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Acid-Base Catalyzed Transesterification of *Archontopheonix cunninghamiana* (Bangalow Palm) Seed Oil

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ABSTRACT: Currently, majority of global energy is sourced from petrochemicals which comes with its attendant environmental challenges. Efforts are therefore focused on developing renewable sources for energy and biofuel is in the front burner for these alternatives. In this paper, we explored *Archontopheonix cunninghamiana* seed; an inedible and waste material, as an opportunity for alternative feedstock for biodiesel production. The oil extraction recorded a low yield which may not be economically viable; the transesterification of extracted oil gave 94.56% yield of biodiesel. Physico-chemical analyses of the produced biodiesel showed viscosity of biodiesel to be 2.64 mm^2/s, which is within American Society for Testing Materials (ASTM) and EN590:1999 petrol diesel specifications. It also has a pour point value of 8 °C which is within acceptable range of European Committee for Standardization EN590:2008 for petrol diesel. The flashpoint was found to be 110 °C which make for easy handling and transportation and agrees with EN14214:2008 minimum value of 101 °C. Spectroscopic studies done on the extracted oil and the produced biodiesel showed successful transesterification of the oil and good biodiesel quality.

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Within the last century, population, economic wealth and energy consumption has grown exponentially. Global population was 1.6 billion in nineteen hundred, 2.5 billion half a century later, and more than 7 billion human beings exist today. Farnoosh (2019) stated that wealth, estimated by the gross domestic product in real value, has been multiplied by a factor of 40 over the same period; energy consumption has been both a support and a consequence of this growth, rising from slightly less than 1 billion tons of oil equivalent at the beginning of the century to more than 13 billion tons today. Majority of our energy sources are from petrochemical, coal and natural gas which leave huge carbon footprints on the environment. British Petroleum (2018) in its yearly forecast, concluded that renewable energy is the fastest-growing energy source, accounting for 40% of the increase in primary energy. Bioenergy according to the International Energy Agency (2017) is the largest source of growth in renewable consumption over the period 2018 to 2023. It was responsible for half of all renewable energy consumed in 2017 – providing four times the contribution of solar photovoltaic (PV) and wind combined and will account for 30% of consumption within the period. Biodiesel, a renewable, biodegradable fuel manufactured domestically from vegetable oils, animal fats, or recycled restaurant grease, as an alternative fuel, has many merits. Agawal and Das (2001) listed some of these benefits to include; it's being derived from a renewable, domestic resource, it relieves reliance on petroleum fuel imports, it is biodegradable and non-toxic. Compared to petroleum-based diesel, biodiesel has a more favorable combustion emission profile, such as low emissions of carbon monoxide, particulate matter and unburned hydrocarbons. Carbon dioxide produced by combustion of biodiesel can be recycled by photosynthesis, thereby minimizing the impact of biodiesel combustion on the greenhouse effect. Transesterification is a catalyzed chemical reaction involving the conversion of a carboxylic acid ester into a different carboxylic acid ester. It is a catalytic process of exchanging the organic alkyl groups of plant oil – an ester with the methyl group of methyl alcohol. When an alcohol usually methanol comes in contact with free fatty acids, they bond to form a biodiesel (Vandkata *et al*., 2012). *Archontopheonix cunninghamiana* commonly called bangalow palm belongs to the palmae family and is largely cultivated for its tall, graceful appearance (Figure 1). The fruits produced by *A. cunninghamiana*, although abundant, do not represent a valuable and nourishing food resource for frugivorous birds. According to Mengardo and Pivello (2012), they contain little

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nutritive mass (only about 7% of the fruit dry weight provides any nourishment for pulp consumers) and are of poor nutritional value. Their analyses of the pulp nutritional value of *A. cunninghamiana* fruit revealed low levels of protein (7.83±1.13 mg/g of dry mass) in comparison with the concentrations of lipids and soluble sugars: 45.14±15.08 mg/g and 63.28±9.80 mg/g of dry mass respectively. They posited that *A. cunninghamiana* has phenological and reproductive traits that possibly enhance its ability to disperse into new habitats and to become an invader specie. The fruit (Figure 2) occurs in a drupe of variable size - 400 to 300 g - with a slender, red or yellow exocarp; a farinaceous mesocarp which is variably orange; and a dark and hard endocarp. The size of the seeds (Figure 3) depends on the ecotype: in the cultivated plants they weigh about 4 g, are recalcitrant and take between 45 and 90 days to germinate (Global Invasive Species Specialist Group, 2019). This work reports the extraction of *Archontopheonix cunninghamiana* seed oil and the conversion of same to biodiesel.

Fig. 1: *A. cunninghamiana* tree

Fig. 2: *A. cunninghamiana* fruits

Fig. 3*: A. cunninghamiana* seeds

MATERIALS AND METHODS

Reagents: Analytical grades of absolute methanol, conc. sulfuric acid, potassium hydroxide, glacial acetic acid, iodine, potassium iodide, sodium thiosulphate, chloroform, isopropyl alcohol (all Sigma Aldrich) were used in this research. The n-hexane used was redistilled before use.

Instrumentation: The following instruments were used for analyses in this work; Perkin-Elmer Fourier Transform – Infra Red (FT-IR) spectrum 2 with serial number 97529, Viscometer bath (Stanhope SETA) maintained at 40 °C, Penski Martens closed cup flash point tester, Gas Chromatograph-Flame Ionization Detection (GC-FID) (HP6890) column type HP INNOWax with carrier gas being Nitrogen gas, Hinotek SYD-510D pour and cloud point tester, water bath, rotary evaporator and analytical balance.

The infrared spectra of *A. cunninghamiana* seed oil and produced biodiesel were measured using a PerkinElmer® Spectrum™ Two FT-IR spectrometer. The samples were injected into the empty accessory to obtain the best contact with the crystal and background spectrum. The spectral collection lasted approximately 5 minutes with all spectra recorded within a range of 4000-550 cm^{-1} with a 4 cm^{-1} resolution.

European Union (EU) norm EN 14103 procedure for gas chromatography analyses was used to analyze the biodiesel using a GC–FID HP6890 apparatus (Agilent Technologies, USA) equipped with data acquisition software HP Chemstation Rev.A.09.01 (1206). Separations were accomplished using a 15 m long HP-INNOWAX capillary column, (30 m X 0.25 mm and 0.25 μm film thickness) at a constant hydrogen flow of 22 psi. Sample (1 μL) was injected in a split ratio of 20:1 with an injector temperature of 250 °C. The temperature program of the oven started with an initial

temperature of 12 °C and was followed by an increase in temperature up to 15 °C.

Plant Material: Ripe fruits of *Archontopheonix cunninghamiana* were harvested from Trans Amadi Industrial layout Port Harcourt, Rivers State, Nigeria and were identified by matching against the database of the Invasive Specie Specialist Group (ISSG) and Palmpedia. The seeds were removed from the fruit and air dried. The air-dried seeds of *A. Cunninghamiana* were pulverized and weighed (3.06 kg).

Extraction of oil from the seed: The pulverized seeds was soaked with 2.8 L of n-hexane in an aspirator bottle for 24 hours; the extract was filtered and concentrated using a rotary evaporator. Soaking and filtration processes were repeated 5 times using fresh n-hexane (2 L) each time (Ndukwe *et al*., 2016). Extracted oil was weighed and the percentage yield calculated.

Characterization of the oil obtained from A. cunninghamiana seed:
Acid value and % Free Fatty Acid (FFA) (ASTM D664): In a conical flask containing 20 ml of isopropyl alcohol (IPA), 0.5 g of *A. cunninghamiana* seed oil was dissolved and 3 drops of phenolphthalein indicator added and stirred vigorously. The solution was titrated with 0.1 N potassium hydroxide until a pink color that stayed for 15 sec was observed. The volume of KOH used was recorded as A. The procedure was repeated for the blank starting with IPA and volume of KOH used recorded as B. Acid value and percentage free fatty acid were obtained using equations 1 and 2 respectively.

$$\text{Acid value} = \frac{(A - B) \times N \times 56.1\ g}{W\ (g)} \quad \ldots\ldots 1$$

$$\%\ \text{FFA} = \frac{(A - B) \times N \times 28.2\ g}{W\ (g)} \quad \ldots\ldots 2$$

Where N = normality of the base;
W = weight of oil used

Iodine Value (Association of Official Analytical Chemists (AOAC) Official Methods Cd 11-92): To a 20 ml of chloroform in a conical flask was dissolved 0.5 g of the *A. cunninghamiana* seed oil. 20 ml of potassium iodide solution was added to it followed by 100 ml of distilled water. The resulting solution was placed in the dark for 30 minutes. The solution was titrated with standardized 0.1 M sodium thiosulphate using 1% starch as indicator and introducing 1 ml of starch test solution, stirred continuously till the solution turned blue black and then colourless which marked the end point (V_1). A blank titration (V_2) was also carried out starting with 20 ml of chloroform. Iodine value (IV) was calculated using equation 3.

$$\text{IV} = \frac{(V_2 - V_1) \times N \times 12.69\ g}{W\ (g)} \quad \ldots\ldots 3$$

Where N = normality of $Na_2S_2O_3$

Saponification Value (AOAC Official Methods Cd 3-25): A. cunninghamiana seed oil (0.25 g) was dissolved in a conical flask with 10 ml of 0.5 N ethanolic solution of potassium hydroxide and the solution was refluxed and allowed to cool. Phenolphthalein (3 drops) was added and the solution titrated with 0.5 N hydrochloric acid (V_1). A blank titration (V_2) was carried out as well (AOAC, 1990). Thus, saponification value (SV) was calculated using equation 4.

$$\text{SV} = \frac{(V_2 - V_1) \times N \times 56.1\ g}{W\ (g)} \quad \ldots\ldots 4$$

Where N = normality of HCl

Peroxide Value (AOAC official Methods Cd 8b-90): In a 25 ml mixture of glacial acetic acid and chloroform (2 volume of glacial acetic acid and 1 volume of chloroform) was dissolved 0.5 g of the *A. Cunninghamiana* seed oil and 1 ml of saturated solution of potassium iodide was added, followed by the addition of 7.5 ml of distilled water. The solution was titrated with 0.1 N sodium thiosulphate until yellow color disappeared. 0.5 ml of starch indicator was added to the solution and the titration continued to end point (V_1). A blank (V_2) was carried out as well (AOAC, 1990). Peroxide value was calculated using equation 5.

$$\text{PV} = \frac{(V_2 - V_1) \times N \times 1000}{W\ (g)} \quad \ldots\ldots 5$$

Where PV=Peroxide value (Milleqv. peroxide/kg);
N = normality of $Na_2S_2O_3$

Two Step Acid-Based Catalyzed Transesterification: Acid pretreatment (acid catalyzed esterification): A. cunninghamiana seed oil (200 ml) was measured into a round bottomed flask, stoppered and was heated on a thermostated magnetic stirrer at 55 °C. A mixture of 4 ml of concentrated sulfuric acid and 20 ml of methanol was added to the round bottomed flask containing the oil. The mixture was placed on the thermostated magnetic stirrer maintained at 400 rpm and refluxed for 1 hour. The refluxed mixture was then

poured into a separating funnel and allowed to stand for 2 hours. The aqueous phase contained in the lower layer was tapped off while the pretreated oil was collected for further analysis (Iloamaeke *et al*., 2016).

Base catalyzed transesterification: Acid pretreated *A. cunninghamiana* seed oil (200 ml) was measured and poured into a round bottomed flask which was immersed in a water bath set at 55 °C and allowed to heat up until the temperature of the water bath was attained. KOH (1.08 g) was weighed and added to a conical flask containing 20 ml of methanol and swirled gently until the KOH pellet completely dissolve, thereby, forming methoxide solution. The methoxide solution was added to the heated oil and refluxed for 1 hour at a temperature maintained at 55 °C. The refluxed mixture was poured into a separating funnel and allowed to stand for 1 hour for separation to occur. The lower layer (glycerol layer) was tapped off and the upper layer (biodiesel) was washed 5 times with warm distilled water. The weight and volume of the obtained biodiesel was measured and recorded.

Characterization of Produced Biodiesel:
Density Measurement (ASTM D445-12): An empty beaker was weighed using an analytical weighing balance. The produced biodiesel (30 ml) was then poured into the beaker, and the combined weight measured. The difference between the weight of the beaker plus biodiesel and that of the empty beaker was obtained and recorded as the weight of the oil. The density was obtained by taking the ratio of the weight of the biodiesel and its volume (Equation 6).

$$\text{Density of biodiesel} = \frac{\text{WBD}}{\text{VMD}} \quad \ldots\ldots 6$$

WBD = Weight of measured biodiesel;
VMD = Volume of measured biodiesel

Kinematic Viscosity Measurement (ASTM D445): The produced biodiesel (10 ml) was poured into a viscometer tube. The tube was then immersed into a viscometer bath maintained at 40 °C. The oil in the tube was sucked up to the upper limit mark using a suction pump and allowed to drop under gravity. A stopwatch was started and the set up monitored till the oil got to the lower limit of the tube and the watch stopped. The time was recorded, and the procedure repeated twice. Kinematic viscosity was calculated using equation 7.

$$\text{KV @ 40 °C } (\text{mm}^2/\text{s}) = \text{T (s)} \times \text{TC} \ldots 7$$

Where T = time and TC = Tube constant

Flash Point (ASTM D93): Flash point of the biodiesel was measured using Pensky-Martens closed cup apparatus. The produced biodiesel was poured into the brass cup to touch the prescribed mark inside the cup and then gently placed into its position until it locked. A thermometer was placed inside through the provided opening. At intervals, lighted flame was passed through the sample in the cup to check for flash, while temperature was being monitored. The temperature at the first distinct flash was taken and recorded. This gave the closed flash point of the biodiesel.

Pour Point (ASTM D6892-03, 2014): The biodiesel in a test jar with a thermometer clamped to it was cooled inside a constant temperature cooling bath, as it cooled it formed wax crystals. The test jar was removed at every degree drop in temperature and tilted to check the surface movement. When the surface did not flow for 5 seconds, the temperature was recorded.

RESULTS AND DISCUSSION

Summary of the physico-chemical properties of *Archontopheonix cunninghamiana* seed oil is presented in table 1. *A. cunninghamiana* seed yielded 3.39% of oil which is low to be explored economically. It has acid value of 13.49 mgKOH/g and %FFA of 6.78. On further pre-treatment with sulfuric acid, the acid value dropped to 1.089 mgKOH/g which is within the permissible limit. According to Iloamaeke *et al*. (2016) higher amount of FFA can lead to emulsification and presents a great difficulty during separation of the biodiesel from glycerol and its washing.

Table 1: Physicochemical properties of *A. cunninghamiana* seed oil

Property	Unit	Value
Color		Orange
Appearance		Liquid
% Yield	%	3.39
Saponification value	mgKOH/g	335.50
Peroxide value	Millieqv. peroxide/kg	205.30
Iodine value	g I_2/100g of oil	112.63
Density @ 25 °C	g/cm^3	0.8589
Pour point	°C	6
Acid Value	mgKOH/g	13.49
% FFA	%	6.78

Table 2 presents the physico-chemical properties of produced biodiesel in comparism with European biodiesel standard EN 14214:2008 and American biodiesel standard as contained in ASTM D6751-07b. A very significant yield of 94.56% was recorded from the transesterification of *A. cunninghamiana* seed oil using potassium methoxide. The European standards for biodiesel EN 14214:2008, outlined the standard density of biodiesel to range from 0.86-0.9 and that for petroleum diesel was pegged from 0.82-0.845. The density values obtained for *A. cunninghamiana*

biodiesel is 0.9091 which is within the above standards. Kinematic viscosity affects the flow properties of the fuel which has impact on the size of the droplets of the fuel during fuel injection. Higher viscosity fuels form larger droplets leading to incomplete combustion of the fuel and exhaust emission become more (Gaonkar and Vaidya, 2016). According to Gerpen (2005), if fuel viscosity is extremely excessive, there will be a degradation of the spray in the cylinder causing poor atomization, contamination of the lubricating oil, and the production of black smoke. Consequently, the kinematic viscosity @ 40 °C of the biodiesel from *A. cunninghamiana* which is 2.64 mm^2/s, is within the EN 14214:2008 and ASTM D6751-07b standards of 3.5-5.0 mm^2/s and 1.9-6.0 mm^2/s respectively. Flash point according to István and Ioan-Adrian (2011), classifies fuels for transport, storage and distribution according to hazard level. Flash point does not affect the combustion directly; higher values make fuels safer with regard to storage, fuel handling and transportation. From table 2, the flash point of biodiesel produced was 110 °C which is within the standard range for biodiesel cited above. The value was higher than that of Petro diesel (55 °C) and this makes it more stable to fire. This flash point gives an indication that the biodiesel produced is not highly flammable but would require safety precautions like any fuel during usage, storage and transportation.

Table 2: Physicochemical properties of Biodiesel from *A. cunninghamiana* seed

Property	Unit	Produced Biodiesel	Standards		
			Biodiesel EN14214: 2008	ASTM D6751-07b	Petrodiesel EN 590:1999
Color		Orange			
Appearance		Liquid			
% Yield	%	94.56			
Flash point	°C	110.0	101	93	55
KV @ 40 °C	mm^2/s	2.64	3.5 - 5.0	1.9 - 6.0	2.0 - 4.5
Density @ 25 °C	g/cm^3	0.9071	0.86 - 0.9		0.82 - 0.845
Pour Point	°C	8		-5 -10	5.6 - 11.1

In table 3, the fatty acid alkyl ester composition of the biodiesel produced using potassium methoxide catalyst is outlined. Gas chromatography analysis using Flame Ionization detection (GC-FID) technique carried out on the produced biodiesel showed that the product from *Archontopheonix cunninghamiana* seed oil consists of high percentage of oleic acid methyl ester (48.26%), followed by linoleic acid methyl ester (23%) which has some levels of unsaturation, and palmitic acid methyl ester (15%). High values of unsaturated fatty acid methyl ester (FAME) components in biodiesel make for lower cetane number according to Altun (2014). He posited that higher cetane number tends to reduce NOx emission. There are also other fatty acid methyl esters in low concentrations.

Table 3: GC-FID result of produced biodiesel

Retention time (min)	Compound	Concentration (%)	No of C-atoms: double bonds
16.040	Palmitic acid methyl ester	15.065	C16: 0
18.057	Stearic acid methyl ester	7.851	C18: 0
18.937	Oleic acid methyl ester	48.263	C18: 1
19.522	Linoleic acid methyl ester	23.530	C18: 2
20.654	Linolenic acid methyl ester	1.055	C18: 3

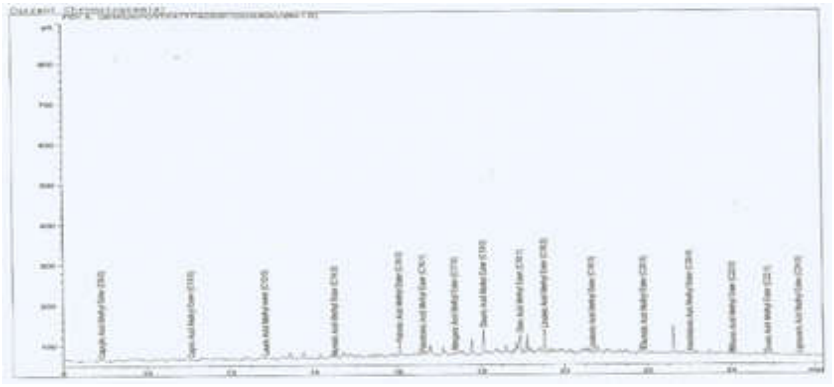

Fig. 4: GC-FID Spectrum of produced biodiesel from *A. cunninghamiana* seed oil

The result of FT-IR analyses (Figures 5 & 6) as presented in table 4 showed a strong absorption peak at 1735 cm^{-1} (in both the oil and the biodiesel) characteristic of C=O stretching of esters, as well as strong absorption at 1480 cm^{-1} attributed to CH_2 bending vibration. Tariq *et al*. (2011) posits that esters have two characteristically strong absorption bands arising from carbonyl (C=O) around 1750-1730 cm^{-1} and that of C-O (antisymmetric axial stretching and asymmetric axial stretching) at 1300-1000 cm^{-1}. According to Sabrina *et al*. (2015), the fingerprint region which lies from 1500-900 cm^{-1} is the main spectrum region that allows for distinction between the oil and its respective fatty acid alkyl ester. In the produced biodiesel spectrum (Figure 6), there are marked absorptions at 900 cm^{-1} attributed to asymmetric stretching of $-CH_3$ and 1050 cm^{-1} assigned to asymmetric axial stretching vibrations of O-C-C bonds, which are absent in the oil spectrum.

Table 4: Comparative FT-IR of *A. cunninghamiana* seed oil and produced biodiesel

Peak (cm^{-1})	Functional group (Oil)	Functional group (Biodiesel)
3450	O-H (stretching)	O-H (stretching)
2850	C-H (single bonded)	C-H (single-Bonded)
1735	C=O (esters)	C=O (esters)
1480	CH_2 (bending)	CH_2 (bending)
1050	C-O	O-C-C (asymmetric axial stretching)
900		$-CH_3$ (asymmetric stretching

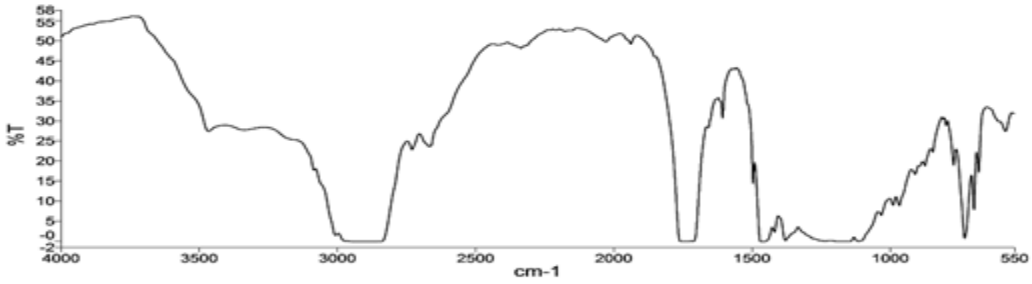

Fig. 5: FT-IR spectrum of *A. cunninghamiana* seed oil

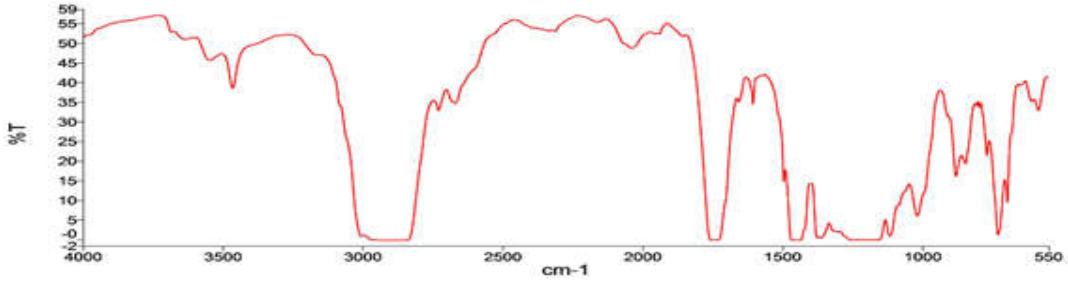

Fig. 6: FT-IR spectrum of produced biodiesel from *A. cunninghamiana* seed oil

Conclusion: *Archontopheonix cunninghamiana* seed recorded a low oil yield and very high biodiesel yield. Results of analyses done on the final product showed a successful transesterification of the oil from *Archontopheonix cunninghamiana* seeds. This was made possible by acid pretreatment which reduced the acid value of the oil significantly to enable successful transesterification. This seed can serve as alternative feedstock for biodiesel however, it is not commercially viable to do so due to its low oil yield.

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