

**PRODUCTION OF PARTICLEBOARD FROM RICE HUSK AND LOCUST
BEANS POD USING GUM EXTRACT FROM GUILL AND PERR**
(Cissus populnea)

BY

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ABSTRACT

The quest for man's comfortability has led to increasing degradation of the environmental standard relative to life coexistence. Owing to the facts that the rate of deforestation is alarming and has led to the depletion of the ozone layer, this creates the need for substitute in wood industry. This study focuses on the production of particleboard from rice husk and locust beans pod using gum extract from guill and perr (*Cissus populnea*). Rice husk and locust beans pod pose to have some of the pozzolanic properties similar to that needed in composite formation, this can be a means of transforming the tagged "Agricultural waste" into furniture's, hereby diverting the wood exploitation into its conservation. The binding agent in particleboard processing cannot be neglected, as it plays a vital role in its longevity and cost of production. From the study, the gum was extracted using a Soxhlet extractor at different operating conditions (temperature, extraction time and sample dosage) with an optimum condition of 75 °C with 40 g sample dosage at 90 minutes for fresh sample using Ethyl acetate as extraction solvent. Proximate/elemental analysis of the extract shows a viscosity of 0.29 N. s/m², density of 1.25 g/cm³, 42.8 w/w % of carbon, Ash content of 3.12 w/w %, protein of 5.56 w/w %, Fat content of 0.68 w/w %, fiber content 1.12 w/w % while an FTIR analysis of the extract shows the presence of carboxylic group at wave number of 1680.64 cm⁻¹. From the various particleboards produced, the result from sample tagged J with 1:3 adhesive to rice husk ratio (wt. %) at 70°C within a pressing time of 10 minutes shows the best Modulus of Elasticity (410 N/mm²), Modulus of Rupture (19.04 N/mm²) with an average density of 1013 kg/m³ and Scanning Electron Microscopy (SEM) showing the bonding effects between the adhesive and the aggregate proportion of rice husk used. This was compared with the conventional boards and it shows similar mechanical properties and met the LD-1 requirement of ANSI A208.1 Standards.

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GLOSSARY OF ABBREVIATIONS

FT-IR: Fourier Transform Infra-Red MOE:

Modulus of Elasticity

TRT: Termite Resistance Test MOR:

Modulus of Rupture SBT: Static

Bending Test

SEM: Scanning Electron Microscopy IB

Strength: Internal Bond Strength NAUF: No

Added Urea-Formaldehyde UF: Urea–

Formaldehyde

MUF: Melamine Urea–Formaldehyde

ANSI A208.1: American National Standard for Particleboard ASTM:

American Standard of Testing and Methods

CPA: Composite Panel Association

AOAC: Association of Official Analytical Chemistry NaOH:

Sodium hydroxide

MW: Molecular Weight VI:

Viscosity index

CHAPTER ONE

1.0

INTRODUCTION

1.1 Background to the Study

The desire of mankind to have shelter and furniture requires construction materials; these construction materials can exist in various resources; ranging from stones to timber. To satisfy this craving of humanity, the quest for those resources sets in; this led to the use of substances that has the potential of being transformed into shelter and other furniture materials (Topbaşlı, 2013). Material which has this property that was found and used from time memorial is timber. Initially, stones and mud were used to construct shelter and domestic utensils which had a major problem of high-weight, poor conformability and less self-reliance (Acharya *et al.*, 2011). Considering this Challenge, attention was then channelled to exploitation of timber for furniture making. Due to this change in trend of life patterning furniture process, the demand for wood became high and led to deforestation (Indah *et al.*, 2018). In wood demand reduction the need for recycling of wood scraps during processing was also considered, this led to the formulation of particleboard. In the last few decades, successful development and improvement of wood based composites panels (with the economic benefit of producing low cost wooden materials) has been a major alternative to solid wood usage (Mohanty *et al.*, 2015). The demand for composite wood products of various varieties like particleboard, plywood, hardboard, oriented standard board, medium density fibreboard and veneer board has equally increased significantly throughout the world.

According to Alma *et al.*, (2005), particleboard is one of the most widely used interior wood composite substrates and is commonly used for cabinetry and furniture manufacture. The raw material for chipboard manufacturing can be forest material from paring and pruning, ragged industrial residues (e.g., slabs, leavings of top cutting, and

residual-roll of wood lamination), thin industrial waste (e.g., sawdust and planer shavings), chips from furniture and carpentry industrial processing, or lignocellulose materials such as sugarcane-bagasse, rice husk and other agricultural residues in a pure mode or mixed with wood particles. Composite boards from oil palm frond agricultural residues have the potential to be used as a wood alternative to overcome the material shortage in the wood industry. Filiz *et al.* (2011) determined a method through infrared spectroscopy to evaluate compositions of agro-based particleboards produced from eucalyptus and pine mixed with sugarcane-bagasse, justifying their possible applications in research. Arruda *et al.* (2011) report that over the past decades, many non-wood lignocellulose biomasses, including bamboo, wheat straw, cotton stalks, sunflower stalk, and kenaf stalk, have been used to produce particleboards or other end products.

Since the beginning of the use of wood from ancient time, the value of the forest has expanded drastically, as the human population and their economic grew. Today, wood is used for tools, paper, buildings, bridges, guardrails, railroad ties, poles, furniture, packaging, and thousands of other products (Abdulkareem *et al.*, 2017). The demand for particleboards in the sectors of housing construction, furniture manufacturing and interior decoration (wall and ceiling panelling) has continued to increase (Stefânia *et al.*, 2015). On the other hand, accelerated deforestation and forest degradation, in addition to a growing demand for wood-based boards, have raised an important issue regarding the sustained supply of raw material to the above sectors for a long time. As a result of these concerns, alternative fibers could play an important role in manufacture of composite panels such as particleboard (Avcı *et al.*, 2013). Research has been carried out on a wide variety of non-wood plant fibers and agro residues from many different regions of the world: buckwheat Bekhta *et al.*, (2013) and cotton straws (Alma *et al.*, 2005).

Particleboard is an engineered panel product in which particles of wood are bonded together to form a panel. Most conventionally, they are produced from wood-based materials, which are formed by using a synthetic resin adhesive (Eom *et al.*, 2005). Particleboards are used in furniture making, ceiling boards and partitions in the building industry. More so, in the development of materials based on renewable resources, the search for lignocelluloses substitute for wood is one of the biggest challenges that academia and wood industries are facing. Owing to the facts that deforestation is encroaching the environment and has led to the depletion of the ozone layer (Bauchongkol *et al.*, 2009). Recycling of Agricultural waste material such as rice husk and locust-beans Pod without any complex treatment can withstand in space of time as a good replacement for wood in particleboard processing. More so, its basic properties such as resistance to termite and fire attack make it most effective.

In Modern industrial processes, particleboards are made mainly from selected particles bonded together with a thermosetting resin. Mostly used resins are formaldehyde based, that are usually produced from petrochemical raw materials (Yang *et al.*, 2007). The concept of substituting the fast depleting Petrochemical raw material (formaldehyde) which are known to emit carcinogenic gases with other environmental friendly adhesive is always desired (Mohanty *et al.*, 2015).

The binding agent in particleboard processing cannot be neglected, as it plays a vital role in it longevity and water permeability. The use of synthetic glue and other chemical bond pose a high cost of production on its processing (Jamaludin *et al.*, 2001). In particleboard production, the extraction of gum from guill and perr (*Cissus populnea*) for particleboard synthesis can be attributed to its high availability, low cost of extraction, gum in nature, resistance to weathering and eco-friendly makes it a good substitute for phenol-formaldehyde resin and other conventional gums used. *Cissus populnea* are plants which

belong to the family of Amplidaceae (vitaceae) Ropelike in nature, has medicinal uses and are grown in most part of the world.

This study is to synthesize wood gum from locally sourced raw materials with the views of using it in furniture work and to achieve self and economic reliance by depleting our dependence on imported varieties of foreign gums based on the growing need for gum in our industries.

1.2 Statement of the Research Problem

Over dependency on forest industries has led to the daily increase in the rate of deforestation. This has led to the depletion of the ozone layer (Bauchongkol *et al.*, 2009). Hence a substitute has to be considered to reduce the adverse effects of it consequences. Likewise, the environment pollution as a result of the disposal of some Agricultural bye products/waste (rice husk and locust beans pod) has been of concern and the need to recycle these materials has to be considered. More so, the high cost of particleboard production is due to the synthetic adhesives used.

1.3 Aim and Objectives of the Study

The aim of this research is to produce particle board from rice husk and locust beans pod using gum extract from guill and perr (*Cissus populnea*) as the binder. This was achieved through the following objectives;

1. To extract gum from guill and perr (*Cissus populnea*), studying the effect of process parameters and to optimize the effects of process variables to determine the optimum conditions for producing high yield of gum using design expert.
2. Comparative study on the different extraction yield of the gum obtained from samples (fresh and dried) of guill and perr (*Cissus populnea*).

3. Characterization of the gum from guill and perr (*Cissus populnea*) using Fourier Transform Infrared Spectroscopy (FT-IR).
4. Production and casting of particle board
5. Characterization of Particleboard (MOE, TRT, MOR, SBT, SEM).

1.4 Scope of the Study

This research work is limited to the production of particle board from rice husk and locust beans pod using gum extract from guill and perr.

1.5 Justification of the Study

The speedy increase in wood demand has placed a big conscience in the wood industries towards its procurement and application. As day-to-day use of wood increases, with little or no recycling measure put in place, has led to deforestation and in turn degradation, thereby exposing the earth surface to solar radiation. In search of alternative to salvage this adverse effect on the human race, recycling of locally sourced material such as rice husk and locust-beans pod without any complex treatment can withstand the test of time as a good replacement for wood in particleboard processing.

The use of rice husk and locust-beans pod which is highly available, eco-friendly and resistance to termite, can be a medium of transforming the tagged; Agricultural waste into furniture, hereby diverting the wood exploitation into its conservation.

In particleboard processing, the use of adhesive plays a vital role in the intermolecular structure of the material. Conventionally, urea formaldehyde is mostly used which also adds to the production cost and thus presents the boards as expensive commodity. Substitution of urea formaldehyde with a gum extracted from guill and perr (*Cissus populnea*) which is less expensive and forms a fire resistance structure can highly be a replacement to the conventional adhesives.

Therefore, reduction in cost of production, waste disposal and big investment opportunities.

CHAPTER TWO

2.0 LITERATURE REVIEW

Composite materials are referred to as an engineering products made from two or more different constituent materials with a significantly different physical or chemical properties and which remain separate and distinct on a macroscopic level within the finished structure (Durand, 2008). Azumah (2014) also stated that particleboards are panel products produced by compressing small particles of wood whiles simultaneously joining them together with an adhesive. Particleboard is a panel product manufactured by spraying wood particles with adhesive, forming them into a mat, and compressing the mat to desired thickness between heated Platens to cure the adhesive (Hoadley, 2000). Particle board is cheaper, denser and more uniform than conventional wood and plywood and is substituted for engineering work when cost is more important than strength and appearance. Particleboard can be made more appealing by painting or the use of wood veneers on visible surfaces.



Plate I: Engineering Particleboards (Stark *et al.*, 2010)

As stated by (Arslan *et al.*, 2007), Particleboard is an engineered wood product manufactured from wood chips and a synthetic resin or other suitable binder, which is pressed and extruded. Particleboard can also be said to be a three-layered board, with

fine particles on the top and bottom surfaces, and larger wood flakes in the middle. Particleboard can be said to be a generic term for a composite panel primarily composed of cellulosic materials (usually wood), generally in the form of discrete pieces or particles, as distinguished from fibre, bonded together with a bonding system, and which may contain additives. The wood particles are pressed and bonded together with resin creating a tight compact panel that can be machined cleanly. The surfaces are sanded smooth at the mill, ready for use or finishing with a high-pressure laminate, decorative foil or timber veneer. To produce panel board with good strength, smooth surfaces, and equal swelling, manufacturers ideally use a homogeneous raw material (Stark *et al.*, 2010).

Particleboard is one of the two main groups of particle composites which are commonly recognized based on size of wood components and the method of Production employed. Normally, flakes, chips or wafers are known as the major constituent in particleboards processing. Likewise, fibreboard is the other group of particle composite, having its major constituents as fibre and fibre bundles. The strength of the particleboard is strongly determined by the binder employed and not necessarily the fibre used, although the particle size and shape also have an influence on the strength (Kent and Riegel, 2007).

The BS EN 309:1992 defines the particleboard as: "panel material manufactured under pressure and heat from particles of wood (wood flakes, chips, shavings, saw-dust, wafer, strands, and similar) and/or other lignocellulose material in particle form (flax shaves, hemp shaves, bagasse fragments and similar) with the addition of an adhesive." Studies from Some other researchers had shown that non wood and agro-fibre can also be used as particleboard such as: (Alma *et al.*, 2005) studied the usability of cotton carpel in the study, urea– formaldehyde (UF) and melamine urea–formaldehyde (MUF) adhesives were used. More so, the chemical composition of cotton carpel and various mechanical

and physical properties of cotton carpel-based particleboards were also studied. From there produced particleboards were characterised, having the minimum physical and mechanical properties as required in the conventional standards for general purpose particleboards. likewise, the usability of Rice husk flour as a partial substitute for the wood particles used as the Feed stock for production of particleboards, by using urea-formaldehyde resin as the composite binder. The study examined the physical and mechanical properties of the rice husk flour-wood particleboard as a function of the type of urea-formaldehyde resin applied.

Rahim *et al.*, (2012) investigated the mechanical and physical performances of three layer experimental particleboards using Rubber wood particles in the face layers while kenaf particles in the middle layer of the Panels boards. In the study, Rates (70:30), (50:50) and (30:70) was the applied ratio rubber wood to kenaf particles respectively. Urea formaldehyde resin was used as the composite binder. The use of bamboo and pine for production of particleboard. In the study, urea-formaldehyde was employed as a binder in the proportion of 10 % of the dry weight of material, bamboo particles originating from the apical part of the culm, and pine particles derived from industrial processing. Tahir and Nasib (2009) more so, produced particleboard composite using kenaf and rubber wood of 12mm thickness, were urea formaldehyde resin was used and mixed with hardener and wax. The mixtures of rubber wood and kenaf particles at ratio 50:50 by weight with resin level 6, 8 and 10 %. Paridah *et al.*, (2014) investigated the effects of Rubber wood: Kenaf stem ratio to resin content on mechanical and dimensional stability properties of hybrid particleboard were determined. In the study, Particleboard produced from kenaf stem and Rubber Wood particle blends at different Wood loading (0 %, 50 %, 70 %, 100 %) and resin levels (6 %, 8 %, 10 %) were determined. the

adhesive used was Urea formaldehyde resin. Laemlaksakul (2010) produced particleboard using bamboo culm, the adhesive used was Urea formaldehyde resin.

2.1 Origin of Particleboard

The introduction of Modern plywood in building industries as an alternative to timber can be traced back to the 19th century, as at the end of the 1940s shortage of wood flakes was recorded which affect its production. Particleboard was intended to be a replacement. produced during World War II at a factory in Bremen, Germany. A German Max Himmelheber invented the particleboard. It used waste materials such as planer shavings, off cuts or sawdust, hammer-milled into chips, and bound together with a phenolic resin. Hammer-milling involves smashing material into smaller and smaller pieces until they pass out through a screen. Most other early particleboard manufacturers used similar processes, though often with slightly different resins. Haygreen and Bowler (1996) are of the opinion that the development of the particleboard industry was stimulated in Europe by lumber shortages and in the United States by large quantities of unused softwood mill residues. In the late 1940s, a number of particleboard plants were built in Europe and United States but the product was crude and the industry struggled to capture new markets. By 1960 the industry had been established and growing rapidly as the world production increased from 0.02 million m³/yr in 1950 to 3 million m³/yr in 1960 to 20 million m³/yr in 1990 (Anon, 2010).

It was found that better strength, appearance and resin economy could be achieved by using more uniform, manufactured chips. Manufacturers began processing solid birch, beech, alder, pine and spruce into consistent chips and flakes. These finer layers were then placed on the outsides of the board, with the central section composed of coarser, cheaper chips. This type of board is known as three-layer particleboard. As the world develops technologically, graded-density particleboard has also been modified. It

contains particles that gradually become smaller as they get closer to the surface (Anon, 2010).

As the world's need for sawn wood increased from 55 million tonnes from 1913 to 62million tonnes in 1950 and then to 102 million tonnes in 1980, particleboard has no record at all in 1913 and 1950 until 1980 when there was a record of 24.1 million tonnes (Azumah, 2014).

2.2 Manufacturing Process of Particleboard

The manufacturing processes of particleboard can be viewed under the following sections:

1. Pre-treatment/Chip preparation section
2. Drying and sifting section
3. Glue regulating and applying section
4. Forming and hot-pressing section
5. Cooling and sizing section
6. Sanding section

Shanghai Jinnan Import and Export Co. Ltd stated that particleboard is made from small diameter wood, branches, wood residues and non-wood agricultural residues via chip preparation, drying, spreading and hot press section. Finished boards can then be further used for furniture, building, packaging, vehicle and ship decoration and lamination (Anon, 2010).

Particleboard or chipboard is manufactured by mixing wood particles or flakes together with a resin and forming the mixture into a sheet. The raw material is fed into a disc

chipper with between four and sixteen radially arranged blades. The chips from disk chippers are more uniform in shape and size than from other types of wood chippers. The particles are then dried, and any oversized or undersized particles are screened out.

Resin is then sprayed as a fine mist onto the particles, amino-formaldehyde based resins are the best performing based on cost and ease of use. Urea Melamine resins offer water resistance with more Melamine offering higher resistance. It is typically used in external applications, with the coloured resin darkening the panel. To further enhance the panel properties, resorcinol resins can be mixed with phenolic resins, but that is more often used with marine plywood applications. Panel production involves other chemicals including wax, dyes, wetting agents and release agents, to aid processing or make the final product water resistant, fireproof, or insect proof. After the particles pass through a mist of resin sufficient to coat all surfaces, they are layered into a continuous carpet. This 'carpet' is then separated into discrete, rectangular 'blankets' which will be compacted in a cold press. A scale weighs the flakes, and they are distributed by rotating rakes. In graded-density particleboard, the flakes are spread by an air jet that throws finer particles further than coarse ones. Two such jets, reversed, allow the particles to build up from fine to coarse and back to fine. The formed sheets are cold-compressed to reduce thickness and make them easier to transport. Later, they are compressed again, under pressures between 2 and 3 MPa (290 and 440 psi) and temperatures between 140 °C and 220 °C (284 and 428 °F) to set and harden the glue. The entire process is controlled to ensure the correct size, density and consistency of the board. The boards are then cooled, trimmed and sanded. They can then be sold as raw board or surface improved through the addition of a wood veneer or laminate surface (Frederick and Norman, 2004).

Particleboard making starts with particles preparation in the laboratory after the trees are harvested and bucked into smaller segments before it is chipped by using a commercial

chipper. The chips are then reduced into particles using laboratory type hammer mill. The air-dried particles are screened on a vibrator screen machine to obtain the sizes of particles of 0.5-1.0 and 1.0-2.0 mm. The accepted particles are dried in an oven with temperature of 60 °C to achieve moisture content (MC) of 4-6 %. A pre-weighted amount of particles comprised are blended initially without binder to enable it to mix thoroughly prior to spraying of the furnish that comprised of urea formaldehyde resin, wax emulsion and ammonium chloride (NH₄Cl). Urea-formaldehyde resin catalyst is applied at a resin content of 10 % resin solids based on oven-dry weight of wood particles. The wax emulsion is applied at 1 % based on the weight of the resin and ammonium chloride (NH₄Cl) catalyst act as hardener was applied at 1 % based on the resin content (Loh *et al.*, 2010).

After blending, the particles are spread evenly into 340mm x 340mm wooden box former using metal cauls Plate as the base to produce a loose mat. The mat formed is initially pre-pressed manually to consolidate the thickness. Distance bars are placed at both sides of the mat in order to get the targeted board thickness during hot pressing. The mat is then hot pressed in a thermal-oil heated hydraulic hot press at an elevated temperature of 145 °C up to 200 °C with a specific pressure of 18 kg cm⁻² to achieve target thickness 12 mm (Mirski *et al.*, 2008). The mat is hot-pressed for 5 min based on the recommendation of the resin supplier. After hot-pressed, the boards are then conditioned in a conditioning room maintained at a relative humidity of 65 ± 5 % and 20 ± 2 °C for 7 to 10 days prior to Properties evaluation (Loh *et al.*, 2010).

The American National Standard for Particleboard (ANSI A208.1) classifies particleboard by physical, mechanical and dimensional characteristics as well as adhesive/binder levels. The Standard was developed through the sponsorship of the Composite Panel Association (CPA) in conjunction with producers, users and general

interest groups. The standard has a tiered system of emission levels allowing either a maximum of 0.18 ppm or 0.09 ppm for industrial grades or 0.20 parts per million (ppm) for manufactured home decking. To meet the needs of the market many particleboard manufacturers have voluntarily developed ultra-low-emitting and no added urea-formaldehyde (NAUF) products, so there are a wide variety of products available today with reduced formaldehyde levels, as well as a growing number of non-formaldehyde alternatives. ANSI A208.1 (2009) stated that formaldehyde shall not exceed 0.3 parts per million (ppm) under test conditions. Test conditions include an air exchange rate of 0.5/hour and test temperatures of 77 °C. Board should meet or exceed this requirement when manufactured. ANSI (2009) also indicated that the moisture content of particleboard should not exceed 10 % moisture content.

2.3 Application of Particleboard

Particleboard been a composite panel product consisting of wood particles such as sawdust, wood chips, sawmills shavings or other agricultural wastes that are bound together with a synthetic resin or other suitable binders under heat and pressure can be seen as an inexpensive alternative to solid wood panelling, and has emerged as a versatile substitute for wood in many applications. In building construction, particleboard find its application for decoration and acoustic purposes, AC duct covering, door panel inserts, wall panels, floor ceiling and roofing tiles (Asha, 2017). Today's particleboard gives industrial users the consistent quality and design flexibility needed for fast, efficient production lines and quality consumer products. Particleboard panels are manufactured in a variety of dimensions and physical properties providing maximum design flexibility for end users. Some of the common uses of particleboard are countertops, door core, floor under laying, manufacture of home decking, office and residential furniture, shelving, store fixtures, stair treads and kitchen cabinets (Davis and Dhingra, 2001).



Plate II: Engineering Particleboard Block (Asha, 2017)

2.4 Rice Husk

Rice husk is the by-product in rice milling operation with an approximately 20 percent of the total weight of the paddy grain being processed. The components of this rice husk are therefore determined by the milling method employed. Despite the abundant nature of this by-product products and its unique physical and chemical properties, it is however not being commercially used in Nigeria. Only a little portion of the rice husk produced is utilized in a meaningful way, the remaining part is burnt into ashes or dumped as a solid waste with little being used in animal feed formulation (Olalere *et al.*, 2015). The reasons behind the use of Rice Husk in the construction industry are its high availability, low bulk density ($90\text{-}150\text{ kg/m}^3$), toughness, abrasive in nature, resistance to weathering and unique composition.



Plate III: Rice Husk (Lee *et al.*, 2003)

The main components in Rice Husk are silica, cellulose and lignin. The composition of Rice Husk as a percentage of weight is shown in Table 2.1 (Jauberthie *et al.*, 2000). Rice Husk contains high concentration of silica in amorphous and crystalline (quartz) forms. The presence of amorphous silica determines the pozzolanic effect of the Rice Husk. Pozzolanic effect exhibits cementitious properties that increase the rate at which the material gains strength. The extent of the strength development depends upon the chemical composition of alumina and silica in the material. The external surface of the husk contains high concentration of amorphous silica which decreases inwards and is practically non-existent within the husk. The elemental composition of the rice husk is summarised in Table 2.1 below.

Table 2.1: Composition of Rice Husk (Anbu *et al.*, 2009)

Composition	wt. %
SiO ₂	18.80 – 22.30
Cellulose	28 -38
Protein	1.9 – 3.0
Fat	0.3 – 0.8
Other nutrients	9.3 – 9.5

2.5 Locust Beans Pod

The locust beans generally referred to as *Parkia biglobosa*, it is a perennial tree which belongs to sub-family of mimosodae and family of leguminosae (fabaceae). The trees are usually carefully preserved by the inhabitants of the area where they grow because they are valuable sources of reliable food, especially the seeds which serves as source of useful ingredients for consumption (Tor *et al.*, 2018). The seeds on fermentation are used in cooking. It has been reported that the husks and pods are good for livestock. The roots, barks, leaves, stems, flowers, fruits and seeds of *Parkia biglobosa* are all used medicinally

to treat a range of ailments, including diarrhea ulcers, pneumonia, burns, coughs, (Tor *et al.*, 2018). *Parkia biglobosa* also called African locust bean tree, Okpeye in Igala, Iyere in Igbo, Iyeke in Igede language in Nigeria, is a leguminous crop peculiar to the tropics, *Parkia biglobosa* is a tree that is not normally cultivated but found in population of two or more in the savannah regions of West Africa. *Parkia biglobosa* is found throughout the savannah lands of North central Nigeria covering Benue, Kaduna, Kwara, Kogi, Nassarawa, and Plateau States. The tree grows to the range of 7 to 20 metres high and bears pods that occur in large bunches and vary from 120 to 300 mm in length. A matured *Parkia biglobosa* of 20 to 30 years can bear about a tone and above of harvested fruits, the tree can start to bear fruits from five to seven years after its planting.



Plate IV: Locust Beans (Source; google/search)

In this study, the pod of *Parkia biglobosa* was considered to be used as one of the feed stock owing to the fact that large quantity of the pod is dumped/burnt after extracting the seed from it, some are washed off into streams hereby polluting the land/water bodies.

More so, *Parkia biglobosa* belong the hard wood (Deciduous tree) category which makes it has high level resistance to termite attack. As reported by Soetan *et al.*, (2014) Table 2.2 shows the elemental composition of the *Parkia biglobosa* consisting of; Calcium, Magnesium, Potassium, Sodium, Phosphorous, Manganese, Iron, Copper and Zinc.

Table 2.2: Composition of the seeds and leaves of locust beans (Soetan *et al.*, 2014)

Parameter	seed	leaf
Ca (%)	0.703	0.346
Mg (%)	0.356	0.184
K (%)	0.211	0.123
P (ppm)	47.392	79.833
Na (ppm)	86.729	21.446
Mn (ppm)	54.811	17.981
Fe (ppm)	69.828	26.334
Cu (ppm)	9.766	2.256
Zn (ppm)	12.156	5.529

2.6 Adhesive

The word adhesive, also known as glue, cement, mucilage, or paste is any compound that adheres or binds particles together when applied to one surface or both surfaces, of two separate items and then resists their separation (Pike and Roscoe, 2013). Adhesives are produced from either natural or synthetic sources. Resin is a natural or synthetic compound which is highly viscous in its natural state and hardens with treatment. Typically, resin is soluble in alcohol but not in water. Adhesives increase the resistant strength and stiffness of the composite sheet. The adhesion of the glue depends on the wood-adhesive bonding chain, the type of materials joined, or conditions under which it is applied. Adhesive bonding performance between wood elements is seen to be significantly influenced by the degree of penetration of the adhesive into the porous

network of interconnected cells. Adhesives used in the manufacture of particleboard should be flexible and soft to respond to the dynamic effects of swelling and shrinkage, yet impart the required strength. The adhesive must also withstand the rigours of particleboard manufacturing with sufficient flow to increase particle coverage (Anbu *et al.*, 2009).

The use of adhesives offers many advantages over binding techniques such as sewing, mechanical fastening, thermal bonding, and others. These include the ability to bind different materials together, so as to distribute stress more efficiently across the joint, the cost effectiveness of an easily mechanized process, an improvement in aesthetic design, and more so, an increased design flexibility. Some of the disadvantages of adhesive use may include decreased stability at high temperatures, relative weakness in bonding large objects with a small bonding surface area, and greater difficulty in separating objects during testing (Kozowyk *et al.*, 2017).

2.6.1 Origin of adhesive

Historians has shown that the use of adhesives was discovered in central Italy when two stone flakes partially covered with birch-bark tar and a third uncovered stone from the Middle Pleistocene era (circa 200,000 years ago) were found. This is thought to be the oldest discovered human use of tar- hafted stones (Mazza *et al.*, 2006). The birch-bark-tar adhesive is a simple, one-component adhesive. The first use of compound adhesives was discovered in Sibudu, South Africa. In this, 70,000-year-old stone segments that were once inserted in axe hafts were discovered covered with an adhesive composed of plant gum and red ochre (natural iron oxide) as adding ochre to plant gum produces a stronger product and protects the gum from disintegrating under wet conditions (Wadley *et al.*, 2009). The ability to produce stronger adhesives allowed mid stone-age humans to

attach stone segments to sticks in greater variations, which led to the development of new tools (Wadley and Lyn, 2010).

More recent examples of adhesive use by prehistoric humans have shown that at the burial sites of ancient tribes. Archaeologists studying the sites found that approximately 6,000 years ago the tribesmen had buried their dead together with food found in broken clay pots repaired with tree resins (Ebnesajjad and Sina, 2010). Another investigation by archaeologists uncovered the use of bituminous cements to fasten ivory eyeballs to statues in Babylonian temples dating to approximately 4000 BC (Mittal *et al.*, 2003).

The first references to adhesives in literature first appeared in approximately 2000 BC. Further historical records of adhesive use are found from the period spanning 1500–1000 BC. Artifact from this period include paintings depicting wood gluing operations and a casket made of wood and glue in King Tutankhamun's tomb (Ebnesajjad and Sina, 2010). Other ancient Egyptian artifacts employ animal glue for bonding or lamination. Such lamination of wood for bows and furniture is thought to have extended their life and was accomplished using casein (milk protein)-based glues. The ancient Egyptians also developed starch-based pastes for the bonding of papyrus to clothing and a plaster of Paris-like material made of calcined gypsum (Mittal *et al.*, 2003).

The Greeks and Romans made great contributions to the development of adhesives, through the use of marquetry and Wood veneering were developed, the production of animal and fish glues refined, and other materials utilized. Egg-based pastes were used to bond gold leaves incorporated various natural ingredients such as blood, bone, hide, milk, cheese, vegetables, and grains (Ebnesajjad and Sina, 2010). The Greeks began the use of slaked lime as mortar while the Romans furthered mortar development by mixing lime with volcanic ash and sand. This material, known as pozzolanic cement, was used

in the construction of the Roman Colosseum and Pantheon (Mittal *et al.*, 2003). The Romans were also the first people known to have used tar and beeswax as caulk and sealant between the wooden planks of their boats and ships (Ebnesajjad and Sina, 2010).

2.6.2 Chemical properties of adhesive

Basically, adhesives compose of carbon, hydrogen, oxygen, small quantities of mineral matter and sometimes a little nitrogen. Generally, gums also contain small quantities of tannin. The chemical composition of the three main exudates gums is complex and varies to some extent, depending on their source and age (Verbeken *et al.*, 2003). Adhesive bonding performance between wood elements is the measure or degree of penetration of the adhesive into the porous network of interconnected cells. Researches made on penetration of wood adhesives shows that the penetration of adhesive into wood can be categorized into two groups:

1. Gross penetration
2. Cell wall penetration.

The gross penetration results from the flow of liquid adhesive into the porous structure of wood, mostly filling cell lumens. Hydrodynamic flow and capillary action could be explained as gross penetration. While the Cell-wall penetration occurs when resin diffuses into the cell wall or flows into micro fissures. In wood, the least resistance to hydrodynamic flow is in the longitudinal direction, following the lumens in the long and slender trachea of softwood, or through the vessels of hardwoods. Since vessels are connected end-to-end with perforation plates and there is no pit membrane, this cell type dominates the penetration of adhesives in hardwoods (Onur, 2016). All of the potential adhesion mechanisms are influenced by penetration. The concept of mechanical interlocking is obviously dependent on penetration of the adhesive phase beyond the

external wood surface. In addition to the degree of penetration of adhesive, the combined adhesion force due to covalent bonding and formation of secondary chemical bonds which is directly related to the area of surface in contact between the adhesive and the cell wall.

Adhesion is the tendency of dissimilar particles or surfaces to bond to one another. The internal forces between molecules that are responsible for adhesion are chemical bonding, dispersive bonding, and diffusive bonding. These intermolecular forces can make cumulative bonding and bring certain emergent mechanical effects (Kamke and Lee, 2007). Cohesion word (cohaerere in Latin language) means “stick or stay together.” Cohesive force is the tendency of similar molecules to stick together. They attract mutually. Cohesive force caused by the shape and structure of molecules, which makes the distribution of orbiting electrons irregular when molecules get close to one another, creating electrical attraction that can maintain a microscopic structure such as a water drop (Onur, 2016). The adhesive and cohesive forces, keep together the adhesive with the substrate (adhesion) and the adhesive to itself (cohesion).

2.6.3 Uses of adhesive

In furniture and forest product industry, “wood adhesives” have played an important role in the development and efficient use of wood. In wood products, the most commonly used material is glue. If we check different wood products (plywood, MDF, particleboard, OSB, structural frame and wooden architectural doors, windows, and frames), adhesives are more important to retain their structure. Significant amounts of adhesives are used in floor coverings, kitchen counters and for ceiling and wall tiles. They are also used in non-structural applications, within car upholstery and accessories (Kamke and Lee, 2007).

2.6.4 Types of adhesive

2.6.4.1 Natural adhesives

Natural adhesives are generally referred to as the organic adhesives; they are set by solvent evaporation. Some of the adhesives that are in this category are animal glues, fish glues, vegetable glues, and casein.

In ancient times, plant and animal glues were prominently used, later they left the place for the synthetic resin. Animal glue has to fight against instability and resistance to hot water and microorganisms and the lack of block board limit their usage, though the use and application of synthetic resin in plywood production eliminate these drawbacks. Adhesives used in the furniture industry prior to 1930s were obtained from crop and animals, those obtained from animals are classified as: Animal glue or gelatine (those obtained from skin, bone, and fish residues), Blood (those obtained from the raw with blood from slaughterhouses), Casein (those derived from an animal milk protein).

Adhesives obtained from animals are of gluten origin and are obtained by boiling collagens in water agent. Animal based adhesives are gelatin-type adhesives, which are sourced from waste and by-products of the animal industries. Raw materials of animal adhesives are hides, sinews, and bones of cattle and other animals. The wastes of leather industry (from tanned hides) are also utilized. Adhesives that are made from hides are of higher grade than glue obtained from bones and tendons (Onur, 2016). In plywood and veneer industries, the glue is applied with a gluing machine. It is more important not to use excess glue unnecessarily. Because, in this case, the deterioration of the balance between the amount of water present in the glue and moisture content of the wood material will cause distortion, such as a crack up, swelling, and corrugated formation of drawbacks. The unfavourable side of animal glues is that it comes off when contacted with water or at 80 °C temperature and due to higher relative humidity of animal glue

bonding there is the loss of precision. Furthermore, animal glues, with the action of microorganisms can easily undergo deterioration.

Botanical adhesives are obtained mostly by processing starchy plants. As well as some of the resins of wood species fall into this group. Plant based adhesives are divided into two groups, the starch based and the cellulose based. The first group refers to those adhesives derived from starch, extracted from plants, such as corn, rice, potatoes, and wheat. They are mostly applied in bookbinding, paper bags, and cardboard boxes. The second group of plant adhesives is derived from cellulose, like; trees, shrubs, or fruits such as bananas, which are used more in stickers affixed to glass.

2.6.4.2 Semi-synthetic adhesives

The Semi-synthetic adhesives are Cellulosic derivatives likewise referred to as thermoplastic adhesives. Thermoplastics are in solid phase at normal temperature. On heating, they soften and melt. They occur as an adhesive solution, dispersion, and in solid form. A liquefied thermoplastic material can provide adhesion when allowed to cool. In solution and dispersion, they remain as a film by the evaporation of the liquid solvent. Usually, most of the thermoplastic adhesives have low/medium shear strength. The excellent resistance to oils is an advantage and very poor resistance to water is a disadvantage of this type of adhesives. Some good examples of this category include polyvinyl alcohol, polyacrylates, silicone resins, polyamides, acrylic acid diesters, polyvinyl acetate (Kumar, 2002).

2.6.4.3 Synthetic adhesives

The synthetic adhesives are generally referred to as the inorganic adhesives. They have many advantages for use in the woodworking industry over the organic adhesives. In the outdoor furniture, synthetic adhesives can be used in joints that remain as strong as the

wood even in unprotected exposure to the weather. Most of the “animal” adhesives can be used in furniture joints for interior use only (Onur, 2016). Inorganic adhesives are based on typical compounds, such as sodium silicate, magnesium oxychloride, lead oxide (litharge), sulphur, and various metallic phosphates. These materials form strong resistant bonds for special applications, and are still widely used. The advent of synthetic organic polymer adhesives during the last two decades has led to a decline in the use of many of the older inorganic adhesives laboratory recipes (Skeist, 2012).

2.6.4.4 Urea formaldehyde adhesives

Urea formaldehyde resins are widely used in chipboard or plywood production. Urea Formaldehyde resin is produced by heating suitable urea and formaldehyde at 115 °C for 5 hours. They are usually produced during the production of high quality glue E3 formaldehyde emissions. Urea Formaldehyde resins came into market in 1930s. Urea Formaldehyde resin can be formulated either for hot pressing or for room temperature curing by different types and amounts of catalyst. Urea Formaldehyde resins is compatible with various low cost extenders or fillers, thus permitting variation in both quality and cost. They are available with solid contents from about 40–70 % percent. They are also marketed as dry powders, with or without incorporating the catalyst. Urea Formaldehyde resins being used as an adhesive provide a number of advantages to manufacturers in the wood industry. The use of Urea Formaldehyde resins adhesives makes it superior to other chips and board them, providing the reasons for the choice of MDF and plywood production as follows:

- i. Low cost
- ii. A very different baking (curing) condition
- iii. Easy to use
- iv. Low energy consumption during production with low firing temperature

- v. Ability to easily dissolve in water
- vi. Microorganisms and abrasion resistance
- vii. Hardness
- viii. Great thermal properties

Amidst all these advantages, Urea Formaldehyde resin has its own disadvantages despite an outstanding advantage. Urea Formaldehyde resins has a high durability, especially in low humidity and high temperature. Hence Urea Formaldehyde resin produced from forest industry products is suitable for indoor use only. The combination of temperature with humidity reduces adhesive property of the urea formaldehyde and melamine urea formaldehyde adhesives in the product and leads to formaldehyde gas emission.

Urea Formaldehyde resins is inexpensive and used where surface smoothness is required but not high water resistance. Urea Formaldehyde resins is extensively used in particleboard manufacturing for interior applications such as furniture and cabinetry. However, it has a disadvantage of formaldehyde emissions at high temperature. Chemical bonds between urea and formaldehyde are weaker than PF and are easily cleaved by moisture.

2.6.4.5 Melamine resin adhesives

Melamine-formaldehyde resin is obtained from poly-condensation of melamine and formaldehyde. In the reaction between formaldehyde and melamine, the melamine (2, 4, 6 - triamino1, 3, 5- triazine) gives derivatives containing different numbers of methyl groups participating amine group. The number of methyl groups may be up to six. The water-soluble methyl melamine cross-linking at elevated temperatures over a methylene or ether bridges are converted to the formaldehyde resin. Melamine-formaldehyde resins are usually used in the impregnated decor paper, barrier lining the balance and

preservation, post-forming craft, and overlays. They are also used in production for hardwood Kraft paper impregnated overlay and coatings for the tray. Melamine-formaldehyde resin adhesives are sold in furniture market as powders. Melamine-formaldehyde resin adhesives are prepared by mixing with water or used with a Melamine-formaldehyde hardener. The appearance of Melamine-formaldehyde adhesive is almost white in colour, but the addition of filler usually gives them a light tan coloration similar to the urea resins. Melamine-formaldehyde resins are considerably more expensive than PF or UF resins.

2.6.4.6 Silicone adhesives

Silicone adhesives are known as polysiloxanes, it has the chemical structure of silicones, silicon (Si) and oxygen (O) atoms are sequentially arranged instead of carbon (C) contained polymer is the common name. The most methyl or phenyl groups are located bound to silicon atoms of the silicone molecules. Silicones are the most fluidic produced in the form of a resin. Silicone fluids are quite stable substances, and they are not affected by water or influenced by rising heat. They are very good electric insulators as well as hydraulic fluids and emulsion-breaking agents, and they are also used to reduce water permeability of various materials, such as paper. Silicone rubbers are also electrically insulating and chemically resistant and maintain flexibility in a wide temperature range. These are the important features. It is most commonly used in protective sheath and insulating varnishes. In furniture industry, silicone adhesives are usually used to: Finish material at counter-tops, bond edges and finish points at parquets, bond kitchen and bath cupboards and bond upholstering fabric of sofas. Single component silicone adhesives require a humidity of 5–95 % to cure. Besides the presence of humidity, a temperature between 5 and 40 °C is required to cure the adhesive.

2.6.5 Guill and perr (*Cissus populnea*)

Guill and perr is a tropical climbing plant usually found in West Africa, particularly in Nigeria. It belongs to the family of Ampelidaceae (Vitaceae). Its sap had been used as soup thickener, for treatment of venereal diseases and indigestion, drug binder and in ethno - medicine for treatment of male infertility (Olutayo *et al.*, 2019).

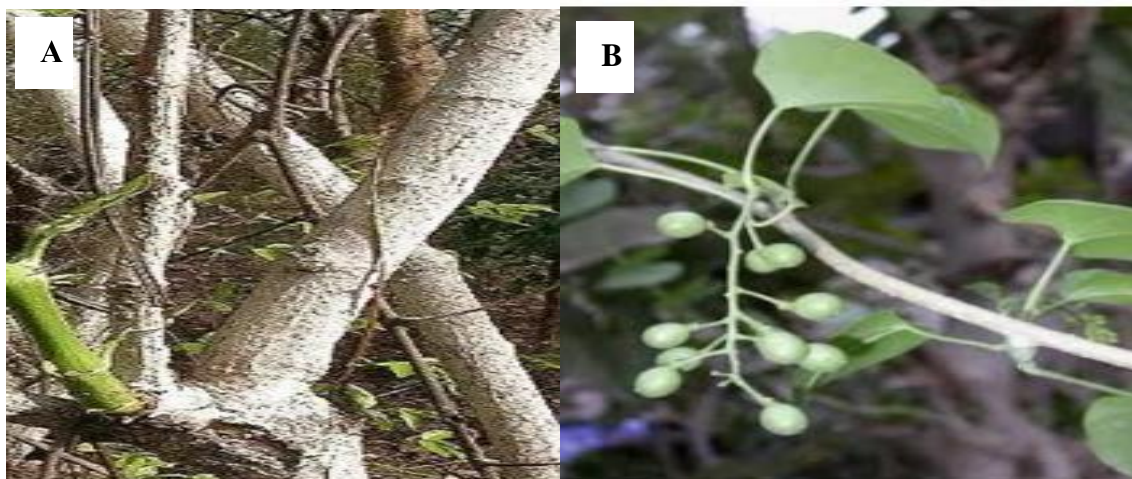


Plate V: The Guill and perr plant (A) Stem (B) Leaves (Oyedemi, 2012)

The demulcent property, biocompatibility, cheapness, lack of toxicity, soothing action and non-irritating nature of the sap favours its application usage in the manufacturing industries, the composition, characteristics and usefulness of guill and perr (*Cissus populnea*) fibre in composites applications are limitless. Natural fibre exhibits a high hydrophilicity due to interaction between the hydroxyl groups of fibre components and water molecules originated from non-crystalline region of the fibre (Batiancela *et al.*, 2014). This initiated poor interfacial adhesion and resistance to water absorption. Many surface modifications (such as acetylation, methylation, cyanoethylation/mercerization, benzylation, permanganate treatment, acrylation) have been used to improve the mechanical properties of fiber, density and water absorption for effective use in composites applications (Azeez and Onukwuli, 2018). Research in polymer composite applications is being directed towards the use of natural fibres as renewable resources for

reinforcement. Lignocellulose fibres also called “plant” or “natural” fibres such as bast, leaf or hard, seed, fruit, wood, cereal straw, and other grass fibres have been area of interest of researchers in polymer composites.

Lignocellulosic fibres are materials rich in lignin, hemicellulose and cellulose that are used for various applications such as yarns and textiles, ropes, twines and nets, non-woven fabrics, tissues, paper and board products, packaging, building and construction materials, fibre boards, insulation, geotextiles, composites and automotive parts (Emeje *et al.*, 2009). In composites, the applications of natural fibres depend on their composition, physical and mechanical properties.

The environmental benefits of natural fibres over synthetic fibres for industrial applications partly depends on the possibilities for replacement of the various fibres, energy requirement for the production process, product performance, functional life time and options for waste disposal (Oyedemi, 2012).

In Brazil, 93 % of natural fibres such as banana, jute, piassava, sponge-gourd, sugarcane, coco-nut, rice straw, sisal, ramie and coconut are replacing glass or other traditional reinforcement materials in composites (Emeje *et al.*, 2009). The advantages of natural fibres include:

- i. Low density
- ii. High toughness
- iii. Comparable specific strength properties
- iv. Reduction in tool wear
- v. Ease of separation
- vi. Reduced energy of fabrication
- vii. Non – toxic

- viii. Low cost
- ix. Availability

2.6.6 Characterization of adhesive

2.6.6.1 Ash content

In accordance with Association of Official Analytical Chemistry (AOAC) method (1990) on the performance of an adhesive, Ash content is determined from the loss in weight that occurs during continuous heating of the sample at high temperature in a furnace for over a period of time, allowing all organic matter to burn off without permitting any appreciable decomposition of the ash constituents.

2.6.6.2 Moisture content

Moisture content refers to the total amount of water present in a sample at normal or atmospheric temperature and pressure. This is done based on the difference between the net weight of sample and the weight after drying to constant (Oyedemi, 2012). It shows the rate at which the sample absorbs water when exposed to the atmosphere.

2.6.6.3 Determination of protein content

The protein content of Adhesives can be determined using the Association of Official Analytical Chemistry (AOAC) method (1990). In this method, the Adhesives are weighed into a digestion tube and an adequate volume of a concentrated sulphuric acid is also added in the tube. The tubes will then be placed in a digester pre-set at 410 °C and digested for few minutes. Little quantity of distilled water will be added to each tube when it has been cooled after digestion to prevent it from caking. Each of the tubes is to be placed in the distilling unit, with NaOH dispensed into it to dilute the solution. The mixture in the tube will further be distilled into Boric acid for few minutes. The mixture is then titrated against HCl until a grey colour will appear.

2.6.6.4 Determination of viscosity

Viscosity is the measure of the degree of fluidity of a liquid or the measure of fluid's resistance to flow. This is typically measured as the time required for a standard quantity of fluid at a certain temperature to flow through a standard orifice, it is a physical property by virtue of which substance is able to form, retain and offer resistance to shearing under heat and pressure (Aminudin *et al*, 2011). Viscosity decreases with increase in the substance temperature.

2.6.6.5 Determination of pH

The pH of any substance shows the degree of Acidity/Alkalinity of that particular substance. In accordance with the Association of Official Analytical Chemistry (AOAC) method (1990), the pH of an adhesive can be determined using a pH meter (Mettler Delta 340, Halstead, England).

CHAPTER THREE

3.0 RESEARCH METHODOLOGY

3.1 Materials

There are three major materials used as the feed stock in this research, for the gum extraction and the particle board production. These materials are:

- Guill and perr (*Cissus populnea*)
- Rice Husk
- Locust Beans Pod

The Guill and perr (*Cissus populnea*) were obtained from Inele Ugoh, Olamaboro local government area of Kogi State. The rice husks were sourced from Gidan Kwano Village of Niger State. The locust beans pod was obtained from Ogaji Ankpa Local Government area of Kogi State. More so, some reagents were used at different point, such as NaOH pellet, Ethyl acetate and Distilled water.

The milled steel was purchased from Minna Niger state while the lubricant was obtained from Mobil chemical market, Niger state and all other reagents used were for analysis unless stated otherwise.

Table 3.1: Materials/reagents used for this research work

Raw materials	Model	Source
Guill and perr	-	Inele Ugo, Olamaboro Kogi State
Rice husk	-	Gidan kwano Community Rice mill, Niger State
Locust beans pod	-	Inele Ugo, Olamaboro Kogi State
Distilled water	-	Chemistry Dept. KSU Anyigba
Sodium hydroxide (NaOH)	Burgoyne andCo,	Ochala Chemical Store, Ankpa Kogi state
Ethyl acetate	Gruppo montedison	Ochala Chemical Store, Ankpa Kogi state

3.1.1 Equipment

Table 3.2: List of equipment, model number and source

Equipment	Model number	Source
Digital weighing balance	Ohaus co operation	Chemistry Dept. KSU
Heating mantle	Gallenkamp	WAFB Dept. FUT
Funnel	Pyrex (England)	Chemistry Dept. KSU
Muslin cloth	-	Chemistry Dept. KSU
Plastic Bowl	-	Chemistry Dept. KSU
Oven	Gallenkamp	Chemistry Dept. KSU
Round bottom flask	Pyrex (England)	Chemistry Dept. KSU
Reflux condenser	Pyrex (England)	Chemistry Dept. KSU
Water bath(stirrer)	Shanghai (china)	Chemistry Dept. KSU
Measuring cylinder	Pyrex (England)	Chemistry Dept. KSU
Beaker	Pyrex (England)	Chemistry Dept. KSU
Conical flask	Pyrex (England)	Chemistry Dept. KSU
Digital pH meter	Jenco, KL-009	Chemistry Dept. KSU
FTIR	XPRT-PRO	Shetco chem. Advance Lab.

3.2 Methodology

3.2.1 Binder (gum)

After sourcing for all the required materials and equipment needed for the extraction of Gum handy, the barks of the fresh Guill and perr (*Cissus populnea*) stems obtained were scrapped off and chopped into smaller sizes (4-8 cm long) with an average weight of 0.2 kg. The required concentration of the reagent was prepared; to prepare 0.1M of NaOH, 4.0 g of NaOH pellet was weighed out using the weighing balance and 1000 ml of distilled water was used to dissolve it.

3.2.1.1 Delignification process

Delignification is the process of reducing the lignin content and other debris attached to the fibre of the sample (Chen, 2006). The chopped samples were treated with 0.1M NaOH in a plastic bowl of 10 L, this was to delignify the guill and perr (*Cissus populnea*) at room temperature for 48 hours, after which samples were washed repeatedly with distilled water to neutralize the sample.

3.2.1.2 Size reduction

Size reduction is the process of crushing or grinding particles of a substance to attain a size smaller than the previous state (Oyedemi, 2012). After the delignification process, samples were divided into two groups. The first group were Oven dried then pounded into powdery form and a mesh sieve was use to obtain a size of 150 µm. The second group was blended using a laboratory blender to obtain a paste.

3.2.1.3 Extraction of gum

After each group of the samples had undergone size reduction. The fresh samples were weighed out ranging from 30 – 50 g respectively and enclosed in a muslin cloth. Likewise, the dried samples were also weighed out ranging from 30 – 50 g respectively

and enclosed with a muslin cloth. Soxhlet extraction apparatus was used for the extraction of the gum using ethyl acetate and distilled water as the extraction solvents respectively. Each of the enclosed samples were placed in the thimble of the soxhlet extractor and the operating conditions were varied, having a temperature range of 90 - 110 °C for water and 65 - 85 °C for ethyl acetate respectively. More so, the operating time was varied from 30 min to 90 min for water and ethyl acetate respectively. Table 3.3 and Table 3.4 show the Variance of variable for response surface model used in the extraction process, comprising of the lower, centre and upper limit for the extraction.

Table 3.3: Variance of variable for response surface model ethyl acetate

Factor	Lower	Centre	Upper
Temperature (°C)	65	75	85
Extraction time (Min)	30	60	90
Amount (wt. %)	30	40	50

Table 3.4: Variance of variable for response surface model for water

Factor	Lower	Centre	Upper
Temperature (°C)	90	100	110
Extraction time (Min)	30	60	90
Amount (wt %)	30	40	50

3.2.2. Particleboard production

The method reported by Azumah (2014) from Shanghai Jinnan Import and Export Co. Ltd in line with the manufacturing process of particleboard was viewed under the following stages:

1. Chip preparation stage
2. Drying and sizing stage
3. Gum regulating and applying stage
4. Forming and hot-pressing stage
5. Cooling and sizing stage
6. Analysis/Characterisation (quality check)

3.2.2.1. Chip preparation stage

Rice husk and locust beans pod are the main feed stock in this particle board production processes. Upon obtaining considerable amount of these feed stocks, stones and other materials were handpicked from the samples. Distilled water was used to wash them separately to remove other significant impurities from the samples. After series of washing both samples were seen to be significantly clean, they were then prepared for drying.

3.2.2.2. Drying and sizing stage

The washed feed stocks were Oven dried at 80 °C within 8 h followed by size reduction stage; at this stage, particles of both samples were pounded separately using a wooden mortar and pestle. When desirable size was obtained after the crushing, the samples were sieve to obtain a particle size of 150 µm in accordance with Loh *et al.*, (2010).

3.2.2.3. Gum regulating and applying stage

In particleboard production, the ratio of gum to feed stock used play a very important role in meeting up to the regulatory standard. Percentage variation for rice husk and locust beans pod were carried out as shown in Table 3.5 and then, the ratio for gum to feed stock was optimised to know the optimum condition where the best yield will be produced as shown in Table 3.6 respectively.

Table 3.5: Percentage variation for rice husk and locust beans pod

Feed Stock	1 st Point	2 nd Point	3 rd Point	4 th Point	5 th Point
Rice Husk	0	25	50	75	100
Locust Beans Pod	100	75	50	25	0
Total (%)	100	100	100	100	100

Table 3.6: Variance for response surface model used in the particleboard production

Factor	Lower	Centre	Upper
Temperature (⁰ C)	60	70	80
Pressing time (Min)	5	10	15
Gum to Feed stock Ratio (wt. %)	1:3	1:6	1:9

3.2.2.4. Forming and hot-pressing stage

From the prepared mixture of feed stock and gum, after completion of mould, aggregate proportion of the mixture were properly poured into the mould as relative heat was applied. This was done in accordance with the result obtained from the Response Surface Methodology via composite design expert. This comprises of Rice Husk to Locust beans to Gum ratio, taking into consideration the temperature of the pressing and time spent during the pressing respectively. In place of the electric hot-pressing machine, the

electrical hot plate was used varying the temperature from 60 - 80 °C, applying pressure from both ends of the mould using bolts and Nuts.

3.2.2.5. Cooling and sizing stage

Cooling is the process of reducing the temperature of a substance. This was done after the forming and hot-pressing stage, to allow the particle board cools off as it dries out. This was done to reduce heat content of the particleboard. After the cooling process was the sizing of the particle board, this was done by trimming out the extruded edges and area of improper alignment as a result of the compression/pressure applied during the hot-press process. An emery paper was used to remove such areas of default.

3.2.3 Analysis on gum

Several characteristic tests were carried out on the gum extracted in accordance with Association of Official Analytical Chemistry (AOAC) method (1990) on the performance of a gum.

3.2.3.1 Determination of ash content

In the determination of the Ash content, the AOAC method (1990) for the determination of Ash content was employed. This was determined from the loss in weight that occurs during continuous heating of the sample at 550 °C in furnace for 4hours, which was enough to allow all organic matter to burn off without permitting any appreciable decomposition of the ash constituents (until the weight was constant). It was then removed and cooled in a desiccator for 30 min and the weights were noted. The calculation is shown below;

$$\% \text{ Ash} = \frac{W_f - W_c}{W_1} \times 100 \quad (3.1)$$

Where: W_c = initial weight of empty crucible

W_1 = Initial weight of sample

W_f = final weight of crucible and sample

Experimentally: Three porcelain crucibles were washed and dried in an oven to a constant weight at 100 °C for 5 min. They were allowed to cool in a desiccator, then labelled I, II and III then weighed (W_c). After noting the weight of the empty crucibles, 3.0 g of the sample were weighed into each of the previously weighed porcelain crucibles and reweighed (W_i). The crucibles containing the samples were then placed in a furnace, which was set at 550 °C for 4 h. They were then removed and allowed to cool in the desiccators then finally weighed (W_f). The percentage ash content was then calculated.

3.2.3.2 Determination of moisture content

The moisture content was carried out using the gravimetric method, based on the difference between the net weight and the weight after drying to constant (at 100 °C for 8 h). Three beakers were properly washed and allowed to dry in an air oven at 70 °C for 5 min to a constant weight. The beakers were allowed to cooled in a desiccator for 10 min, then labelled A, B and C, then weighed (W_1). 3.0 g of the sample was accurately weighed into the previously labelled samples and reweighed (W_2). Then the beakers were placed in an oven maintained at 100 °C for 8 h. They were removed and transferred to desiccators to cooled, finally weighed (W_3). The percentage moisture content was then calculated.

$$\% \text{ Moisture} = \frac{W_2 - W_3}{W_2 - W_1} \times 100 \quad (3.2)$$

3.2.3.3 Determination of lipid content

The percentage fat was determined using an automated method (AOAC, 1980). This is done by the continuous extraction of fat content from the sample using suitable solvent at 40 - 70 °C in a soxhlet extractor (n-hexane). 2.0 g of sample were placed in the extraction thimbles, and then placed in the extraction jacket, a clean dried 500 ml round bottom flasks containing 300 ml n-hexane. The round bottom flasks and the condenser were connected to the soxhlet extractor and cold-water circulation was put on. The heating mantle was switched on the heating rate was adjusted until the solvents were refluxing at a steady rate. Extraction was carried out for 6 h. The solvents were recovered and the extract was dried in the oven at 70 °C for 1 h.

The percentage fat was calculated as thus:

$$\% \text{ Fat} = \frac{W_f - W_c}{W_i} \times 100 \quad (3.3)$$

Where: W_c = initial weight of empty crucible

W = Initial weight of sample

W_f = final weight of crucible and sample

3.2.3.4 Determination of protein content

The protein content of the sample was determined using the Association of Official Analytical Chemistry (AOAC) method (1990). 1.0 g of the sample was weighed into a digestion tube and 15 ml of concentrated sulphuric acid and 5 kjeldahl tablet were also placed in the tube. The tubes were then placed in a digester pre-set at 410 °C and digested for 45 min. 75 ml of distilled water was added to each tube when it has been cooled after digestion to prevent it from caking. Each of the tubes was placed in the distilling unit, with 50 ml of 40 % NaOH dispensed into it to dilute the solution. The mixture in the tube

was further distilled into 25 ml of 4 % boric acid for 5 min. The mixture was then titrated against 0.47 M of HCl until a grey colour was obtained. A blank sample which was subjected to the procedures was also titrated with 0.47 M of HCl. The percentage total nitrogen was computed as follows:

$$\% \text{ Total Nitrogen} = \frac{(14.01 + S_1 - B_1 \times M)}{10 \times S_w} \times 100 \quad (3.4)$$

Where: S_1 = Sample initial titre

B_1 = blank titre

M = Molarity of HCl

S_w = Sample weight

3.2.3.5 Determination of crude fibre

The crude fibre content was determined according to AOAC methods (1990). The defatted sample (1 g) was weighed into a 600 ml beaker. 100 ml Trichloroacetic acid (TCA) reagent (obtained from mixing 500 ml glacial acetic acid, 450 ml water and 50 ml concentrated HNO_3 and then dissolving 20 g of trichloroacetic acid in the mixture) was added. The mixture was then allowed to boil and reflux. The flask was removed, cooled slightly and then filtered. The residue was then washed six times with hot distilled water and once with methylated spirit. The filter paper with the sample was transferred into a porcelain crucible and dried in an oven for 24 h. After cooling in a desiccator, the sample was weighed (weight A). It was then ashed in a muffle furnace at 600 °C for 4 h. After cooling in a desiccator, it was weighed again (weight B). The loss in weight during incineration is equivalent to the amount of crude fibre. The percentage fibre was obtained from equation below.

$$\% \text{ Crude fibre} = \frac{A - B}{\text{weight of sample}} \times 100 \quad (3.5)$$

3.2.3.6 Determination of the carbohydrate content

The starch and sugar contents were quantitatively determined using the method described by Dubois *et al.* (1956). 0.02 g of starch was weighed into a centrifuge tube and the powder was wetted with 1ml of ethanol. Distilled water (2 ml) was then added followed by 10 ml of hot ethanol and vortex. This was centrifuged for 10 min at 2000 rpm. The supernatant was decanted into a test tube and made up to 20 ml extract to determine the sugar content. For the assay, 0.8 ml of distilled water was added to aliquot (0.2 ml) of supernatant followed by 0.5 ml of 5 % phenol and vortex. Concentrated H₂SO₄ (2.5 ml) was added and vortex. The sediment obtained after centrifuging was hydrolysed with per-choric acid and used to estimate starch content. Phenol-sulphuric reagent was used for colour development and glucose standards were used for estimation of sugar. The resulting mixtures were cooled and read at absorbance of 490 nm. The free sugar content was calculated as:

$$\% \text{ sugar} = \frac{(A-I) \times DF \times V}{B \times W \times 10^6} \times 100 \quad (3.6)$$

Where: A is the absorbance of sample

I is the intercept of sample

DF is the dilution factor

V is the volume

B is the slope of the glucose standard curve

W is the weight of the sample.

3.2.3.7 Determination of Swelling Index

In determining the swelling index, 2.0 g of the sample was poured into a 10 ml measuring cylinder and the volume occupied was noted (V_1). 8ml of distilled water was added and the dispersion was well shaken. Water was added to make up to 10 ml. The dispersion was allowed to stand for 24 h and the volume (V_2) was noted. The swelling index (S) was calculated from the equation stated below:

$$S = \frac{V_1 - V_2}{V_1} \times 100 \quad (3.7)$$

3.2.3.8 Determination of viscosity and specific gravity

Viscosity was determined using a homogenous and well blended mixture of the samples at a room temperature, using a Viscometer (8-speed model 800, serial 03-857, Ofite Testing Equipment Inc., Houston, Texas, USA). The spindle was set at 300 r.p.m. and 600 r.p.m speeds in succession. The viscosity was calculated from the readings obtained.

A density bottle of 25 ml capacity was weighed empty and the mass, M_0 was recorded. The bottle was then filled with distilled water and weighed, the mass M_1 was recorded. After these, the bottle was also filled with Sample and weighed, the mass M_2 was recorded. This procedure follows the ASTM D1298 standard, the specific gravity was calculated using the equation below.

$$\text{Specific gravity} = \frac{M_2 - M_0}{M_1 - M_0} \times 100 \quad (3.8)$$

3.2.3.9 Determination of pH

The pH of any substance shows the degree of Acidity/Alkalinity of that particular substance. The pH of the sample was determined in duplicate using a pH meter (Mettler Delta 340, Halstead, England).

3.2.4 Analysis on particleboard

Several characteristic tests were performed on the particleboard produced in accordance with DIN EN 312 (German Institute for Standardization (DIN) 2003), which requires that particleboards for construction purpose in a wet area without much load-bearing function must undergo three main standard tests. These standard tests carried out on the produced particleboards are:

1. Bending test
2. IB strength test
3. Swelling test

These tests were necessary to ascertain if the panels satisfy the minimum required values of MOR, IB strength, and swelling in thickness due to moisture absorption. The DIN EN 312 particleboard requirement is equivalent to the American National Standard A208.12009 (CPA 2009) for wood particleboard classes M-2 and M-3 (Xu *et al.*, 2013). More so, other physical and mechanical tests were carried out including:

1. Moisture content test
2. Particleboard density
3. Water absorption test

This was done in accordance to ANSI A208.1-2009, an American Standard of Testing and Methods (ASTM) which were compared with the minimum required for particleboard production by the norm of these Standards Institute. Particle board Morphology Characterization using SEM was also carried out on the sample to analyse the sample surface morphology and compatibility.

3.2.4.1 Bending test

The test was carried out on each of the samples to determine their response to the applied pressure from the machine. This was done using the Instron machine, the test shows the Modulus of rupture on the samples. Modulus of rupture (MOR) is a measure of the ability of a sample to resist a transverse (bending) force perpendicular to its longitudinal axis. The bending strength of each test piece was calculated from the formula show below.

$$\text{MOR} = \frac{3 f_{\max} L_1}{2bt^2} \quad (3.9)$$

Where: f_{\max} is the maximum load

L_1 is the distance between the centres of the supports, in millimetres

b is the width of the test piece in millimetres

t is the thickness of the test piece in millimetres.

3.2.4.2 IB strength test

In accordance with DIN EN 312, Internal Bond Strength of a sample is the ability of the material to withstand internal stress within the sample in relation to its gum bond. Internal Bond Strength of a sample generally correlates with the density in the panel core. This was investigated by using Instron machine to compress the sample to determine the IB strength.

3.2.4.3 Swelling test

A swelling test was conducted to determine any change in sample's dimension in accordance with DIN EN 312. The samples were carefully measured out and dimensions of 100mm by 50mm with 15mm thickness each were obtained. The samples were submerged under the distilled water after taking their measurements at room temperature for 24 hours to determine long term water resistance properties, respectively. The

samples were taken out and surfaces were dried using a clean dry cloth after going through an immersion process. The weight and thickness of the samples were measured before and immediately after soaking. The thickness swelling was calculated as follows:

$$(\%)S = \frac{(T_f - T_o)}{T_o} \times 100 \quad (3.10)$$

Where: S is the swelling (%)

T_f is the final thickness after soaking in the period of 24h

T_o is initial thickness

3.2.4.4 Modulus of elasticity (MOE)

The MOE of a material show the extent to which a material can be stretched before yielding or breaking. In accordance with DIN EN 312, this was done using the Instron machine. The modules of elasticity of each test piece were calculated from the formula below:

$$MOE = \frac{f_{\max} L_1^3}{4bdt^3} \quad (3.11)$$

Where: f_{\max} is the maximum Load

L_1 is the distance between the centres of the supports, in millimetres

b is the width of the test piece in millimetres

t is the thickness of the test piece in millimetres.

d- is the deformation occurring against load

3.2.4.5 Moisture content test

Moisture content refers to the total amount of water presence in a sample at normal or atmospheric temperature and pressure. This is done based on the difference between the net weight of sample and the weight after drying to constant (Oyedemi, 2012). It shows the rate at which the sample absorbs water when exposed to the atmosphere. The moisture content of samples was carried out in accordance to ANSI A208.1-2009, an American Standard of Testing and Methods (ASTM). The weight of each samples were carefully measured out and dimensions of 100mm by 50mm with 15mm thickness each were obtained to determine the moisture content of each. The samples were then dried for 12 h at 80 °C. After the drying, the oven dry weight for each sample was taken. They were weighed again for every 4 hours until a constant weight was obtained. The moisture content for each sample was calculated as follows:

$$\% \text{ Moisture} = \frac{W_1 - W_2}{W_2} \times 100 \quad (3.12)$$

Where: W_1 is the weight of sample before drying

W_2 is the constant weight of sample obtained after Oven dried.

3.2.4.6 Particleboard density

The density (ρ) of a material is seen to be the ratio of its Mass to the Volume occupied by the material. Hence the density was evaluated by measuring the weight the sample and their corresponding dimensions. From the values obtained, density (ρ) was calculated using the following expression:

$$\text{Density } (\rho) = \frac{\text{Mass of Sample}}{L \times B \times H} \quad (3.13)$$

Where: L is the Length of sample

B is the breadth of the sample

H is the height of the sample

3.2.4.7 Water absorption test

The water absorption test was conducted to determine the rate of water penetration into the sample which will in turn result in physical change in samples dimension. In accordance with the DIN EN 312, the weight of each samples were carefully measured out after which, they were submerged in the distilled water after taking their measurements at room temperature for 12 h to determine long term water resistance properties, respectively. The samples were taken out and surfaces were dried using a clean dry cloth after going through an immersion process. They were weighed again for every 4 hours until a constant weight was obtained. Water absorption at time (t) was calculated as follows:

$$W.A(t) = \frac{(W_f(t) - W_o)}{W_o} \times 100 \quad (3.14)$$

Where: W.A (t) is the water absorption (%) at time (t),

W_o is the initial weight

$W_f(t)$ is the weight of the samples at a given immersion time, t.

3.2.4.8 Scanning electron microscopy

The surface morphology of the samples was observed using SEM machine, this was carried out to check the surface morphology of the samples. Moderate resolution scanning electron microscope (SEM) of JEOL JSM – 630 J model was used to study the morphology of surfaces of the samples.

CHAPTER FOUR

4.0

RESULTS AND DISCUSSION

4.1 Extraction Yield of Gum

After the delignification and the extraction of Gum from Guill and perr (*Cissus populnea*) Plant, the percentage yield from both dried and fresh samples was calculated as shown in Table 4.1 and Table 4.2, using ethyl acetate and water as extraction solvent respectively.

Table 4.1: Extraction yield of gum using ethyl acetate

Std	Mass of Fresh Sample (g)	Mass of Dried Sample (g)	Production Yield from Fresh Sample (%)	Production Yield from Dried Sample (%)
1	50.00	50.00	70.80	60.00
2	50.00	50.00	70.40	65.20
3	30.00	30.00	72.00	71.33
4	30.00	30.00	73.33	70.00
5	40.00	40.00	84.15	72.00
6	50.00	50.00	76.40	66.80
7	30.00	30.00	74.00	70.00
8	30.00	30.00	72.67	66.67
9	50.00	50.00	76.80	68.40
10	40.00	40.00	80.50	67.50
11	40.00	40.00	82.00	68.50
12	40.00	40.00	78.50	66.50
13	30.00	30.00	71.33	67.33
14	50.00	50.00	77.60	67.20
15	40.00	40.00	81.00	65.00

Table 4.2: Extraction yield of gum using water

Std	Mass of Fresh Sample (g)	Mass of Dried Sample (g)	Production Yield from Fresh Sample (%)	Production Yield from Dried Sample (%)
1	50.00	50.00	68.40	67.60
2	50.00	50.00	72.80	70.40
3	30.00	30.00	73.33	70.00
4	30.00	30.00	74.67	71.33
5	40.00	40.00	77.50	75.50
6	50.00	50.00	73.60	66.80
7	30.00	30.00	76.00	72.67
8	30.00	30.00	72.67	69.33
9	50.00	50.00	74.80	70.00
10	40.00	40.00	74.50	67.00
11	40.00	40.00	68.00	70.50
12	40.00	40.00	78.00	68.00
13	30.00	30.00	74.00	67.33
14	50.00	50.00	77.60	69.20
15	40.00	40.00	73.50	69.50

From Table 4.1 (Ethyl Acetate), the optimum yield was obtained at the samples dosage of 40 g (highest point) and 50 g (lowest point) from the fresh sample respectively. Likewise, the optimum yield from the dried sample were obtained from sample dosage of 40 g (highest point) and 50 g (lowest point) respectively.

More so, from Table 4.2 (water), the optimum yield was obtained at the samples dosage of 50 g (highest point) and 40 g (lowest point) from the fresh sample respectively. Likewise, the optimum yield from the dried sample were obtained from sample dosage of 40 g (highest point) and 50 g (lowest point) respectively. These results obtained can be attributed to the effects of the process variables, such as; Temperature and extraction time. Table 4.2 shows the effect of process variables on the extraction yield using Response surface methodology.

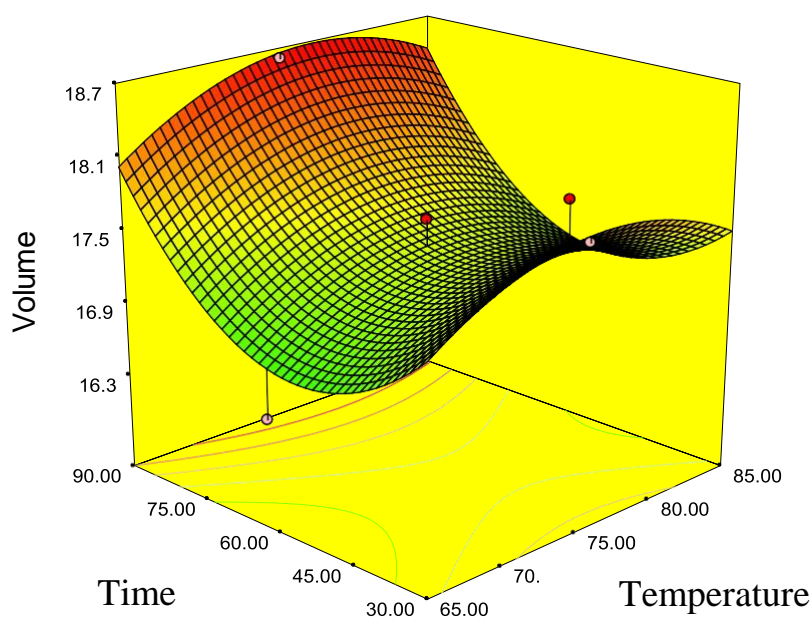


Plate VI: Extraction response using ethyl acetate fresh sample

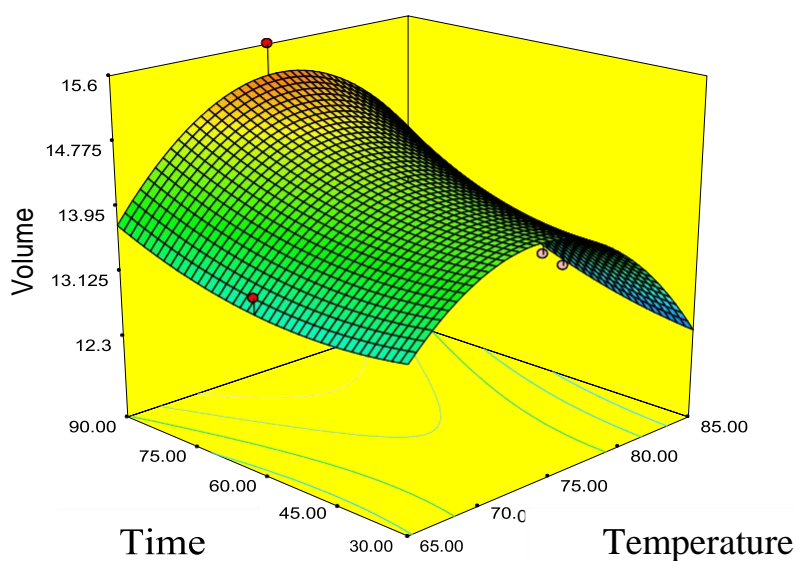


Plate VII: Extraction response using ethyl acetate for dried sample

From Plate VI and Plate VII, it was indicated Statistical that the highest yield (84 %) was obtained at 75 °C with 40 g sample dosage at 90 min for fresh sample using ethyl acetate as extraction solvent. While the lowest yield (60 %) was obtained at 85 °C with 50 g sample dosage at 30 min from dried sample using ethyl acetate as extraction solvent respectively.

Table 4.3: Extraction response using ethyl acetate for fresh and dried samples

Std	Run	Factor 1 Temperature (⁰ C)	Factor 2 Time (Minutes)	Factor 3 Amount (g)	Fresh Sample (Wt. %)	Dried Sample (Wt. %)
6	1	85.00	30.00	50.00	70.80	60.00
5	2	65.00	30.00	50.00	70.40	65.20
13	3	75.00	60.00	30.00	72.00	71.33
4	4	85.00	90.00	30.00	73.33	70.00
12	5	75.00	90.00	40.00	84.15	72.00
7	6	65.00	90.00	50.00	76.40	66.80
2	7	85.00	30.00	30.00	74.00	70.00
3	8	65.00	90.00	30.00	72.67	66.67
8	9	85.00	90.00	50.00	76.80	68.40
11	10	75.00	30.00	40.00	80.50	67.50
15	11	75.00	60.00	40.00	82.00	68.50
9	12	65.00	60.00	40.00	78.50	66.50
1	13	65.00	30.00	30.00	71.33	67.33
14	14	75.00	60.00	50.00	77.60	67.20
10	15	85.00	60.00	40.00	81.00	65.00

Similarly, Table 4.3 indicates the optimum points for the yield as reflected on Plate VI and Plate VII showing highest yield of 84 % and lowest yield of 60 % respectively.

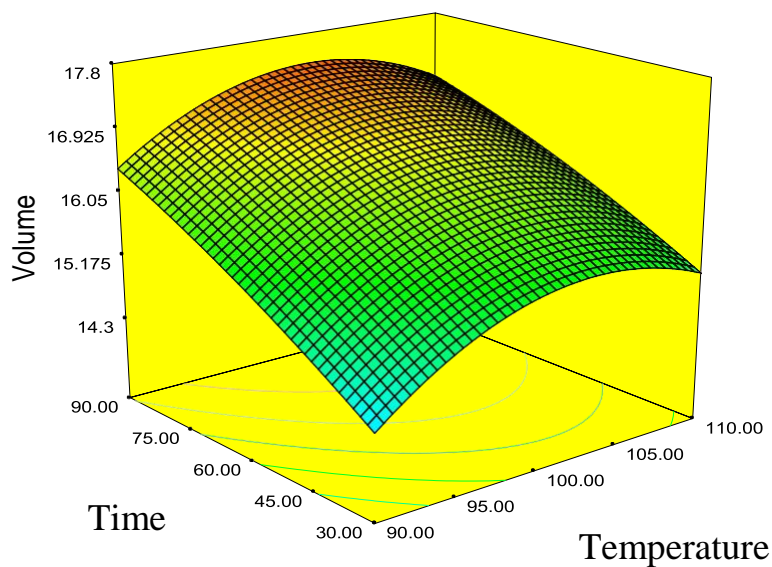


Plate VIII: Extraction response using water for fresh sample

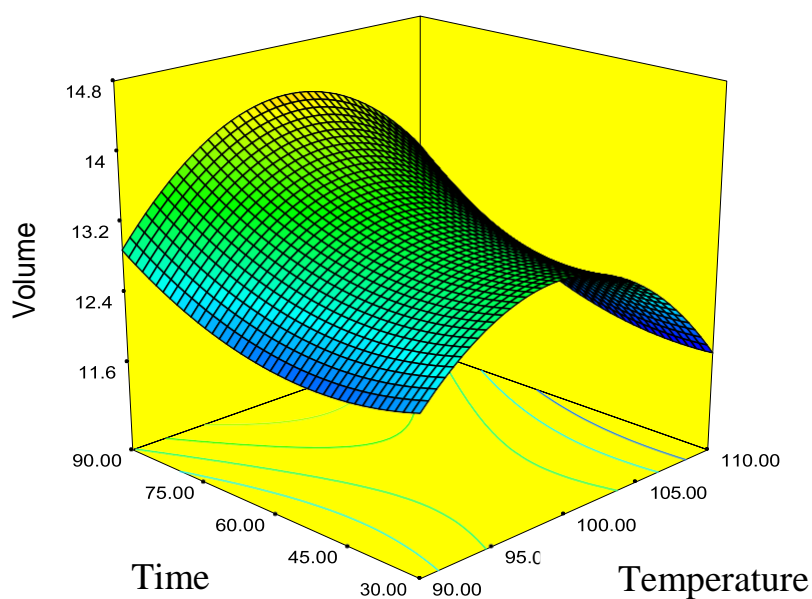


Plate IX: Extraction response using water for dried sample

The Plate VIII and Plate IX, indicate the optimum point of highest yield (78 %) was obtained at 100 °C with 40 g sample dosage at 60 min for fresh sample using water as extraction solvent. While the lowest yield (66 %) was obtained at 90 °C with 50 g sample dosage at 90 min from dried sample using ethyl acetate as extraction solvent respectively.

Table 4.4: Extraction response using water

Std	Run	Factor 1 Temperature ° (°C)	Factor 2 Time (Minutes)	Factor 3 Amount (g)	Fresh Sample (Wt. %)	Dried Sample (Wt. %)
6	1	110.00	30.00	50.00	68.40	67.60
5	2	90.00	30.00	50.00	72.80	70.40
13	3	100.00	60.00	30.00	73.33	70.00
4	4	110.00	90.00	30.00	74.67	71.33
12	5	100.00	90.00	40.00	77.50	75.50
7	6	90.00	90.00	50.00	73.60	66.80
2	7	110.00	30.00	30.00	76.00	72.67
3	8	90.00	90.00	30.00	72.67	69.33
8	9	110.00	90.00	50.00	74.80	70.00
11	10	100.00	30.00	40.00	74.50	67.00
15	11	100.00	60.00	40.00	78.00	70.50
9	12	90.00	60.00	40.00	69.00	68.00
1	13	90.00	30.00	30.00	74.00	67.33
14	14	100.00	60.00	50.00	77.60	69.20
10	15	110.00	60.00	40.00	73.50	69.50

Similarly, Table 4.4 indicates the optimum points for the yield as reflected on Plate VI and Plate VII showing highest yield of 78 % and lowest yield of 66 % respectively. This maintained that ethyl acetate has sustainable activity and shows a good extraction process variable to use in the extraction technique.

4.1.1 Effect of extraction temperature on gum yield

The extraction temperature plays a very vital role in gum extraction from both fresh and dried sample using ethyl acetate and water as extraction solvent respectively. Using the ethyl acetate, from the fresh sample at lower temperature of 65 °C there was no significant yield of the Gum extracted, despite the high time of extraction and sample dosage. The temperature proportion was varied from 65 - 85 °C, this was used as result of the fact that ethyl acetate has a boiling point of 64.8 °C and more so a temperature of 70 °C and 90 °C was reported by Cosreanue *et al.*, (2015). Table 4.3 shows the effect of temperature variation on the percentage yield obtained using ethyl acetate. From the Table, for the values of temperature chosen for this particular study, yield is slightly increased up to 60 °C temperature and after that, the yield (94 %) then decreases. This could be due to the fact that the rate of extraction is greatly influenced by temperature thereby resulting to higher yield. The yield proportional increases with increase in extraction, also temperatures greater than 85 °C has lower yield, owing to the fact that there is a high chance of losing the solvent since it is above its boiling point. Likewise, using water shows that from the fresh sample at lower temperature of 90 °C below the boiling point, an average yield was observed despite the high time of extraction and sample dosage this could be due to the hydrophilic nature of the sample. The temperature proportion was varied from 90-110 °C, since water has a boiling point of 100 °C, all so a similar temperature range was reported by Oyedemi (2012). Table 4.4 shows the effect of temperature variation on the percentage yield obtained using water. From the Table, for the values of temperature chosen for this particular study, the highest yield (78 %) was observed at 100 °C in the fresh sample while an optimum yield of 72.67 % was recorded at 110 °C in the dry sample respectively.

4.1.2 Effect of extraction time on gum yield

In most chemical processes, time of reaction contribute a lot to its conversion or yield. For this study, the reaction time was varied from 30 - 90 minutes. The effect of extraction time variation on conversion efficiency is shown on Table 4.2. The yield obtained is increased generally with increase in the extraction time to a certain time. This is seen in both the fresh and dried samples respectively. The yield of gum increases as the extraction time increases from 30 to 90 min and then the yield stabilizes. Longer extraction time above the optimum yield will not have any positive impact on the yield obtained as this will rather increase the production cost.

4.2 Analysis on Gum

4.2.1 Proximate analysis on gum

Proximate analysis as seen on Table 4.5, the Ash, Moisture, Protein, Fat, Fibre and carbohydrate content of the gum were: 3.12 %, 12.74 %, 5.56 %, 0.68 %, 1.12 % and 76.78 % w/w respectively. These values compare favourably with those reported in literature. The carbohydrate content of the gum is slightly less than the values reported by Petrie (2006) and Oyedemi (2012), ranging from 82.31- 87.81 % w/w. It is also slightly greater than 51.9 - 68.3 % reported by Wagner (2008). Nadir and Songklod (2010) had noted that carbohydrates are the major components of Gums. Moisture content is a measure of the amount of water molecule present in a sample at normal or atmospheric temperature and pressure. The mean moisture content from the results showed that the moisture content of the gum was 12.74 % while that of Oyedemi (2012) was reported to be 12.57 %. Excessive moisture in a material could lead to activation of enzymes and the proliferation of microorganisms thereby affecting the Gum (Petrie, 2006). The control of relative humidity is therefore important during storage of the Gum for optimum shelf-life. The ash content of the Gum was slightly higher than that of

Oyedemi (2012) but lower than the value reported by (Petrie, 2006). The ash content is an index of mineral contents of the substance (Nemu, 2002). Protein content of the Gum sample from the result was 5.56 % and can be attributed to the low degree of Delignification of the samples.

Table 4.5: Proximate analysis of gum

Composition	Percentage (w/w %)
Ash	3.12
Moisture	12.74
Protein	5.56
Fat content	0.68
Fiber Content	1.12
Carbohydrate content	76.78

4.2.2 Physiochemical analysis of gum

The Physiochemical Analysis carried out on the gum was to indicate if the extract obtained contain some of the properties displayed by similar product reported in literature. This can be seen on Table 4.6. From the analysis, the Density, pH, Viscosity and Specific gravity of the gum were; 1.25 (g/cm³), 6.5, 0.294 (N.s/m²), 1.25 respectively. The result shows that the pH value is within the range as compared with the other gums that have been reported in literature. Petrie, (2006) reported a pH range of plant gum of 5.69-6.49. Oyedemi (2012) likewise gave a pH range of the purified and crude gum to be in the acid range (6.5 – 6.8). The pH of the polyamine – gums formulated with mucuna and African yam bean were 6.3 and 5.5 respectively (Tamakloe, 2000).

Table 4.6: Physiochemical analysis of gum

Composition	Value
pH	6.5
Specific Gravity	1.25
Viscosity	0.294 (N.s/m ²)
Density	1.25 (g/cm ³)

Table 4.7: Elemental composition of gum

Element	Composition (w/w %)
Oxygen	47.6
Carbon	42.8
Hydrogen	8.2
Nitrogen	1.4

4.2.3 Fourier transform infra-red (FT-IR) spectrum

The FT-IR spectrum of the extract, locust beans pod, rice husk, and particleboard are shown in Figure 4.1. The finger print region of the spectrum consists of characteristic peaks between 800 and 3400 per cm; these peaks are attributed to the C-N stretching, the C-O bond stretching, the OH stretching, N-H bond stretching and C-H bond stretching respectively. The FT-IR spectra of the extract confirmed the presence of intense bands of functional groups. This report was compared with that of Emeje *et al.*, (2009), as similar properties were observed with a sharp band at 2939 per cm for methyl C-H characteristic stretching associated with aromatic rings. The broad band at 3286 cm⁻¹ is due to the hydrogen-bonding that contributes to the complex vibration stretches associated with free inter and intra-molecular bound hydroxyl groups which make up the gross structure of carbohydrates (Unsal *et al.*, 2009). This is all consistent with a polysaccharide structure that is neither a starch nor cellulose, but does have some peptide cross links and some amino-sugars. The essentially neutral pH of this material leads us to conclude that there can be very few free carboxyl groups to contribute to hydrogen bonding which can be attributed to its adherent properties.

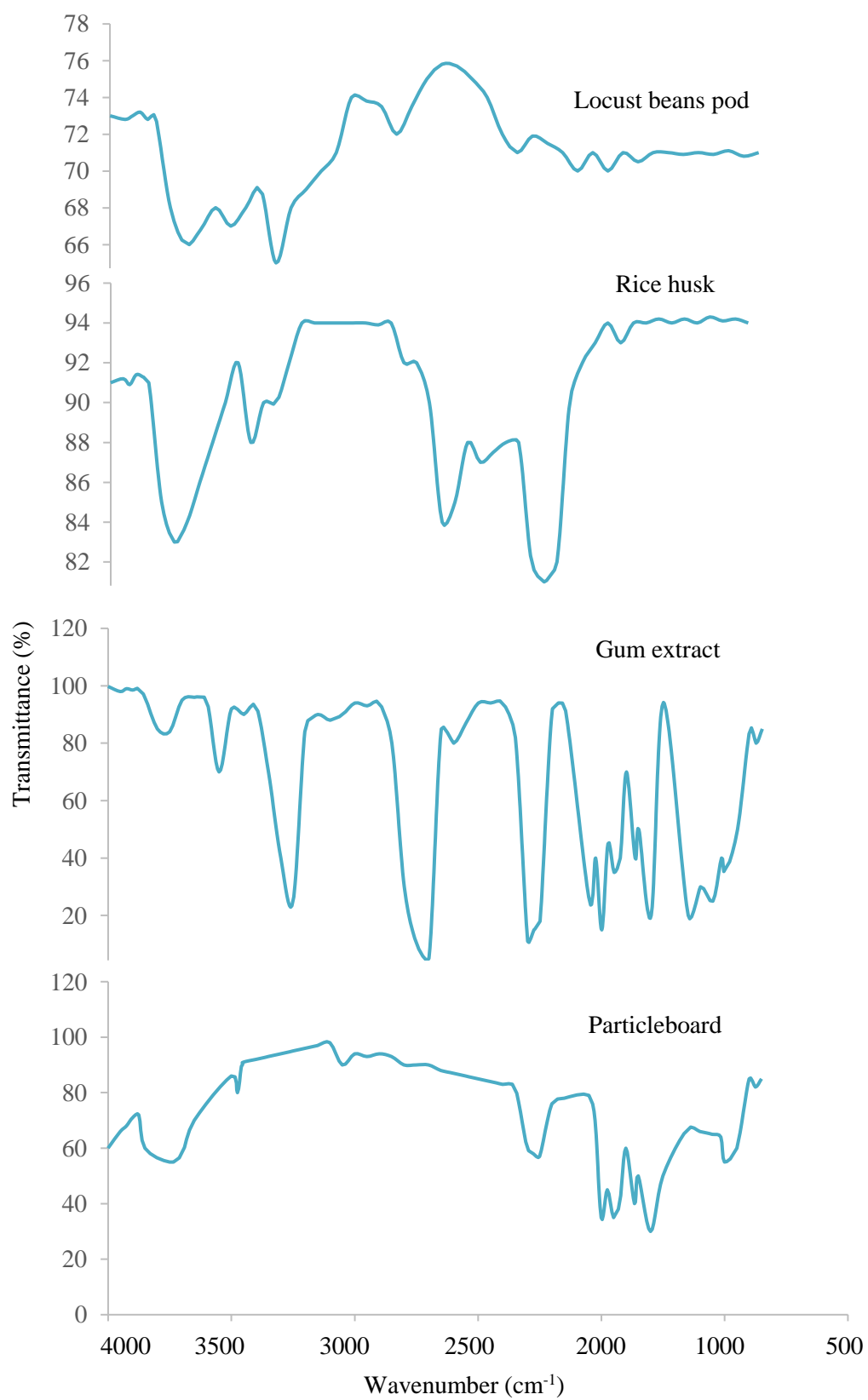


Figure 4.1: FT-IR spectrum

4.3 Analysis on the Feed Stock

Table 4.8: Proximate Analysis of rice husk

Composition	Percentage (w/w %)
Ash	26.18
Moisture	4.55
Volatile matter	52.38
Fixed Carbon	8.04
Lower heating value	8.85

Table 4.9: Proximate Analysis of locust beans pod

Composition	Percentage (w/w %)
Ash	4.68
Moisture	4.88
Volatile matter	92.38
Fixed Carbon	48.04
Lower heating value	33.85

From the particleboard produced, some of the product didn't meet up to formation standard as a result of the percentage variation for rice husk and locust beans pod in relative to the gum ratio. In the result obtained, it was observed that some of the products were found to be inappropriate while other appeared appropriate. This is as a result of the different mixing ratio, temperature applied and pressing time applied. From the result, product with high dosage of locust beans pod tends to be inappropriate at high temperature compared to others. More so, the highest number of inappropriate products were observed from the 50 -50 % mixing at low gum ratio. Appropriate and moderate products were obtained at high gum ratio, low temperature, high rice husk to locust beans pod dosage and moderate pressing time. Samples of the appropriate products were then further subjected to other mechanical and instrumental test to detect if it meets up the regulatory standard. Table 4.8 shows details of selected products for further analysis.

Table 4.10: Produced particleboard with varying process parameters

Rice Husk (wt. %)	Locust Beans Pod (wt. %)	Gum to Feed stock Ratio (wt. %)	Pressing Temperature (°C)	Pressing Time (Min.)	Product Tag
25	75	6	70	10	A
50	50	9	60	15	B
75	25	3	80	5	C
100	0	6	70	10	D
50	50	6	70	10	E
75	25	9	60	5	F
100	0	3	80	15	G
100	0	9	60	10	H
50	50	9	80	5	I
75	25	3	70	10	J
50	50	6	60	15	K
75	25	9	80	5	L
50	50	3	70	5	M
75	25	6	60	15	N
100	0	9	80	5	O
75	25	3	60	10	P
25	75	9	70	5	Q
50	50	3	60	15	R
75	25	6	80	5	S
100	0	9	70	10	T

4.3.1 Bending test

The Mechanical and physiochemical analysis were carried out on each of the samples produced Tagged A-T in order to determine their response to the applied pressure from the pressure press machine. Table 4.8 shows the optimum load bearing for samples, the highest MOR of 19.04 N/mm² which was recorded for sample J with rich husk to locust beans dosage of 75-25 %, with high gum ratio at 70 °C within a pressing time of 10 min, followed by sample N with a MOR of 18.99 N/mm² with rich husk to locust beans dosage of 75-25 %, having an average gum ratio at 60 °C within a pressing time of 10 min. More

so, sample with the least MOR of 8.16 N/mm² was recorded for sample M with a low gum ratio at 80 °C within a pressing time of 5 min. From the result, it can be seen that the gum ratio plays a very important role in the resistance to stress property of the

particleboard. As compared with literature, Azumah (2014) recorded its highest bending mean stress at a high gum ratio. More so, the effect of the pressing temperature can also be seen as higher temperature weakens the gum bonding capacity, hereby reducing its bending stress.

4.3.2 Swelling test

From Table 4.9, it shows that the optimum thickness of the sample after immersed in water were obtained for sample O and sample A, at 19.67 % and 3.33 % respectively. Research has shown that the hydrophilic nature of rice particles is higher than that of the locust beans pod, this is also confirmed from the result. Sample O with higher ratio of rice husk to locust beans pod has the highest swelling thickness percentage while samples with low ratio of rice husk to locust beans pod has a low swelling percentage. Hence, an average value of swelling thickness percentage is observed in samples with moderate ratio of rice husk to Locust Beans Pod respectively.

4.3.3 Modulus of elasticity (MOE)

Similarly, like the MOR, Table 4.9 also shows the optimum load bearing for samples, the highest MOR of 19.04 N/mm² which was recorded for sample J with rich husk to Locust beans dosage of 75-25 %, with high gum ratio at 70 °C within a pressing time of 10 min, followed by sample H with a MOR of 16.7 N/mm² having a high gum ratio at 60 °C within a pressing time of 10 min. More so, the least MOR of 8.16 N/mm² was recorded for sample M with a low gum ratio at 80 °C within a pressing time of 5 min. From the result, it can be seen that the gum ratio plays a very important role in the resistance to stress property of the particleboard. As compared with literature, Azumah (2014) recorded its highest bending mean stress at a high gum ratio. More so, the effect of the pressing temperature can also be seen as higher temperature weakens the gum bonding capacity, hereby reducing its bending stress.

Table 4.11: Mechanical properties of produced particleboard

Product Tag	Rice Husk (wt %)	Locust Beans Pod (wt %)	MOE (N/mm ²)	Swelling test (%)	MOR (N/mm ²)
A	25	75	230	3.33	10.67
B	50	50	191	6.67	8.86
C	75	25	333	13.33	15.47
D	100	0	272	16.67	12.62
E	50	50	210	10.00	9.73
F	75	25	320	6.67	14.83
G	100	0	233	13.20	10.79
H	100	0	360	17.27	16.71
I	50	50	250	7.33	11.62
J	75	25	410	12.93	19.04
K	50	50	206	5.73	9.55
L	75	25	344	12.20	15.95
M	50	50	176	7.27	8.16
N	75	25	409	12.00	18.99
O	100	0	304	19.67	14.09
P	75	25	360	12.67	16.68
Q	25	75	230	4.00	10.67
R	50	50	191	6.67	8.86
S	75	25	333	10.40	15.47
T	100	0	272	17.33	12.62

4.3.4 Moisture content test

The water content of each of the samples was determined using the equation reported by Huang *et al.*, (2004). Table 4.9 shows the moisture content of the particleboard produced. It was observed that the particleboard with the highest amount of moisture content was obtained from the particleboard containing high quantity of rice husk (100:0 %) at low temperature and pressing time. While the lowest moisture content was obtained from particleboards that contain high amount of locust beans pod (25:75 %) at high temperature and pressing time. From the result it shows the effect of temperature on the moisture

content of a sample. More so, it shows that rice husk is more hydrophilic than locust beans pod.

4.3.5 Water absorption test

The water content of each of the samples were determined using the equation reported by Huang *et al.*, (2004). Table 4.10 shows the moisture content of the particleboard produced. It was observed that the Particleboard with the highest amount of Moisture content was obtained from the particleboard containing high quantity of rice husk (100:0 %) at low temperature and pressing time. While the lowest Moisture content was obtained from particleboards that contain high amount of locust beans pod (25:75 %) at high temperature and pressing time. From the result it shows the effect of temperature on the Moisture content of a sample. More so, it shows that rice husk is more hydrophilic than Locust beans pod.

4.3.6 Particleboard density

From the standard equation for calculating the density (ρ) of a material as a ratio of its mass to the volume occupied, the density was evaluated from its uniform volume (75cm^3) and a varying mass ranging from 72-86 g respectively. Table 4.10 shows the corresponding densities of the particleboard produced. It was observed that the highest density is obtained from the particleboard containing high quantity of locust beans pod (25:75 %). The lowest density was obtained from particleboards that contain high amount of rice husk (100:0 %). This can be attributed to the reason that locust beans pod has a higher density to rice husk sample. Since density is directly proportional to mass of sample, likewise particleboards samples with higher masses has higher densities.

Table 4.12: Physical properties of produced particleboard

Product Tag	Moisture content (wt. %)	Water absorption (%)	Swelling test (%)	Density (Kg/m ³)
A	2.38	4.65	3.33	1147
B	5.26	10.00	6.67	1067
C	8.33	12.82	13.33	1040
D	11.76	13.16	16.67	1013
E	5.41	10.26	10.00	1040
F	8.57	10.53	6.67	1013
G	8.82	13.51	13.20	987
H	12.50	13.89	17.27	960
I	8.11	7.50	7.33	1067
J	8.57	10.53	12.93	1013
K	5.26	7.50	5.73	1067
L	8.82	13.51	12.20	987
M	5.41	10.26	7.27	1040
N	5.56	10.53	12.00	1013
O	8.82	16.22	19.67	987
P	8.33	7.69	12.67	1040
Q	2.38	7.14	4.00	1120
R	5.26	7.50	6.67	1067
S	8.33	10.53	10.40	1013
T	11.76	13.89	17.33	960

4.3.7 Scanning electron microscopy

The surface morphology of the Particleboard was observed using SEM, Figure 4.2 shows the scanning electron micrographs of Rice husk and Locust Beans Pod source. As seen, the surface appearance reflects the degree of compatibility of the Gum to the feed stocks. This depends on the type of Adhesive used and also be attributed to the mineral composition of the material in relative to its porosity.

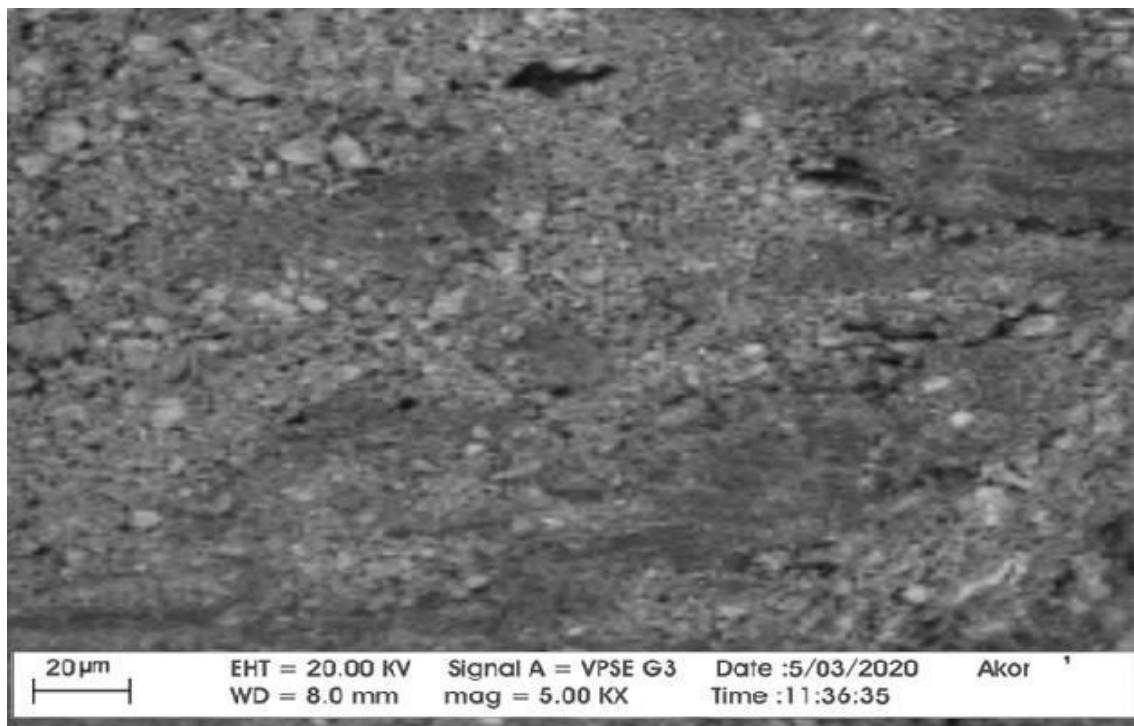


Figure 4.2: SEM morphology of particleboard from rice husk

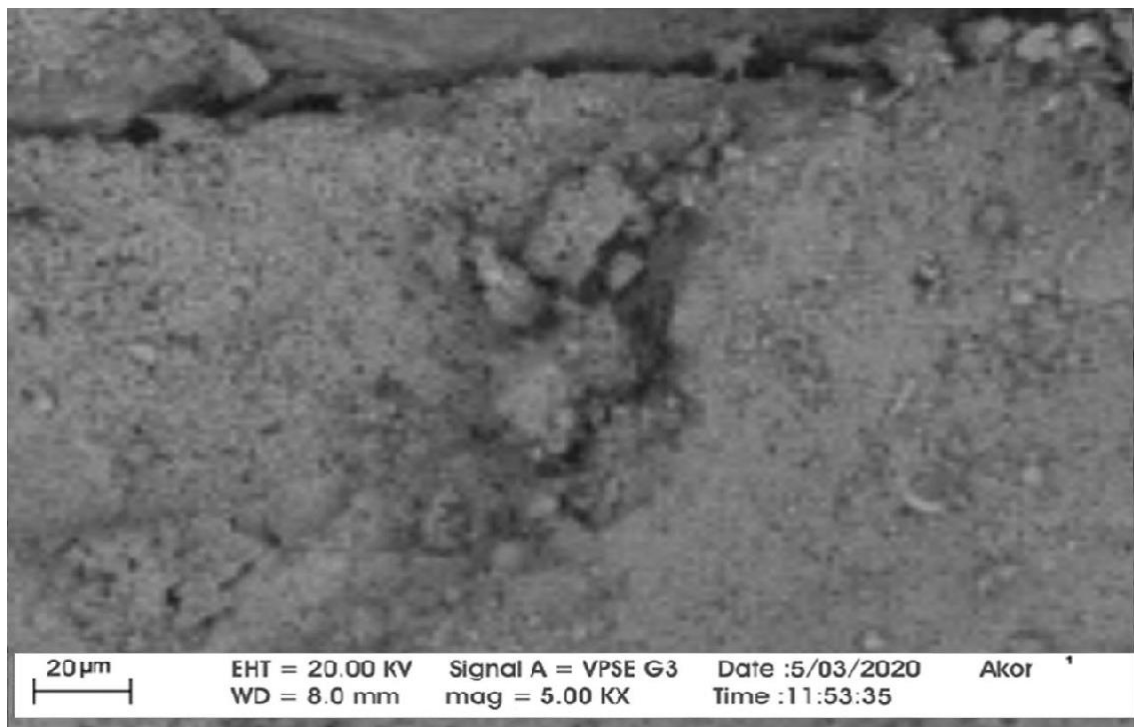


Figure 4.3: SEM morphology of particleboard from locust beans pod

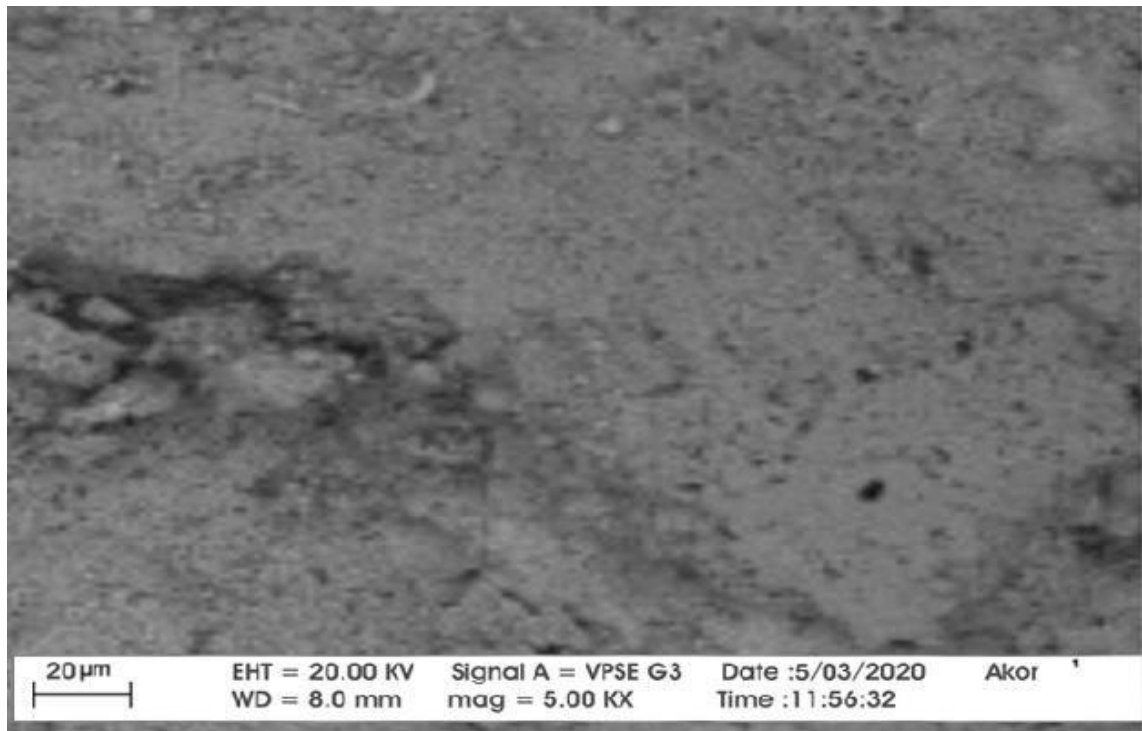


Figure 4.4: SEM morphology of particleboard from rice husk and locust beans pod

4.3.8 Termite resistance test

Termite resistivity test was carried out from the result obtained, it shows that samples with high concentration of locust beans pod had no attack of the termite while those with high amount of rice husk pod shows little signs of attack after observing for 2 months. Hence, samples with an average percentage ratio of rice husk to locust beans pod will stand the test of time in resisting the attack from termite.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In this study, the following conclusions were drawn;

1. The Gum was successfully extracted from Guill and perr (*Cissus populnea*) plant studying the effect of process parameters via RSM and the optimum yield (78 % for highest yield) was obtained at 100 °C with 40 g sample dosage at 60mins for fresh sample. While the lowest yield (66 %) was obtained at 90 °C with 50 g sample dosage at 90 min from dried sample using water as extraction solvent respectively.
2. Comparative study on the yield of the Gum obtained from different Samples (fresh and dried) shows higher percentage of gum in fresh sample to dried sample.
3. From the Characterization of the Gum using Fourier Transform Infrared Spectroscopy (FT-IR), shows the presence of carboxylic group in the substance at similar stretching with literature.
4. From the Characterization of Particleboard, it was indicated that sample J has the best MOE of 410 N/mm², MOR of 19.04 N/mm², with an average density of 1013 kg/m³ respectively.

Hence, the particleboard produced shows similar mechanical and physiochemical properties with the conventional boards and its mechanical properties met the LD-1 requirement of ANSI A208.1 Standards.

5.2 Recommendations

The following recommendation were made for further studies

1. The yield of Gum from other sources that has mucous membranes and belongs to the family of Amplidaceae (*Vitaceae*) should be investigated for comparative study.

2. Effect of other extraction solvent should also be investigated.
3. For the Particleboard production, other sources of feedstock should be used with the Gum to monitor the performance yield obtained.
4. Other methods of production can be used to check the performance of the Gum.

5.3 Contribution to Knowledge

The effects of process variables to determine the optimum conditions for producing high yield of gum extract from *Cissus populnea* was successfully studied and it was observed that for fresh sample using ethyl acetate solvent, an optimum yield of 78 % was obtained at 100 °C with 40 g sample dosage at 60 minutes. While from dried sample using water as extraction solvent, the lowest yield of 66 % was obtained at 90 °C with 50 g sample dosage at 90 minutes respectively.

Application of adhesive extracted from *Cissus populnea* in particleboard production using rice husk and locust-beans pod as stock feed was successfully studied and it was observed that from the various particleboards produced, the result from sample tagged J with 1:3 adhesive to feedstock ratio (wt. %) at 70°C within a pressing time of 10 minutes with rich husk to locust beans dosage of 75-25 % respectively.

Characterization and comparative study on the particleboard produced to checkmate the durability and ability to withstand the environmental conditions shows an optimum Modulus of Elasticity of 410 N/mm², Modulus of Rupture as 19.04 N/mm² and with an average board density of 1013kg/m³ respectively.

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