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NUTRITIONAL EVALUATION OF CROP RESIDUES USING NEAR INFRARED REFLECTANCE SPECTROSCOPY

BY

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DECLARATION

I, Ayuba Abdul Rahman hereby declare that this dissertation is the result of my own original work and that no part of it has been presented for another degree in this university or elsewhere. All sources of information cited and assistance received in the preparation of this work have been duly acknowledged and referenced.

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DEDICATION

I dedicate this work to my parents, Mr. Abdul Rahman Zefo and Ms. Fati George.

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LIST OF ACRONYMS

ADF – Acid Detergent Fibre

AIBPs – Agro-industrial By-products

ADL – Acid Detergent Lignin

AOAC – Association of Official Analytical Chemistry

CF – Crude Fibre

CO_2 – Carbon dioxide

CP – Crude Protein

CT – Condensed Tannins

CV – Cross Variation

DM – Dry Matter

DMD – Dry Matter Digestibility

FAO – Food and Agriculture Organization

GDP – Gross Domestic Product

GSS – Ghana Statistical Service

IITA – International Institute of Tropical Agriculture

ILRI – International Livestock Research Institute

ITC – International Trypanotolerance Centre

IVOMD – In-vitro Organic Matter Digestibility

IVDMD – In-vitro Dry Matter Digestibility

IVGPT - In-vitro gas production technique

MoFA – Ministry of Food and Agriculture

NDF – Neutral Detergent Fibre

NIRS – Near-infrared Reflectance Spectroscopy

OM – Organic Matter

OMD – Organic Matter Digestibility

RPD – Residual Predictive Deviation

SEC – Standard Error of Calibration

SECV – Standard Error of Cross Validation

SEP - Standard Error of Prediction

SSA – Sub-Saharan Africa

WSC – Water Soluble Carbohydrates

ABSTRACT

Traditionally, the nutritional constituents of forages were determined by tedious wet-chemistry methods which did not match with current analytical requirements in terms of quickness, easiness, cheapness, effectiveness and accuracy offered by the near infrared reflectance spectroscopy (NIRS). Two studies were carried out to the use of NIRS for evaluating the nutritional composition (CP, IVOMD and NDF) of crop residues. In the first study, 561 samples of sorghum and 1010 samples of groundnut residues with already determined wet-chemistry values were subjected to NIRS scan to determine the prediction power and analytical efficiency of the NIRS technique in relation to the routine methods. The results showed higher R^2 of 0.89 and 0.86 for CP and NDF respectively and a moderate R^2 of 0.36 for IVOMD. The 1010 groundnut samples analysed with NIRS produced more consistent values (CV% of 15.88, 7.54 and 11.19 for CP, IVOMD and NDF respectively) than values obtained by the wet-chemistry methods (CV% of 18.18, 8.68 and 13.35 for CP, IVOMD and NDF respectively). There was a higher correlation ($r = 0.89$, 0.88 and 0.82 for CP, IVOMD and NDF respectively) between values measured with both techniques ($p<0.05$). In the second study, comparative review was done with NIRS and routine methods viz., accuracy, speed and cost. The results showed that the routine methods are not only time-consuming, they are expensive and also less consistent than NIRS method. In conclusion, the study indicates that NIR spectroscopy calibrated properly for groundnut haulms and sorghum stover and thus has potential for effectively quantifying the nutritional components of sorghum and groundnuts residues with acceptable accuracy, precision and repeatability.

Keywords: Crop Residues, Forage, Livestock, NIR Spectroscopy.

CHAPTER ONE
1.0 INTRODUCTION

1.1 Background

Most nations in the developing world have insufficient feed resources to increase livestock output to meet the rising demand caused by population expansion. In both quality and quantity, forage production remains a key obstacle to the production of livestock in the tropics partly because of seasonal shifts, frequent droughts, and wild bushfires. Long periods of drought and low forage supply have compounding effects on the output of free-ranging animals, necessitating nutritional interventions such as the formulation of animal diets from easily accessible materials to reducing nutritional stress in animals (Konlan *et al.*, 2016).

In many parts of the world especially Africa, the use of crop residues in supplementary feeding serves as a nutritional intervention. Crop leftovers have substantial potential and are readily accessible. In the tropics, they play a significant role in ruminant feeding (Suskombat, 2004). In semiarid and sub humid regions, the most prevalent crop residues are legume haulms (cowpea, peanut) and cereal (millet, maize, rice, and sorghum) stovers and straws. The crop residues are fed in two different ways, they are either grazed on the crop field in situ or stored in barns from which animals are fed from on a daily (Schiere and Kater, 2001). Crop residues in Africa have not been used to its potential because of the heavy tasks associated with transporting, conserving and storing it (Jayasuriya, 2002). The commonest way to conserving residues in Africa is either by drying it on the farm field (sun-curing) or by storing the residues atop a shed in the homestead (Addah and Ayatunde, 2021). This way, the residues are rendered unprotected against the bare sun and the rains causing leaching of the nutrients thereby reducing the quality and quantity of the residues (Addah and Ayatunde, 2021). Poor feed offered to animals translate to low intake and poor animal performance. The quality of crop residues can be tested by chemically judging its composition in the laboratory, conducting a feeding trial or in-vitro digestion trials. Feeding trials are expensive and time consuming whiles laboratory methods are based on destructive determinants and exposes laboratory technicians to chemicals (Kong *et al.*, 2005). Chemical methods and feeding trials are challenging when you look at their cost-effectiveness and time needed to complete one analysis as

against other newly emerging methods of analysis (Singh *et al.*, 2018). In addition, there is poor availability of laboratories in Africa. By combining laboratory data with spectrum information, near-infrared spectroscopy (NIRS) a high-throughput technology which is cost-effective, quick with higher precision, may represent a valuable way of determining the chemical composition of residues by the smallholder farmer (Ramirez *et al.*, 2015; Wu *et al.*, 2023). This technology is non-consumptive and applies the instrumental approach for the quick and accurate determination of the nutritional contents and other related feeding value characteristics of forages assuming the correct protocols are applied correctly (Kong *et al.*, 2005). The near infrared reflectance spectroscopy system of analysis is based on the use of the instrumentation technique for assessing the composition of a sample with minimal or no sample preparation. This new technology operates with the premise that the major chemical constituents of a sample (C, H, O, and N) have a characteristic and unique absorption features in the near infrared region which separate those (Ramirez *et al.*, 2015). In animal production the application of the near infrared spectroscopy to analysing forages and feeds has gained increased interest and consideration. It is used throughout the entire supply chain of animal production to assess the nutritional values of feedstuffs to maximize animal performance, to monitor the evolution of silage at fermentation, nutritional analysis of animal products (milk, meat and eggs), and to identify potential food frauds (Ramirez *et al.*, 2015, 2010). The near infrared method of analysis has four primary benefits: speed, easy preparation of samples, multiple nutritional assessments with a single operation, and non-consumption of the samples further assessment with the same or a different technique. Near infrared reflectance spectroscopy offers a high capacity for prediction; however calibration must be performed with care to avoid failures in prediction. Many researchers have already predicted the forage quality parameters through NIR spectroscopy, for instance CP, NDF and OMD. For CP in various forages and feedstuffs NIRS technique successfully predicted the nutritive values of forages with R^2 (coefficient of determination) values of 0.90 or higher and standard errors well within wet chemistry errors (Mekoya et al., 2008).

On the one hand feeding trials take days to weeks to complete, laborious and exhaustive. On the other hand chemical methods are expensive and limited by speed, which is a critical factor, and are based on destructive determinants. In this regard, the application of the near infrared reflectance spectroscopy (NIRS) may prove to be an important solution to assessing the nutritional information of forages.

It is worth noting, however, that most of the studies that correctly predicted the composition of forages mainly did so using samples from the temperate. There is not much of such research done here in the tropics, especially on crop residues and this has left a gap in literature on predicting the quality parameters in tropical crop residues with the NIRS method. Moreover, the samples used in those studies were from carefully undertaken experimental fields. As a result there is the likelihood that such results might not be similar to samples taken from farmers' actual, natural farm fields from which the residues are sourced for feeding livestock.

On this basis, this study aimed to predict some quality parameters of groundnut and sorghum crop residues using NIR spectrometer on ground samples.

1.2 Objectives

1.2.1 Main Objective

The primary purpose of this was to assess the accuracy of NIRS in predicting the chemical composition of sorghum and groundnut residues.

1.2.2 Specific Objectives

Study 1

1. To predict the CP, NDF, and IVOMD contents of sorghum residues.
2. To determine the efficiency of NIRS in estimating the CP, NDF, and IVOMD of groundnut residues with respect to the traditional wet-chemistry techniques.

Study 2

To review different feed evaluation methods with emphasis on economic considerations.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Livestock Production

In most Sub-Sahara African countries the keeping of farm animals is important to their economies as it accounts for nearly a third of the gross domestic product (GDP) of agriculture and employs more than 50% of residents in the rural communities (Winrock International, 1992; FAO, 2012). It was reported that almost all of the agricultural produce from Africa are reportedly produced by smallholder farmers (FAO, 2012). In this region, the people's high carbohydrate diet is compensated with quality livestock and livestock products even though about 82% of the dietary protein demand is not met (Karbo and Agyare, 2002). Farming accounts for a fifth of Ghana's GDP, with poultry and livestock providing 6% (MoFA, 2016). Ghana's agriculture sector growth in recent times has impacted its economy so well reducing unemployment by 0.4% from 2014 to 2015 (Oppong-Anane, 2013). Cattle, goats, sheep, poultry and pigs are the major types of livestock reared in Ghana and they found in all the agro-ecological zones (MoFA, 2016). Through utilization of crop residues, provision of traction, and soil fertility regeneration, livestock production makes a considerable contribution to agricultural intensification and crop production sustainability (FAO, 2012). The importance of keeping livestock by the smallholder farmer is that they improve the firmness and resilience of farms, serves as a source of quick income, reduces crop failure risk, and whiles enabling the producers the ability to buy farm inputs (Karbo and Agyare, 2002). As a result most of the smallholder farmers especially in the northern Savanna agro-ecological zones supplements cropping with cattle production (Oppong-Anane, 2013). Farm animal rearing stabilises the socioeconomic capacity of these households by serving as dependable income sources when crop prices are low especially during harvest and they cannot sell much (Oppong-Anane, 2013).

2.2 Production of Small Ruminant

In regions whereby farmers do not typically rear cattle, small ruminants serve significant purposes in sustaining the economies of majority of West African rural households (Lebbie, 2004; Tweneboah, 2000). Small ruminants are kept for money generation, for household use, religious

purposes and for recreational purposes (Ozung *et al.*, 2011). Small ruminants (7.8 million) outnumber cattle (about 1.5 million) in Ghana (VSD, 2009). The production of small ruminants in Ghana relies on traditional extensive systems in which animals are fed household waste and farm residues such as yam, plantain, rice straw, groundnut haulms, cocoyam and cassava peels, these due to their inadequate levels in terms of quantities and quality, results in lower productivity and loss of animal life, particularly lambs (Baiden and Obese, 2010). Small-scale small ruminant farmers in Ghana encounter numerous obstacles when attempting to generate income from their livestock. This occurs as a result of the slow growth, irregular weight gains due to cyclical variations in nutrition, and deficiencies in reproduction (Annor *et al.*, 2007). Ghana imports about 70% of its livestock and livestock products because of its inability to meet the protein requirement of its populace (Okai *et al.*, 2005). The fact that holdings are modest does not appear to motivate livestock keepers to enhance husbandry methods. In the highly rainfall regions where crop farming is done, sheep and goats are tethered in the growing season to prevent crop damage (Konlan *et al.*, 2015). The roles, current systems of production, and required resources to support the rearing of small ruminants are unlikely to experience significant change in the near future. Small ruminant production has been an essential part of majority of peri-urban and urban dwellers and their families for a long time (Baah *et al.*, 2012). According to Oppong-Anane (2011), around one-fourth of all the 13.3 million sheep and goats produced in Ghana are raised by peri-urban and urban populaces.

2.3 Ruminant Nutrition

There are numerous variables that hinder livestock output in sub Saharan Africa, but the insufficient feed resource is the most significant. Even though there is abundance of forages in the humid season, access is limited in some parts of northern Ghana due to compound crop farming (Awuma, 2012). Throughout the agricultural season, these writers claim that feed is inaccessible to cattle in certain communities (MoFA, 2011; Oppong-Anane, 2013, Ansah and Issaka, 2018). In northern Ghana, livestock keepers rely mostly on natural pasture, and residues of crops and by products as the primary source of feed, which provide around half of the overall DM demands of ruminants

annually (Smith, 2002; Oppong-Anane 2013; Konlan *et al.*, 2016; Amole and Ayantunde, 2016). During the dry season, the majority of farmers perform supplemental feeding utilising crop waste. This feed issue, together with other limiting variables, has a substantial impact on the low livestock productivity. Northern Ghana's savanna region is characterised by bush burnings which causes a decline in the nutritional content of green forage due to senescence. Majority of smallholder farmers are unable to provide the nutritional requirement of their livestock during the dry and harsh weather conditions due to the inadequacy of the prevalent management system. In the dry season, the majority of livestock farmed in Ghana's northern parts depend on low quality feedstuffs (Karbo and Agyare, 2002). This circumstance results in cyclical weight increase during the farming season and decreases during the hot season (Annor *et al.*, 2007). Ansah and Issaka (2018) determined in a previous study that grazing on unimproved natural pasture is the most prevalent feeding method in Ghana's Kumbungu District in the Northern Regionand grazing accounted for 80% of the yearly feeding regiment of sheep, goats and cattle in Ghana's Upper West and Northern regions (Konlan *et al.*, 2016). According to farmers, the amount and quality of natural pasture provide challenges throughout the year for grazing. During the rainy season, most grazing fields are abundant with fresh, high-quality forage; however, the utilization of these lands for planting food crops leads to the weeding of the vegetation reducing the livestock's access to quality vegetative forages that serve as feed for them. The protein value of forage is low, with greater values of fibre and weak digestive ability, especially during the dry season (Castillo-Caamal *et al.*, 2003). The exploitation of wastes of crop occurs immediately prior to the latter part of the year and during the first two months of the following year. For the period of the period of crop production, sheep, goats and cattle are restricted and given green fodder (Konlan *et al.*, 2016). Extensive study has been conducted to identify solutions that will benefit livestock farmers in managing the feeding of their animals strategically. The preservation of forage produced during the rainy season for nourishing to stall-fed or partially grazed animals during the dry season is a viable strategy. Other technologies that contribute to the forage stock include boosting the nutritional value of agricultural wastes by physical and chemical

treatment, improved management of rangelands, and the use of agro-industrial by-products by peri-urban farmers (Smith, 2002).

2.4 Feed Resources for Ruminant Production

2.4.1 Natural Pastures

Conferring to Oppong-Annane (2001), one-quarter of Ghana's lands is utilised for natural grazing. The architecture of plant growth in these agro-ecosystems matches well with patterns of the rain (Fianu *et al.*, 2001). Natural pastures accessibility in northern Ghana associates really well with the rainfall patterns. The supply of feed increased throughout the wet season but decreased greatly during the dry harsh times of the year. Most communities prohibit cattle from accessing natural pastures around cultivated crops during the rainy season to prevent damage to crops (Konlan *et al.*, 2016). In the prime of the growing year and just prior to harvesting, ruminants had access to approximately 80% of feed resources from natural pastures; consequently shortages of feed were severe in the late dry season, primarily from February to April (Konlan *et al.*, 2016). Availability of natural pastures in the Northern and Upper West regions was approximately 20%, but only 10% in the Upper East Region (Konlan *et al.*, 2016). The annual herbage output in the northern Savanna ecological zone is calculated at 2.2 tonnes dry matter (DM) per hectare (Oppong-Anane, 2001). The amount of available vegetation determines the pasture's possible carrying capacity. The condition of the vegetation has an effect on the productive ability of small ruminants on pasture. The quality and quantity of pastures is influenced by seasons and climates, accidental or intentional burning, and the content of leguminous forages. Seasonally, however, the nutritional value of natural pasture in the northern Savanna zone changes considerably (Karbo and Agyare, 2002). In regions where the availability of herbage is sufficient throughout the dry season because of good vegetative growth during the rainy season in the interior Savanna zone, it is frequently lacking in protein, vitamins, and minerals due to the seven-month dry season (Smith, 2002). Also variable, phosphorus (P) content ranges from 0.2 to 0.1% DM (Fleischer *et al.*, 1996). Grass attains its maximum quality during stem elongation, and its quality declines after heading. Changes in species composition, such

as hosting a legume such as *Stylosanthes guianensis* in a natural pasture to increase the protein yield per hectare, and improvements in management, such as grazing control or the application of fertilisers, can increase the yield and quality of natural pastures (Ibrahim, 1998).

2.4.2 Cultivated and Improved Pasture

Grasslands are intensively used in sub-Saharan Africa, though they are only good at sustaining the appropriate level and duration of sheep and goat output. The purpose of cultivating pastures or fodder crops is to increasing the production of forage per unit area and the nutritive value of the grasses (Ibrahim, 1998). Improved or farmed forages are more productive and have a higher nutritional value than their wild counterparts. Numerous fodder plants have been tested for their adaptability within distinct ecological zones, and certain helpful species have been chosen for each zone. Introducing enhanced forages and pastures to rural farming communities has been limited by land scarcity and crop-dominated agriculture, and the adoption of this technology by smallholder mixed farmers has been generally delayed (Mekoya *et al.*, 2008). The yield of improved pasture and forage is between 6-8 tonnes and 3-5 tonnes DM per hectare, while that of tree legumes ranges between 10-12 tonnes DM per hectare (Alemayehu, 2003). Despite the significance of enhanced pasture and forage plants, their introduction has been limited due to the lack of available land and the prevalence of crop farming (Alemayehu, 2002). As a result of the kind of soil and temperature, pasture development in highland regions is more challenging than in humid, warmer, and lower regions. As a result of seed dissemination by wind, precipitation, and ruminant droppings, however, leguminous plants now occupy a broader region. According to studies, pigeon pea (*Cajanus cajan*) and Leucaena (*Leucaena leucocephala*) are partially produced as fodder crops (Karbo and Agyare, 2002; Konlan *et al.*, 2016). This calls for additional research to encourage livestock farmers to develop more fodder plants. Pigeon pea (*Cajanus cajan*) and *Leucaena leuccocephala* are the predominant fodder plants grown on a few farms in Northern Ghana (Konlan *et al.*, 2016). Also *Gliricidia sepium* and *Ficus gnaphalocarpa* are offered. Estimations indicate that the average area devoted to leucaena agriculture is substantially greater in Upper East region of Ghana (Konlan *et*

al., 2016). In addition, *Andropogon gayanus* has been utilised to enhance natural pastures in the Sudan Savannah region (Oppong-Anane, 2001). According to Konlan *et al.* (2016), the average area of pigeon pea cultivation in Northern Ghana is comparable across regions and serves mostly as a barrier between two farmers' fields or between different crops.

2.4.3 Conserved Forage

The production of fodder and forage is an activity that is only viewed being done as on the fringe and it is coupled with different sources of agricultural activities aimed at boosting food production (Nitis, 1999). Consideration must be given to the conservation of fodder generated during the wet season, which can be given to animals maintained throughout the dry season. This may be the only technique capable of ensuring that the severe nutrient needs of animals kept under the varied systems of animal productions especially those in the smallholder dairy production in semiarid tropical climates, are met (Dube, 1995). Supplemental forage diets should be supplied to animals throughout the shortage periods that are brought on by restricted development of pastures or when the conditions of the pastures are not good. The preserved forms of forage are hay, haylage, and silage. Albeit the various approaches that have been acknowledged as effective and best ways to preserve and store forages, it is essential to keep in mind the following: the nutritive values of forages reduce during conservation and can rarely match that of fresh forages due to the conservation methods employed which renders the loss and waste of important nutrients such as protein, sugars, and fat (Romero *et al.*, 2015). It is difficult to produce hay from forages in Southern Africa due to the conservation of forages at its optimal return, content of protein, and digestibility. Nevertheless, harvesting often occurs during the foggiest portion of the wet season (Topps and Oliver, 1993). Forage sorghum, *pennisetums* and elephant grasses need too long a time to dry due to the their bulkiness but forages with thin stems such as are able to dry given a day or two (Mhere et *al.*, 1999). If harvesting is postponed until later in the season, there is a significant decrease in quality and an increase in leaf cracking and loss, particularly in legume forages (Maclaurin and Wood, 1987).

2.4.3.1 Hay

Hay is defined as feed preserved in aerobically dry or low-humidity settings. Forages when cut fresh often have moisture content between 75 and 85% (Collins and Coblentz 2013). Thus, the objective of producing hay is to rapidly eliminate moistness to obtain moisture level of at least 20% or less or to achieve 80% DM concentration or more. The act of lowering wetness is known as curing and is often achieved using solar radiation (field curing) or artificial drying in a barn using either hot or cooled air. A dry matter concentration of more than 80% (preferred: less than or equal to 15% moisture levels) restricts respiration in the plants and permits the nearly total conservation of plant nutrients over longer periods. There are three types of factors that influence the moisture loss process in hay production: (1) management related, (2) forage related, and (3) weather related (Rotz, 1995; Collins and Owens, 2003).

2.4.3.2 Silage

In the majority of tropical nations, silage utilisation is limited. Nonetheless, there is sufficient evidence that grasses like as elephant grass (*Pennisetum purpureum*) can be used to produce silage of acceptable quality (Pond *et al.*, 2004). Tropical grasses with about 3% (fresh weight) of soluble carbohydrates provide enough lactic acid generation and to prevent secondary fermentations that result in excessive butyric acid production (Wilkinson, 1983). In Ghana, the native forages used for silage have lower CP and poor digestibility (Rymer *et al.*, 2005) but if harvested at right stage can be very beneficial in producing silage (Addah *et al.*, 2014). High-moisture forages are used to generate silage, which are preserved by acids produced through fermentation in an anaerobic environment. Throughout the process of ensiling, the simple carbohydrates in the forage are digested by non-oxygen using bacteria into organic acids and this increases the acidity of the silage to proportions that inhibit the reproduction of unwanted microbes. This is because yeasts known for spoiling silage remains inactive so long as there is oxygen and the silage will be maintained if oxygen remain absent in the bag. The addition of oxygen to a silo encourages yeast growth and may cause spoiling (Adesogan and Newman, 2004). An ideal crop for ensiling should have more simple

carbohydrates to undergo fermentation, should not easily undergo change in pH, and possess a DM concentration of about 20% (McDonald *et al.*, 1991). Silage is known to be a valuable and economical feed for cattle operations (Adesogan and Newman, 2004).

Water soluble carbohydrates (WSC) are important for silage production and for the insufficiency of it during harvesting it do not make legumes and grasses from the tropics an ideal for silage preparation (Adesogan and Newman, 2004). When the simple carbohydrates in the silage are left to be acted upon by oxygen-utilizing bacteria it reduces the DM which in turn affects the quantity of nutrients in the silage that are digestible and beneficial to the animal (Addah *et al.*, 2014). More heat is produced coupled with a higher pH when the simple carbohydrates and lactic acid are digested by oxygen-utilizing yeasts producing CO_2, ethanol and water (McDonald, 1991). As a result, their buffering capacity is enhanced, leaving their proteins susceptible to proteolysis (Fianu *et al.*, 2001). Corn and sorghum have higher moisture content and this limits their ability to be used for high quality hay production (Adesogan and Newman, 2004). The harvesting of these grasses as silage could reduce harvesting losses and provide a more timely harvest, hence improving fodder quality (Adesogan and Newman, 2004). Maize is the predominant crop used for ensiling in the southern Africa because it is produced in greater quantity, has more energy and fermentation characteristics (Maasdorp and Titterton, 1997). However, it is limited with a lower CP content which is about 8% (Topps and Oliver, 1993). In the semiarid parts of the tropics, maize is highly susceptible to moisture stress; therefore, its suitability as a silage crop is doubtful. In general, yields are low and energy values are significantly lower than in regions with higher precipitation. Mhere *et al.* (1999) explored the possibility of using other drought resistant and high yielding crops for silage making and found that forage sorghum, forage pernisetums and grain sorghum were all ideal for ensiling but their quality in nutrients such as the lower protein of the forage sorghum (7% DM) and pennisetum (9.5% DM) are a limiting factor in their usage for silage production (Mhere *et al.*, 1999). The addition of crops rich in protein to the cereal crops is one of the ways to increasing the CP of silage. This can be accomplished either by intercropping cereals with legumes or by cultivating cereals and legumes separately and combining them during ensiling. One way of having

lower CP in silage is losses due to physical and chemical means. Kohler *et al.* (2013) reported an average loss of 8% DM in corn silage. Fermentation, wilting, reheating and respiration are regarded as the primary causes of silage DM losses (Spiekers *et al.*, 2009). Some of the methods to improving the use of forages include chopping, grinding, and steaming, thus reducing the particle size (Addah *et al.*, 2014) but one must be careful to not reduce particle size to finer as it may impede rumen degradability (Collins and Coblentz, 2013). Albeit the low level of education of Ghanaian farmers on handling agro-chemicals (Addah *et al.*, 2021), hydrolytic agents such as NaOH, NH_3 and urea are noted for solubilising hemicellulose and lignin (Collins and Coblentz, 2013).

2.4.5 Crop Residues

The five regions up-north Ghana are suitable for livestock production (Karbo and Bruce, 2000) but the long spells of dry seasons that results in feed shortage poses major threat to livestock farmers in these regions (Oppong-Anane, 2013). Albeit the high cost of feedstuff, famers resort to using crop residues which decline in quality (Larbi *et al.*, 2002). There are numerous crop wastes that can be utilised for sheep and goat grazing. In crop–livestock systems, small ruminants are better utilizers of these agricultural by products. Due to the construction of infrastructure, the dwindling accessibility of natural pasture, particularly in metropolitan areas, has increased the pressure on urban farmers to find alternative feed sources for their livestock and has contributed to the rising crop residue demand . Similar tendency were observed in the Ethiopian highlands, where almost 70% of crop residues are used as animal feed (Zinash and Seyoum, 1991). Annually, 8 million tonnes DM of cereal stalks and 3.5 million tonnes DM of residues from legume, root, and tuber crops are theoretically accessible as animal feed in Ghana, according to estimates (Oppong-Anane, 2010). It is estimated that about 5 million tonnes DM of crop residue are created annually in northern Ghana (MoFA, 2011; Karbo and Agyare, 2002). Cassava, a tuber crop that is processed in about five tonnes daily in Techiman and Wenchi (Seidu *et al.* 2012) is produced across Ghana and the peels serves as a rich source for readily fermentable carbohydrates that has the potential of supplementing

the energy of tropical forages (Onua and Okeke, 1999). If not properly tested the high hydrogen cyanide content (Cardoso *et al.*, 2005) coupled with its perishability (Tewe, 1992) and low CP content (Konlan *et al.*, 2016) can reduce feed intake and performance by interfering in the utilization of essential amino acids (Enneking and Wink, 2000).

Due to collecting difficulties and alternate applications, such as domestic fuel (e.g., sorghum and millet stalks) and thatch roofing, the fraction of these crop leftovers that are consumed by animals is extremely low (MoFA, 1998). It has been determined that agricultural residues and agro by-products in various preserved forms play an essential role in the nutrition of ruminant livestock and guarantee year-round feed availability. It is also the most affordable method of decreasing the rising expense of feeding ruminants in tropical regions (Attoh-Kotoku, 2003). Tolera *et al.* (2012) reported that agricultural leftovers provide around 50% of Ethiopia's overall feed supply. Few farmers have given crop residue conversion significant thought, but a more concerted effort is required for farmers to adopt this technique in order to solve the dry season feed problem (Konlan *et al.*, 2016). There are numerous crop wastes that can be utilised for sheep and goat grazing. In crop–livestock systems, sheep and goats make better use of these agricultural by-products. Farmers without animals may use crop wastes to increase soil fertility or sell them. In Northern Ghana, crop remains are mostly fed to livestock at the end of the year and throughout the first two months of the following year (Konlan *et al.*, 2016). The gathering of leguminous crop residues for supplemental feeding during the dry season is a suitable technique that stresses the integration of crops and livestock (Jayasuriya 2002; Smith, 2002). According to a study conducted by Ansah and Issaka (2018) in the Kumbungu District of Ghana, the most often farmed food crop is maize, which produces crop residue and crop by-products. The majority of non-legume crop wastes were left on the field as mulch and for animals to graze. The approach could result in a significant amount of wasted feed due to the residue's susceptibility to contamination with urine and faeces, but it would also enrich the soil with organic carbon, phosphate, and nitrogen. An earlier study revealed that 25 to 50% of crop residue generated in the humid forest and savanna zones of West Africa might be used as animal feed without significantly impacting crop production (Larbi *et al.*, 2002). Included

among the common crop wastes were groundnut haulm, cowpea haulm, and soybean residue. These are supplemental food sources. The commonest cereal stovers are fed on-site as opposed to being harvested. Straw which is a by-product of cereal crops is fed to ruminants as roughage. In Africa, the subsequent grains are used as straw: millet, maize, sorghum, wheat, teff, rice and barley. On a smaller scale, residues from sugarcane, banana, cocoa, cassava, legume crops and cotton are also utilised. Cereal crop residue has minimal nutritional significance. The energy value of cereal residues lies between 5.5 and 9.6 MJ ME/kg DM (Zinash and Seyoum, 1991). Energy levels vary based on cereal variety and residue conservation practices following harvest. High levels of lignin in straw reduce its digestion. Straws of the majority of the cereal crops are deficient in minerals especially phosphorous whiles virtually all do not have significant protein values. Straw's high nutritional value and coarse physical structure hinder the activity of microorganisms in the rumen and contribute to the slow rate of digestion. All of these variables contribute to poor voluntary intake (Ibrahim, 1998). According to a study conducted by Konlan *et al.* (2016), a greater proportion of cereal crop wastes remain on farmers' fields. This available fraction of feed is then used to raise cattle as they are allowed to grase on. Crop by-products are considered to have low metabolizable energy of about 7.5 MJ/kg DM, low crude protein ranging from 20–60 g CP/kg DM, moderately high digestibility ranging from 30–45% OMD but high value of fibre (>700 g of cell wall material/kg DM). Such materials as feed sources limits the daily feed intake of sheep and goats to not more than 20 g DM /kg live weight (Owen and Jayasuriya, 1989) and greatly decreases animals' aptitude of meeting their nutritional needs, resulting in weight loss (Smith, 2002). The paucity of fermentable carbohydrates in the majority of excesses of crops is evidenced by the comparatively low digestibility of organic matter (Jayasuriya, 2002). Reduced protein concentration, high fibre content, and low digestibility of nutrients have been reported in fodder, especially during the dry season (Castillo-Caamal *et al.*, 2003). Some farmers have limited storage space for crop residues that are low in fibre and high in protein, such as the tops and haulms of groundnut, soya beans and cowpea. More than 20% of the farmers had easy access to the crop remnants of legumes, while the majority of farmers do not save crop residues after harvesting the

main crop (Awuma, 2012). Processing techniques, such as chemical treatment, have the ability to increase the nutritional value of crop waste for animal feed (Annor *et al.*, 2007). The benefits of alkaline treatment with urea of high-fiber crop wastes have been extensively explored and are well-established (Owen and Jayasuriya 1989). However, adoption of this technology is minimal in Northern Ghana; hence, farmers cannot fully use the potential of these crop leftovers (Konlan *et al.*, 2016). Adams and Ohene-Yankyera (2014) suggested that improved extension education on agricultural residue usage is necessary. In addition, few farmers in northern Ghana have addressed crop residue conservation to alleviate the chronic feed shortage during the dry season (Konlan *et al.*, 2016).

2.4.5.1 Sorghum Stover

Stover consists of the field remnants of major grains like corn and sorghum. In many regions of the world, these wastes constitute the majority of the available fodder. In small-scale production systems, stover is typically handled and dried in its long, un-chopped state prior to storage, frequently by stooking in or on the field's perimeter (Awuma, 2012). Large-scale systems also bale and ensile the material, with or without urea treatment. Sorghum is a cereal crop that thrives primarily in arid regions of Asia, Africa, and parts of America and Australia. Immediate following crop harvest, the stover is also harvested, preferably by cutting it as green as possible and then drying it for later use as corn feed (Konlan *et al.*, 2016). In some regions, grazing is practised, but it is not preferred due to the potential of waste caused by trampling and manure, as is always the case while grazing Stover.

2.4.5.2 Corn Stover

Corn stover comprises of the entire plant remains remaining in the field after harvesting corn. It consists of the stems, leaves, husks, and cobs. Corn stover is widely dispersed, abundant, and inexpensive, with significant development and utilisation potential (Konlan *et al.*, 2016). After the corn harvest, many small farms either graze the corn stover on the field or harvest or dry it in the field or on the homestead as a cost-effective resource for winter cattle fodder (Oppong-Anane, 2013). In bigger farms, it is either baled after drying or ensiled. Maize stover contains more nutrients than most straws. Sweet-corn cobs, which are taken while the plant is still green, provide a

substantial amount of high-quality roughage as a by-product. This stover is left to grow a few days after the cobs have been harvested. The stover produced by these plants is superior to that of a fully grown crop (Zinash and Seyoum, 1991). The findings indicate that the nutritious content of various maize plant sections varies considerably. The husks are more digestible and nutrient-dense than the leaves, but they only account for around 12% of the field's residue (Savadogo *et al.,* 2000). The leaves may sustain an adult cow that is not lactating.

2.4.5.3 Pulse and Leguminous Crop Residues

The leaves and stems of certain regularly grown pulses, such as soybean, green and black grammes, and pigeon pea, are also effective meals (Ibrahim, 1998). Green pea haulm vines are useful by-product that is best preserved as fodder. Pea straw and soybean hulls picked by hand are important sources of roughage. Many of these have a higher feeding value than cereal straws, but are significantly harder to retrieve. In humid areas, leaves tend to discolour or fall off during or before harvest, whereas they shatter in dry circumstances. When the final harvest drying occurs on the homestead, it is easier to recover leaves and stems from threshing. However, harvesting success is highly dependent on favourable weather, and leaves are frequently infected or wilted at harvest time (Castillo-Caamal *et al.,* 2003).

Soybean Pasture

The soybean is a leguminous plant with a maximum height of 1 metre. Soybean is predominately an oil seed containing approximately 20% oil (Dijkstra *et al.*, 2005). The principal soybean wastes consist of oil harvest by-products such as cakes and grain harvest by-products such as stems, leaves, and hulls, which can be grazed, ensiled, or dried to form hay. The foliage is highly palatable, has a high nutritional value, and is easily digestible. To feed dry cows and heifers, the stubble can be grazed or chopped. Soybean straw, which is the by-product of threshing the beans, can be fed to cattle as roughage.

2.4.5.4 Restrictive Factors Related to Crop Residues

Despite the widespread use of crop wastes in animal feeding, the absolute conversion of these residues into marketable animal products is limited by a number of issues (Addah and Ayatunde, 2021). For example, cereal crop residues are weak in protein; however cell wall as neutral-detergent

fibre amounts for up to 80 % of the DM and is a significant source of energy for ruminants. However, the capacity of rumen microorganisms to digest cell wall polysaccharides (cellulose and hemicellulose) is restricted by the presence of phenolic and other aromatic chemicals, commonly known as lignin (Ansah *et al*., 2018). Sorghums are notorious for prussic acid poisoning when late rains and hot heat drive regrowth of plant stubble. Trypsin inhibitors and non-starch polysaccharides are anti-nutritional substances produced by soybean. Feeding tubers, maize cobs, and other large pieces of food has caused ruminants to choke due to oesophageal obstruction, which occurs when animals only partially swallow solid bits of food such as potato tubers (Oppong-Anane, 2010.

2.4.5.5 Handling of Crop Residues

In light of the majority of the aforementioned issues, numerous innovative strategies have been created to improve the exploitation of crop wastes and agricultural by-products (Dijkstra *et al.*, 2005). These strategies collectively increase the availability of nutrients to animals by improving their digestibility (Charray *et al.*, 1992). The primary purpose of physical treatment is to minimise residual size and increase the surface area exposed to digestive enzymes in the animal body, while the other three methods aim to improve the digestion process in the animal body. Physical, chemical, physicochemical, and biological treatment methods are depicted in the diagram that follows.

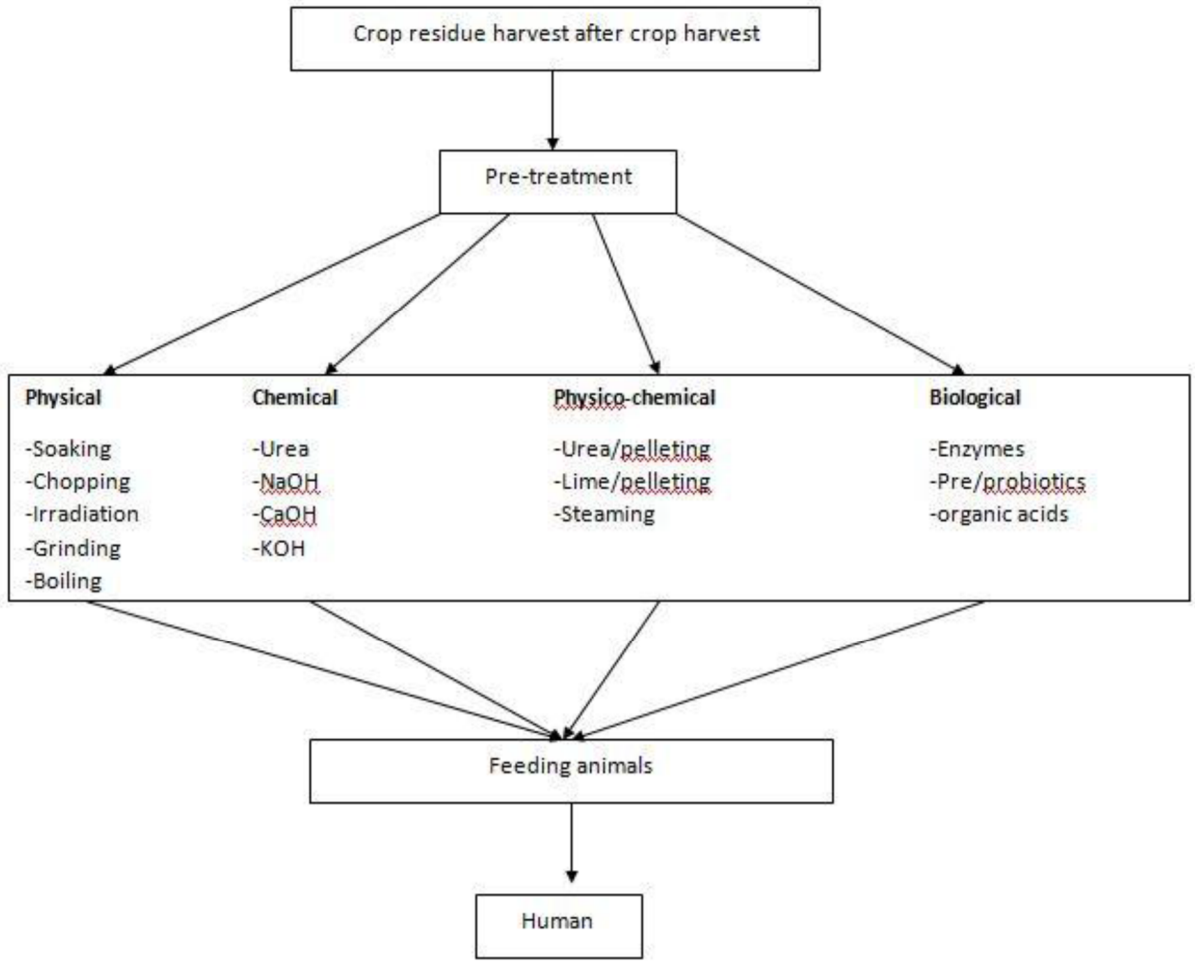

Figure 1: Methods of Crop Residue Treatment

2.4.5.6 Factors Affecting Quality and Quantity of Crop Residues

The quantity of accessible crop residues is influenced by all factors that typically influence crop yield. The selective grazing behaviour of animals is another element that leads to the consumption of just particular portions or fractions of crop waste (Ansah *et al.*, 2018). During grazing, trampling contributes to the loss of edible material. Collecting and processing the residues (e.g., milling) increases the amount of residue consumed by the animal, but is related with a decrease in animal performance since the animals are compelled to consume lower-quality material (Cottle, 2013). According to Tesfaye and Musimba (2006) several variables impact the quality of residues, including: 1. nutrient leaching and rain damage can drastically reduce the nutritional value of crop residues. The method of harvesting has been demonstrated to considerably alter the quality of residues. 3 The cultivar has an important role. 4 Low crop harvest yields higher quality residues because nutrients were not translocated from the stem and leaves to the grain. In addition to these

considerations, the technological measures to boost intake and quality include the addition of additives, such as spraying residues with molasses or feeding a rumen-stimulating lick such as urea molasses brick.

2.4.6 Agro-Industrial By-Products (AIBP)

There are also by products of the agro-industries (AIBPs) that are produced by the agricultural processing enterprises and households and can be used as feed for livestock. In Ghana, more than an estimated 80 % of rural farmers rely on this agro-industrial by products in feeding their ruminants (Teye *et al.*, 2011). Nevertheless, these AIBPs have not received a great deal of study attention despite their tremendous potential for boosting livestock production, particularly in the business to fatten sheep and goats for sale. Rural farmers in the northern parts of Ghana commonly nourish their animals with millet, maize and sorghum brans, leftover grains from the brewery and soybean cake as well as cotton seed cake (Ansah *et al.*, 2012; Oppong-Anane, 2013). Also, by products of the agro industrial processing such wastes of corn processing, rice, brewer's spent grain, and the brans of maize and sorghum and rice are often less fibrous and have a comparatively large quantity of crude protein (Jayasuriya, 2002). The majority of smallholder farmers do not have access to this set of by-products of the agricultural processing industry during the critical periods of feed scarcity (Konlan *et al.*, 2016).

However, when accessible, cottonseed cake promises to be a great source of nutrients for small ruminants. Cottonseed cake has high levels of protein (25 - 40%), cellulose (25 - 30%) and fat (10 - 23%). After a livestock has become accustomed to it its consumption increases. The finest results are produced by combining salt and molasses with the cake. This was tested with West African Dwarf ewes, and a mixture of cottonseed cake and molasses in a 50:50 ratio produced positive results (Charray *et al.*, 1992). Due to its high protein content, the amount of cottonseed cake can be decreased without negative consequences. Unfortunately, the kernel and seed coat of whole cotton seed and other cotton by-products contain gossypol (FAO, 2014). This free gossypol in the seed (0.03 - 0.3%) is toxic especially to monogastrics and pre-ruminants (Aydin *et al.*, 2008). When the gossypol concentration in cotton seed high (50 and 100 ppm), it becomes toxic to poultry and

swine, respectively. Church (1991) reported that gossypol detoxification by rumen microorganism renders raw cotton seed harmless for adult sheep, goats, and cattle.

Most oilseed cakes (oil meals) are by-products of processing groundnut, sunflower, and soybean oil crops. These cakes are high in protein and fatty acids, similar to cottonseed cakes. Oil meals can be fed solely or in a mixture with molasses for good results. Osuji *et al.* (1993) found that Menz sheep in the Ethiopian highlands utilised sunflower cake successfully in terms of rumen microbial N synthesis, N retention, and growth. The addition of tiny quantities of energy, such as broken maize grain, enhanced microbial nitrogen synthesis, nitrogen retention, and live weight gain. Cost and availability will impact the likelihood that farmers will embrace the cakes.

When beer is brewed, the discarded grains and yeast are the leftovers. These are readily accepted as feed by sheep and goats. The increasing number of breweries in sub-Saharan Africa is the source of these by-products (including home-made installations). Home installations produce by-products that are higher in energy and protein than industrial by-products (Ayantunde *et al.*, 2008).

Sugarcane factories produce dry sludge, molasses, and bagasse as by-products. Sheep are able to consume all three by-products. The majority of farmers utilise molasses, a thick, dark brown liquid containing 50 – 65% sugar and little protein and water (Romney *et al.*, 1993). Thus, it is a high-calorie feed. When added to coarse and less frequently absorbed cottonseed cakes, it boosts their consumption. It can be added to the forage during the silage-making process. Therefore, molasses is a good feed when supplemented with protein and minerals. To raise the profitability of small ruminant fattening operations, research and extension education are required to enhance farmers' understanding of the optimum use of these feedstuffs to boost ruminant productivity.

Table 2.1: The nutritional composition of commonly fed crop leftovers and agro-industrial by-products in smallholder agriculture

Type of Feed	DM%	OM%	Ash%	CP%	NDF%	ME (MJ/Kg)
Crop residues						
Cowpea haulm	94.0[3]	80.0[6]	20.0[6]	18[1]	45.6[6]	7.7[6]
Groundnut haulm	94.6[2]	94.0[2]	0.6[2]	18.2[1]	42.2[2]	10[2]

Sorghum stover	93.1[12]	98.97[6]	0.13[6]	4.4[5]	73.6[6]	6.9[6]
Maize stover	90.4[8]	84.3[8]	7.1[10]	5.3[5]	71.9[8]	8.8[11]
Millet stover	-	79.3[15]	20.7[15]	3.6[15]	59.7[15]	6.07[15]
Rice stover	-	80.2[15]	19.8[15]	4.8[15]	48.9[15]	6.2[15]
Agro-industrial by-products						
Rice bran	91.8[4]	74.9[4]	16.9[4]	6.7[4]	-	11.3[4]
Wheat bran	92.7[4]	87.5[4]	5.2[4]	15.5[4]	36.7[6]	16.5[4]
Pito mash	92.9[4]	88.5[4]	4.4[4]	28.8[4]	-	17.8[4]
Pigeon pea waste	90.0[3]	-	-	7.5[3]	-	-
Rice straw	93.7[3]	90.6[1]	10.4[1]	3.4[3]	19.8[6]	7.3[1]
Yam peels	95.3[7]	90.2[7]	9.8[7]	4.9[7]	-	10.2[7]
Cassava peels	86.3[3]	-	5.7[9]	4.6[3]	19.6[9]	17.7[9]
Maize bran	90.5[13]	95.7[13]	4.3[13]	11.6[13]	31.9[14]	10.5[13]
Cotton seed cake	91[13]	94.4[13]	5.6[13]	41.1[13]	-	7.8[13]
Soyabean meal	90[13]	95.9[13]	4.1[13]	51.1[13]	-	9.9[13]

DM: Dry matter, OM: Organic matter, CP: Crude Protein, NDF: Neutral detergent fibre, ME: Metabolisable energy

[1]Onwuka *et al.*, (1997), [2]Romney *et al.*, (1993), [3]Karbo *et al.*, (1997), [4]Abarike *et al.*, (2012), [5]Mosimanyana and Kiflewahid (2006), [6]Tunde and Ayantunde, (2016), [7]Omole *et al.*, (2013), [8]Faftine and Zanetti (2010), [9]Oppong-Anane (2013), [10] Laureano-Pérez *et al.*, (2005), [11]Tesfaye and Musimba (2006), [12]Savadogo *et al.*, (2000), [13]Donkoh and Attoh-Kotoku (2009), [14]Mlay *et al* (2005), [15]Amole and Ayantunde (2016b).

2.6 Nutrient Intake and Digestion in Farm Ruminants

Supplementation of concentrates increases production and rumen microbial nitrogen efficiency and increases digestibility of DM to about 15% in ruminants (Wanapat *et al.*, 2007). Agreeing to Fernandez-Rivera *et al.* (1995), the digestibility of DM for ruminants fed diverse forage species and crop wastes in confinement was 58%, 55%, and 58%, respectively (Huang *et al.*, 2008). Studies of

in vivo digestibility involving total faeces collection from sheep and goats foraging on natural pastures are relatively problematic because of the preciseness of collection of the faeces; however errors are decreased when performed with care (Cottle, 2013). However, it is simple to collect faeces from sheep and goats that are fed at will in detention for evaluating fodder use and nutritional quality (Coleman, 2005). The daily mean faecal production of goats, sheep and cattle has been calculated to be 2,385, 197 and 345 g DM/d for cattle, goats and sheep fed ad libitum a variety of forage species under intensive management (Fernandez-Rivera *et al.*, 1995). The daily faeces production of Djallonkè × Sahelian and Djallonkè hybridised sheep reared in a semi intensive management estimated by the International Trypanotolerance Centre (ITC, 2014) ranged from 219 to 475 g DM/d and 358 to 616 g DM/d correspondingly. According to the ITC (2014), the faeces of sheep contain 2.0±0.4% nitrogen and 0.9±0.2% phosphorus. Ayantunde *et al.* (2008) also observed a 170% increment in nitrogen intake, 24% increase in faecal nitrogen, and sheep nourished with a basal diet of wild hay and millet bran and groundnut haulm supplementation had 260% increase in the production of nitrogen in the urine under a restricted feeding regime. Consequently, nitrogen consumption increase with bran of cereals and residues of leguminous crop additions. Also, it has been found that the faeces of sheep fed browses contain more N than those given agricultural wastes (Somda *et al.*, 1993). Tannins, lignin and similar phenolic chemicals influence nitrogen up take and shift urine nitrogen excretion to nitrogen in faeces excretion linked with feed particles that are undigested in the stools (Reed *et al.*, 1990). The concentration of nitrogen in faeces of goats, sheep and cattle is less during the dry season than the wet and humid season, and there are annual variations as a result of variations in the yearly patterns of rainfall (Somda *et al.*, 1993; Schlecht *et al.*, 1995). Somda *et al.* (1993) postulated that the transfer of urine nitrogen to faecal nitrogen and from faecal soluble nitrogen to insoluble nitrogen makes the nitrogen more accessible for reutilizing in crop-livestock schemes when sol fertility is replaced by faecal matter. According to Mariana (2008), African dairy cattle hold marginal dietary nitrogen and have a faeces nitrogen concentration of approximately 83%. Reynolds and De Leeuw (1995) have determined the nitrogen production levels of tropical livestock and concluded that goats, sheep and cattle in small holder systems retain

not more than 20% of absorbed nitrogen for productive uses. This indicates that livestock in the tropical system have a restricted capacity to utilise dietary nitrogen or that it is in an indigestible form. It has been found that the N content of dried and fresh faeces samples is comparable (Schlecht *et al.*, 1995), since nitrogen losses as a result of drying are around 1.9 g N/kg DM of faecal matter and its statistically insignificant. Therefore, the nitrogen content of dried faeces does not require compensation for losses associated with drying samples (Schlecht *et al.*, 1995).

2.7 Near Infrared Reflectance Spectroscopy

Near infrared reflectance spectroscopy (NIRS) is a new non-destructive technique for analysing the physiochemical properties of samples using the infrared portion of the electromagnetic spectrum (750 nm to 2500 nm). This infrared spectroscopic system is a multiple based analytical technique which permits the precise simultaneous prediction of multiple parameters at a given time (Givens *et al.*, 1997). The approach is non-destructive, fast, cost-effective and accurate in comparison to conventional analytical methods (Yakubu *et al.*, 2020). Due to its multiple advantages, the NIRS technology has gained widespread acceptance in recent years as the primary method for online feed analysis (Huang *et al.*, 2008). NIRS machines of the past were sophisticated, prohibitively expensive, and intended for laboratory usage exclusively. They offered extremely exact analytical data, but only expert staffs were able to operate them. The evolution of this technique has resulted in a significant decline in the weight and bulkiness of NIRS devices, resulting in the creation of field-portable instruments. In addition, their performance has improved as a result of the acceleration in spectra acquisition, enhanced transmission, processing of signal, and software and hardware advancements. Measurement for NIRS is taken in transmission, transmittance or reflectance mode. The transmittance mode provides data on the full sample bulk traversed by the light, whereas the latter just provides information from the sample's surface. The combination of the two different modes gives the transflectance mode and is particularly suited to liquids such as milk (Tsenkova *et al.*, 2001).

Calibration is essential for effective usage of the NIRS method (Corson *et al.*, 1997). Obtaining robust and accurate calibration equations is difficult since it takes significant quantity of samples in building one. The goal of the calibration is to encompass all changes of physical and/or chemical attributes. To adapt equipment to new measurement and sample conditions, constant calibration is necessary. But when calibration equations are established, near infrared spectroscopic machine provides a quick and cost effective assay that is able to screen large samples (Restaino *et al.*, 2008) and is straightforward to apply. Additionally, daily device diagnostics are essential to safeguard accurate and consistent results (Stuth *et al.*, 2003). The inability of the NIRS system to absorb mineral the near infrared region, this approach has a low sensitivity for predicting mineral concentration. However, the combination of UV light, electronic nose technique and X-ray fluorescence which are also detection techniques offer a possible solution to this issue (Huang *et al.*, 2008). In the majority of research the quality of near infrared reflectance spectroscopy calibration is evaluated in terms of precision and linearity. The coefficient of determination (R^2), the ratio of performance to deviation demonstrate linearity (RPD) and the calibration standard error (SEC) are all linear statistics that are used to assess NIRS calibration strength (Restaino *et al.*, 2008).

2.7.1 Application of NIRS Technology in Forage Research

To effectively use near infrared reflectance spectroscopy Brunno-Soares *et al.* (1998) proposed four requirements which must be obeyed and rigorously adhered to by researchers and this has over the years become cardinal rules. These principles are;

1. Ensure that the samples used for calibration characterizes the samples in the population being analysed adequately;
2. Perform precise chemical analyses on the samples used for calibration;
3. Selection of the appropriate data processing procedures to glean the information from the near infrared spectra; and
4. Choose the appropriate wavelengths.

2.7.1.1 Forage Plant Breeding

Near infrared reflectance spectroscopy was first used in genetics and breeding to assess the quality of fodder in a report by Marum *et al.* (1979). The report tried to analyse plant cell wall heritability and quality in reed canary grass (*Phalaris arundinacea L.*). During the creation of calibration equations for scanning monochromator equipment, all the 72 samples in the population to be assessed were used. Correlations between laboratory and NIRS values were at or above r = 0.90 for two fractions of fibre and CP, and lignin, *in vitro* digestibility (IVDMD) and silica were at or above r = 0.73. In general, the SEC values of calibration for all components were smaller than 1% except IVDMD which had 1.7%. These errors were smaller than those often stated in conventional laboratory assays. When Marum *et al.* (1979) employed calibration models grounded on a year old samples to assess quality of forages using samples from the preceding year, the SEP were more than twice the following year's. This conclusion supported the necessity of developing unique calibration formulae for each year. Significant variations in the spectral characteristics of reed canary from year to year were linked to variations in the colour of the samples of those years and these variations were related to differences in the processes of drying the samples between those years. The results demonstrated that forage samples intended for NIRS analysis must be processed, stored, and handled uniformly. Each time a particular process of preparing samples is left out of sample set for calibration, the calibration equation must either be modified to account for the alteration or a new model must be developed to characterize the new population. In addition, if plants leading to an unidentified set of samples are grown in an environment that is not involved in the calibration set, the models will typically be improved by adjusting to the new environment.

2.7.1.2 Ruminant Nutrition

Pasture quality was reportedly predicted by near infrared reflectance spectroscopy *in vivo* (Brunno-Soares *et al.*, 1998). It is argued that though there might be some disparities with the *in vivo* response of animals, however, the obtained R^2 values were higher; 0.72, 0.64 and 0.78 respectively for digestible energy, intake and digestibility of legume forages and grass by sheep. Shenk *et al.* (1991) discovered that near infrared reflectance spectroscopic machine that was fixed with a

scanning monochromator was able to accurately predict the digestibility and energy intakes of temperate hays that were dried on the field as it did for *in vitro* and fiber digestibility. In addition, Brunno-Soares *et al.* (1998) had established that a scanning monochromator NIRS instrument had the ability to predict ruminant animal responses (digestible energy, intake, and digestible energy intake) to both sole and mixed forage-based diets with the same accuracy as wet-chemistry analyses. Burdick *et al.* (1987) utilized a scanning filter apparatus for the assessment of *in vivo* digestibility in Bermuda grass hays using near infrared reflectance spectroscopy, after selecting the primary and secondary wavelengths with a scanning monochromator. 1.78 to 2.50 standard errors were smaller than those obtained in animal feeding studies (3.1). With fixed filter equipment, NIRS prediction of fodder quality *in vivo* has been less successful. By using fixed filter monochromator, Winch and Major (1987) discovered that the SEC values in assessing the *in vivo* digestibility of leguminous crops and grasses were not acceptable. By analysing oesophageal fistula samples, Lindberg (1981) employed near infrared reflectance spectroscopy to assess fodder intake of animals grazing on arid or semiarid rangeland. They discovered that near infrared reflectance spectroscopic studies of browse species and forb were as accurate at predicting intake as traditional chemical analyses. To reliably determine the *in vivo* values for unknown substances, data on animal responses for the near infrared reflectance spectroscopy model calibration ought to be gathered with the same precision as chemical constituent data. Using reliable base data, scanning monochromator equipment may estimate the intake and digestibility of forages with the same efficiency as *in vitro* techniques or even better, and in a significantly shorter amount of time. Due to the fact that near infrared reflectance spectroscopy permits daily monitoring of the quality of pasture in studies of animal nutrition, feeds can be rapidly modified in large scale feeding trials to correct for variations in the quality forages and to maintain the initial nutrient levels specified for certain treatments. Feed composition as well as faecal constituents can be examined using near infrared reflectance spectroscopy (Williams and Norris, 2001).

2.7.2 Future NIRS Applications

2.7.2.1 Feed Ingredients

The analysis of feed ingredients will be a future application of NIRS and this will play an important role. The majority of the works done till now has focused on forages, particularly in connection with management of forage, plant breeding and studies of conservative forages. Not many studies were conducted on feeding regimement. Forage is frequently a component of a complete diet, and the primary reason for analysing its constituent parts is to determine their quality for ration balancing (Chukwu and Durojaiye, 2014). The ingredients and the overall proportion must be analysed for various compositional and quality factors. An additional significant facet of nutritional constituent analysis is providing the computer with the necessary data for it to spontaneously choose the correct equation when analysing a mixed ration. However, it is critical to use practical information when one was supposed to use theoretical data through this method (Brunno-Soares *et al.*, 1998). It is important to look for crucial details revealed by the analysis about the feed ingredient and the ration. Quantitative and qualitative information should both be looked out for. Frequently, a desired outcome may be nominal rather than actual. As crucial as a quantitative examination is the knowledge that an element exceeds or falls short of an acceptable threshold. In principle, the near infrared spectroscopic instrument will act as an analyst would react to specific data, i.e., it should be able to determine whether the determined value is sufficient. Devices containing such expert systems will necessitate more advanced and adaptable software and computers that are more capable of running the software. It is conceivable that more than one type of analytical instrument will be connected to the computer information system. There will be two types of future systems for which we are developing prototype technology (Meuret *et al.*, 1993). The first is the previously described huge integrated system. The second type of instruments will be small, possibly portable, single-use, and dedicated to a particular analysis or commodity. These two types of instruments will be able to meet the requirements of the scientist, producer, and regulatory groups for feed and feed component analysis.

2.7.2.2 High Moisture Feedstuffs

For ruminants, feed items containing a substantial volume of water are an essential source of nutrients. High moisture feeds contain the majority of the nutrient properties necessary for measuring intake and digestibility in dry feeds (Enneking and Wink, 2000). The solubility and degradability of protein, as well as the concentration of the fibre components, influence the digestibility of both dry and high-moisture meals. In the case of silages, the use of NIRS immediately on wet materials saves time and improves prediction accuracy (Rushing et *al.*, 2016). Before NIRS may be used to assess feeds and forages for general ruminants, it must embrace the analysis of wet silages. However, there are difficulties in analysing high-moisture materials such as silages. In contrast to dried samples, which can be finely ground with a Wiley mill, it is difficult to grind wet samples to sufficient homogeneity, so wet sample sampling is not accurate. If the issues with grinding wet samples can be resolved affordably and effectively, the NIRS prediction of wet samples will be significantly enhanced (Abrams *et al.*, 1988). Near-infrared Reflectance Spectroscopy promises to be a precise and speedy method for evaluating certain chemical constituents in feeds with high moisture content. However, care must be taken when preparing wet samples for NIRS analysis, as well as identifying the constituents that are significant and can be measured satisfactorily using NIRS.

2.7.2.3 Mixed Feeds

Near-infrared Reflectance Spectroscopy analysis of mixed concentrates will allow feed millers to verify the accuracy of the feed formulation and provide livestock farmers with the ability to evaluate the accuracy of purchased concentrates. In addition, it will assist in determining whether or not complete rations have been correctly prepared. Initial investigations into the applicability of NIRS in evaluating mixed feeds yielded disappointing results. It was determined that the poor performance was due to the lack of uniformity in the mixed feeds (Castillo-Caamal *et al.*, 2003). This, however, implies that uniformly ground materials and complex regression techniques are essential for evaluating the chemical components of mixed feeds and minimizing variations in NIRS-obtained analytical values, while requiring the use of uniformly ground materials.

2.7.3 Analysis Procedures of NIRS Instruments

The importance of analysing the nutrient constitution of livestock feed lies in the decision making procedure to increase the quantity and quality of output. The majority of research conducted to date on the usefulness of NIRS for assessing the chemical components of feed ingredients has focused on forages. In most situations, forage comprises an important part of the entire diet; therefore, it is necessary to evaluate its elements to discover how they help to balancing diets. As early as the 1970s, near infrared spectroscopy was utilized for forage analysis (Brunno-Soares *et al.*, 1998), and several studies have demonstrated its utility in the cattle business (Brogna *et al.*, 2009). Prior research created calibration equations for feed and/or raw materials from various environments. These objectives were attained, and the numerous positive results proved the potential of NIRS for predicting the nutrient composition of feed, utilizing multiple tools and data processing methods. The findings of these and other studies have been documented and reviewed (Givens *et al.*, 1999). When it started, near infrared reflectance spectroscopy was only used to NIRS calibrations by using grounded and dried samples , this came with many disadvantages such as lengthy time spent on drying the samples and the possible alterations of the nutritional value due to higher temperature treatment of feeds, which could result in unacceptable results (Sprague *et al.*, 2003). The primary issue with silages is volatile compounds such as alcohols, esters, organic acids, amines, and ammonia that are lost (Givens *et al.*, 1999). Organic acids and alcohols are essential energy sources for ruminants and are also important silage fermentation indicators (Masoero *et al.*, 2007). In order to avoid an underestimation of these characteristics, improved NIRS devices have enabled the analysis of fresh, non-dried materials. Multiple investigations (Griggs *et al.*, 1999; Abrams *et al.*, 1988) have demonstrated that the presence of moisture does not significantly affect the prediction of components. This was accomplished by the enhancing the spectrophotometers' capacity to reduce water interference and developing the correction of light scattering which is chemo metric software (Griggs *et al.*, 1999; Murray and Cowe, 2004). Abrams *et al.* (1988) found that NIRS technology successfully evaluated the nitrogen content, DM and insoluble nitrogen of wet fodder even though

there was the influence of water on the near infrared spectra but it failed to assess the composition of fermented silage when. In his study on un-dried and fermented silage, Sinnaeve *et al.* (1994) accurately predicted fermentation products with R^2 of 0.93, 0.90, 0.85 and 0.86, respectively, for NH_3, pH, lactic acid, and acetic acid. In addition, the study showed that reflectance mode predicted forages more accurately than transmittance mode. Park *et al.* (1998) found that employing non-dried grass silages in an NIRS study enhanced the prediction several different chemical constituents, such as fermentation characteristics.

According to Sørensen (2004), NIRS can accurately predict pH, lactic acid, alcohol, acetic acid, and NH3-N in grass and corn silage. Near infrared reflectance technology is currently used to analyse the nutritional composition of natural pastures (Parrini *et al.*, 2017; Parrini *et al.*, 2019). Recently, various reviews on the use of NIRS technology in portable equipment have been published (Pasquini, 2018; Teixeira dos Santos *et al.*, 2013). Berzaghi *et al.* (2005) used a diode spectrophotometer, Zeiss Corona 45 (Carl Zeiss, Germany), with wavelength ranging between 960 and 1700 nm to study the practicality of using portable spectrophotometer to analyse the nutritional composition of corn silage without sample preparation.. In this study, 388 samples of corn silage were gathered and analysed over four years and a vast geographical area and the standard cross validation error (SECV) values for estimation errors were relatively low, indicating that predictions were accurate. The R^2 values for DM, NDF, and protein were respectively 0.87, 0.88, and 0.76. It was argued that the decreased protein figures were as result of the partial unpredictability of protein in corn silage. Mertens and Berzaghi (2009) determined the ability of both the laboratory spectrophotometer and portable spectrophotometer to predict and determine the dry matter of silage. It was concluded that the prediction errors of the diode array was almost about half more than the laboratory spectrophotometer, the overall error for moisture was however less. In view of the fact that the change in silage moisture from day to day can be greater than 10 %, a portable instrument allowed for a major modification of the feed, resulting in a significant enhancement in the accuracy of the amount of diet delivered (Mertens and Berzaghi, 2009). In a separate study, Patton *et al.* (2018) evaluated the predictive capacity of three portable instruments of NIRS to that of a

laboratory NIRS on fodders and found that the portable instrument sensors were less accurate than the laboratory equipment. The research included the examination of 96 samples of perennial Loliulm. The samples were analysed using an NIRS laboratory instrument with a wavelength range of 1100–2498 nm. PN-A predicted CP and water-soluble carbohydrates (WSC) similarly to Lab-6500, but was unable to predict DM and ADF. Lab-6500 projected values for all four parameters analysed were not replicated by PN-B. PN-C projected a mean DM comparable to that of NIR Systems 6500, but not for CP and ADF. The use of portable NIRS instruments was complicated by the inability to transfer robust calibration curves from a laboratory instrument to a portable instrument. Marchesini *et al.* (2018) investigated the feasibility of transferring calibration curves from a laboratory instrument to two versions (PL1 and PL2) of a next generation diode array portable spectrometer. Using the FOSS calibration curves as a reference, the transfer quality of the calibration equations between the two poliSPEC versions differed due to the differences in construction. PL2 was the successor to PL1 in terms of generation. PL2's superior structural qualities allow it to perform more effectively than PL1. The PL1 version of poliSPEC made the most accurate forecasts for DM, NDF, and ash, while the PL2 version also made accurate predictions for CP and ADF. Utilizing PL1 as a reference tool also yielded positive outcomes.

2.7.3.1 Sampling

A precise evaluation of the quality of forage is impossible without the analysed forage in the lab diligently resembles the average of the overall inhabitants from which it was sampled from (Mertens and Berzaghi, 2009). The greatest cause of error in any analytical system of forages is the improper sampling techniques and insufficient subsample numbers. Population sampling varies at each sampling location and between sampling locations. A cargo of baled hay is representative of several causes of variation (Marchesini *et al.*, 2018). Variations across bales are caused by the fact that different portions of the load originated from different regions of the field or even from separate fields. In addition, one portion of the cargo might have been bare to more moisture or sunlight than another. Consequently, sampling a single bale from the load may result in an analysis that substantially overestimates or underestimates the quality of the entire load. Variation inside the

bale (intra-site variation) may have multiple origins (Marchesini *et al.*, 2018). The stones outside the bale may have been subjected to weathering or oxidation. Depending on the interior's moisture content, oxidation or fungal growth may have occurred. In any case, sampling from the surface will produce inaccurate findings (Patton *et al.*, 2018). Hay's physical characteristics also contribute to diversity within a single site. It consists of leaves, stems, and infecting plants and is not uniform. Careless sampling from the bale frequently results in a sample with an atypical number of stems, making the hay appear inferior than it actually is.

Hand sampling typically does not produce representative samples of baled hay; a coring instrument is required. This apparatus is comprised of a stainless-steel tube with removable boring teeth at the sampling end and a fitting at the opposite end. There are two types of fittings available. One is compatible with an electric drill, while the other is compatible with a manual bracing. Due to its lack of power, the latter is more difficult to use, especially when sampling densely packed bales. According to the author, one core extracted from each of twenty randomly chosen bales will adequately represent one lot of hay. A lot of hay is defined by the following characteristics: it comes from the same field and cutting, is harvested within 48 h, and is of uniform maturity. The corer should be inserted from the long end of the bale, and the sample should be extracted from the middle. Using the included wooden rod, the corer is emptied into a sample collection bag following sampling. Then, it is determined whether sufficient samples have been obtained. Occasionally, when bales are lightly packed, the corer will collect little hay, striking primarily air pockets. When this occurs, it is necessary to resample the bale from a little different location. The collected samples must be combined in a single bag. In the sample bag, the composite sample should be thoroughly mixed. Although mixing can be achieved by hand stirring, sample rotation is the preferred method. The sample is placed in a plastic bag that is significantly larger than the sample taken, and the top is sealed with a small hole. The bag is then inflated to its full size, sealed, and rotated both vertically and horizontally to evenly disperse the sample. Sending the full sample to the testing laboratory is preferred. If the sample is too large, random subsamples may be gathered from various locations within the bag. Analysis should occur as soon as practicable following sample.

Sampling silage is more difficult than sampling than sampling hay. Regardless of the type of silo, only a fraction of the silage mass is acceptable. Due to the high moisture content, coring devices frequently fail to perform satisfactorily, and silage degrades rapidly when removed from the silo's anaerobic environment. For these reasons, Marum *et al.* (1979) reported that silage quality estimations are frequently less trustworthy than hay quality estimates. From the bunker silo, just the surface of the silage bulk is available for sampling. Two samples should be collected from the top, middle, and bottom of the distribution. Surface material should not be omitted because it is also fed. In an upright silo, an even lower amount of silage is available for sample. Typically, access to the material is restricted to an access door or the bottom unloader. The optimal time for sampling is during feeding, when the material is most concentrated, being discharged from the silo six or more random samples should be collected by hand at this time. If sampling is required at the door, the sample should be taken from as deep into the silage as practicable, given that silage near the door has frequently been exposed to oxygen (Mertens and Berzaghi, 2009). Silage is more difficult to combine than hay. If subsampling is required, silage samples should be well mixed by hand, reaching all the way to the bottom of the bag to prevent particles from settling. It is optimal to send the full sample for analysis. Before being sent to the laboratory, samples must be dried at 60 º C for at least 48 h.

2.7.3.2 Sample Preparation

Consistency in technique is essential to successfully use near infrared reflectance spectroscopy to analyse feed samples (Singh *et al.,* 2018). As a result of the near infrared reflectance spectroscopy been sensitive to each of the physical and chemical sample aspect, bands will vary depending on the procedure for drying and grinding the sample. Variations in these processes can easily provide erroneous findings. Marum *et al.* (1979) provided an illustration of this issue by noting that equations derived from fodder samples that were dried in non-cloth material such as paper were not able to analyse samples that were dried in cloth materials. Prior to grinding, samples that contain less than 88% DM must be dried in a 60º C oven with forced air. This temperature is high enough to force out the majority of the sample's water and sufficiently okay to not affect the chemistry of the

sample (Givens et *al.*, 1999). Once samples have been dried, they should be allowed to acclimate to the moisture and temperature. Samples that are moistened (such as silage) must be dehydrated for at least 48 h. Usng a microwave oven as an alternative drying process accelerates the speed of examination (Patton *et al.*, 2018). The drying time is decreased from more than a day to a few minutes. Nevertheless, using microwaves irresponsibly might end up burning the sample as a result of the excessive radiations of the microwave exposed to the samples (Givens et *al.*, 1999). Afterwards, the samples must be dehydrated to a DM level of between 90 and 94%. Important to the use of the microwave oven is trial and error, which allows the operator to determine the appropriate drying time (Marchesini *et al.*, 2018).

When doing near infrared reflectance spectroscopic analysis, samples must be grounded using a cyclone mill with a 1-mm screen. Due to the fine-grained particle size produced by the cyclone mill, unpublished studies have proven that the cyclone mill improves the precision of analysis than the standard Wiley mill. Cleaning and blowing the mill after treating each samples ensures that there is no cross-contamination (Nocek, 1988) and the periodical replacement of the crushing ring ensures that the particles sizes are always consistent (Hoden, 1999). Samples should not be disposed immediately as it is frequently necessary to preserve samples for extended period of times in the event that provision of the samples to others for recalibration is needed. An important point to note is that the reserved samples stay unmodified in their chemical composition. Alterations in moisture levels of the sample because of anaerobic and aerobic organisms causing degradation in the sample's composition might cause alterations in spectra. In order to reduce microbial activity, it is required to store samples in a container that is airtight and that contains at least 88 % (Marum et al., 1979). The preferred storage container is the retort pouch, which consists of aluminium foil inserted between layers of two plastics. The bag is closed on all sides before being filled with a sample and heat-sealed on the last side. Care must be taken not to crimp the edges of the sealing surface during the sealing process, as this will allow moisture to enter or exit the bag. When a sample is required, the end of the pouch can be severed with a paper cutter, a bit of the stored sample can be extracted, and the pouch can be resealed.

2.7.3.3 Instrument Operation

Near infrared reflectance spectroscopy measurements are extremely sensitive to instrument and sample temperature. Consequently, all Instruments are equipped with temperature controls (Brunno-Soares *et al.*, 1998). These controls function properly at temperatures between 25 and 5^{o}C. However, the equipment does not regulate sample temperature, thus the user must ensure that samples are at room temperature prior to analysis. If the room environment cannot be maintained according to specifications, an alternative approach must be used to calibrate the instrument so that it is applicable across a range of environmental circumstances (Marchesini *et al.*, 2018). Same-sample spectra should be acquired under varied room conditions. These samples will have identical chemical values; hence, when the computer calibrates the wavelengths, the effects of fluctuating ambient temperature and humidity will be reduced. This method would also be applicable to studies of samples with varying temperatures. Instrument warm-up is essential, and the required time varies by instrument type. It is advised that the majority of instruments operate continually. Continuous operation is optimal for the electronic components, and the average lifespan of a light bulb is now roughly three years (Brunno-Soares *et al.*, 1998). However, if the instrument is turned on from a cold state, the warm-up time must be at least 15 min. and may be as much as 1 min. for certain instruments. The SCAN software activates the bulb within the Pacific Scientific 6350 monochromator. Connecting the instrument to AC electricity and inputting the word SCAN into the computer to activate the warm-up period completes the warm-up. The required warm-up process for other instruments can be found in their respective manuals. Before beginning each work session, two diagnostic tests should be done after the instrument has been adequately warmed. First, the instrument's decibel level must be determined. Second, the correctness of the wavelength must be confirmed. Other diagnostic tests that can be performed on monochromators include optimal data scaling and instrument-to-computer communications (Patton *et al.*, 2018)

2.7.3.4 Selection of Equation

Narrow- and broad-based equations are the two fundamental types of analytical equations utilized in NIRS. Equations with a narrow base established for a definite population has restricted utility outside its application to the samples that are not in the same population. It is even challenging to

build a broad-based equation for an unlimited population; nonetheless they have several applications when built. Broad-based equations include those established by the individual labs to analyse daily forage samples and service calibration curves developed by public laboratories to analyse research forage samples from several scientists. Equations derived for populations that are indefinite are called universal equations. The term "universal" indicates that the calibration equation can be applied to a substantial number of samples (Brunno-Soares *et al.,* 1998). A perfect example is a universally established hay equation that include widespread amount of samples from an extensive locations with varied species of forages and species. Considerable amount of the dissatisfaction expressed regarding the use of NIRS stems from a lack of comprehension of the universal equation concept. The calibration samples needs to cover all variables that influence the physico-chemical characteristics of the samples to be analysed (Brunno-Soares *et al.*, 1998). This criterion is closely tied to the equation's precision. The data in Table 2 illustrate several characteristics of narrow- and universal-based equations. Hay samples were used in a factorial field experiment aimed at testing the universal equation hypothesis (Templeton *et al.*, 1983). The samples were sourced from various years, species, drying techniques and harvests. In the trial, the parameters were species (alfalfa, red clover, bird's foot trefoil, orchard grass, and timothy), treatment, and time.

Table 2.2: Prediction standard errors for a single value of five forage quality parameters predicted by NIRS

		Quality Parameters				
Used to design equation	Equations used on	CP	NDF	ADF	Lignin	IVDMD
Birds foot trefoil	Alfalfa	0.40	1.75	2.93	0.69	2.14
Orchard grass	Red-clover	1.67	15.90	4.24	2.08	3.88
Orchard grass	Timothy	0.40	1.35	1.57	0.54	2.28
Oven drying at 65º C	Oven drying at 75º C	0.50	1.62	0.93	0.62	2.23
Oven drying at 65º C	Field-drying for 3 days	0.99	4.46	3.19	1.34	5.69

Harvest 1	Harvest 2	0.94	2.52	1.52	1.42	3.63
Half of samples	Other half	0.66	1.97	1.02	0.59	1.80

CP = crude protein; NDF = neutral detergent fibre; ADF = acid detergent fibre; IVDMD = *in vitro* digestibility (Yakubu et *al.*, 2020)

Table 2.2 above demonstrates that a single specie equation designed for analysing only one samples of only plant like the orchard grass, was not able to yield correct findings when applied to an unlike plant species like the clover. Similarly, equations derived from oven dried samples could not produce accurate results when applied to samples that were dried on the field. Nevertheless, the universally derived equation which included half of the samples, reflecting all bases of physic-chemical variance, was accurate enough for all samples in the experiment. These equations will be required to make pre-calibrated instruments accessible to both the public and private sector. It is conceivable, with practice, to gather a collection of samples with widespread properties devoid of employing the factorial method. The vital point is to recollect the simple theory underlying the idea of the standardization. Equation for instrument calibration might be obtained from the instrument's owner or bought from public or private sources. Whether the equation for the analysis is to be acquired from a private or public source, it is advisable to take the following precautions:

- Appropriateness of the equation for the instrument to be used.
- There exists an acceptable method for transfer into the instrument the equation of the analysis.
- The equation of analysis was made for its intended use.
- The equation contains all necessary analysis.
- The type and dependability of the chemical analysis utilized in the calibration technique are beyond question.
- The equation guarantees a level of accuracy and precision that has been demonstrated.

2.7.3.5 Validation of Equation

Validation of equations is done with three basic steps. To assist in the selection of the right equations validation must be done with a subset of the analysed population that did not take part in

the equation for calibration (Marum et *al.,* 1979). Samples used in validation during routine near infrared reflectance spectroscopy analysis are usually smaller than calibration set samples and unknown to the calibration set. Examining residuals for larger t and H outliers is the foremost step in applying validation to selecting equations. Obtaining larger value for t with fewer samples suggest that the laboratory data did not adequately match the samples and the samples measurements must be replaced with new measurement of the same population in the analysis where as numerous samples for validation that has larger *t* values indicates over fitting and it means the equation is definite to only the samples that were used for calibration (Bruno-Soares et al., 1998). Bad measurements cannot be overlooked as fewer bad sample measurements can gravely affect the validation statistics (Singh *et al.,* 2018). Large values of *H* for fewer samples indicate that they have different characteristics to the calibration samples and should be assessed whether there was an omission if only their t values are larger too. The statistic that truly defines the ability and accuracy of equations to predicting an unknown sample from the similar populations is the standard error of prediction (SEP). The general rule is to select the equation with the smaller SEP. Another important statistic is the slope of the regression line which correlates the laboratory determined values to the NIRS determined figures. The slope must be closer to 1.0. Should it be significantly different from 1.0, which would indicate that the high and low values were constantly over or under estimated. Finally, the equation is validated with a different set of samples.

2.8 Review of Feed Evaluation Methods, Economic Considerations and Speed of Analysis

2.8.1 Introduction

Evaluation of feedstuffs offers information on their quality. It is essential for determining the ability of feedstuffs to meet the nutritional needs of animals (Beever and Moud, 2000). There have been numerous ways for evaluating feed throughout the years. Numerous modifications have been made to improve accuracy and precision (Dijkstra *et al.,* 2005). The nutritional value of ruminant feeds is determined by their chemical makeup and concentration of the different elements (Chumpawadae *et al.,* 2007). Animal performance can be projected with certain accurateness based on examination of the feed.

This study attempts to examine the available methods for evaluating feed, with an emphasis on performance and economic factors.

1. Chemical Procedure

Feed evaluation has been based on the proximate composition of feedstuffs (CP, CF, minerals, ash, NFE and moisture) for a very long period. The Weende technique classifies carbohydrates as two indigestible coarse fibres and a soluble nitrogen-free extract (Williams and Norris, 2001). Chemical analysis of ruminant feeds involves determining the feed's DM, organic matter (OM), structural carbohydrate, soluble carbohydrate, crude fat, CP, and OM (France *et al.*, 2000). Wood and Badve (2001) also devised the detergent analysis, a technique that more precisely classifies carbohydrates into cell content and cell wall using NDF. Lignin, hemicellulose, and celluloses are the primary constituents of NDF, whereas hemicellulose and celluloses are the primary constituents of ADF. Animal intake and in-vivo digestibility cannot be predicted just by chemical composition. Using the Kjedahl method and acid digestion and distillation, CP is estimated from the nitrogen fraction of the feedstock using the chemical approach. This approach measures nitrogen instead of protein. The measured nitrogen is multiplied by 6.25 to estimate the feed's protein level.

2 Digestibility Methods

2.1 In-vivo digestibility

A feeding trial is the usual method for measuring digestibility. It is believed that feeding the proper animal is a more reliable method for determining the nutritional value of any diet. Protein is the most often measured nutrient using the in-vivo approach. Several different methods, such as total faecal collection, the use of chromic oxide as a marker, and indigestible ADF/NDF as a marker, have been developed and modified over time to increase the assessment of in-vivo digestibility (Church, 1991). To quantify digestibility, the recovery of these markers is evaluated.

2.2 In-Situ Procedure

The in-sacco method for measuring protein digestion in ruminants involves suspending a nylon bag carrying test feed in the rumen of an animal with a fistula. According to Nocek (1988), the nylon bag approach provides increased contact between the feed and the ruminal environment. After incubating the nylon bags in the rumen for varying amounts of time, the degree and digestion rate of the test material can now be determined. Post-ruminal washing, reduction in bag pore size, and sample size to bag surface area are suggested to limit variability due to microbial contamination, sample preparation, processing, and bag type discrepancies (Lindberg, 1981; Nocek, 1988).

2.3 In-vitro Digestibility

For assessing in-vitro digestibility, the in-vitro digestibility (IVDMD) and in-vitro gas production (IVGPT) methods are utilised. Tilley and Terry's two-stage in-vitro digestibility method is a frequently used in-vitro digestibility method (IVDMD) for determining feed digestibility. With this procedure, meals are incubated in rumen fluid for 48 h before being digested with pepsin. The widespread adoption of the Tilley and Terry approach is due to the simplicity and utility of the data it produces (Wood and Badve, 2001). Holden (1999) reported a higher association between in-vitro and in-vivo digestibility in his investigation.

In-vitro gas production technique (IVGPT) includes fermenting feed samples using natural rumen microorganisms under controlled laboratory conditions employing a variety of treatments. With a mixture of rumen fluid, buffer, and minerals, a sample of feed is incubated at 39 °C for varying amounts of time (24, 48, 72, 96, or 144 h) (Beever and Moud, 2000). Measuring the total gas produced during incubation per gram of DM of deteriorated feed samples (Wood and Badve, 2001). The purpose of the IVGPT is to increase the fermentation of feed in the rumen (Rymer *et al.*, 2005).

2.8.2 Economic Considerations, Efficient Analysis and Swiftness

Conventional wet-chemistry laboratory procedures are more expensive than NIR. As the per-sample cost of regular wet-chemistry procedures is substantially more than that of the near infrared reflectance method (Table 2.3). Wet-chemistry procedures are extremely time-consuming, requiring a whole day to produce results. The in-vivo approach (total collection technique) is the most accurate method for assessing the digestibility of feed among the available methods. Nevertheless,

it has proven to be time-consuming, arduous, and costly. It is not applicable to all actual feeding circumstances and cannot be performed as standard laboratory work (Zewdie, 2019; France *et al.*, 2001). The in-situ digestibility approach is costly, time-consuming, and involves the use of rumen fistulated animals with low repeatability from laboratory to laboratory (Wood and Badve 2001).

Sample preparation time for NIR is less than 2 minutes, and many analytes can be asessed from a single sample, making NIR as quick as regular techniques (Table 2.3).

The results of a study by Singh *et al.* (2018; Table 2.3) indicate that the non-destructive method of NIRS could be used to determine the proximate compositions (protein, oil, and fibre content) of cereals, grains, and legumes over the destructive (traditional) method, as the analytical results obtained for these parameters were significantly correlated for both techniques. The cost-effectiveness and speed of NIR analysis compared to conventional methods (Table 4.6) make this approach suitable for use in feed analysis in the livestock and agriculture industries (Singh *et al.*, 2018). Yang *et al.* (2017) saved around 80% of typical laboratory costs when estimating the fodder quality characteristics of *Lolium multiflorum* by employing the NIRS approach. The scientists noted that no chemical waste was produced by the process compared to traditional wet-chemistry, and they proposed that this should be an incentive for environmental friendliness and lowering reagent costs.

Table 2.3: Operating Cost and Speed of Different Methods

Technique	Chemicals/sample	Instrumental cost	Total cost/Analyte	Cost/sample	Time/sample
Spectrophotometer (protein)	8	0.3	9	92	6 h
Soxhlet (oil)	40	8	48	92	6 h
Gravimetric method (fibre)	28	7	35	92	6 h
NIRS	-	2	2	2	5 min

Singh *et al.*, 2018

2.8.3: Conclusion

A feed evaluation method provides data on feed materials to meet the nutritional needs of the animal. Different methods have been established and improved over the years to assess the chemical composition of feeds. Digestibility, chemical and enzymatic approaches are the main feed evaluation methods. For the past few decades the near infrared reflectance spectroscopy has been found to be preferable to the traditional wet-chemistry methods. It needs no sample preparation and non-destructive. In terms of cost, analysis time, and safety, the near infrared reflectance spectroscopy is preferable for accurate and speedy calculation of feed composition when the samples are homogeneous.

CHAPTER THREE

3.0 MATERIALS AND METHODS

Study 1: Prediction of Sorghum and Groundnut Residues Using NIRS

3.1: Materials

A total of 561 sorghum and 1010 groundnut samples were obtained from sorgum stover and groundnut haulms, respectively. The materials were obtained from an on-farm breeding and agronomic trials conducted in Kaduna, Sokoto, Kano, Zamfara, Katsina, and Plateu all in the northern parts of Nigeria, West Africa.

3.2: Sample Preparation and Wet-chemistry Analyses

Each sample, about 100 g was dried in a forced air oven at 60º C to constant weight, ground with a Wiley mill to pass through a 1 mm sieve before analysing for CP, IVOMD and NDF. Analysis was done with reference to Association of Official Analytical Chemist (AOAC, 1995) standards and according to Van Soest *et al.* (1991) for CP, NDF and IVOMD respectively.

3.2.1: Determination of Crude Protein

The CP contents were assessed by wet-chemistry analysis with reference to the Kjeldahl method (AOAC, 1990) with the KjeltecTM 8400 analyzer unit (FOSS, Hoganas, Sweden). In a 250 ml TKN digestion tube ground samples of 0.5 g each were added with 10 ml conc. H_2SO_4 acid and two digestive tablets (Beijing Jinyuanxingke Technology, Beijing, China). Processing of blanks with all the reagents was done simultaneously. After that the test tubes were digested in the digestion block (Tecato TM digestor auto; FOSS, Hoganas, Sweden) at 420 ºC for 90 min when the samples were clear and green. KjeltecTM 8400 analyser (FOSS, Hoganas, Sweden) was used to was employed to carry out the Kjeldahl technique. The CP was calculated using the equation below:

$$\text{CP (\%DM)} = \frac{(V1-V2)\times C\times 1.4007\times 6.25}{\text{M}} \times 100$$

Where V1 = Volume of HCL required, V2 = Volume of HCL required for blank, C = Molarity of HCL used, 1.4007 = milliequivalent weight of N×100, 6.25 = average coefficient of nitrogen conversion into proteins, and M = weight of sample.

3.2.2: Determination of Neutral Detergent Fibre

The NDF content was determined by using Goering and Van Soest (1970) and Van Soest *et al.* (1991) described method with 0.5 g ground samples in Automatic Fiber Analyzer (ANKOM 2000 Fiber Analyzer; ANKOM Technology, NY, USA). The contents of NDF were calculated using:

$$\text{NDF (\%DM)} = \frac{(M2-(M1\times C1))}{M} \times 100$$

Where M = weight of sample, M1 = weight of bag, M2= weight of organic matter after extraction by neutral detergent, and C1= Ash corrected blank bag factor (weight of extracted blank bag/weight of original blank bag).

3.2.3: Determination of In vitro Organic Matter Digestibility

The IVOMD was determined by the Tilley and Terry method modified by Alexander and McGowan (1996). Briefly, 0.5 g of milled samples were weighed and incubated at 39 ºC for 48 h in an incubation flask. Then 10 ml of rumen liquor and 40 ml of McDougall buffer were added to the solution followed by the addition of HCL and the solution was further incubated for 48 h with 50 ml of pepsin. The solution was filtered at the end and the residue obtained was poured into a crucible which was combusted at 105 ºC, increased at 500 ºC, weighed, and used to compute the IVOMD.

3.3: Spectra Data Measurement

Near infrared reflectance spectroscopy analysis was performed using bench top FOSS DS-2500 scanning monochromator (FossNIR –System, Hillerod, Denmark). The FOSS DS-2500 monochromator was turned on and preheated for an hour before spectra collection begun. Three subsamples were exposed to an electromagnetic scan in the absorbance mode for each sample with the monochromator covering a range of 850-2500 nm. About 100 g of the ground samples of the residues was scanned using a slurry cup with quartz window of about 12.6 cm^2 area. Each sample's spectra was measured and adjusted against the background spectrum at ambient temperature. As a means of evaluating the possibility for prediction, the final spectrum of each sample was the mean of the three measurements. In this experiment about 70% of the samples of each crop were selected

randomly to include in the calibration set whiles the remaining 30% formed the validation set (Wu *et al.*, 2023).

3.4: Development of Calibration Models

In this work, the samples were divided into two sets for each constituent: a larger set (calibration set) for the development of calibration models, and a smaller set (validation set) for testing the correctness of the calibration equations presented in Tables 4.1 and 4.2, respectively for sorghum. The means of these three measurements was used for multivariate analysis using PROC REG procedure of SAS.

3.5: Review of Analytical Methods

An extensive reading of journal papers and books on different analytical methods was done to conduct a cost benefit analysis.

3.6: Data Analysis

The accuracy of the prediction of the sorghum samples was determined using the determination coefficient (R^2), standard error of prediction (SEP), and Residual Predictive Deviation (RPD) between wet-chemistry laboratory values and the anticipated NIRS values.

All the 1010 groundnut samples were normally distributed and analysis between and within samples were undertaken using ANOVA and Pearson correlation to test for relationship between values obtained with routine methods and NIRS measured values. All the components were tested for CV% for within location and overall measurements on each method. Meta-analysis was applied in reviewing the differences in cost-effectiveness, swiftness and environmental friendliness of the various methods of feed evaluation. The databases were configured from literature publications for pigeon pea, soybean, rice and *Lolium multiforum* studies.

CHAPTER FOUR

4.0 RESULTS

4.1 Prediction of Sorghum and Groundnut Residues

4.1.1: Chemical Characteristics of Sorghum Residues

The wet-chemistry values of the calibration set samples (n=164) are shown in Table 4.1. Due to the heterogeneity in different parts (leaves, shoot and roots), developmental stages and location of sorghum residues, the CP, NDF, and IVOMD parameters exhibited wide variation. The wet-chemistry values of samples used for CP calibration ranged from 0.6% to 7.4%, with a mean of 4.2% and a SD of 1.4. The calibration samples for IVOMD ranged from 47.6% to 68.2%, with a mean of 58.8 % and a SD of 3.8 whiles the NDF calibration curves displayed values between 54.7 and 85.5 %, with a mean of 69.2 % and a SD of 4.0.

Table 4.1: Composition summary of sorghum residues used for calibration, including sample count (n), mean, standard deviation (SD), minimum, and maximum values.

Parameter (DM %)	n	Mean	SD	Min	Max
CP	164	4.254	1.463	0.61	7.49
IVOMD	164	58.876	3.847	47.64	68.28
NDF	164	69.256	4.042	54.74	85.59

n= number of samples, SD= standard deviation, min= minimum, max= maximum

The samples used to validate the calibration equations had amplitude components that were more similar to those in the calibration set. The range of CP values was from 1.1 to 6.9%, with a mean of 4.1% and a SD of 1.4. In vitro organic matter digestibility values varied from 50.8 to 67.0%, with an average of 58.8%. The range of NDF values was 57.4% to 76.8%, with a mean of 68.8%.

Table 4.2: Composition summary of sorghum residues utilized for validation, including sample count (n), mean, standard deviation (SD), minimum and maximum values.

Parameter (DM %)	n	Mean	SD	min	Max

CP	58	4.102	1.370	1.06	6.85
IVOMD	58	58.842	3.492	50.79	67.03
NDF	58	68.815	3.971	57.36	76.79

N= number of samples, SD= standard deviation, min= minimum, max= maximum

4.1.2: NIRS Calibrations and External Validation Models

Identified spectral points in the infrared area carrying information on the analysed sorghum samples were used to create multivariate calibration for regression model development against the laboratory determined values. The spectrum used to generate IVOMD calibration was incompatible with the reference data, although the spectrum used to develop the CP and NDF models was linear. The calibration set (Table 4.3) displayed an R^2 of 0.83%, SEC of 0.60%, and RPD of 2.43% for CP. For IVOMD calibration displayed an R2 of 0.28%, SEC of 3.27%, and RPD of 1.17%. The NDF calibration had a R^2 of 0.79%, a standard error of estimate (SEE) of 1.79%, and a relative RPD of 2.25%.

Table 3.3: Statistics of NIRS Calibrations for CP, NDF, and IVOMD in Sorghum Stover

Parameter (D %)	N	SEC (%)	R^2	RPD (%)
CP	164	0.60	0.83	2.43
IVOMD	164	3.27	0.28	1.17
NDF	164	1.79	0.79	2.25

N= number of samples, SEC= standard error of calibration, R^2= coefficient of determination, RPD= residual predictive deviation.

The external validation set (Table 4.4) was comprised of 58 independent samples from the calibration set. As an improvement over the calibration model, the validation set demonstrated the highest coefficient R^2 of 0.89% (Figure 2), with SEP of 0.47% and RPD of 3.06%, for CP. In vitro organic matter digestibility validation demonstrated a R^2 of 0.30% (Figure 3), SEP of 3.00, and RPD of 1.16%, whereas NDF validation had a R^2 of 0.86% (Figure 4), SEP of 1.50%, and RPD of 2.57%.

Table 4.4: External validation statistics obtained from regression equations of laboratory values of CP, IVOMD and NDF in sorghum and NIRS predicted values of the validation set.

Parameter (DM %)	N	SEP (%)	R^2	RPD (%)	RER
CP	58	0.47	0.89	3.06	14.47
IVOMD	58	3.00	0.30	1.16	5.41
NDF	58	1.5	0.86	2.57	12.95

N= number of samples; SEP= standard error of calibration; R^2= coefficient of determination, RPD= residual predictive deviation (SD/SEP); RPD= range error ratio (range/SEP).

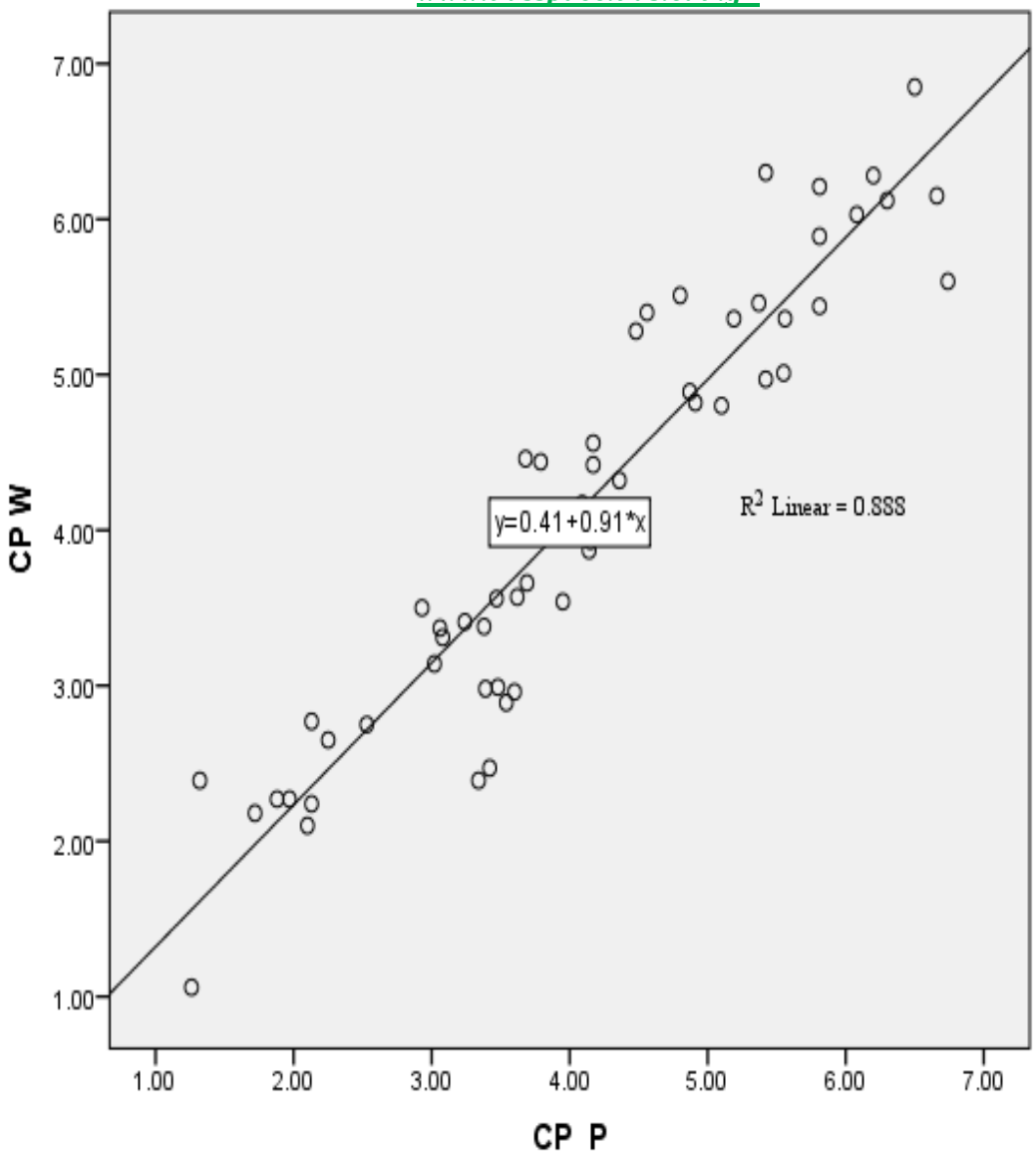

Figure 2: Relationship between predicted values (x axis) and laboratory determined values (y axis) in the calibration set for CP in sorghum.

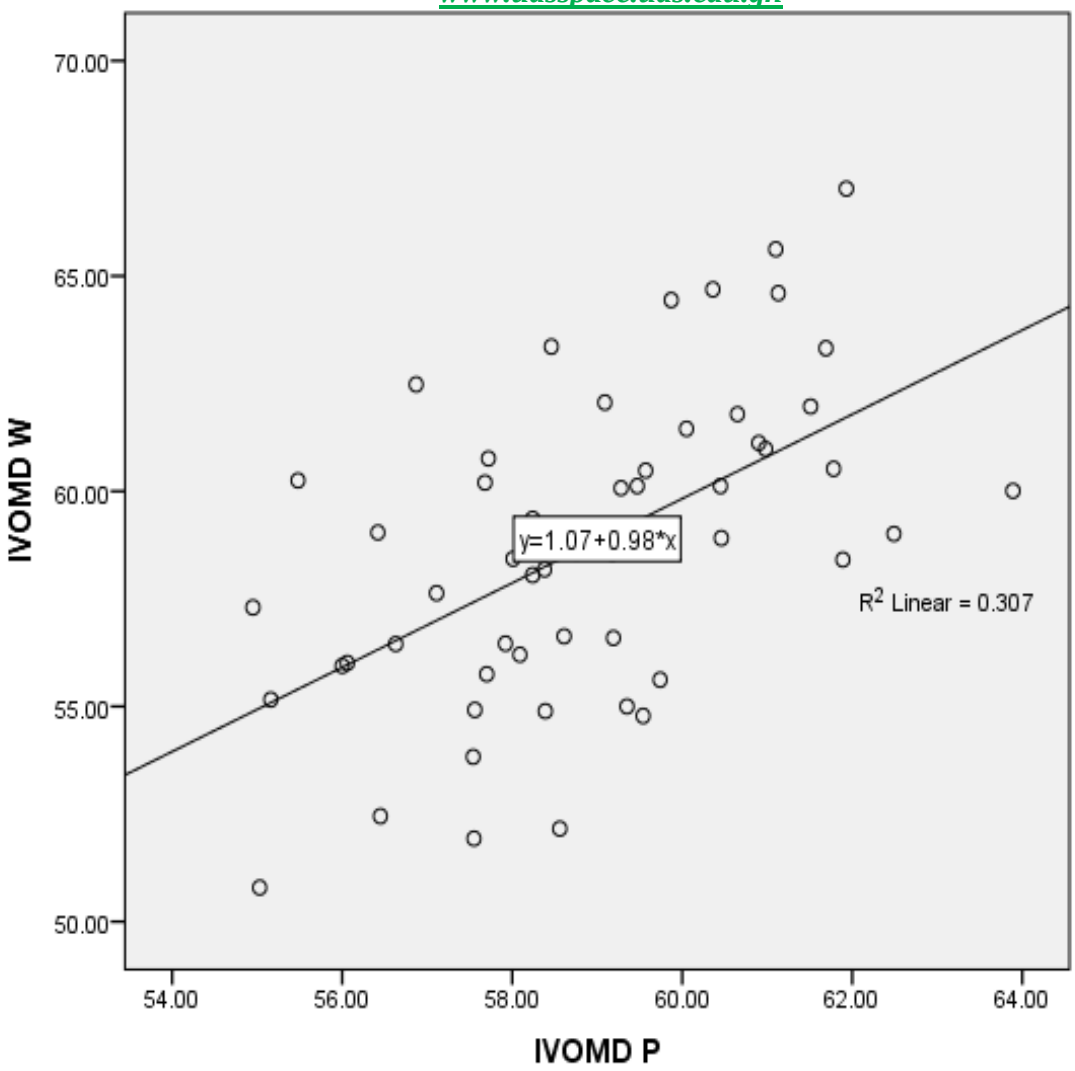

Figure 3: Relationship between predicted values (x axis) and laboratory determined values (y axis) in the calibration set for IVOMD in sorghum.

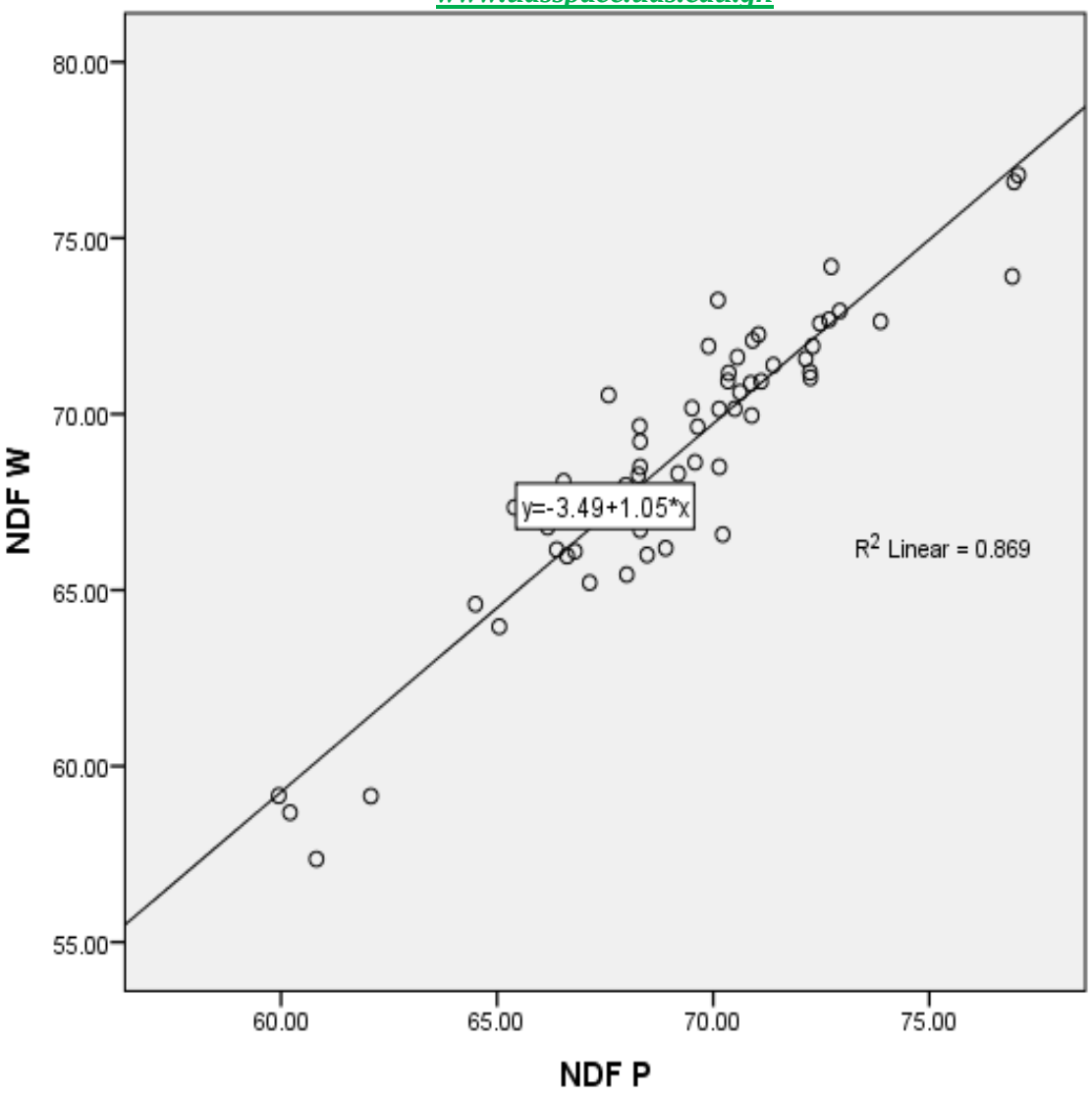

Figure 4: Relationship between predicted values (x axis) and laboratory determined values (y axis) in the calibration set for NDF in sorghum.

4.1.3: Analytical Efficiency of NIRS and Routine Methods for Estimating Groundnut Residues

Table 4.5 shows an analysis of the CP and NDF concentration and IVOMD in 1010 groundnut residues. The groundnuts samples used ranged from 3.08 to 13.88 for CP, 26.03 to 75.02 for IVOMD and 28.87 to 70.30 for NDF. The CV%, a measure of variation in repeated examinations, was found to be lower in NIR spectroscopy than in the wet-chemistry procedures employed to determine the CP, IVOMD, and NDF of groundnut residues.

Similar trends were seen at each of the six locations listed in Table 4.5. Wet-chemistry values for CP, IVOMD, and NDF of groundnut samples from Kaduna varied from 3.08 to 12.48%, 37.24 to 71.71%, and 34.00 to 70.30%, respectively. The CP, IVOMD, and NDF contents of groundnut

residues from Kano ranged from 4.75 to 13.88%, 26.03 to 71.85%, and 28.87 to 63.56%, respectively. In contrast, the CP, IVOMD, and NDF contents of groundnut residues from Katsina ranged from 5.93 to 1.35%, 58.20 to 72.27%, and 33.34 to 44.85%, respectively. Similarly, the CP, IVOMD, and NDF values of groundnut residues from Plateau ranged from 4.19 to 11.47%, 45.89 to 75.02%, and 31.64 to 60.56%, respectively, when analysed using either the conventional we-chemistry approach or NIR spectroscopy. Table 4.5 displays the analytical data for both Sokoto and Zamfara groundnut residues. In general, and in all sites, NIR spectroscopy produced more repeatable results than other techniques, however for the Zamfara state, the traditional we-chemistry method was more consistent (CV% = 4.69) than NIR spectroscopy (CV% = 5.17) with respect to IVOMD analysis. There was a higher correlation between the measured values of CP, IVOMD, and NDF for NIRS and their corresponding we-chemistry values (Table 4.6).

Table 5.5: Nutrient Composition of Groundnut Residues Measured with Traditional and NIRS Methods

Location		CPWET	CPNIRS	WETIVOMD	NIRSIVOMD	WETNDF	NIRSNDF
Kaduna	Mean	7.18	7.17	57.12	56.97	49.39	49.30
	CV%	23.25	21.33	9.75	9.05	14.41	12.08
	N	287	287	287	287	287	287
	Min	3.08	3.54	37.24	41.28	34.00	36.70
	Max	12.48	9.49	71.71	70.31	70.30	65.09
	SEM	0.10	0.09	0.33	0.30	0.42	0.35
Kano	Mean	8.35	8.19	60.17	59.75	45.72	45.81
	CV%	17.84	15.5	10.10	7.73	14.58	11.24

	N	239	239	239	239	239	239
	Min	4.75	4.75	26.03	46.96	28.87	35.5
	Max	13.88	12.43	71.85	68.49	63.56	62.25
	SEM	0.09	0.08	0.39	0.29	0.43	0.33
Katsina	Mean	8.57	8.63	63.13	63.09	40.82	40.94
	CV%	5.25	3.93	2.15	1.74	4.45	3.88
	N	117	117	117	117	117	117
	Min	5.93	7.41	58.20	57.30	33.34	35.99
	Max	10.35	10.35	72.27	65.96	44.85	46.62
	SEM	0.04	0.03	0.13	0.10	0.17	0.14
Plateau	Mean	7.92	7.74	60.04	60.39	46.84	47.10
	CV%	18.05	13.43	8.44	6.45	10.61	7.47
	N	153	153	153	153	153	153
	Min	4.19	5.46	45.89	51.51	31.64	36.61
	Max	11.47	9.65	75.02	70.43	60.56	55.70
	SEM	0.11	0.08	0.41	0.32	0.40	0.28
Sokoto	Mean	8.13	8.12	60.08	61.57	44.34	44.42
	CV%	9.47	7.14	4.11	3.05	8.47	7.06
	N	106	106	106	106	106	106
	Min	5.91	5.91	56.16	54.57	33.77	37.22
	Max	11.33	9.12	71.27	65.56	54.50	53.58
	SEM	0.07	0.05	0.24	0.18	0.36	0.30
Zamfara	Mean	8.07	8.09	59.37	59.56	46.44	46.50
	CV%	12.39	10.38	4.69	5.17	6.97	6.75
	N	108	108	108	108	108	108

	Min	4.85	6.82	52.19	52.87	34.93	33.58
	Max	11.72	11.72	66.77	70.39	54.47	54.47
	SEM	0.09	0.08	0.27	0.29	0.31	0.30
Overall	Mean	7.92	7.87	59.73	59.62	46.29	46.36
	CV%	18.18	15.88	8.68	7.54	13.35	11.19
	N	1010	1010	1010	1010	1010	1010
	Min	3.08	3.54	26.03	41.28	28.87	33.58
	Max	13.88	12.43	75.02	70.43	70.30	65.09
	SEM	0.04	0.03	0.16	0.14	0.19	0.16

CV% = Coefficient of variation, SEM = Standard error of means, Min = Minimum value, Max = Maximum value, CPWET = Wet-chemistry values for crude protein, CPNIRS = Crude protein values determined by near infrared spectroscopy, WETIVOMD = Wet-chemistry values for digestibility, NIRSIVOMD = Digestibility values determined by near infrared spectroscopy, WETNDF = Wet-chemistry values for neutral detergent fibre, NIRSNDF = near infrared values for neutral detergent fibre.

Table 6.6: Measures of Association between we-chemistry and NIRS Methods

Component	R	R Squared
Crude Protein	0.89	0.77
Digestibility	0.88	0.78
Neutral Detergent Fibre	0.82	0.67

Significant at 0.05

CHAPTER FIVE
5.0 DISCUSSIONS

5.1: Prediction of the Chemical Composition of Sorghum Residues with NIRS

Baseline nonconformities and interferences from the NIRS machine have a tendency to cause mistakes in the set of spectrum utilized for calibration (Siesler *et al.*, 2008); hence, spectral pre-treatment techniques were required to reduce the influence of the undesired components. In this work, mathematical pre-treatment was applied to the data prior to processing to aid in the calibration development (Neves *et al.*, 2012) and to improve reliability. Different chemical connections, primarily C-H, O-H, and N-H, were measured spectrally at different wavelengths (850-2500 nm) (Bokobza, 2002). All of the primary chemical constituents of feedstuffs have unique infrared absorption characteristics due to the bending and stretching of H bonds associated with C and N.

For the creation of appropriate forage calibration equations, a considerable sample range of all the analysed components is required. Variations in nutrient concentrations were caused by factors such as geographic location, soil structure, environmental circumstances, and variances in growth stages. The calibration equations were confirmed using samples from the same population, but these samples were excluded from the calibration set. External validation is a crucial step for enhancing the performance of prediction equations (Lobos *et al.*, 2013). The parameters were modified by removing outliers from the spectral limits of the calibration models' extreme values. Outlier identification is crucial for enhancing the quality of calibration equations, which are dictated by calibration quality (Li *et al.*, 2016).

In general, the predictive ability and reproducibility of the spectroscopic spectral data and the standard laboratory values used to build the calibration equations were satisfactory. The CP component of feedstuffs has been accurately determined by near infrared reflectance spectroscopy in multiple studies (Andueza *et al.*, 2011; Rushing *et al.*, 2016; Chen *et al.*, 2015) and, according to Yang *et al.* (2017), may have been the most extensively studied variable in feed materials. Consistently good R^2 and RPD values were obtained in this work for the CP prediction equation,

which had R^2 and RPD values of 0.89 (Figure 2) and 3.06, respectively. Considering the RER results, a further evaluation of the prediction models' precision was conducted. The RER value obtained from the CP calibration model fell within the AACC (1999) quality threshold values for RER-based model performance. Method 39-00.01 of the AACC (1999) indicates that RER value of 4 indicates that the model is satisfactory for screening samples; RER value of 10 indicates high acceptability for quality control; and RER value of 15 indicates that the calibration is suitable for quantification. In conclusion, the CP model had a more stable performance and greater predictive ability than other models and can be widely used to assess the forage quality of sorghum.

The mean, SD and range values for IVOMD of sorghum are presented in Table 4.1. The data indicated that IVOMD calibration set did not compare well with the corresponding values in the prediction set. Calibration statistics as well as prediction statistics to validate the developed equations are shown in Table 4.3 and 6 respectively. The performance of the equations for IVOMD was not successful. Though the RER value of 5.41 was within the acceptable threshold for sample screening (AACC, 1999) the calibration statistics (R^2 = 0.3, SEP = 3.0, RPD = 1.16) (Figure 3) indicated a poor prediction ability of the IVOMD models. This result did not agree with Snyman and Henda (1992). They had reliable prediction of digestibility for most forage but for maize residues. The correlation coefficient (r^2) values were generally high (> 0.9) while the SEP values were relatively low. The explanation was that the closeness of the slope to 1 and the bias to 0 supported the high prediction of digestibility. Also, the near infrared reflectance spectroscopy determination of digestibility is contingent on the premise that all sources of random variation within and between trials have been maintained to a minimum and that the same digestibility approach was used in population calibration and validation. Any bias in the original digestibility data will result in a bias in the digestibility prediction. Yakubu *al,* (2020) asserted that calibration is merely a mathematical method that teaches the NIRS system to precisely estimate the chemical constituents of plant samples founded on the link that exist between lab values and the near infrared reflectance spectroscopic spectra. It has been established that the disparities of digestibility that exist within trials is usually insignificant; but, one's inability to reduce all sources of random variant

will upsurge the scale of the errors. The important cause factors of variations include but not limited to sample transfer, determination of dry matter and the random analytical errors of weighing. Minson *et al.* (1983) reported orderly biases in depending on the near infrared reflectance spectroscopy to predict digestibility values of plant samples of different maturities, species and physical form. Though, a lot of the biases were because of changes in the sizes of the particle since log (1/R) was used. The authors did not expect NIRS be able to identify variations in digestibility caused by physical factors such as pelleting and grinding. The pelleting processes as well as heating perhaps changed the construction of the components of the forage, by so doing causing the differences in particle size. Pelleting is associated with increased feed intake and reduced digestibility because of its faster passage rate triggered by physical action. This analysis is supported by the data from Minson *et al.* (1983), who found that pelleting reduced DMD by 4.2 %, however near infrared reflectance spectroscopy predicted only a 1.3 % reduction.

Neutral Detergent Fibre content is considered a limiting factor for the estimation of the nutritive values of feedstuff and as such affects digestion of forages by livestock. Several studies (Yang *et al.*, 2017; Rushing *et al.*, 2016; Cheng *et al.*, 2015; Stubbs *et al.*, 2010) have been able to accurately predict NDF concentrations of forages with NIRS. In this particular study, the performance of the prediction models for NDF (R^2 = 0.86, RPD = 2.57) was considered successful. In consistent with the previous studies the prediction model for NDF was precise. In *Lolium multiforium,*Yang *et al.* (2017) constructed NIRS equations by using partial least square regression (PLS) which was able to accurately predict NDF (R^2v = 0.91, RPD = 3.44). By using multiple-linear regression (MLR) and principal component regression (PCR) Cheng *et al.* (2015) constructed NIRS models which successfully predicted NDF in *Leymus chinensis.* Similar results were found in Yang *et al.* (2017), however, interestingly, the coefficient of determination in the calibration process (R^2cal = 0.94) was higher than the validation (R^2val = 0.91) and according to Keim *et al.* (2015) could be due to the fact that the whole set of samples used are not evenly distributed in terms of composition. It is recommended that an appropriate sample set for near infrared reflectance spectroscopic analysis must be extensive and consistently dispersed in terms of their conformation and the extreme values

represented when separating the sample set to calibration and validation subset (Murray, 1988). In this particular study, the performance of the prediction model for NDF (R^2 = 0.86, RPD = 2.57) (Figure 4) was successful. In consistent with the previous studies the prediction model for NDF was precise.

5.2: Analytical Efficiency of NIRS and Routine Methods in Estimating the Composition of Groundnut Residues

This study presents a depth comparative analytical view on the CV % of nutrient concentrations obtained with routine methods against NIRS derived values, with a significant number of groundnut residues. Generally, NIR spectroscopy produced more consistent results than other techniques with crude protein (CP) measures showing the weakest relationship (CV% Routine: 18.18, CV% NIRS: 15.88). There was a higher correlation between the measured values of CP, IVOMD, and NDF and their corresponding routinely measured values (Table 4.6). NIRS is considered a secondary method because its evaluation is based on reference as compared to the routine methods; as a result cannot be more accurate than the methods with which it was based (Singh *et al.*, 2018). However, it can reduce analytical variations with better precision and consistency (Singh *et al.*, 2018) as seen with this result (Table 4.5). Several scientists working on a variety of crops, such as Soybean (Lee *et al.*, 2011), Brown Rice (Bagchi *et al.*, 2015), and strawberry (Jin, 1994), have demonstrated that NIR reflectance technique can be adopted successfully over conventional methods for various biochemical quality parameters. Interestingly, NIRS derived values for samples obtained from Zamfara State reported higher errors compared to the routine method. Crude protein (CP) had generally, inconsistent results with both NIRS and conventional method; although they had acceptable SEM. Davis *et al.* (2012) found that NIRS analysis of CP underestimated CP by up to 22 g/kg DM. Rosenthal (1973) stated that NIRS instruments may be used to promptly and precisely assess the DM, EE, and CP contents in grain and grain products using the NIR approach. In consistent to that, NIRS was able to analyse CP, IVOMD, and NDF levels in groundnut residues according to accepted analytical methods, as demonstrated by the findings of the present study.

The accuracy of NIRS was comparable to that of conventional techniques, with the added benefits of near-instantaneous, non-destructive, and chemical-free analysis.

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

In this study three optimal models (Y = 0.41+0.91X, Y = 1.07+0.98X and Y = -3.49+1.05X) were developed based on laboratory data to predict the CP, IVOMD and NDF respectively of sorghum (561 samples) and groundnut (1010 samples) residues. The samples were separated into two sets for each component: a larger set (of calibration) for the development of calibration equations and a smaller set (for validation) for which the derived equations were tested. The models produced higher relative coefficient of determination (CP = 0.89 and NDF = 0.86) but 0.30 for IVOMD and ratio of prediction deviation (RPD), CP = 14.47, IVOMD = 5.41and NDF = 12.95.

In conclusion, the development of calibration equations for CP, IVOMD and NDF of crop residues including sorghum and groundnut is possible. It is fast, non-destructive and chemical free but allows the prediction of values with high correlation.

6.2 Recommendations

- It is recommended that further studies should be carried out using locally acquired samples in other to increase the robustness of the models and usability in this region.

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