

JOURNAL OF ENGINEERING, EMERGING TECHNOLOGIES AND APPLIED SCIENCES (JEETAS)

A PUBLICATION OF THE FACULTY OF ENGINEERING, NIGER DELTA UNIVERSITY

NOVEMBER 2023

VOLUME 01

ISSUE 02

ISSN: 1116 - 4514

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Soxhlet Extraction of Oil from Monkey Sugarcane (*Costus afer*) Leaves

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Abstract:

Bio-oil is a mixture of organic components obtained from biomass components such as esters and amongst others, Soxhlet extraction is the most common method of bio-oil production from various feedstock. The aim of this study is to extract the bio-oil of monkey sugarcane leaves by a soxhlet extractor and examine the physicochemical properties. The average percentage of oil yield was 5.2% and a total weight of 26.09g of bio oil was extracted from 500g of dried *Costus afer* leaves. The physicochemical properties studied and results obtained using ASTM and other standards are; boiling point (125°C), acid value (0.9mgKOH/g), FFA (0.49mgKOH/g), water content (0.53%), saponification value (146.3mgKOH/g), flash point 187°C and pour point (5°C). The bio-oil produced has comparable properties with that produced from other agricultural products and so could serve as a good feedstock for biodiesel production.

Keywords — Monkey sugarcane, agbodou, soxhlet apparatus, oil extraction, methanol

I. INTRODUCTION

Bio-oil can be extracted from oleaginous plants, algae, municipal wastes or other organisms via different methods such as pyrolysis, soxhlet extraction method etc. which can be further used as feedstock for biodiesel production. Bio-oil is a complex mixture of organic components obtained from biomass components such as esters, alcohols, organic acids etc. The process of bio-oil extraction by the use of a soxhlet apparatus is one of the most conventional methods for vegetable oil extraction from materials that are oleaginous in nature (Ratna et al., 2015). Dried and particle size reduced samples which are weighed into a thimble in contact with the solvent for the oil to be moved from the solid matrix to the fluid medium. The solvent selection is based on the ultimate leaching properties of the sample. Oliveira et al., (2013) presented a report on the analysis of the mass transfer that takes place during solvent extraction in a packed column in a related study.

Franz von Soxhlet in 1879 invented the soxhlet extraction apparatus. Originally, it was designed to extract lipids from solid materials. This apparatus is normally used in situations where the solubility of the compound is limited in a particular solvent. Unmonitored operations while recycling a small amount of solvent to dissolve a larger amount of material is possible in this method. This apparatus has a percolator for solvent circulation, thimble that retains the solid to be extracted, siphon mechanism, which periodically empties the thimble. Much work has been done by different researchers on the use of soxhlet apparatus for the extraction of bio oil from different feedstocks. However, the main objective of this study is to examine the extraction process of bio oil from monkey sugarcane (*Costus afer*) leaves using the soxhlet extraction method to obtain the rate of oil yield and investigate the physicochemical properties of the bio-oil.

II. MATERIALS AND METHODS

A. Methods

The materials used in this study include; Fresh monkey sugarcane leaves, stopwatch, attrition mill, sieve shaker, sieves, soxhlet extraction apparatus, rotary evaporator, beakers, funnel, weight balance, plastic, distilled water, measuring cylinders, conical flask, spatula and, oven, methanol etc.

1. **Monkey Sugarcane Leaves (Costus afer):**

The leaves of monkey sugarcane were harvested from a nearby bush at Tantua in Amassoma community in Southern Ijaw Local Government Area, Bayelsa State, Nigeria. Amassoma is an Ijaw speaking community situated in Lat. 4.97oN, Long 6.11oE, and 79 meters above sea level. The samples were taken to the Processing Laboratory of Agricultural and Environmental Engineering, cleaned and oven dried at an average relative humidity of 65% and an average temperature of 60°C for a period of six (6) hour in the Processing Laboratory of the Agricultural and Environmental Engineering Department, Niger Delta University, Wilberforce Island Bayelsa State. The crispy leaves were then grinded and stored in air tight polyethene bags to prevent the absorption of moisture. Plate 1 presents the fresh monkey sugarcanes, Plate 2 and 3 present the grinding process and sieved monkey sugarcane leaves respectively.



Figure 1: Monkey sugarcane



Figure 2: Dry sieved monkey sugarcane leaves

2. **Extraction of Bio-Oil:** In this work, the extraction of the bio-oil was done with the soxhlet extraction apparatus from the monkey sugarcane (*Costus afer*) leaves, using methanol as solvent. In a soxhlet apparatus, the extractor thimble is fitted in between a round bottom flask at the bottom and a bulb condenser at the top. The monkey sugarcane leaves were properly dried and ground with the attrition mill and sieved with the 75-micron British Standard sieve size and kept at air tight environment (i.e. inside a nylon paper) to avoid moisture absorption before used for the study. The properly processed *costus afer* samples were used for the extraction. A dried thimble that was free from any form of visible impurities was weighed and then, 100 g of the sample was added into the thimble and weighed again. The 500 ml round bottomed flask which was also free from any form of visible impurities was also weighed and then, methanol was used to fill the 500 ml round bottomed flask up to 250ml of the flask, thereby making the feed to solvent ratio to be in line with the method used by Nibe et al., (2023).

The soxhlet extractor with a reflux condenser was fitted up and the heat source was adjusted to about 80oC so that the solvent boils gently and, this was allowed to siphon and left for about 30 minutes to 1 hour. The condenser was detached and the thimble was removed. The methanol was also allowed to siphon over the barrel before the condenser was detached. Then, a rotary evaporator was used to dry the flask that contains the oil at 80oC for a period of 30 seconds and cooled in a desiccator and then weighed. The thimble was placed in a beaker in an oven at 50oC and dried to constant weight with the sample (i.e. monkey sugarcane leaves powder), it was then cooled in a desiccator and weighed as was applied by Adegbe et al., (2016). The extraction was performed in triplicate using the soxhlet extraction apparatus, and the mean values and standard deviations were calculated based on equation (1) as presented by Ratna et al., (2015) to obtain the percentage oil extracted after the process of extraction.

$$\text{Percentage oil yield} = \frac{w_2}{w_1} \times 100 \quad (1)$$

where,

w_1 = weight of monkey sugarcane leave powder in grams

w_2 = weight of the oil produced in grams



Figure 3: Setting up Soxhlet extraction apparatus



Figure 4: Bio-oil extraction



Figure 5: Rotary evaporator for oil separation



Figure 6: Extracted monkey sugarcane bio-oil

III. RESULTS AND DISCUSSION

A. Bio Oil Extraction

Based on the experimentation for the bio oil extraction using the soxhlet apparatus as was also adopted by Nibe et al., (2023), the calculations of the oil yield from the monkey sugarcane leaves is presented in Table 4.1, where the mass of the sample collected per run, the mass of the dry sample after extraction, and the oil yield from each run of the sample denoted with M1 – M5. An average of 5.22g was obtained as mass of the oil yield and the average percentage of oil yield was 5.2%. Also, the average mass of the oven dried waste of the sample after extraction was 94.74g. A total volume of 26.09g of the bio oil was extracted from 500g of dried monkey sugarcane leaves as shown in Table 4.1. However, a total volume of 1000ml of the bio oil was extracted from the study.

Table 1: Bio-oil extraction result from monkey sugarcane (*Costus afer*) leaves

Sample ID	Weight of sample (g)	Weight of dry waste (g)	Weight of oil yield (g)	Percentage oil yield (%)
M ₁	100	94.58	5.23	5.2
M ₂	100	94.89	5.11	5.1
M ₃	100	94.44	5.54	5.5
M ₄	100	94.68	5.32	5.3
M ₅	100	95.11	4.89	4.9
Total	500	473.7	26.09	26.0
Mean	100	94.74	5.22	5.2

B. Physiochemical Properties of Monkey Sugarcane (*Costus Afer*) Bio Oil

The quality of the bio-oil produced from the monkey sugarcane leaves was analyzed by evaluating physical and chemical properties of the bio oil are discussed and presented in Table 2. The boiling point which is an important property of a bio oil selection for biodiesel production was investigated in this study. The boiling point for the production of the monkey sugarcane leaves bio oil

was 125°C as presented in Table 2. This value of the boiling point is lower than the boiling points obtained by Zahir et al., (2014) for corn oil and mustard oil which were reported as 140 and 170°C respectively. Due to the fact that oils are denser than water, it is therefore expected that the boiling point of monkey sugarcane is higher than that of water (100°C) which is in line with the reports presented by Zahir et al., (2014) in a similar study on corn oil.

Acid value as a property of bio oil for biodiesel production cannot be overemphasized. The volume of catalyst used in the transesterification reaction is dependent on the acid value of the bio oil. Hence, higher acid value will require more catalyst which will also increase the cost of biodiesel production process. The presence of high acid values in bio oil can lead to production of biodiesel with undesirable properties. The acidic impurities may affect the combination characteristics of biodiesel, which can contribute to engine deposit and enhance the corrosion of fuel system components. The acid value of the monkey sugarcane bio oil as presented in Table 2 is 0.9mgKOH/g which is lower, compared to bio oil from other agro-products such palm oil (4.217mgKOH/g) and tallow oil (1.113mgKOH/g).

Free fatty acid (FFA) value obtained for the monkey sugarcane leaves bio oil was 0.49mgKOH/g. Oils with high levels of FFA are more susceptible to oxidative aging, they become rancid more quickly. Patrick, (2013) reported that, base catalysed transesterification reaction requires low percentage of FFA of less than 1% for materials to be used as feedstock for biodiesel production.

Also the result of average percentage moisture content of the monkey sugarcane bio oil was 0.53%. This is also in conformity with the result obtained by other researchers in literature. High content of moisture in the bio oil can contribute to lower density, combustion reaction rate and corrosion tendencies. The result of the density test on the monkey sugarcane bio oil is 0.918 g/cm³. The density is a property that is normally used together with other physical properties to characterize the bio oil of a particular feedstock.

Saponification value describes the average molecular weight of the fatty acids present in specimen as triglycerides. Higher saponification value depicts lower average length of fatty acids, the higher the mean molecular weight of triglycerides and vice-versa. This study showed to have a saponification value of 146.3mgKOH/g. This value was lower compared to corn oil (153.8mgKOH/g) but higher than mustard oil (125.6mgKOH/g) as reported by Zahir et al., (2014).

Iodine numbers are often used to determine the amount of unsaturation in fats, oils and waxes. The iodine value obtained in this study was 11.4g. This value is lower than the value for corn oil (15.96g) but higher than that of mustard oil (8.10g) obtained by Zahir et al., (2014). Peroxide value gives the initial evidence of rancidity in unsaturated fats and oils. This study obtained a peroxide value of 0.35meq/kg. This value is seen to be higher than the peroxide value obtained by Zahir et al., (2014) for corn oil (0.162meq/kg) but lesser than the value for mustard oil (0.83meq/kg).

Also, the information on the ash content of a bio oil used for biodiesel production is very important because, the high ash content of the bio oil can contribute to the corrosion of the processing equipment used in biodiesel production. In this study, the ash content of the monkey sugarcane bio oil was obtained as 0.003%.

The flash point of the monkey sugarcane bio oil was 187oC and this result is lower compared to the result obtained by Demirbas, (2008) who used vegetable cooking oil as a feedstock and got a flash point of 212oC for the bio oil. This means that, the bio oil is less flammable, at lower temperature and is good and safe for handling operations.

The pour point of the monkey sugarcane bio oil was also investigated and the result was 5oC. The pour point of this study was also lower than that obtained by Demirbas, (2008) for waste cooking oil which was 10oC. Since, the pour point is the temperature below which a liquid loses its flow characteristics, the bio oils obtained in this study can therefore be suitable for use even in cold countries.

Table 2: Physicochemical Properties of the Bio-oil

S/No	Oil Properties	Value	ASTM
1.	Boiling point (°C)	125	
2.	Density (g/cm ³)	0.918	---
3.	Ash content (%)	0.003	D482
4.	Acid value (mgKOH/g)	0.90	D664
5.	Water content (%)	0.53	
6.	FFA (mg KOH/g)	0.49	
7	Saponification value (mgKOH/g)	146.3	
8.	Iodine value (g)	11.4	
8.	Peroxide value (meq/kg)	0.35	
9.	Flash point (°C)	187	D93
10.	Pour point	5	D97
11.	Kinematic Viscosity @ 40°C (mm ² /s)	41.5	D445
12	Ph	7.3	

C. Viscosity of Monkey Sugarcane Bio Oil

Viscosity is one of the fundamental qualities that characterize flow behavior of a fluid (Burubai and Amber, 2013). It is a measure of a fluid’s ability to resist motion when a shearing stress is applied. The viscosity of the bio oil at ambient temperature, was tested using a rotary viscometer (BLS-8S model) at different spinning; 6, 12, 30, and 60 rpm, and the result is presented in milli-Pascal seconds as shown in Figure 7. It was observed that, the viscosity of the oil reduced with the increase in r.p.m of the spindle of the rotary viscometer. Hence, the oil contained can be described as a Non-Newtonian fluid (Burubai and Amber, 2013), specifically a shear-thinning fluid otherwise called a dilatant. They obey the power law model. Also, Figure 7 shows a curve for predicting the viscosity of the oil using power equation from the relationship between rpm and viscosity; the power equation on the graph can be used to predict the viscosity of the oil in mPa.s unit.

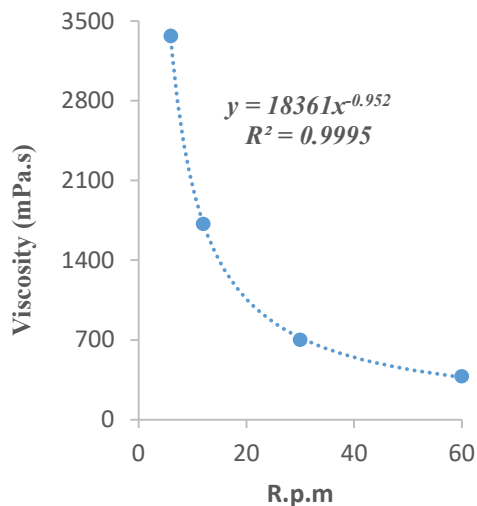


Figure 7: Relationship between rpm and viscosity

IV. CONCLUSION

The soxhlet extractor is a common laboratory device for the extraction of bio-oils from oleaginous materials. Application of this method for oil extraction from *Costus afer* showed an average mass of oil yield as 5.22g and the average percentage of oil yield was 5.2%. Also, the average mass of the oven dried waste of the sample after extraction was 94.74g. A total weight of 26.09g of the bio oil was extracted from 500g of dried monkey sugarcane leaves. The physicochemical properties obtained were; boiling point (125oC), acid value (0.9mgKOH/g), FFA (0.49mgKOH/g), water content (0.53%), saponification value (146.3mgKOH/g), flash point 187oC, pour point (5oC) and the pH value was 7.3.

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