MJ/kg are widely acceptable (Ismail 2013). The results obtained from analysis done shows that the composite briquette (R:W) of 60:40 and 90:10 calorific values falls within the acceptable value 17.54 MJ/kg and 17.05 MJ/kg respectively (Tanko *et al*., 2020) FTIR spectra of the composite briquettes revealed the participation and presence of –NH_2, -NH, -CO, -COO, -OH, -CH_3, SO_2(Sarma *et al*., 2014). The biomass sample of rice and wheat husks can be used as alternative fuel for domestic cooking in both rural and urban household in Nigeria.

5.3 Recommendations

The production of briquettes samples composite using wheat husk and rice husk was a more efficient means of utilizing the biomass materials due to the higher heating values generated.

1. There is a need to carry out more characterisation on this composite briquette (biomass) by using different particle size and shape (rectangular, circular, and triangular)as this could enhance their calorific values.
2. Subsequent research or analysis on this composite briquette should focus more on how to reduce the ash and moisture contents of 60:40 and 90:10 in order to get higher calorific values.

REFERENCES

Ahiduzzaman, M., Baqui, M.A. Kamruzzaman, M. & Dasgupta, N. (2012).Prospect of rice husk briquette fuel in Bangladesh.J. *of Agricultural Engineering, IEB, Vol.35/AE. Pp.57-63.*

Andrzej K. Bledzki, Abdullah A. Mamun&Jurgen Volk. (2010).Barley husk and coconut shell reinforced polypropylene composites: The effect of fibre physical, chemical and surface properties.

Avella M, Rota G, Martuscelli E, Raimo M, Sadocco P &ElegirG. Poly(3-hydroxybutyrate-co-3-hydroxyvalerate) and wheat straw fiber composites: thermal, mechanical properties and biodegradation behavior. *J mater sci 2000;35:829-36.*

AyhanDemirbas (2010).Progress and recent trends in bio-diese fuels, *Energy conversion and management" review* 50,2010, pp. 14-34

Bhattacharya, S. C. & Abdul Salam, P. (2002). Low greenhouse gas biomass option for cooking in the developing countries. *Biomass bioenergy 22:305-317*.

BledzkiA.K, MamunA.A&Jaszkiewicz A, Volk J. PLA(2009)*composite reinforced by wheat and rice husk. In: ANTEC@NPE conference proceedings, Chicago, USA.*

Colin W, Harold C, Charles E.W. Encyclopedia of grain science (2004). Missouri: *Elsevier Academic Press.*

DaianeCalheiro, Amanda G. Kieling,& Carlos A. M. Moraes. (2015). Characterization of rice husk ash produced using different biomass combustion techniques for energy.*http://dx/doc.org/10.1016/j.fuel/2015.10.086Publication/Elsevier.*

Food and Agriculture Organization of the unitednations – *FAO (Trade andMarkets Division).Rice Market Monitor, Rome, FAO; 2012. <http://www.fao.org/economic/est/publications/rice-publications/rice-market-monitor- rmm/en/> [accessed 20.12.13].*

Food and Agriculture Organization of the united nations. *Natural forestmanagement. wwwfao.org.Retrieved 15 April 2018*

Gabriele D. & Luca T. J. (2013).Development and Evaluation of a new Advance Solid Bio-fuel and Related Production Processes. International Journal of RenewableEnergy Research, Vol.3 No.2 gabriele.digiacomo@univaq.it, luca.tagileri@univaq.it.

Garrido, M.A.; Conesa, J.A.; Garcia, M.D. Characterization and production of fuel briquettes made from biomass and plastic wastes.*Energies 2017, 10, 850. [CrossRef]*

Gurbuz-Beker U, S. Kuc ¸ukbayrak, A.&Ozer, (1998).Briquetting of Afs ¸in–Elbistan lignite, *Fuel Processing Technology 55 1998 117–127.*

Imeh E. Onukak, Ibrahim A. Mohammed-Dabo, Alewo O. Ameh, Stanley I. R. Okoduwa&Opeoluwa O. Fasanya, (2017). Production and characterization of biomass briquettes from tannery solid waste: *recycling 2017, 2,17; doi:10.3390/recycling2040017.*

Imeh, S.T. and Tembe, E.T. (2011): Combustion properties of Briquettes produced from sawdust of *khayasenegalensis and Gmelinaarborea*pp14-18.

International Energy Agency (IEA) (2011). CO_2emissions from fuel combustion: highlights.*https://www.iea.org/publications/freepublications/publication/CO2emissionfr mfuelcombustionHIGHLIGHTS.pdf.Accessed 21 Mar 2015.*

International Energy Agency (IEA) (2019). CO_2 emission from fuel combustion: *highlights.https://www.macrotrends.net/countries/NGA/Nigeria/carbon-co$_2$-emissions. Pdf Accessed 2019.*

Hughes, E. (2000). Biomass co-firing: Economics, policy and opportunities. *Biomass bioenergy*19:457-465.

Javed, S.H., Shah, F.H., Mansha, M., (2011).Extraction of Amorphous Silica from wheat husk by using $KMNO_4$.*Department of chemical and mechanical Engineerings, University of Engineering and Technology Lahore, Pakistan.Journal of faculty of Engineering & Technology, JFET 18 (2011) 39-46*.

JiZang, Tiantian Wu, Huihuib Song, Nan Zhou, Shisuo Fan, ZhengxinXie& Jun Tang, (2019). College of Resource and environmental, Anhui agricultural university, key laboratory of anhui province, Hefei 230036, China.

Lim J.S, Manan Z. A,Alwi S.R.W,&Hashim H (2012). Review on utilization of biomass from rice industry as a source of renewable energy. *Renew sust Energy Rev 2012:16:3084-94.*

Mckendry, P., (2002). Energy production from biomass (part 1): *overview of biomass. Bioresources Technology*, Vol. 83, pp. 37-46.

MEE (2020). Fourier Transform Infrared spectroscopy (FTIR): Retrieved from the Materials Evaluation and engineering. Inc. www.mee-inc.com/about/us

Muhammad, Y., Faizal, M., &Marsi, N., (2013). Characteristics of composite Rice straw and coconut shell as Biomass Energy Resources (Briquette) Case Study: MuaraTelangVillage, Burryuasin of South Sumatra. *International journal on Advanced Science, Engineering Inormation Technology, Vol. 3 No. 3 ISSN: 2088-5334.*

Muthukumar, S., &Lingadurai, K., (2014). Investigating the mechanical behavior of coconut shell and groundnut reinforced polymer composites. *Global Journal of Engineering Science and Researches, ISSN: 2348-8034.*

Nicholas, A.M. &Folorunsho, O.A., (2012). Utilizing Rice Husk Briquette in firing Crucible furnace for low temperature melting metals in Nigeria.*Engineering technology and Applied Science Research, Vol.2, pp. 265-268.*

Ogwu, I. Y., Tembe, E. T., &Shomkegh, S. A. (2014*).Department of Forest Production and Products and Departmentbof Social and Environment Forestry University of Agricultural Makurdi.*

RavindranathN.H,&HallD.O, (1995). Biomass, Energy, and Environment -A Developing Country Perspective from India, *Oxford Univ. Press, Oxford, 1995, ISBN: 0-19-8564368.*

Rbeck, (1999).California Test 226. Business, Transportation and Housing Agency, pp. 1 – 2.
Richard C. Brundle, Charles A. Evans Jr. & Shaun Wilson (1992).*Encyclopedia of materials characterization.Manning publications co. Greenwich CT 06830 pp. 70-146.*

Sarma, P.J, Kumar, R. &Pakshirajan, K.(2014). Batchand continuous removal of copper and lead from aqueous solution using cheaply available agricultural waste materials.*Department of biotechnology, Indian Institute of Technology Guwahati 781039, assam, India*.

SchiffmanY.M.,&D'AlessioG.J, (2003). Limits to Solar and Biomass Energy Growth, D.C. *Heath and Company, Lexington,, ISBN: 0-669-05253-1.*

Sivakumar, V*;*Asaithambi, M; Sivakumar, P (2012).Physic-chemical and adsorption studies of activated carbon from agricultural waste.*Adv. Appl. Sci. Res 3(1): 219-226.*

Stevens M.P. (2012). Polymer chemistry: *An introduction. Oxford: Oxford university press.*

Stevenson G. G, Pre-Investment Analysis for Biomass Fuels (1996).*CA paper presented at thInternational Workshop on Biomass Fuel Briquetting in Developing ountries, Khartoum.*

TankoJamilu (2020).Preparation and characterization of composite briquette of rice husk and coconut shell, unpublished research thesis department of physics ABU Zaria.

Teixera C. M, Kirsten F. V, & Teixeira P.C.N (2012).Evaluation of moringooleifera seed flour potential biodiesel producer microalgae. *J. Appl. Physical* 24:557-563.

United Nation Development Program me (UNDP) (2004): Energy for Sustainable Development in Asia and Pacific Region: *Challenges and Lessons from UNDP Projects. p.2.*

Vassilev S. V, Baxter D. &vassilav C. G, (2013). An overview of the behavior of biomass during combustion part 1. Phase *–Mineral transformations of organic and inorganic matter. Fuel 112:391-449.*

Wilson HK. Grain crops. 2nd ed. London: McGraw-Hill Book Company; 2004.