

Biodiesel Synthesis by Ethanolysis of *Hura crepitans* Seed Oil Unfit for Consumption in Benin

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Abstract: To reduce fossil fuel dependence and greenhouse gases, biomass energy is in high demand. *Hura crepitans* (HC) is a widely distributed plant species in Benin. But its seed oils are reputed to be purgative and unfit for consumption. So, we collected the seeds of HC in Agame (South of Benin). They were extracted and the seed oils have been converted into biodiesel. First, the quality indices (acid, peroxide, iodine and saponification) were determined. Then, elementary physicochemical parameters and fuel properties of the extracted oil have been highlighted according to standardised methods. Transesterification parameters of the seed oils (alcohol/oil and catalyst/oil ratios, temperature and yield) were also studied. The fatty acids of vegetable oil and the characteristics of its obtained biodiesel were finally identified. It appears that HC seeds have a lipid potential of 52.54%. Its oil is unsaturated and dominated by linoleic acid (54.13%). The yield of the transesterification reaction is 81.47%. The fuel parameters of the obtained biodiesel are: acidity (0.41%); density at 26°C (0.887); cetane number (54.44) compared to those of HC seed oils: acidity (4.81%), density at 26°C (0.929) and cetane number (44.53). The biodiesel obtained by transesterification with potash has much better parameters that comply with biodiesel standards. These results suggest that biodiesel of HC could be proposed to power Diesel engines without a preheating system.

Keywords: *Hura Crepitans*, Transesterification, Biodiesel, Fuel Parameters

1. Introduction

Throughout the world, the industrial revolution has given a prominent place to energies such as nuclear, fossil, natural gas and coal [1]. In order to anticipate the decline of these energies and reduce greenhouse gases, the exploration of renewable energies, particularly biodiesel, has been launched. Despite of the high cost of their production, food and arable land competitions, biofuels remain a credible alternative source because they are renewable [2-4]. Indeed, with their biodegradability and low toxicity to animal species and micro-organisms, they present little or no environmental risk

unlike fossil oil [5]. A wise choice of unused or poorly consumed forest oil resources is necessary to limit controversy. However, whatever their origin, the combustion of crude vegetable oils produces oxygenated hydrocarbons at low temperatures, which leads to the thermal braking of diesel engines. They undergo physical and chemical changes in the combustion chambers, especially in engines, with the formation of gums, triglycerides and a large amount of carbon, etc. on metal parts, which reduces their volatility [6, 7]. So, it is necessary to proceed its transformation into biodiesel. Several conversion methods such as: transesterification, interesterification, microemulsification

and pyrolysis are possible [8-10]. Of all these processes, transesterification is the most widely used because of its simplicity, the best yields it offers and the quality of the biodiesel obtained [11]. This reaction is influenced by several factors whose control is essential to optimize performance. These factors include: alcohol/oil and catalyst/oil ratios; reaction temperature; water content of vegetable oil and alcohol; reaction time; amount of free fatty acids; and agitation of the reaction medium. To produce biodiesel that is 100% renewable in origin, vegetable oil ethyl esters are preferred. Indeed, it can be obtained by using bioethanol from sugar biomass or lignocellulosic biomass and are less toxic than methanol [9, 10, 12]. Benin is full of oilseed raw materials with little or no value due to the lack of chemical and technical knowledge. Present study aims to produce biodiesel from unconventional vegetable oil of *Hura crepitans* harvested in Benin.

2. Material and Methods

2.1. Plant Material

The plant material investigated consists of *Hura crepitans* seeds called bombardier in French. In Beninese vernacular languages, the tree is known as: “Wuntin” in Fongbe and “kekefotin” in Goungebe. The seeds are collected at “Agame” (southern Benin) and shelled. The almonds are sun dried, crushed and the flour obtained is sieved with a sieve of 0.8 or 1 μm diameter and stored at 40°C in the oven until the end of the extraction.

2.2. Extraction of Vegetable Oil

The extraction of vegetable oil was carried out under atmospheric pressure at $69 \pm 1^\circ\text{C}$ for 6 hours with Soxhlet using hexane according to the standard ISO 659 method (1989).

2.3. Determination of Physicochemical Properties of Seed Oil

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

The acidity, peroxide and saponification value (SV) are determined respectively according to French standards T 60-204; T 60-220 and T 60-206. The iodine value was evaluated by the Winkler method.

The lower calorific value (1) of the crude oil in kJ/kg was calculated by the approximate formula [13] using the previously determined iodine value (IV) and saponification value (SV).

$$\text{LCV} = 47645 - 4.187 \text{ IV} - 38.31 \text{ SV} \quad (1)$$

The cetane number (2) was calculated by the Klopfenstein formula [13]. This formula uses the value of the saponification index of the extracted vegetable oils.

$$\text{CN} = 46.3 + \frac{5458}{\text{SV}} - 0.225 \text{ IV} \quad (2)$$

The refractive index (3) is determined using the Perkins mathematical formula reported by Babatunde and Bello [14].

$$\text{RI} = 1.45765 + 0.0001164 \text{ IV} \quad (3)$$

2.4. Fatty Acids of Seed Oil and Ethyl Ester of Biodiesel Determinations

Fatty acids profile was determined through the fatty acid methyl esters (FAMES). Thus, FAMES were obtained by using Ackman method [15] as previously reported by Lepage and Roy [16] and Masood *et al.* [17].

FAMES of seed oil and ethyl esters of biodiesel were both analyzed by coupling gas chromatography (Thermo Fischer Scientific Ultra Brand) with mass spectrometry (GC / MS). Chromatographic analysis was performed on Trace GC Ultra equipped with an ASI 3000 auto sampler and with Polaris Q spectral mass detector, all from Thermo Fischer Scientific Ultra Brand. The coupling and the automatic control of the devices have been done by the software EXCALIBUR 2.0 Thermo Fisher. The Split/splitless injector was set at 250°C and the ion source temperature set at 250°C. Ultrapure Helium Alpha-gas 2 was the gas carrier set at 1ml/min constant flow with automatically adjusted pressure. Injections were on split mode. GC was fitted with a fused silica capillary column (DB-FFAP) 30m×0.25mm inner diameter (ID) x 0.25 μm film thickness (J & W Scientific, Agilent Technologies). Initial oven temperature was 130°C. The program temperature was as follows: equilibration time: 0.5 min; linear increase to 178°C at 4°C /min, followed by linear increase to 210°C at 1°C/min, followed by an increase to 245°C at 40°C/min and final 13min hold. The duration of the analysis was 60 min. The injected volume was 1 μL and the injected amount 10 $\mu\text{g/mL}$. Positive ionization of the FAMES was performed by electronic impact (EI), with 70 eV energy and full scan detection mod. Mass spectra range was 50-650 m/z; scan 0.58 sec.

Precise identification of the analytes was achieved by their relative retention times and mass spectra on the spectral mass database NIST libraries for fatty acid composition. External fatty acid standards were the 28 FAMES compounds NU-CHEK-PREP inc Elysian USA, (GLC reference standard 462) and the Supelco 37 component FAMES mix (CRM 47885).

2.5. Transesterification

Transesterification reaction was done by following the diagram of figure 1. Acidity greater than 1% has been evaluated in advance for crude vegetable oil. This required neutralization of the vegetable oil with an ethanolic solution of potassium hydroxide before preheating. Under stirring, an ethanolic solution of 1.1% potassium hydroxide in an ethanol/oil ratio of 6 was added. After 1 hour of agitation of the mixture at 45°C, a settling time of 16 hours or more was followed. A separation of the raw biodiesel from the alcohol-rich aqueous phase was performed. After washing with warm distilled water, the product is subjected to one hour of distillation at 100°C. Thin layer chromatography of the

product was then performed using the Ichihara and Fukubayashi [18] method to verify the purity of the biodiesel.

The process was stopped after a good yield of ethyl esters had been obtained and resumed if not.

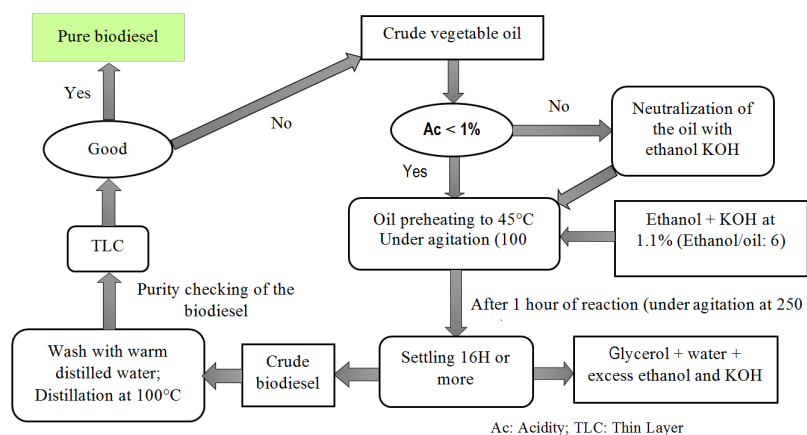


Figure 1. Transesterification process.

3. Results and Discussion

3.1. Physical and Chemical Characteristics of *Hura crepitans* Vegetable Oil

Physical and chemical characteristics of *Hura crepitans* (HC) are shown on Table 1. The lipid potential of HC seeds is 52.54%. The acidity of vegetable oil above 1% requires its neutralization before its use in diesel engines [19]. The high peroxide value (>10 meqO₂/kg) indicates that vegetable oil has a high concentration of peroxide and hydroperoxide which are primary products of autoxidation. A use in food is therefore ruled out at this stage. This could be related to its exposure to air, light, heat or its composition in unsaturated fatty acids. This index makes it

possible to predict future behaviour in terms of fat stability and to take conservation measures [20-22]. The high iodine index shows the unsaturated character. This parameter reflects the fluidity of vegetable oil [23]. This is confirmed by the fatty acid composition of vegetable oil which shows the predominance of unsaturated fatty acids (84.90%), particularly linoleic acid (54.13%) [23, 24]. These results are significantly different from those of Adewuyi et al. [13] and Owolabi et al. [25] who worked on the same species in Nigeria. This could depend on several parameters including the variety, climate, soil growing conditions and morphology of the matrices studied [26, 27]. Indeed, although the study conducted by Owolabi et al. [25] showed a much unsaturated oil, saturated fatty acids are also well represented (34.12%).

Table 1. Physico-chemical characteristics, oil yield and fatty acids of *Hura crepitans* kernel

	Yield (%)	HC	HC [28-30]
		52.54 ± 0.22	36.40 - 47.80
Quality indices	Free fatty acids (% Linoleic acid)	4.81 ± 0.14	1.64 - 4.10
	Peroxide value (meq O ₂ /kg)	28.02 ± 0.38	2.14 - 9.10
	Iodine value (g I ₂ /100g)	123.21 ± 0.41	122.08 - 149.10
	Saponification value (mg KOH/g)	210.29 ± 1.27	202.00 - 210.38

Table 1. Continued.

Acides gras		HC	HC [15]	HC [25]
		(%)	(%)	(%)
Saturated fattyacids	Tridecylic acid (C13:0)	0.13 ± 0.01	ND	ND
	Palmitic acid (C16:0)	9.14 ± 0.08c	12.20b	21.67a
	Margaric acid (C17:0)	0.02 ± 0.01b	ND	0.31a
	Stearic acid (C18:0)	5.63 ± 0.06b	5.10b	9.66a
	Arachidic acid (C20:0)	0.07 ± 0.00c	0.20b	2.48a
	Tricosylic acid (C23:0)	0.07 ± 0.00	ND	ND
Total of saturated fatty acids		15.06 ± 0.00c	17.50b	34.12a
Unsaturated fattyacids	Palmitoleic acid (C16:1, n-7)	0.09 ± 0.00b	0.10b	0.57a
	Oleic acid (C18:1, n-9)	27.34 ± 0.00a	27.20b	26.91c
	Linolelaïdic acid (C18:2)	0.92 ± 0.01	ND	ND
	Linoleic acid (C18:2, n-9, 12)	54.13 ± 0.04a	52.80b	36.61c
	α- linolenic acid (C18:3)	2.36 ± 0.03a	1.80b	0.75c
	Gondoic acid (C20:1)	0.06 ± 0.00	ND	ND
Total of unsaturated fatty acid		84.90 ± 0.00a	81.90b	64.84c

HC: *Hura crepitans*; ND: No Detected.

The data in a line followed by different letters are significantly different ($p < 0.05$). The values are averages of three repetitions \pm standard deviation.

3.2. Some Fuel Characteristics of *Hura Crepitans* Vegetable Oil

The comparative study of the fuel characteristics of HC vegetable oil recorded in Table 2 shows fuel parameters that generally comply with DIN 51605 for pure fuel rapeseed oil [7] except for the determined acidity, which remains higher.

A neutralization of the vegetable oil is therefore necessary before its use in diesel engines to avoid corrosion of metal parts. But this has consequences for the operation of engines that will use it directly in cold conditions [7, 31]. Compared to the biodiesel standard EN 14214 from the same sources, the acidity is even higher (4.81%) as well as the relative density (0.929) at 26°C. There is also a lower cetane number than that recommended by the biodiesel standard EN 14214. A conversion of vegetable oil into biodiesel is therefore necessary for diesel engines to improve their fuel parameters.

Table 2. Some fuel characteristics of *Hura crepitans* vegetable oil.

Characteristics	HC	Specification for vegetable rapeseed oil	Specification for biodiesel [31]
		DIN 51605 [7]	EN 14214
Moisture content (%)	0.06 \pm 0.00a	0.075a	Max: 0.05a
Ethyl linolenate content (%)	2.36 \pm 0.03b	-	Max: 12a
Polyunsaturated ester content (C=C \geq 4, %)	ND	-	Max: 1
Acidity (%)	4.81 \pm 0.14a	Max: 1.5b	Max: 0.5c
Density (à 26°C)	0.929 \pm 0.001a	0.90 – 0.93a	0.86 – 0.90b (*)
Lower calorific value (MJ. Kg ⁻¹)	39.07 \pm 0.05	-	Min: 35
Cetane number	44.53 \pm 0.06b	Min: 35c	Min: 51a
Refractive index	1.472 \pm 0.001	-	ND

HC: *Hura crepitans*; ND: No Detected; Max: maximum limit; Min: minimum limit; (*): density at 20°C.

The data in a line followed by different letters are significantly different ($p < 0.05$). The values are averages of three repetitions \pm standard deviation.

3.3. Transesterification Reaction

3.3.1. Effect of the Molar Ratio Ethanol/Oil

Figure 2 shows the influence of the ethanol/oil molar ratio on the conversion rate of HC vegetable oil. The reaction time

has a positive impact on the reaction performance. Within the first 20 min for the 4:1 molar ratio and 50 min for two other ratios (6:1; 8:1), the oil conversion rate increases rapidly before beginning its progressive evolution. The molar ratio of 8:1 slightly increases yield within the first 10 min. This molar ratio has no major effect on the conversion rate of vegetable oils into biodiesel in general. Thus the molar ratio ethanol/oil of 6 is retained for the rest of the operations.

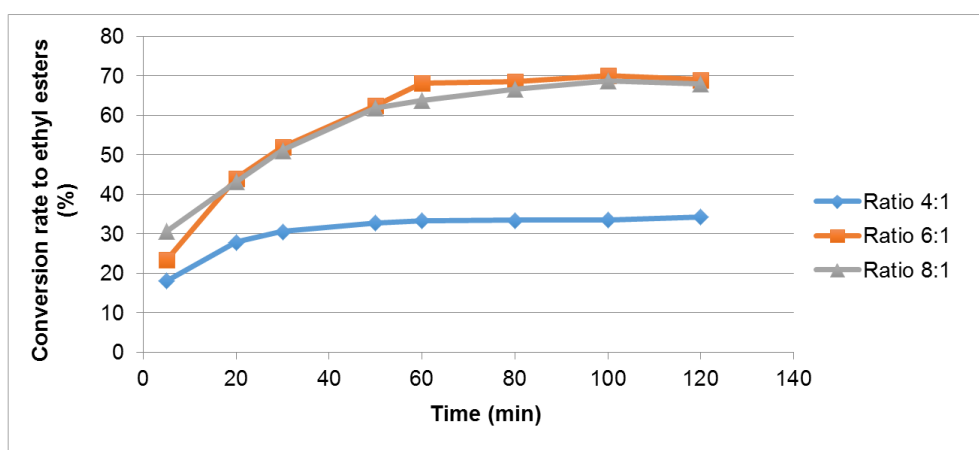


Figure 2. Evolution of the molar ratio on the conversion of vegetable oil from *Hura crepitans* to biodiesel (35°C; anhydrous ethanol; 1% KOH; 250 rpm).

3.3.2. Effect of Temperature and Reaction Time on Transesterification

Figure 3 shows the influence of temperature and reaction time on the conversion rate of HC vegetable oil. Within the first 5 min, the conversion rate from vegetable oil to biodiesel is proportional to the temperature of the reaction. Beyond this time, a temperature above 45°C is not cost-

effective. After 60 min of reaction, the temperature increase is without any major positive impact or on the contrary reduces the conversion efficiency. 45°C is therefore the most suitable temperature for converting HC vegetable oil into biodiesel under predefined reaction conditions. The high content of unsaturated fatty acids may also explain the conversion rate of vegetable oils at this temperature [32].

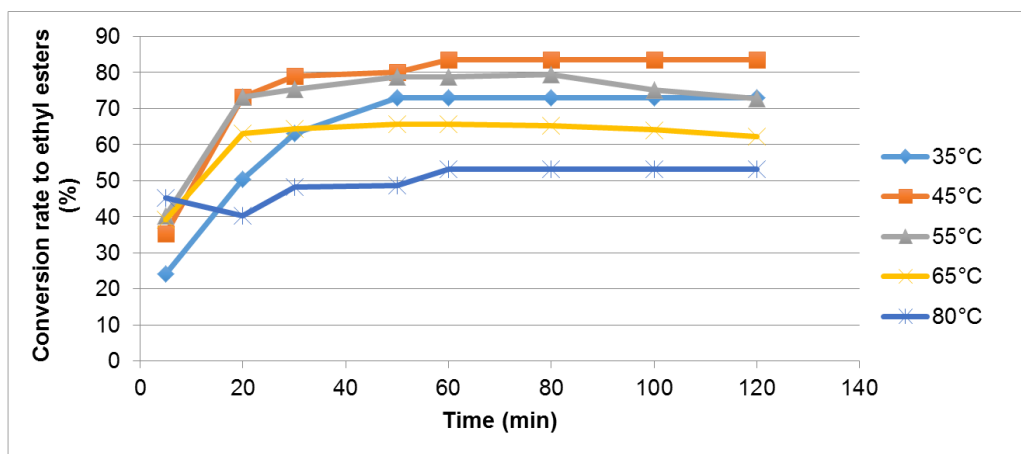


Figure 3. Influence of temperature and reaction time on the conversion rate of *Hura crepitans* vegetable oil to biodiesel (anhydrous ethanol; ethanol molar ratio: oil of 6; 250 rpm).

3.3.3. Effect of Catalyst Concentration and Reaction Time on the Conversion Rate of Vegetable Oils to Biodiesel

In order to select the amount of catalyst required for the transesterification reaction, a study was conducted and results are presented in Figure 4. From its analysis, it appears that the reaction efficiency is proportional to the catalyst concentration. This is more pronounced at the beginning of the reaction until about 50 min. From 80 min onwards, the additional addition of potassium hydroxide (KOH) from 1%

to 1.1% and 1.2% (w/w) seems unsuccessful on the yield with vegetable oil of *Hura crepitans*. It is also noted that 0.9% KOH (w/w) does not offer a good yield. Similarly, 1.2% KOH is disadvantageous for the transesterification of vegetable oil from *Hura crepitans* after 30 min of reaction. This could be due to a possible competition between the saponification reaction and transesterification [33]. This oil has a good yield after 60min reaction with 1.1% catalyst. In view of these results, the latter grade was therefore used.

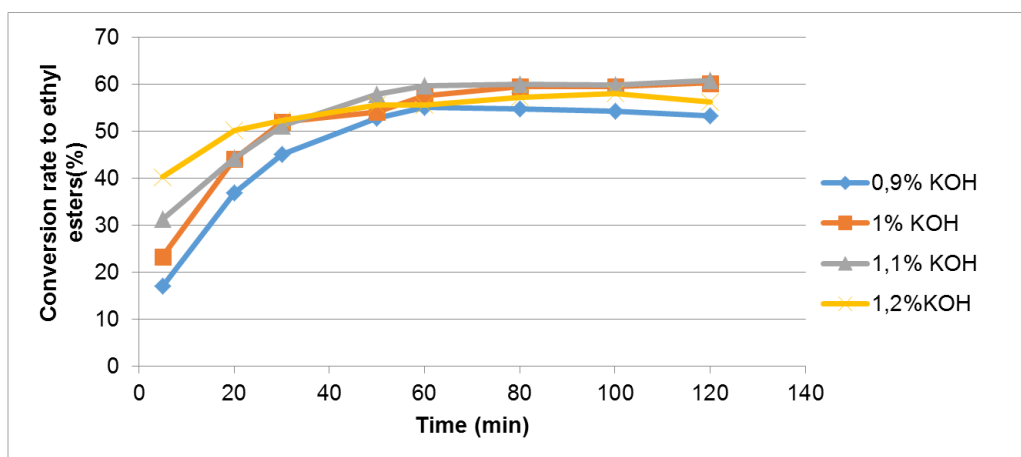
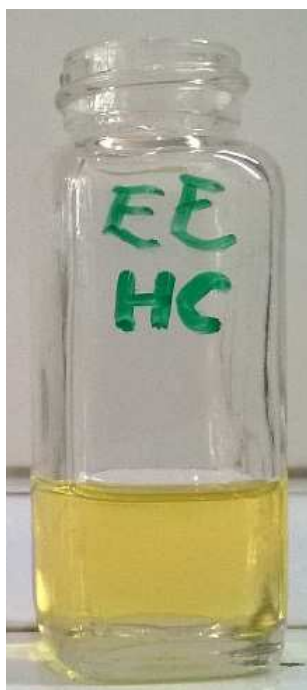


Figure 4. Conversion rate of *Hura crepitans* vegetable oil for four (04) different concentrations of KOH (anhydrous ethanol; ethanol molar ratio: oil of 6; 250 rpm; 45°C).

3.3.4. Ethanolysis of *Hura Crepitans* Oil Under Optimal Conditions

The conversion of HC vegetable oil gave a yield of 81.47% after one hour of reaction with 1.1% catalyst and a reaction temperature of 45°C. It should be noted that settling began 10 to 20 min after the final product was introduced into the separating funnel. The conversion rate of non-conventional HC vegetable oil is greater than 80% obtained by Nitiëma-Yefanova et al [34] with *Balanites aegytiaca* vegetable oil. It should be noticed that Nitiëma-Yefanova worked under the following conditions: 35°C; 1.1% KOH; an

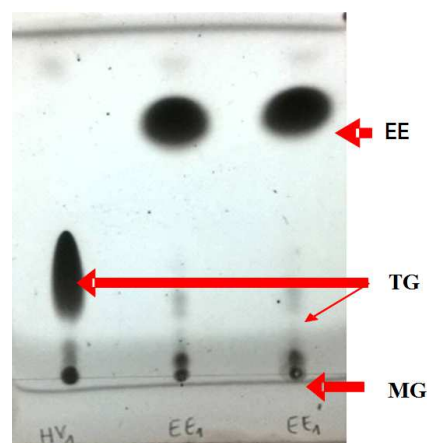
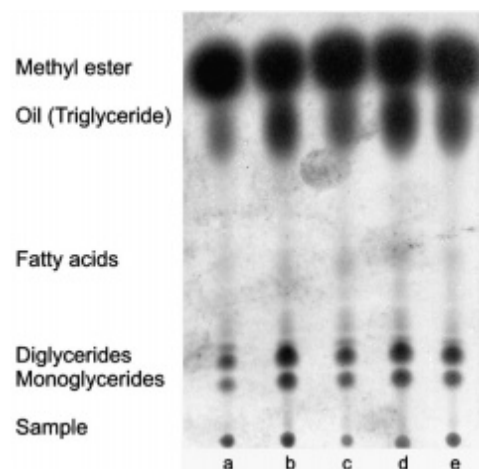
ethanol/oil molar ratio of 6 and a reaction time of 120 min. The same work by this author showed that this yield increased from 80 to 93% after the addition of glycerol. The latter made it possible to facilitate the separation of the biodiesel-rich organic phase from the aqueous phase; thus an additional production cost. This shows that some of *Hura crepitans*' Biodiesel would end up in the wash water. The efficiency of the transformation could be improved if we use glycerol for separation and less water for washing with the risk that biodiesel is richer in phospholipids, glycerol, free fatty acids, residual KOH, etc. [35].

a. Settling of *Hura crepitans*' biodiesel after washingb. *Hura crepitans*' biodiesel**Figure 5.** Separation of *Hura crepitans*' biodiesel.

3.4. Some Fuel Characteristics of Biodiesel from *Hura Crepitans* Vegetable Oil

The chromatogram in Figure 6a. shows the migration of

the stains of the different compounds resulting from the thin layer chromatography of the biodiesel obtained in order to verify its purity. In comparison with Figure 6b, there is a preponderance of ethyl esters [18, 36] as confirmed by the result in Table 3, with an ester yield of 98.48%. The analysis of this table shows a reduction in acidity (0.41%), density (0.887) and an increase in the cetane number to 54.44. These values are similar to those of Adewuyi *et al.* [13] who worked on HC from Nigeria. All these data, like most of the others in Table 3, comply with biodiesel standards.

a. Chromatogram of *Hura crepitans*' biodiesel

b. Chromatogram of soya biodiesel [36]

Figure 6. Thin layer chromatography of biodiesels.**Table 3.** Some characteristics of *Hura crepitans*' biodiesel and standards.

Characteristics	HCB	Biodiesel specification [31]
		EN 14214
Ethyl ester content (EE, %)	98.48 ± 1.27a	Min: 96.50a
Triglycerides (TG, %)	0.32 ± 0.02a	Max: 0.20a
Diglycerides (DG, %)	ND	Max: 0.20
Monoglycerides (MG, %)	0.40 ± 0.02b	Max: 0.80a
α+γ –ethyl linolenate (C18:3, %)	0.30 ± 0.01b	Max: 12a
Ethyl timnodonate (C20:5, %)	0.02 ± 0.00b	Max: 1a
Moisture content (%)	0.03 ± 0.00b	Max: 0.05a
Acidity (%)	0.41 ± 0.06a	Max: 0.50a
Density (at 26°C)	0.887 ± 0.01a	0.86-0.90a (*)
Iodine value (g I ₂ /100g)	118.03 ± 1.48a	Max: 120a

Characteristics	HCB	Biodiesel specification [31]
		EN 14214
Lower calorific value (MJ/kg)	41.12 ± 0.07a	Min: 35b
Cetane number	54.44 ± 2.00a	Min: 51a
Refractive index	1.471 ± 0.000	ND

HCB: Hura crepitans' biodiesel; ND: Not Detected; (*): density at 20°C.

The data in a line followed by different letters are significantly different ($p < 0.05$). The values are averages of three repetitions ± standard deviation.

4. Conclusion

The kernels of *Hura crepitans* seeds have a high lipid potential of nearly 52.54%. Vegetable oil is highly unsaturated and deserves protection from air, light and heat. This oil has good physicochemical and energy characteristics, but its direct use in diesel engines is not recommended due to its high acidity and density and lower cetane number as compared to the data provided by the European standard EN 14214. A yield of 81.47% biodiesel, containing 98.48% ethyl esters, is obtained in a single step by transesterification of vegetable oil in order to reduce production costs. Among all the treatment variants adopted, it was found that the maximum yield of ethyl esters was obtained using an ethanol/oil molar ratio of 6 and 1,1% (m/m) KOH at a reaction temperature of 45°C. The minimum reaction time required to obtain a maximum ester yield was 60 min. The fuel characteristics of biodiesel are also improved with a reduction in acidity, density and an increase in cetane number. Biodiesel, in view of the results obtained, could be proposed to power Diesel engines without a preheating system.

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