

Spectroscopic Analysis of Oil Extracted from Seeds of *Hildegardia barteri* (Mast.) Kosterm

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Abstract

Though, there has been alarming concern for increase in environmental pollution from domestic and industrial use of fossil fuel but all hands have been on deck to ensure provision of measures that will mitigate through different researches. Replacement of fossil fuel combustion with bio-resources products has been a sustainable measure. In this study, sun-dried and oven-dried seeds of *Hildegardia barteri* (mast.) Kosterm were subjected to mechanical and n-hexane solvent extraction of oil content. Extracted oil samples were spectroscopically analyzed to detect the possible functional group using Fourier transform infrared (FT-IR) spectrometer so as to explore its domestic and industrial use. Results from this study shows that all *Hildegardia barteri* oil samples had a prevalence of single bond compounds which suggests more of saturated fatty acids. The presence of aliphatic amine group in oven-dried solvent extracted (OD+SE) sample distinct it from other samples; sun-dried solvent extracted (SD+SE) sample, was characterized with presence of sulphate group and an unidentified group; the presence of phosphate group in sun-dried mechanical extracted (SD+ME) oil and oven-dried and mechanical extracted (OD+ME) sample has nitrile group and also unidentified group which indicates the presence of undesirable impurities proves the necessity for degumming or refining of oil.

The mechanically extracted *H. barteri* oil samples were observed to have more impurities than the solvent extracted oil which could have resulted from contamination during handling and extraction processes. However, there were prevalence of alcohol (O-H), alkane (C-H), alkyne (C≡H), aldehyde (C=O stretch), esters (C-O), ether (C-O-C) and carboxylic acids (C-O) in all oil samples of *H. barteri* as observed from the spectra analysis. The results indicate stability of the oil but a need for further refining when there is need for industrial applicability.

Keywords: FT-IR analyses; *Hildegardia barteri*; Seed oils; Extraction methods; Drying methods

Introduction

Lipids (fats and oils)-one of the major components of food which has functional properties governed by their molecular structure and intra and intermolecular interactions within the food system, and the amounts present, define the characteristics of food products. The quality of these properties includes chemical, biological and microbial factors, e.g. stability of oil, which implies their shelf life and is connected with irreversible chemical and enzymatic reactions [1].

Oils and fats which constitute human diet, is not limited to consumption purpose as past and recent researches have shown growing concerns for oils as source of material for industrial purpose [2]. The main factor for this concern is due to environmental issues that regard fossil fuel as major contributor of greenhouse gases which are characterized by trace elements such as Lead, Sulphur, Arsenic, and Carbon.

Fourier Transform Infrared (FTIR) spectrometer is one of the spectroscopic methods that is known to be a rapid and non-destructive powerful analytical tool studying edible oils and fats reference. FTIR is also an excellent tool for quantitative analysis, since the intensities of the spectral bands are proportional to concentration. For this reason, FTIR has been used to distinguish oils from different botanical origins

using non-supervised classificatory techniques [3,4]. It suggests functional groups or compounds that are present which are further categorized into either saturated or unsaturated compounds. The FTIR spectroscopy has an excellent potential in providing qualitative and quantitative data for fuels including biodiesel without much rigor in sample preparation; it is used extensively as a quantitative analytical procedure for assessing edible oil quality parameter [5,6].

Hildegardia barteri is a lesser-used but primarily an ornamental tree species with one-seeded pods which has been attested to have oil contents [7,8] ranging between 19-39%. The species belongs to the family Malvaceae, having its geographical range from Ivory Coast to south-eastern Nigeria within West Africa and is called the Krobo Christmas tree. The observed oil yield values fall within the reported range for commercial vegetable oils considered suitable for commercial and industrial applications [9].

Hence, the need for explicit spectrophotometry evaluation of the oil obtained for its domestic and industrial applications potentials.

Materials and Methods

Seed collection and drying

The fresh matured sample materials were collected from the University of Ibadan, Nigeria and de-hulled. De-hulled samples were randomly divided into two halves. The first half was spread in the sun

of ambient temperature of 32°C for average of 9 days due to inconsistent weather condition while the other half was oven dried at 40°C for 6 hours to attain a constant weight.

Extraction methods

Two different methods Soxhlet Extraction using n-Hexane Solvent for 12 hours [10] and Mechanical Process using a fabricated manually operated mechanical extractor were used to extract oil samples from both oven and sun dried kernels of *H. barteri*; this, giving a total of four oil samples. They are: Oven dried and solvent method (OD+SE), Sun dried and solvent method (SD+SE), Oven dried and mechanical method (OD+ME) and Sun dried and mechanical method (SD+ME)

Spectroscopy analysis

The spectroscopy analysis performed on the oil samples was FT-IR. It was used to determine the possible functional groups present in the samples. The response of the functional groups were characterized by observing the transmission of infrared radiations and comparing it with known standards in order to identify the type and the nature of functional groups present in the samples. The absorption frequency spectra were recorded and plotted as transmittance against wave number.

Results and Discussion

The extracted oil yield from the different samples ranges between 19.52-30.53%, having the sun dried and solvent extracted (SD+SE) sample gave the highest yield while the sun dried and mechanical extracted (SD+ME) sample gave the least yield. The difference between the yields of SD+SE and SD+ME was as a result of the extraction methods. The result corroborates the findings that solvent extraction method is known for its high yielding oil output [11]. The overall yield compares well with the report on the seed composition of kernels of *H. barteri* (39% oil yield) [12] and also corresponds with oil yield values that fall within the reported range for commercial vegetable oils such as cotton seed (19.50%), soybeans seed (19.0%), palm oil (48.65%) and groundnut (49.0%) [13].

Spectra analysis (FT-IR)

Tables 1-4 are results of the FT-IR analysis performed on the oil samples, which show suggestions of likely functional groups or compounds that are present as interpreted by Pavia et al. [14].

Oven Dried and Solvent Extracted (OD+SE) oil sample

In Table 1, compounds observed from the FT-IR analysis of oven dried and solvent extracted oil sample (OD+SE) were Alcohols (O-H), Esters (C-O) and Alkynes (C≡H). The types of bonds among the compounds found mostly were single bond. In Figure 1, the absorption peaks shown are the wave numbers which depict corresponding functional groups as interpreted in Table 1.

Sample	Wavenumber (Cm ⁻¹)	Functional Group	Compound
OD+SE	3473.88	O-H	Alcohols
	3299.37	O-H	Alcohols
	2689.07	C-H	Alkanes

2033.61	C≡H	Alkynes
2112.04	C≡H	Alkynes
1432.16	C-H (bend)	Alkanes
1733.66	C=O	Aldehyde
1172	C-O	Alcohols
	C-O	Esters
	C-O	Ether
	C-O	Carboxylic acids
	C-H-(CHX)	Alkyl halides
	C-N (stretch)	Aliphatic Amines
710.77	C-Cl	Alkyl Halides

Table 1: Functional groups in OD+SE Sample.

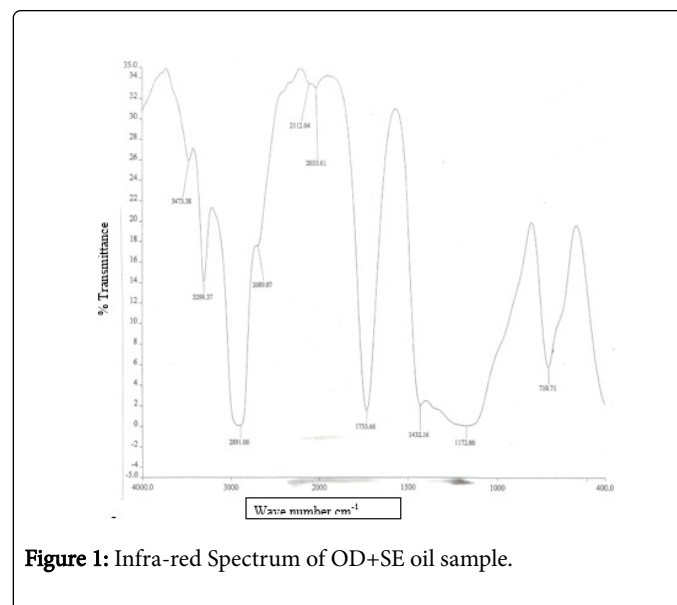


Figure 1: Infra-red Spectrum of OD+SE oil sample.

The prevalence of the single bond compounds suggests that the oil sample has more percentage of saturated fatty acid compounds than the unsaturated compounds and this is in conformity with 77.04 and 22.96% respectively for saturated and unsaturated fatty acid composition of crude fat, reported on extract from kernels of *H. barteri* [15]. The nature of these saturated chemical groups indicates lower iodine value and higher cloud point. This depicts that the oil is an essential oil and is more stable in the presence of oxygen [16]. That is, the occurrence of oxidative rancidity in the oil sample will be low and this extends the shelf life of oils not in use. Another implication of the presence of these saturated chemical groups is that the free flow of the oil may be affected particularly during cold weather operations in that it may tend to be semi-solid.

Sun Dried and Solvent Extracted (SD+SE) sample

Table 2 shows the FT-IR analysis of the (SD+SE) sample. The result suggests the abundance of C-H (stretch) aldehyde, C≡H alkynes and CH₂X alkyl halide groups and the prevalence of saturated bonds in the

oil sample. In Figure 2, absorption peaks shown are the wave numbers which depict corresponding functional groups as interpreted in Table 2.

Sample	Wavenumber (Cm ⁻¹)	Functional Group	Compound
SD+SE	3474.29	O-H	Alcohols
	3300.23	C≡H	Alkynes
	2889.78	C-H(stretch)	Aldehydes
	2700.28	C-H	Aldehydes
	2330.53	Unidentified	
	2033.61	C≡H	Alkynes
	1738.74	C=O	Ester
	1432.16	CH ₃ (bend) or S=O	Alkane, Sulphate
	1167	C-H (-CH ₂ X)	Alkyl halide
	710.82	C-X	Alkyl halide

Table 2: Functional groups in SD+SE Sample.

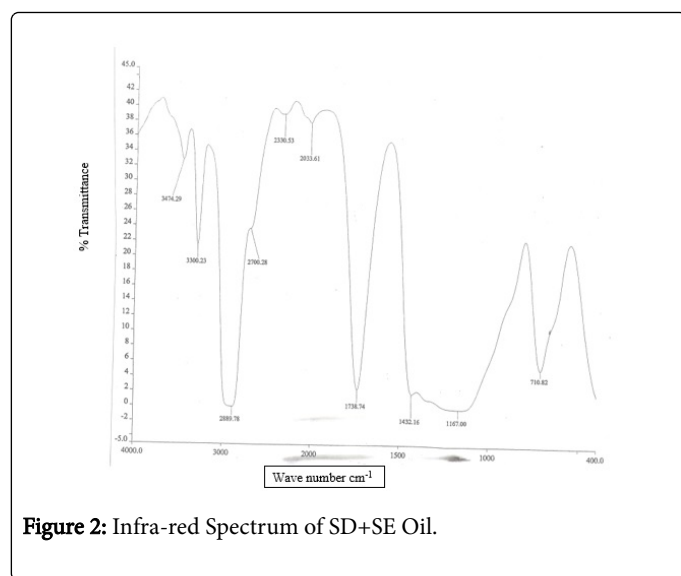


Figure 2: Infra-red Spectrum of SD+SE Oil.

Except for the unidentified group (2330.53 cm⁻¹) and sulphate group which may indicate undesirable contaminant in this sample, all other chemical compounds suggested to be present are similar to that of OD+SE sample. In some cases, the presence of these contaminants could be traced to desirable or undesirable chemical or biochemical reactions from the process of sun drying which may lead to changes in colour, texture, odour or other properties of food products [17,18]. However, from literature review [19], the elemental analysis carried out shows that the sulphur contained in oil of *H. barteri* seeds are of lower values which fall within permissible limit.

The presence of more saturated chemical groups indicates higher cloud point which could affect the performance of biofuel during cold weather operations [20]. The ester C=O and alkyne C≡H groups which are characterized by unsaturated bonds, may not have significant effect in keeping the oil that could be used for production of biodiesel as reported for rubber biodiesel [13]. However, the need for injecting cold

flow improver into the rubber biodiesel for better performance was suggested. This could also be applicable to the biodiesel that could be produced from *H. barteri* seeds.

Sun Dried and Mechanically Extracted (SD+ME) sample

The most abundant functional groups observed in the sun-dried mechanical extracted oil sample were Alcohol (O-H), Esters of either C-O or C=O, Alkanes (C-H), Alkyne (C≡C or C≡H) and Phosphate (P=O) (Table 3). Figure 3 shows absorption peaks which depict corresponding functional groups as interpreted in Table 3.

Prevalence of single (saturated) bonds indicates that the oil is a stable and not a dry oil. It is also an indication of low iodine value and higher cloud point (B100.research.com) which may affect the free flow of the fuel particularly during cold weather operations [20].

The appearance of P=O group verifies the presence of phosphate which is usually in form of phospholipids (a key component) in most vegetable oils. But the use of vegetable oils for biofuel purpose, need to be devoid of phosphorus because it tends to form harmful deposits on engine parts. However, removal of these phospholipids and reduction of phosphorus can be done through a refining processes known as degumming and neutralization [7].

Sample	Wavenumber (Cm ⁻¹)	Functional Group	Compound
SD+ME	3474.33	O-H	Alcohols
	3299.97	O-H	Alcohols
	2890	C-H	Alkanes
	2689.07	C-H	Alkanes
	2380.95	C=O	Aldehyde
	2100.84	C≡H	Alkynes
	2033.61	C≡C (stretch)	Alkynes
	1736.73	C=O	Ester
	1429.37	CH (bend)	Alkanes
	1172	C-O	Alcohols, Esters
		C-O	Ether, Carboxylic acid
		P=O	Phosphate
	710.45	C-Cl	Alkyl halides

Table 3: Functional groups in SD+ME Sample.

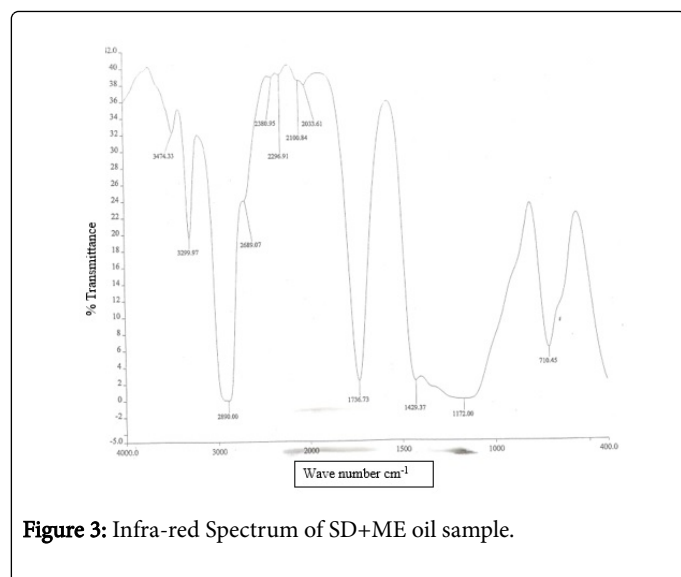


Figure 3: Infra-red Spectrum of SD+ME oil sample.

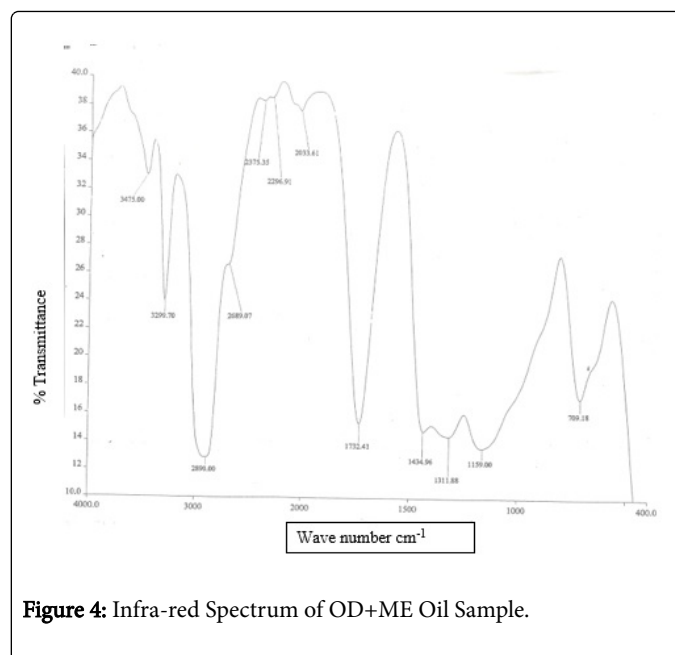


Figure 4: Infra-red Spectrum of OD+ME Oil Sample.

Oven Dried and Mechanically Extracted (OD+ME) sample

The FT-IR analysis obtained in the OD+ME sample suggested the following functional groups: Alkynes ($C\equiv C$), Aldehydes ($C=O$ or $C-O$) and Alkyl halides $C-X$. In Figure 4 appearance of absorption peaks which are the wave numbers depict corresponding functional groups as interpreted in Table 4.

Sample	Wavenumber (cm^{-1})	Functional Group	Compound
OD+ME	3475	O-H	Alcohol
	3299.7	$C\equiv C$	Alkynes
	2890	C-O	Aldehyde
	2689.07	C-H	Alkanes
	2375.35	Unidentified	
	2296.91	$N\equiv C$	Nitriles
	2033.61	$C\equiv C$	Alkynes
	1732.41	$C=O$	Aldehyde
	1434.96	C-H-(CH_2)	C-H-(CH_2)
	1311.88		C-N-(CH_2X)
	1159	C-X	Alkyl Halides
	709.18	C-X	Alkyl Halides

Table 4: Functional groups in OD+ME Sample.

Infra-red spectrum of OD+ME oil sample

Appearances of nitriles NC and unidentified groups could indicate presence of undesirable impurities that would have contaminated the oil during handling and extraction process. Like the previous samples, the result also indicates more of saturated bonds than the unsaturated bonds in the oil sample.

The presence of single bond compounds especially alkanes indicates prevalence of properties such as cloud points that affect the performance of biodiesel during cold weather engine operation [20]. However the presence of groups with unsaturated bonds especially carbon to carbon double bonds ($C=C$, $=C-C$) which are the most abundant could cause the fuel to remain in liquid state in a cold weather condition but may be liable to possible oxidation during storage [13].

Generally, the prevalence of saturated bonds indicates that all the oil samples are essential or stable and not dry oil that vaporize.

Conclusion

The Fourier Transform-Infrared Spectroscopy is a standard method that studies the functional groups that are present in the oil samples of *H. barteri*. The presence and the nature of single bond functional groups observed, among other factors provide information on the stability of the oil and suitability for potential use for biodiesel fuel.

Observations made from the spectra analysis of the oil samples suggested prevalence of alcohols, esters, alkanes, alkynes, aldehydes and alkyl halides as common functional groups in which majority are characterized by saturated bonds. Thus, the oil from *H. barteri* fruit is an essential or stable oil. While oil samples with sulphate and unidentified groups which are termed impurities can be ascribed to the effect of handling and extraction methods used.

However, more spectroscopy analyses such as use of Nuclear Magnetic Resonance (NMR), Gas chromatography mass spectroscopy (GCMS), Liquid chromatography mass spectroscopy and (LCMS) will

be explored for different compounds on this species in the future research.

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