Vol. 9(1), pp. 1-6, May 2018
DOI: 10.5897/JPTAF2018.0136
Article Number: 841A23356999
ISSN 2360-8560

http://www.academicjournals.org/JPTAF

ACADEMIC JOURNALS
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Journal of Petroleum Technology and Alternative Fuels

Full Length Research Paper

Biodiesel potentials of two phenotypes of *Cyperus esculentus* unconventional oils

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Received 23 January, 2018; Accepted 23 April, 2018

Two varieties of *Cyperus esculentus*, identified by their phenotype (black or yellow) were collected from Benin. To investigate their biodiesel profiles, the roots were extracted with hexane. Physicochemical properties and fuel profiles of obtained oils (YCE and BCE, respectively *Yellow C. esculentus* and Black *C. esculentus*) were determined by following standards methods. Although the major physicochemical properties (Iodine value: YCE: 72.89± 0.00 vs BCE: 79.73± 0.00 g I_2/100 g-oil and Saponification value: YCE: 178.54 ± 0.6 vs BCE: 178.09 ± 0.13 mg KOH/g-oil) were relatively closed, slight difference were observed with their fatty acids profiles. Major fatty acids were oleic acid (YCE: 63.50 ± 0.15 vs BCE: 63.42 ± 0.11 g / 100 g- oils) and palmitic acid (YCE: 16.50 ± 0.26 vs BCE: 15.94 ± 0.11 g / 100 g- oils). These profiles should be correlatived with the density (≈0.89 g.cm^{-3} at 40°C), the viscosity (≈24 mm^{2}s^{-1} at 40°C) and the cetane number (YCE: 60.47vs BCE: 59.01) of the oils. Their calorific values (YCE: 36361vs BCE: 37452 KJ/Kg) indicate that they could be used for biodiesel purposes. However, *C. esculentus* oils should be treated before their conversion in biodiesel in order to reduce their high level in minerals.

Key words: *Cyperus esculentus*, unconventional oils, fuel profiles, physicochemical properties, phenotype.

INTRODUCTION

The oil shock of the 70 years revealed that an economical growth founded on the fossil energy abundance alone should not be done continually. Besides, the use of fossil energy generates environmental problems (Mecher et al., 2006; Goldemberg, 2008; Balat and Balat, 2009). As alternatives, biofuels are elected today due to their biodegradability, the easy reproducibility of their production techniques and the highest of their lubricating properties (Steenblik, 2006).

In Africa, the use of biofuels is known. In the beginning of the Second World War, to avoid the restocking difficulties in classic fuels, the Society of Construction of the Abidjan's harbor (Ivory Coast) used close to 100 tons of palm oils in powerful motors (Cirad, 2008). However, the vegetable oil used without a previous refinement could damage the motors. Indeed, in the rooms of

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combustion, the vegetable oils undergo physicochemical changes which explain the formation of undesirable matters such polymerized fatty acids and carbon (Kulkarni and Dalai, 2006). A better choice of the vegetable oil made by following their physicochemical properties is therefore important.

From the North to the South of Benin, the tubers of *Cyperus esculentus* are harvested. The cakes are used like fuels. The phenotype of this species depends on the zone of culture. So, in the South of Benin (Ouedeme-Adja) the phenotype of *C. esculentus* is yellow dominance while in Mallanville (in the North of Benin), these tubers are valued for their black color.

The fatty acid profile of *C. esculentus* vegetable oils have been described to be highly oleic (65.6%) (Kim et al., 2007). *C. esculentus* vegetable oils present very good biofuel profile for the diesel motors (Zhang et al., 1996; Barminas et al., 2001) in relation to their density closed to diesel oil (in 40°C) (Wauquier, 1994) and their high caloric value, superior to 35000 kJ/kg (CIRAD, 2014).

To our knowledge, no study has been done on the biofuel profile of *C. esculentus* tubers harvested in Benin. So, this work aims to evaluate the physicochemical and the biofuel profiles of two species of *C. esculentus* harvested in Benin.

MATERIALS AND METHODS

Samples collection

Fruits of black variety of *C. esculentus* were collected in *Ouedeme* (Southern of Benin) otherwise the yellow variety was collected in Mallanville (Northern of Benin). Once collected fresh, the tubers are manually sorted, washed, sun-dried and stored in a dry place.

Oil extraction

Oil extraction from the *C. esculentus* tubers has been done for 6 h with Soxhlet apparatus by using Hexane at 69±1°C under atmospheric pressure.

Physicochemical properties

The water content, density and viscosity were determined according to the standardized methods (DIN EN ISO 12937, NF T 60-214, NF ISO 3104).

The acid, peroxide and saponification index of the oils were determined following the French standards T 60-204; T 60-220 and T 60-206. The iodine value was evaluated by the Winkler method.

The calorific value was determined by the method of Batel et al. (1980) and the cetane number was calculated from the Klopfenstein equation (1982).

Mineral elements

The mineral elements contents of *C. esculentus* oils were dosed according to the dry procedure by Inductive Coupled Plasma (ICP) on a Varian-Vista device equipped with the Coupled Charge Device (CCD) detector.

Fatty acids contents

To determine the fatty acid composition, 1 µl of a hexane solution of methyl esters was injected into an Agilent 6890 HP GC series (Agilent, USA) equipped with an Innowax (Agilent, USA) type column, 30 m long, 0.32 mm internal diameter having a film thickness of 0.25 µm. The injector was in split mode, ratio 1/80 at 250°C as temperature. The carrier gas was helium with a flow rate of 1.5 $ml.min^{-1}$.

RESULTS AND DISCUSSION

Oil extraction yield

Table 1 shows the moisture and oil content of *C. esculentus* tubers. The black variety contains up to four times more moisture than the yellow one (1.25 ± 0.12%). The oil contents of both varieties are relatively close to 28%. According to the variability observed between oil and moisture contents, it would be interesting to evaluate the impact of roasting on the yield of *C. esculentus* tubers oils. Studies have shown that the yield of seed oil may depend on several parameters, including the edaphic conditions of culture and the morphology of the matrices studied (Abaza et al., 2002). Nevertheless, the oil content of *C. esculentus* from our studies is higher than 15.9% found for *C. esculentus* tubers harvested in Ghana (Yeboah et al., 2012).

Physicochemical properties

Iodine value

The iodine value reflects the unsaturation of the oil. The more the oil is unsaturated the higher its iodine number is. According to the results recorded in Table 2, the iodine numbers determined are in the order of 72.89 g I_2/100 g-oil for the yellow variety and 79.73 g I_2/100 g-oil for the black variety.

These values are slightly lower than the data (88 - 90 g I_2/100 g-oil) reported by Barminas et al. (2001) on *C. esculentus*, tubers harvested in Nigeria. They are even lower (113 g I_2/100 g-oil) than that of the same species studied in Egypt (Arafat et al., 2009).

However, the iodine values of *C. esculentus* from our work comply with the limit (120 g I_2/100 g-oil) set by EN 14214 standards (CIRAD, 2014) and corroborate the composition of these oils, which are rich in unsaturated fatty acids (studied below). This could justify the fluidity of *C. esculentus* oils (Saillard, 2012).

Saponification values

Saponification values (≈178 mg KOH/g-oil) indicate that

Table 1. Moisture content and volatile matter (Te) and oil extraction yield of nutgrass tubers (Th).

Harvest area	Yield		
	Variety	Te (%, g/g)	Th (%, g/g)
Ouedeme	BCE[a]	5.50±0.76	28.00±0.01
Malanville	YCE[b]	1.25±0.12	27.49±0.89

[a]YCE: Yellow *Cyperus esculentus;* [b]BCE: Black *Cyperus esculentus.*

Table 2. Physicochemical properties and fatty acids profile of vegetable oils.

Physicochemical property	YCE[a]	BCE[b]
Peroxide value (meq0_2/kg-oil)	8.75 ± 1.63	7.95 ± 0.10
Iodine value (g I_2/100 g-oil)	72.89± 0.00	79.73± 0.00
Saponification value (mg KOH/g-oil)	178.54 ± 0.6	178.09 ± 0.13
Palmitic acid (C16 :0)	16.50 ± 0.26	15.94 ± 0.11
Stearic acid (C18 :0)	06.87± 0.08	09.84 ± 0.05
Oleic acid (C18 :1)	63.50 ± 0.15	63.42 ± 0.11
Linoleic acid (C18 :2)	12.70 ± 0.07	09.01 ± 0.03

[a]YCE: Yellow *Cyperus esculentus;* [b]BCE: Black *Cyperus esculentus*.

there is no significant difference between the two varieties (Table 2). These values are lower than 209 mg KOH/g-oil, of the vegetable oil of *C. esculentus* harvested in Egypt (Arafat et al., 2009). They are similarly lower than those 196 and 208 mg KOH/g-oil, of vegetable oil of *Jatropha curcas* conventionally used for biodiesel (Kpoviessi et al., 2004). This could reflect a low content of our samples in short chain fatty acids (Ferhat et al., 2014).

Peroxide values

The peroxide values determined for the vegetable oils of *C. esculentus* are 8.75 meq 0_2 / kg-oil for the yellow variety and 7.95 meq 0_2 / kg-oil for the black variety (Table 2). They are below the limit of 10 meq 0_2 / kg-oil, which is the maximum level allowed for most conventional oils (Ferhat et al., 2014). The analysis of these values shows that the oils did not undergo major degradation by oxidation (Abdelaziz and Djamila, 2016).

Fatty acids composition

Fatty acids influence the fuel properties of the oil such as viscosity, oxidation stability, cetane number, exhaust emissions, heat value, cloud point, pour point, etc (Bello et al., 2012). The compositions (in g / 100 g- oils) of fatty acids are presented in Table 2. The unsaturated fatty acids are oleic acid (YCE: 63.50% and BCE: 63.42%) and linoleic acid (YCE: 12.70% and BCE: 09.01%). The particularly high proportions of oleic acid indicate that these oils are stable to oxidation and can be stored for a long time (Lamaisri et al., 2015). However, Gopinath et al. (2010) found that high unsaturation of the oil could reduce the cetane number of its biodiesel. The cetane number is related to the ignition delay. It is high with a decreasing length of the carbon chain (Islam et al., 2013). The proportions of palmitic acid (YCE: 16.50 ± 0.26%, BCE: 15.94 ± 0.11%) and stearic acid (YCE: 06.87 ± 0.08%, BCE: 09.84 ± 0.05%) slightly different from the two varieties, can give a similar view of their cetane number (Table 3).

Fuel properties of vegetable oils

Water content, acid number, density, viscosity, cetane number, phospholipids, Na+K, Ca+Mg and calorific value of the oils were determined. The results are presented in Table 3.

Water content, density and viscosity

The water content (%, wt) of both oils (YCE: 0.06 and BCE: 0.05) are lower than those of *Terminalia belerica* (0.15%) and *J. curcas* (0.17%) seed oils reported by Sarin et al. (2010).

It was proved that the richness of oils in monounsaturated fatty acids such as oleic acid increases

Table 3. Fuel properties of two varieties of *Cyperus esculentus* vegetable oils compared to vegetable oil of rapeseed and biodiesel.

Fuel property	Unconventional vegetable oils studied		Pre-Standard DIN 51605 of rapeseed oil[c]	Biodiesel standards ASTM D 6751 / EN 14214 biodiesel[c]
	YCE[a]	BCE[b]		
Water content (%)	0.06	0.05	Max : 0.075	Max :- / 0.05
Acid value (mg KOH.g^{-1})	1.77	1.56	Max : 3	Max : 0.5
Density (g.cm^{-3}, 40°C)	0.896	0.898	0.9 – 0.93	-/ 0.86 – 0.9
Viscosity (mm^{2}s^{-1}, 40°C)	23.91	24.43	-	1.9 – 6 / 3.5 - 5
Cetane number	60.47	59.01	Min : 39	Min : 47 / 51
Phospholipids (ppm)	23.00	25.00	Max : 12	Max : 10
Na + K (ppm)	101.20	112.30	-	Max : 5
Ca + Mg (ppm)	110.00	99.00	Max : 20	Max : 5
Calorific value (KJ.kg^{-1})	36361	37452	37400 [c]	Min : - / 35000

[a]YCE: Yellow *Cyperus esculentus;* [b]BCE: Black *Cyperus esculentus*; Min: minimum limit value; Max: maximum limit value; [c].
Source: CIRAD (2008).

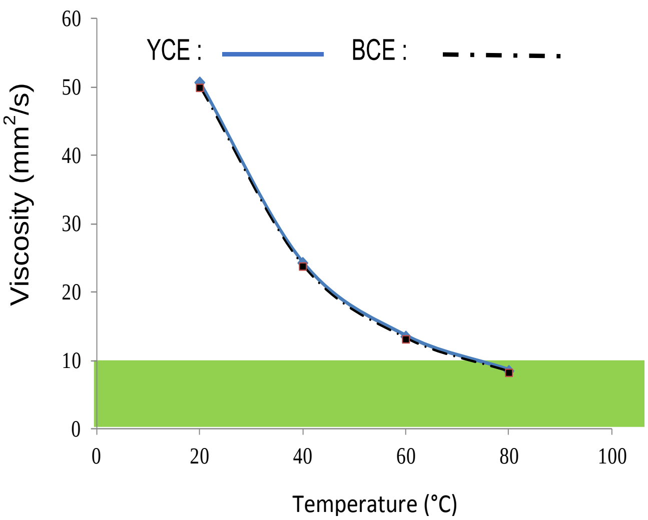


Figure 1. Influence of temperature on the viscosity of vegetable oils of YCE and BCE.

its viscosity (Fasina et al., 2006). According to biodiesel standards ASTM D 6751 and EN 14214 biodiesel, the viscosities (≈24 mm^{2}s^{-1} at 40°C) and densities (≈ 0.89 g/cm^3 at 40°C) are quite high. High level of viscosity should create problems of pumping and of out-flow through the hoses and the filters. However, at 40°C the viscosities are lower than most of unconventional seed oils such as Jatropha: 37.0 mm^{2}s^{-1}; Sunflower: 33.9 mm^{2}s^{-1}, Soybean: 32.6 mm^{2}s^{-1} and Rapeseed: 37.0 mm^{2}s^{-1} (Sarin et al., 2010).

The viscosity of oils is meaningfully more elevated than the viscosity of diesel in ambient temperature. Chalatlon et al. (2011) proposed a reduction of this viscosity by heating the oils.

The variation of viscosities of *C. esculentus* oils with the temperature (Figure 1) in a view to identify the appropriate heating temperature of the oils has been evaluated.

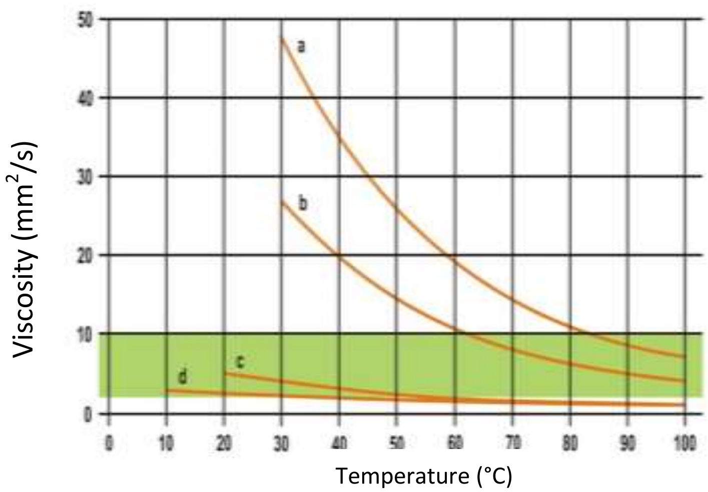
The two curves (YCE and BCE) juxtapose themselves. This means that YCE and BCE oils have the same behavior when the temperature is increasing.

The viscosity of *C. esculentus* oils decreases, by following an exponential equation: $y = 86.603e^{-0,031x}$ (where y is kinematic viscosity and x, the temperature) with increasing temperature.

Figure 2 is the CIRAD standard curves applying for Colza oils, Gasoil half blend, Biodiesel and Gasoil. The green zone represents the zone of normalized viscosity. By following this standard, we identify the minimal heating temperature for *C. esculentus* oils as 70°C (9.89 cst).

Cetane number and calorific value

The cetane number of our studied oils are 60.47 and



a = rapeseed oil, b = Gasoil half Blend, c = Biodiesel, d = Gasoil

Figure 2. Viscosity of fuel as a function of temperature.
Source: CIRAD (2008).

59.01 respectively for YCE and BCE. Srivastava and Prasad (2000) reported lowest cetane number (< 39) for linseed, rapeseed, soya bean and Babassa seed oils harvested in India (Srivastava and Prasad, 2000).

Vegetable oils studied have a calorific value of 36361 kJ/kg for the yellow variety of *C. esculentus* and 37452 kJ/kg for the black variety. These values are higher than the minimum predicted by the German pre-standard (35000 kJ/kg) for a biodiesel (CIRAD, 2014) but lower than that of *C. esculentus* from Nigeria studied by Barminas et al. (2001) which is 40700 kJ/kg.

Acid value

The acid value of a vegetable oil is a function of its free fatty acids and characterizes the state of alteration of the oil by hydrolysis (Bettahar et al., 2016). The acid values (mg KOH/g) for the two oils (< 2 mg KOH/g-oil) are below the limit of 3 mg KOH/g-oil for rapeseed and slightly exceed 0.5 mg KOH/g-oil fixed for the reference biodiesel (CIRAD, 2014). These acid numbers, however, remain lower than those of *J. curcas* oils from eight localities in Benin studied by Kpoviessi et al. (2004).

Phospholipid, (Na + K) and (Ca + Mg) content

The phospholipid content (>20 ppm), the Na + K content (>100 ppm) and the Ca + Mg content (>99 ppm) are extremely high for both studied oils. Their values pass the data foreseen by the norms.

The oils should be refined through several processes to decrease these values. Cuvelier and Maillard (2012) proposed chelating agents such as EDTA (Ethylene-Diamine-Tetra-Acetic acid) and citric acid to reduce metallic cations in matrices.

In the same way, in spite of Argemone seed oils having high toxicity and viscosity, Pramanik et al. (2012) revealed that the fuels properties of their oils were closer to those of traditional transesterified biodiesel. They proposed several processes to lower the bothersome parameters of Argemone seed oils.

Conclusion

The vegetable oils extracted from the two varieties (yellow and black) of *C. esculentus* tubers presented quite physicochemical and biodiesel profiles. The properties such as water content, cetane number and calorific value have been seen better than those of which were related for unconventional seed oils such as Argemone, *Terminalia belerica*, linseed, rapeseed, soy bean and Babassa.

However, by varying the viscosities of the oils depending on the temperature, we reveal that the two species had an identical behavior. These vegetable oils can be used as biofuel if they were preheated to 70°C, at least. In the same way, their use as biofuel requires a pretreatment considering the high level of their mineral compounds. The biofuel potentials of these refined oils and their blend are in progress.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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