

Evaluation of Tigernut Waste for Production of Bioproducts

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Abstract:- Conversion of biomass into fuels and value added bioproducts is highly essential considering the menace of the pollution associated with fossil sourced fuels and Chemicals. Biomass conversion technology is an emerging innovation in the global energy sector. Besides the inherent advantages identified with biomass resources such as renewability, abundance and intoxicity, the resource is a means of sure way of growing national economy. In this study, assessment of monomeric sugars in tigernut chaff and its potential for the production of some bioproducts (biodiesel and bioethanol) was investigated. Tigernut chaff was defatted by solvent extraction method; the oil was trans-esterified using homogenous and heterogeneous catalyst obtained from waste chicken egg shell. The biodiesel production was optimized by varying the reaction time, catalyst type and concentration. The defatted chaff was hydrolysed using 4% H₂SO₄. The effect of particle size and reaction time on the release of monomeric sugars as well as sugar degradation products in the hydrolyzed sample was evaluated. Identification and quantification of the monomeric sugar was done using a dual wavelength UV-Visible spectrophotometer. The hydrolysed biomass was fermented to bioethanol using *Saccharomyces cerevisia* and the crude bioethanol was purified with Bio-CaO. From the results obtained, the optimum yield of the biodiesel was established at 0.9 w/v catalyst concentration, 60°C reaction temperature and 60min reaction time with potassium methoxide catalyst. The properties of the biodiesel obtained were consistent with the specifications of American Standard for Biodiesel Testing Materials (ASTM D 6751). The hydrolysis was found to be most effective at 1.11µm particle size and 120 min reaction time. The results revealed that tigernut waste can be used to produce biofuels and also as platform material for domestic and industrial purposes due to the concentration of monomeric sugar present it contains.

Keywords: Transesterification, Hydrolysis, Monomeric sugars and Bioethanol

I. INTRODUCTION

Generation of value-added products from biomass resources through biorefining processes is now an emerging trend in bioenergy research. As essential as this research thrust is to the overall benefits of global development in terms of provision of sustainable energy sources and gainful management of agricultural wastes the process of achieving this task is still challenging. The depletion of fossil fuel reserves and global concern about climate change have necessitate the need for alternative fuels and bio chemicals to replace the fossil-based products.

Tigernut (*Cyperus esculentus*) is a tuber, it belong to the family of *Cyperaceae* (Devries and Feuke, 1999). It is cultivated in Nigeria and other parts of West Africa, East Africa, parts of Europe, particularly Spain as well as in the Arabian Peninsula (Abaejoh *et al.*, 2006). Tigernut has been cultivated both as livestock feed and for human consumption. Proximate analysis showed that the nut contains the following: Moisture 3.50- 3.78%, Crude Protein 7.15 -9.70%, Lipid 32.13- 35.43%, Crude Fibre 6.26 -5.62% , Carbohydrates 46.99- 41.22%, Ash 3.97- 4.25% , Energy (kJ) 1343- 1511(Oladele and Aina, 2007). Tigernuts have excellent nutritional qualities with a fat composition similar to olives. Since the tubers contain 20 – 36% oil, *Cyperus esculentus* has been suggested as a potential crop for the production of biodiesel (Zhang *et al.*, 1996; Ugheoke *et al.*, 2007). One of the secondary waste products of *Cyperus esculentus* is the chaff. Tigernut chaff are potentially a rich source of fibre which can be converted to value added products after the milk and oil have been removed from the tubers.

Transesterification reaction of a fat or oil with an alcohol in the presence of catalyst (KOH, NaOH, NaOCH₃, etc.) to form fatty acid methyl esters and glycerol is the most widely used industrial method for production of biodiesel from oils/fats with lower alcohols in the presence of homogeneous or heterogeneous catalysts (Meher *et al.*, 2006). Several oils, both edible and non-edible such as sunflower oil (Arzamendi *et al.*, 2008), palm (Li and Xie, 2006), jatropha (Tiwari *et al.*, 2007), have been transesterified for biodiesel production, and it consists of a sequence of three consecutive reversible reactions where triglycerides are converted to diglycerides. Diglycerides are converted to monoglycerides followed by the conversion of monoglycerides to glycerol. In each steps an ester is produced and thus three ester molecules are produced from one molecules of triglyceride and three molecules of alcohol is required to complete the reaction, but some extra amount of alcohol should be added to the reaction to move the reaction in forward direction.

In view of the environmental pollution i.e odor from biodegradation of waste eggshell without any pretreatment a great deals of efforts have been performed on transforming the eggshell wastes to a valuable product (Tsai *et al.*, 2006). Chicken eggshell is one of the agricultural waste, it has been reported that it contains chemically (by weight) calcium carbonate (94%), magnesium carbonate (1%), calcium

phosphate (1%) and organic matter (4%) (Stadelmam, 2000). Calcium oxide (CaO) is one of the most promising alkaline earth metal oxides with high basicity, which is suitable for biodiesel production (Bal *et al.*, 2001; Tsai *et al.*, 2008; Syazwani *et al.*, 2015). Mussel shells, Mollusk shells and Chicken egg shell can be calcined to produce CaO used as a heterogeneous catalyst in biodiesel production (Nakatani *et al.*, 2009; Gogate and Golge, 2012; Rezaei *et al.*, 2013).

Colorimetric methods by Miller in 1959 with the use of dinitrosalicylic acid (DNS) assay, the orcinol-sulfuric acid method described by Scott and Melvin in 1953, has been the long aged methods used for monomeric sugar analysis. Douglas in 1981 developed a method called the phenol-sulfuric acid method. These methods cannot be used to quantify pentoses and hexoses separately. The Douglas developed another method which is the phloroglucinol-glacial acetic acid method, which was later used for the determination of pentose. Modern equipments are also available in this modern days for example high performance liquid chromatography (HPLC), gas chromatography (GC) and high performance anion exchange chromatography (HPAEC). Although HPLC and HPAEC are considered to be the best methods for sugars analysis, both qualitatively and quantitatively (Douglas, 1981) their cost is high because of the analytical columns, effluent reagents, and instrument maintenance. Recently, Chi *et al.* (2013), develop a simple, rapid, and low-cost method for sugar analysis which is the UV method. The aim of this research work is utilize tigernut chaff being discarded as waste for production of biodiesel and bioethanol and to determine the concentration of the monomeric sugar present in the chaff using a dual wavelength Uv-visible spectrophotometer.

II. MATERIALS AND METHODS

2.1 Sample preparation

Tiger nut chaff was collected from milk processing lab of Food Science Department Federal University of Technology, Akure, Ondo state Nigeria. The chaff was dried for 5 days in an oven at 70 °C, after which it was milled using Eurolex3025 grinder. The oil of the tigernut chaff was extracted using the method described by Barminas *et al.* (2001). Two hundred grams (200g) of the tigernut cake was transferred into the Soxhlet thimble carefully fixed to a round bottom flask. 500mL petroleum ether (b.p 40-60 °C) was poured inside the round-bottom flask and heated on a thermostatically controlled heating mantle (60°C) and refluxed until the oil was fully extracted. This procedure was repeated using n-hexane as extractant. The extraction was done in batches. After extraction, the defatted tigernut chaff was dried in a thermostatically-controlled oven at 60°C for 3h. The dried defatted tigernut chaff was subsequently milled using grinder. The extracted Oil was characterized with Fourier Transform Infra-red (FTIR).

2.2 Catalyst Preparation

Bio-CaO was prepared from egg shell as described by Achanai *et al.* (2013). The bio-catalyst was produced by a process called calcination-hydration-dehydration treatment of egg shells. Egg shells were washed thoroughly with tap water to remove any unwanted material adhered onto its surface, and then rinsed with distilled water. The washed egg shell was dried in a thermostatically controlled oven set at 105 °C for 24h. The dried egg shells was milled with blender (Eurolex 360) to reduce the particle size and there after calcined in a muffle furnace (LH.Thermo GmbH, LM 412.27, model DC021032) under static air conditions at 900°C for 3h to transform the calcium species in the shell into CaO particle. The calcined product was refluxed with water at 60°C for 6 h, filtered and then dried in a thermostatically controlled oven at 120 °C overnight. The solid product was dehydrated and calcined at 700°C for 3 h to change the hydroxide form to oxide form. The CaO produced was identified with XRD (Bruker AXS analyzer with a VANTEC-1 Detector) using a double-crystal wide-angle goniometry with the 2 θ scan from 10°–80° at a scan speed of 5° 2 θ /min in 0.05°/ 0.03°2 θ increments using CuK α radiation ($\lambda=0.15406\text{nm}$). The detected peak positions were compared with those of the International Center for Diffraction Data Standard (JCPDS) patterns to identify the crystalline phases.

2.3 Transesterification of the extracted oil

Transesterification reaction was carried out as described by Ugheoke *et al.* (2007). 20mL of the extracted tigernut oil was weighed. A laboratory scale reactor which was constructed with a 500mL conical flask, equipped with thermometer and mounted on a magnetic stirrer hotplate was used to carry out the reaction. The use of homogeneous catalyst (KOH & NaOH) and heterogeneous catalyst (Bio-CaO) was employed. 10mL of oil was poured into the lab scale reactor and heated to 45 °C to improve the miscibility of oil with methanol. Potassium hydroxide (KOH) and sodium hydroxide (NaOH) were prepared in the concentration range of 0.3% w/v, 0.5% w/v, 0.7% w/v, 0.9% w/v, 1.1% w/v, and 1.3% w/v. The potassium methoxide and sodium methoxide were introduced gently into the heated oil inside the reactor, the entire content was brought to a temperature 60°C at a heating rate of 20 °C/min, then held at this temperature at different time interval (30, 60, 90, 120 and 150 min) and agitation speed of 1500rpm. CaO derived from the egg shell was weighed in 5g, 10g, 20g, 30g and 40g and dissolved in methanol. The calcium methoxide was introduced into the oil inside the reactor at temperature 60°C at different time interval of 90min, 180min, 270min, and 360min. Agitation speed of 1500rpm was set for the reaction. The reaction product was left overnight in a separatory funnel so as to separate glycerol from the biodiesel. The collected biodiesel was washed with acetic acid and further washed thoroughly with distilled water until tested neutral to litmus paper to remove impurities. The percentage yield of the biodiesel was calculated and characterized with FTIR for structural determination. Fuel properties of derived

biodiesel such as viscosity, specific gravity and refractive index were carried out as following the procedure described by AOAC (2001).

2.4 Optimization and Acid hydrolysis of Tigernut Chaff

One hundred grams (100g) of tigernut chaff was weighed in accurately and in triplicate, it was pretreated using Screening and ultrasonication method: the milled cobs were screened using U.S standard sieve No.20 using different particle sizes (1.11 μ m, 1.14 μ m and 1.17 μ m) and added to 1L of water respectively so as to obtain a slurry concentrations mL/g. Each of the samples was poured into troughs and then homogenized thoroughly by stirring. Acid hydrolysis was carried out using a laboratory scale reactor built with pressure pot and operated under the following reaction conditions: Temperature (120 °C), acid concentration (4 % w/v) and contact time (60 min and 120 min), respectively using heat stirrer under pressure. After the reaction time was over, the hydrolysed sample was cooled, tested with litmus paper, the pH was maintained at pH 6 after which the hydrolysate was filtered and kept frozen for analysis (Adedara *et al.*, 2020). The surface morphology of the pre and post hydrolysed tigernut chaff was determined with SEM (Tescam 600 Scanning Electron Microscopy (FEI Company Hillsboro) and FTIR analysis was also carried out on the pre and post hydrolysed cob with Perkin Elmer Fourier Transform Infra-red (FTIR) so as to monitor the effect of the hydrolysis. A colored reagent was prepared for U.V measurement method described by Chi *et al.*, 2013. Identification and quantification of the monomeric sugars (glucose, xylose, and arabinose) was carried out using a dual wavelength Uv-visible spectrophotometer (PerkinElmer Lambda XLS).

2.5 Preparation of colour reagent

Coloured reagent was prepared was prepared by dissolving 2g of phloroglucinol in 110mL of glacial acetic acid followed by adding 30mL of anhydrous ethanol and 2mL of concentrated hydrochloric acid. 5 sets of standards were prepared. 1mL each of sugar standards (glucose, xylose and Arabinose), degradation products standards (Hydromethylfufural and fufural) and the hydrolysed samples were mixed with 15mL of colour reagent in a 50mL test tube it was then placed in a boiling water bath for 20 min, the mixture was cooled with cold water for 6 min after which spectra scanning was conducted for the standards and the samples in the wave length range of 300nm to 700nm. Thereference was mixture of distilled water and colour reagent under the same condition as above.

2.6 Fermentation of the hydrolysed sample

Fermentation was carried out as described by Ogunsuyi and Badiru. (2016) and Adedara *et al.* (2020). The hydrolyate obtained was fermented in a 2 L capacity fermentation tank using brewers yeast (*saccharomyces cerevisea*), the fermented broth was distilled at temperature of 80°C using a simple distillation set-up and further purified by refluxing it

for 3hr with Bio-Cao obtained from calcined chicken egg shells after which it was further distilled. The bioethanol was characterized with FTIR and GCMS.

III .RESULTS AND DISCUSSION

Table 1 shows the result of the physicochemical properties of the extracted Tigernut chaff oil. The refractive index, viscosity, specific gravity, moisture content, acid value, saponification value were consistent with those obtained by Sabinus (2012) and ASTM standard values for oil used for biodiesel.

Table1: Physicochemical Properties of the Extracted Tigernut Chaff Oil

RESULTS PARAMETER	RESULT(Mean± SD)
Specific Gravity	0.885±1.41
Viscosity (centipoise)	31.195±0.01
Moisture Content (%)	1.25±0.35
Colour (425nm)	Yellow
Refractive Index	1.4665±0.00
Acid Value (mgKOH/g)	1.990±0.01
Free fatty acid (mgKOH/g)	0.995±0.01
Saponification Value (mg/g)	175.500± 2.53

3.1 X-ray Diffraction (XRD) Analysis

Figure 1 show the conversion of chicken egg shells into bio-catalyst through calcination process and the XRD pattern of the calcined shell. Sharp XRD reflection at 111 and 200 orientation implies that the material was well crystallized. Prominent peaks can be seen at 2 θ equal to 31.609 and 34.111. Comparing the XRD peaks with the standard peak patterns of JCPDS (Joint Committee on Powder Diffraction Standards) files, the peak at 31.6 (111) corresponds to calcium oxide (CaO) or the lime of the face-centered cubic form (Fm-3m; JCPDS No. 01-077-2010) and the peak at 34.194 (101), correspond to calcium hydroxide (Ca(OH)₂ or portlandite) of the hexagonal form (P-3m1; JCPDS No. 01-087-0673). There was emergence Ca (OH)₂ which according to Tangboriboon *et al.*, (2012) could be due to the absorption of moisture during storage. The result indicated that the egg shell was converted to CaO in the course of the reaction.

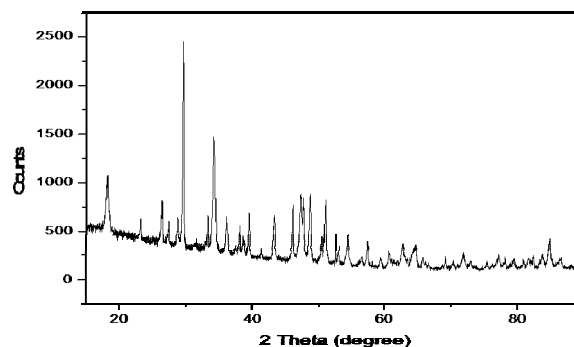


Fig 1: XRD pattern of calcined chicken egg shell.

3.2 FTIR Analysis of the Extracted Oil and Biodiesel Produced

Figures 2 and 3 shows the FTIR spectra of the crude extract of tigernut chaff oil and the transesterified oil. The difference noted in the structural compositions of the extracted and the transesterified oils could be attributed to the variation in the ester group of the oils. According to Soereset *al.*(2008), the oils are chemically similar in the region of 1800 cm^{-1} - 1700 cm^{-1} but different in the finger print region of 1500 cm^{-1} - 900 cm^{-1} . The ester group is described as $\text{R}^1\text{-C}(\text{OCH}_3)=\text{O}$ in biodiesel. R^1 represents long chains of hydrocarbons.

From the spectra below, the major difference between the oil and biodiesel is seen with emergence a new signal at 1437.19 cm^{-1} with its deformation vibration which is the methyl ester group. The next visible transformation was the broad signal at 1163.38 cm^{-1} in edible oil separated into two concrete signals at 1171 cm^{-1} and 1197 cm^{-1} .

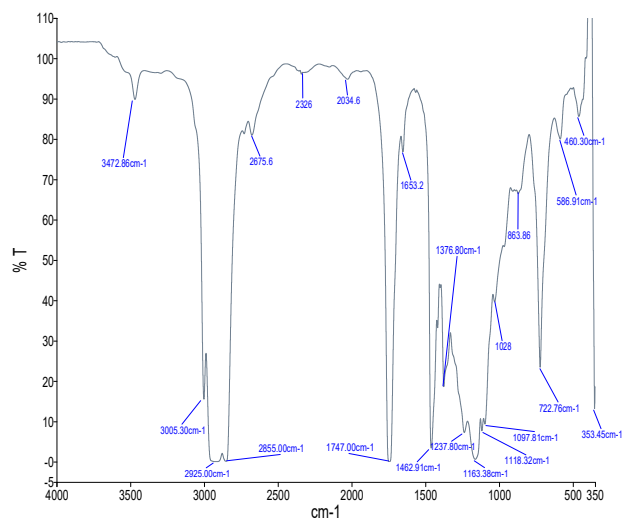


Fig 2: FTIR spectrum of the crude tigernut chaff oil

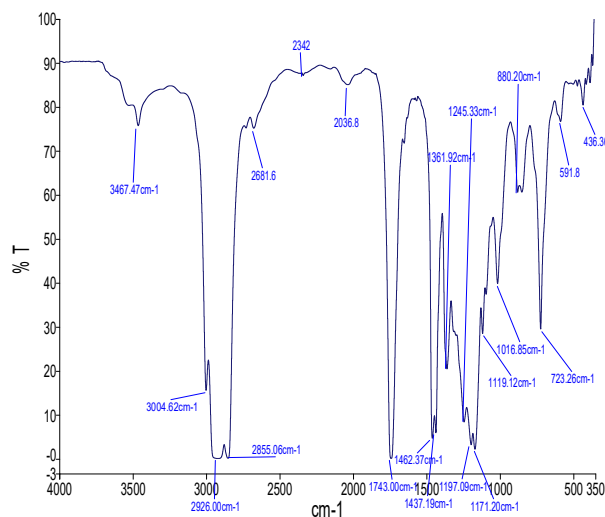


Fig 3: FTIR spectrum of tigernut derived biodiesel

3.3 Fuel Properties of the Biodiesel Produced

Table 2 shows the fuel properties of the derived biodiesel from tigernut chaff oil. The viscosity of the biodiesel obtained fall within the range of ASTM standard (4-6Cent) specified for biodiesel as presented on the table, this implies that the derived biodiesel will combust completely, atomize effectively in engines without any leakage in the injection pump plunger. The specific gravity of the derived biodiesel (0.823, 0.820 and 0.800) was comparable with 0.84 reported by Enweremadu and Alamu (2010) for biodiesel produced from shea nut.

The acid values obtained were 0.72, 0.74 and 0.72mgKOH/g for NaOMe, KOMe and Bio CaO, respectively. The acid values obtained were comparatively lower than 2.30 reported for crude tigernut biodiesel by Aldo *et al.* (2012), this implies that the biodiesel will not cause corrosion in the diesel engine. The refractive indices were similar to those reported by Sanjay *et al.* (2014) for *Gmelinaarborea* and *Tabernaemontanadivaricata* seeds which were 1.450 and 1.446, respectively. In addition, the refractive indices of the derived biodiesel (1.456, 1.448 and 1.482) were consistent with standard values (1.245-1.675). The cloud points (5.8°C , 6.0°C and 5.7°C) were also consistent with standard values. The flash points of the derived biodiesel were quite comparable with 6.5°C reported for biodiesel produced from shea nut butter by Enweremadu and Alamu (2010). The moisture content obtained compared favorably with 0.05max of ASTM standard for biodiesel and convectional diesel. It can be seen that the fuel properties of the tigernut biodiesel are within the range of recommended values for fuel as described by Chongkhong *et al.* (2007). The results also agree with the results obtained by Achanai *et al.* (2013) for *cyperus tuber* oil.

Table 2: Fuel Properties of the Biodiesel Produced

Parameters	KOH	NaOH	BIO-CaO	ASTM BIODIESEL	CONVENTIONAL DIESEL
Viscosity ($\text{mm}^2\text{ s}$)	$4.74^{\pm} \pm 0.08$	$4.72^{\pm} \pm 0.07$	$4.84^{\pm} \pm 0.03$	1.9-6.0	4-6.0
Specific gravity	$0.823^{\pm} \pm 0.01$	$0.820^{\pm} \pm 0.02$	$0.800^{\pm} \pm 0.00$	NA	0.875-0.9
Flash point ($^{\circ}\text{C}$)	$173^{\pm} \pm 0.01$	$171^{\text{ab}} \pm 0.03$	$170^{\text{b}} \pm 0.01$	130minimum	60-80
Cloud point ($^{\circ}\text{C}$)	$5.8^{\text{a}} \pm 0.58$	$6.00^{\pm} \pm 0.08$	$5.7^{\text{a}} \pm 0.01$	-3-15	-35-5
Refractive Index	$1.456^{\text{a}} \pm 0.01$	$1.448^{\text{a}} \pm 0.01$	$1.482^{\text{b}} \pm 0.01$	1.245-1.675	NA
Acid Value(mEq/g)	$0.72^{\text{a}} \pm 0.01$	$0.70^{\text{a}} \pm 0.01$	$0.72^{\text{a}} \pm 0.01$	0.80 max	0.50
Moisture (%)	$0.03^{\text{b}} \pm 0.01$	$0.032^{\text{b}} \pm 0.01$	$0.04^{\text{a}} \pm 0.01$	0.05 max	0.05max

3.4 Effect of Catalyst Concentration on Percentage Yield of Biodiesel

Figure 4 depicts the effect of homogenous catalyst concentration on the biodiesel yield. The percentage yield of the derived biodiesel increased steadily from 47% to 88% (KOMe) and from 33% to 86% (NaOMe) as the catalysts

concentration increased from 0.3% w/v to 0.9% w/v. Thereafter, a decrease was observed when the catalyst concentration increased above 0.9% w/v. Therefore, it was obvious that too high concentration of homogenous catalyst favours formation of soap during transesterification reaction thereby leading to a decrease in volume of the biodiesel produced. The yield of biodiesel obtained was consistent with 85% obtained by Darnoko (1999) for palm oil and comparably higher than 67% obtained by Ugheokeet *al.* (2007).

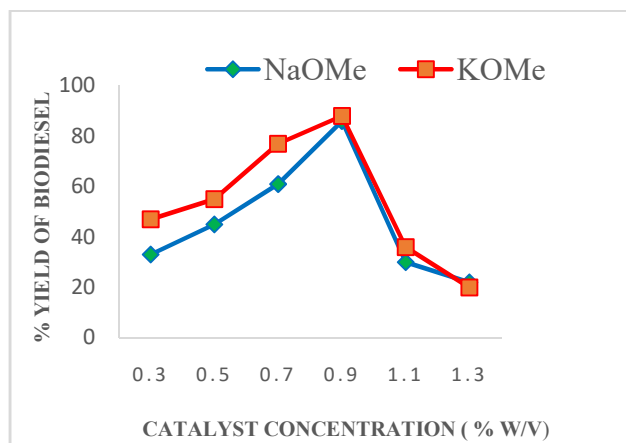


Fig 4: Effect of homogenous catalyst concentration on the percentage yield of biodiesel

Figure 5 shows the effect of heterogeneous catalyst on biodiesel yield. The biodiesel yield increased from 22% to 81% with catalyst concentration of 5% w/v to 20% w/v and decreased to 46% with catalyst concentration of 40% w/v. The loadings of 30% w/v and 40% w/v created catalyst accumulation on the wall of the glass reactor, creating diffusion problems during in the course of the reaction decreasing the catalyst activity (Buasriet *al.*, 2012). The result obtained is consistent with 92.92 and 94.49% obtained by Achanaiet *al.* (2013) for transesterification of palm oil with duck and chicken egg shells bio-catalysts respectively.

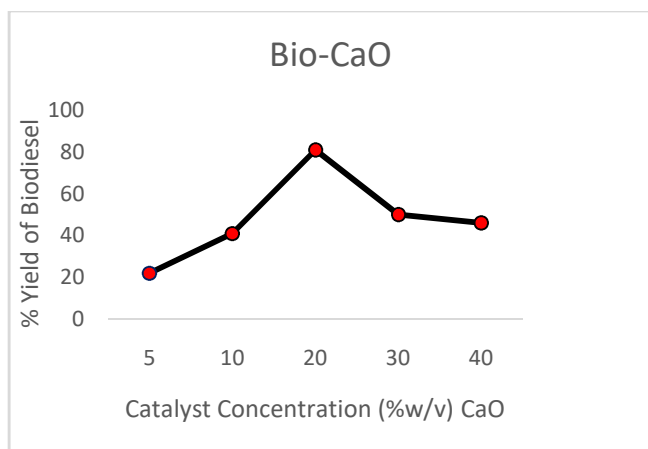


Fig 5: Effect of heterogeneous catalyst concentration on the percentage yield of biodiesel

3.5 Effect of Reaction Time on the Percentage Yield of Biodiesel

Figure 6 shows the effect of reaction time on biodiesel yield. A gradual increase in the percentage yield of the biodiesel was noted as the reaction time was raised from 30 min to 90 min for the homogenous catalyst (NaOMe and KOMe) and from 90 min to 270 min for heterogeneous catalyst (Bio-CaO). Thereafter, the biodiesel yield decreased as the contact time was raised above 90 min and 270 min for the homogenous and heterogeneous catalysts respectively. This is due to the fact that transesterification reaction between oil and alcohol becomes reversible when the reaction proceeds at longer time (Buasri *et al.*, 2012; Santana *et al.*, 2012).

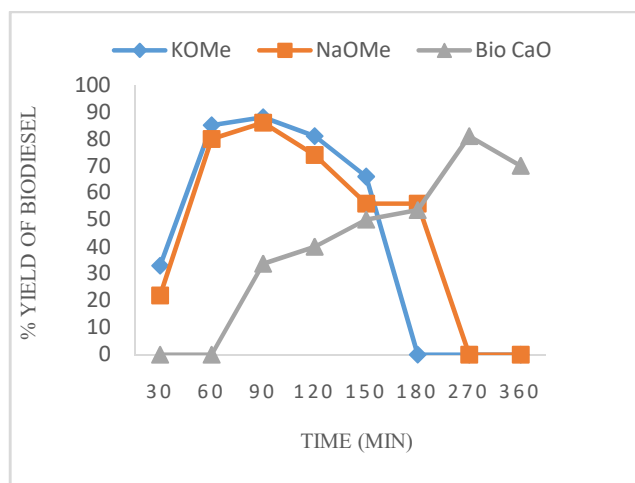


Fig 6: Effect reaction time on the percentage yield of biodiesel.

3.6 FTIR Analysis of Pre and Post Hydrolysed Biomass

The absorption bands shown at around 3300 cm^{-1} indicate OH absorption frequency with strong and intense band in post hydrolysed sample a than in pre hydrolysed (Figures 7) these bands can be attributed to OH absorption frequencies in the monomeric sugars and lignin. The neighboring peaks around 2800 cm^{-1} in post hydrolysed sample and could be due to stretching vibrations of C-H of CHO, this could be because of the release of arabinose and xylose during the hydrolysis process. The absorption bands around 1750 cm^{-1} may be due to carbonyl specific absorptions (C=O). The bands at around 1630 cm^{-1} indicates the presence of C=C stretching vibration of alkanes which is present in pre and post hydrolyzed biomass but more sharp and intense in post hydrolysed biomass. The bands at around 1400 and 1300 cm^{-1} correspond to C-H vibration of CH_3 , it was intense in the post hydrolysed biomass than in the pre hydrolysed biomass. Absorption band with great intensity around 1000 - 1100 was observed in post hydrolysed biomass which correspond to C-O stretch

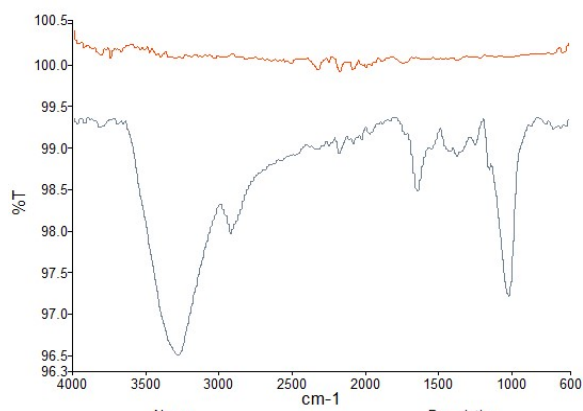


Fig 7: FTIR Spectra of Pre and Post Hydrolyzed Tigernut Chaff

Pre hydrolysed tigernut chaff. Pre hydrolysed tigernut chaff

Plates 1 and Plate 2 reveal the surface morphologies of tigernut chaff before hydrolysis and after hydrolysis. The surface of the sample shows a well-organized structure before hydrolysis and a disorganized structure after hydrolysis. The result indicates that the hydrolysis technique used was efficient.



Plate 1: SEM images of Tigernut chaff before hydrolysis

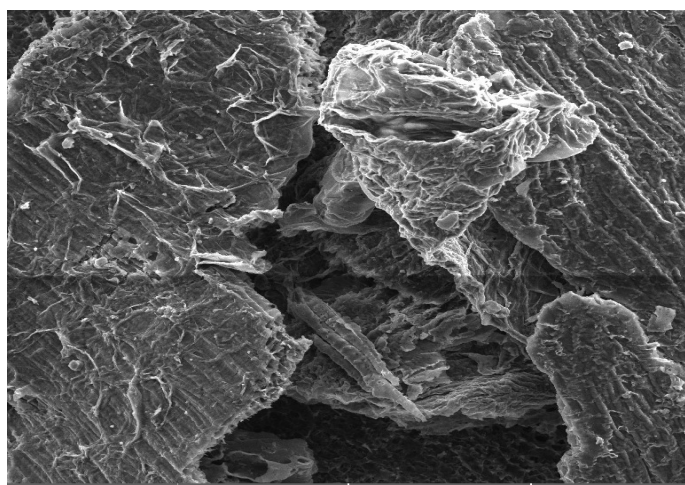


Plate 2: SEM images of Tigernut chaff after hydrolysis

3.8 Effect of Particle Size on Sugar Level of the Hydrolysate

Figure 8 shows the effects of different particle sizes (1.11 μm , 1.14 μm and 1.17 μm) on sugar level of tigernut chaff with respect to time. The highest concentration of monomeric sugars were obtained with the smallest particle size of 1.11 μm . This implies that an increase in the surface area of the biomass sample enhanced the release of the monomeric sugars. As the particle size increased from 1.11 μm to 1.17 μm at 120°C and 120min, glucose concentration decreased from 13.64 g/L to 9.36 g/L, xylose decreased from 9.36 g/L to 8.75 g/L and arabinose decreased from 7.92 g/L to 6.86 g/L. The concentration of degradation products such as furfural increased from 0.03 g/L to 0.05 g/L upon an increase in the particle size of the biomass. However, the sugar concentrations obtained in this study were higher than those reported by Gamezet *et al.* (2006) for sugarcane baggase.

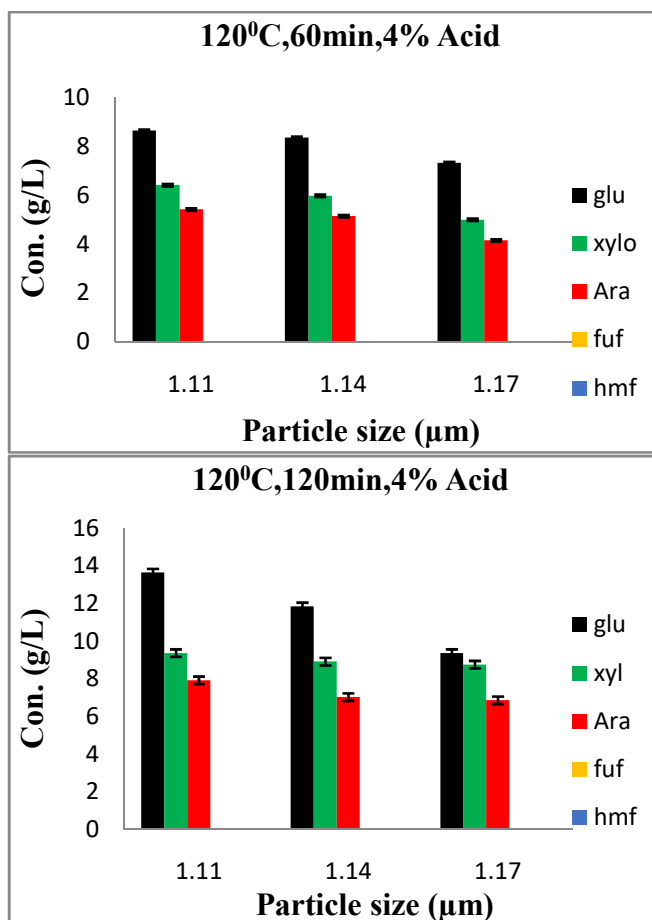


Fig 8: Effect of particle size on sugar level of Tiger nut Chaff Hydrolysate

3.9 Effect of Reaction Time on Sugar Level of the hydrolysate.

Figure 9 shows the effect of time on sugar level. The total concentration of all the monomeric sugars increased when a longer reaction time was used. Additionally, when the reaction was allowed to proceed at a reaction time beyond 120min, the concentration of glucose diminished. However,

remarkable increase in the concentration of xylose and arabinose was noticed while the degradation product (HMF) increased slightly under these higher operating conditions.

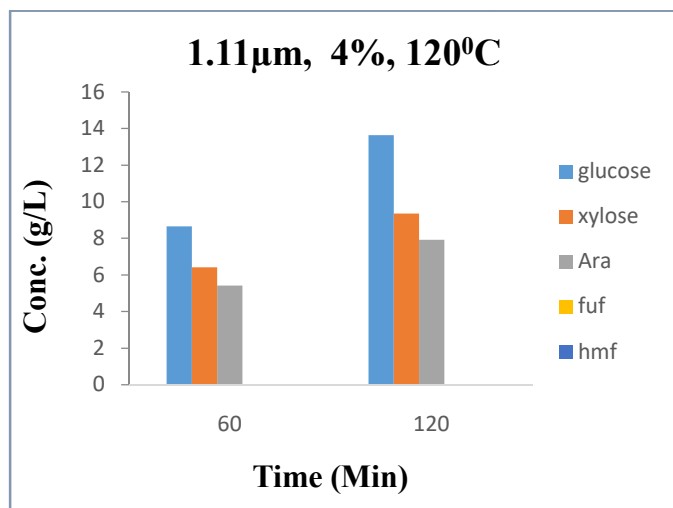


Fig 9: Effect of reaction time on sugar level of tigernutchaff hydrolysate

3.10 FTIR Analysis of Bioethanol

Figure 10 and 11 depicts the FTIR and GCMS spectra obtained from the bioethanol produced with absorption band in the following regions: 3328.78 cm^{-1} (O-H stretch of alcohol), 2973.16 cm^{-1} (C-H stretch of alkane) and 1067.11 cm^{-1} , 1044.87 cm^{-1} (C-O stretch of alcohol). Figure 11 revealed the retention time, molecular weight and the structure of the bioethanol produced, the structure obtained from the spectrum confirmed that the compound produced was ethanol

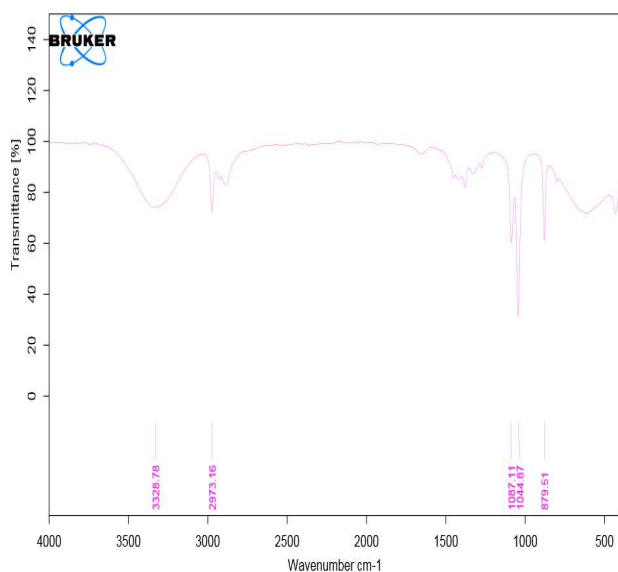


Fig 10: FTIR Spectrum of Purified Bioethanol obtained from Tigernut Chaff.

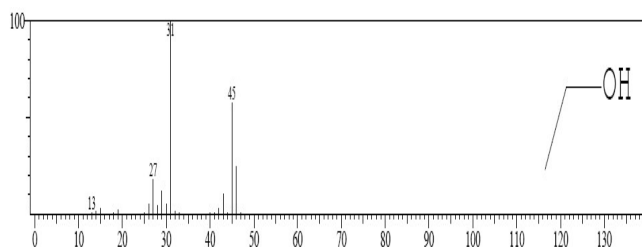
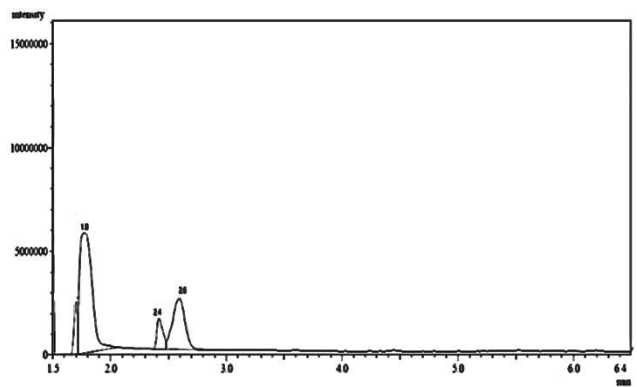


Fig 11: GCMS Spectrum of Purified Bioethanol obtained from Tigernut Chaff.

IV CONCLUSION

This study confirmed the suitability of the oil extracted from tigernut chaff for the production of biodiesel considering the quality and the physicochemical properties which conformed to standard values of oil-feed for biodiesel as specified by ASTM. The conversion process of the crude oil to biodiesel was established to be optimum under experiment conditions of 0.9% catalyst concentration (NaOMe), 90min reaction time and reaction temperature of 60°C during which 88% of the oil extract was converted into biodiesel. Also the Bio-CaO efficiently converted the crude oil into biodiesel with a yield of 81%, thus confirming the effectiveness of the in-house catalyst for transesterification process. The fuel properties of the derived biodiesel also conform to ASTM standard values, which implies that the derived biodiesel can conveniently run in diesel engines like their petroleum-based counterparts. The results from this study indicated that the best condition for the release of monomeric sugars (glucose, xylose and arabinose) from tigernut chaff were $1.11\mu\text{m}$ particle size and 120 min reaction time. The availability of the biomass coupled with the high yield of the bioethanol product (85%) derived from the biomass proved the resourcefulness of the biomass as potential feedstock for bio-based products such as biofuels.

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