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THERMOPHYSICAL PROPERTIES OF NON-EDIBLE VEGETABLE OIL AND DERIVED ETHYL BIODIESEL OBTAINED FROM *CHRYSOPHYLLUM ALBIDUM* KERNELS

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ABSTRACT

The aim of this study was to evaluate the thermophysical properties of vegetable oil and derived ethyl biodiesel obtained from *Chrysophyllum albidum* kernels. Extracted oil was ethyl-transesterified and the effect of temperature and pressure on the thermophysical properties such as density, kinematic viscosity and isothermal compressibility (K_T) was investigated both on the vegetable oil and on the derived ethyl biodiesel. The fatty acid ethyl ester composition of the formulated biodiesel was also evaluated using gas chromatography-mass spectrometry (GC-MS). Results obtained indicated that the yield of oil extraction is 33.23±3.26%, m/m, with peroxide index of 10.46± 0.12meq O_2/Kg-oil. This peroxide index indicated that the oil was not very suitable for feeding and may be subject to rapid oxidation. The main fatty acid ethyl esters in the oil are linoleate (21.22%), linolenate (25.62%), oleate (19.7%) and palmitate (15.98%). The kinematic viscosity values of the extracted oil varied between 12.08 and 88.65 cst, while those of the derived ethyl biodiesel varied between 2.75 and 10.36 cst for temperatures between 293.15 and 353K. The K_T decreases with increasing pressure at constant temperature while it increases with temperature at constant pressure. Data from this study will be useful in the prediction of diesel engine performance and their real-time simulations.

KEYWORDS: *Chrysophyllum albidum*, vegetable oil, biodiesel, thermophysical properties, Benin

1. INTRODUCTION

African ecosystems abound with numerous multipurpose plant species that remain in the wild and under-exploited [1]. Benin, like other tropical countries, has a wide variety of oil plants such as soy, palm and cotton seeds [2]. Vegetable oils and animal fats are used in food, cosmetic products and in many industrial sectors [3]. Vegetable

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oil is also used as a raw material for the production of biodiesel [4]. Indeed, biofuels are often produced from food plants, such as sunflower, soybean, rapeseed, wheat, beet and sugar cane. As a consequence, food prices have reached their highest levels [5] which is more dangerous for food security in developing countries. Therefore, non-edible vegetable oils or second-generation feedstocks have become more attractive for biodiesel production. In order to explore non-edible feedstocks for biodiesel production, several researches have been undertaken on many non-edible feedstocks.

African star apple (*Chrysophyllum albidum*) is an evergreen tree native to many parts of tropical Africa that belongs to the Sapotaceae family [6]. It reaches average dimensions between 25 and 37 meters, with a circumference at maturity varying on average between 1.5 and 8 meters. Its wood generally has deep stripes on the trunk and stems. Its bark is thin, pale to brownish green and has a gummy latex. The seeds of African star apple are underutilized and are discarded after consumption of its juicy pulp [7].

In Benin, *Chrysophyllum albidum* has multiple uses and is marketed by the population. Several studies reported that the seeds are a good source of vegetable oil [8,9]. Others studies have also focused on various aspects related to these resources, including their contribution to the diet of local populations, their diversity and their geographical distribution [10,11,12]. Ochigbo and Paiko [13] reported the effect of solvent mixture on the characteristics of oils extracted from *Chrysophyllum albidum* seeds [14]. Previous work has reported the fatty acid composition and quality indices of vegetable oil extracted from *Chrysophyllum albidum* seeds [15,16], which can be used for biodiesel production. Thus, the present study aims to investigate the thermophysical properties of non-edible vegetable oil and derived ethyl biodiesel obtained from *Chrysophyllum albidum* kernels.

2. MATERIALS AND METHODS

2.1 Materials

Chrysophyllum albidum fruits were collected at Southern Benin between 6°30′11″ North and 2° 08′ 42″ East, and between 6°38′ 00″ North and 1° 43′ 00″ East. The seeds contained in these fruits were extracted and then sun-dried. The fleshy white kernels of these seeds were also extracted and then dried in an oven at 40°C for 24 hours. They were then ground using a Moulinex blender. The oleaginous paste was used for extraction of the vegetable oil by the Soxhlet method using hexane at 69 °C under reduced pressure.

3.2 Production of biodiesel

The transesterification reactions were carried out in two steps. In the first step, the fatty acids of the vegetable oil were esterified by homogeneous acid catalysis using an ethanol:oil molar ratio of 30:1 in the presence of concentrated H_2SO_4 (1% w/w oil) and moderate stirring (250 rpm at 78°C/1 hour). Then the reaction was neutralised with 25% (w/w) with sodium bicarbonate solution for 5 min under slow stirring, and the phases were separated in a separating funnel. The resulting ester was dried and then filtered with anhydrous sodium sulphate (Na_2SO_4). The residual solvent was evaporated under reduced pressure and the ester-rich phase was then weighed. The unreacted triglycerides and partial glycerides (mono- and di-glycerides) contained in the ester-rich phase were transesterified in a second step. The reaction was continued by using the ethanol/oil molar ratio (6 and 8), the concentration of the 1.1% KOH catalyst (m/m oil) and the temperature 60 °C for 2 hours under moderate stirring (250 rpm). The final mixture was separated in a separating funnel and the ester-rich phase was dried with anhydrous sodium sulphate and filtered through filter paper [17,18].

3.3 Preparation of fatty acid ethyl esters

400µL of KOH (85%, Sigma Aldrich) 2 N solution in ethanol (99.8%) was mixed with 60 mg of oil and the mixture was refluxed at 70°C under vigorous stirring. After one hour of reaction, the mixture was cooled to room temperature. Then 5 ml of n-heptane (Pesti-S grade, Biosolve) and 1 g of sodium sulphate were added to the mixture, vortexed for 10 seconds and left to stand for 20 min. The ethyl esters dissolved in heptane formed the top layer which was separated from the decanted glycerol at the bottom of the screw tube. 100 µL of ethyl esters extracted from the top layer was diluted in 900 µL with n-heptane for analysis [17,18].

3.4 GC/MS analysis conditions

The fatty acid profile of the non-conventional vegetable oil was carried out by Scion 486 GC coupled to BRUKER Daltonic tims TOF HRMS equipped with an electrospray ionisation source (GC-APCI). 1µL of diluted sample (20x in heptane) was injected in split mode (x20) into the GC-TOFHRMS system, on a Restek RXi Sil-5MS

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column 30m long, 0.25mm internal diameter and 0.25µm film thickness. The injector temperature was 260°C. The carrier gas used was helium at a constant flow rate of 4.0 $mL.min^{-1}$.The initial oven temperature was programmed to be 80°C for 1 minute followed by an increase from 25°C per minute to 140°C, then an increase from 20°C per minute to 200°C and finally an increase from 15°C per minute to 310°C with a hold time of 5 minutes. The mass spectrometer worked in scan mode from 100 to 1000 m/z at 3 Hz. The temperature of the transfer capillary was 200°C and the corona needle at 1800nA. Chromatograms and mass spectra are analysed using Bruker Compass Data Analysis V4.3 software [17,18].

3.5 Density measurements

The density (ρ) was measured with an Anton Paar K.G. DMA 45 vibrating tube density meter as a function of pressure and temperature for non-conventional vegetable oil and ethyl biodiesel. An additional DMA 512 cell was adapted to this instrument, which allows pressure measurements up to 400 bar. In this study, the device allowed us to carry out measurements by varying the temperature from 293.15 to 353.15 K and the pressure from 0.1 to 40 MPa. The DMA 45 was connected to an mPDS 2000V3, allowing the vibration period to be measured with some accuracy. A Julabo Polystat 36 thermostat bath is used to control the temperature of the vibrating tube cell. The temperature is measured inside the cell by an AOIP PN 5207 thermometer with an uncertainty of 0.05 K. A positive displacement piston pump is used to apply pressure to the system, measured by an HBM PE 200/2000 transducer with an uncertainty of 0.1%. Before and after each manipulation (sample loading), the densimeter and all capillaries are cleaned with petroleum ether and hexane to remove all traces of residues of the substance previously studied. Once this cleaning process is complete, a vacuum is applied to the system before introducing the sample to be studied. When thermal equilibrium is reached, the period of vibration of the cell is determined at different pressures, starting with 0.1 MPa, followed by the highest pressures. Then the temperature of the liquid bath is changed and a new isotherm is studied [17,18].

3.6 Measurement of the kinematic viscosity

The kinematic viscosity of our samples was determined with a Ubbelohde viscometer from Schott-Geräte. A kinetic energy correction is applied according to the diameter of the capillary tubes used. For this purpose, approximately 15 ml of the filtered sample was introduced into the reservoir (capillary tube). The maximum filling volume is limited by the marks on the tank. After filling, the viscometer is hung with its holder in a transparent thermostat from Schott-Geräte. To avoid measuring errors in the viscometer, the temperature in the thermostat is kept constant at ± 0.01 °C [17,18].

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3. RESULTS AND DISCUSSION

3.1 physicochemical parameters of the vegetable oil and convertibility

Table 1 presented the results of some physicochemical parameters of the vegetable oil extracted from *Chrysophyllum albidum* kernels and the conversion rate of the oil into ethyl biodiesel. Results indicated that seeds of *Chrysophyllum albidum* collected at Benin have a good lipid potential (33.23 ± 3.30%) with a moisture content of 2.64±0.08%. Acid, iodine and peroxide values of the vegetable oil are respectively 3.6 ± 0.23 mg KOH/g-oil, 33.21 ± 0.08 mg I_2/100 g-oil and 10.46 ± 0.12 meq O_2/Kg-oil. Based on these results, this raw material can also be valorized as a good source for vegetable oil and biodiesel production [14]. Audu et al. [19] reported similar values of acid (33.12±0.25 mgKOH/g), iodine (60.05±0.02 mgKOH/g), and peroxide index (2.10±0.10 m Eq/kg) to those found in the present study.

Results also indicated that a good rate of ethyl esters was obtained (96.2% (m/m)) in basic homogeneous catalysis at 333.15 K with a molar ratio (6:1) and 1% (m/m) of the vegetable oil (Table 1). Deng et *al.* [20] reported that esterification catalyzed by an esterifying acid followed by transesterification is the only possible way to obtain a maximum rate of conversion of oil into biodiesel. Similar results were also reported by Anastopoulo et *al.* [21] in the ethanolysis of rapeseed and sunflower oil, as well as in the ethanolysis of peanut oil [22]. The purification of the ethyl esters by using an aqueous solution of phosphoric acid at 1% (m/m) allowed to obtain a biodiesel yield of 86.05%.

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Table 1. Vegetable oil extraction yield, quality indices and conversion rate of vegetable oil to ethyl esters by the combined basic catalysis transesterification reaction

Parameters	Values
Moisture content (%)	2.64± 0.08
Oil yield (%)	33.23 ± 3.26
Acid (mg KOH/g-Huile)	3.6 ± 0.23
Iodine (mg I_2/100 g-Huile)	33.21 ± 0.08
Peroxide (méq O_2/Kg-Huile)	10.46± 0.12
Free Fatty acid content (%)	0.16±0.02
Conversion rate (%)	96.20±0.34
Purification yield (%)	86.45±1.03
Biodiesel (%)	86.05±2.28

3.2 Fatty acid profile

The results of identification of ethyl esters are presented in Table 2. From results it appeared that biodiesel derived from *Chrysophyllum albidum* vegetable oil is rich in mono and polyunsaturated esters. Linolenic (C18:3), linoleic (C18:2) and oleic (C18:1) acids are the major fatty acids. The saturated fatty acids in high proportions are palmitic (C16:0) and stearic (C18:0) acids. Avram et *al.* [21] also reported the presence of oleic acid, linoleic acid, and linolenic acid in oil extracted from rapeseed. GC-MS analysis of fatty acids in soursop seed oil revealed the presence of unsaturated fatty acids (73.42%) and saturated fatty acids (26.68%). From results it appears that the biodiesel from *Chrysophyllum albidum* has useful and similar characteristics to standard biodiesels [23].

Table 2. Ethylic ester composition

Fatty acids	Value (%)
Myristate (C14:0)	2.28
Palmitate (C16:0)	15.98
Stearate (C18:0)	4.19
Arachidate (C20:0)	2.26
Behenate (C22:0)	0.82
Eicosatrienoate (C23:0)	1.68
Lignocerate (C24:0)	1.76
Palmitoleate (C16:1)	1.99
Oleate (C18:1)	19.7
Gondoate (C20:1)	2.02
Erucate (C22:1)	0.50
Linoleate (C18:2)	21.22
Linolenate (C18:3)	25.62

3.3 Kinematic viscosity of vegetable oil and formulated ethyl biodiesel (BE100)

The kinematic viscosity values of vegetable oil and formulated ethyl biodiesel (BE100) are presented in Table 3 and Figure 1. From results, it appears that the kinematic viscosities of the oil and that of the formulated ethyl biodiesel decrease with increasing of the temperature. Indeed, the kinematic viscosity of oils in general is higher than that of petroleum diesel and is around 30 to 40 cSt at 313.15 K. It is known that most stationary (low speed) engines operate with fluids having a viscosity between 13.00 and 17.00 cSt at 313.15 K. Therefore, the use of this vegetable oil directly in engines, requires preheating to reach an acceptable viscosity before its injection into engines.

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Table 3. Kinematic viscosity (mm^2/s) of vegetable oil and ethyl biodiesel (BE100) at atmospheric pressure

Temperature (K)	Vegetable oil	Ethyl biodiesel (BE100)
293.15	88.65	10.36
313.15	37.48	6.40
333.15	19.76	3.58
353.15	12.08	2.75

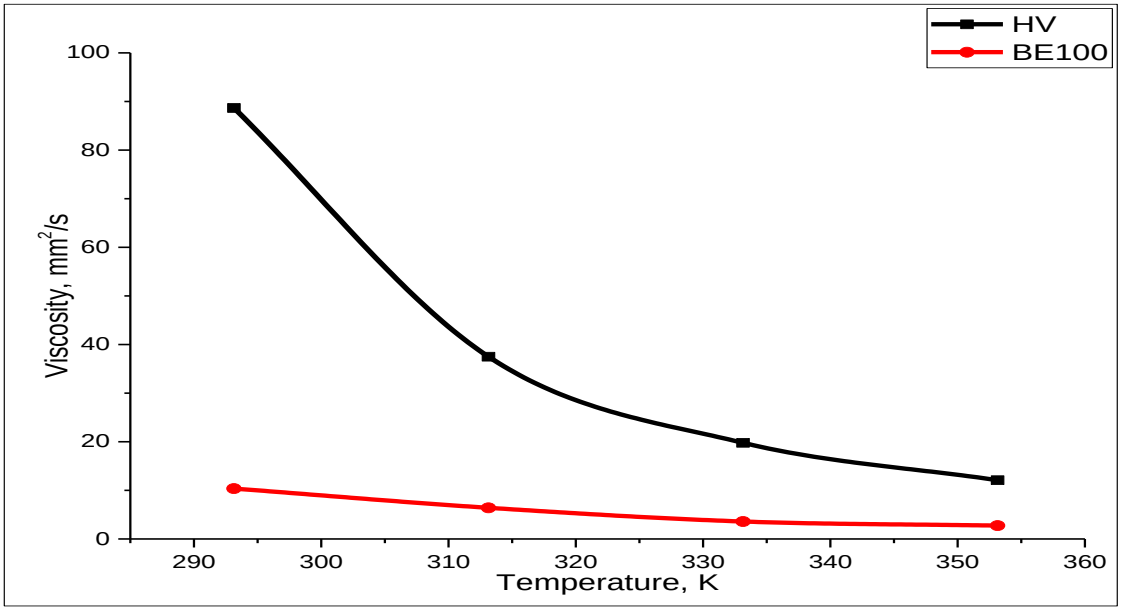

Figure 1. Effect of temperature on the kinematic viscosity of vegetable oil (HV) and formulated ethyl biodiesel (BE100) at atmospheric pressure.

3.4 Influence of fatty acids on the density of vegetable oil and formulated ethyl biodiesel

The results of effect of temperature and pressure on the density of the oil and formulated ethyl biodiesel are presented respectively in Tables 4 and 5. From results, it appears that at constant temperature, the increase in pressure from 0.1 MPa to 40 MPa leads to the compression of samples (HV and BE100) and consequently, the increase in density. On the other hand, when the measurements are made at constant pressure with a variation of temperature, the density of the samples decreases. Figures 2,3 and 4 indicated the effect of temperature on the density of the vegetable oil and the derived biodiesel at constant pressure (0.1 MPa). As expected, the density decreases with the increase of the temperature. Rodríguez-Antón et *al.* [24] reported experimental data on methyl esters of rapeseed and petroleum diesel at atmospheric pressure and 293.15 K. Thus, they reported densities of 883 kg/m^3 for rapeseed methyl esters and 835 kg/m^3 for petroleum diesel. Indeed, density (ρ) is an important property that influences the dissolution of fuel injected into the engine cylinder. In addition, vegetable oils are naturally denser than biodiesel and fossil diesel. In addition, biodiesel is denser and naturally less compressible than fossil diesel. The relatively high density of biodiesel compromises its use as an alternative fuel. The molecular structure and fatty acid composition are responsible to the increase of the density of biodiesel. It appears that biodiesel rich in saturated fatty acid esters is less dense while those with a higher unsaturated fatty acid ester composition are denser. Recent researches on biodiesel derived from vegetable oil from *Ceiba pentandra* and *Afzelia africana* seeds indicated similar results and confirms the general trend regarding the influence of temperature and pressure on density [1, 2]. The source, quality and nature of the raw materials would explain these measurements of density.

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Table 4. Effect of temperature and pressure on the density of vegetable oil

Pressure (MPa)	Temperature (K)			
	293.15	**313.15**	**333.15**	**353.15**
0.1	914.65	902.00	888.47	875.12
10	921.18	907.70	894.63	881.87
20	925.40	913.26	900.79	888.16
30	930.81	918.43	906.03	894.25
40	935.30	923.37	910.94	899.28

Table 5. Effect of temperature and pressure on the density of ethyl biodiesel (BE100)

Pressure (MPa)	Temperature (K)			
	293.15	**313.15**	**333.15**	**353.15**
0.1	908.30	894.11	879.87	866.41
10	913.65	899.71	884.86	873.49
20	918.16	905.21	892.43	880.07
30	921.75	910.53	898.01	886.70
40	927.86	915.61	903.36	892.01

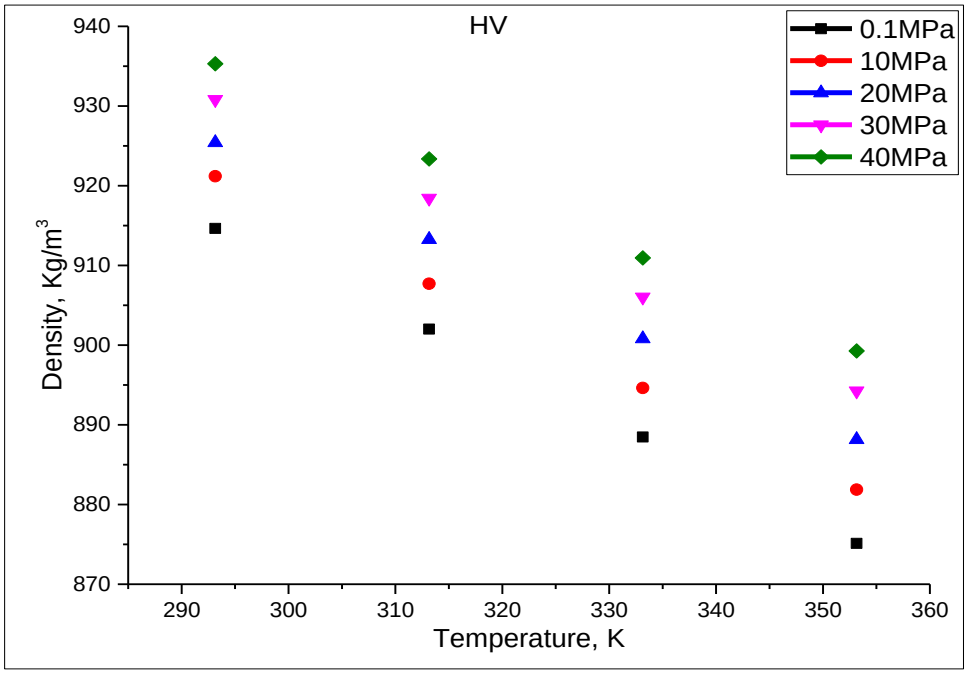

Figure 2. Density variation of vegetable oil (HV) with temperature and pressure

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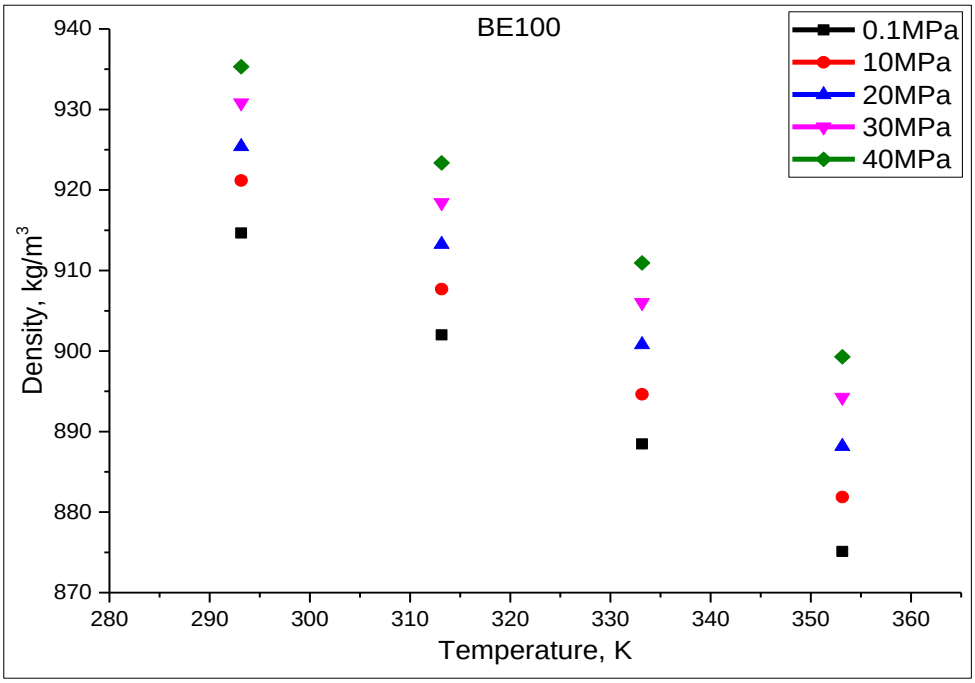

Figure 3. Density variation of the ethyl biodiesel (BE100) with temperature and pressure

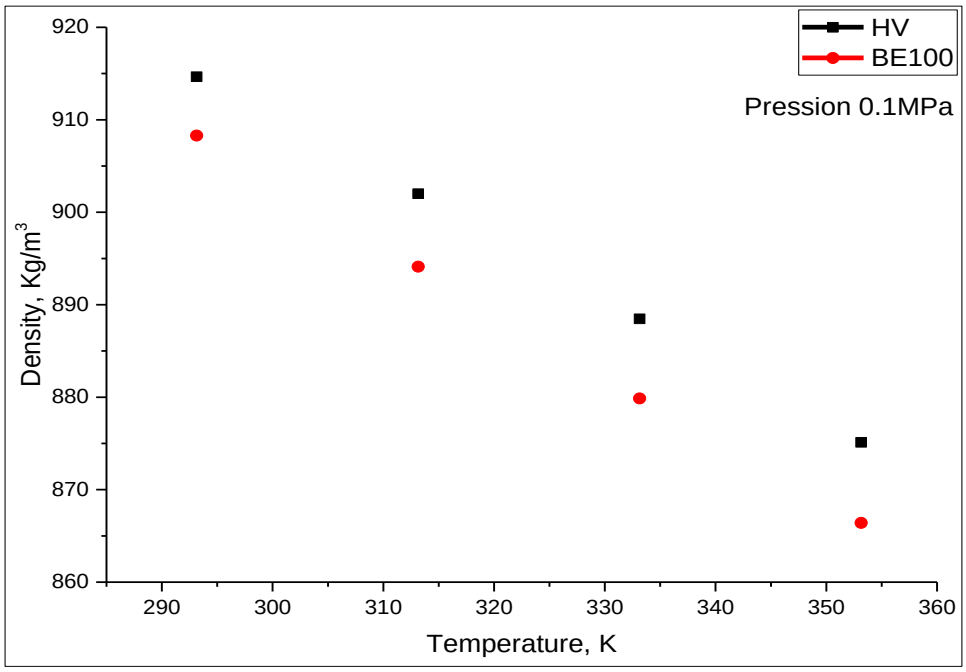

Figure 1. Effect of temperature on density of vegetable oil (HV) and derived ethyl biodiesel (BE100).

3.5 Isothermal compressibility

Table 6 presented the results of the isothermal compressibility coefficient of formulated ethyl biodiesel (BE100) and Figure 5 indicated the variation of the isothermal compressibility coefficient with temperature (293.15 to 353.15 K) and pressure (0.1 to 40MPa). Results indicated that the isothermal compressibility (K_T,) decreases with increasing pressure at constant temperature while it increases with temperature at constant pressure. In general,

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derived ethyl biodiesel (BE100) has a slightly lower isothermal compressibility than petroleum diesel. Results of previous researches on the isothermal compressibility of biodiesel from *Ceiba pentandra* and *Afzelia africana* seeds indicated similar trends as a function of pressure at constant temperature [14,15]. This finding would reflect a better performance of BE100 in diesel engines.

Table 6. Isothermal compressibility (10^3 k_T/ Mpa) of formulated ethyl biodiesel (BE100) as a function of temperature and pressure

Pressure (MPa)	Temperature (K)			
	293.15	**313.15**	**333.15**	**353.15**
0,1	0.621	0.699	0.786	0.884
10	0.588	0.630	0.689	0.762
20	0.558	0.573	0.613	0.670
30	0.531	0.525	0.553	0.598
40	0.506	0.486	0.504	0.541

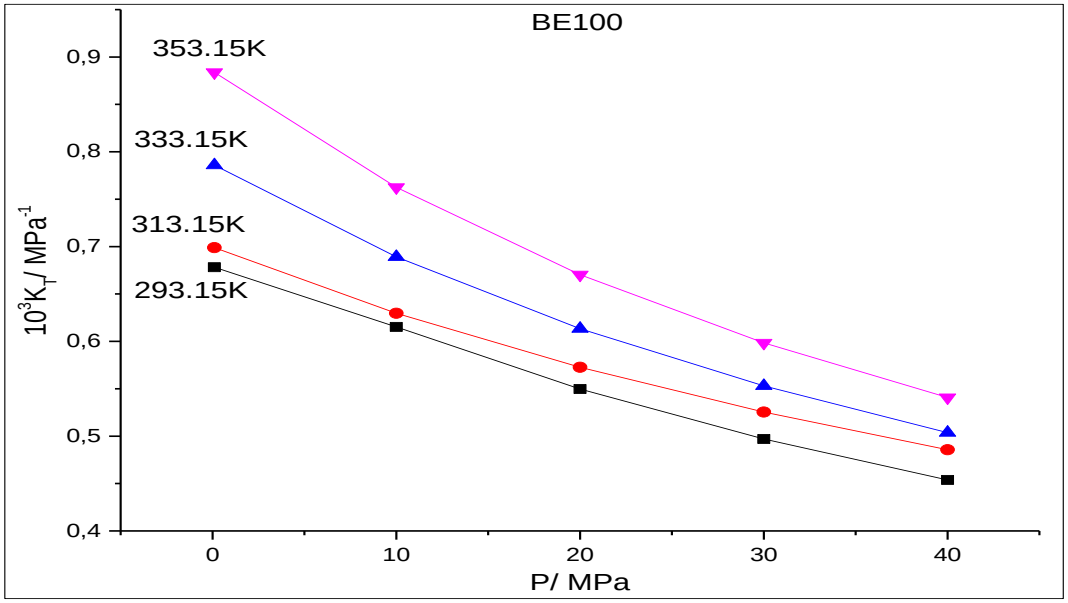

Figure 5. Variation of the isothermal compressibility of BE100 with temperature and pressure

4. CONCLUSION

This study is of major interest in the field of biofuels in order to generate a database of thermophysical and fuel properties in the improvement of diesel engine performance and the behavior of the derived biodiesel formulated in these engines. Thus, the investigated vegetable oil is denser than the derived biodiesel and they are both denser than pure diesel. However, the formulated biodiesel has more interesting properties and is closer to pure diesel. The isothermal compressibility of biodiesel derived from *Chrysophyllum albidum* seed oil increases with temperature at constant pressure and decreases with increasing pressure along the isotherm. Results from this study can be used to reassure present and future users about the use of biodiesel in diesel engines.

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