## MACHINE PARAMETERS FOR KENAF PELLET PRODUCTION FOR REMEDIATION OF CRUDE OIL POLLUTED WATER BODIES

BY

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### **CERTIFICATION**

This is to certify that this study was carried out by Kadiri Azeez Oluwaseun with matriculation number 162287 in the Department of Agricultural and Environmental Engineering, Faculty of Technology, University of Ibadan, Nigeria.

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## **DEDICATION**

This work is dedicated to God Almighty, the all-knowing and sustainer of all.

and

The Love of humanity (to those who went all the way to help others without requesting any compensation)

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### **ABSTRACT**

Remediation of crude petroleum polluted water for agricultural activities is a challenge. Kenaf has been found to have potentials for remediation and pelletising it will improve handling and recovery. Literature is sparse on the appropriate operating parameters for kenaf pelleting machine. This study was designed to investigate the effects of Screw Pitch (SP), Speed of Rotation (SR), and Die Diameter (DD), on kenaf pellets properties suitable for crude oil spill remediation.

A pelleting machine was designed for kenaf using standard procedures. The machine was evaluated using response surface methodology. The variables were SP (40, 50, 60, 70 and 80 mm), SR (40, 50, 60, 70 and 80 rpm) and DD (27.5, 30.0, 32.5, 35.0 and 37.5 mm). Thirty experimental combinations were generated from the variables to get responses on machine efficiency (Pelleting Efficiency (PE) and Percentage Recovery (PR)), pellets' mechanical properties (Force, Deflection, Energy, Young modulus and Durability Index, (DI)) and remediation potentials (oil recovered and changes in pH). At different combinations, the machine was used to pelletise samples of kenaf, starch and water (1:1-2:3) mixture. Machine efficiency and mechanical properties were determined using ASABE and Universal Testing Machine. The oil recovered was determined by comparing oil contents before and after remediation using a spectrophotometer according to AOAC standards, while changes in pH were determined by comparing pH values before and after remediation. The process was simulated and optimised using artificial neural network and its accuracy determined using mean square error (MSE) and coefficient of determination ( $R^2$ ). Data were analysed using ANOVA at  $\alpha_{0.05}$ .

The PE and PR ranged 82.3-95.8% and 68.4-88.3%, respectively. The PE and PR increased with increase in DD and SR. Forces at peak, yield and break ranged 84-280, 108-342 and 142-504 N, respectively. Deflection at peak and break were 2.01-5.48 and 3.89-10.24 mm, respectively. Minimal Energy to peak, yield and break were 4.93, 6.75 and 9.42 Nmm/s, respectively. The Young's modulus and DI were 0.03 N/mm² and 98.9 %, respectively. The SP, DD and SR significantly affected the mechanical properties. Oil recovery ranged between 97.6 % and 99.8 %, while changes in pH ranged 0.01-0.22, signifying the occurrence of remediation. Increase in SP and DD yielded an increment in oil removal and increased changes in pH, while a rise in SR led to a reduction in quantity of oil removed and an increase in the changes in pH. The MSE and R² of the models varied between 0.0003 and 125545469.3; 0.2589 and 0.9978, respectively. The optimal operating conditions for the production of kenaf pellets for remediation were 54 mm SP, 68 rpm SR and 34 mm DD.

Kenaf pellets were good absorbents for remediation of crude oil polluted water bodies. Optimal conditions for the production of durable kenaf pellets for the effective remediation of crude petroleum polluted water bodies were established. An efficient pelleting machine for the production of kenaf pellets was developed.

**Keywords:** Kenaf, Polluted water, Remediation, Kenaf pelletising, Screw pitch

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### LIST OF ABBREVIATIONS

AML Acute Myeloid Leukaemia

ANN Artificial Neural Network

ANOVA Analysis of Variance

AOAC Association of Official Analytical Chemists

ASAE American Society of Agricultural and Biological

/ASABE Engineers

BR Starch/kenaf ratio

BTEX Benzene, Toluene, EthylBenzene and Xylene

CT Computational Time

DD Die Diameter

DI Durability Index

DPR Department of Petroleum Resources

IAR&T Institute of Agricultural Research and Training

MAD Mean Absolute Deviation

MAPE Mean Absolute Percentage Error

MSE Means Square Error

NDDC Niger Delta Development Commission

NEPC National Environment Protection Council

NMA Nigerian Maritime Agency

NOSDRA National Oil Spill Detection and Response Agency

OTA Office of Technology Assessment

PAHs Polycyclic aromatic hydrocarbons

PE Pelleting Efficiency

PR Percentage Recovery

PRB Permeable Reactive Barriers PRB

R<sup>2</sup> Coefficient of Determination

RCoV Relative Coefficient of Variation

rMBE Relative Mean Bias Error

RMSE Root Mean Square Error

RPM Revolution Per Minute

RSM Response Surface Design

SFE Supercritical Fluid Extraction

SP Screw Pitch

SR Speed of Rotation/ Pelleting Speed

UTM Universal Testing Machine

WHO World Health Organization

# CHAPTER ONE INTRODUCTION

### 1.1 Background of Study

Agricultural machines have over the millennia been used to enhance agricultural operations. Depending on their categories, they are used on farms to improve production, soil and site preparation, enhance farm operations such as dispersal of chemicals and manures, precision farming, harvesting and processing in order to increase the value of the agricultural products. In some cases, they are used to increase the shelf life of agricultural produce which are generally perishable (Srivastava *et al.*, 2006).

Existing designs of machines are subject of various research studies for various reasons. Such researches may seek to suggest modifications for machine performance and efficient management of energy consumption, determine best operating conditions for the machines, analyse effects of variations in parameters (operating, machine and material) on the machines' performance, review of ergonomic properties.

The screw type pelleting machine was initially designed to mould grounded kenaf fibres into pellets which can be easily handled and utilized for the intended purposes, which according to preliminary studies (Kadiri, 2014) was for controlling the effects of oil spills via absorption of the spilled crude oil.

Oil spillage has over decades been a source of environmental concern and will continue to occur due human activities and equipment failure in the excavation, transportation and storage of petroleum products (Fingas, 2001). The type of crude oil, weather condition and strength of sea are main factors that determines the rate of spread of spill.

Crude oil spillage in Nigeria is unavertable as the nation's dependence of crude oil which despite its utilization in various industries, its exploration has brought untold hardship to the life of the rural communities around which it is being discovered (Usman *et al.*, 2015). It is often referred to as black gold and is a major source of income, energy resources,

employment as well as international recognition to countries endowed by it, this makes it the most dependable resource in the world (Tugenhat and Hamillton, 1975; Akinlo, 2012; Kadiri, 2014).

Despite its high level of significance and utility, it has its own disadvantages as it is capable of rendering the environment inhabitable in the form of oil spills, gas flaring, land degradation, noise pollution, soil fertility loss, fire outbreaks, water pollution and other forms of environmental hazards (BOSR, 2008; Kadafa *et al.*, 2012; Amnesty International, 2013; Kadiri, 2014). Oil spillage is a major form of environmental pollution and defined as the discharge of liquid petroleum into the environment due to human activities Adelana *et al.*, 2011). However, oil spillage remediation and management techniques have been initiated and utilized over the years to reduce the undesirous effects of oil spills, make the affected environments ecological habitable as well as reduce the cost associated damages in oil affected regions.

Neglect of oil spill and poor allocation of resources to the affected regions such as the Niger delta, has over the years been the reason for disputes, armed conflicts as well as crime rate increment in countries producing crude oil. This can only be minimized effectively through management of oil spills as well as efficient bioremediation of the affected areas.

Although, there are different policies put in place to tackle and minimize the effects of oil spill through the use of its agencies such as Niger Delta Development Commission (NDDC), Department of Petroleum Resources (DPR), Federal and State Ministries of Environment, National Oil Spill Detection and Response Agency (NOSDRA) and Nigerian Maritime Agency (NMA) (Adelana *et al.*, 2011; Amnesty International, 2013), poor attitude to cleaning up and control of oil spills in the regions are still discouraging the residents in the affected regions.

Oil spillage in Nigeria takes two major forms, oil spills on land and oil spills on water bodies, the former can be reasonably curtailed as its rate of spread is not as ferocious as the later. Oil spill on water bodies is capable of reaching a distance of 150 km after 104 hours in wet seasons and a similar distance in 162 hours during dry season, thus is obtained using a simulation model according to Nwilo and Badejo, (2008).

Oil spilled on water can be curtailed through a number of ways, including mechanical, chemical (gelling, chemical dispersion e.g., use of detergents, sinking), natural means (wind dispersal, evaporation, as well as photochemical oxidation), controlled burning (combustion), absorbent materials such as agricultural fibres (wool, jute, kenaf etc.) (ASAE, 2010), artificial fibres (polypropylene, sol-gels) and the use of bioremediation (oil eating bacteria) (US Congress OTA, 1991; Chol and Cloud, 1992; Cai *et al.*, 2010; Karan *et al.*, 2010; Adelana *et al.*, 2011; Dave and Ghaly, 2011; David and Joel, 2013; Kadiri, 2014; Walther, 2014).

There is a need to remediate oil spilled water bodies using cheap, available, natural, environmentally-friendly, efficient and effective means, which will include the use of natural fibres. One of the sources of natural available fibre being used for the environmental remediation of oil contaminated bodies is the kenaf plant, the fibres are used for cordage, making ropes as well as paper making (Robinson, 1988; LeMahieu *et al.*, 1991). According to Dyke *et al.*, 2010, the long fibres are capable of absorbing oil. Also, further studies (Kadiri, 2014) have showed that the fibres from the stem are capable of absorbing petroleum.

Although the wool as an absorbent is also effective, the cost of wool production coupled with its competitive use as a clothing raw material and the fact that it would be ineffective as a proper combustion material, implies it use is limited when compared to the use of kenaf. The use of chemical dispersants is also common but the residual effects of its use as well as the cost of acquiring the chemicals may be too high while burning will produce acidic gases.

### 1.2 Problem Statement

The use of dried harvested kenaf as a sorbent material for the environmental remediation of oil spilled water bodies comes with advantages, some of which includes; its natural availability, the ability to recover some of the oil absorbed as well as its use as a biofuel after the absorption and oil recovery. However, its utilization for remediation requires processing to improve its handling, dispersion as well as absorption of crude oil. Mustapha (2014) and Kadiri (2014) designed and fabricated machines for processing decorticated kenaf stems. Kenaf stem were milled into granules using a plate mill (Mustapha, 2014)

while pellets were produced from granulated kenaf fibres. This was done to achieve easier handling, dispersion as well as recovery of kenaf after use. Nevertheless, the machine currently needs to be modified as it got stuck frequently. Corn starch and cissus bonded pellets also had low durability while the oil absorption of the animal feed bonded pellets was relatively low; it requires an inlet chute (hopper) and the pelleting process needs to be optimized (Kadiri, 2014).

### 1.3 Justification of Study

The optimization of the pelleting process for kenaf bonded pellets would aid in improving the production of durable pellets for use in oil polluted water bodies. This will be a major boost in the clean-up of polluted regions like the Niger Delta where oil spillage has made ecological existence of some organisms increasingly difficult. Effective durable oil absorbing pellets can easily be used and gathered. The absorbed oil in the gathered pellets can then be squeezed out and the pellets reused or combusted as fuel.

### 1.4 Objectives of the Study

The main objective is to improve the process of pelleting kenaf. The specific objectives will include:

- design and fabricate a screw type pelletizer;
- evaluate the screw type pelletizer;
- optimize and determine the effects of variations in machine and operating parameters such as kenaf-starch ratio, pelleting speed, die diameter and the screw pitch on the process of pelleting kenaf using Response Surface Methodology;
- determine the mechanical properties and remediation ability of the pellets formed using the varied parameters; and
- develop mathematical models associating pelleting speed, die diameter, screw pitch and kenaf-starch proportion used on the machine performance and Mechanical Properties and remediation potentials of the pellets using Response Surface Methodology.

### 1.5 Scope of Study

A screw type pelletizer was designed, modified and fabricated to allow for variations in machine parameters. Kenaf pellets were produced from dried harvested Kenaf stems. This study explored the pelleting process of Kenaf stems taking into consideration the effect(s) the variations in machine and operating parameters had on the performance of the pelletizer (pelleting efficiency and percentage recovery) as well as the mechanical and remediation properties of the produced pellets.

The machine and operating parameters varied include the pelleting speed, Die Diameter, screw pitch, and quantity of binder added. Models associating performance of the pelletizer as well as mechanical and remediation properties of the pellets with the varied machine parameters was established.

## CHAPTER TWO LITERATURE REVIEW

### 2.1. Kenaf (Hibiscus cannabinus)

Kenaf is a multipurpose shrub capable of reaching maturity in 4-5 months, it is an excellent source of cordage (used for ropes, sacks, twines etc.), canvas, paper making, source of food, source of edible oils, used in composite manufacture and used in the process of cleaning up oil spills from water bodies (Dempsey, 1975; Princen, 1982; Seale *et al.*, 1996; Han *et al.*, 1999; Webber *et al.*, 2002; Euchora, 2004; Bioenergy Crops, 2012; Alexopoulou, 2013; AbdulHalip *et al.*, 2014; Kadiri, 2014 and Millogo *et al.*, 2015) Although the time of origination of the kenaf is unknown, the origin of kenaf has been traced to the Eastern Central African (Tanzania, Kenya, Uganda, Rwanda) region where kenaf and similar crops from the same family such as okra and cotton are still grown in the wild (Princen, 1982; LeMahieu *et al.*, 1991; *Vision Paper*, 1998).

The kenaf plant belongs to *Malvaceae* family, the same family of okra, cotton and the hollyhock. The botanical name, *Hibiscus cannabinus* L. for kenaf was coined from its distinctive hibiscus flower and its exhibition of split of split leaves similar to the cannabis plant (Princen, 1982; AMRC, 2002; and Alexopoulou *et al.*, 2013). The plant has over 200 known varieties and has been found to easily adapt in different conditions based on the varieties, it is also described a lover of tropical and temperate region (Tahir *et al.*, 2015).

The kenaf plant is capable of reaching 8-14 feet and in certain conditions reach 20 feet (LeMahieu *et al.*, 1991; Rowell and Stout, 1998; Bioenergy Crops, 2012; Kadiri, 2014). It is capable of yielding between 10-20 tonnes per hectare of dry fibre annually. The harvested stem contains three distinct layers; the bast which is about 35-40% of the total mass of the stem consists long fibres usually utilized for cordage and woven for products such as rugs, mats etc. (Robinson 1988), the core (rest of the stem mass) usually consisting of shorter fibres and is the main part of the stem which is used for making composites, oil absorption,

animal beddings and the pith usually whitish in colour (Alexopoulou *et al.*, 2013; Lips and van Dam, 2013).

The plant is also said to be medicinal for fever, nausea and bruises as it contains saponins, phospholipids, tocopherol, phytosterols and polyphenolics. It is also used as an additive for food (Mohammed *et al.*, 1995, Alexopoulou *et al.*, 2013). It is portrayed in Plates 2.1 and 2.2.

### 2.2. Kenaf Processing

Due to the multipurpose nature of the kenaf plant several processing equipment can be used for the various parts of the plant. The stem fibres which are primary raw materials for the production sacks can be removed by retting, The oil contained in the seeds can be extracted using extraction. The stems can be decorticated to separate the bast from the core and pith and; the core can be reduced to particles using size reduction mills as depicted in Plate 2.3 (Kadiri, 2014). These reduced particles can be used for absorption of chemical and oil, production of composites, gasification, pyrolysis and other purposes (Alexopoulou *et al.*, 2013). Kenaf oil and milk are sourced from milled kenaf seeds. Kenaf milk is produced by using a combination of cleaning, hydration, milling, filtration, homogenization and pasteurization (Karim *et al.*, 2020). Kenaf oil is extracted using Supercritical Fluid Extraction (SFE) and can be consumed as edible oil or used in the production of resins and other chemicals (Mariod *et al.* 2017). The whole plant is also dried, crushed and pelleted to be used as animal feed as well as biomass for energy production (Liang *et al.*, 2003; Basri *et al.*, 2014; Kayembe, 2015)



Plate 2.1: Kenaf Plants at the Faculty of Agriculture.

Source: Kadiri, 2014



Plate 2.2: Dried Harvested Kenaf stem.

Source: Kadiri, 2014

Plate 2.3: Ground Dried Kenaf Stem (Core & Pith).

(Source: Kadiri, 2014)

### 2.3. Kenaf Pelleting

The process of producing kenaf pellets is necessary for improving handling. Pelleting can be done using three basic different mechanisms as described below by Saravacos and Kostaropoulos (2002), Pietsch (2005) and BEPEX (2011)

- I. Gear Pelletizers: As described by Saravacos and Kostaropoulos (2002) and BEPEX (2011), and shown in Figure 2.1a (III), they are also known as the Schueler pellet press, and are equipped with conical hoppers which installed directly above the gear mechanism. This feature enables the materials to fall freely towards two counterrotating gears or two gears, one rotating and the other being stationary with already bored nozzles. These gear-bored nozzles serve as dies. The gear pelletizer is used notably for production of dust-free uniform, highly durable cylindrical pellets. However, the gear and components are subject to continuous wear thus requiring constant replacement which makes it relatively expensive to operate.
- II. Rotary Pelletizers: Rotary pelletizers are commonly used for the production of pellets as depicted in Figure 2.1a (I and II). It is however of two types; the flat-die type and the ring-die mills. The material is fed from the top in both cases, but in the former as described by Hasting and Higgs (1980), Obernberger and Thek (2010), Romallosa and Cabarles (2011) and BEPEX (2011), the extrudates are pushed vertically downwards through a flat plate equipped with the required opening. The ring-die type according to Obernberger and Thek (2010), on the other expels the materials by pressing it against the walls of the cylindrical casing.
- III. Screw Type Pelletizers: They have a wide range of applications. According to Huber (2000), Morad *et al.*, (2007), BEPEX (2011), Sakai (2013), Oduntan *et al.*, (2014) and Muhammad *et al.*, (2016), its main components include a prime mover, a barrel (comprising a feeding zone and a kneading zone), a speed reduction mechanism, a die (shape forming) and sometimes a cutter and a heating panel as depicted in Figure 2.1b. Screw pelletizers are classified into two main categories, which are the single screw and the twin-screw pelletizers. The former can be further reclassified based on design into solid single screw extruder, interrupted flight extruder-expanders and single segmented-screw extruders (Mian 2000) while the latter can be reclassified into co-rotating and counter-rotating twin screw Extruders.

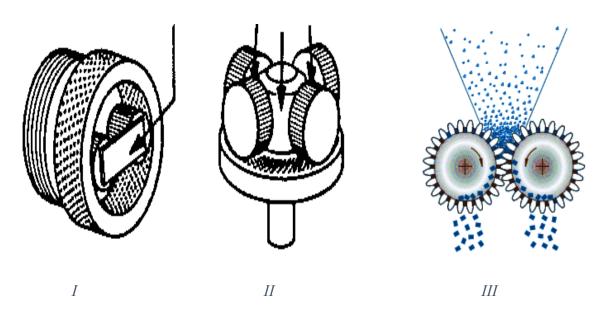


Figure 2.1a: Principle of Operation of Rotary and Gear Pelletizers (I is the Ring-Die pelletizer, II is Flat Die Pelletizer while III is the gear pelletizer)

Sources: Obernberger and Thek (2010) and BEPEX (2011)

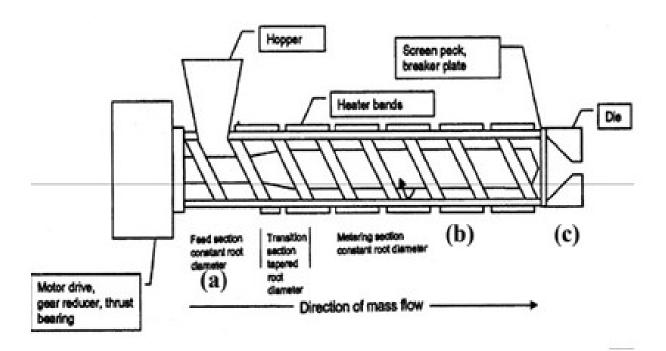


Figure 2.1b: Schematic Representation of a Single Screw Extruder showing the Feed Section (a), Metering Section (b) and the Die Section (c).

Source: Yamsaengsung and Noomuang (2010)

The factors affecting pelletizing (Malinowski and Smith, 1975; Hasznos *et al.*, 1992; Sonaglio *et al.*, 1995; Saravacos and Kostaropoulos, 2002) include:

- Resistance in pushing the materials through the die openings
- Residence time of the materials within the pelletizer
- Pressure exerted by the pelletizer.
- Additional process required for pelleting
- Moisture content of the material to be pelletized.
- Binder used in the pelleting process.
- Configuration of the pelletizer.

**Resistance of materials:** The resistance of the materials being pushed through the die opening is an important criterion in pelleting as it highlights the flowability of the extrudate. It depends majorly on the particle size, nature of the extrudate, binder utilized, loading rate of the extrudate as well as the moisture content of the extrudate. This is because negligence of this factor can lead to clogging, poor pellet formation and poor pellet quality.

Resident time/space within the pelletizer: This is another imminent factor affecting extrusion and is solely based on the pelletizer's configuration. A longer time/space denotes a higher tendency of extrudate loss thus implying a reduction in percentage recovery although there is also a larger exposure to heat via friction between the shaft and barrel which might also tend to increase pelleting efficiency. The space between the shaft and the die should likewise not be too much, this is essential to reduce the amount of extrudates left in the pelletizer after operation.

Kenaf is reported to be pelleted for use as animal feed (Phillips, 2012) as well as for oil absorption from water bodies (Kadiri, 2014). The use of cissus, corn starch and animal feed as binders for kenaf stem is also reported, alongside its effects on the durability and oil absorptivity of the kenaf pellets in Kadiri, 2014. Figure 2.3 depicts the procedures in the pelleting of kenaf.

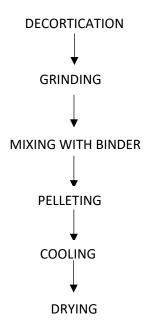


Figure 2.3: Processes Involved in producing Kenaf Pellets

**Pelletising pressure:** The extruding pressure is another significant factor in extrusion, it is a measure of the interaction between the torque of the prime mover as well as the orifice in the die. It is also pertinent to note that the torque is an affiliation of the power generated by the prime mover and its angular speed (revolution/minutes) as expressed mathematically in equation (2.I). This thus implies that the extruding pressure is affected by the speed of the prime mover, the power rating generated by the prime mover and the size of the orifice on the die.

Torque 
$$T = \frac{9.5488 \times P(Kw)}{S(rpm)}$$
 2.I
$$P = \frac{T}{A}$$

Thus the pressure of extrusion can be obtained by combining both equations above into equation (2.2) below

Torque 
$$T = \frac{9.5488 \times P(Kw)}{A(m^2) \times S(rpm)}$$
 2.2

Where P is the Power generated by the prime mover (in Kilowatts)

A is the size of the die orifice (in metres squared)

But

S is the speed of the electric motor (in revolutions per minutes)

Additional processes required: Some other processes may be added to aid the extrusion process, this depends on the desired products and process. For instance, heat bands are used to generate additional heat necessary for reshaping polyethylene materials as well as formation of metallic ore pellets, likewise the water and other coolants are added as lubricants in heavy-duty extruders, while the use of cutters at the end of the pelletizers have also been utilized particularly for the production fish feeds.

**Extrudate's moisture content:**. The moisture content of the extrudate is a prominent factor that requires preliminary investigations and experimentations, this is because excess moisture can lead to slurry, half-shaped and weak pellets while inadequate moisture led to broken pellets and clogging and subsequently wastage of extrudates.

**Binder:** the nature and amount of the binder is one of the commonly overlooked factors. Binders are required to be adhesive but it is also necessary that the binders should aid the flowability of the extrudates while being not too adhesive to the barrel, shaft and cone of the extruders. This is majorly to reduce the losses due to extrudate retention within the

extruder. This implies that binder selection also requires preliminary trials and experimentations and varies with the nature of extrudates. For instance, the use of sticky and adhesive binders such as lignin and cissus gum would work for soft fibrous materials like sawdust.

**Extruder configuration:** This is another factor that depends on the purpose of extrusion as well as the nature of the extrudates. The extruder's configuration simply comprises the type of extruder shaft (twin screw (counter rotating or same direction) or single screw (see Figure 2.4)), types of barrels (as shown in Figure 2.5) (Rokey, 2000)

### 2.3.1. Selection of the Desired Pelleting Machine

The pelleting mechanisms available locally are of three; the screw-type, the gear type as well as the rotary pelleting machine. However, the screw type pelletizer is preferred for this study because of its technical and economic advantages, some of which include production of variable sizes of pellets at cheaper cost, stable and durable pellet production as well as less wear and tear to the machine. Figure 2.6 portrays an autoCAD schematic diagram for an extruder

Saravacos and Kostaropoulos (2002) stated that the selection of the extruder should involve some economic and technical criteria. These criteria are listed below.

Economic considerations in the selection of the pelleting machine. These are enumerated below

- The cost of the screw pelletizer is consistent with its quality and capacity.
- Binders and other additives must be affordable and readily available.
- Energy consumed when compared to the production capacity must be low.
- Wear and tear of the machine components must be low.
- Component part of the machine must be cheap and easily replaceable.

Technical considerations in the selection of the pelleting machine. These are enumerated below –

- Noise level should be kept below 85 Decibels (85db).
- Machine must be hermetically sealed where the major material is powdery.
- Capacity of the machine must be coherent with the required type of processing.

- Desired product quality must be attained.
- Foundation must be able to withstand machine vibrations.

According to and Rokey (2000) and Firdaus *et al.*, (2017), there exists different screw and barrel configurations which include constant pitch, constant root diameter configuration; constant pitch, variable root diameter configuration as well as variable pitch, variable root diameter configuration, as depicted in Figure 2.4, while Figure 2.5 shows the available barrel types used commonly in the extruding operations.

The study made use of the constant pitch, constant root diameter configuration fitted in a smooth bore barrel all made from mild steel. The die would be extruded to aid formation of pellets.

### 2.3.2. Binder Selection

The binders are used to increase the bonding strength of the pellets. It is also desirable to use organic naturally existing binding agents to ensure that environmentally friendly pellets are produced. Naturally existing edible gums and mucilage such as Terminalia gum obtained from the incised trunk of, Terminalia randii (Combretaceae), tragacanth gum from stems of Astragalus gummifer (Leguminoseae), corn starch obtained from grains of Zea mays, cassava starch obtained from the tubers of Manihot esculenta, Corn fibre gum, Xantham gum obtained from fermenting the bacteria Xanthamonas campestetris, Mango gum extracted from the stem of Mango (Magnifera indica) tree of the Anacardiaceae family, Lignosulphonates, Cissus gum obtained from stem of Cissus populnea (Ampelidaceae), Mucuna gum obtained from the cotyledon of Mucuna flagillepes (Papilionaceae) Grewia gum from the inner bark of Grewia mollis (Tillaceae), Flaxseed gum obtained from the hulls of flaxseed, Cordia mucilage obtained from the raw fruits of Cordia obliqua (Boraginaceae), waterleaf mucilage obtained from the leaves of Talinum Triangulare, mucilage from Sida acuta (Malvaceae), Mucilage from Corchorus Olitorious leaves, Potato starch and Potato peel residue, Detarium gum obtained from the cotyledons of the seed of *Detarium microcarpum (Caesalpinioideae*) amongst others (Sivak and Preiss, 1998; Yamakazi et al., 2008; Okore, 2009; Qian et al., 2010; Saha and Bhattacharya, 2010, Sivakumar et al., 2010; Adeleye et al., 2011 Kuokkanen et al., 2011; Oladipo and Nwokocha, 2011; Fernandez and Banu, 2012; Foster and Mitchell, 2012; Soladoye and

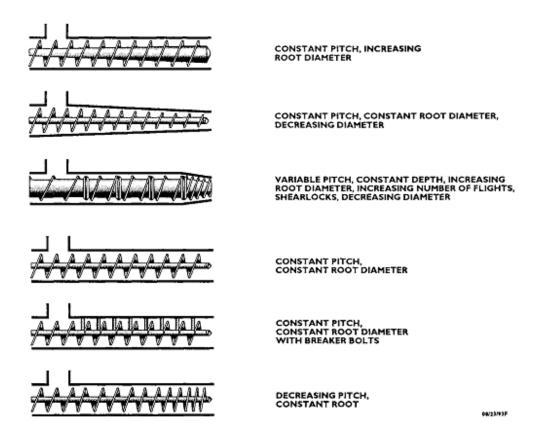


Figure 2.4: Some Possible Barrel Screw Combination for the Single Screw Extruder.

Source: Rokey (2000)

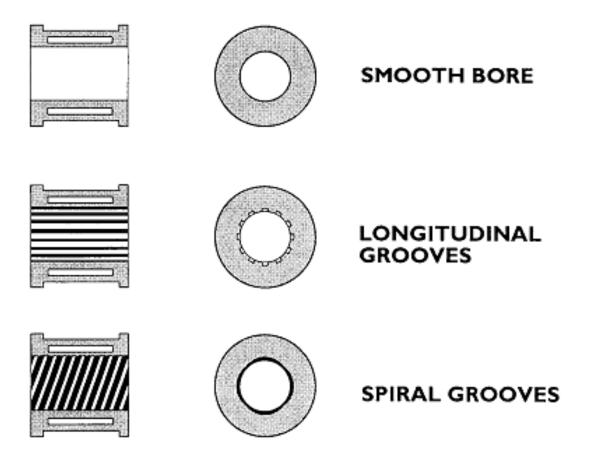


Figure 2.5: Types of Barrels used in Extruding Process

Source: Rokey (2000)

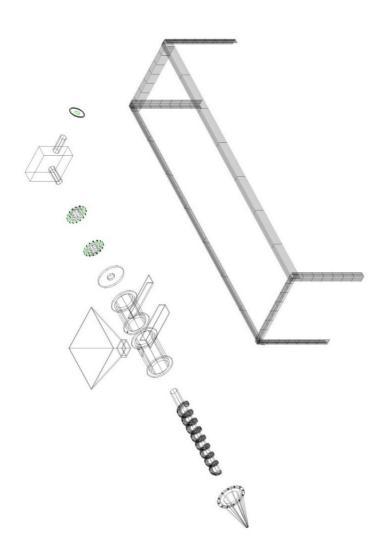


Figure 2.6: AutoCAD Schematic Drawing of the Expected Screw-Type Pelleting Machine

Chukwuma, 2012; Adetuyi and Dada, 2014; Aguoru *et al.*, 2014; Choundary and Pawar, 2014; Kadiri, 2014; Ordu and Chukwu, 2015; Olorunnisola and Asimiyu, 2016).

The choice selection of binder for this study should therefore exhibit strong adhesive properties, cheap, readily available as well as being biodegradable, thus, cassava starch obtained as the bye product from the runoff water from a cassava processing centre can be considered as it is readily available as cassava is being processed on daily basis.

### 2.3.3. Pellet Optimization

Pellet optimization is an important aspect of the pelleting process as it measures the functionality of the pellets as it relates to its purpose. However, optimization techniques and parameters vary and the main purpose is to determine if certain parameters would significantly affect the utilization of these aggregates. Aggregates have been found to be assessed on durability, dissolution, energy conversion, strength and uniformity, reduced harmful emission etc. (Raji *et al.*, 2008; Holubcik *et al.*, 2012; Vigants *et al.*, 2017; Bartocci *et al.*, 2018; Lang *et al.*, 2020; Mazumder *et al.*, 2021; Liu *et al.*, 2023)

Optimization of the Kenaf pelleting process is also paramount to determine the best parameters in respect to Kenaf pelleting. Changes in the parameters are associated with certain effects on the machine performance and the properties of pellets in terms of the mechanical properties, bulk density and absorption capacity of the pellets. These parameters include Die Diameter and geometry, pelleting speed, screw pitch, quantity of binder used, types of binder used, particle size, die pressure, moisture content, die temperature etc. (Shaw, 2008; Duncan, 2010; Lee *et al.*, 2013; Akdeniz and Shishvan, 2015; Labbé *et al.*, 2020).

This study seeks to provide an insight in respect to optimizing Kenaf processing although similar work has been done on wood pellets (Holubcik *et al.*, 2012; Ahn *et al.*, 2014), animal feed (Wood, 1987; Vukmirović *et al.*, 2017), water hyacinth (Davies and Davies, 2013) biomass and agricultural waste (Shaw, 2008), corn stover (Tumuluru, 2014; Djatkov *et al.*, 2018), Olive tree pruned residues (Carone *et al.*, 2011) Agro-Industrial by-products (Jan *et al.*, 2016) and some other composite materials (Forero-Nuñez *et al.*, 2015),

#### 2.3.4. Pelletizer Evaluation

Evaluation of the pelletizer is a paramount assessment of the pelletizer based on desired objectives. These objectives vary and are subjected to changes based on the machine and operating parameters used for pelleting. Pelletizers can be evaluated based on production capacity, pelleting efficiency, specific energy consumption, production costs, non-pelleting loss (percentage recovery), etc. (Abo-Habaga *et al.* 2017; Ikubanni *et al.*, 2019; Malgwi *et al.*, 2020).

Pelleting efficiency and percentage recovery as prescribed by Ugoamadi (2012), Kadiri (2014), and Oduntan *et al.*, (2014) are basic assessment standards for the evaluation of the pelletizer. The pelleting capacity is the mass of pellets produced per unit time usually measured in kilograms per hour. The pelleting efficiency is the percentage of pellets produced in mass with respect to the mass of output while the percentage recovery is the proportion of output to the extrudate introduced into the pelletizer. Similar work has also been done on fish feed pelletizers (Davies and Davies, 2011; Ojo *et al.*, 2014), sludge (Sial, *et al.*, 2007) and sugarcane bagasse (Aloria *et al.*, 2017).

#### 2.3.5. Pellets Evaluation

Evaluation of the pellets based on the mechanical properties as well as the remediation ability is a very important aspect as it forms a basis for the assessment of the kenaf pellets for use as absorbents for remediation in remediation of oil polluted water bodies. These mechanical properties are indications of how the pellets would behave when exposed to load while in a static position. These values are very important when determining stacking height and the choice of packaging materials during storage.

The durability of the pellets is an indicator of the pellets' reaction would be when exposed to dynamic loads. It is a measure of the pellets resistance to wear and tear when subjected to motion. It is a paramount factor in choosing packaging materials and fragility classification during handling and transportation. Mechanical properties have always been determined using the universal testing machine where the material to be tested is subjected to compressive and tensile forces along vertical and horizontal axis to determine the points of change in the structure of its structure. While the durability of pellets can be done in quite

a number of ways including tumbling, exposure to pneumatic force as well as rotary force (Abdulmumini *et al.*, 2020; Graham *et al.*, 2017; Obernberger and Thek, 2010; Temmerman *et al.*, 2006). Pellets with high strength and durability can reduce losses during handling and transportation (Peng *et al.*, 2013).

Similar work has been done on Poppy waste (Kazimirova *et al.*, 2017), Wood pellets (Jonsson, 2009; Duncan, 2010; Oveisi-Fordiie, 2011; Ståhl *et al.*, 2012; Jezerska *et al.*, 2014), Organic manure, and biomass (Nováková and Brožek, 2008; Tenorio *et al.*; 2015 and Gaitán-Alvarez *et al.*, 2017)

The intended use for the pellets for the remediation of oil spilled water bodies will also necessitate the evaluation of Kenaf pellets based on the remediation ability when in contact with both water and oily hydrocarbons (petroleum). This is achievable by determining certain criteria which may include quantity of BTEX compounds removed by the pellets, the amount of hydrocarbon/oil removed by the pellets, the changes in the pH as well as changes in the turbidity of the samples before and after using the Kenaf pellets.

Similar works of remediation have been carried out on Modified Ostrich bone waste (Shakeri *et al.*, 2016), Rejected feminine hygiene napkins (RECYC PhP, 2018), Modified Ordered Mesoporous Carbon (Lian *et al.*, 2019) and b-cyclodextrin modified resin (Yang *et al.*, 2016). Kenaf pellets (portrayed in Plate 2.4) also has great absorption ability, a feature that makes wood pellets excellent bedding materials for animals as well as a suitable clean up material (Kadiri, 2014; *Wood pellets*, 2016)



Plate 2.4: Starch, Animal Feed and Cissus bonded Kenaf Pellets During Oil Absorption Test (Source: Kadiri, 2014)

# 2.4. Oil Spill and Remediation

The exploration and conveyance of crude oil in Nigeria has resulted in catastrophic environmental degradation since its commencement in 1958 after oil discovery in 1956 at Oloibiri. Some of these oil pollution disasters includes Shells' Forcados storage tank #6 in July 1979 in Delta, Elf Obagi blowout of 1972, Texaco Funiwa blowout of 1980, Agip leakage of 1980, Bodo oil spills of 2008, Mobil Qua Iboe (Idoho) spill of 1998 in Akwa Ibom and the pipeline rupture of 2001 (Fingas, 2001; Aghalino and Eyinla, 2009; Etkin, 2011; David and Joel, 2013; Pegg and Zabbey, 2013;). It is also alarming to note an estimated 9-13 million barrels of oil has been spilled in fifty (50) years after the commercial production of crude that is (1958-2008) averaging 260,000 litres annually, surprisingly, these figures exclude minor spills (FME, NCF, WWF UK, CEEP-IUCN, 2006; Baird, 2010; Kadafa, 2012). Despite these startling facts, no serious action has been taken to curtail these spills or remediate the contaminated environs. This is further portrayed by the fact that engineers have not yet arrived to commence clean-up on a site when the remediation program was launched in August 2017 (Wikipedia, 2019), coupled with the poor condition of the surrounding waters containing 53.9mg/L and 62.7mg/L far back as 1993 (Daniel-Kalio and Braide, 2002). According to Agbonifo, (2016) and Adelana et al, (2011), these spills are attributed to quite several reasons which includes

- Sabotage (Destructive vandalizing and theft)
- Regulatory/ Institutional failure
- Corrosion and Ageing pipelines
- Equipment Failure

Oil spillage has been associated with serious devastating impact on the environment including altering soil micronutrients, soil aggregation properties, biota and crop growth (Udoh and Chukwu, 2014). Oil spills also have significant impact on the natural resources on which many poor communities in the Niger delta depends on including portable water, fishing, farming activities as well as the degradation of the ecological system, thus, impacting the biodiversity and environmental integrity of the Niger delta as well as health, livelihood and food security of people living in oil polluted regions (Digha *et al.*, 2017)

Hydrocarbon contamination of soil has been determined to cause death and mutation due to accumulation of pollutants in animal and plant tissues via extensive damage to the local ecological system (Otaiku, 2019). Oil spill has also been found to have significant impact on the physical, environmental, mental, communal, fiscal and health of the Niger Delta populace via destruction of arable land and polluting fishing creeks (Otaiku, 2019; Akpoghelie *et al.*, 2021). In summary, crude oil spillage contaminates wildlife, destroys habitats, toxifies the environment, disrupts food chain, affects the economy as well as constitutes long-term environmental damage thus it is vital to minimize damage to ecological systems and enhance recovery of polluted sites via preventive and rehabilitative measures.

The frequency of the causes of these spills are portrayed in Table 2.1., Table 2.2 shows common categories of petroleum and their group composition while Table 2.3 shows the effect of oil spillage on plants in the Niger delta.

**Table 2.1: Potential Causes of Oil Spill in Nigeria** 

<b>Potential Causes of Oil spillage</b>	Frequency (%)
Sabotage/Bunkering	36.0
Engineering	0.5
Human Error	2.0
Corrosion	36
Equipment Failure	6.0
Others	2.5
(9, 11, 1, 2011)	

(Source: Adelana et al., 2011)

**Table 2.2: Composition of Some Categories Oils and Petroleum Products** 

	_		_				
Group	Compoun d Class	(%) Gasolin e	Diese l	Light Crud e	Heav y Crud e	Intermediat e Fuel Oils (IFO)	Bunke r C
Saturates		50- 60	65- 95	55 - 90	25 - 80	25 -35	20 -30
	Alkanes	45 -55	35- 45				
	Cyclo- alkanes	5	30- 50				
	Waxes		0- 1	0-20	0- 10	2- 10	5- 15
Olefins		5- 10	0 - 10				
Aromatics		25- 40	5- 25	10- 35	15- 40	40- 60	30-50
	BTEX	15- 25	0.5- 2.0	0.1- 2.5	0.01- 2.0	0.05- 1.0	0.00- 1.0
	PAHs		0-5	10-35	15-40	40-60	30-50
Polar Compoun d			0- 2	1-15	5-40	15-25	10- 30
	Resins		0-2	0- 10	2- 25	10-15	10- 20
	Asphaltene s			0- 10	0-20	5- 10	5-20
Metals				30- 250	100- 500	100-1000	100- 2000
Sulphur		0.02	0.1- 0.5	0 -2	0- 5	0.5- 2	0 2- 4

(Source: Daniel-Kalio and Braide, 2002)

Table 2.3: Effects of oil spill on certain plants in Niger Delta

S/N	Plants	Description of oil spill effects
1	Plantain	Young plants in the immediate vicinity of the oil spill were killed
		from the root upwards. Those further away became discoloured,
		brown with leaves that are severely scorched.
2	Cassava	Cassava plants in the immediate vicinity of the oil spill were
	& Sugar Cane	destroyed irrespective of age. The root system and edible
		tuberous roots rotted. Standing dead plants were defoliated and
		their stems became ash-brown or dark in colour.
3	Raffia Palm	At the immediate vicinity of the oil spill, both young and mature
		plants were killed. Standing dead plants became defoliated,
		turned dark in colour, with dead inflorescence hanging down.
		Further away, the leaves turned brown were are scorched. Trunks
		were also discoloured.
4	Abura	Young plants at the immediate vicinity of the oil spill were killed.
		Standing plants were completely defoliated and stems became
		dark-brown in colour.
5	Alstonia sp.	No Alstonia sp. were found at the immediate vicinity of the oil
		spill. Further away, however, the plants became pale-green in
		colour.
6	Anthocleista	Plants in the immediate vicinity of the oils spill were destroyed.
	sp.	Standing plants were defoliated and their stem became darkened.
7	Panicum sp.	Plants in the vicinity of the oil spill were killed and masses of
		them, pinkish-brown in colour, became dislodged and were
		decaying. Standing dead plants had pinkish brown colour at the
		base, near the oil/water level, but yellowish-green to yellow
		leaves above. Further away, the plants still showed chlorotic
		response to oil.

(Source: Daniel-Kalio and Braide, 2002)

# Oil Spill on Land

Oil spills on land affects the growing condition of plants by diminishing essential nutrients, properties and resources such as soil pH, soil air, soil water, Total organic carbon, total organic matter, electric conductivity, etc (Anyadiegwu and Ohia, 2018; Akpoghelie *et al.*, 2021). Oil spills has also been found to be responsible textural changes in the soil (Edem and Oshunsanya, 2020) Oil spilled hydrocarbon also affects food crops, animals (both aquatic and terrestrial) resulting in ill-health and in severe cases death (Ordinioha and Brisibe 2013) as shown in Plates 5 and 6.

### Oil Spill on Water

Organic Contaminations of water bodies has been significant over decades because the resulting contamination not only affects animal but also human welfare and health with certain organic compounds such as Benzene, Ethylbenzene Toluene and Xylene highlighted amongst Organic contaminants necessitating restrictions in Safe Drinking Water Act of 1986 (Reed *et al.*, 1992; Schroeder, 2018). Benzene, Toluene, Xylene and Ethylbenzene are also said to possess low odour and taste and render water inconsumable even at low levels of contamination (WHO, 2005). Table 2.4 shows Characteristics, Transport Parameters and Maximum Contamination Level of BTEX members.



Plate 2.5: Oil Spill in Ogoni Waters

(Source: Vanguard, 2021)



Plate 2.6: Unrestrained Oil Well Head Spilling Oil in Kegbara Dere, Ogoni (2007)

(Source: BBC News, 2011)

Contact with Benzene either through consumption, inhalation or skin contact can cause increased apprehension, vertigo, lethargy, headaches, central nervous system impairment, misperception, skin irritation, damage to the cornea and catalepsy in lower doses while higher doses can result in cancer of blood forming organs especially Acute Myeloid Leukaemia (AML), impairment of immune (lymphatic) system and death and in the case of animals, it causes damaging effects on foetus such as delayed bone formation, bone marrow damage and low birth weight (Beth *et al.*, 1997; Dehghani *et al.*, 2018)

Xylene exposure can cause irritation of nose, eyes, throat and the skin, difficulty in breathing, impaired memory, stomach upset, impaired muscular coordination, giddiness, confusion, changes in sense balance, impairment of the central nervous system, increased pervasiveness of anxiety, damage to foetus in pregnant animals such as decreased weight, delayed skeletal development, skeletal changes and death in quite a number of cases. It is non carcinogenic in nature (Mike *et al.*,1995: NEPC, 2003).

Low consumption of inhalation of and physical contact with Toluene can cause headaches, giddiness, impairment of brain ability, memory loss, tiredness, weakness, confusion, difficulty in breathing, nausea, skin irritation and loss of appetite while prolonged consumption and inhaling of Toluene has resulted in hearing loss, astigmatism, brain damage, loss of muscular coordination, memory loss, poor balance, damage to liver, kidneys and lungs and death (Alfred *et al.*, 2000).

Exposure to Ethylbenzene for a short-term period is known to cause throat and eyes irritation, sluggishness, dizziness, skin irritation, damages to kidney and liver, blood changes and impairment of the nervous system in animals (Jessilyn and Julia, 1999).

The main goal of remediation is to remove to the best ability the quantity of toxic material in the environment, some of which are hydrocarbons containing Benzene, Toluene, Ethylbenzene and Xylene (BTEX) compounds. Some of these compounds are colourless and not easily detected by vision thus increasing the chances of causing harmful effects to the ecological system.

Table 2.4: Characteristics, Transport Parameters and Maximum Contamination Level for BTEX Compounds.

	Benz ene	Tolu ene	Ethylb enzene	o-Xylene	p-Xylene	m- Xylene
IUPAC Name	Benz ene	Meth yl Benz ene	Ethylbe nzene	1,2- Dimethyl benzene	1,4- Dimethyl benzene;	1,3- Dimethyl benzene
Molecula r formula	C <sub>6</sub> H <sub>6</sub>	C <sub>6</sub> H <sub>5</sub> CH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub> C <sub>2</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>4</sub> CH <sub>3</sub> CH <sub>3</sub>	C <sub>6</sub> H <sub>4</sub> CH <sub>3</sub> CH <sub>3</sub>	C <sub>6</sub> H <sub>4</sub> CH <sub>3</sub> CH <sub>3</sub>
Molecular weight (g/mol)	78	92	106	106	106	106
Colour	Colo urles s	Colo urles s	Colourl ess	Colourles s	Colourles s	Colourles s
Physical Stat e	Liqui d	Liqui d	Liquid	Liquid	Liquid	Liquid
Melting Point (°C)	5.5	-95	-95	-25	13.3	-47.9
Boiling Point (°C)	80.1	110. 6	136.2	144.4	138.37	139.3
Density at 20°C (g/cm³)	0.87 87	0.86 69	0.8670	0.8801	0.8611	0.8642
Aqueous solubility at 25°C (mol Lw <sup>-1</sup> )	0.02 24	0.00 60	0.0016	0.0018	0.0017	0.0015
Vapor pressure at 25°C (kPa)	12.5 9	3.72	1.23	0.89	1.17	1.10
Air– Water partition	0.22	0.25	0.32	0.20	0.28	0.30

at 25°C (L <sub>w</sub> L <sub>a</sub> -1)						
Octanol-						
water	2.17	2.69	3.20	3.16	3.27	3.30
partition						
at 25°C Log(						
K <sub>ow</sub> )						
Maximu	5	1000	700		10000	
m						
National						
Contami						
nation						
Level(μ						
g/L)						

**Source**: Mike et al., (1995); Beth et al., (1997); Jessilyn and Julia, (1999); Alfred et al., (2000) and Schroeder (2018)

Remediation of oil contaminated water bodies can be *in situ* or *ex situ*. The former implying onsite where the spill occurred while the latter involves the design of a system for treating the contaminated water samples (David and Joel, 2013; Akpor *et al.*, 2014). Kenaf (*Hibiscus cannabinus*) was identified by (Wikipedia, 2019; Janice, 2002) as one of the indigenous plants in Africa for environmental remediation in Nigerian oil regions. Kenaf pellets are also reported to have the capacity to absorb seven (7) times their weight of hydrocarbon (Kadiri, 2014).

The utilization of absorbents for the remediation has gained more influence as a form of *in situ* treatment because of the tendency to retrieve the oil from the water at limited expense, however, the use of naturally available absorbents is encouraged due to availability, biodegradability, efficiency and cost of production. The plant fibres are thus assessed based on properties such as absorbency, biodegradability, eco-friendly compatibility, effectiveness, cost and availability, pellet durability. These absorbent materials are sometimes subjected to certain treatments to enhance these properties, these treatments can be physical, chemical or biological and they include; carbonization, hydrothermal treatment, drying, crushing, mercerization, acetylation, grafting, acidification, aerogelization, cationization as well as inoculation with petroleum degrading microbes (Nguyen *et al.*, 2023).

#### **Absorption vs other Methods of Remediation**

Although, this study focuses on the usage of absorbents, it is pertinent to note that in ideal cases, remediation involves more than one method. Other methods of remediating contaminated water bodies in comparison to the use of absorbents are discussed below.

I. Mechanical Method: The mechanical method of remediation involves the removal of contaminated water. This can be done using several equipment which includes skimmers, water pumps, filters and artificial (collapsible dams) (FPRG, 2004) as depicted in Plate 2.6. The removed contaminated water samples are then transported to treatment facility designed to remove the contaminants. The treated water samples are then returned to the water body. This method is often expensive as the cost involved includes transportation of the samples, cost of treatment chemicals,



Plate 2.6: Spill Task Containment Oil Boom

(Source: Safetynigeria, 2022)

- operational costs and thus is not recommended for developing countries and regions with limited resources
- II. On site Burning: Burning involves the use of ignited oxidation to remove hydrocarbon from the surface of water. It is easy to commence but must be carried it in a controlled environment to prevent fire disaster (Walther, 2014). It is cheap but it does not completely remove the hydrocarbon, it increases global warming and there is no recovery of the spilled hydrocarbon as compared to the use of absorbents.
- III. Permeable Reactive Barriers (PRB): This is an *in-situ* method of treatment where influents are treated by passing through permeable layers. These barriers are made of materials such as Zeolite, Single-walled Carbon Nanotubes, Multiwalled Carbon Nanotubes, Hydrophobic Granular Activated Carbon, Activated Carbon Fibres, Zero Valence Iron, Iron Oxides/Oxyhydrates, that remove the contaminants via filtration (Shakeri *et al.*, 2016; Vaezihir *et al.*, 2020). These are however commonly used in groundwater sources such as wells, boreholes and washbores contaminated with BTEX and heavy metals.
- IV. Chemicals: The use of chemicals has also been recorded. Chemicals used in remediation are either solidifiers, dispersants or oxidants. The solidifiers such as Oil Bond, ALSOCUP, Enviro Bond 403 and Gelco 200 are coagulants used for spilled liquid hydrocarbon (Akpor *et al.*, 2014; Sundaravadivelu *et al.*, 2016) while dispersants are used for thinning the films of hydrocarbon, thus quickening the natural disintegration of oil film on water. Oxidants commonly used are oxygen (Hypolimnetic oxygenation) and Nitrates for the oxidation of oil spilled on water (Søndergaard *et al.*, 2008). However, recent developments have shown that nanoparticles used in nanophotocatalytic process of treating wastewater such as Maghemite are capable of effectively degrading BTEX in produced water (Sheikholeslami *et al.*, 2018).
- V. Bioremediation: This is the utilization of organisms (plants and microbes) to curtail oil spillage. Certain oil-eating microbes (bacteria, yeast and fungi) have been used in remediating hydrocarbon polluted water sources. Some of these oil-eating microbes include *Bacillus, Brevibacterium, Vibrio, Pseudomonas, Proteobacteria, Deinococcus-thermus, Arthrobacter, Aspergillus, Candida, Penicillum* etc. (Akpor

- et al., 2014; Akmirza et al., 2017; Atohunjere, 2018). Bioremediation using certain plants have also been reported, with the likes of Bulwort, Pondweed, Coontail, Eurasian water, parrot feather, and Hydrilla (Akpor et al., 2014).
- VI. Natural Degradation: This method involves allowing nature to take its natural course. This process is slow and involves physical, chemical and biological transformation all of which encompasses digestion, oxidation and biodegradation (Kao *et al.*, 2006 and Walther, 2014). This is only advised in situations where the oil spill is not affecting the ecological stability of the affected region. It is usually aided by natural occurring movements such as wind, weather changes, water waves etc.

## 2.5. Experimental Design and Data Analysis

The experimental schedule for trials is an important aspect of mathematics and statistics that is incorporated in science and technology. The main objective is to integrate mathematical concepts of selection such as permutations, combinations and probability with a reduced tendency of repetition and exclusion of redundant variables. This implies that repetitions are eliminated while variables with lower probabilities are likely excluded.

This also applies to this research where questions on the production and utilization of kenaf pellets in remediating oil polluted water bodies arose. There came the formulation of these questions into objectives. These formulations led to the introduction of optimization where certain parameters were varied at various levels. This is corresponding to the translation into a statistical model which is succeeded by development of a statistical model using Response Surface Methodology (RSM), this is presented in the Appendices as a design schedule using *Design Expert* software

#### 2.5.1. Experimental Design

A proper experimental design is a schedule of trials that is deliberately constructed in anticipation for an actual series of experiments. It is a major tool for the effective utilization of a scientific method having provisions for variations in parameters. These number of variations may either be even or odd for which only one per parameter will be selected for each trial. This is the mathematical concept of combination (Hinkelmann and Kempthorne, 2008; Lawson, 2015; Selvamuthu and Das. 2018) as portrayed in Figure 2.7.

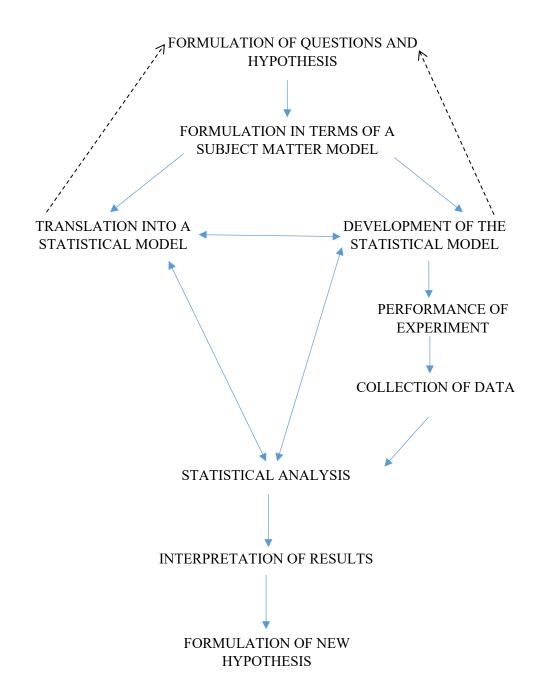


Figure 2.7: Stages in Scientific Experimentation

(Source: Hinkelmann and Kempthorne (2008))

The experimental design should also eliminate repetition while concentrating on selection of variables with a higher probability, thus eradicating perplexing effects associated with variations in the factors. Some of these experimental designs may include factorials, Response Surface Design and mixture and may be applied using experimental design software such as *Design Expert*, R, *Slmul8 Stat*, SPSS, STATA, Minitab etc.

#### 2.5.2. Data Analysis

Data analysis, processing and interpretation of results is another essential phase and it marks the completion of the experiment. It occurs after the collation of data from the experiments which is the outcome from the performance of the experiments. Data analysis involves classification of the obtained results, establishing the relationships between the variables and presenting them with the aid of charts, graphs, and models further explained by an associated report entirely based on the analysis with references to similar external sources of information in a bid to support decision making.

There are four (4) broad categories of data analysis (Calzon, 2021; Johnson, 2021; Stevens 2022), namely:

- Descriptive analysis
- Diagnostic analysis
- Predictive analysis
- Prescriptive analysis

**Descriptive Analysis:** This is a category of data analysis that evaluates past experiments with a view to present details on occurrences and is usually presented with the aid of central tendencies and dispersion as well as charts and other graphical means (Heumann *et al.*, 2016)

**Diagnostic Analysis:** This is the category of data analysis usually associated with the reason for an occurrence. This is often presented with figures and quotations. Examples are analysis of sales decline, reduction in production quality etc (Stevens 2022).

**Predictive Analysis**: This is a category of data analysis that evaluate historical data with a view to envisage future events. An example of predictive analysis is probability determination and is often presented with figures.

**Prescriptive Analysis**: This is possibly the most advanced category of data analysis as it examines historical data with a view to provide insights on the best approach to certain situations. It involves computer programs like regression, artificial intelligence and machine learning. In summary, it generates models which can be used to predict outcomes in scenarios.

Programs like R, SPSS, MATLAB, Python, Excel, SAS etc (Johnson, 2021) are capable of doing these categories of data analysis making cumbersome work look really simple and can also cover a wide range of profession. This makes data analysis a vital aspect of research in the various professions.

Artificial Neural Network, an aspect of Artificial Intelligence will be was used for the data analysis. It is an innovation of data processing that emulates biological transmission of responses via neurons, that is, it is an imitation of the human neurological system into data analysis (Graupe, 2013; Kim. 2017; Aggarwal, 2018). The development of artificial intelligence was initiated by Nicolas Rashevsky in 1930s via Neurodynamic studies and has within a century evolved into a more complex but significant aspect of modern-day technology (Alaloul and Qureshi, 2020).

Artificial Neural Network has been reported to be used either in unison or along with other models in various aspects and fields such as extraction of oil from African oil bean kernels (Ogunlade, 2018), modelling ecological conditions (Jørgensen, 2016), granulation of moist pharmaceutical formulations (Ismail *et al.*, 2020), fatigue behavior of composites (Diniz and Júnior, 2019), strength of concrete (Golafshani *et al.*, 2019; Abass *et al.*, 2019), efficacy of a multistage moving bed biological process (Almomani, 2020), anticipated wind and solar energy site assessment (Adedeji *et al.*, 2020) etc. Figure 2.8 depicts the data processing pattern of the ANN

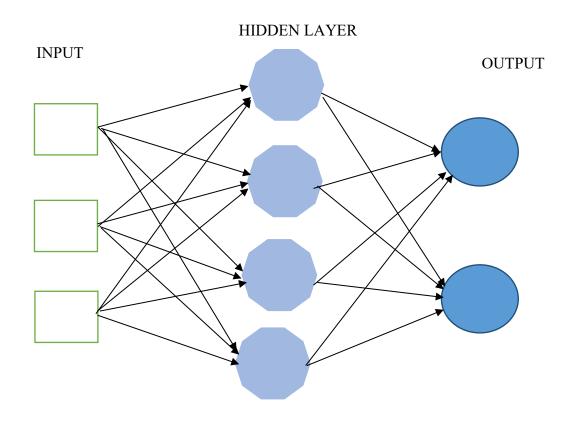


Figure 2.8: Pattern of Artificial Neural Network

## **CHAPTER THREE**

### MATERIALS AND METHODS

# 3.1. Design of the Screw Type Pelletizer

Mild steel was used as a primary constructing material for designing food and creating the pelletizer because of its availability and durability. Apart from the bearings and shaft, the pelletizer was fabricated using mild steel in resemblance to the AutoCAD diagram in Appendix I.

Predesign tests were carried out on the Kenaf materials to attain certain information. Physical properties such as angle of repose and the bulk density of Kenaf-binder mixture were also determined. The angle of repose of the input materials (mixture of Kenaf and binder) was ascertained in a similar method as prescribed by Mohsenin (1986), while the bulk density of the mixture was obtained using procedures from ASAE standard 269.4

#### 3.1.1. Hopper design

The screw type pelletizer will have a new hopper designed and fabricated using mild steel plate as described by Gale (2009) and Oduntan *et al.*, (2014). A hopper equipped with a feed gate was designed, the feed gate is to control the feed rate during the optimization of the pelleting process. The hopper will be fitted above the barrel such that the hopper empties directly into the barrel through an opening 75 mm by 60 mm. (Kadiri, 2014; Aremu *et al.*, 2014) stated that Equation (3.1) will be used to determine the hopper's volume.

$$H_V = \frac{c_M(m^3/hr)}{N_C(min/hr)} \times C_S$$
 3.1

Where  $H_V$  is the Hopper's Volume

 $C_M$  is the machine's capacity

N is the frequency of loading the hopper per hour

 $C_s$  denotes Safety Constant (1.5)

Nonetheless, equation (3.2) can be used to determine the machine's capacity, CM.

$$C_M = \frac{Feed\ Rate}{Bulk\ Density\ of\ the\ Mixture}$$
 3.2

Where the bulk density obtained was 385 kg/m<sup>3</sup>

Feed rate used is 120 kg/hr (Maximum for the study)

$$C_M = \frac{120}{385}$$

$$C_M = 0.312 \text{m}^3/\text{hr}$$

Applying equation 3.1, it is desired that the hopper be emptied every minute, that is N = 60 Hopper's volume becomes

$$H_V = \frac{0.312}{60} \times 1.5$$

$$H_V = 0.0078 \text{ m}^3/\text{min}$$

The angle of inclination used in the design of the hopper was 65 degrees after taking into consideration, the angle of repose determined earlier. According to Ugoamadi (2012), Equation 3.3 describes the relationship between hopper volume, height, and orifices sizes at the hopper ends for a truncated pyramid.

$$V_H = \frac{h}{3} \left[ A_1 + A_2 + \sqrt{A_1 A_2} \right]$$

$$V_H = 0.0078 \text{ m}^3/\text{hr}$$

$$h = 0.3 \text{ m}$$

$$A_2 = 0.075 \text{ m} \times 0.06 \text{ m} = 0.0045 \text{ m}^2$$

$$A_1 = 0.072546 \text{ m}^2 \approx 7.25 \times 10^2 \text{ m}^2$$

Therefore, the area of the orifice at the hopper's top will be  $7.25 \times 10^{-2}$  m<sup>2</sup>

### 3.1.2. Screw and barrel design

The screw design desired for the pelletizer will be achieved by emulating formula displayed in Oduntan *et al.*, (2014) and in a constant pitch, constant root diameter pattern described by Harper (1980) and Rokey (2000) as single screw pelletizer.

The barrel used in this study is a smooth bore type made of mild steel and has dimensions of 355 mm long, as well as 80 mm and 110 mm as internal and external diameters respectively. According to Bortolamasi and Fottner (2001), equation 3.4 shows a correlation between the screw pitch and screw diameter at the feeding section.

$$P_S = \frac{2}{3}D_S$$

This can be restated as

$$D_S = \frac{3}{2}P_S \tag{3.4}$$

This is also in conformity with Huber (2000) which depicts that at the feeding section, screw pitch of greater or equals to  $1 \ge 1$  is used for single flight screws.

Given that the internal diameter of the barrel is the same as the major diameter of the screw conveyor i.e., 80 mm, according to Ugoamadi (2012) the minor diameter of the screw conveyor can be obtained as

$$Q = \frac{\pi}{4} (D_S^2 - d_S^2) \times P \times N \times f \times 60$$

This can be rewritten as

$$d_S = \sqrt{\left(D_S^2 - \frac{4Q}{\pi \times P \times N \times m \times 60}\right)}$$
 3.5

Where  $Q = Input Rate m^3/hr$ 

 $D_{\rm s}$  = Diameter of the Barrel

 $d_s$  = Screw conveyor's minor diameter

P = Screw conveyor's Screw Pitch

m = Material Property

N= pelletising speed

Considering the extreme scenario where Q is maximum (when input is 120 kg/hr.), P and N are least (0.04 m and 40 rpm) respectively

$$Q = \frac{\text{Input}}{\text{Density of Preconditioned Mixture}}$$

$$Q = \frac{120}{380}$$

$$d_S = \sqrt{\left(0.08^2 - \frac{4 \times 120 \div 385}{3.142 \times 0.04 \times 40 \times 1 \times 60}\right)}$$

$$d_S = \sqrt{2.26664 \times 10^{-3}}$$

$$= 0.04761 \text{m}$$

$$\approx 48 \text{ mm}$$

The diameter of the screw conveyor is obtained from deducting the minor diameter from the major diameter of the conveyor. This is obtained as

$$Dsc = 80 - 48 = 32 \text{ mm}$$

Helix angle  $\theta$  can be obtained using trigonometric relations from the equation 3.6

$$\theta = \cos^{-1}\left(\frac{D_b - D_{sh}}{D_{sc}}\right) \tag{3.6}$$

Where  $D_b$  = Barrel's Diameter (80 mm)

 $D_{sh}$  = Shaft's Diameter (50 mm from 3.1.4)

 $D_{sc}$  = Diameter of Screw Conveyor (32 mm)

Inputting the above values,

$$\theta = \cos^{-1} \left( \frac{0.08 - 0.05}{0.032} \right)$$
$$\theta = \cos^{-1} 0.9375$$
$$\theta = 20.36^{\circ}$$

The Length of the conveyor according to Oduntan *et al.*, (2014) can be obtained through equation 3.7 below

$$S = 3.42(0.5d + tl)\theta$$
 3.7

Where d is the diameter of the shaft (m) (from 3.1.4)

t is the tangent of the tapering angle (for this case 1)

 $\theta$  denotes angle of helix (rads)

1 denotes the shaft's dimension (m)

Inserting the respective numerical values in equation 3.7

$$S = 3.42(0.025 + (1 \times 0.455))0.3554$$
$$S = 0.5834m$$

## 3.1.3. Belt and pulley system

The Pulley system (used to determine the pelleting speed) will be arranged using the formula presented by Kadiri (2014) and Aremu *et al.*, (2014) as depicted in Equation (3.8)

$$N_1 D_1 = N_2 D_2 3.8$$

Where  $N_1$  = Pulley Speed on Prime mover

 $D_1$  = Pulley Diameter on Prime mover

 $N_2 = Gear box Pulley Speed$ 

 $D_2$  = Gear box Pulley Diameter

The belt length selection was determined according to Aremu et al., (2014) using Equation 3.9

$$L = \frac{\pi}{2}(D_1 + D_2) + 2s + \frac{(D_1 - D_2)^2}{4x}$$
 3.9

Where  $D_1$  = Prime mover's Pulley Sheave diameter

 $D_2$  = Gear box Pulley's Sheave diameter

L = Length of Belt

s = Distance between the centres of both pulleys

## 3.1.4. Shaft design

The forces as well as component of the extruder's shaft is represented in Figures 3.1 and 3.2 below

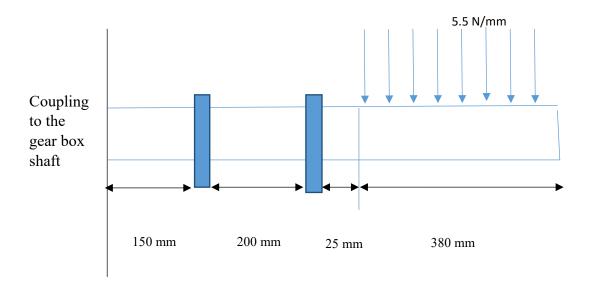


Figure 3.1: Schematic Diagram of the Pelletizer's Shaft

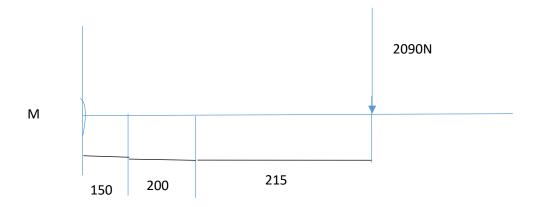


Figure 3.2: Forces and their Adjudged Points of Action on the Pelletizer's Shaft

Where M = Turning Moment on the Shaft from the gear box

W = weight of the Screw conveyor on the Shaft

For a pulley driven by a motor using belts, there exists 2 sides with different tensions, say  $T_1$  and  $T_2$  for the tight and slack side respectively.

According to (Ugoamadi, 2012), Ti can be obtained from the mathematical expression

$$T_1 = T_{max} - T_C \tag{3.10}$$

$$T_1 = \sigma A - mV^2 \tag{3.11}$$

Where  $\sigma = Maximum \ Safe \ strength \ of \ belt \ 2.5 \ N/mm^2$ 

A= Cross sectional Area of the Belt 375 mm<sup>2</sup>

m= mass per unit length of the belt 0.375 kg/m

V= Velocity of the Belt m/s

However, Odesola *et al.*, (2016) stated that the Velocity of a belt during transmission can be mathematically derived from the expression

$$V = \frac{\pi \times D \times N}{60}$$
 3.12

Where D= Diameter of Driving Pulley and

$$N = Speed in rpm$$

For this design, the diameter of the Pulley is 100 mm = 0.1 m and N is 1420 rpm.

$$V = \frac{3.142 \times 0.1 \times 1420}{60}$$

$$V = 7.44 \text{ m/s}$$

Imputing the 7.44m/s for V in equation 3.7,

$$T_1 = (2.5 \times 375) - (0.375 \times 7.44^2)$$
  
 $T_1 = 937.5 - 20.7576$ 

$$T_1 = 916.7424 \text{ N}$$

Odesola *et al.*, (2016) stated that the relationship between  $T_1$  and  $T_2$  is expressed mathematically in formula 3.13

$$\frac{T_1 - mV^2}{T_2 - mV^2} = \ell \frac{f \times \alpha}{\sin(0.5\theta)}$$
3.13

Where f is coefficient of friction between the belt and pulley = 0.25 (Khurmi and Gupta, 2005 and Ugoamadi, 2012)

 $\alpha$ = angle of wrap of the belt around the pulley

 $\theta$  = groove angle of the pulley 35°

According to Khurmi and Gupta (2005) and Odesola *et al.*, (2016), angle of wrap is obtained from equation 3.14

$$\alpha = 180^{\circ} - 2\sin^{-1}(\frac{R-r}{c})$$
 3.14

For this design R and r are the same in value, this makes  $\alpha$ = 180 (3.142 rads)

Imputing these values in equation 3.9, we get

$$\frac{915.82 - 0.375 \times 7.44^{2}}{T_{2} - 0.375 \times 7.44^{2}} = \ell \frac{0.25 \times 3.142}{\sin(0.5 \times 35)}$$

$$\frac{915.82 - 20.7576}{T_{2} - 20.7576} = \ell^{2.6122}$$

$$\frac{895.0624}{T_{2} - 20.7576} = 13.6288$$

$$13.6288(T_{2} - 20.7576) = 895.0624$$

$$13.6288T_{2} - (13.6288 \times 20.7576) = 895.0624$$

$$13.6288T_{2} - 282.9012 = 895.0624$$

$$13.6288T_{2} = 1177.984$$

$$T_{2} = 86.432$$

Equation 3.15 can be used to calculate the diameter of the shaft according to Khurmi and Gupta (2005), Ugoamadi (2012), Oduntan *et al.* (2014), and Odesola *et al.* (2016).

$$D = \sqrt[3]{\frac{16}{\pi\tau} \left( \sqrt{(K_b M_b)^2 + (K_t M_t)^2} \right)}$$
 3.15

Where  $\tau$  = Shaft material's Maximum Permitted Stress (42MPa i.e. 42N/m<sup>2</sup>)

 $K_b$  = bending shock and fatigue factor (1.5)

 $K_t$  = twisting's combined shock and fatigue factor (10)

 $M_b$  = shaft's maximum bending moment

 $M_t$  = shaft's maximum twisting moment

From Figure 3.2, maximum bending moment on the shaft  $M_b$ , can be obtained in equation 3.16 as

$$M_b = W_{SC} \times d \tag{3.16}$$

Where  $W_{SC}$  = Weight of Screw Conveyor

d =Distance of point of force to the nearest support

$$M_b = 2090N \times 0.215m$$
  
 $M_b = 449.35$ Nm

The belt and pulley system was connected to the gear box which was then transmitted to the shaft via coupling. This suggests that the twisting moment applied to the gear box and the shaft of the pelletizer is the same. Equation 3.17 can be used to get the shaft's Maximum Twisting Moment, Mt, according to Khurmi and Gupta, (2005) and Ugoamadi, (2012)

$$M_t = (T_1 - T_2) \frac{D}{2}$$
 3.17  
 $M_t = (916.7424 - 86.432) \times 0.05$   
 $M_t = 830.3104 \times 0.05$   
 $M_t = 41.6655Nm$ 

Inserting the obtained value of  $M_b$  and  $M_t$  in equation 3.15, we get

$$D = \sqrt[3]{\frac{16}{3.142 \times 42 \times 10^6}} \left( \sqrt{(1.5 \times 449.35)^2 + (10 \times 41.6655)^2} \right)$$

$$D = \sqrt[3]{\frac{16}{1.31964 \times 10^8}} \left( \sqrt{674.025^2 + 416.655^2} \right)$$

$$D = \sqrt[3]{1.21245 \times 10^{-7}} \left( \sqrt{454309.7004 + 173601.5557} \right)$$

$$D = \sqrt[3]{1.21245 \times 10^{-7}} \left( \sqrt{627911.2561} \right)$$

$$D = \sqrt[3]{1.21245 \times 10^{-7} \times 792.4085159}$$

$$D = \sqrt[3]{9.607557 \times 10^{-5}}$$

$$D = 0.0458m \approx 46 \text{ mm}$$

Therefore, a shaft of 50 mm diameter is required

#### 3.1.5. Power requirement

The total power required to operate the machine is summation of the energy expended in conveying the materials along the barrel, the force required to power the shaft and the force required to extrude the materials through the die.

Force required to push Kenaf fibre is a function of the moisture content as well as the size of the die opening i.e.

$$F = P \times A \tag{3.18}$$

Where P is the compressive strength of the material at a particular moisture content and A is the cross-sectional area of the die opening

It is anticipated that the introduction of the binders to the ground Kenaf will like increase the moisture content to about 35% wb. Dauda *et al.*, (2014) reported that the compressive stress on Kenaf at 35% moisture content is 9.24MPa.

The cross-sectional area to be considered is the smallest of the Die Diameters to be used in the study, this is obtained as 30 mm

The minimal force needed to extrude Kenaf pellets is thus obtained mathematically as

$$F = P \times A$$

$$F = 9.24 \times 10^{6} \times \frac{\pi D^{2}}{4}$$

$$F = 9.24 \times 10^{6} \times \frac{3.142 \times 0.04^{2}}{4}$$

$$F = 9.24 \times 10^{6} \times 1.2568 \times 10^{-3}$$

$$F = 11612.832 \text{ N}$$

The power required to extrude the Kenaf mixture is obtained using equation 3.19

$$E = \frac{F \times N \times D}{60}$$
 3.19

Where D is the diameter of the shaft

N is the speed of pelleting

$$E = \frac{11612.832 \times 120 \times 0.05}{60}$$
$$E = 1161.2832 \text{ W}$$

The power expended in conveying the mixture along the barrel according to Odesola *et al.*, (2016) as expressed in equation 3.20.

$$E_r = \frac{QL}{367}(\omega_o + \sin\beta)$$
 3.20

Where Q denotes Feed Rate (2 kg/min)

L stands for Screw conveyor length (0.58 m from 3.1.1)

 $\omega_{\rm o}$  is transportation constant (4.0)

 $\beta$  is barrel's inclination angle (0°)

The power consumed in transporting the materials along the barrel thus becomes

$$E_r = \frac{120 \times 0.5834}{367} \times 4$$

$$E_r = 0.7630 W$$

Odesola *et al.*, (2016) stated that the power expended in powering the pelletizer's shaft according to Odesola *et al.*, (2016) is represented in mathematical expression 3.21 as

$$E_s = 2M \left(\frac{\pi N}{60}\right)^3 d^2 \tag{3.21}$$

Where M denotes the shaft's mass

N is the shaft rotating speed

d is the shaft's diameter

M can be obtained via equation 3.22

$$M = \rho \times V_{\rm s} \tag{3.22}$$

Where  $\rho$  is the density of the metal used for making the shaft (7900kg/m3)

 $V_S$  is the volume of the shaft  $(\pi r^2 l)$  (r is the radius while l, the length)

$$M = 7900 \times 3.142 \times 0.025^2 \times 0.455$$
$$M = 7.06 \, Kg$$

Inserting the values into equation 3.21

$$E_s = 8 \times 7.06 \left(\frac{3.142 \times 120}{60}\right)^3 0.025^2$$
$$E_s = 8.76 W$$

The total power required for the process becomes

$$E_T = E_S + E_R + E$$

$$0.763 + 8.76 + 1161.2832$$

$$E_T = 1170.81 \text{ W}$$

The power required to power the pelletizer according to Ugoamadi, (2012) is expressed in equation 3.23

$$P(hp) = \frac{E_T \times K_S}{746}$$
 3.23

Where  $K_S$  is Service Factor (1.875)

$$P(hp) = \frac{1170.81 \times 1.875}{746}$$

$$P(hp) = 2.943 Hp \approx 3 HP$$

Therefore a 3 horse power electric motor was suitable for the pelletizer

#### 3.1.6. Die diameter

The Pellet sizes were determined by the opening in the cone. The cone is made from mild steel fitted to the barrel using nuts and bolts via flanges. Pellet sizes will be varied by changing the sizes, this was achieved by changing the cones used during the optimization process, pellet sizes of 28, 30, 32.5, 35 and 37 mm are to be produced.

## 3.2. Evaluation and Pre-Selection for Optimization

The binder used was locally sourced, economical as well as environmentally friendly. For this reason, cassava starch was chosen, cassava starch was obtained from a cassava processing centre at Ajetunmobi axis, Water Junction, Ibadan North Local Government, Ibadan. The starch was initially air dried to reduce the moisture content.

The machine was evaluated to determine the conditions and parameters before optimization. A water/kenaf ratio of 3:1 was selected after considering the texture of the mixture. A Kenaf/water ratio of 4:1 was too watery while a 2:1 water Kenaf ratio was too dense and got dried easily in the pelletizer. Hence, the choice of 3:1 for the optimization process.

Control sample pellets made from blended Kenaf particles was also made during the evaluation and subsequently a particle size distribution was carried out. Kenaf particles of (1.00 mm), 2.36 (2mm), 3.35(3 mm), 4.00 mm and 4.75 (5 mm) were tested during which 2.36 mm sized particles gave a homogeneous mixture ideal for pelleting process thus resulting in the choice of 2 mm particles for this study.

# 3.3. Optimization of the pelletizer

Certain parameters were varied for the purpose of optimizing the pelleting process. For instance, the Screw pitch, cone size (pellet diameter) as well as the pulley ratio (pelleting speed) were varied collectively as machine parameters. Also varied was the kenaf-starch proportion. The desired values are presented in Appendix I.

A nominal sample mass of 600 g of dried Kenaf particles will be used for each trial, water of 1800 g per trial was used. The starch-kenaf ratio was 1:1, 1:1.25, 1:1.5, 1:1.75 and 1:2. The screw pitch and pelleting speed was varied. The screw pitch of 40, 50, 60, 70 and 80 mm were used while the pelleting speed of 40, 50, 60, 70 and 80 rpm were used for the experiment. Pellets were produced using 28, 30, 32, 35 and 37 mm diameter openings in protruding cones, this was to enhance the formation of pellets. The experiment schedule was designed using Stat-Ease Design Expert 10.

The experiment encompasses three categories, namely:

- Performance evaluation of the pelleting machine
- Mechanical evaluation of the pellets
- Remediation characteristics of the pellets

#### 3.3.1. Response Surface Methodology (RSM)

The experiment would be designed using Response Surface Methodology, a tool available on Design Expert Version 10.0. Table 5 shows the schedule of parameters and the values.

**Table 3.1: Experimental Schedule for the Pelleting Process** 

Parameters	Values				
	1	2	3	4	5
Screw Pitch (mm)	40	50	60	70	80
Pelleting Speed (rpm)	40	50	60	70	80
Die Diameter (mm)	27.5	30.0	32.5	35.0	37.5
Starch/Kenaf ratio	1:1.00	1:1.25	1:1.50	1:1.75	1:2.00

# 3.4. Performance Evaluation of the Pelleting Machine

According to Aremu *et al.*, (2014), Davies and Davies (2011) and Ojomo *et al.*, (2010) the pelletizer was evaluated based on the Pelleting Efficiency and Percentage Recovery.

## 3.4.1. Pelleting Efficiency

The pelletizer's propensity to generate pellets is known as the "Pelleting Efficiency," and it may be mathematically calculated using the equation 3.24

$$\eta \% = \frac{M_P}{M_Q} \times 100\%$$
 3.24

Where η symbolizes Pelleting Efficiency

M<sub>P</sub> denotes the mass of pellets produced

Mo is the output mass

#### 3.4.2. Percentage Recovery

The percentage of the extrudate recovered following each test is known as the percentage recovery. Percentage recovery is mathematically gotten from the Equation 3.25

$$R(\%) = \frac{M_o}{M_E} \times 100\%$$
 3.25

Where *R*% denotes Percentage Recovery

*M<sub>O</sub>* represents output Mass

 $M_E$  is the extrudate mass

## 3.5. Mechanical Properties of Pellets

The formed pellets were tested using a Computer Controlled Universal Testing Machine Model WDW-50 (Instron) at the timber mechanics laboratory, Forest Research Institute of Nigeria, Jericho, Ibadan, Oyo State as stipulated by Harun and Afzal (2016), Akinoso and Raji (2001) and Liu *et al.*, (2014). Compressive forces were applied to each pellet at a speed of 5mm per minute and the following data were obtained from the charts and figures from the Instron testing machine.

- Force at Peak (N)
- Deflection at Peak (mm)

- Energy to Peak (Nm)
- Force at Yield (N)
- Energy to Yield (Nm)
- Force to Break (N)
- Deflection at Break (mm)
- Energy to Break (Nm)
- Young Modulus

The forces (force at peak, force at yield and force to break) and deflection (deflection at peak, deflection at yield and deflection at break) would be obtained via direct reading from the universal testing machine.

The energy values would be obtained from the mathematical expressions 3.26, 3.27 and 3.28 while the young modulus would be attained using Equation 3.29

Energy to Peak

$$= \frac{Force \ to \ Peak \times Deflection \ at \ Peak}{Time \ taken \ to \ Peak}$$
 3.26

Energy to Yield

$$= \frac{Force\ to\ Yield \times Deflection\ at\ Yield}{Time\ taken\ to\ Yield}$$
3.27

Energy to Yield

$$= \frac{Force\ to\ Break \times Deflection\ at\ Break}{Time\ taken\ to\ Break}$$
 3.28

Young Modulus

$$= \frac{Stress \ at \ Yield}{Strain \ at \ Yield}$$
 3.29

# **Durability**

The mechanical durability of the pellets as described by (Graham *et al.*, 2017; Larsson and Samuelsson, 2017; Ahn *et al.*, 2014; Liu *et al.*, 2013 and Mina-Boac *et al.*, 2006) was determined by either agitating the pellets for a period of 10 minutes at low speeds ranging 50-70 RPM or by agitating the pellets at high speeds for a relatively short period. For this experiment, 75g of kenaf pellets were placed in a durability testing apparatus as designed

in Appendix I (Fig A2) and tumbled at a speed of 1180 RPM at a torque of 0.03311Nm for a duration of 1 minute. The tumbled pellets were then sieved using a 4.75 mm aperture screen to remove crumbs. The durability was mathematically determined via equation 3.30

$$D(\%) = \frac{M_{at}}{M_{bt}} \times 100$$
 3.30

Where D = Durability of the pellets

 $M_{at}$  = Mass of samples after tumbling

 $M_{bt}$  = Mass of samples before tumbling

#### 3.6. Remediation Potentials

Crude oil was obtained from Conoil regional office in Port Harcourt, Rivers State while the reagents (1,1,1-trichloroethane and pH buffer solution) were sourced from a retail outlet in Ring Road area of Ibadan. A contaminated water solution is made by introducing 50 ml of crude oil to 200 ml of deionized water and stirred vigorously for 1 min, 30grams of Kenaf pellets are then added to the contaminated sample and was allowed to absorb the oil before being removed after a period of 5 minutes. The remaining oil remediated sample is then determined using the following procedure.

Remediation of the polluted water using the kenaf pellets was investigated under two categories, namely:

- Amount of Oil removed
- Changes in the pH value

#### 3.6.1. Ratio of Oil Removed.

The quantity of oil removed by the kenaf pellets was determined using HACH DR/2000 spectrophotometer. The remediated sample is measured into a 500ml capacity separatory funnel and 35ml 1,1,1-trichloroethane was added. The reagent 1,1,1-trichloroethane serves as a solvent for the oil content, bonding with the hydrocarbon and subsequently precipitating to the bottom of the water if the mixture is left undisturbed for a 10 minutes period. Plate

3.1 and Figure 3.3 shows the picture and molecular structure of the reagent, 1,1,1-trichloroethane respectively.

The water is separated from the oil/1,1,1-trichloroethane mixture in the separatory funnel with the aid of the attached tap. The oil content in the oil/1,1,1-trichloroethane mixture is then determined by comparing to a buffer of 25 ml 1,1,1-trichloroethane as stipulated in the procedure manual of the HACH DR/2000 spectrophotometer.

This process is repeated for each set of pellets produced and the amount of oil removed was determined by the mathematical expression

Ratio of Oil Removed = 
$$\frac{Initial \ Amount \ of \ Oil \ in \ Water \ (ppm)}{Final \ Amount \ of \ Oil \ in \ Water \ (ppm)}$$
3.31

## 3.6.2. Changes in pH Value

The pH value of the samples was determined using an RoHS ATC pen type pH meter before and after remediation and the differences in the values obtained. A simulated crude oil spill was created using 50 ml of crude oil and 200 ml of deionized water. The pH meter was first calibrated using a standard buffer solution of 7.0 before use. The probe was then inserted to determine the pH of the polluted water and then recorded. Kenaf pellets were then added to the simulated solution and remediation was allowed to take place for a 5 minutes period, the pH of the remediated sample was then determined after calibration using a standard buffer solution. The process was repeated for the sets of pellets before and after remediation.

# 3.7. Analysis of Data

The analysis of the recorded data was done using Artificial Neural Network, an aspect of Machine Learning (Alaloul and Qureshi, 2020), the objective is to use data obtained from the experiments to predict anticipated properties of kenaf pellets using various parameters using Artificial intelligence.

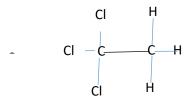


Figure 3.3: Molecular Formular of 1,1,1-TriChloroethane



Plate 3.1: 1,1,1-Trichloroethane

#### **Model Architecture**

The multilayer perceptron artificial neural network (MLP-ANN) model of two hidden layers was developed to model each of the responses relative to their predictors (input variables). Different model architectures were tested on each response to determine which architecture best predicts the response variable. For each tested architecture, several runs and trials were carried out to ensure that overfitting and underfitting of one architecture is avoided. All the responses were trained using the Levenberg Marquardt backpropagation algorithm. This technique was adopted due to its effectiveness in earlier studies and its laudable merits. For instance, the Levenberg Marquardt backpropagation algorithm is characteristically known for its quick convergence compared to the gradient descent-based models. The convergence when adequately monitored does not occur at the local optimal. Further to this, the Levenberg Marquardt backpropagation algorithm is very much effective in solving non-linear problems and this is evident in the relationship between the inputs and the outputs of each of the models developed. This technique is also known to perform effectively in cases where paucity of data exists.

The logsigmoid and softmax transfer functions were used for the first and second hidden layers of all the models developed. These transfer functions translate the input signals to output signals. These activation functions have been observed to be very efficient for continuous data, hence the reason for their choice in this research (Ansari *et al.*, 2018; Abass *et al.*, 2019; Uslu, 2020, Mesgari *et al.*, 2020).

#### **Model Training**

The dataset was first divided into training datasets and testing datasets in the ratio 70:30 respectively. Each model was trained with the 70% of the dataset. Prior to the model training phase, normalization of all the dataset was carried out. The normalization process ensures that the effect of large data points do not mask data points with lower numerical values. The data normalization was carried out by mapping each row minimum and maximum values to a range between -1 and 1 such that all data values fall between these range. This was carried out using to the equation (3.31).

$$y = \frac{(y_{max} - y_{min})(x - x_{min})}{(x_{max} - x_{min})} + y_{min}$$
 3.32

These normalized data were used to train each of the models. A reverse normalization was carried out after each model training to obtain the original values of the data. The weight and bias of each ANN model were initialized and the models were trained with the developed architecture until a lower mean square error is obtained. A maximum number of iterations/epochs of 2000 was set. This is to allow for adequate training in case where increased number of iterations is needed before convergence. These original values, according to (Desai et al., 2008; Ansari et al., 2018; Nazerian et al., 2018; Ray et al., 2020) can be obtained using Equation 3.33

$$Y_j = \sum_{n=1}^{j} A_{IJ} B_{IJ} + K_J 3.33$$

Where Y = the total weight at a particular point

A =the weight at the previous node (s)

B =the weight attributed to the connector(s) linking the node(s)

K =value attributed to the bias

## **Model Testing**

Each model obtained as best for every response was tested using the test dataset (30% of the whole dataset). This process was carried out to investigate the effectiveness of the trained model on unfamiliar datasets. Inputs for each response variable are supplied to the model to predict the corresponding output and this were compared with the expected experimentally determined response graphically and statistically.

#### **Model Performance Evaluation**

Though graphical presentation of a comparison plot between the ANN predicted response and the experimentally determined response was plotted for each response, it is highly essential that the predicted results be statistically evaluated. This was carried out using known statistical performance metrics associated with regressive predictive models like ANN. To avoid bias, a nexus of statistical performance evaluation metrics was adopted for the model evaluation. These include the root mean square error (RMSE), mean absolute

deviation (MAD), mean absolute percentage error (MAPE), relative coefficient of variation (RCoV), and relative mean bias error (rMBE) (Adedeji *et al.*, 2020; Adeleke *et al.*, 2022). These are described in Equations (3.34-3.38):

$$RMSE = \sqrt{\frac{\sum_{k=1}^{N} (y_k - \hat{y}_k)^2}{N}}$$
 3.34

$$MAD = \frac{1}{N} \sum_{k=1}^{N} |y_k - \bar{y}_k|$$
 3.35

$$MAPE = \frac{1}{N} \sum_{k=1}^{N} \left| \frac{y_k - \hat{y}_k}{y_k} \right| \times 100\%$$
 3.36

$$rMBE = \frac{1}{N} \sum_{k=1}^{N} \left( \frac{\hat{\mathbf{y}}_k - \mathbf{y}_k}{\mathbf{y}_k} \right)$$
 3.37

$$RCoV = \frac{median|\hat{y}_k - y_{k-} \widehat{medium}|}{y_{k-} \widehat{medium}}$$
 3.38

Where N = number of experiment/Trials,

 $y_k$  = observed values of responses

 $\hat{y}_k$  = predicted Value of responses

 $y_k = \widehat{medium} = \text{median of the predicted responses}$ 

 $\bar{y}$  = mean value of responses

Further to this, the computational time (CT) of each of the model was also reported. This is very essential in determining the efficiency of each model. The number of iterations achieved before the stopping criterion was achieved was also reported

Graphs were plotted and models generated to describe the relationship between the varied parameters (Screw pitch, Die Diameter, pelleting speed, particle size as well the Kenaf-

starch proportion) and the perceived responses (which include Pelleting Efficiency, Percentage Recovery, Force at Peak (N), Deflection at Peak (mm), Energy to Peak (Nm), Force at Yield (N), Energy to Yield (Nm), Force to Break (N), Deflection at Break (mm), Energy to Break (Nm), Young Modulus, Durability, ratio of oil removed and the change in pH).

#### **Mathematical Models**

Mathematical models associating the responses (which include Pelleting Efficiency, Percentage Recovery, Force at Peak (N), Deflection at Peak (mm), Energy to Peak (Nm), Force at Yield (N), Energy to Yield (Nm), Force to Break (N), Deflection at Break (mm), Energy to Break (Nm), Young Modulus, Durability, ratio of oil removed and the change in pH) with the varied models using *Design Expert* response surface methodology central composite design (CCD) were also established and their respective coefficient of determination (R<sup>2</sup>) listed.

# CHAPTER FOUR RESULTS AND DISCUSSION

# 4.1. Machine Operation and Evaluation

The kenaf pelleting machine is an innovative designed and locally fabricated purposely for agglomeration of ground kenaf stems with the objective of improving handling. The efficiency of machine as well as the pellets were evaluated as specified in Sections 3.4-3.7.

# 4.1.1 Machine Description and Operation

The machine was fabricated as designed in Appendix I, consisting of the prime mover (Electric motor), a gearbox and the screw type pelletizer and shown in Plate 4.1. The machine is a 3HP, 3-phase electric motor. It was mounted below while the gearbox and pelletizer were positioned on the frame; both gearbox and pelletizer were connected to each other using sprockets and double chain, while electric motor transmitted power to the gearbox via belt and pulley.

The pelletizer was fabricated in conformation with the design calculations which comprises the hopper capacity, barrel size and length, belt and pulley assembly, the shaft size as well as minimum power requirement. The angle of repose of Kenaf-starch mixture on mild steel surface was found to be 50 degrees (50°) while the bulk density was calculated as 385 kg/m<sup>3</sup>.

Slight modifications were made to encompass variations in parameters associated with optimization, these include screw pitch, pelleting speed as well as Die Diameter. Variations in screw pitch were obtained by using augers constructed on removable pipes displayed in Plate 4.2. The cone had a single die in the form of a protruding pipe attached to it, this was to enhance the production of kenaf pellets. The openings on the cones were to accommodate variations in compliance with the experimental schedule in Table 3.1. Plate 4.3 shows some of the cones used for the production of kenaf pellets.



**Plate 4.1: The Pelletizer** 



Plate 4.2: Augers used in the Pelletizer.



Plate 4.3: Various Cones with the different Die Diameters

# **Pre-Optimization Evaluation of the Machine**

The pelletizer was appraised using equations 3.24 and 3.25. The following data were obtained from its evaluation.

milled kenaf mass = 200 g

Quantity of water added= 600 g

Quantity of Starch powder= 200 g

Mass of Extrudates = 1000 g

Broken pellets = 11.8 g

Pelletised mass = 536.9 g

Duration for extrudate to leave Hopper = 645 s

# **Pelleting Efficiency**

The Extruder's Pelleting Efficiency was

$$\eta \% = \frac{M_P}{M_O} \times 100\%$$

$$\eta \% = \frac{536.9}{1000} \times 100\%$$

$$\approx$$
 53.7%

## **Percentage Recovery**

Extruder's Percentage Recovery was

$$R(\%) = \frac{M_o}{M_T} \times 100\%$$

$$R\% = \frac{548.7}{1000} \times 100 \approx 54.9\%$$

#### 4.1.2 Pellet Production and Evaluation

The kenaf fibres were grounded with the aid of an electric motor-powered plate mill and sieved manually using 2.36 mm aperture sieves, the sieved kenaf was then mixed with water and raw cassava starch at specified mix ratio after which it was pelletized. The pelleting efficiency and percentage recovery were obtained from the equations postulated in Section 3.5. The pelleting efficiency and percentage recovery values served as criteria for evaluating the performance of the pelletizer. These values are recorded and tabulated in Table 4.4 while the calculated values for their trial were presented in Appendix II.

The obtained pellets were first weighed immediately after production (Plate 4.4), sun dried till the pellets were dried enough for storage (Plate 4.5) and then stored in a waterproof storage facility (Plate 4.6) before the tests could be carried out. Some of the pellets produced are shown in Plate 4.7.

Mechanical Properties of the stored pellets were determined using a computer controlled Universal Testing Machine (UTM) at the Forestry Research Institute of Nigeria, Jericho (Plate 4.8). Machine parameters determined included force at peak, force at yield, force at break, deflection at peak, deflection at Break, energy to peak, energy to yield and energy to break.

The durability of the pellets was also determined using a durability determining apparatus specifically designed and fabricated as specified in Appendix I. The durability index was mathematically obtained from Equation 3.26. The apparatus is portrayed in Plate 4.9. The values of the mechanical properties and durability index were recorded and presented in Table 4.5.



Plate 4.4: Weighing of Freshly Produced Kenaf Pellets



Plate 4.5: Sun Drying of Kenaf Pellets



Plate 4.6: Kenaf Pellets Ready for Storage



Plate 4.7: Dried Starch bonded Kenaf pellets

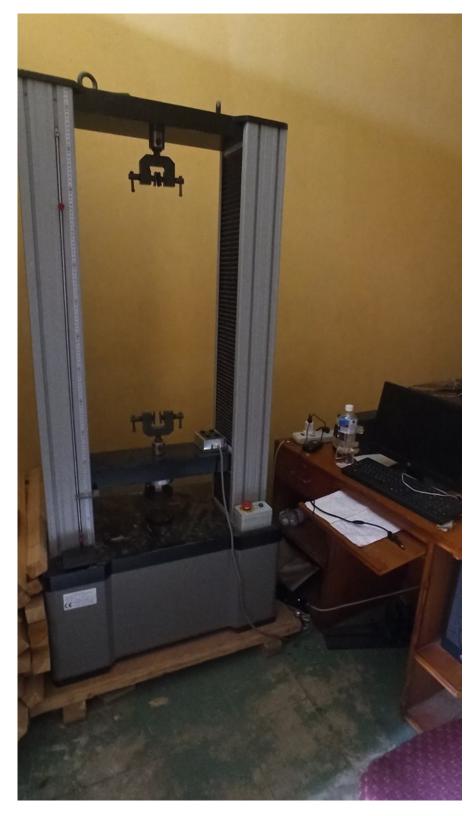


Plate 4.6: Computer Controlled Universal Testing Machine



**Plate 4.7: Durability Testing Apparatus** 

The Pellet samples were also evaluated based on their remediation ability. This was achieved by quantifying the extent of oil removal as well as the changes in the pH of the simulated oil polluted water as specified in Section 3.7. The ratio of oil and change in pH of the crude oil polluted water were recorded and tabulated in Table 4.2.

Plates 4.10-4.14 shows the stages involved in the remediation process. Plate 4.10 portrays the measured separate crude oil and water samples as specified in Section 3.7 while Plate 4.11 shows the simulated oil spill and the pH meter calibrated. Plate 4.12 shows the effect of Kenaf Pellets in the simulated oil spill.

Plate 4.13 is a pictorial representation of the oil precipitate obtained after the stabilization period and blank sample of the reagent, 1,1,1-trichloroethane while Plate 4.14 reveals the utilization of the HACH Dr 2000 Spectrometer taking readings of the oil content of the precipitate after using the blank sample as a buffer. These procedures were also recorded in Section 3.7.



Plate 4.8: Crude Oil and Water before Mixture



Plate 4.9: Simulated Oil Spilled water and Calibration of pH meter



**Plate 4.10: Remediation Process** 



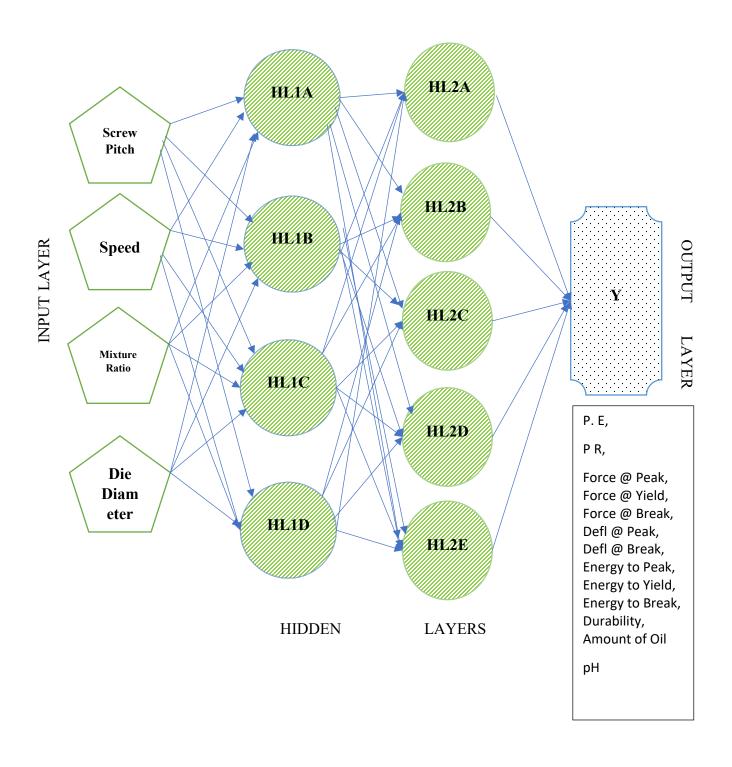
Plate 4.11: Oil Precipitate in Reagent and Blank Reagent Samples



Plate 4.12: Reading of Oil in Water PPM after Remediation.

## 4.1.3 ANN Data Analysis

Data analysis was carried out at 95% significant level ( $\alpha_{0.05}$ ). Two different topologies were utilized for this work, 4-3-5-1 and 4-4-5-1. The better was chosen for each of the responses. This signifies that there were 4 inputs (screw pitch, die diameter, pelleting speed and kenaf/binder ratio), two hidden layers (one with eight nodes and the other with nine nodes) and an output layer signifying each response (pelleting efficiency, percentage recovery, force at peak, force at yield, force at break, deflection at peak, deflection at break, energy to peak, energy to yield, energy to break, durability, amount of oil removed and changes in the pH)



**Figure 4.1: 4-4-5-1 ANN Topology** 

The values Mean Square Error (MSE), Root Mean Square Error (RMSE), Mean Absolute Deviation (MAD), Mean Absolute Percentage Error (MAPE), Relative Coefficient of Variation (RCoV) and Relative Mean Bias Error (rMBE) for both test and Train data were rounded off to the nearest 4<sup>th</sup> decimal figures and tabulated in Table 4.1 and 4.2. This was to ensure uniformity in presentation. Likewise, the values of computational time (CT) were approximated to the nearest 2 decimal figures in Table 4.3.

# 4.2. Effect of Optimization on Performance Evaluation of the Machine

The effect of variations in the operating and machine parameters were observed on the performance of the pelletizer in the form of the Pelleting Efficiency and the Percentage Recovery. The outcome of optimization on the performance of the machine is tabulated in Table 4.4.

## **Effect of Optimization on the Pelleting Efficiency**

There were significant changes in the Pelleting efficiency when different parameters were varied. These changes are portrayed in Tables 4.4, Figures 4.2 and 4.3 and further buttressed by the presence of statistical variables (MSE, RMSE, RCoV, rMBE, MAD as well as MAPE) as presented in Tables 4.1 and 4.2.

**Table 4.1: Performance Metrics ANN Test Data** 

Responses	MSE	RMSE	MAD	MAPE	RCoV	rMBE
1	11.4548	3.3845	2.6200	2.5484	0.0077	1.3798
2	32.4263	5.6945	4.4315	5.5122	0.0223	1.0984
3	4628.1640	68.0306	39.6251	20.2976	0.08465	-4.3775
4	0.1607	0.4009	0.2778	10.3853	0.1292	-4.3760
5	83.8048	9.1545	5.9634	45.0496	0.1400	5.1405
6	28291.7800	168.2016	108.7210	47.4964	0.0644	-3.4256
7	31.9233	5.6501	4.0513	29.7762	0.2294	12.0317
8	8032.2480	89.6228	48.5457	18.2933	0.1686	-1.5548
9	0.8873	0.9420	0.6514	6.5692	0.0911	-1.7706
10	65.9829	8.1230	6.4972	29.7330	0.3204	0.2958
11	0.0003	0.0164	0.0117	40.9374	0.0679	10.3731
12	0.2188	0.4678	0.3936	0.3911	0.0018	-0.0543
13	125545469.3	11204.7075	7384.2318	65.2924	0.1979	25.8221
14	0.0025	0.0499	0.0442	94.7241	0.1607	11.0226

**Table 4.2: Performance Metrics ANN Train Data** 

Responses	MSE	RMSE	MAD	MAPE	RCoV	rMBE
1	1.0989	1.0483	0.7086	0.7534	0.0077	-0.1131
2	6.0308	2.4558	1.8405	2.2948	0.0227	-0.1022
3	1092.5797	33.0542	19.9778	8.9534	0.0876	3.0942
4	0.4404	0.6637	0.4542	14.3729	0.1299	-1.9398
5	12.3819	3.5188	1.8487	12.1028	0.1400	-4.3036
6	1034.2850	32.1603	18.2839	5.4060	0.0720	4.0931
7	21.5056	4.6374	3.5255	24.2608	0.2193	4.9767
8	4886.7329	69.9052	49.2209	18.9076	0.1777	-2.7161
9	1.5668	1.2517	0.8637	12.4827	0.0979	0.4264
10	75.3938	8.6830	5.3992	19.0113	0.2586	-3.4170
11	3.363E-05	0.0060	0.0033	10.1792	0.0773	2.1212
12	0.0760	0.2756	0.1790	0.1626	0.0018	0.0692
13	83729290.61	9150.3711	4948.4256	27.0181	0.2969	-5.8588
14	0.0005	0.0232	0.0112	40.5258	0.1607	-0.3707

Table 4.3: Iterations, Computation Time and Topology for each Response

Responses	Iterations	CT (seconds)	Topology
1	28	2.09	4-3-5-1
2	15	2.15	4-3-5-1
3	24	1.99	4-3-5-1
4	38	1.93	4-3-5-1
5	73	2.16	4-3-5-1
6	137	1.24	4-3-5-1
7	22	1.12	4-4-5-1
8	22	1.17	4-4-5-1
9	24	1.14	4-4-5-1
10	12	2.30	4-3-5-1
11	16	2.27	4-3-5-1
12	20	2.14	4-3-5-1
13	67	2.25	4-3-5-1
14	15	2.12	4-3-5-1

**Table 4.4: Effect of Optimization on the Performance Evaluation of the Machine** 

	Factor 1	Factor 2	Factor 3	Factor 4	Response 1	Response 2
Run	A:Screw Pitch	B:Die Diameter	C:Pelleting Speed	D:Starch/Kenaf Ratio	Pelleting Efficiency	Percentage Recovery
	mm	mm	rpm		%	%
1	70	35	70	1.75	95.6	87.05
2	40	32.5	60	1.5	92.14	85.6
3	80	32.5	60	1.5	92.84	84.93
4	50	35	70	1.75	95.76	81.18
5	70	30	50	1.75	93.87	77.98
6	50	30	50	1.25	92.62	73.95
7	70	35	70	1.25	91.55	71.92
8	60	32.5	60	1.5	91.62	82.36
9	70	35	50	1.75	94.81	80.26
10	60	32.5	60	1.5	91.62	82.36
11	70	30	50	1.25	90.68	70.03
12	50	30	70	1.75	93.66	82.98
13	50	35	70	1.25	94.03	75.49
14	60	32.5	60	1	95.06	86.88
15	70	30	70	1.75	91.72	74.88
16	60	32.5	40	1.5	90.97	78.45
17	50	30	70	1.25	89.61	75.21
18	60	32.5	60	1.5	91.62	82.36
19	50	35	50	1.25	85.74	69.94
20	60	32.5	60	2	95.62	88.27
21	50	30	50	1.75	90.47	82.22
22	70	30	70	1.25	92.66	83.74
23	60	32.5	60	1.5	91.62	82.36
24	60	37.5	60	1.5	82.36	68.49
25	60	32.5	60	1.5	91.62	82.36
26	70	35	50	1.25	85.96	70.85
27	50	35	50	1.75	90.63	87.36
28	60	27.5	60	1.5	88.76	82.59
29	60	32.5	60	1.5	91.62	82.36
30	60	32.5	80	1.5	92.46	73.89

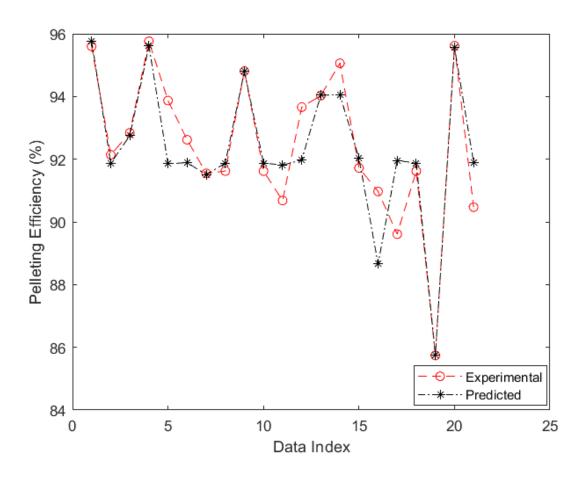


Figure 4.2: Pelleting Efficiency Training Data

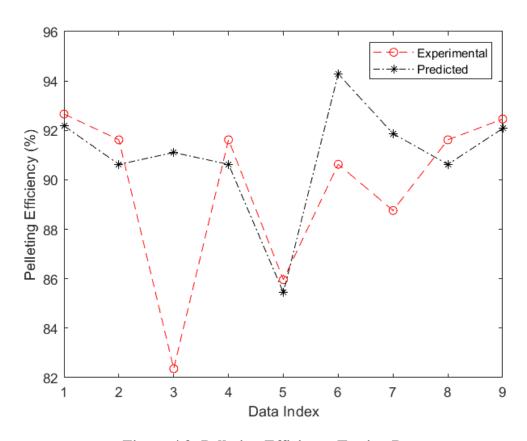


Figure 4.3: Pelleting Efficiency Testing Data

Pelleting Efficiency was peak at 95.76 and least at 85.74% signifying a range exceeding 10%. The maximum predicted Pelleting efficiency for data obtained from training was 95.77% and 94.28% for data testing. Similarly, the least predicted Pelleting Efficiency resulting from training data was 85.74% which corresponded to the least experimental test for the same trial and 85.44% for data used for testing the ANN model.

The ANN analysis was done in a computational time of 2.09s, using 28 iterations as well as an array of 4-3-5-1 (Table 4.3) and thereby yielded MSE values of 1.0989 and 11.4548 for predicted training and testing data respectively, RMSE values of 1.0483 and 3.3845 for data training and data testing respectively. Similarly, MAD values of 0.7086 and 2.6200 were also obtained for both training and testing of data in a corresponding manner while MAPE for both training and testing data yielded 0.7534 and 2.5484 respectively. RCoV values were 0.0077 for both training and testing data whereas rMBE values were -0.1131 and 1.3798 for data training and data testing congruently.

From Table 4.4, it is also pertinent to note that higher binder ratio resulted in higher Pelleting efficiency in the trials evidenced in the comparison of Runs #1 and #7, Runs #4 and #13 and Runs #20 and #8. However, the law of diminishing returns applies at a certain stage predicted at 95.77% Pelleting Efficiency. This is similar to findings in (Radeva *et al.*, 2018).

Table 4.4 also portrays the effects on Die Diameter on the Pelleting Efficiency noticeable in the pair of Runs #6 and #19 where there is a decrement from 92.62% to 85.74%, Runs #15 and #1 presented an increment of 3.88% from 91.72% to 95.6% while a comparison of Runs #28 and #10 revealed an increment of 2.86% from 88.76% to 91.62%. It can be argued that an increase in Die Diameter would present an increase in Pelleting efficiency under optimal conditions.

Table 4.4 presents the effects of Pelleting Speed on the pelleting efficiency. A study of Runs #16 and #18 reveals an increase from 90.97% to 91.62%, likewise Runs #19 and #13 showed a rise from 85.74% to 94.03% while Runs #5 and #15 indicated a decline from 93.87% to 91.72%.

Another study of Table 4.4 with the aim to investigate the effect of screw pitch on the pelleting Efficiency commencing with Runs #2 and #25 reveals a decline from 92.14% to

91.62%, Runs #6 and #11 92.62% to 90.68% whereas Runs #2 and #3 showed an increment from 92.14% to 92.84%.

In general, the pelleting efficiency of pelleting kenaf was above 85% which is synonymous with the pelleting efficiency for wood pellets (Whittaker and Shield, 2017; Tumuluru, 2018; Wang *et al.*, 2020).

# **Effect of Optimization on the Percentage Recovery**

The Percentage Recovery of extrudates during the pelleting process also varied significantly during optimization as evidenced in Table 4.4, Figures 4.4 and 4.5 and bolstered by values for RMSE, MSE, MAPE, MAD, RCoV and rMBE shown in Table 4.1 and 4.2.

Experimental values for Percentage Recovery were between 68.49% and 88.27% culminating in a difference of nearly 20% while the predicted values were 87.75% maximum and 68.42% minimum for trained data and 87.56% maximum and 75.59% minimum for tested data using the ANN model.

The ANN analysis for Percentage Recovery was achieved within a Computational Time (CT) of 2.15s using 15 iterations and a network arrangement 4-3-5-1. Obtained MSE values were 6.0308 and 32.4263 for trained data and test data respectively while The RMSE was 2.4558 and 5.6945 for data training and testing congruently. Similarly, rMBE values of -0.1022 and 1.0984 were recorded for both data training and data testing while RCoV values of 0.0227 and 0.0223 were documented for trained data and tested data sets correspondingly. Finally, MAPE values were 2.2948 and 5.5122 for data training and data testing respectively whereas MAD values of 1.8405 and 4.4315 for trained data and tested data were respectively registered.

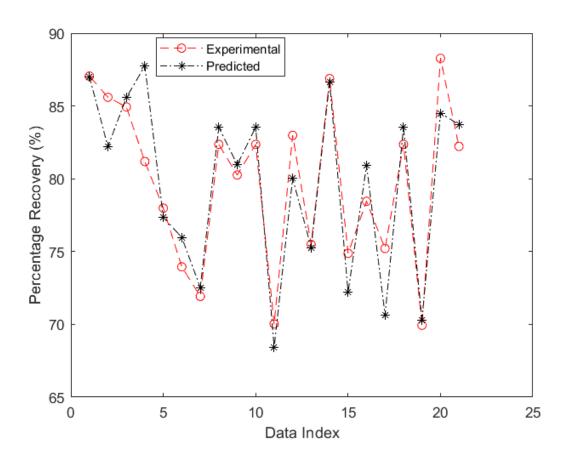
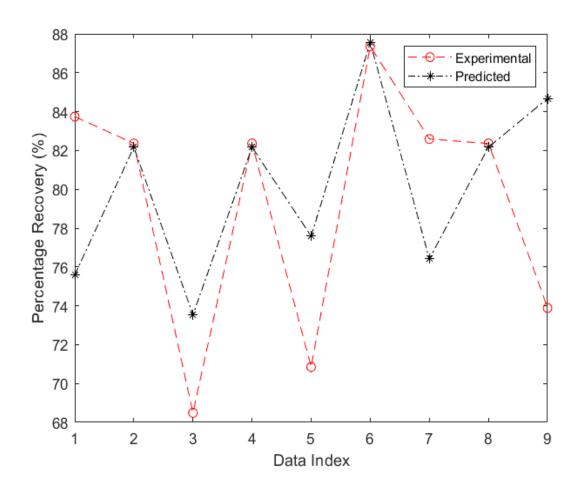


Figure 4.4: Percentage Recovery Training Data



**Figure 4.5: Percentage Recovery Testing Data** 

A study of Table 4.4 indicates screw pitch exhibited curve-like effect on the percentage recovery when other factors were constant, this is visible in the examination of Runs #2, #8 and #3 where there was an initial decline from 85.6% to 82.36% and then a rise to 84.93% for a simultaneous increase in screw pitch from 40 mm to 60 mm and 80 mm. However, a comparative study of #19 and #26 reveals a rise from 69.94% to 70.85% while comparison of Runs #21 and #5 indicated a decline of over 4% from 82.22% to 77.98% both occurring for an increment from 50 mm to 70 mm.

Table 4.4 also displays the consequences of varying Die Diameter on the percentage recovery noticeable from the pair of Runs #28 and #8 where a slight reduction from 82.59% to 82.36% was indicated, Runs #6 and #19, waning of 4% from 73.95% to 69.94% and Runs #12 and #4 revealed a decrease from 82.98% to 81.18%. Thus, implying a tendency in reduction of percentage recovery for increment in Die Diameter.

From Table 4.4, it can also be deduced that the pelleting speed was also significant on the percentage recovery with a rise evidenced in Runs #16 and #29 from 78.45% to 82.36%, Runs #6 to #17 from 73.95% to 75.21% and Runs #11 and #22 from 70.03% to 83.74% for corresponding increment in pelleting speed. Thus, inferring those changes in the pelleting speed is directly proportional to changes in Percentage recovery.

Finally, information from Table 4.4 correlating Kenaf/starch ratio and percentage recovery can be envisaged in examining Runs #14, #25 and #20 reporting an initial decline from 86.88% to 82.36% and then a rise to 88.27%, Runs #6 and #21 portrayed an incline from 73.95% to 82.22% and similarly, Runs #17 and #12 reflected an increase from 75.21% to 82.98% for increment in Kenaf/Binder ratio. It can thus be deduced that there is a direct correlation between the Kenaf/binder ratio and the Percentage Recovery.

The pelleting machine also portrayed a Percentage Recovery exceeding 68%, thus implying that majority of the extrudate were recovered despite moisture loss due to heat and residual matter trapped within the extruder after operation.

# 4.3. Effect of Optimization on the Mechanical Properties and Durability of the Pellets

The consequences in varying the various machine and operating parameters on the mechanical properties and durability of kenaf pellets were displayed in Table 4.5, the mechanical properties studied include the Forces (Peak, yield and Break), Deflection (Peak and Break), Energy to attain (Peak, Yield and Break) and Durability.

#### **Effects of Optimization on Force at Peak**

Force determined at peak of the pellets varied based on the values of the parameters used in producing the pellets. These values are displayed by the combination of Table 4.5 and Figures 4.6 and 4.7. The effect of these variations on the Force at peak is reinforced by the presence of statistical metrics MSE, RMSE, MAD, MAPE, RCoV and rMBE portrayed in Tables 4.1 and 4.2.

Experimental data were within the range of 84-280 N resulting in a difference of almost 200 N while the predicted maximum and minimum values for data training were 259.46 N and 84 N respectively while predicted maximum and minimum values for data testing were 284.39 N and 69.60 N congruently.

The ANN analysis for Force at peak was conducted within a computational time (CT) of 1.99s using 24 iterations and an analysis situs of 4-3-5-1, Calculated MSE values were 1092.5797 and 4628.1640 for data training and data testing respectively while RMSE values were 33.0542 and 68.0306 for trained and tested data congruently whereas MAD values were 19.9778 and 39.6251 for training data and testing data correspondingly. MAPE values of 8.9534 and 20.2976 were obtained for training data and testing data harmoniously with RCoV values for trained data and tested data pegged at 0.0876 and 0.08465 respectively and rMBE values of 3.0942 and -4.3775 were obtained for training and testing data congruently.

An examination of Table 4.5 with respect to changes in force at peak due to variations in Kenaf /binder ratio, a comparison of Runs #14, #8 and #20 indicates a rise in force determined at peak point from 140 N to 228 N and subsequently 252 N. Similarly, a comparative study of Runs #7 and #1 depicts an increment from 116 N to 172 N and Runs

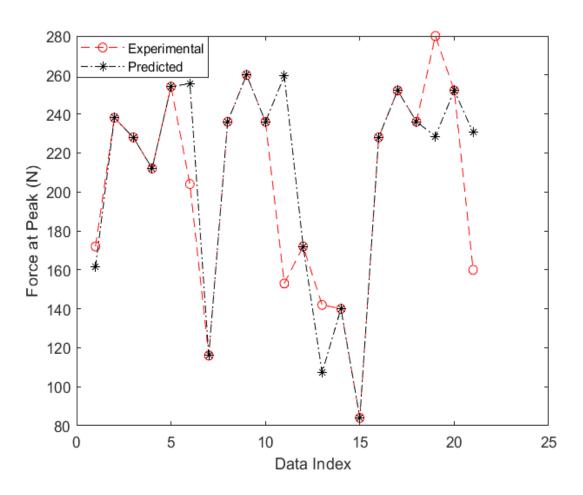


Figure 4.6: Force at Peak Training Data

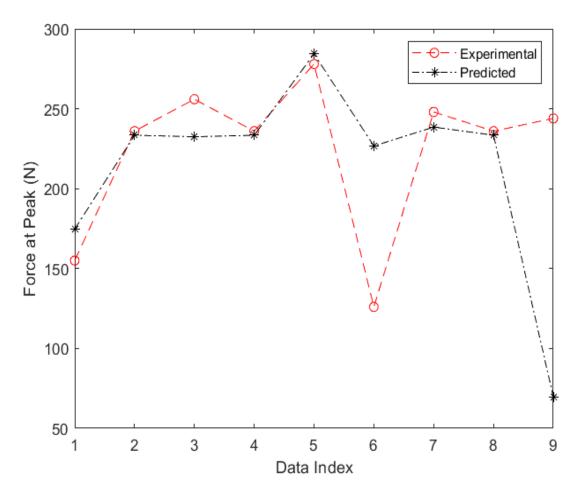


Figure 4.7: Force at Peak Testing Data

#11 and #5 portrayed an increase from 153 N to 254N, all occurring with simultaneous increment in amount of binder.

Table 4.5 also shows the correspondence between the screw pitch and Force at peak evidenced in the comparison of Runs #2, #10 and #20 depicting a decline from 238 N to 236 N and then 228 N, similarly, Runs #13 and #7 showed a reduction of 26 N in Force at peak from 142 N to 116 N as well as Runs #6 and #11 with a decline from 204 N to 153 N for corresponding increase in a screw pitch, thus signifying an inverse relationship between the screw pitch and Force at Peak.

Information from Table 4.5 can also be used to describe the relationship between the pelleting speed and the force at peak as indicated in the assessment of Runs #16, #18 and #30 displaying an increment from 228 N to 236 N and a subsequent figure of 244N, Runs #27 and #4 showed a rise from 126 N to 212 N. However, a decline from 280 N to 142 N was observed in the evaluation of Runs #19 and #13, all of which were reported for increase in Pelleting Speed.

Finally, Table 4.5 with regards to changes in Forces at peak caused variations in Die Diameter visualised in the evaluation of Runs #28, #8 and #24 which displayed an initial decline from 248 N to 236 N and then a surge to 256 N, Runs #21 and #27 portrayed a decline from 160 N to 126 N whereas a comparison of Runs #6 and #19 depicted a rise from 204 N to 280 N.

#### **Effects of Optimization on Deflection at Peak**

The deflection at Peak also varied when the various parameters were varied as portrayed in Table 4.5. Figures 4.8 and 4.9 depicts the plots of trained and tested data respectively. The presence of these variations is evidenced by the availability of the statistical metrics rMBE, MSE, RMSE, RCoV, MAPE and MAD as displayed in Tables 4.1 and 4.2.

The ANN analysis was achieved within a Computational Time (CT) of 1.93s using 38 iterations and a topology of 4-3-5-1 as presented in Table 4.3. Tables 4.1 and 4.2 depicted values of the MSE as 0.4404 and 0.1607 for trained and tested data accordingly, RMSE as 0.6637 and 0.4009 for training data and testing data congruently, MAD as 0.4542 and 0.2778 for training data and testing data simultaneously, MAPE as 14.3729 and 10.3853 for

trained data and tested data harmoniously, RCoV values as 0.1299 and 0.1292 for both trained and tested data harmoniously and rMBE values as -1.9398 and -4.3760.

Experimental values for deflection at peak was 2.008 mm minimum and 5.481 mm maximum, culminating in a range of 3.473 mm, maximum predicted test values 4.048 mm while the minimum was 2.179 mm (range of 1.869 mm) where as predicted training values were 4.043 mm for maximum and 2.286 mm minimum (1.757mm).

Table 4.5 portrays the link between the deflection at peak and the changes in Kenaf/starch ratio, this is evidenced in Run #14, #18 and #20 where a continuous rise from 2.008 mm to 3.517 mm and then 3.813 mm was recorded. The scenario was also similar for Runs #11 and #5 where an increment from 2.418 mm to 3.497 mm was registered. However, the pair of Runs #22 and #15 reported a decline from 3.4081 mm to 2.378 mm.

Table 4.5 also shows the relationship between the pelleting speed and the deflection at peak as indicated in the assessment of Runs #16, #8 and #30; there was an incline from 3.368 mm to 3.517 mm and subsequently 3.628 mm deflection recorded at peak point for the pellets. This is also similar to Runs #11 and #22 which also witnessed a rise from 2.418 mm to 3.041 mm.

Table 4.5 also displays the correlation between the diameter of the pellets and the deflection, specified in the examination of Runs #28, #29 and #24 with a reported rise from 2.705 mm to 3.517 mm and 3.649 mm, similarly, Runs #11 and #26 registered an increment from 2.418

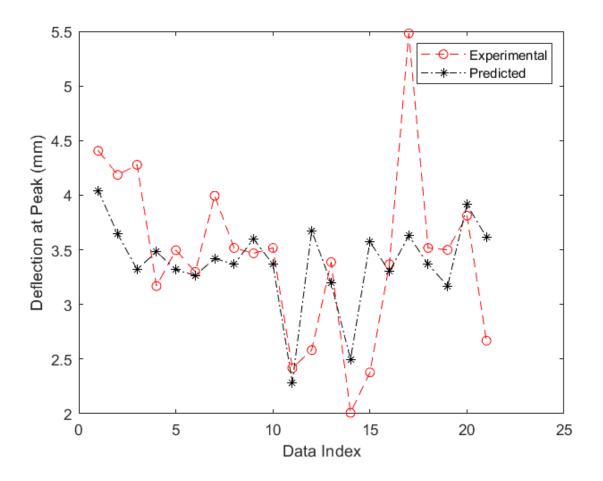


Figure 4.8: Deflection at Peak Training Data

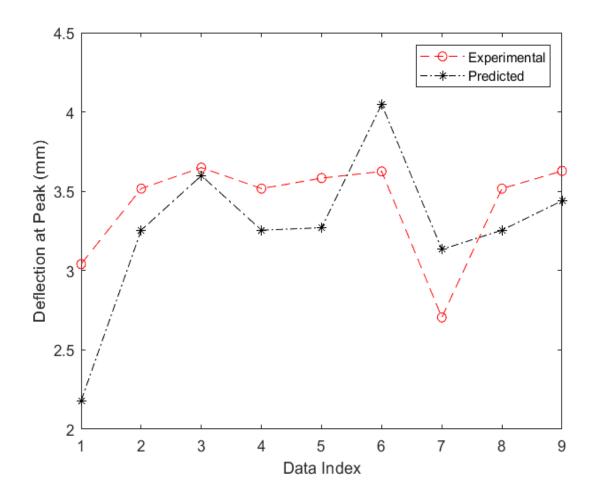


Figure 4.9: Deflection at Peak Testing Data

mm to 3.583 mm and Run #21 and #27 revealed a surge from 2.668 mm to 3.625 mm deflection at the point of peak.

Finally, with respect to Deflection at Peak, Table 4.5 reveals the association of changes in the screw pitch on the deflection suffered by pellets at the peak point as realised from Runs #2, #29 and #3 with a recorded fall from 4.186 mm to 3.517 mm followed by a rise to 4.278 mm. Runs #17 and #22 also had a decline from 5.481 mm to 3.041 mm likewise the comparison of Runs #6 and #11 which revealed a decline from 3.928 mm to 2.418 mm.

# Effects of Optimization on Energy to Peak

The energy to peak for starch bonded kenaf pellets also varied significantly for each of the pellets, these were presented by the combined utilization of Table 4.5 and Figures 4.10 and 4.11, while the variations are bolstered by the presence of MSE, RMSE, MAPE, MAD, RCoV and rMBE variables displayed in Tables 4.1 and 4.2.

Maximum experimental value was 29.074 Nmm/s while the minimum was 4.927 Nmm/s exhibiting difference of 24.147 Nmm/s. Predicted maximum values were 29.306 Nmm/s and 34.891 Nmm/s for data training and data testing respectively while predicted minimum values were 9.287 Nmm/s and 4.891 Nmm/s.

ANN analysis for Energy to peak was obtained within a Computational Time (CT) of 2.16s using seventy-three (73) iterations and a topology of 4-3-5-1. MSE values of 12.3819 and 83.8048 for data training and data testing respectively while RMSE values were 3.5188 and

**Table 4.5: Mechanical Propeties and Durability of Kenaf Pellets** 

	Fact or 1	Fact or 2	Facto r 3	Factor 4	Resp onse 3	Respo nse 4	Resp onse 5	Resp onse 6	Resp onse 7	Resp onse 8	Respo nse 9	Resp onse 10	Resp onse 11	Resp onse 12
R un	A:Sc rew Pitch	B:Di e Diam eter	C:Pell eting Speed	D:Starch/ Kenaf Ratio	Force @ Peak	Defle ction @ Peak	Energ y to Peak	Force @ Yield	Energ y to Yield	Force  @ Break	Defle ction @ Break	Energ y to Break	Youn g Modu lus	Durab ility
	mm	mm	rpm		N	mm	Nmm /s	N	Nmm /s	N	mm	Nmm /s	$\frac{N}{m}$	%
1	70	35	70	1.75	172	4.406	14.31	174	14.47 9	178	4.925	14.79 8	0.022 5	98.86
2	40	32.5	60	1.5	238	4.186	15.68 2	266	19.10 4	285	9.812	23.86 1	0.046 2	99.56
3	80	32.5	60	1.5	228	4.278	16.56 3	254	21.88 9	272	9.624	24.44 2	0.045 8	99.16
4	50	35	70	1.75	212	3.168	7.301	340	16.80 1	388	8.671	21.29	0.030 5	98.3
5	70	30	50	1.75	254	3.497	17.86 4	288	22.95 8	324	9.58	27.00 8	0.041 2	98.76
6	50	30	50	1.25	204	3.298	14.62 6	248	16.07 6	277	8.564	19.70 9	0.034 8	98.64
7	70	35	70	1.25	116	3.994	9.642	120	9.996	190	9.907	15.82	0.011 9	99.42
8	60	32.5	60	1.5	236	3.517	13.20 7	284	15.36 9	292	9.483	16.85 2	0.051 5	98.7
9	70	35	50	1.75	260	3.468	18.13 7	291	22.59	313	9.638	25.56 9	0.042 6	99.24
10	60	32.5	60	1.5	236	3.517	13.20 7	284	15.36 9	292	9.483	16.85 2	0.051 5	98.7
11	70	30	50	1.25	153	2.418	8.408	181	10.04	223	6.385	14.00 7	0.036	98.62

12	50	30	70	1.75	172	2.582	12.28 4	242	19.26 3	266	8.823	22.01 2	0.047 6	99.43
13	50	35	70	1.25	142	3.387	11.82 9	180	14.98 7	276	8.513	22.96 8	0.024 2	98.15
14	60	32.5	60	1	140	2.008	6.538	163	8.226	204	7.881	11.62 9	0.031 6	98.55
15	70	30	70	1.75	84	2.378	4.927	108	6.745	142	3.89	9.421	0.043	98.96
16	60	32.5	40	1.5	228	3.368	16.69 4	272	20.36	309	8.796	24.80 7	0.042 7	99.26
17	50	30	70	1.25	252	5.481	14.57 2	252	13.71 8	252	5.856	14.86 4	0.055	99.31
18	60	32.5	60	1.5	236	3.517	13.20 7	284	15.36 9	292	9.483	16.85 2	0.051 5	98.7
19	50	35	50	1.25	280	3.5	27.09 8	342	32.17 3	504	9.932	44.18 6	0.046 1	99.26
20	60	32.5	60	2	252	3.813	29.07 4	318	36.81 8	486	10.23 8	48.57 4	0.056 4	99.72
21	50	30	50	1.75	160	2.668	12.50	234	18.92	256	8.244	20.87 9	0.048 6	99.63
22	70	30	70	1.25	155	3.041	9.82	181	12.18 7	227	8.686	15.99 4	0.039	98.84
23	60	32.5	60	1.5	236	3.517	13.20 7	284	15.36	292	9.483	16.85 2	0.051 5	98.7
24	60	37.5	60	1.5	256	3.649	19.06 4	328	31.66	440	8.993	45.74 2	0.026 2	98.69
25	60	32.5	60	1.5	236	3.517	13.20 7	284	15.36 9	292	9.483	16.85 2	0.051 5	98.7
26	70	35	50	1.25	278	3.583	25.84 2	336	29.40 8	402	9.982	38.85	0.048	99.54
27	50	35	50	1.75	126	3.625	10.03 1	184	14.90 8	196	7.55	15.98 2	0.018 5	98.56

28	60	27.5	60	1.5	248	2.705	9.115	264	11.26	274	7.965	12.99	0.049	99.72
29	60	32.5	60	1.5	236	3.517	13.20	284	O		9.483	•	-	98.7
20							7		9			2	5	
30	60	32.5	80	1.5	244	3.628	19.24 4		23.61 8		9.642			99.12

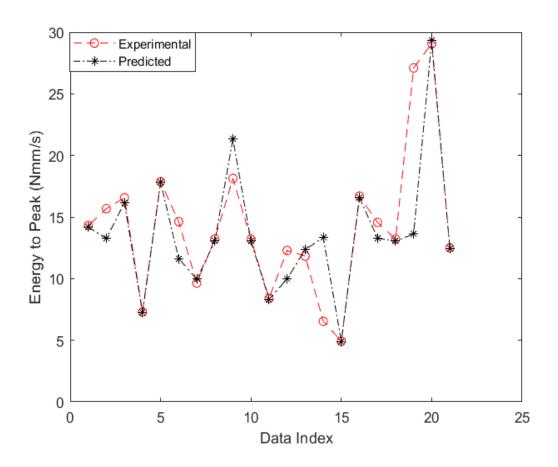


Figure 4.10: Energy to Peak Training Data

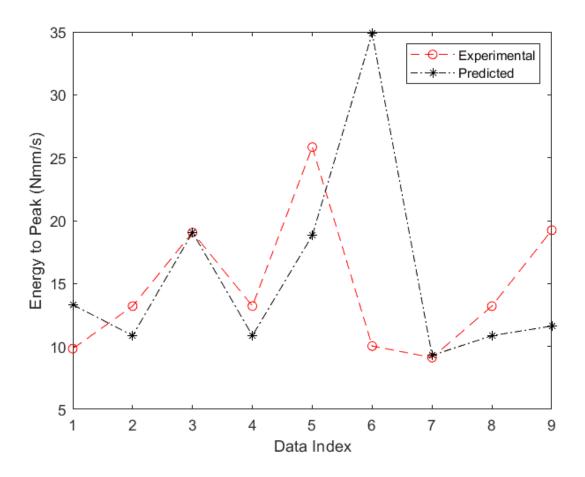


Figure 4.11: Energy to Peak Testing Data

9.1545 for trained and tested data congruently whereas MAD values were 1.8487 and 5.9634 for data training and data testing respectively. MAPE values were 12.1028 and 45.0496 for trained data and tested data while RCoV values were 0.1400 for both data training and testing respectively whereas rMBE values were -4.3036 and 5.1405 for both trained and tested data congruently.

Table 4.5 provides information on the relationship between the screw pitch and the energy to peak as evident in the comparison of Runs #2, #25 and #3 with an initial decline from 15.682 Nmm/s to 13.207 Nmm/s before a rise to 16.563 Nmm/s. similarly, a reduction was also recorded from 12.284 Nmm/s to 4.927 Nmm/s in the study of Run #12 and #15, while Run #4 and #1 yielded an increment from 7.301 Nmm/s to 14.313 Nmm/s.

Table 4.5 is also fortified with details associating the Die Diameter with the energy required to reach the peak of the pellets. This is manifest in the comparative study of Runs #28, #18 and #24 with an increase from 9.115 Nmm/s to 13.207Nmm/s and furthermore 19.064 Nmm/s. Similarly, arise from 17.864 Nmm/s to 18.137 Nmm/s was reported when #5 and #9 were compared. Likewise, an incline from 8.408 Nmm/s to 25.842 Nmm/s was recorded from a comparative study of Runs #11 and #26 for a purported rise in the screw pitch.

Table 4.5 also portrays the correlation between the pelleting speed and changes in energy to peak of kenaf starch bonded pellets as obvious in the assessment of #16, #18 and #30 with an initial decline from 16.694 Nmm/s to 13.207 Nmm/s before a surge to 19.244 Nmm/s. Runs #21 and #12 also reveals a reduction from 12.502 Nmm/s to 12.284 Nmm/s, likewise Runs #6 and #17 also reported a minor slump from 14.626 Nmm/s to 14.572 Nmm/s, thus signifying a decline in the energy to peak for an increase in the pelleting speed.

Lastly, Table 4.5 reveals the association between the amount of binder and the energy to break, which is apparent from the comparative study of #14, #18 and #20 with a reported increment from 6.538 Nmm/s to 13.207 Nmm/s and subsequently 29.074 Nmm/s. However, a decline from 14.626 Nmm/s to 12.502 Nmm/s was reported for the pair of Runs #6 and #21 congruently which is also similar to a decline from 25.842 Nmm/s to 18.137 Nmm/s reported for Runs #26 and #9.

#### **Effects of Parameter Variation on Force at Yield**

The determination of force at yield was also found to be affected significantly by the varied parameters with details presented in Table 4.5 and Figures 4.12 and 4.13. The presence of these changes in values are confirmed by the values of MSE, RMSE, MAD, MAPE, RCoV and rMBE duly presented in Tables 4.1 and 4.2.

Experimental values for force at yield climaxed at 342 N and a minimum of 108 N with a difference of 234 N. Predicted Trained data peaked at 416.12 N and a least value of 120 N whereas predicted tested data peaked at 508.568 N and least at -264.263 N.

Table 4.3 reveals that the ANN analysis was completed in 1.24s using One hundred and Thirty-seven (137) iterations in a 4-3-5-1 topology. MSE values of 1034.2850 and 28291.7800 for data training and data testing congruently while RMSE of 32.1603 and 168.2016 were obtained for trained and tested data respectively whereas MAD values of 18.2839 and 108.7210 were recorded for trained and tested data. MAPE were 5.4060 and 47.4964 for data training and data testing harmoniously while obtained values for RCoV remained at 0.0720 and 0.0644 for trained data and tested data whereas rMBE were 4.0931 and -3.4256 for data training and data testing.

Experimental values reported in Table 4.5 depicts the linkage between the Force at yield and the amount of binder added as evidenced in the comparative study of Run #14, #10 and #20, where a continual rise was recorded from 163 N to 284 N and subsequently 318 N. Similarly, there was an increase of 54 N from 120 N to 174 N when assessing #7 and #1 just as Runs #11 and #5 reported an incline of 107 N from 181 N to 288 N, thereby signifying that there was an increase in Force at yield for an increment in amount of binder.

Table 4.5 also shows the association between the pelleting speed and force registered to stress the pellets to yield point, depicted by the study of #16, #25 and #30 with an initial rise from 272 N to 284 N followed by a slump back to 272 N. Runs #11 and #22 generated

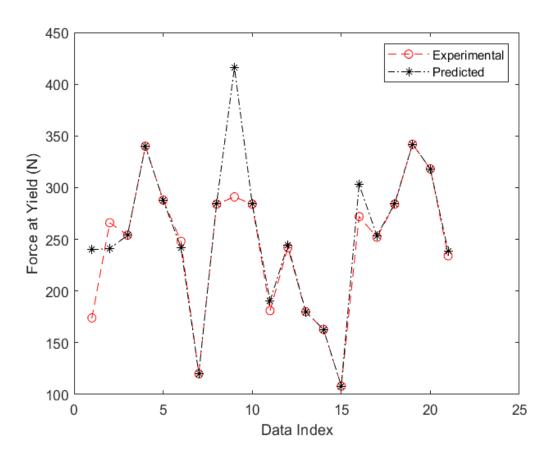


Figure 4.12: Force at Yield Training Data

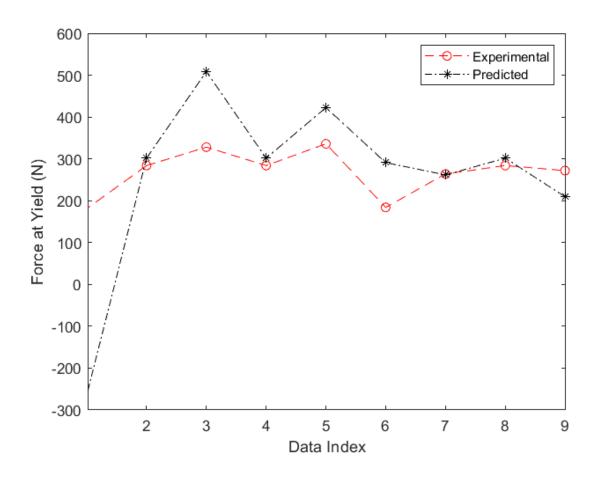


Figure 4.13: Force at Yield Testing Data

pellets with the same force at yield at 181 N while Runs #26 and #7 revealed a decline from 336 N to 120 N, all of which occurred when the pelleting speed was increased.

It is also pertinent to note that there is a correlation between the Die Diameter and the force at yield as displayed in Table 4.5, Runs #28, #25 and #24 portrayed a continual increment from 264 N to 284 N and subsequently 328 N; Runs #6 and #19 produced a similar trend with a rise from 248 N to 342 N likewise Runs #5 and #9 recorded a slight rise from 288 N from 291 N. All of which resulted with an increase in the diameter of pellets.

Finally, Table 4.5 reveals the relationship between the screw pitch and Force at yield for kenaf starch bonded pellets visualized in the comparative study of Runs #2, #8 and #3 which revealed an initial rise from 266 N to 284 N and a subsequent decline to 254 N, Runs #21 and #5 displayed an increment from 234 N to 288 N whereas Runs #6 and #11 depicted a decline from 248 N to 181 N, all of which were reported for an increase in the Screw Pitch.

## **Effects of Optimization on Energy to Yield**

The energy required to stress the pellets to yield point was also determined to be affected by variations in machine and operating parameters as visualized in Table 4.5 as well as Figures 4.14 and 4.15. The presence of the effects were supported by the values of RCoV, MAPE, rMBE, MAD, MSE and RMSE portrayed in Tables 4.1 and 4.2.

Experimental values for energy to yield was maximum at 36.818 Nmm/s and minimum at 6.745 Nmm/s. Predicted maximum and minimum values were 36.652 Nmm/s and 11.836 Nmm/s for data training and 33.926 Nmm/s and 11.432 Nmm/s for data testing respectively.

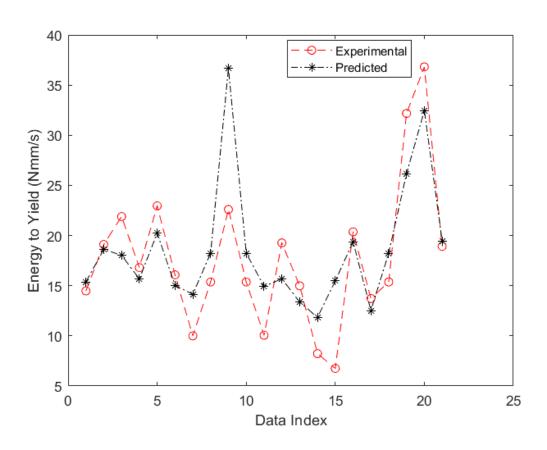


Figure 4.14: Energy to Yield Training Data

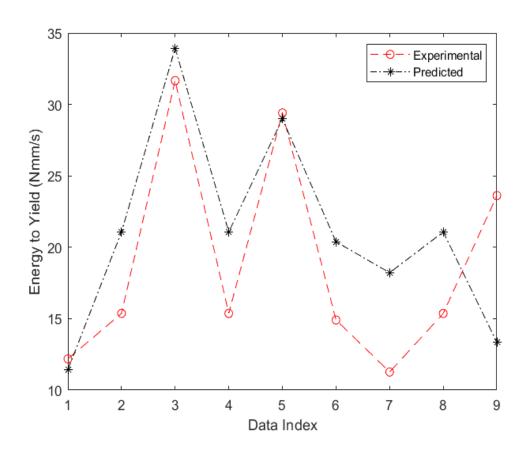


Figure 4.15: Energy to Yield Testing Data

The ANN analysis of energy to yield was achieved in a computational time (CT) of 1.12s using twenty-two (22) iterations and a network situs of 4-4-5-1 as displayed in Table 8. MSE values of 21.505 and 31.9233 were recorded for trained and tested data respectively, RMSE of 4.6374and 5.6501 for data training and data testing congruently, MAD of 3.5255 and 4.0513 were obtained for trained and tested data simultaneously. MAPE values of 24.2608 and 29.7762 were reported for data that has been trained and tested respectively, RCoV of 0.2193 and 0.2294 for data training and data testing simultaneously while rMBE were 4.9767 and 12.0317 for trained data and tested data respectively.

Table 4.5 comprises information associating the consequences of changing the screw pitch on the changes in energy expended to press the pellets to yield point. This is visible in the assessment of Runs #2, #18 and #3 which reveals a dip from 19.104 Nmm/s to 15.369 Nmm/s and a surge to 21.889 Nmm/s. Runs #27 and #9 reported a rise from 14.908 Nmm/s to 22.593 Nmm/s, likewise #21 and #5 where an increment from 18.920 Nmm/s to 22.958 Nmm/s was also recorded.

Table 4.5 also portrays the correlation between the Die Diameter and the energy to yield of the pellets as evidenced in the comparative study of Runs #28, #29 and #24 which revealed the incessant rise from 11.268 Nmm/s to 15.369 Nmm/s to 31.668 Nmm/s, likewise a rise from 13.718 Nmm/s to 14.987 Nmm/s was recorded for the assessment of Runs #17 and #13 whereas the study of Runs #21 and #27 revealed a decline from 18.92 Nmm/s to 14.908 Nmm/s. All of which occurred with an increase in Die Diameter.

Table 4.5 is also fortified with details on the relationship between the pelleting speed and the energy to yield as seen in the comparative study of #16, #18 and #30 with an initial decline from 20.366 Nmm/s to 15.369 Nmm/s followed by a surge to 23.618 Nmm/s. A slump from 22.958 Nmm/s to 6.745Nmm/s was reported for the comparison of Runs #5 and #15 whereas a rise was recorded during the comparative study of Runs #11 and #22 from 10.049Nmm/s to 12.187Nmm/s.

Data from Table 4.5 also provides evidence pertaining to the quantity of binder and its subsequent effect on the energy to yield of the pellets as noticed in the investigation of Runs #14, #18 and #20 which displayed a continuous rise from 8.226 Nmm/s to 15.369 Nmm/s to 36.818 Nmm/s. Similarly, the comparison of Runs #11 and #5 yielded a surge from

10.049Nmm/s to 22.958 Nmm/s likewise that of Runs #6 and #21 which revealed a rise from 16.076Nmm/s to 18.92 Nmm/s.

# **Effects of Optimization on Force at Break**

The force required to break the kenaf pellets was another result reported to be disrupted by the variations in the parameters evaluated during the pelleting of kenaf stem particles Information regarding the Force at peak are displayed by the amalgamation of Table 4.5 and the pair of Figures 4.16 and 4.17. The presence of these effects was reinforced by the values of statistical metrics MSE. RMSE, MAD, MAPE, RCoV and rMBE.

Experimental data on the force to break provides minimum and maximum values of 142 N and 504 N respectively with a range of 362 N. Predicted maximum and minimum values were 429.296 N and 214.755 N for trained data respectively while the predicted maximum and minimum values for tested data were 415.434 N and 215.025 N congruently.

The ANN analysis was conducted within a computational time (CT) of 1.17s using twenty-two (22) iterations and a topology of 4-4-5-1 (Table 8). MSE values of 4886.7329 and 8032.2480 for trained and tested data respectively, RMSE of 69.9052 and 89.6228 for data training and data testing simultaneously, MAD of 49.2209 and 48.5457 for data training and data testing congruently, MAPE of 18.9076 and 18.2933 for trained data and tested data harmoniously, RCoV of 0.1777 and 0.1686 for data training and data testing respectively while rMBE values were -2.7161 and -1.5548.

Table 4.5 is garnished with details unfolding the relationship between the force at break and the amount of binder used in the pelleting process as shown in the comparative study of Runs #14, #18 and #20 where there was continual increment from 204 N to 292 N to 486 N. However, Runs #6 and #21 revealed a dip from 277 N to 256 N just as another dip was recorded from 190 N to 178 N when Runs #7 and #1 when compared.

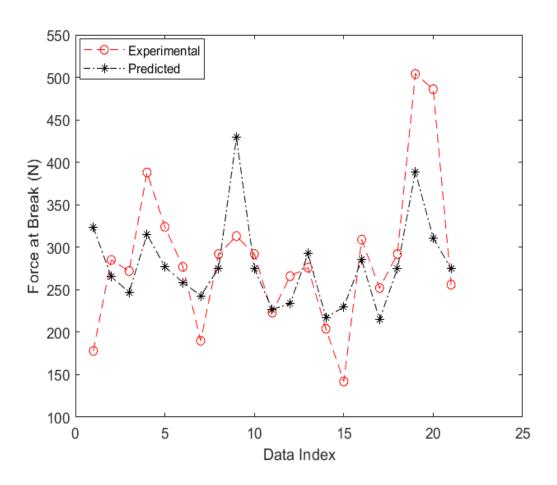


Figure 4.16: Force at Break Training Data

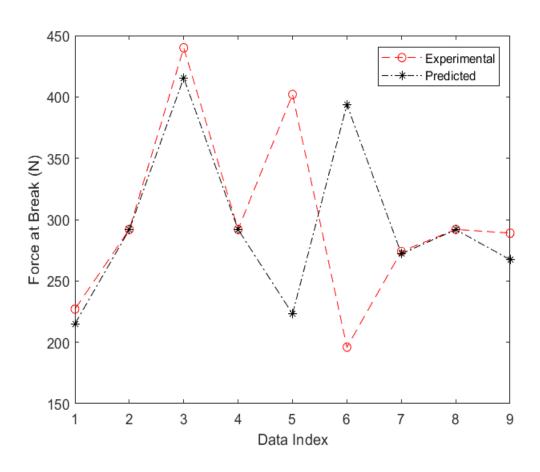


Figure 4.17: Force at Break Testing Data

Table 4.5 also portrays the correlation between the pelleting speed and the force at break as revealed in the assessment of Runs #16, #18 and #30 where continuous decline from 309 N to 292 N and subsequently 289 N were recorded. Similarly, Runs #26 and #7 depicted a decrease from 402 N to 190 N whereas the comparison of #11 and #22 showed a slight rise from 223 N to 227 N.

Information presented in Table 4.5 also uncovered the association between the pellet diameter and the force required to break the pellets. This is visualized in the examination of Runs #28, #25 and #24 where an incessant rise is noticed from 274 N to 292 N and subsequently 440 N just as #11 and #26 revealed an increment from 223 N to 402 N, likewise Runs #12 and #4 depicting an augmentation of 122 N from 266 N to 388 N. All of which were accompanied with a rise in the Die Diameter.

Finally with respect to Force at break, Table 4.5 also presents the effect accompanied by alterations in the screw pitch. Runs #2, #10 and #3 depict an initial incline from 285 N to 292 N followed by a drop to 272 N. A drop in the force at break was also recorded during the comparison of Runs #19 and #26 from 504 N to 402 N just as the assessment of Runs #6 and #11 resulted in a decline from 277 N to 223 N, all of which occurred as a result of increment in the screw pitch.

#### **Effects of Optimization on Deflection at Break**

The deflection at the point of fracture of the pellets differed for the pellets produced during optimization, as effected by the changes in the parameters and these variations are presented using the combination of Table 4.5 and Figures 4.18 and 4.19. Tables 4.1 and 4.2 portray the statistical indices used to analyze these variations.

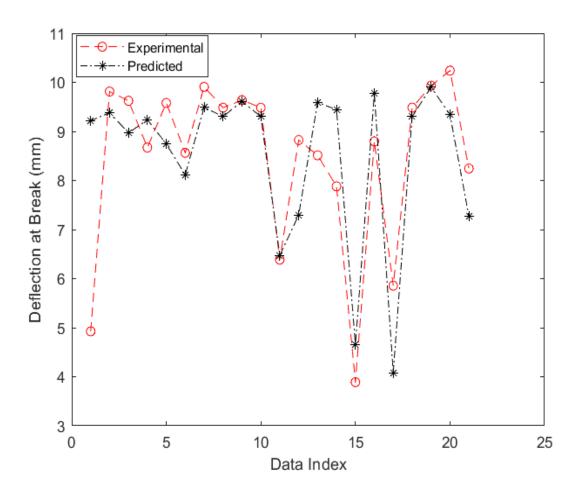


Figure 4.18: Deflection at Break Training Data

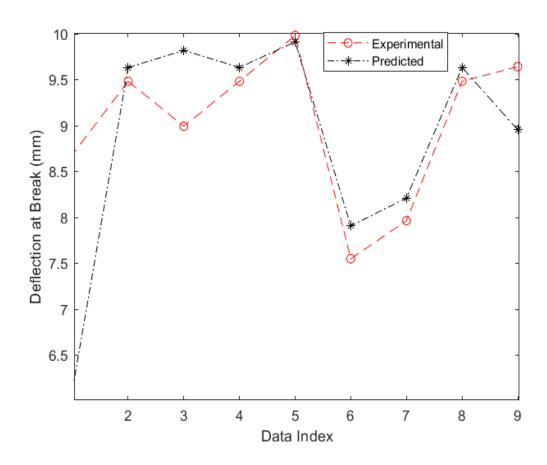


Figure 4.19: Deflection at Break Testing Data

Experimental data on deflection at break point maxed at 10.238 mm and a minimum of 3.89 mm exhibiting a range of 6.348 mm. Predicted maximum and minimum values for training data were 9.900 mm and 4.077 mm respectively while predicted peak and least values for tested data were 9.912 mm and 6.123 mm simultaneously.

The ANN analysis was conducted with a computational time (CT) of 1.14s using twenty-four (24) iterations and a network situs of 4-4-5-1 as reported in Table 4.3. MSE values of 1.5668and 0.8873 were recorded for trained data and tested data respectively while RMSE of 1.2517 and 0.9420 were obtained for data training and data testing congruently, MAD values of 0.8637 and 0.6514 were registered for trained and tested data harmoniously. Similarly, MAPE values of 12.4827 and 6.5692 were obtained from data training and data testing respectively while RCoV of 0.0979 and 0.0911 were recorded for data training and data testing simultaneously and rMBE values of 0.4264 and -1.7706 were achieved for trained and tested data congruently.

Information provided in Table 4.5 displays the relationship between the screw pitch and the pellets' deflection at break point, this is visualized in the assessment of Runs #2, #10 and #3 where an initial slump from 9.812 mm to 9.483 mm followed by a rise to 9.624 mm were recorded. A rise from 5.856 mm to 8.686 mm was reported in the comparative study of Runs #17 and #22 likewise the comparison of Runs #27 and #9 portrayed a surge from 7.55 mm to 9.638 mm. all occurring in line with an increment in the screw pitch.

Table 4.5 also provides details on the association between the pellet diameter and the deflection at break point as seen in the examination of Runs #28, #25 and #24, with an initial rise from 7.965 mm to 9.483 mm and a subsequent slump to 8.993 mm. Runs #21 and #27 also revealed a reduction from 8.244 mm to 7.55 mm whereas the comparative study of Runs #17 and #13 portrayed a rise from 5.856 mm to 8.513 mm, all of which were achieved simultaneously with an increase in diameter of the pellets.

Table 4.5 is also furnished with information revealing the correlation between the pelleting speed and the deflection at fracture point of kenaf starch bonded pellets as noticed in the comparative study of Runs #16, #25 and #30 which revealed an incessant increase from 8.796 mm to 9.483 mm and subsequently to 9.642 mm. However, Runs #19 and #13 displayed a reduction from 9.932 mm to 8.513 mm whereas the comparison of Runs #21

and #12 portrayed a reduction from 8.244 mm to 8.823 mm, all occurring with an harmonious increase in pelleting speed.

Finally, Table 4.5 also provides information pertaining to the relationship between the changes in the amount of binder and the deflection recorded at the point of fracture, as visualized in Runs #14, #18 and #20 with incessant rise from 7.881 mm to 9.483 mm and a further value of 10.238 mm. Similarly, the comparative study of Runs #13 and #14 portrayed an increment from 8.513 mm to 8.671 mm likewise Runs #11 and #5 revealed a rise in values from 6.385 mm to 9.580 mm deflection at break point, all of which occurred with a corresponding increase in quantity of binder added.

# **Effects of Optimization on Energy to Break**

The energy required to press the pellets to break was also determined to differ according to adjustments in the various parameters used in this study. The effects of these variations are reported with the aid of Table 10 in collaboration with Figures 29 and 30. Evidences of these variations are portrayed in Tables 6 and 7.

Figures from the experiment revealed a maximum of 48.574 Nmm/s and a minimum of 9.421 Nmm/s thus having a range of 39.153 Nmm/s. Predicted maximum and minimum values were 35.404 Nmms and 16.252 Nmm/s respectively for trained data while the predicted maximum and minimum value were 35.979 Nmm/s and 16.926 Nmm/s congruently for tested data.

The ANN analysis is obtained in a computational time (CT) of 2.3s using twelve (12) iterations and a topology of 4-3-5-1. MSE values of 75.3938 and 65.9829 for trained data and tested data respectively, RMSE of 8.6830 and 8.1230 were recorded for data training and data testing congruently, MAD of 5.3992 and 6.4972 for trained data and tested data, MAPE values of 19.0113 and 29.7330 were registered for both data training and data testing respectively, RCoV of 0.2586 and 0.3204 was obtained for data training and testing

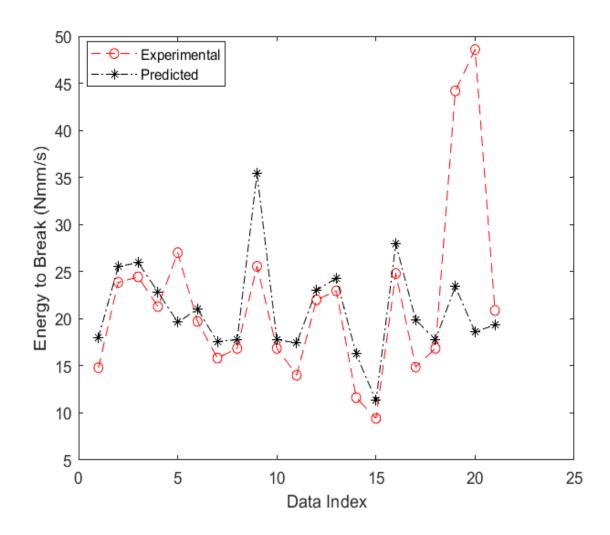


Figure 4.20: Energy to Break Training Data

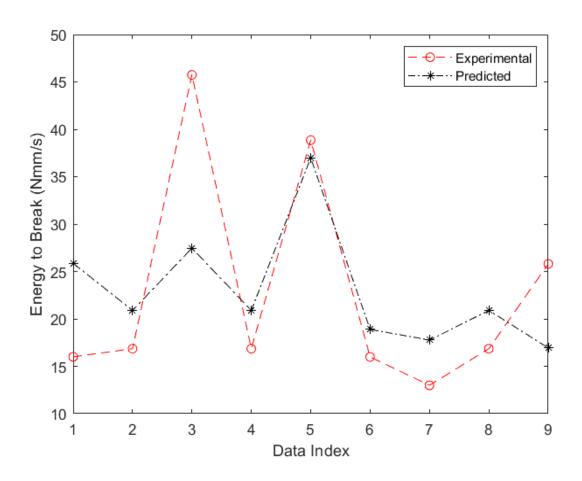


Figure 4.21: Energy to Break Testing Data

accordingly, while rMBE values of -3.4170 and 0.2958 were recorded for trained data and tested data respectively

Experimental results tabulated in Table 4.5 gives an insight into the correlation between the Energy required to break the pellets and the quantity of binder added. This is noticeable in the evaluative study of Runs #14, #18 and #20 where an incessant rise from 11.629 Nmm/s to 16.852 Nmm/s and further 48.574 Nmm/s was recorded. Correspondingly, Runs #11 and #5 depicted an increment from 14.007 Nmm/s to 27.008 Nmm/s likewise Runs #17 and #12 which also portrayed a surge from 14.864 Nmm/s to 22.012 Nmm/s. All being exhibited with a rise in the amount of starch added.

Table 4.5 also provides details on the relationship between the pelleting speed and the minimum energy essential to cause fracture as visualized in the assessment of Runs #16, #23 and #30 with an initial slump from 24.807 Nmm/s to 16.852 Nmm/s followed by a surge to 25.801 Nmm/s. Runs #6 and #14 reported a nose-dive from 19.706 to 14.864 Nmm/s whereas an increment was noticed from 15.982 Nmm/s to 21.29 Nmm/s in the comparative study of #27 and #4. All accompanied with an increment in the pelleting speed.

Table 4.5 also reveals the association between the Die Diameter and the energy to break of the pellets. Comparisons are based on increment in Die Diameter as discovered in the assessment of #28, #25 and #24, where a continual increase from 12.991 Nmm/s to 16.852 Nmm/s is followed by a surge to 45.742Nmm/s. A similar trend was noticed in the comparative study of #11 and #26 where a surge from 14.007 Nmm/s to 38.856 Nmm/s was recorded whereas the comparison of #Runs 5 and #9 revealed a slight drop from 27.008 Nmm/s to 25.569 Nmm/s.

Lastly with regards to energy to break, Table 4.5 portrays the affiliation between the screw pitch on the auger and the minimal energy required to fracture the pellets as revealed in the assessment of Runs #2, #23 and #3, with an initial slump from 23.861 Nmm/s to 16.852 Nmm/s followed by a surge to 24.442 Nmm/s. #4 and #1 also portrayed a reduction from 21.29 Nmm/s to 14.798 Nmm/s with a similar trend noticed in the comparative evaluation of Runs #6 and #11 with a fall from 19.709 Nmm/s to 14.007 Nmm/s. all of which were exhibited with increment in screw pitch.

# **Effects of Optimization on Young Modulus**

The young modulus otherwise known as the Modulus of Elasticity was also determined to be affected by the changes in the parameters associated with the pelleting process of kenaf. Experimental data are presented in Table 4.5 while Figures 4.22 and 4.23 represent the graphical representation of both data used in training and testing the ANN models respectively. The statistical metrics presented in Tables 4.1 and 4.2 are indications of effects caused by these variations.

Table 4.3 revealed that the ANN analysis required a computational time (CT) of 2.27s using sixteen (16) iterations in a topology of 4-3-5-1. MSE values of 3.363×10<sup>-5</sup> and 0.0003 for trained data and tested data were reported correspondingly, RMSE of 0.0060 and 0.0164 for data training and data testing congruently, MAD of 0.0033 and 0.0117 for trained data and tested data harmoniously, MAPE values were 10.1792 and 40.9374 for data training and data testing simultaneously, RCoV values were 0.0773 and 0.0679 for trained data and tested data correspondingly while rMBE values of 2.1212 and 10.3731 were registered for data training and data testing simultaneously.

Experimental data indicates a peak young modulus of 0.0564 N/mm² and a least value of 0.0119 N/mm² thus having a range of 0.0445 N/mm². Predicted maximum and minimum values used for data training were 0.0571 N/mm² and 0.0209 N/mm² respectively whereas the predicted maximum and minimum values for testing data 0.0602 N/mm² and 0.0330 N/mm² congruently.

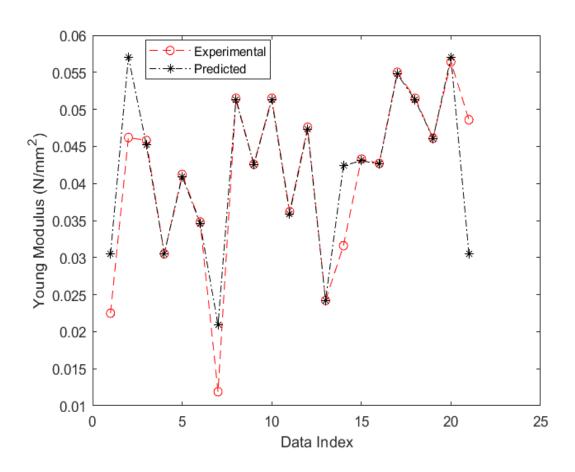


Figure 4.22: Young Modulus Training Data

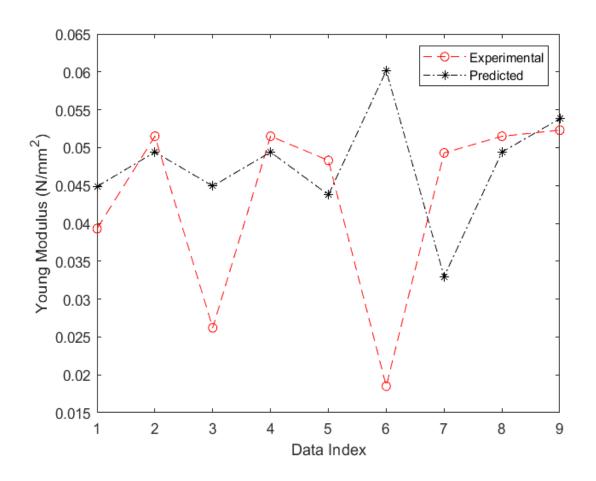


Figure 4.23: Young Modulus Testing Data

Table 4.5 is equipped with experimental data describing the correlation between the screw pitch and the young modulus as compared with an increment in the screw pitch on the auger. This is revealed in the comparative study of Runs #2, #29 and #3 with an initial rise from 0.0462 N/mm² to 0.0515 N/mm² and a decline to 0.0458 N/mm². Runs #27 and #9 depicted a surge from 0.0185 N/mm² to 0.0426 N/mm². Similarly, the comparison of Runs #6 and #11 portrayed a rise from 0.0348 N/mm² to 0.0362 N/mm².

Table 4.5 also provides information on the effects of changes in the young modulus caused by alterations in the size of the die. Runs #28, #25 and #24 revealed an initial rise from 0.0463N/mm² to 0.0515 N/mm² followed by a slump to 0.0262 N/mm². Runs #6 and #19 portrayed a surge from 0.0348 N/mm² to 0.0461 N/mm² just as an increment from 0.0412 N/mm² to 0.0426 N/mm² was observed in the assessment of Runs #5 and #9. All of which occurred when the Die Diameters were increased.

Table 4.5 is also furnished with experimental results portraying the association between the pelleting speed and the young modulus as evidenced in the comparative study of #16, #29 and #30 which depicts a continuous increase from 0.0427 N/mm² to 0.0515 N/mm² and then 0.0523 N/mm². However, the assessment of Runs #26 and #7 revealed a slump from 0.0483 N/mm² to 0.0119 N/mm². Likewise, the comparison of #21 and #12 which also reported a decline from 0.0486 N/mm² to 0.0476 N/mm². All exhibited with an increment in the pelleting speed.

Lastly, the changes in young modulus were examined against changes in the amount of binder added. This was achieved by evaluating changes in the young modulus as affected by increase in the quantity of starch added in the pelleting process. This is visualized in the assessment of Runs #14, #18 and #20 which portrayed an incessant increment from 0.0316 N/mm² to 0.0515 N/mm² and a further value of 0.056 N/mm². The comparative study of Runs #13 and #4 revealed a rise from 0.0242 N/mm² to 0.0305 N/mm², likewise the comparison of Runs #7 and #1 which showed an incline from 0.0119 N/mm² to 0.0225 N/mm².

The Modulus of elasticity (Young Modulus) was also observed to vary reliably with the quantity of starch added whereas it was relatively inconsistent for the other variables.

Similarly, the observed durability of the starch bonded kenaf pellets were above 98% which is classified as high (Lee *et al.*, 2021).

# **Effects of Optimization on Durability**

The durability of the kenaf pellets produced differed with variations in the machine and operating parameters. This is evidenced by the presence of statistical indices (MSE, RMSE, MAPE, MAD, RCoV and rMBE) as listed in Tables 4.1 and 4.2. The experimental results are reported in Table 4.5 while Figures 4.24 and 4.25 represent the graphical representation of data used for training and testing the ANN model respectively.

The minimum and maximum experimental values of the durability were 98.15% and 99.72% yielding a range of 1.57%. Predicted maximum and minimum data used for training were and 98.40% and 99.88% respectively while predicted maximum and minimum data used for testing were 99.30% and 98.52% congruently.

Information from Table 4.3 revealed that the ANN analysis was conducted within a computational time (CT) of 2.14s using twenty (20) iterations and a network situs of 4-3-5-1. MSE values of 0.0760 and 0.2188 were recorded for trained data and tested data respectively, RMSE values were 0.2756 and 0.4678 for trained data and tested data congruently, MAD values were 0.1790 and 0.3936 for data training and data testing harmoniously, MAPE values were 0.1626 and 0.3911 for trained data and tested data, RCoV values were 0.0018 for both trained data and tested data while rMBE values were 0.0692 and -0.0543 for both data training and data testing.

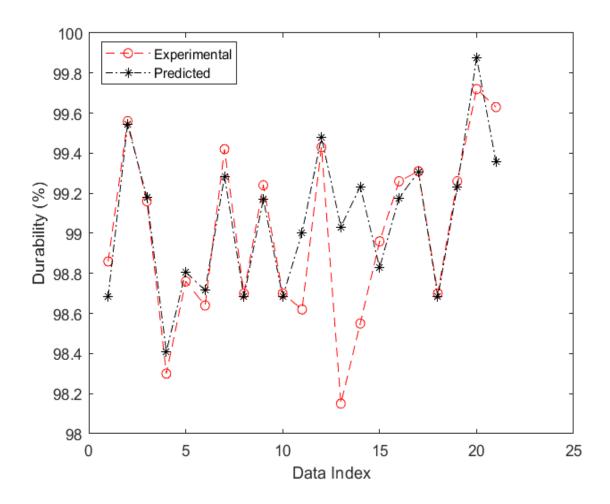


Figure 4.24: Durability Training Data

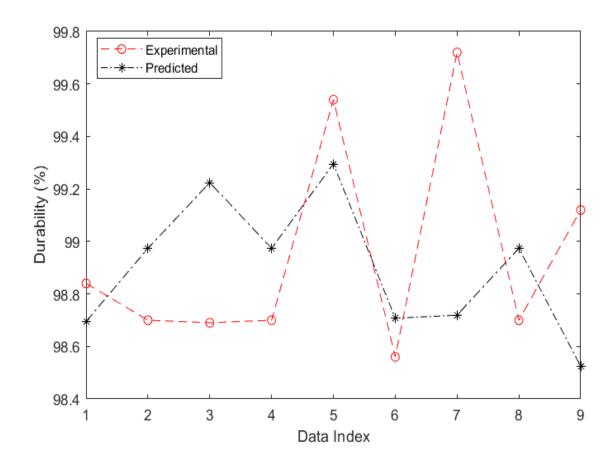


Figure 4.25: Durability Testing Data

Table 4.5 comprises information relating changes in the screw pitch with corresponding variations in the durability of starch bonded kenaf pellets. These can be visualized in the comparative study of Runs #2, #29 and #3 which revealed an initial dip from 99.56% to 98.7% followed by a rise to 99.16%. The assessment of Runs #13 and #7 portrays an incline from 98.15% to 99.42% whereas the pair of Runs #12 and #15 yielded a reduction from 99.43% to 98.96%. It is also pertinent to note that the above assessments were achieved with respect to increment in the screw pitch.

Table 4.5 also portrays the relationship between the Die Diameter and the durability of the pellets with a view to determine the changes in durability with respect to increase in the pellet diameter. This can be seen in the assessment of Runs #28, #25 and #24 which portrayed an incessant decline from 99.72% to 98.7% and further to 98.69%. Similarly, a reduction from 98.96% to 98.86% was revealed in the comparison of Runs #15 and #26, just as the pair of Runs #17 and #13 displayed a drop from 99.31% to 98.15%.

Information recorded in Table 4.5 also describes the correlation between the pelleting speed and the durability of the pellets. The comparative study was done with the goal of expressing the variation with respect to an increase in the pelleting speed. This can be noticed in the assessment of Runs #16, #29 and #30 which depicts an initial drop from 99.26% to 98.7% followed by a rise to 99.12%. A comparative study of Runs #6 and #17 portrays a rise from 98.64% to 99.31% whereas a dip from 98.56% to 98.3% was revealed by the comparison of Runs #27 and #4.

Finally, Table 4.5 also contains experimental results showing the connection between the amount of starch added during the pelleting process to the effective changes in the durability of the pellets. Evaluations were done with respect to increase in the quantity of binder added as observed in the assessment of Runs #14, #18 and #20 where a continuous increment from 98.55% to 98.7% to a further 99.72% was revealed. The comparative study of #22 and #15 also portrays a slight increase from 98.84% to 98.96% whereas the comparison of Runs #7 and #1 indicated a dip from 99.42% to 98.86%. Changes in Forces (Peak, Yield and Break) were directly proportional to the quantity of starch added within the experimental range while an undulating relationship was noticeable for the remaining factors varied during the pelleting process. Changes in the deflection (Peak and Break) were also determined to be

inconsistent for all the factors and the same can be said for changes in the energy required (to attain Peak, Yield and Break).

## 4.4. Effect of Optimization on Remediation Potentials of the Pellets

The variation of the machine and operating parameters also had significant effects on the remediation potential of the pellets, which was categorized based on the amount of oil removed and the recorded changes in the pH of the polluted water samples. These effects are tabulated in Table 4.6

#### Effects of Variations on Amount of Oil removed

The quantity of oil removed was determined to vary for the different sets of pellets produced. The experimental values are presented in Table 4.6, while the graphical illustrations for the trained and tested are portrayed in Figures 4.26 and 4.27 congruently. The presence of the variations is bolstered with the presence of statistical metrics (RMSE, MSE, MAD, MAPE, RCoV and rMBE).

Experiment results peaked at 50000 multiples (99.8%) while the least was 4166.67 multiples (97.6%) culminating a range of 45833.33 multiples. The predicted maximum and minimum values for the data used for training were 29409.50 multiples (99.66%) and 4463.30 multiples (97.76%), respectively whereas the predicted maximum and minimum values for data used for testing were 57580.44 multiples (99.83%) and 4552.79 multiples (97.80%), congruently.

From Table 4.3, it can be deduced that the ANN analysis was conducted in a computational time (CT) using sixty-seven (67) iterations and a topology of 4-3-5-1. MSE values were 83729290.61 and 125545469.3 for trained data and tested data respectively:

**Table 4.6: Pelleting Parameters and Remediation Potential of Kenaf Pellets** 

	Factor 1	Factor 2	Factor 3	Factor 4	Response 13	Response 14
Run	A:Screw Pitch	B:Die Diameter	C:Pelleting Speed	D:Starch/Kenaf Ratio	Oil in Water	pН
	mm	mm	rpm			
1	70	35	70	1.75	6060.61	0.09
2	40	32.5	60	1.5	20000	0.06
3	80	32.5	60	1.5	16666.7	0.08
4	50	35	70	1.75	14285.7	0.22
5	70	30	50	1.75	33333.3	0.02
6	50	30	50	1.25	4761.91	0.07
7	70	35	70	1.25	20000	0.18
8	60	32.5	60	1.5	25000	0.12
9	70	35	50	1.75	28571.4	0.01
10	60	32.5	60	1.5	25000	0.12
11	70	30	50	1.25	16666.7	0.06
12	50	30	70	1.75	4347.83	0.08
13	50	35	70	1.25	5263.16	0.20
14	60	32.5	60	1	11111.1	0.06
15	70	30	70	1.75	12500	0.07
16	60	32.5	40	1.5	13333.3	0.05
17	50	30	70	1.25	5555.55	0.01
18	60	32.5	60	1.5	25000	0.12
19	50	35	50	1.25	33333.3	0.01
20	60	32.5	60	2	22222.2	0.03
21	50	30	50	1.75	4444.44	0.09
22	70	30	70	1.25	5000	0.04
23	60	32.5	60	1.5	25000	0.12
24	60	37.5	60	1.5	50000	0.02
25	60	32.5	60	1.5	25000	0.12
26	70	35	50	1.25	33333.3	0.02
27	50	35	50	1.75	4255.32	0.09
28	60	27.5	60	1.5	4166.67	0.02
29	60	32.5	60	1.5	25000	0.12
30	60	32.5	80	1.5	22222.2	0.07

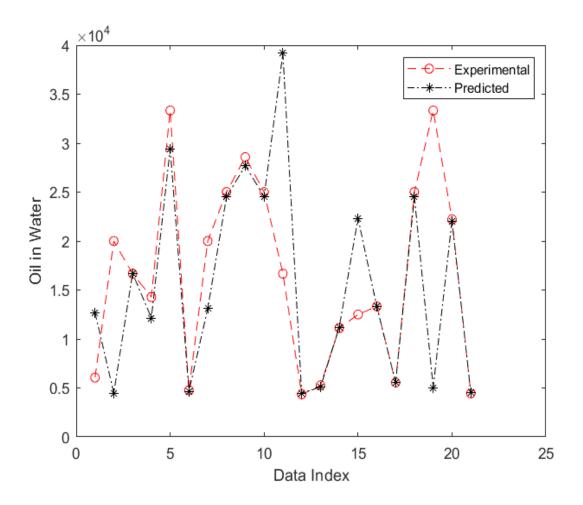


Figure 4.26: Oil in Water Training Data

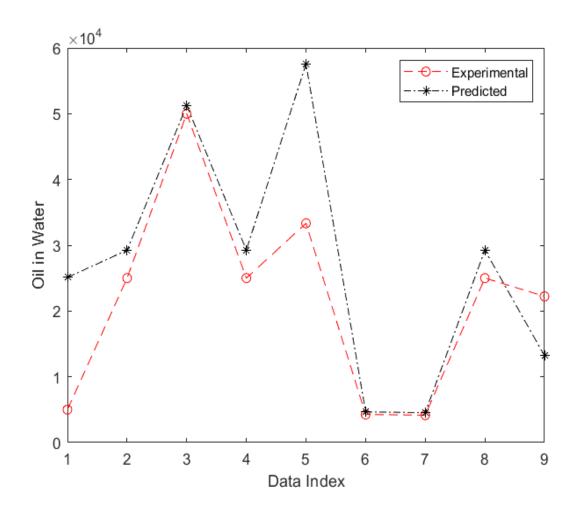


Figure 4.27: Oil in Water Testing Data

RMSE values were 9150.3711 and 11204.7075 for data training and data testing harmoniously: MAD values were 4948.4256 and 7384.2318 for trained data and tested data simultaneously: MAPE values were 27.0181 and 65.2924 for data used for training and testing congruently: RCoV values were 0.2969 and 0.1979 for trained data and tested data simultaneously while rMBE values were -5.8588 and 25.8221 for data training and data testing concurrently.

Table 4.6 consists information that can be used to describe the relationship between the screw pitch and the amount of oil removed. The assessment is reported based on the changes in the quantity of oil removed with respect to increase in the screw pitch. This is evidenced in the comparative study of Runs #2, #29 and #3 where a continual oil removal improvement is revealed from 20000 multiples (99.5%) to 25000 multiples (99.6%) to a further 33333.3 multiples (99.70%). There was a significant improvement in remediation from 4387.83 multiples (97.72%) to 12500 multiples (99.2%) when comparing Runs #12 and #15 whereas the comparison of Runs #19 and #26 portrayed no improvement as the oil removal still remained pegged at 33333.3 multiples (99.70%).

Table 4.6 portrays the correlation between the Die Diameter and the quantity of oil removed. This is demonstrated in the assessment of Runs #28, #25 and #24 displaying an incessant improvement in crude oil removal from 4166.7 multiples (97.6%) to 25000 multiples (99.6%) to a further value of 50000 multiples (99.8%). The comparative study of Runs #22 and #7 revealed an enhanced performance from 5000 multiples (98%) to 20000 multiples (99.5%) whereas the pair of #21 and #27 showed a dip from 4444.44 multiples (97.75%) to 4255.32 multiples. (97.65%) with all the mentioned evaluation based on an increase in Die Diameter.

Table 4.6 is also indicative of the connection between the pelleting speed and the quantity of oil removed by the pellets. Evaluations were based on the increment in the pelleting speed and the corresponding response to the amount of oil removed as evidenced in the comparative study of #16, #29 and #30 which revealed an initial rise from 13333.3 multiples (99.25%) to 25000 multiples (99.6%) followed by a fall to 22222.2 multiples (99.55%). Comparisons of Runs #11 and #22 depicted a reduction from 16666.7 multiples (99.4%) to

5000 multiples (98%), which is similar to the results exhibited by the pair of Runs #21 and #12 which portrayed a dip from 4444.44 (97.75%) to 4347.83 (97.70%).

Finally, Table 4.6 contains experimental data associating the amount of starch added with the amount of oil removed as assessed with a simultaneous increase in the quantity of binder added. This is evident in the evaluation of Runs #14, #18 and #20 where an initial surge from 11111.1 multiples (99.1%) to 25000 multiples (99.6%) followed by a dip to 22222.22 multiples (99.55%) was recorded. The comparison of Runs #16 and #21 portrayed a decline from 4761.9 multiples (97.90%) to 4444.44 multiples (97.75%) whereas that of Runs #13 and #4 revealed a surge from 5263.16 multiples (98.1%) to 14285.7 multiples (99.30%).

# Effects of Variations on Changes in pH

The changes in pH of the remediated water also varied for the different pellets used in the remediation process, these changes are presented in Table 4.6. Figures 4.28 and 4.29 portrays the graphical representation of data used for training and testing the mathematical models respectively. Indications of the variations in the changes of the pH are the presence of the statistical metrics reported in Table 4.1 and 4.2 respectively.

Information from the field experiments revealed maximum and minimum changes to pH values to be 0.22 and 0.01 resulting in a range of 0.21. Predicted data used in training were 0.2 and 0.01 for maximum and minimum values respectively whereas the predicted data used in testing were 0.15 and 0.03 as peak and least values congruently.

The ANN analysis was conducted in a computational time (CT) of 2.12s using fifteen (15) iterations and a network situs of 4-3-5-1. MSE values were 0.0025 and 0.0005 and for tested and trained data accordingly, RMSE values were 0.0232 and 0.0499 for data training and data testing congruently, MAD values were 0.0442 and 0.0112 for data testing and data training and accordingly, MAPE values were 40.5258 and 94.7241 for trained data and

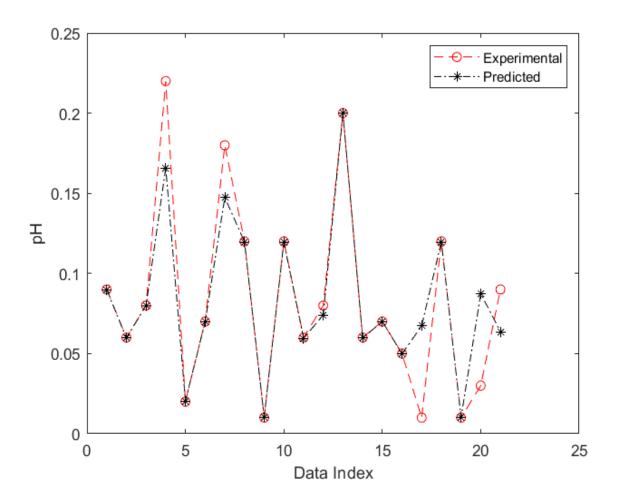


Figure 4.28: Changes in pH Training Data

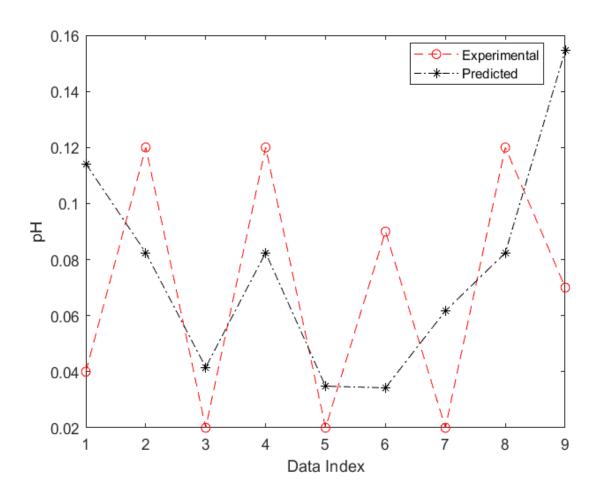


Figure 4.29: Changes in pH Testing Data

tested data. Simultaneously, RCoV values were 0.1607 for both trained data and tested data congruently while rMBE values were -0.3707 and 11.0226 for data training and data testing simultaneously.

Table 4.6 comprises experimental results concerning the relationship between the amount of starch added and the changes in pH during the remediation process. Evaluation is done in lieu of a simultaneous increment in the amount of binder added as observed in the assessment of #14, #18 and #20 where an initial rise from 0.06 to 0.12 followed by a slump to 0.03 was recorded. The comparative study of Runs #19 and #27 portrayed an increment from 0.01 to 0.09 which was similar to the result exhibited by pair of Runs #6 and #21 which revealed an increment from 0.07 to 0.09.

It can also be deduced from Table 4.6 that a correlation existed between the Die Diameter and changes in pH. Changes in pH were examined against increase in the pelleting speed as noticed in the comparative study of Runs #28, #25 and #24 which revealed a surge from 0.02 to 0.12 succeeded by a slump back to 0.02. The comparison of Runs #17 and #13 depicted a surge from 0.01 to 0.2 likewise the pair of Runs #22 and #7 which portrayed an increment from 0.04 to 0.18.

Table 4.6 also shows the association between the pelleting speed and the changes in the pH value of the remediated water. Changes in pH were investigated against increment in the pelleting speed as perceived in the assessment of Runs #16, #25 and #30 which portrayed an initial increase from 0.05 to 0.12 followed by a slump to 0.07. The comparison of Runs #5 and 15 revealed a rise from 0.02 to 0.07 just as the comparative study of Runs #26 and #7 depicted a surge from 0.02 to 0.18.

Finally, from Table 4.6, there was a discovered connection between the changes in the screw pitch and the changes in the pH with changes in pH examined against a simultaneous increase in the screw pitch. This is evident in the comparative study of #2, #23 and #3 which portrays a preliminary increment from 0.06 to 0.12 trailed by a decline to 0.08. The comparison of Runs #6 and #11 reveals a dip from 0.07 to 0.06, similarly, the pair of #27 and #9 exhibited a nose-dive from 0.09 to 0.01.

The quantity of oil removal was found to be directly proportional to the changes in the screw pitch on the auger. There was a potential inverse proportionate relationship between the quantity of oil removed and the pelleting speed whereas the other factors were determined to have an inconsistent association with the amount of oil removed. However, the rule of diminishing returns was observed in the assessment of pH where there was initial increment in changes in pH followed by a reduction noticeable at extreme values of the variation for all the four factors used in the optimization study.

#### 4.5 Mathematical Models

Responses such as machine performance, mechanical properties and remediation potentials of kenaf pellets were also found be affected by operating and machine parameters (Screw pitch SP, Die diameter DD, pelleting speed SR and binder ratio BR). These effects were defined using mathematical equations and coefficient of determination, R<sup>2</sup>. Mathematical models portraying the association between the varied parameters and the responses during the optimization were summarily displayed. The coefficient of determination R<sup>2</sup> was also listed

## Relationship between Parameters and Pelleting Efficiency

The parameters were found to have a correlation with the pelleting efficiency as depicted by the modelled equation 4.1 having a  $R^2$  value of 0.7785.

Pelleting Efficiency

$$= \sqrt[3]{ 50698.53911 - 616.91883 \text{ SR} - 44072.95770 \text{ BR} \atop +19.62943 \text{ DD} \times \text{SR} + 634.70581 \text{ DD} \times \text{BR} \atop -32.4316 \text{ 4 DD}^2 + 8279.43698 \text{ BR}^2 + 0.078096 \text{ SR}^2 \times \text{BR} \atop (4.1)}$$

# Relationship between Parameters and Percentage Recovery

The correlation between the parameters and PR were portrayed in Equation 4.2 and had a  $R^2$  value of 0.5705.

Percentage Recovery =

$$\sqrt[3]{76710.00781 - 1.03642E + 05 BR + 1825.17832 DD \times BR} - 67.50$$

$$-42.15898 DD^{2} + 0.020817 SR^{2} + 16223.48556 BR^{2}$$
(4.2)

## Relationship between Parameters and Force at Peak

The relationship between the parameters and force at peak as described by equation 4.3 with  $R^2$  of 0.2976

## Relationship between Parameters and Deflection to Peak

The association between the parameters and deflection at peak was described by Equation 4.4 with R<sup>2</sup> value of 0.9978

$$D_P = -229.37300 + 13.24020 \text{ DD} + 5.70976 \text{ SR} - 43.95060 \text{ BR}$$
  
 $-0.146547 \text{ SP} \times \text{DD} - 0.067826 \text{ SP} \times \text{SR} + 3.47467 \text{ SP} \times \text{BR}$   
 $-0.140201 \text{ DD} \times \text{SR} - 0.354657 \text{ DD} \times \text{BR} - 1.70333 \text{ PS} \times \text{BR}$   
 $+0.024197 \text{ SP}^2 - 0.132827 \text{ DD}^2 - 2.53154 \text{ BR}^2$   
 $+0.001254 \text{ SP} \times \text{DD} \times \text{SR} - 0.030502 \text{ SP} \times \text{DD} \times \text{BR}$   
 $+0.004076 \text{ SP} \times \text{SR} \times \text{BR} + 0.041940 \text{ DD} \times \text{SR} \times \text{BR}$   
 $+0.000171 \text{ SP}^2 \text{ SR} - 0.021837 \text{ SP}^2 \text{ BR} + 0.001970 \text{ SP} \times \text{DD}^2$ 

# Relationship between Parameters and Energy to Peak

In general, the relationship between the parameters and energy to break was described by the mathematical model in equation 4.5 with a  $R^2$  value of 0.3744.

$$E_P = -29.75679 + 1.72869 DD + 0.001915 DD \times SR \times BR - 0.000278 DD^2 \times SR$$
 (4.5)

# Relationship between Parameters and Force to Yield

The relationship between the Force at yield point and the parameters are depicted by the equation 4.6 with a coefficient of determination of 0.3566

$$Mean(F_Y)$$

5.21033 + 0.090618 SP + 0.201420 DD + 0.127182SR + 1.31450 BR $+ 0.000904 SP \times DD - 0.001416 SP \times SR + 0.012046 SP \times BR - 0.001443 DD \times SR$   $= e^{-0.001937 DD \times BR} + 0.028559 SR \times BR - 0.000505 SP^2 - 0.002196 DD^2 - 0.000397 SR^2$ 

## Relationship between Parameters and Energy to Yield

Overall, Equation 4.7 was used to portray the relationship between the parameters and energy to yield point. The coefficient of determination was reported to be 0.3947.

$$E_Y = -50.53655 + 2.53047 \text{ DD} + 0.004728 \text{ DD} \times \text{SR} \times \text{BR} - 0.000432 \text{ DD}^2 \times \text{SR}$$

(4.7)

# Relationship between Parameters and Force at Break

The mathematical equation 4.8 was used to define the association between pelleting parameters and the force at rupture point. It had a  $R^2$  value of 0.3594.

$$3.20326 + 0.095628 \text{ SP} + 0.217800 \text{ DD} + 0.044864 \text{ SR} \\ + 1.02981 \text{ BR} - 0.001131 \text{ SP} \times \text{SR} + 0.014811 \text{ SP} \times \text{BR} - 0.113956 \text{ DD} \times \text{BR} \\ Mean(F_R) = e \\ + 0.033154 \text{ SR} \times \text{BR} - 0.000479 \text{ SP}^2 - 0.000305 \text{ SR}^2$$

(4.8)

## Relationship between Parameters and Deflection at Break

In totality, the parameters and deflection at the point of rupture were determined to be related using the Equation 4.9 and a R<sup>2</sup> value of 0.2589.

$$D_R = +5.74410 + 0.000129 \text{ DD}^2 \times \text{SR} - 1.35677E - 06 \text{ DD}^2 \times \text{SR}^2$$
 (4.9)

#### Relationship between Parameters and Energy to Break

Equation 4.10 depicts the relationship between the pelleting parameters and the energy to break the pellets. It exhibited a coefficient of determination of 0.3716

$$E_B = -71.31892 + 3.40170 \text{ DD} + 0.003733 \text{ DD} \times \text{SR} \times \text{BR}$$
  
- 0.000441 DD<sup>2</sup> × SR (4.10)

#### **Relationship between Parameters and Young Modulus**

The association between the parameters and the modulus of elasticity was defined using equation 4.11 and had a  $R^2$  value of 0.7172.

Young Modulus

$$= -1.37251 + 0.001536 \, \mathrm{SP} + 0.058840 \, \mathrm{DD} + 0.011085 \, \mathrm{SR} \\ + 0.171450 \, \mathrm{BR} + 0.000080 \, \mathrm{SP} \times \mathrm{DD} - 0.000038 \, \mathrm{SP} \times \mathrm{SR} \\ + 0.000720 \, \mathrm{SP} \times \mathrm{BR} - 0.000227 \, \mathrm{DD} \times \mathrm{SR} - 0.003180 \, \mathrm{DD} \times \mathrm{BR} \\ + 0.000700 \, \mathrm{SR} \times \mathrm{BR} - 0.000025 \, \mathrm{SP}^2 - 0.000734 \, \mathrm{DD}^2 \\ - 0.000021 \, \mathrm{SR}^2 - 0.048400 \, \mathrm{BR}^2$$

## Relationship between Parameters and Durability

Equation 4.12 was used to describe the association between the pellets durability and the parameters varied during the pelleting process. The coefficient of determination was observed to be 0.4281

$$D = \sqrt[3]{9.82954E + 05 - 1.77819 \text{ SP} \times \text{DD} - 9.14795 \text{ DD} \times \text{SR} + 168.48279 \text{ DD} \times \text{BR}}$$

$$(4.12)$$

## Relationship between Parameters and Oil Removed

Overall, the effects of parameters on the oil removed are modelled by equation 4.13 with a  $R^2$  value of 0.6607

Oil in Water

$$= \sqrt[1.2]{ -3.32032E + 05 + 2894.02594 DD - 0.986244 SP^2 \times SR +8.82228 SP \times DD^2 + 4.71509 DD \times SR^2 - 0.137156 DD^2 \times SR^2}$$

(4.13)

#### Relationship between Parameters and pH Changes

The relationship between the parameters and changes in pH was established using the equation 4.14 with a coefficient of determination of 0.5743.

$$pH = \sqrt[1.5]{\frac{1.22473 - 0.000421 \text{ SP} - 0.037983 \text{ DD} - 0.021404 \text{ SR}}{-0.000060 \text{ SP} \times \text{BR} + 0.000693 \text{ DD} \times \text{SR}}}$$

(4.14)

#### 4.6 Other Relevant Information

Information pertaining to the design of the pelletizer and durability testing apparatus such as the AutoCAD drawings are provided in Appendix I. Appendix II comprises the complete table showing the results and various varied parameters as well as calculations from the experimental tests which were used to obtain the results.

Information such as the regression plots for the various responses, predicted data for training and testing as well as the source code for the Artificial Neural Network using MATLAB are provided in Appendix IV.

The coefficient of determination (R<sup>2</sup>) of the responses were between 0.2589 and 0.9978.

# CHAPTER FIVE SUMMARY, CONCLUSION AND RECOMMENDATIONS

#### 5.1. Summary

A single screw extruder was designed and fabricated for producing kenaf pellets for the environmental remediation of oil polluted water bodies. The extruder was evaluated and pelleting process was optimized by varying machine and operating parameters. Results were analyzed using artificial neural network.

#### 5.2. Conclusions

A screw type pelletizer with detachable auger was designed and fabricated with a primary objective to optimize pelleting of grounded kenaf stem thus encompassing provisions for change in speed, changes in die diameter as well as changes in the quantity of starch added. This became necessary in order to improve handling as well as absorption rate during the remediation of oil polluted water bodies.

The pelletizer was evaluated to have pelleting efficiency of above 85% while the Percentage Recovery exceeded 68%, thus implying that majority of the extrudate were recovered despite moisture loss due to heat and residual matter trapped within the extruder after operation.

Mechanical properties of kenaf pellets were also found to significantly affected by the changes in the machine and operating parameters. For example, changes in forces (peak, yield and break) were proportional to changes in kenaf/starch ratio. The durability of the pellets also exceeded 98% signifying high resistance to wear and tear when subjected to dynamic load.

The remediation potentials of the pellets were also determined to be significantly affected by variations in the parameters. Oil recovery was least at 97% signifying effectiveness in

remediation. There were also significant changes in the pH of the polluted water before and after remediation.

Optimal conditions for the production of pellets were found out to be 54 mm Screw Pitch, pelleting Speed 68 rpm and Die diameter of 34mm

#### **5.3.** Recommendations

Recommendations pertaining to this study for future research would include:

- Evaluation of other systems of densification such as hydraulic piston and mechanical piston press, pellet mills (ring-die and flat-bed) as well as the gear type (Obernberger and Thek, 2010; Gilvari *et al.*, 2019).
- There is also a need to explore the possibility of some pretreatment before pelleting such as torrefaction, stabilization, pyrolysis, hydrothermal carbonization etc. (Gilvari *et al.*, 2019; Lee, 2021; Riva *et al.*, 2021).
- The need to try cheaper organic binding agents most especially residual biomass such as brown sugar powder, microalgae, rapeseed cake (residue after oil extraction), miscanthus, molasses, residual corroded corn starch (bye-product from pap preparation) (Anukam *et al.*, 2021).
- The multifunctional characteristics of the kenaf fiber also necessitate the need to explore other cheaper fibrous sources for remediation, these may include sugar cane residues and coconut husks.

## **5.4 Contributions to Knowledge**

The study contributed to knowledge, more information on the production of Kenaf pellets in the following ways:

- ▲ Development of economical, effective, and efficient production of durable oilabsorbing kenaf pellets used in the clean-up of oil spilled on water bodies as the binder to be used were obtained from wastewater of cassava processing.
- ▲ Provided information on the effects that variations in machine and operating parameters (such as pelleting speed, screw pitch and Die Diameter and kenaf-binder conditions) had on the kenaf pelleting process as well as information on the optimization of kenaf pellets production.

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# APPENDICES APPENDIX I: AUTOCAD DIAGRAMS

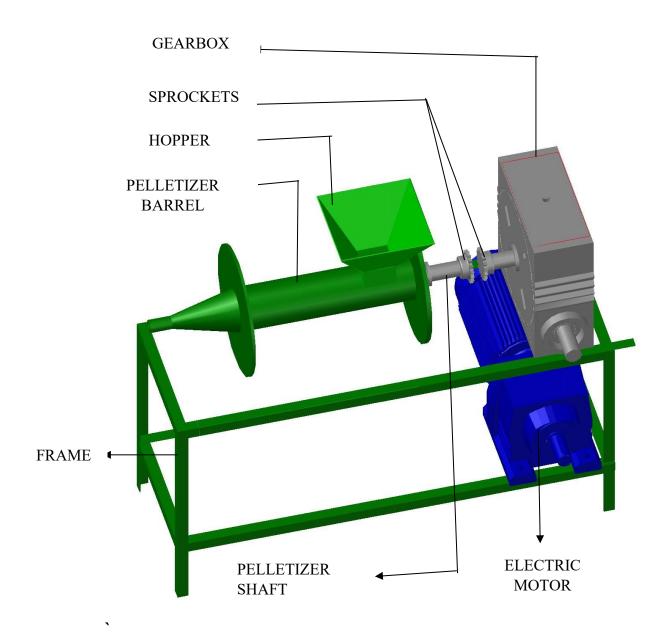


Figure A.1: AUTOCAD Diagram for the Pelletizer

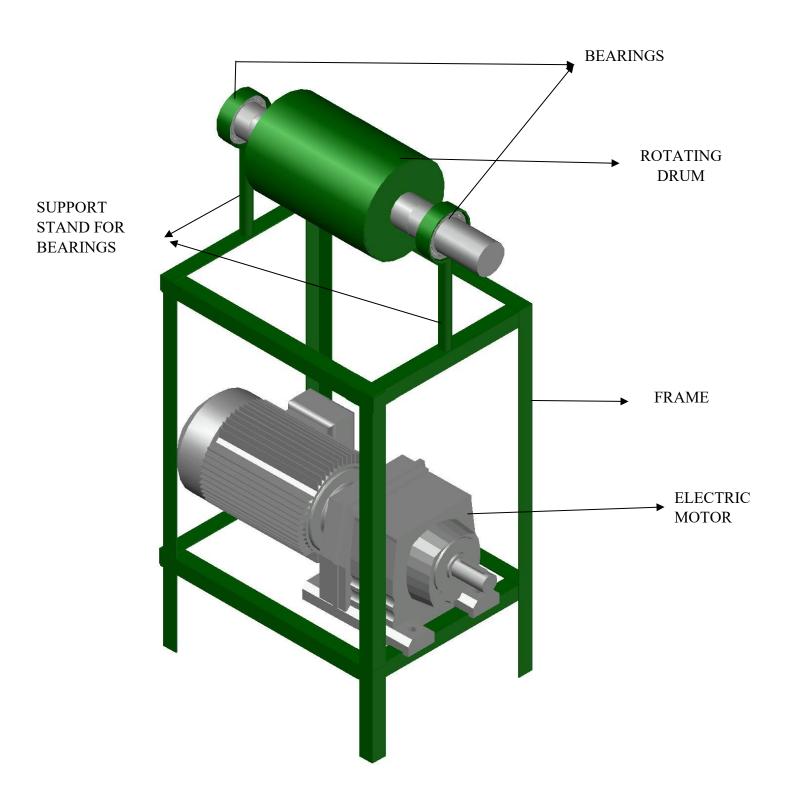


Figure A.2: Durability Testing Apparatus

## APPENDIX II: PARAMETERS AND RESULTS

		Factor 1	Factor 2	Factor 3	Factor 4	Response 1	Response 2	Response 3	Response 4	Response 5	Response 6	Response 7	Response 8	Response 9	Response 10	Response 11	Response 12	Response 13	Response 14
Std	Run	A:Screw Pitch	B:Die Diameter	C:Pelleting Speed	D:Starch/Kenaf Ratio	Pelleting Efficiency	Percentage Recovery	Force @ Peak	Deflection @ Peak	Energy to Peak	Force @ Yield	Energy to Yield	Force @ Break	Deflection @ Break	Energy to Break	Young Modulus	Durability	Oil in Water	pН
		mm	mm	rpm		%	%	N	mm	Nmm/s	N	Nmm/s	N	mm	Nmm/s	N/mm2	%		-
16	1	70	35	70	1.75	95.6	87.05	172	4.406	14.313	174	14.479	178	4.925	14.798	0.0225	98.86	6060.61	0.09
17	2	40	32.5	60	1.5	92.14	85.6	238	4.186	15.682	266	19.104	285	9.812	23.861	0.0462	99.56	20000	0.06
18	3	80	32.5	60	1.5	92.84	84.93	228	4.278	16.563	254	21.889	272	9.624	24.442	0.0458	99.16	16666.7	0.08
15	4	50	35	70	1.75	95.76	81.18	212	3.168	7.301	340	16.801	388	8.671	21.29	0.0305	98.3	14285.7	0.22
10	5	70	30	50	1.75	93.87	77.98	254	3.497	17.864	288	22.958	324	9.58	27.008	0.0412	98.76	33333.3	0.02
1	6	50	30	50	1.25	92.62	73.95	204	3.298	14.626	248	16.076	277	8.564	19.709	0.0348	98.64	4761.91	0.07
8	7	70	35	70	1.25	91.55	71.92	116	3.994	9.642	120	9.996	190	9.907	15.82	0.0119	99.42	20000	0.18
27	8	60	32.5	60	1.5	91.62	82.36	236	3.517	13.207	284	15.369	292	9.483	16.852	0.0515	98.7	25000	0.12
12	9	70	35	50	1.75	94.81	80.26	260	3.468	18.137	291	22.593	313	9.638	25.569	0.0426	99.24	28571.4	0.01
25	10	60	32.5	60	1.5	91.62	82.36	236	3.517	13.207	284	15.369	292	9.483	16.852	0.0515	98.7	25000	0.12
2	11	70	30	50	1.25	90.68	70.03	153	2.418	8.408	181	10.049	223	6.385	14.007	0.0362	98.62	16666.7	0.06
13	12	50	30	70	1.75	93.66	82.98	172	2.582	12.284	242	19.263	266	8.823	22.012	0.0476	99.43	4347.83	0.08
7	13	50	35	70	1.25	94.03	75.49	142	3.387	11.829	180	14.987	276	8.513	22.968	0.0242	98.15	5263.16	0.2
23	14	60	32.5	60	1	95.06	86.88	140	2.008	6.538	163	8.226	204	7.881	11.629	0.0316	98.55	11111.1	0.06
14	15	70	30	70	1.75	91.72	74.88	84	2.378	4.927	108	6.745	142	3.89	9.421	0.0433	98.96	12500	0.07
21	16	60	32.5	40	1.5	90.97	78.45	228	3.368	16.694	272	20.366	309	8.796	24.807	0.0427	99.26	13333.3	0.05
5	17	50	30	70	1.25	89.61	75.21	252	5.481	14.572	252	13.718	252	5.856	14.864	0.055	99.31	5555.55	0.01
26	18	60	32.5	60	1.5	91.62	82.36	236	3.517	13.207	284	15.369	292	9.483	16.852	0.0515	98.7	25000	0.12
3	19	50	35	50	1.25	85.74	69.94	280	3.5	27.098	342	32.173	504	9.932	44.186	0.0461	99.26	33333.3	0.01
24	20	60	32.5	60	2	95.62	88.27	252	3.813	29.074	318	36.818	486	10.238	48.574	0.0564	99.72	22222.2	0.03
9	21	50	30	50	1.75	90.47	82.22	160	2.668	12.502	234	18.92	256	8.244	20.879	0.0486	99.63	4444.44	0.09
6	22	70	30	70	1.25	92.66	83.74	155	3.041	9.82	181	12.187	227	8.686	15.994	0.0393	98.84	5000	0.04
29	23	60	32.5	60	1.5	91.62	82.36	236	3.517	13.207	284	15.369	292	9.483	16.852	0.0515	98.7	25000	0.12
20	24	60	37.5	60	1.5	82.36	68.49	256	3.649	19.064	328	31.668	440	8.993	45.742	0.0262	98.69	50000	0.02
30	25	60	32.5	60	1.5	91.62	82.36	236	3.517	13.207	284	15.369	292	9.483	16.852	0.0515	98.7	25000	0.12
4	26	70	35	50	1.25	85.96	70.85	278	3.583	25.842	336	29.408	402	9.982	38.856	0.0483	99.54	33333.3	0.02
11	27	50	35	50	1.75	90.63	87.36	126	3.625	10.031	184	14.908	196	7.55	15.982	0.0185	98.56	4255.32	0.09
19	28	60	27.5	60	1.5	88.76	82.59	248	2.705	9.115	264	11.268	274	7.965	12.991	0.0493	99.72	4166.67	0.02
28	29	60	32.5	60	1.5	91.62	82.36	236	3.517	13.207	284	15.369	292	9.483	16.852	0.0515	98.7	25000	0.12
22	30	60	32.5	80	1.5	92.46	73.89	244	3.628	19.244	272	23.618	289	9.642	25.801	0.0523	99.12	22222.2	0.07

#### **APPENDIX III: CALCULATIONS**

#### TEST 04

Mass of grounded kenaf = 601g

Mass of Starch = 1052g

Mass of Water added = 1803.2g

Total Mass of Input = 3456.2g

Unpelleted Mass = 119.03g

Mass of Pellets = 2686.85g

Total Mass of Output = 2805.88g

### **Pelleting Efficiency**

$$\eta \% = \frac{2686.85}{3456.2} \times 100\%$$
$$= 95.76\%$$

#### **Percentage Recovery**

$$R\% = \frac{2805.88}{3456.2} \times 100\%$$
$$= 81.18\%$$

**Energy to Peak** 

$$=\frac{212\times3.168}{94.787}=14.572$$

**Energy to Yield** 

$$=\frac{340\times6.6230}{133.349}=16.801$$

**Energy to Break** 

$$=\frac{388\times8.6710}{158.024}=21.290$$

**Young Modulus** 

$$=\frac{0.398}{13.070}=0.0305$$

$$=\frac{51.9}{52.8}=98.3\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 750g

Mass of Water added = 1800g

Total Mass of Input = 3150g

Unpelleted Mass = 142g

Mass of Pellets = 2236g

Total Mass of Output = 2378g

## **Pelleting Efficiency**

$$\eta \% = \frac{2236}{3150} \times 100\%$$
$$= 94.03\%$$

**Percentage Recovery** 

$$R\% = \frac{2378}{3150} \times 100\%$$
$$= 75.49\%$$

**Energy to Peak** 

$$=\frac{142\times3.3870}{40.66}=11.829$$

**Energy to Yield** 

$$=\frac{180\times4.3420}{52.148}=14.987$$

**Energy to Break** 

$$=\frac{276\times8.5130}{102.299}=22.968$$

**Young Modulus** 

$$= \frac{0.210}{8.684} = 0.0242$$

$$=\frac{63.5}{64.7}$$
= 98.15%

Mass of grounded kenaf = 600.1g

Mass of Starch = 1050.2g

Mass of Water added = 1800.3g

Total Mass of Input = 3450.6g

Unpelleted Mass = 181.5g

Mass of Pellets = 2861.8g

Total Mass of Output = 2863.3g

**Pelleting Efficiency** 

$$\eta \% = \frac{2119.8}{2863.3} \times 100\%$$
$$= 93.66\%$$

**Percentage Recovery** 

$$R\% = \frac{2863.3}{3450.6} \times 100\%$$
$$= 82.98\%$$

**Energy to Peak** 

$$=\frac{172\times2.582}{36.153}=12.284$$

**Energy to Yield** 

$$=\frac{242\times5.873}{73.782}=19.263$$

**Energy to Break** 

$$=\frac{266\times8.823}{106.619}=22.012$$

**Young Modulus** 

$$=\frac{0.224}{4.706}=0.0476$$

$$=\frac{52.3}{52.6}=99.43\%$$

Mass of grounded Kenaf = 600g

Mass of Starch = 750.1g

Mass of Water added = 1800g

Total Mass of Input = 3150.1g

Unpelleted Mass = 246.1g

Mass of Pellets = 2123.2g

Total Mass of Output = 2369.3g

## **Pelleting Efficiency**

$$\eta \% = \frac{2123.2}{2369.3} \times 100\%$$
$$= 89.61\%$$

#### **Percentage Recovery**

$$R\% = \frac{2369.3}{3150.1} \times 100\%$$
$$= 75.21\%$$

**Energy to Peak** 

$$=\frac{252\times5.481}{94.787}=14.572$$

**Energy to Yield** 

$$=\frac{252\times5.518}{95.186}=13.718$$

**Energy to Break** 

$$=\frac{252\times5.8560}{99.282}=14.864$$

**Young Modulus** 

$$=\frac{0.607}{11.036}=0.0550$$

$$=\frac{42.6}{42.9}=99.31\%$$

Mass of grounded kenaf = 600.2g

Mass of Starch = 750.4g

Mass of Water added = 1800.6g

Total Mass of Input = 3151.2g

Unpelleted Mass = 193.69g

Mass of Pellets = 2445.12g

Total Mass of Output = 2638.81g

**Pelleting Efficiency** 

$$\eta \% = \frac{2445.12}{2638.81} \times 100\%$$
$$= 92.66\%$$

**Percentage Recovery** 

$$R\% = \frac{2638.81}{3151.2} \times 100\%$$
$$= 83.74\%$$

**Energy to Peak** 

$$=\frac{155\times3.041}{47.999}=9.82$$

**Energy to Yield** 

$$=\frac{181\times5.337}{79.268}=12.187$$

**Energy to Break** 

$$=\frac{227\times8.686}{123.279}=15.994$$

**Young Modulus** 

$$=\frac{0.416}{10.585}=0.0393$$

$$=\frac{59.6}{60.3}=98.84\%$$

Mass of grounded kenaf = 600.5g

Mass of Starch = 1050.875g

Mass of Water added = 1801.5g

Total Mass of Input = 3683.7g

Unpelleted Mass = 228.4g

Mass of Pellets = 2529.9g

Total Mass of Output = 2758.3g

## **Pelleting Efficiency**

$$\eta \% = \frac{2529.9}{2758.3} \times 100\%$$
$$= 91.72\%$$

#### **Percentage Recovery**

$$R\% = \frac{2758.3}{3683.7} \times 100\%$$
$$= 74.88\%$$

**Energy to Peak** 

$$=\frac{84\times2.3780}{40.541}=\quad 4.927$$

**Energy to Yield** 

$$=\frac{108\times3.000}{48.033}=6.745$$

**Energy to Break** 

$$=\frac{142\times3.8960}{58.723}=9.421$$

**Young Modulus** 

$$=\frac{0.260}{6.000}=0.0433$$

$$=\frac{57.1}{57.7}=98.96\%$$

Mass of grounded kenaf = 600.5g

Mass of Starch = 750.7g

Mass of Water added = 1801.5g

Total Mass of Input = 3152.7g

Unpelleted Mass = 191.5g

Mass of Pellets = 2075.8g

Total Mass of Output = 2267.3g

## **Pelleting Efficiency**

$$\eta \% = \frac{2075.8}{2267.3} \times 100\%$$
$$= 91.55\%$$

**Percentage Recovery** 

$$R\% = \frac{2267.3}{3152.7} \times 100\%$$
$$= 71.92\%$$

**Energy to Peak** 

$$=\frac{116\times3.9940}{48.053}$$
= 9.642

**Energy to Yield** 

$$=\frac{120\times5.8920}{70.731}=9.996$$

**Energy to Break** 

$$=\frac{190\times9.9070}{118.983}=15.820$$

**Young Modulus** 

$$= \frac{0.140}{11.748} = 0.0119$$

$$=\frac{51,2}{51.5}=99.42\%$$

Mass of grounded kenaf = 600.5g

Mass of Starch = 1050.875g

Mass of Water added = 1801.5g

Total Mass of Input = 3683.7g

Unpelleted Mass = 141.1g

Mass of Pellets = 3065.5g

Total Mass of Output = 3206.6g

## **Pelleting Efficiency**

$$\eta \% = \frac{3065.5}{3206.6} \times 100\%$$
$$= 95.60\%$$

#### **Percentage Recovery**

$$R\% = \frac{3206.6}{3683.7} \times 100\%$$
$$= 87.05\%$$

**Energy to Peak** 

$$=\frac{172\times4.4060}{52.948}=14.313$$

**Energy to Yield** 

$$=\frac{174\times4.5140}{54.246}=14.479$$

**Energy to Break** 

$$=\frac{178\times4.925}{59.241}=14.798$$

**Young Modulus** 

$$=\frac{0.203}{9.028}=0.0225$$

$$=\frac{43.3}{43.8}=98.86\%$$

Mass of grounded kenaf = 600 g

Mass of Starch = 750g

Mass of Water added = 1800.1g

Total Mass of Input = 3150.1g

Unpelleted Mass = 314.2g

Mass of Pellets = 1888.9g

Total Mass of Output = 2203.1g

## **Pelleting Efficiency**

$$\eta \% = \frac{1888.9}{2203.1} \times 100\%$$
$$= 85.74\%$$

## **Percentage Recovery**

$$R\% = \frac{2203.1}{3150.1} \times 100\%$$
$$= 69.94\%$$

#### **Energy to Peak**

$$=\frac{280\times3.500}{36.164}=27.098$$

**Energy to Yield** 

$$=\frac{342\times4.3420}{46.155}=32.173$$

**Energy to Break** 

$$=\frac{504\times9.9320}{113.288}=44.186$$

**Young Modulus** 

$$=\frac{0.400}{8.684}=0.0461$$

$$=\frac{53.7}{54.1}=99.26\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 1050g

Mass of Water added = 1800g

Total Mass of Input = 3450g

Unpelleted Mass = 282.4g

Mass of Pellets = 2731.6g

Total Mass of Output = 3014g

## **Pelleting Efficiency**

$$\eta \% = \frac{2731.6}{3014} \times 100\%$$
$$= 90.63\%$$

**Percentage Recovery** 

$$R\% = \frac{3014}{3450} \times 100\%$$
$$= 87.36\%$$

**Energy to Peak** 

$$=\frac{126\times3.625}{45.535}=10.031$$

**Energy to Yield** 

$$=\frac{184\times5.8180}{71.809}=14.908$$

**Energy to Break** 

$$=\frac{196\times7.550}{92.589}=15.982$$

**Young Modulus** 

$$=\frac{0.215}{11.636}=\ 0.0185$$

$$=\frac{54.7}{55.5}=98.56\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 1050g

Mass of Water added = 1800g

Total Mass of Input = 3450g

Unpelleted Mass = 270.3g

Mass of Pellets = 2566.3g

Total Mass of Output = 2836.6g

**Pelleting Efficiency** 

$$\eta \% = \frac{2566.3}{2836.6} \times 100\%$$
$$= 90.47\%$$

**Percentage Recovery** 

$$R\% = \frac{2836.6}{3450} \times 100\%$$
$$= 82.22\%$$

**Energy to Peak** 

$$=\frac{160\times2.668}{34.146}=12.502$$

**Energy to Yield** 

$$=\frac{234\times5.790}{71.609}=18.920$$

**Energy to Break** 

$$=\frac{256\times8.2440}{101.081}=20.879$$

**Young Modulus** 

$$=\frac{0.563}{11.590}=0.0486$$

$$=\frac{53.9}{54.1}=99.63\%$$

Mass of grounded kenaf = 600.2g

Mass of Starch = 900.3g

Mass of Water added = 1800.6g

Total Mass of Input = 3301.1g

Unpelleted Mass = 222.1g

Mass of Pellets = 2603.6g

Total Mass of Output = 2825.7g

**Pelleting Efficiency** 

$$\eta \% = \frac{2603.6}{2825.7} \times 100\%$$
$$= 92.14\%$$

**Percentage Recovery** 

$$R\% = \frac{2825.7}{3301.1} \times 100\%$$
$$= 85.6\%$$

**Energy to Peak** 

$$=\frac{238\times4.186}{63.529}=15.682$$

**Energy to Yield** 

$$=\frac{266\times6.421}{89.405}=19.104$$

**Energy to Break** 

$$=\frac{285\times9.812}{117.196}=\qquad 23.861$$

**Young Modulus** 

$$= \frac{0.552}{11.948} = 0.0462$$

$$=\frac{45.7}{45.9}=99.56\%$$

Mass of grounded kenaf = 600.1g

Mass of Starch = 900.2g

Mass of Water added = 1800.3g

Total Mass of Input = 3300.6g

Unpelleted Mass = 200.7g

Mass of Pellets = 2602.5g

Total Mass of Output = 2803.2g

**Pelleting Efficiency** 

$$\eta \% = \frac{2602.5}{2803.2} \times 100\%$$
$$= 92.84\%$$

**Percentage Recovery** 

$$R\% = \frac{2803.2}{3300.6} \times 100\%$$
$$= 84.93\%$$

**Energy to Peak** 

$$=\frac{228\times4.278}{58.889}=16.563$$

**Energy to Yield** 

$$=\frac{254\times7.112}{82.528}=21.889$$

**Energy to Break** 

$$=\frac{272\times9.624}{107.100}=24.442$$

**Young Modulus** 

$$=\frac{0.538}{11.747}=0.0458$$

$$=\frac{59.0}{59.5}=99.16\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 900g

Mass of Water added = 1800g

Total Mass of Input = 3300g

Unpelleted Mass = 233.7g

Mass of Pellets = 2355.1g

Total Mass of Output = 2588.9g

**Pelleting Efficiency** 

$$\eta \% = \frac{2355.1}{2588.9} \times 100\%$$
$$= 90.97\%$$

**Percentage Recovery** 

$$R\% = \frac{2588.9}{3300} \times 100\%$$
$$= 78.45\%$$

**Energy to Peak** 

$$=\frac{228\times3.368}{45.988}=16.698$$

**Energy to Yield** 

$$=\frac{272\times5.786}{77.275}=20.366$$

**Energy to Break** 

$$=\frac{309\times8.796}{109.564}=\qquad 24.807$$

**Young Modulus** 

$$=\frac{0.516}{12.079}=\ 0.0427$$

$$=\frac{53.7}{54.1}=99.26\%$$

Mass of grounded kenaf = 600.2g

Mass of Starch = 900.3g

Mass of Water added = 1800.6g

Total Mass of Input = 3301.1g

Unpelleted Mass = 183.9g

Mass of Pellets = 2255.3g

Total Mass of Output = 2439.2g

**Pelleting Efficiency** 

$$\eta \% = \frac{2255.3}{2439.2} \times 100\%$$
$$= 92.46\%$$

**Percentage Recovery** 

$$R\% = \frac{2439.2}{3301.1} \times 100\%$$
$$= 73.89\%$$

**Energy to Peak** 

$$=\frac{244\times3.628}{46.000}=19.244$$

**Energy to Yield** 

$$=\frac{272\times7.162}{82.482}=23.618$$

**Energy to Break** 

$$=\frac{289\times9.642}{108.001}=25.801$$

**Young Modulus** 

$$=\frac{0.595}{11.377}=0.0523$$

$$=\frac{56.2}{56.7}=99.12\%$$

Mass of grounded kenaf = 600.2g

Mass of Starch = 900.3g

Mass of Water added = 1800.6g

Total Mass of Input = 3301.1g

Unpelleted Mass = 305.7g

Mass of Pellets = 2420.7g

Total Mass of Output = 2726.4g

**Pelleting Efficiency** 

$$\eta \% = \frac{2420.7}{2726.4} \times 100\%$$
$$= 88.79\%$$

**Percentage Recovery** 

$$R\% = \frac{2726.4}{3301.1} \times 100\%$$
$$= 82.59\%$$

**Energy to Peak** 

$$=\frac{248\times2.705}{73.597}=9.115$$

**Energy to Yield** 

$$=\frac{264\times6.512}{152.571}=11.268$$

**Energy to Break** 

$$=\frac{274\times7.965}{167.994}=12.991$$

**Young Modulus** 

$$=\frac{0.521}{10.568}=\ 0.0493$$

$$=\frac{35.7}{35.8}=99.72\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 900g

Mass of Water added = 1800g

Total Mass of Input = 3300g

Unpelleted Mass = 398.7g

Mass of Pellets = 1861.5g

Total Mass of Output = 2260.2g

**Pelleting Efficiency** 

$$\eta \% = \frac{1861.5}{2260.2} \times 100\%$$
$$= 82.36\%$$

**Percentage Recovery** 

$$R\% = \frac{2260.2}{3300} \times 100\%$$
$$= 68.49\%$$

**Energy to Peak** 

$$=\frac{256\times3.649}{49.000}=19.064$$

**Energy to Yield** 

$$=\frac{324\times5.729}{58.614}=31.668$$

**Energy to Break** 

$$=\frac{440\times8.993}{86.505}=45.742$$

**Young Modulus** 

$$=\frac{0.296}{11.298}=0.0262$$

$$=\frac{60.2}{61.0}=98.69\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 900g

Mass of Water added = 1800g

Total Mass of Input = 3300g

Unpelleted Mass = 141.6g

Mass of Pellets = 2725.4g

Total Mass of Output = 2867.0g

**Pelleting Efficiency** 

$$\eta \% = \frac{2725.4}{2867.0} \times 100\%$$
$$= 95.06\%$$

**Percentage Recovery** 

$$R\% = \frac{2867.0}{3300} \times 100\%$$
$$= 86.88\%$$

**Energy to Peak** 

$$=\frac{140\times2.008}{42.998}=6.538$$

**Energy to Yield** 

$$=\frac{163\times4.889}{96.877}=8.226$$

**Energy to Break** 

$$=\frac{204\times7.881}{138.251}=11.629$$

**Young Modulus** 

$$=\frac{0.349}{11.044}=\ 0.0316$$

$$= \frac{61.1}{62.0} = 98.55\%$$

TEST 08, 10, 18, 23, 25, 29

Mass of grounded kenaf = 600.2g

Mass of Starch = 900.3g

Mass of Water added = 1800.6g

Total Mass of Input = 3301.1g

Unpelleted Mass = 227.8g

Mass of Pellets = 2490.9g

Total Mass of Output = 2718.7g

**Pelleting Efficiency** 

$$\eta \% = \frac{2490.9}{2718.7} \times 100\%$$
$$= 91.62\%$$

**Percentage Recovery** 

$$R\% = \frac{2718.7}{3301.1} \times 100\%$$
$$= 82.36\%$$

**Energy to Peak** 

$$=\frac{236\times3.517}{62.846}=13.207$$

**Energy to Yield** 

$$=\frac{284\times7.036}{130.017}=15.369$$

**Energy to Break** 

$$=\frac{292\times9.483}{164.315}=16.852$$

**Young Modulus** 

$$=\frac{0.538}{10.447}=0.0515$$

$$= \frac{58.7}{59.5} = 98.7\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 1200g

Mass of Water added = 1800g

Total Mass of Input = 3600g

Unpelleted Mass = 139.2g

Mass of Pellets = 3038.5g

Total Mass of Output = 3177.7g

**Pelleting Efficiency** 

$$\eta \% = \frac{3038.5}{3177.7} \times 100\%$$
$$= 95.62\%$$

**Percentage Recovery** 

$$R\% = \frac{3177.7}{3600} \times 100\%$$
$$= 88.27\%$$

**Energy to Peak** 

$$=\frac{252\times3.813}{33.049}=29.074$$

**Energy to Yield** 

$$=\frac{318\times6.936}{59.907}=36.818$$

**Energy to Break** 

$$=\frac{486\times10.238}{102.435}=48.574$$

**Young Modulus** 

$$=\frac{0.578}{10.248}=\ 0.0564$$

$$=\frac{70.3}{70.5}=99.72\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 1050g

Mass of Water added = 1800g

Total Mass of Input = 3450g

Unpelleted Mass = 165g

Mass of Pellets = 2525.2g

Total Mass of Output = 2690.2g

**Pelleting Efficiency** 

$$\eta \% = \frac{2525.2}{2690.2} \times 100\%$$
$$= 93.87\%$$

**Percentage Recovery** 

$$R\% = \frac{2690.2}{3450} \times 100\%$$
$$= 77.98\%$$

**Energy to Peak** 

$$=\frac{254\times3.497}{49.722}=17.864$$

**Energy to Yield** 

$$=\frac{288\times5.887}{73.805}=22.958$$

**Energy to Break** 

$$=\frac{324\times9.58}{114.926}=\qquad 27.008$$

**Young Modulus** 

$$=\frac{0.484}{11.262}=0.0412$$

$$=\frac{63.7}{64.5}=98.76\%$$

Mass of grounded kenaf = 600.2g

Mass of Starch = 750.2g

Mass of Water added = 1800.6g

Total Mass of Input = 3151g

Mass of Pellets = 2001.1g

Total Mass of Output = 2206.7g

Unpelleted Mass = 205.6g

**Pelleting Efficiency** 

$$\eta \% = \frac{2001.1}{2206.7} \times 100\%$$
$$= 90.68\%$$

**Percentage Recovery** 

$$R\% = \frac{2206.7}{3151} \times 100\%$$
$$= 70.03\%$$

**Energy to Peak** 

$$=\frac{153\times2.418}{44.001}=8.408$$

**Energy to Yield** 

$$=\frac{181\times4.226}{76.121}=10.049$$

**Energy to Break** 

$$=\frac{223\times6.385}{101.651}=14.007$$

**Young Modulus** 

$$=\frac{0.412}{11.381}=0.0362$$

$$= \frac{57.1}{57.9} = 98.62\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 1050g

Mass of Water added = 1800g

Total Mass of Input = 3450g

Unpelleted Mass = 143.7g

Mass of Pellets = 2625.2g

Total Mass of Output = 2768.9g

**Pelleting Efficiency** 

$$\eta \% = \frac{2625.2}{2768.9} \times 100\%$$
$$= 94.81\%$$

**Percentage Recovery** 

$$R\% = \frac{2768.9}{3450} \times 100\%$$
$$= 80.26\%$$

**Energy to Peak** 

$$=\frac{260\times3.468}{49.715}=18.137$$

**Energy to Yield** 

$$=\frac{291\times6.218}{80.088}=\quad 22.593$$

**Energy to Break** 

$$=\frac{313\times9.638}{117.996}=\qquad25.569$$

**Young Modulus** 

$$=\frac{0.454}{10.657}=0.0426$$

$$=\frac{52.3}{52.7}=99.24\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 750g

Mass of Water added = 1800g

Total Mass of Input = 3150g

Mass of Pellets = 1919.5g

Total Mass of Output = 2233.1g

Unpelleted Mass = 313.6g

**Pelleting Efficiency** 

$$\eta \% = \frac{1919.5}{2233.1} \times 100\%$$
$$= 85.96\%$$

**Percentage Recovery** 

$$R\% = \frac{2233.1}{3150} \times 100\%$$
$$= 70.89\%$$

**Energy to Peak** 

$$=\frac{278\times3.583}{38.545}=25.842$$

**Energy to Yield** 

$$=\frac{336\times5.728}{65.445}=29.408$$

**Energy to Break** 

$$=\frac{402\times9.982}{103.273}=38.856$$

**Young Modulus** 

$$=\frac{0.412}{11.381}=0.0483$$

$$= \frac{43.5}{43.7} = 99.54\%$$

Mass of grounded kenaf = 600g

Mass of Starch = 750g

Mass of Water added = 1800g

Total Mass of Input = 3150g

Mass of Pellets = 1919.5g

Total Mass of Output = 2233.1g

Unpelleted Mass = 313.6g

**Pelleting Efficiency** 

$$\eta \% = \frac{1919.5}{2233.1} \times 100\%$$
$$= 85.96\%$$

**Percentage Recovery** 

$$R\% = \frac{2233.1}{3150} \times 100\%$$
$$= 70.89\%$$

**Energy to Peak** 

$$=\frac{204\times3.298}{45.999}=14.626$$

**Energy to Yield** 

$$=\frac{248\times4.926}{75.992}=16.076$$

**Energy to Break** 

$$=\frac{277\times8.564}{120.363}=19.709$$

**Young Modulus** 

$$=\frac{0.398}{11.437}=0.0348$$

$$= \frac{50.8}{51.5} = 98.64\%$$

## **APPENDIX IV: OTHER RESULTS**

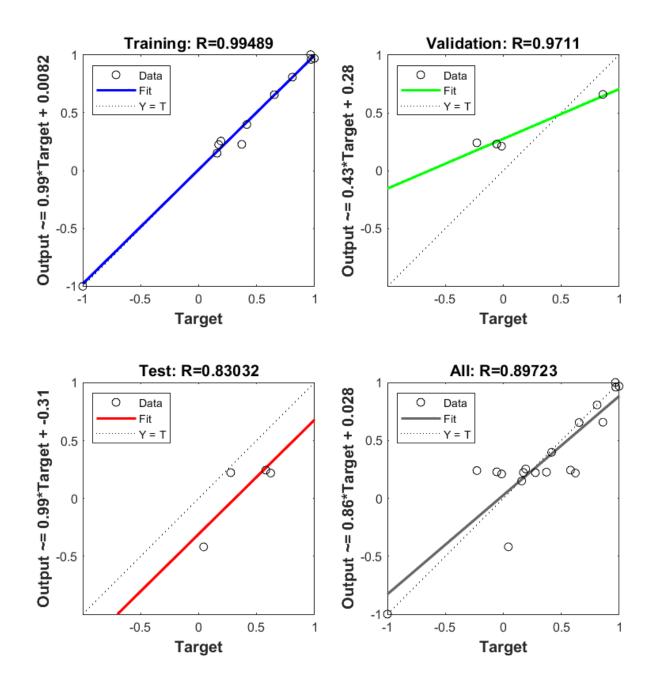
## 1. Response 1 Pelleting Efficiency

Experimental and Predicted Training Data for Pelleting Efficiency

Experimental	Predicted
95.6	95.77138
92.14	91.86856
92.84	92.75073
95.76	95.61118
93.87	91.85194
92.62	91.89061
91.55	91.50601
91.62	91.87664
94.81	94.79894
91.62	91.87664
90.68	91.8107
93.66	91.97633
94.03	94.03959
95.06	94.05226
91.72	92.02812
90.97	88.6565
89.61	91.95694
91.62	91.87664
85.74	85.74407
95.62	95.56648
90.47	91.90298

## Experimental and Predicted Testing Data for Pelleting Efficiency

Experimental	Predicted
92.66	92.18113
91.62	90.62508
82.36	91.10611
91.62	90.62508
85.96	85.44397
90.63	94.28185
88.76	91.86691
91.62	90.62508
92.46	92.0795



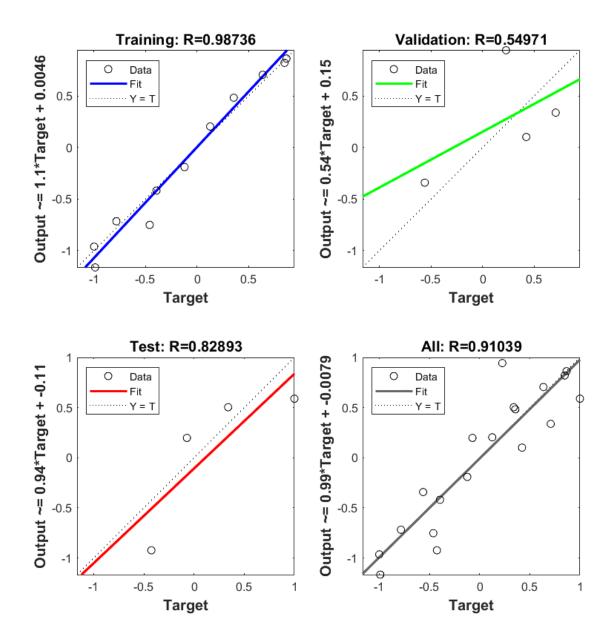
Regression Plot for Pelleting Efficiency

Response 2, Percentage Recovery
 Experimental and Predicted Training Data for Percentage Recovery

Experimental	Predicted
87.05	87.01756
85.6	82.20201
84.93	85.57002
81.18	87.75429
77.98	77.35098
73.95	75.96903
71.92	72.52811
82.36	83.53299
80.26	80.97207
82.36	83.53299
70.03	68.4233
82.98	80.03215
75.49	75.26602
86.88	86.62724
74.88	72.20737
78.45	80.91902
75.21	70.64737
82.36	83.53299
69.94	70.2753
88.27	84.5046
82.22	83.72285

### Experimental and Predicted Testing Data for Percentage Recovery

Experimental	Predicted
83.74	75.58591
82.36	82.16588
68.49	73.52882
82.36	82.16588
70.85	77.58185
87.36	87.56096
82.59	76.41222
82.36	82.16588
73.89	84.67524



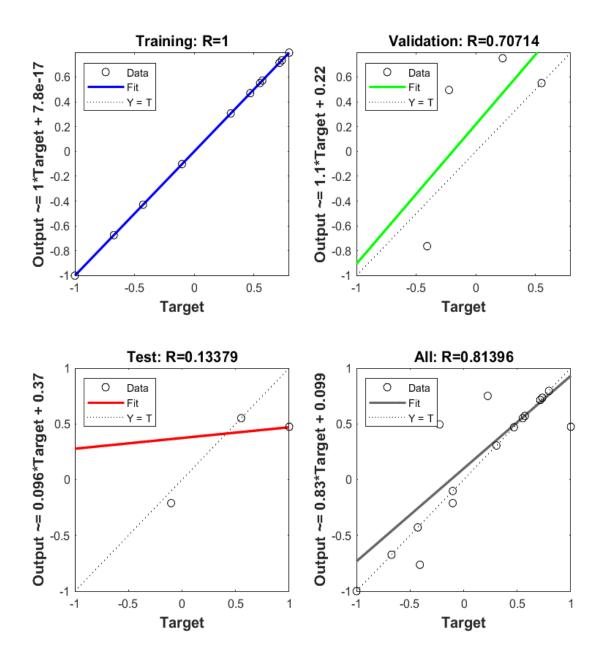
Regression Plot for Percentage Recovery

Response 3, Force at Peak
 Experimental and Predicted Training Data for Force at Peak

Experimental	Predicted
172	161.3785
238	238
228	228
212	212
254	254
204	255.6034
116	116
236	236
260	260
236	236
153	259.4597
172	172
142	107.1999
140	140
84	84
228	228
252	252
236	236
280	228.5046
252	252
160	230.5122

Experimental and Predicted Testing Data for Force at Peak

Experimental	Predicted
155	174.705
236	233.4407
256	232.4848
236	233.4407
278	284.385
126	226.7352
248	238.5604
236	233.4407
244	69.60067



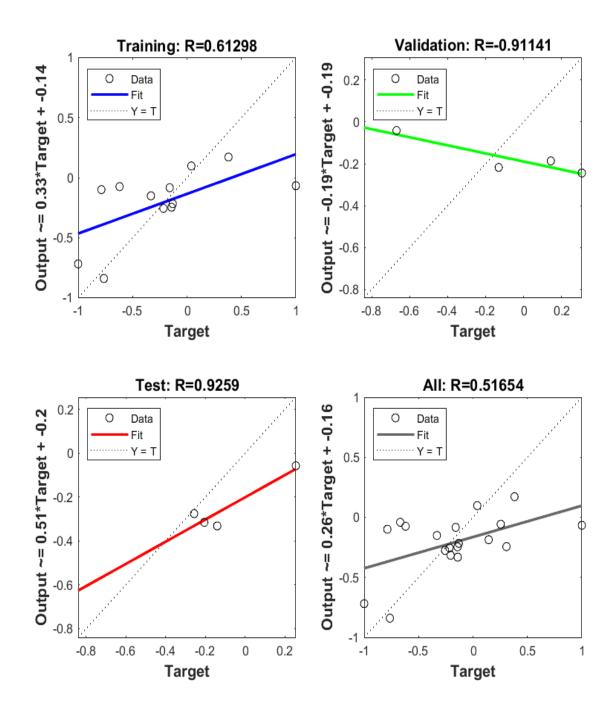
Regression Plot for Force at Peak

Response 4, Deflection at Peak
 Experimental and Predicted Training Data for Deflection at Peak

Experimental	Predicted
4.406	4.042558
4.186	3.644196
4.278	3.320643
3.168	3.48208
3.497	3.317098
3.298	3.265235
3.994	3.420936
3.517	3.367126
3.468	3.599062
3.517	3.367126
2.418	2.286068
2.582	3.673295
3.387	3.196366
2.008	2.496318
2.378	3.571701
3.368	3.300918
5.481	3.628536
3.517	3.367126
3.5	3.167945
3.813	3.914009
2.668	3.615259

Experimental and Predicted Testing Data for Deflection at Peak

Experimental	Predicted
3.041	2.179283
3.517	3.254839
3.649	3.598784
3.517	3.254839
3.583	3.270527
3.625	4.047698
2.705	3.133589
3.517	3.254839
3.628	3.440566



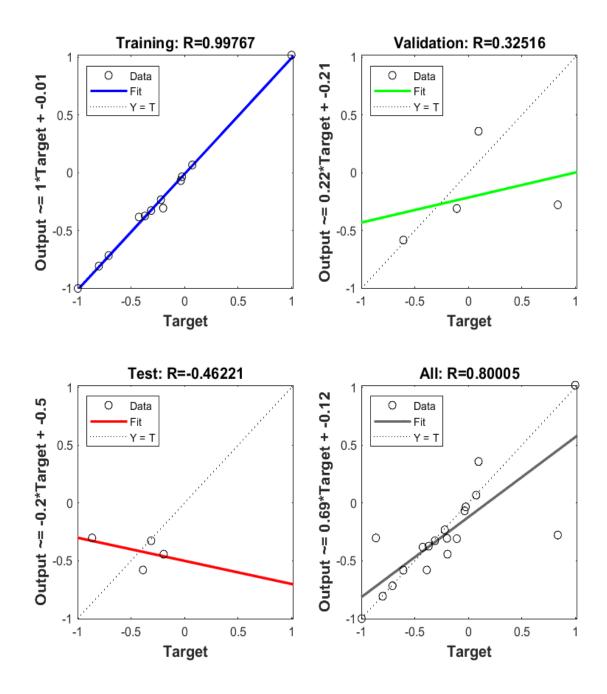
Regression Plot for Deflection at Peak

Response 5, Energy to Peak
 Experimental and Predicted Training Data for Energy to Peak

Experimental	Predicted
14.313	14.1766
15.682	13.25793
16.563	16.16787
7.301	7.231756
17.864	17.80517
14.626	11.633
9.642	9.953351
13.207	13.04221
18.137	21.33051
13.207	13.04221
8.408	8.331856
12.284	9.979903
11.829	12.36293
6.538	13.3324
4.927	4.890631
16.694	16.57672
14.572	13.2914
13.207	13.04221
27.098	13.63874
29.074	29.30592
12.502	12.47106

# Experimental and Predicted Testing Data for Energy to Peak

Experimental	Predicted
9.82	13.31206
13.207	10.85845
19.064	19.0585
13.207	10.85845
25.842	18.81637
10.031	34.89063
9.115	9.286507
13.207	10.85845
19.244	11.62092



Regression Plot for Energy to Peak

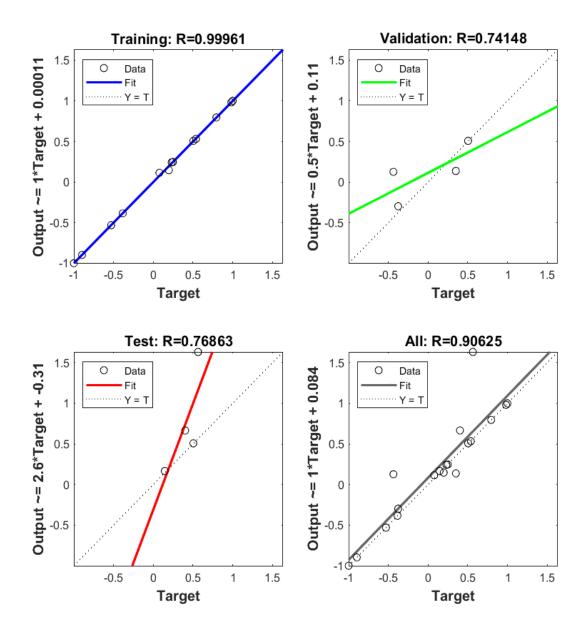
6. Response 6, Force at Yield

Experimental and Predicted Training Data for Force at Yield

Experimental	Predicted
174	239.8701
266	241.1014
254	253.9985
340	339.8876
288	287.3798
248	242.1666
120	119.9977
284	284.4615
291	416.1197
284	284.4615
181	190.1854
242	244.6275
180	180.0393
163	162.899
108	108.0727
272	302.9584
252	253.4681
284	284.4615
342	342.0106
318	318.2158
234	238.3893

Experimental and Predicted Testing Data for Force at Yield

Experimental	Predicted
181	-264.263
284	301.7162
328	508.5683
284	301.7162
336	422.4239
184	291.0256
264	261.6808
284	301.7162
272	209.6192



Regression Plot for Force at Yield

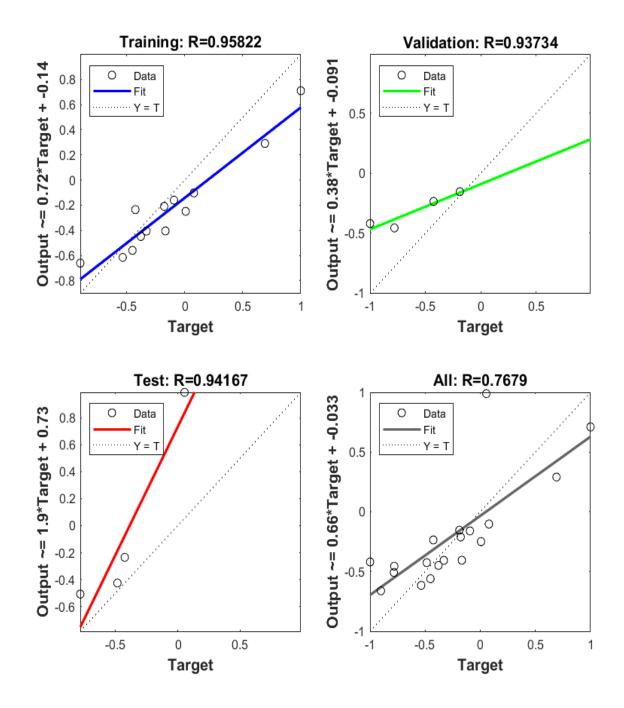
7. Response 7, Energy to Yield

Experimental and Predicted Training Data for Energy to Yield

Experimental	Predicted
14.479	15.38002
19.104	18.61487
21.889	18.02855
16.801	15.67113
22.958	20.23874
16.076	15.02945
9.996	14.14375
15.369	18.24004
22.593	36.65195
15.369	18.24004
10.049	14.92897
19.263	15.68926
14.987	13.35991
8.226	11.8364
6.745	15.47026
20.366	19.37719
13.718	12.52257
15.369	18.24004
32.173	26.15259
36.818	32.46336
18.92	19.46569

Experimental and Predicted Testing Data for Energy to Yield

Experimental	Predicted
12.187	11.43191
15.369	21.07361
31.668	33.92573
15.369	21.07361
29.408	28.99509
14.908	20.38365
11.268	18.21696
15.369	21.07361
23.618	13.34311



Regression Plot for Force at Yield

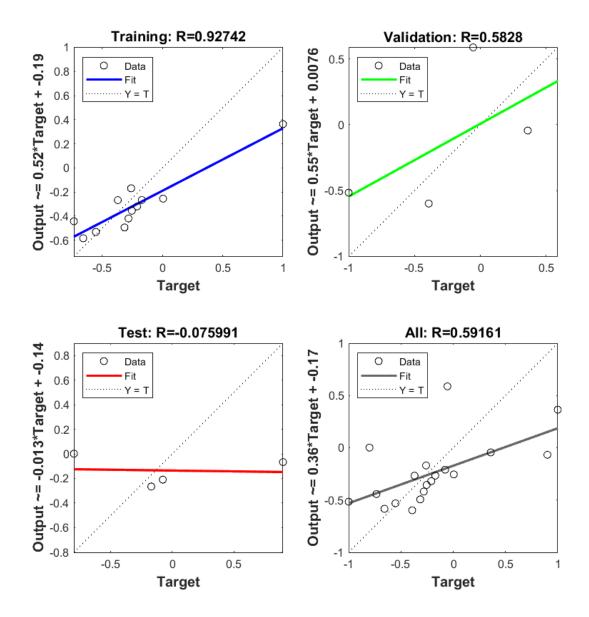
8. Response 8. Force at Break

Experimental and Predicted Training Data for Force at Break

Experimental	Predicted
178	323.32
285	265.2379
272	247.1665
388	315.0007
324	276.8836
277	258.7204
190	242.8939
292	274.8939
313	429.2964
292	274.8939
223	226.8628
266	233.635
276	292.4234
204	217.3983
142	229.6625
309	285.0866
252	214.7553
292	274.8939
504	388.8375
486	310.938
256	274.6622

Experimental and Predicted Testing Data for Force at Break

<b>Experimental</b>	<b>Predicted</b>
227	215.0248
292	291.7792
440	415.4341
292	291.7792
402	223.1216
196	393.6621
274	272.0999
292	291.7792
289	267.2791



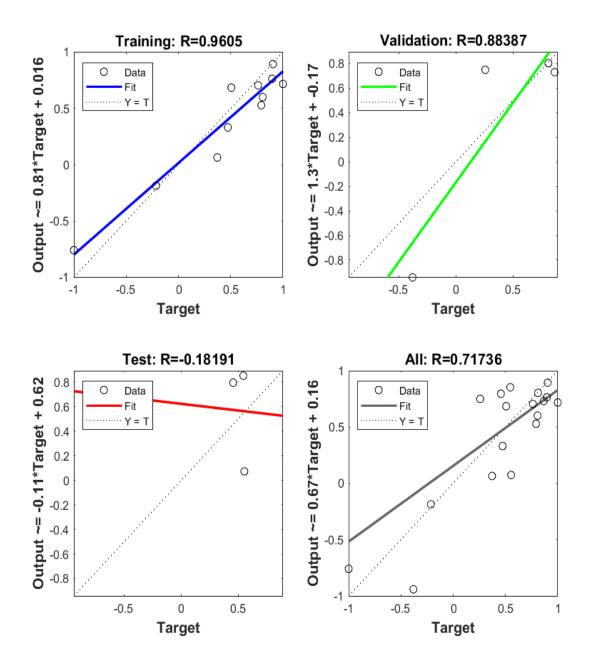
Regression Plot for Force at Break

Response 9, Deflection at Break
 Experimental and Predicted Training Data for Deflection at Break

Experimental	Predicted
4.925	9.215613
9.812	9.381321
9.624	8.970711
8.671	9.236476
9.58	8.742797
8.564	8.114957
9.907	9.489551
9.483	9.301564
9.638	9.612578
9.483	9.301564
6.385	6.472228
8.823	7.297227
8.513	9.590124
7.881	9.443189
3.89	4.654398
8.796	9.770188
5.856	4.077245
9.483	9.301564
9.932	9.900351
10.238	9.342213
8.244	7.269893

Experimental and Predicted Testing Data for Deflection at Break

Experimental	Predicted
8.686	6.123036
9.483	9.633549
8.993	9.818925
9.483	9.633549
9.982	9.912213
7.55	7.906912
7.965	8.212424
9.483	9.633549
9.642	8.953913



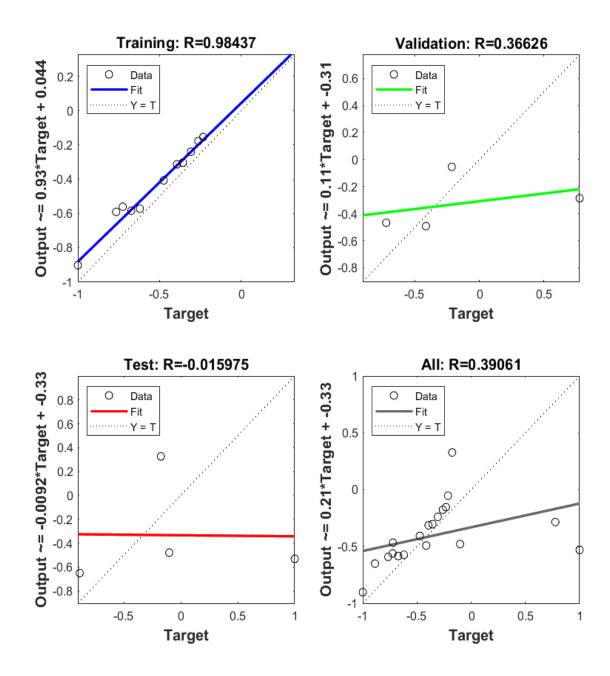
Regression Plot for Deflection at Break

10. Response 10, Energy to BreakExperimental and Predicted Training Data for Energy to Break

Experimental	Predicted
14.798	18.00845
23.861	25.52177
24.442	25.97094
21.29	22.84937
27.008	19.61599
19.709	21.02485
15.82	17.55641
16.852	17.77904
25.569	35.40446
16.852	17.77904
14.007	17.42071
22.012	23.04758
22.968	24.29695
11.629	16.25178
9.421	11.31192
24.807	27.95372
14.864	19.86365
16.852	17.77904
44.186	23.40656
48.574	18.60225
20.879	19.36034

Experimental and Predicted Testing Data for Energy to Break

Experimental	Predicted
15.994	25.85562
16.852	20.88819
45.742	27.45991
16.852	20.88819
38.856	36.97891
15.982	18.88127
12.991	17.76488
16.852	20.88819
25.801	16.92587



Regression Plot for Energy to Break

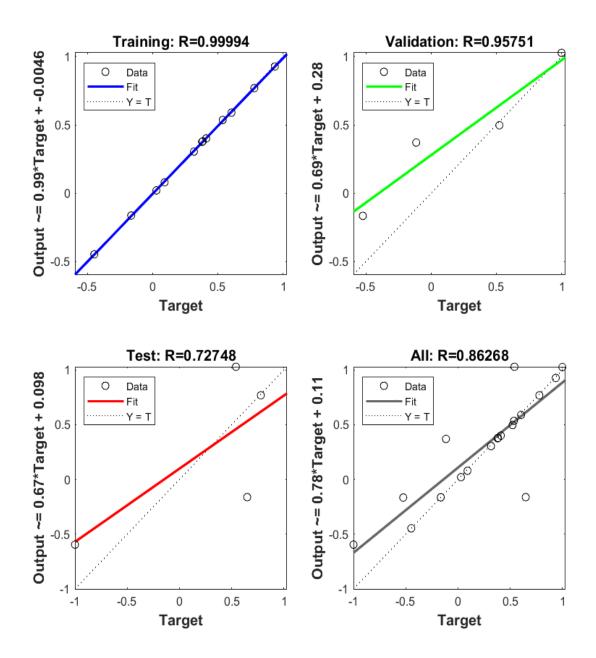
11. Response 11, Young Modulus

Experimental and Predicted Training Data for Young Modulus

Experimental	Predicted
0.0225	0.030472
0.0462	0.057067
0.0458	0.04522
0.0305	0.030525
0.0412	0.040935
0.0348	0.034619
0.0119	0.020909
0.0515	0.051265
0.0426	0.042527
0.0515	0.051265
0.0362	0.035934
0.0476	0.047273
0.0242	0.024213
0.0316	0.042399
0.0433	0.043075
0.0427	0.042609
0.055	0.054777
0.0515	0.051265
0.0461	0.046069
0.0564	0.057004
0.0486	0.030557

Experimental and Predicted Testing Data for Young Modulus

Experimental	Predicted
0.0393	0.044867
0.0515	0.049404
0.0262	0.044922
0.0515	0.049404
0.0483	0.043759
0.0185	0.060155
0.0493	0.03296
0.0515	0.049404
0.0523	0.053813



Regression Plot for Young Modulus

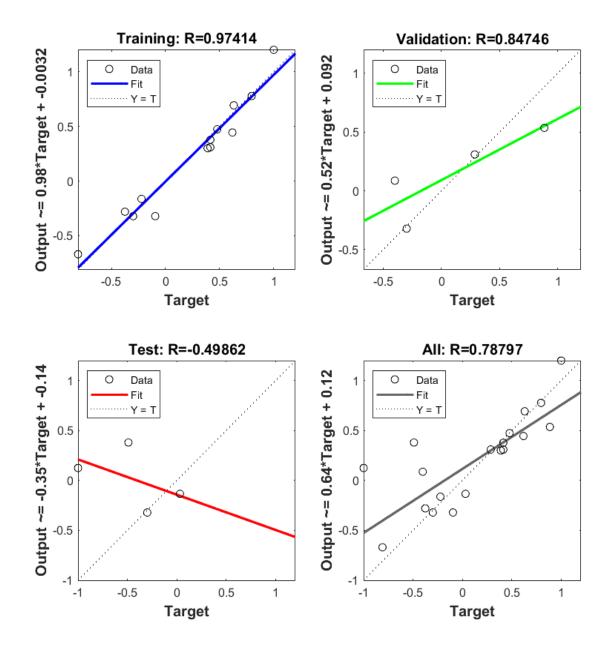
12. Response 12, Durability

Experimental and Predicted Training Data for Durability

Experimental	Predicted
98.86	98.68312
99.56	99.5452
99.16	99.17817
98.3	98.40931
98.76	98.80716
98.64	98.71508
99.42	99.28395
98.7	98.68286
99.24	99.17073
98.7	98.68286
98.62	99.0034
99.43	99.47876
98.15	99.0326
98.55	99.23321
98.96	98.82965
99.26	99.17743
99.31	99.30696
98.7	98.68286
99.26	99.23177
99.72	99.8772
99.63	99.35572

Experimental and Predicted Testing Data for Durability

Experimental	Predicted
98.84	98.69505
98.7	98.9737
98.69	99.22418
98.7	98.9737
99.54	99.29559
98.56	98.7076
99.72	98.71921
98.7	98.9737
99.12	98.52399



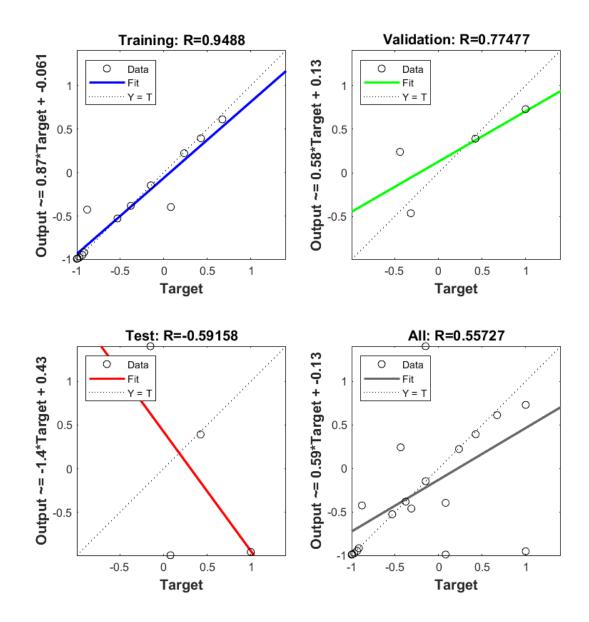
Regression Plot for Durability

13. Response 13, Oil in WaterExperimental and Predicted Training Data for Oil in Water Removal

Experimental	Predicted
6060.61	12658
20000	4481.803
16666.7	16694.19
14285.7	12144.4
33333.3	29409.5
4761.91	4668.342
20000	13086.58
25000	24523.43
28571.4	27694.01
25000	24523.43
16666.7	39173.37
4347.83	4463.297
5263.16	5089.009
11111.1	11189.73
12500	22334.76
13333.3	13289.82
5555.55	5542.179
25000	24523.43
33333.3	5024.72
22222.2	22051.65
4444.44	4534.868

Experimental and Predicted Testing Data for Oil in Water Removal

Experimental	Predicted
5000	25112.8
25000	29245.68
50000	51239.94
25000	29245.68
33333.3	57580.44
4255.32	4651.269
4166.67	4552.785
25000	29245.68
22222.2	13192.27



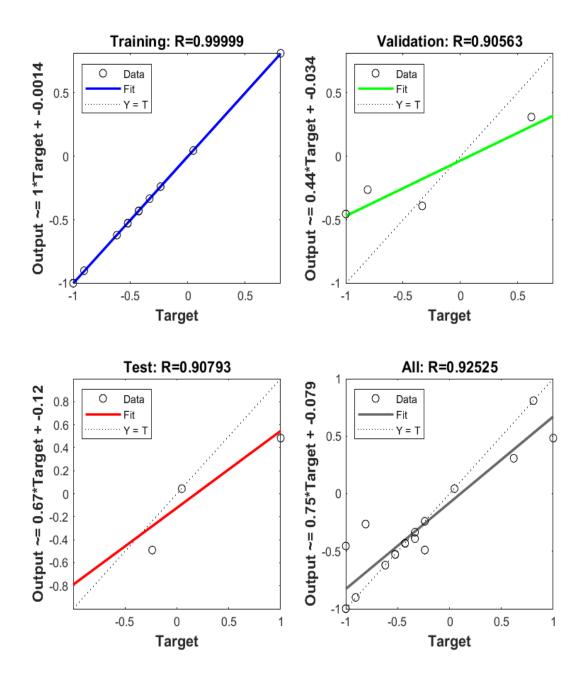
Regression Plot for Oil in Water Removal

14. Response 14, Changes in pHExperimental and Predicted Training Data for Changes in pH

Experimental	Predicted
0.09	0.089891
0.06	0.059719
0.08	0.079945
0.22	0.165798
0.02	0.020412
0.07	0.06966
0.18	0.147373
0.12	0.119698
0.01	0.010157
0.12	0.119698
0.06	0.059565
0.08	0.073983
0.2	0.200046
0.06	0.05976
0.07	0.069956
0.05	0.049824
0.01	0.067275
0.12	0.119698
0.01	0.010054
0.03	0.087334
0.09	0.063665

Experimental and Predicted Testing Data for Changes in pH

Experimental	Predicted
0.04	0.114164
0.12	0.082422
0.02	0.041558
0.12	0.082422
0.02	0.034846
0.09	0.034261
0.02	0.061681
0.12	0.082422
0.07	0.154563



Regression Plot for Changes in pH

### **MATLAB CODE**

```
%% ANN CODE
% Leave one out Validation Method.
clear;clc
close all
tic
Data= xlsread('Raw Data.xlsx','D5:U34');
TrainData= Data(1:21, :);
TestData = Data(22:end,:);
TestX= TestData(:,1:4);
TestY = TestData(:,18);
p= TrainData(:,1:4);
t= TrainData(:,18);
%%
% p= Input(5:20,:);
% t= Output(5:20,1); % Inputs
ActualTrain= t;
TestInput= TestX;
TestOutput= TestY;
%% Training Data
trnData = [p t];
```

```
% Save Network
 save('Resp14');
%% Simulate Network and Post Processing
%
a1=sim(net8,pn);
 anew1=mapminmax('reverse',a1,ts);
 YPredTrain=anew1';
%
   NewDataTrain=[ActualTrain w1];
%
   xlswrite('Simulated ResultTrain.xlsx', NewDataTrain, 'Sheet1')
figure, plotregression(ActualTrain,anew1);
 [m,b,r]=postreg(anew1,ActualTrain')
 NewData= [ActualTrain YPredTrain];
xlswrite('PredictedANN Train.xlsx', NewData, 'Resp14') % Writes the result into an
Excel
t=t';
error = (t-YPredTrain);
%
    MeanSquareError=mse(error)
%
    RMSE= sqrt(MeanSquareError)
  MSE Forecast=mean(error.^2)
 RMSE Forecast=sqrt(MSE Forecast)
 MAD Forecast = mad(error)
 MAPE_Forecast= (sum((abs(error)./t))/length(t))*100
```

```
VAF= (1-(var(TestOutput-anew))/(var(TestOutput)))*100
  RCov= MAD Forecast/median(t)
  rMBE= (mean(YPredTrain-t)/mean(t))*100;
PerfMetricTrain= {'MSE','RMSE','MAD', 'MAPE','Rsq','RCoV','rMBE';MSE Forecast,
RMSE Forecast, MAD Forecast, MAPE Forecast, Rsquare, RCov, rMBE \};
  xlswrite('Performance MetricsANN Train.xlsx',PerfMetricTrain, 'Resp14')
% = [MSE Forecast, RMSE Forecast,
MAD Forecast, MAPE Forecast, Rsquare, RCov, rMBE]
 n1=1:length(YPredTrain);
%% PLOTTINGS
figure,
  plot(n1,t,'--or',n1,YPredTrain,'-.*k');
legend('Experimental','Predicted');
    xlabel('Data Index')
      ylabel('pH')
%%
fprintf('==
                      ===== TESTING
%% TESTING
%%%%%%%%%%%%%%%%%% TESTING
```

[Rsquare rootmse] = rsquare(t,YPredTrain)

```
TestData= mapminmax(TestX');
  a=sim(net8,TestData);
  YPred=mapminmax('reverse',a,ts);
  YPred=YPred';
%
    NewData= [TestY w];
NewData= [TestY YPred];
xlswrite('PredictedANN Test.xlsx', NewData, 'Resp14') % Writes the result into an Excel
error = (TestY-YPred);
%
     MeanSquareError=mse(error)
%
     RMSE= sqrt(MeanSquareError)
  MSE Test=mean(error.^2)
  RMSE Test=sqrt(MSE Test)
  MAD Test = mad(error)
  MAPE Test= (sum((abs(error)./TestY))/length(TestY))*100
  [Rsquare rootmse] = rsquare(TestY,YPred)
   VAF= (1-(var(TestOutput-anew))/(var(TestOutput)))*100
  RCov= MAD Forecast/median(TestY)
  rMBE= (mean(YPred-TestY)/mean(TestY))*100;
```

```
PerfMetricTest= {'MSE','RMSE','MAD', 'MAPE','Rsq','RCoV','rMBE';MSE Test,
RMSE Test, MAD Test, MAPE Test, Rsquare, RCov, rMBE \;
  xlswrite('Performance MetricsANN Test', PerfMetricTest, 'Resp14')
% = [MSE Forecast, RMSE Forecast,
MAD Forecast, MAPE Forecast, Rsquare, RCov, rMBE]
% Prediction Plot
  n=1:length(TestY);
     n=1:length(ActualLM');
% t1 = datetime(2014,12,01) + calmonths(1:length(TestOutput));
%% PLOTTINGS
figure,
  plot(n,TestY,'--or',n,YPred,'-.*k');
legend('Experimental','Predicted');
    xlabel('Data Index')
      ylabel('pH')
%%
toc
```