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Cashew Nut Oil: Extraction, Chromatographic and Rheological Characterisation.

Vincent Okechuwku ANIDIOBU[1], Chioma Oluchi ANIDIOBU[2]

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Abstract

Oil was extracted from cashew nuts. The physicochemical parameters of the oil were determined. A chromatographic assay of the oil was carried out using Gas Chromatography-Mass Spectrometry. Seventeen compounds were detected: Phenol, Phenol 2-methyl-, Cyclohexene 4, 4-dimethyl-, m-Fluoro-2-diazoacetophenone 4-dimethyl-, Tetradecanoic acid, Phenol 4-octyl-, n-Hexadecanoic acid. Others are 9, 12-Octadecadienoic acid (Z, Z) - methyl ester, Hexadecanoic acid methyl ester, Methyl stearate, Dodecanoic acid methyl ester, 9, 12, 15-Octadecatrienoic acid methyl ester, 9, 12, 15-Octadecatrienoic acid (Z, Z, Z)-, Oleic acid, Octadecanoic acid, Tetracosanoic acid and 9-Octadecenoic acid methyl ester. Among the components are omega three and omega six essential free fatty acids. The rheological profiling and flow properties of cashew nut oil were determined using a Programmable Rheometer. Cashew nut oil exhibits slight dilatant behaviour at the low end of shear rate. The long chain and high molecular weight of its constituents controlled its rheology. Long-chained 9-Octadecenoic acid methyl ester, 9, 12-Octadecadienoic acid (Z, Z) - methyl ester, Tetracosanoic acid and methyl stearate, coupled with their high molecular weights are responsible for the shear thickening effect observed. Two models, Carreau-Yasuda and Ostwald-de Waele Power Law were employed to fit the rheological data. The Carreau-Yasuda model followed well the data.

Keyword: Cashew nut oil, GC-MS, Rheology of cashew nut oil, Ostwald-de Waele Power Law model, Carreau-Yasuda model

Major Classification Code: Food Science, Food Engineering, Health Science, Environmental Engineering

1. Introduction

Cashew (*Anacardium Occidentale L*) is a tropical evergreen shrub plant originating from Brazil (Dike et al., 2019; Taiwo, 2015). It is found around latitude 23° North and South of the equator (Pandian et al., 2021). It is a low water requiring plant grown profitably in regions with an average precipitation of 50-350 cm (Idah et al., 2014). Cashew as food is an excellent source of healthy omega-three fatty acid (Kyei et al., 2019). As a cash crop, it is a good source of income having over a million participants in the industry with trading value of 24 billion Naira ($60 million United States of America Dollars) in Nigeria (Adeigbe et al., 2016). Other countries like Vietnam, India and Philippines are significant producers of cashew nuts (Taiwo, 2015; Dike et al., 2019; Shahbandeh, 2022). Vietnam has maintained a

1 First Author. Principal Lecturer, Food Process Engineering Section Department of Food Technology, Federal Polytechnic Ado-Ekiti, Ekiti State, Nigeria. Email: drvinanidiobu.class@gmail.com
2 Second Author. Lecturer II, Department of Science Laboratory Technology, Federal Polytechnic Ado-Ekiti, Nigeria, Email: chyanidiobu@gmail.com

giant spot as the global biggest producer and exporter of cashew products in 2020, exporting an estimated 2,600,000 tones of cashew nuts worth over 3.2 billion USD (FAOSTAT, 2020).

Three main products obtained from raw cashew nuts are cashew nut oil (CNO), cashew nut shell liquid (CNSL) and cashew nut kernels (Akinhanmi et al., 2008). A fourth product, the cashew fruit or apple, is eaten raw or processed for fruit juice (Idah et al., 2014).

The nut is one of the most essential products from the cashew industry, mainly used as confectionery or for extraction of the highly valuable cashew nut oil. The fat content of the cashew nut is completely natural, very good for the human body. The cashew nut oil is laden in Linoleic acid (Omega-three) and is the least harmful to the heart and arteries. Its oil is said to constitute around 47% total weight of the nut (Achal, 2002). In tropical Africa, especially Nigeria, men above forty-five and women above fifty suffer predominantly from high blood pressure due to the accumulation of cholesterol from fats and oils and from other sources consumed over the years (Adeloye et al., 2020).

Cashew contains 'good fat', which has the correct blend of saturated to monounsaturated and polyunsaturated fat ay the ratio of 1:2:1. This mixture is nutritionally good for human ingestion (Akinhanmi et al., 2008). The comparative quantity of good fat in cashew nuts is helpful to improving quality health when ingested and the comparative profusion of fat in cashew nuts does not imply a nutritional risk (Achal, 2002). In the past, Idah et al. (2014), Kyei et al. (2019), and Dike et al. (2019) worked on extraction and Physicochemical characterization of cashew nut oil.

Gas Chromatography (GC) is an essential analytical tool that separates constituents in a gas mixture. GC is a chromatographic method used to isolate and analyse substances that can be vaporised devoid of breakdown. GC is used for determination of the purity of substances or separating the different constituents of a substance and relative amounts of different components of a mixture. Pure compounds from a mixture of substances can be produced using the instrumentality of GC. Two techniques are blended in Gas Chromatography-Mass Spectroscopy (GC-MS) into a single analytical method to evaluate a blend of chemicals. Gas chromatography isolates a mixture of components, while mass spectroscopy characterises each component individually. Quantitative and qualitative analyses of samples are achieved by combining both GC and MS techniques (Pearson, 1981).

Deformation and flow of fluid materials is described as Rheology. A nice introspection on rheology is crucial to gain insight in many scientific and engineering processes (Anidiobu, 2016). Molecular interactions and structural rearrangements within the fluid triggered by their flow and deformation lead to the observed outcome during the rheological measurements (Bird et al., 2006).

The objective of this work is to assess cashew nut oil's chemical and molecular composition from the gas chromatography-mass spectrophotometer. Also, the flow consistency and the fluid's rheological characterization were carried out after the oil was extracted.

2. Materials and Methods

2.1 Instrumentation and Methodology of Gas Chromatography-Mass Spectrophotometer Analysis

GC-MS is a distinctive separation method applied for detection and quantification, focused on samples. The samples are expected to be unstable or thermally volatile but should resist the harsh partitioning environment of the gas chromatograph. The Gas-chromatographic apparatus has an injection port from where the process is initiated. The next operation was, evaporation and separation of the sample components occur one by one, and finally, this equipment identifies the separated fragments of the corresponding sample. An explicit spectral peak is generated for each component and electronically transmitted on the screen.

The analysis was executed using a 3800/4000 gas chromatograph-mass spectrometer (Santa Clara, California, USA) model operational with an Agilent silica capillary column (30 x 0.25 mm ID x 0.25μm film thickness, 5% Phenyl, 95% Dimethyl Polysiloxane). Nitrogen gas of 99.999% purity was used as the carrier gas during the experiment at 1ml/ min of constant flow rate at the injector temperature of 290ºC. Ionisation of electron took place when the ion inception temperature was 150ºC. During the experiment, the temperature algorithm was as follows: from 50ºC it was raised to 220ºC at 2ºC/ min and held for 10min; from 220ºC to 280ºC with 4ºC/min and maintained for 10 min. Identification and categorization of unknown compounds were made by referring to the retention times of authentic compounds and the spectral data collected from NIST and Wiley Spectral library search program.

Replicates samples and were infused into the equipment continuously as one batch at random. In addition, the prepared pool of samples was used as quality controls and inserted at regular intervals all through the experiment to provide data from which the repeatability can be assessed and calculated (Cock & Rede, 1996; Ronald, 2007).

2.2 Physical and Chemical Properties of Cashew Nut Oil

Cashew nut oil was Characterised by analysing its physicochemical properties. The physical properties were analysed by standard tests as described by AOAC (1990). Some of the properties include: specific gravity, boiling point, refractive index and density of the oils. As part of the chemical analyses, the saponification value and the pH of the oils as follows were determined. The method described by Strong and Kock (1974) was used to determine the saponification value while the method described by Aremu et al. (2006) was used to determine the PH value of the oil.

2.3. Rheological Measurement Procedure

The samples were kept at rest for 30 minutes to achieve complete structural recovery. Brookfield DV-3T Ultra Programmable Rheometer was applied to determine the sample's rheology. The theory of operating the Rheometer is to impel a spindle deep in the fluid in the course of a calibrated spring. The spring's deflection determines the viscous drag of the testing material aligned with the spindle. The spindle shapes, sizes, speeds are the variables that project the measurement range of the rheometer. 600 ml beaker fluid container was used during the experiment. The rheological readings were taken at 24mm cone radius and at 0.8 cone angle that gives the gap height of 0.1 mm at the boundary of the cone. The rheology of cashew nut oil was investigated at 27°C.

2.3.1. Ostwald-de Waele Power Law Model (PLM)

The expression of Power law model is given as follows:

$$\tau = \eta \gamma^{n} \qquad (1)$$

From equation (1),

η is the experimental viscosity (Pa.s), (mPa.s as applicable)

τ is the fluid's shear stress (N/m^2)

n Changes with the behaviour of the fluid (dimensionless)

The fluid is Newtonian when the behaviour index equals to one. When *n* is less than 1, the fluid exhibits psuedoplastic behaviour; if the value of n is higher than 1, the fluid is dilatant.

The basic simplification of the two-parameter power-law expression was shown by Anidiobu (2016) as follows:

$$\eta \gamma = \eta_o \gamma^{n} \qquad (2)$$

Making η the subject,

$$\eta = \eta_o \gamma^{n-1} \qquad (3)$$

Taking the natural logarithm of equation (3)

$$\ln \eta = \ln \eta_o + (n-1)\ln \gamma \qquad (4)$$

Plot of $\ln \eta$ against $\ln \gamma$ gives a straight-line graph with slope of *(n-1)* and intercept, $\ln \eta_o$.

2.3.2.Carreau-Yasuda Model (CYM)

The following parameters are found in the expression of Carreau-Yasuda model as expressed by Yasuda (1979): α, λ, n, η_0, and η_∞.

$$\eta(\dot{\gamma}) = \eta_\infty + (\eta_o - \eta_\infty)\left[1 + (\dot{\gamma}\lambda)^a\right]^{\frac{n-1}{a}} \qquad (5)$$

From equation (5), the parameters η_o and η_∞ are the zero and infinite shear rates. The variable λ is the viscous relaxation time. It defines the location of the switch from dilatant to psuedoplastic behaviour. The power-lawslope is represented by (*n*-1). The variable, n, is the behaviour index and it changes with the composition of the fluid. The parameter "α"is dimensionless, introduced by Yasuda to the Carreau equation.

2.3.3 Rheological Curve Fitting

The linear least square method was applied to fit the Power law model. The Carreau-Yasuda model was fit using the method of Morrison (1999).

3. Experimental Results

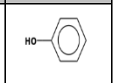
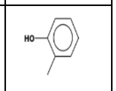
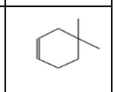
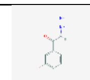
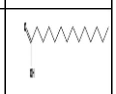
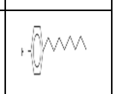
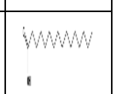
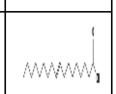
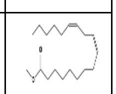
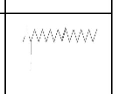
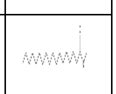
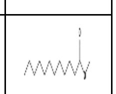
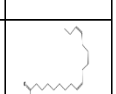
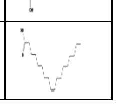
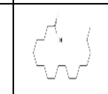
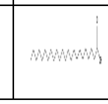
3.1. Chromatographic Assay Results

Figure 1 is the GC-MS Chromatograph of cashew nut oil. It shows seventeen compounds eluted at different retention times and peak areas. The qualitative and quantitative compositions are discussed here.

GC-MS analysis revealed seventeen compounds in cashew nut oil, as shown in Table 1. Phenol (1.08% composition), Phenol 2 methyl (7.15% composition), and Phenol, 4-octyl- (0.52 % composition) are antioxidants present in the analysed cashew nut oil with enormous health benefits (Trevisan et al., 2005; Shahidi & Ambigaipalan, 2015). The compounds found in cahew nut oil vary structurally from plain phenolic molecules to high-molecular-weight complex polymers. It is clearly evidential that consuming various foods containing phenolic compounds possibly will lower the risk of health challenge as a result of their antioxidant activity (Vermerris & Nicholson, 2008). If present in foods, antioxidants control rancidity increase thereby extending the shelf life of the food product, slows the advance of toxicity, and maintain nutritional quality in the products (Shahidi & Ambigaipalan, 2015). Oxidation of lipid is a significant cause of food value decline and production of off odours and off flavours, diminishing shelf life, changing texture and colour, and diminishing the nutritional value of food

(Alameda et al., 2009). The presence of these antioxidants in cashew nut oil prevents it from oxidation.

Table 1: GC-MS Summary of the Chromatographic Assay of Cashew Nut Oil

Peak #	RT	Compound Detected	Mol. Formula	MW	Peak Area%	Comp %wt	m/z	Structures
1	6.52	Phenol	C_6H_6O	94	4.35	1.08	65, 66, 94	
2	16.61	Phenol 2-methyl-	C_7H_8O	108	6.33	7.15	51, 79, 108	
3	21.96	Cyclohexene, 4,4-dimethyl-	C_8H_{14}	110	5.94	1.41	41, 56, 110	
4	23.00	m-Fluoro-2-diazoacetophenone	$C_8H_5FN_2O$	164	4.75	1.50	107, 108, 164	
5	28.74	Tetradecanoic acid	$C_{14}H_{28}O_2$	228	3.96	0.11	43, 73, 228	
6	31.00	Phenol, 4-octyl-	$C_{14}H_{22}O$	206	6.58	0.52	77, 107, 206	
7	34.09	n-Hexadecanoic c` acid	$C_{16}H_{32}O_2$	256	7.92	9.17	43, 73, 256	
8	36.31	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	270	5.15	8.21	43, 73, 282	
9	38.00	9,12-Octadecadienoic acid (Z, Z)-, methyl ester	$C_{19}H_{34}O_2$	294	17.42	25.25	41, 67, 294	
10	39.00	9-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	296	8.51	13.06	55, 69, 296	
11	40.44	Methyl stearate	$C_{19}H_{38}O_2$	298	5.74	7.00	43, 74, 298	
12	42.50	Dodecanoic acid methyl ester	$C_{13}H_{26}O_2$	214	4.55	4.08	41, 74, 214	
13	45.42	9,12,15-Octadecatrienoic acid, methyl ester	$C_{19}H_{32}O_2$	292	2.37	4.02	41, 79, 292	
14	49.00	9,12,15-Octadecatrienoic acid, (Z,Z,Z)-	$C_{18}H_{30}O_2$	278	1.98	2.09	67, 79, 278	
15	54.00	Oleic Acid	$C_{18}H_{34}O_2$	282	6.14	7.35	41, 56, 282	
16	56.00	Octadecanoic acid	$C_{18}H_{36}O_2$	284	3.56	5.78	43, 73, 284	
17	67.02	Tetracosanoic acid	$C_{24}H_{48}O_2$	368	0.79	0.26	43, 57, 368	

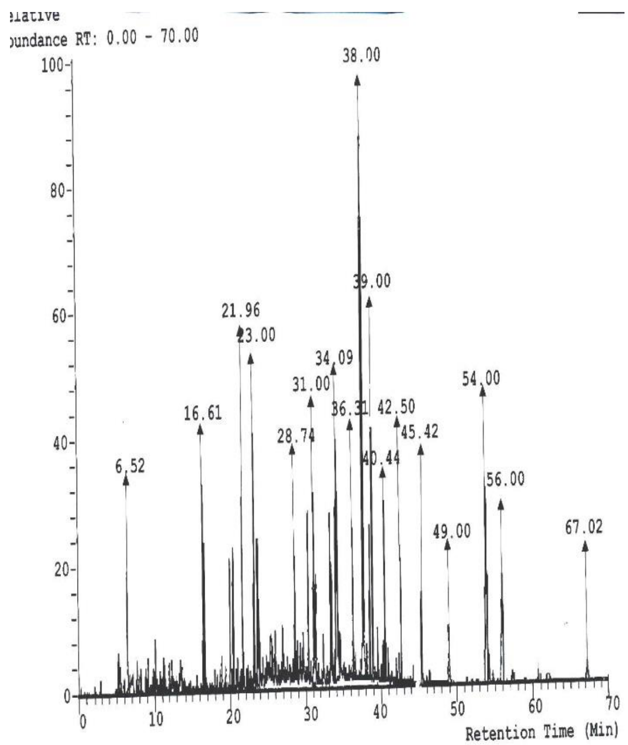


Figure 1: Chromatograph of Cashew Nut Oil

Likewise, using the GC-MS, 1.5% of mono fluoro-2-diazoacetophenone with a molecular weight of 164g/mol was also detected in the cashew nut oil. It also has an excellent antioxidant capacity (Shahidi & Ambigaipalan, 2015).

Also, 1.41% of 4, 4-dimethyl Cyclohexene was detected in the cashew nut oil with a molecular weight of 110g/mol.

0.11% of Tetradecanoic acid is present in the cashew nut oil. Tetradecanoic acid, also known as Myristic acid, is a distinctive saturated fatty acid with the molecular formula of $CH_3(CH_2)_{12}COOH$. Myristates is a common name for its salts, esters, or tetradecanoates (Playfair, 2009). Myristic acid is contained in cashew nut oil, 8–14% bovine, palm kernel oil, butterfat, coconut oil and breast milk (Beare-Rogers et al., 2001). Captivatingly, some workers point to myristic acid's helpful effects on High-Density Lipoprotein

(HDL) cholesterol and hence improving HDL (good cholesterol) to total cholesterol ratio, which is good for the cardiovascular health of humans (Kromhout et al., 1995).

9.17% of n-Hexadecanoic or Palmitic acid with a molecular weight of 256g/mol was found in cashew nut oil. It is the most common saturated-free fatty acid in plants, animals and microorganisms (Gunstone et al., 2007). Palmitic acid can be formed by many varieties of plants and organisms, typically at small quantities (Anneken et al., 2006). Also, 5.15% of Hexadecanoic acid, methyl ester with a molecular weight of 270g/mol, is present in the cashew nut oil. A similar result was earlier obtained by Bello and Anjorin (2012) from cashew nuts in Ondo State.

25.25% of Linoleic acid or 9, 12-Octadecadienoic acid (Z, Z)- methyl ester with a molecular weight of 294 g/mol was detected using GC-MS as one of the major compounds in the cashew nut oil. Linoleic acid is an omega-six fatty acid essential to humans which is a polyunsaturated in nature(Haynes, 2016). It is a whitish liquid virtually insoluble in water but soluble in many organic solvents. Consumption of non-rancid nuts such as cashew or its oil, high in omega-6 fatty acid, is linked to lower risk of some ailments, such as cardiovascular diseasesincluding coronary heart disease (CHD), stroke, heart attacks,cancer, and lowers rates of premature death (Aune et al., 2016; Luo *et al*., 2014; Bakalar, 2017; Huang *et al*., 2018). Due to the composition of Linoleic acid in cashew nut oil, it is highly recommended for its health benefits.

13.06% of methyl ester or 9-Octadecenoic acid or Oleic acid with a molecular weight of 296 g/mol was detected using GC-MS in the cashew nut oil. Oleic acid occurs naturally in animal , vegetable fats and oils. Although commercial samples may be yellowish, it is odourless, and colourless oil. Also, oleic acid is categorised as a monounsaturated omega-nine fatty acid. Unlike omega-three fatty and omega-six fatty acids, omega−nine fatty acids are not categorised as essential fatty acids (EFA). Consequently, they can be obtained by the human body from unsaturated fat in the diet and are, therefore, not really essential. Thus, it lacks an omega−six double bond that keeps them from taking part in the reactions that form the eicosanoids. Oleic acid is the highest monounsaturated fat in the human diet (Schwingshackl & Hoffmann, 2014). Consumption of monounsaturated fat has been related with low-density lipoprotein cholesterol and possibly with increased high-density lipoprotein cholesterol (Aladedunye & Przybylski, 2013). Studies imply that Oleic acid could be accountable for the hypotensive (blood pressure-reducing) effects of olive oil which is considered a health benefit (Teres et al., 2008). A 2017 review suggests that oleic acid-rich diets are good for regulating body weight (Huang et al., 2018).

7% of Methyl stearate with a molecular weight of 298g/mol was detected in the sample. This saturated compound, otherwise called fatty acid methyl ester, is the natural component of biodiesel. 4.08% of Dodecanoic acid methyl ester was contained in the sample. It is one of the esters of saturated fatty acids in cashew nut oil.

4.02% of 9, 12, 15-Octadecatrienoic acid methyl ester was detected in the sample. This polyunsaturated ester, or methyl linolenate, has a molecular high weight of 292g/mol. It is also an omega-3, essential fatty acid. (Aung *et al.*, 2018*;* Grey and Bolland, 2014*;* Alvarez Campano et al.,2019*).*

Oleic acid is a fatty acid occurring in animal and vegetable fats and oils. Although commercial samples may be yellowish, it is an odourless, colourless oil. Similarly, oleic acid is grouped as a monounsaturated omega-9 fatty acid, abbreviated having a lipid number of 18:1 *cis*-nine.

5.78% of Octadecanoic acid, with a molecular weight of 284g/mol, was detected in the sample. It is a polyunsaturated fatty acid having an 18-carbon unbranched backbone with three double bonds. Likewise, 0.26% of Tetracosanoic or Lignoceric acid was detected. It is also a saturated fatty acid having the formula $C_{23}H_{47}COOH$ and a molecular weight of 368g/mol.

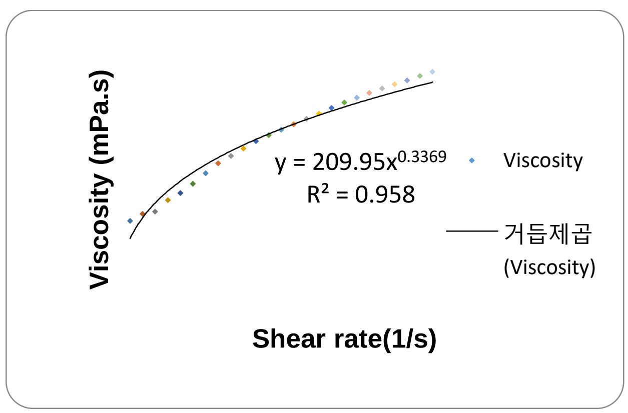


Figure 2: Rheological Curve-fit of CNO using Ostwald-de Waele Power Law Model

Figure 2 shows the rheogram of cashew nut oil. It shows that the viscosity increased from 140mPa.s to 354.4mPa.s with increasing shear suggesting shear-thickening flow behaviour.Franck (2004) produced rheological signatures of polymeric substances that look like those of cashew nut oil obtained during the experimental runs. Triantafillopoulos (1988) had a model for interpreting the rheological outcome of solutions of polymers. His interpretations can be borrowed to explain the rheological outcome of cashew nut undergoing deformation.

Deriving inference from Frank (2004) and Triantafillopoulos (1988), it suggests that rheology of the cashew nut oil is controlled by the high molecular weight of long-chained 9, 12-Octadecadienoic acid (Z, Z) - methyl ester, 9-Octadecenoic acid methyl ester, and methyl stearate. These three compounds shared 45.31% composition of the cashew nut oil. Though Tetracosanoic acid occupied only 0.26% composition of the cashew nut oil with a molecular weight of 326g/mol, it has the most extended chain length to induce a reversible shear hardening (dilatants) effect upon flow at low rates of shear.

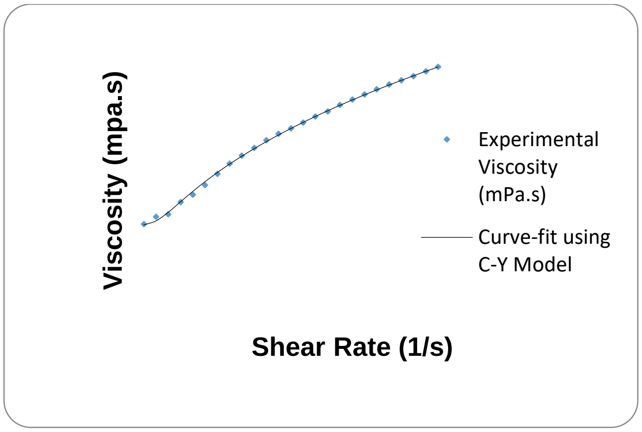


Figure 3: Rheological Curve-fit of CNO using Carreau Yasuda Model

In Figure 2,Ostwald-de Waele Power Law Model fit the rheological data. It can be observed that the goodness of fit, R^2 is 0.958. The curve fit produced a behaviour index of 1.336 (higher than 1), indicating a shear-thickening flow behaviour. Obtaining a behaviour index of one would have indicated a Newtonian flow, while less than one would indicate shear-thinning flow behaviour.

Table 2: Summary of Rheological Curve-fit of Cashew nut and Cashew Nut Shale oils using *Ostwald-de Waele* Power-Law Model

Sample Details	η_o (mPa.s)	n
Cashew nut oil (CNO)	209.9	1.336

The zero shear viscosity was obtained as 209.9mPa.s from the curve-fit. The details are in Table 2. Minor discrepancies are observed between the experimental and the predicted viscosities in Figure 2 at higher shear rates. As a result of the discrepancies, in Figure 3, another model, Carreau-Yasuda, was used to fit the data. The behaviour index of 1.423, indicating shear-thickening flow behaviour, was obtained at almost a perfect fit. The details are in Table 3.

Table 3: Summary of Rheological Curve-fit of Cashew nut and Cashew Nut Shale oils using the Carreau-Yasuda Law Model

Sample Details	η_o (mPa.s)	η_∞ (mPa.s)	λ	a	n
Cashew nut oil (CNO)	138.10	6.67	2.3857	3.461	1.423

Table 4 is the physicochemical parameters of cashew nut oil. The colour of the cashew nut oil is yellow while has a sweet inviting odour, making it easier for consumption. The boiling point of the oil was found to be 93±0.87°C. The result is comparable to that of Idah et al (2014).

Table 4: Physicochemical Properties of Cashew nut oil

S/N	Properties	Cashew nut oil
1.	Colour	Yellow
2.	Odour	sweet
3.	Boiling Point	93.00±0.87°C
4.	Refractive Index	1.43±0.00
5.	Density at 29°C	890.0± 10 kg/m^3
6.	Saponification value (mgKOH)	159.00±1.00
7	pH	5.69±0.17

The refractive index of 1.43±0.00 obtained is similar to that of Tejas et al. (2012). Density is a physical attribute of a material that reflects its intrinsic property. The density of the oil was 890 kg/m^3. The result was also found to be similar to Idah et al. (2014) and Tejas et al. (2012) who worked on cashew nuts from Nigeria. The saponification value was found to be 159.00±1.00. This low value suggests that it is not good for soap production.

4. Conclusion

A chromatographic assay of cashew nut oil revealed seventeen compounds. Phenol, Phenol 2 methyl, and Phenol, 4-octyl- are antioxidants present in the analysed cashew, which have immense health benefits. Some essential fatty acids were detected. 9, 12, 15-Octadecatrienoic acid, methyl ester was also detected in the sample. Polyunsaturated ester, or methyl linolenate, an omega-three essential fatty acid, was also present. Linoleic acid, a polyunsaturated omega-six fatty acid is essential for humans. Consumption of non-rancid nuts such as cashew or its oil, high in omega-6 fatty acids, is related with a lower risk of cardiovascular diseases. The outcome of the chromatographic assay suggests that the oil is safe for human consumption. On rheological characterisation, cashew nut oil exhibited shear thickening behaviour at low shear rates. The Carreau-Yasuda model gave a better curve fit compared to Ostwald-de Waele Power Law Model.

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