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Contribution to the extraction and oil of the physico-chemical characterization of seeds of *Ricinus communis L.* wild flora of Congo-Brazzaville for the production of the biodiesel

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ABSTRACT: The fall in the use of fossil fuels and the increasing need to safeguard the environment lead to the valorization of alternative sources of energy.Biodiesel is an interesting alternative to the traditional diesel bus; at the same time, it makes it possible to reduce CO2 emissions by 80% and minimize its impact on greenhouse gases and climate change. This work aims to determine the physico-chemical characteristics of the oil of castor seeds (Ricinus communis L.) wild flora of Congo-Brazzaville for the production of the biodiesel. The extraction of oil from castor seeds was made thanks to the method known as Soxhlet, starting with a non-polar organic solvent (hexane) after several cycles of siphonage. After extraction, extracted oil was characterized on parameters such as color, density, viscosity, acid value, acidity, the index of saponification, and pH.

The objective of this work is to contribute to the extraction and the physico-chemical characterization of seeds of Ricinus communis L. (Ricin). wild flora of Congo-Brazzaville for the production of the biodiesel. The extraction of oil starting from castor seeds was made thanks to the method known as Soxhlet, starting from a non-polar organic solvent (hexane) after several cycles of siphonage. After extraction, extracted oil was characterized on the parameters such as the color, the density, viscosity, the acid value, acidity, the index of saponification, and the pH. The output of the extraction of oil starting from seeds of Ricinus communis L. of the wild flora of Congo is 33.43%. This extracted oil has a pale yellow color, a density of 0.95 at 25°C, an acidity of 1.69%, and a low kinematic viscosity of 6.16 mm²/s at 28°C. It has a high acid value of 3.28 mg KOH/g when compared with an oil intended for the production of the biodiesel (2 mg KOH/g). Oil obtained is acid with pH = 5, lower than the oil pH intended for the production of the biodiesel to which the value must be close to 7 and rich in fatty acids for long-chain carbonaceous with an index of saponification of 152.40 mg KOH/g. Thus, the utilization of this oil for the production of biodiesel requires the application of a process of cross-esterification upstream.

Keywords: *Extraction, oil of Ricinus communis L, soxhlet, characterization, biodiesel*

INTRODUCTION

The *Ricinus communis* (Ricinus) is a plant that belongs to the Euphorbiaceae family. It comes from tropical Africa, but you can also find it under subtropical. The only one that represents the Ricinus species is a tree with large web beds. This plant is largely cultivated in most areas of tropical and subtropical drought, as in many moderate areas equipped with a hot summer. The Ricinus is present along the African continent of the Red Sea Atlantic coast and on the islands of the Indian Ocean (Maroy, 2011). The seeds of *Ricinus communis (Ricinus)* are significant oleaginous seeds that contain 35 to 50 % oil and 18 % of proteins (Okechukwu et al., 2015, Rousnet, 2008) The principal fatty acid is 2-hydroxy-CIS - Øctadecenoic, known as a ricinolic acid (Akpan et al., 2006). According to the United Nations, the world population exceeded 8 billion people in November 2022, one billion more than the year 2010. So far, most of this global energy request has always been fulfilled by fossil sources of energy such as oil, coal, and natural gas, which constitute 80% of the consumption of the entire energy (Planet-Energies, 2019; Khiari, 2016) The use of these energy sources for emission and accumulation of Gases for the greenhouse, according to the example of CO2, SO2 and NOx as well as the climatic changes and causes serious damage in the environment (Khiari, 2016; Brunschwig et al, 2011).

These problems that are involved in the climatic changes reduce the use of the sources of fossil energy and increase environmental supply. It is of the utmost importance to valorize alternative energy sources (Brunschwig et al, 2011). The biocarburants resulting from biomass seem to be one of the most interesting solutions that can replace conventional fuel, reduce CO2 emissions by 80% and minimize the effects on the effect of greenhouse and the changes, but also relatively relatively relative to become energy independent (Brunschwig et al., 2011). The biodiesel is a mixture of ethyl or methyl plant oil esters (EEHV or EMHV) or animal fat (Stickerer et al, 2008). It does not become more permissible from a variety of raw materials, the energy of the edible (soybean oil, colza and a sunflower) (the oil of the seeds of Jatropha Curcas and Ricinus) (Okechukwu et al., 2015). The Ricin forms one of the sources of non-approved vegetable oil (Guergour, 2011). Ricin's oil can be processed to produce biodiesel, and the oil cake, the remaining al., 2015; the agro-alimentary (Kadi, 2020). The Republic of Congo, in tropical Africa, can be a great producer of Biodiesel thanks to the use of Ricin. In this instruction, the government of the Republic of the Congo and the Italian group Eni-Congo signed an agreement on the next production of organic carburants based on castor oil in 2021. Empty creates 90,000 direct jobs and will also stimulate the rural economy (French News, 2023; MCI, 2023; Energies Media, 2023). The production of biodiésel is therefore also a social part and a share in the development of the Congo. This work is to extract and characterize the oil of the Congo's wild flora for the production of biodiésel.

MATERIEL AND METHODS

Biological material

The biological material used was seeds of *Ricinus communis L*, which were collected on wild seedlings at the edge of the Congo River in Brazzaville, the capital of the Republic of Congo (Fig.1). The fruits of dry ricinus were collected the first two weeks of September 2023.

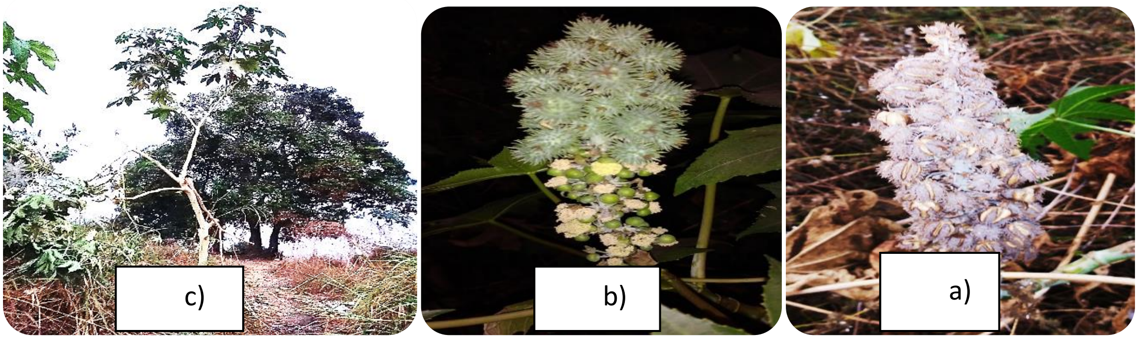

Fig. 1. a) Plant of Ricinus Communis L (Cornice, edge of the Congo river); b) fruits of wet ricinus, and c) fruits of dry Ricinus

Dehusking, drying, and crushing of seeds of *Ricinus communis L*

After the collection of the fruit of ricin, the seeds were extracted by dehusking (Fig. 1a) and then dried with the sun (Fig. 2b) and then in a drying oven at 60°C for 6 hours (Fig. 2c) in order to reduce to the maximum their moisture. Boudjemaa and Abdelkrim, 2015. In order to facilitate the broadcasting capacity of the solvent of extraction, the dried castor seeds underwent an operation of crushing. This operation was carried out using a mortar (Fig. 2d).

Fig.2. a) Dehusking;b) Drying with the sun;c) drying with the drying oven ;d) crushed Castor seeds

Extraction of the castor oil to the soxhlet

The extraction of the oil of castor seeds was made in the following way:

A mass of 30 g of crushed castor seeds (mass mg) was weighed using a balance with precision, then introduced into the porous cartridge, then, the whole was put in the balloon with 250 ml of hexane (solvent) Fig.3a. The balloon was then heated (beforehand weighed with vacuum mo) with 69 ± 1 °C to begin the cycle from extraction. After 6 hours and several siphonages, one noticed that the solvent became limpid and one stopped operation (Sidohounde et al., 2019). The cartouche of the soxhlet was withdrawn, then, the balloon was again heated to recover solvent. Recovery is just made in extreme cases siphonage. After the operation of extraction, the hexane mixture + extracted oil was obtained in the balloon (Fig.3b) and the seed oil cakes in the cartridge into porous (Fig.3c). Lastly, after recovery of solvent, the hexane traces in the mixture with oil were evaporated in the drying oven during 15 min to obtain pure extracted oil, then the balloon was weighed (mass m1) obtained was stored in the bottles (Fig.3d).

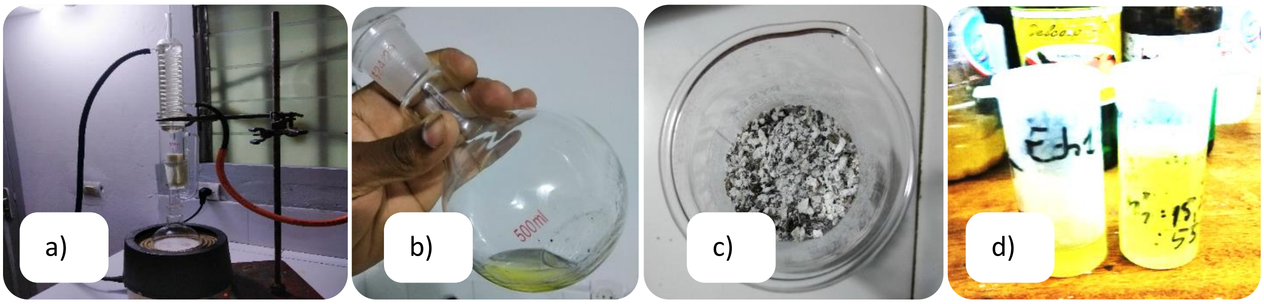

Fig.3 a) Assembly of the extractor soxhlet ; b) Mix extracted oil + hexane; c) Oil cakes; d) Castor oil extracted

Castor oil output extracted

Castor oil output extracted η_h was calculated using equation 1:

$$\eta_h(\%) = \frac{m_h}{m_g} \times 100 \qquad (Eq.\ 1)$$

with m_0 : mass empty test-tube (g) ; m_1 : mass test-tube containing oil (g)

$m_h = m_1 - m_0$: mass of extracted oil (g) ; m_g : initial mass of castor seeds (g).

Characterisation of the castor oil extracted

Density et voluminal mass

The measurement of the density of the castor oil was carried out by measurement of the volume and the mass of a certain quantity of oil. It is calculated by taking into account a water-like liquid of reference to the same temperature (Boudjemaa and Abdelkrim, 2015).

$$\rho_h = \frac{m_h}{V} = \frac{m_1 - m_0}{V} \qquad (Eq.\ 2)$$

with : ρ_h : voluminal mass of oil (g/cm^3); V: volume of oil (cm^3).

$$d = \frac{\rho_h}{\rho_w} \qquad (Eq.\ 3)$$

with : d : density of oil ; ρ_h : voluminal mass of oil (g/cm^3) ; ρ_w: voluminal mass of water is equal to 1 g/cm^3.

pH

The pH was measured using paper pH. A small volume of oil was taken and put in contact with paper pH in a beaker. Thereafter, the paper was colored. By comparing the color of the paper pH colored by the sample to the various colors on the limp of paper pH, the pH of the sample could be determined..


Fig. 4 : Mesure du pH avec du papier pH

Viscosity (μ_h)

The measurement of viscosity was made the method with fall of ball based on the limiting velocity measurement of fall v of a ball of ray r and density (ρ_b) in a known liquid of density (law of Stoke) by equations 4 and 5 (Wikihow, 2024).

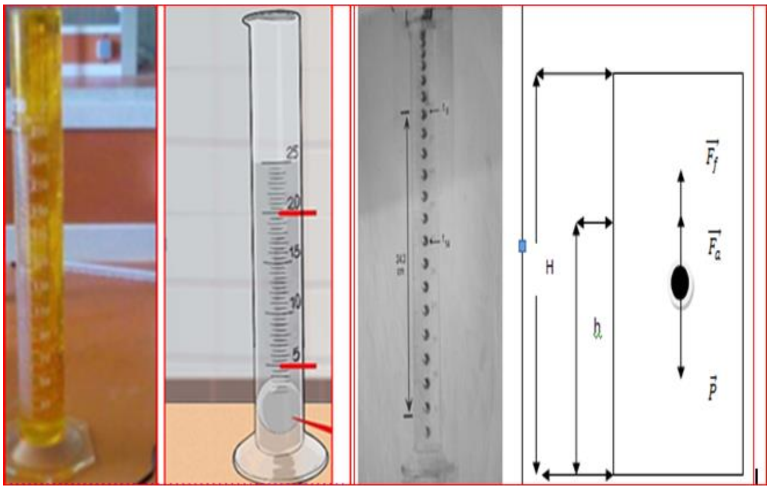

Fig. 5 : Procedure and device of measurement of viscosity (Wikihow, 2024)

Dynamic viscosity is calculated with the following relation:

$$\mu = \frac{2(\rho_b - \rho_h) \times g \times a^2}{9 \times v} \qquad (Eq.4)$$

witth : ρ_h: voluminal mass of oil (g/cm^3). ; ρ_b:voluminal mass of ball (g/cm^3) ; g: constant of equal gravity 9,8 m/s² ; a: ray of the sphere (m) ; v: speed (m/s).

The dynamic unit of viscosity is Pascale second (Pa.s).
One calculates kinematic viscosity (μ_h en m^2/s) by the following relation:
One calculates kinematic viscosity (by the following relation :

$$\mu_h = \frac{\mu}{\rho_h} \qquad \textbf{(Eq. 5)}$$

Acid index (IA)
In an Erlenmeyer, a mass of 1g of oil was weighed then 25 ml of alcohol solution (Ethanol 95°) and some phenolphthalein drops (an indicator) were added. The mixture obtained (solution) was titrated with the KOH with 0.1N (Solution prepared 5 days in advance) until the appearance of a persistent pink coloring (10 seconds) and the volume of KOH for neutralization was noted. The acid value was calculated starting from equation 6 (Lembrouk and Aichouba, 2012).

$$IA = \frac{(56{,}1 \times N_{KOH} \times V_{KOH})}{m_{ech}} \qquad (Eq. 6)$$

$$Acidity\ (\%) = \frac{IA}{2} \qquad (Eq.7)$$

With N_{KOH} : normality of KOH with *0.1N* ; V_{KOH} :volume of KOH necessary to titration (*ml)*.
m_{ech} : mass sample *(g)* ; *IA* : Acid index of *mg KOH/g*.

Index of saponification (IS)
A mass of 1 G of castor oil was weighed and introduced into a round balloon à.fond then 25 ml of solution KOH in ethanol with 0.5 N y have were added. The mixture was carried to boiling in a heating balloon surmounted by a cooling agent with backward flow for 30 min. After cooling, the mixture was recovered in a bécher, while adding some goutes indicator (phenolphthalein).The solution was titrated with the hydrochloric acid HCl with 0.5 N until the disappearance of the pink color and reappearance of the initial color of the mixture (transparent) while noting the volume HCl used (Lembrouk and Aichouba, 2012).

The index of saponification is given by the formula below :

$$\mathrm{IS} = \frac{56{,}1 \times (V_0 - V) \times N_{HCl}}{m_{éch}} \qquad (Eq.8)$$

With : V_0 : volume of *HCl* used for the dummy trial (ml) ; *V* : volume of *HCl* used for the sample to analyze (ml) ; m_{ech} : masse de l’échantillon (g) ; N_{HCl} : normality of hydrochloric acid HCl (N) ; *IS* : Index of saponification (mg KOH/g)

Statistical analysis
Values represented are the means and standard deviations for three replicates. Statistical analysis was carried out by Excel Version 8.0 software. Significance was defined at p<0.05.

RESULTS AND DISCUSSION

Output of castor oil extraction η_h

The oil output extracted seeds of Ricinus communis L. wild flora of Congo east of 33.43%. This castor oil output obtained after extraction is included/understood in the interval of the average output of crude oil, which is located around 28 to 47%, quoted in the literature. Indeed, our output was lower by 47.7% is obtained by Bouhafs and Hadjaidji (2017), but higher than the output obtained by Anuforo et al. in 2015, which amounted only to 28.48%. This difference compared to the literature could be explained by the fact that the seeds were crushed in a coarse way, which had as a consequence bad maximum diffusion of solvent in seeds crushed to facilitate extraction to the maximum of this oil. With that can be added the fact that the castor oil is made up of non-polar and polar fatty acids, these last not being able to be extracted completely with the hexane (a non-polar solvent). A use in proportion of 50:50 of hexane with ethyl acetate (polar solvent) for the extraction by maceration of the oil remainder in the residues of seeds crushed after extraction by soxhlet would certainly increase this output (Hundie and Akuma, 2022).

Physico-chemical characteristics of the castor oil

The physicochemical characteristics of the castor oil of the wild flora congolaise for the production of biodiesel are presented in Table 1. It acts in particular: the color, density at 25°C, the index of saponification, viscosity at 28°C, the acid value, the acidity and pH of the castor oil extracted, compared with those of the crude oil used for the synthesis of the biodiésel obtained in the literature.

Table 1: Physico-chemical characteristics of the castor oil extracted compared with those found in the literature (Al-Akraa and Islam, 2022; Brahimi, 2017; Hadjaidji, 2017; Okechukwu and al., 2015 ; Ramanamihaja, 2006 ; Bouhafs and; Boudjemaa and Abdelkrim, 2015).

Physico-chemical characteristics	Unit	Castor oil (present work)	Crude oil (Literature)
Output	%	33.43	28- 47.70
Color		Pale yellow	
Density with 25°C		0.95	0.86-0.96
pH		5	5.5-6.11
Viscosity kinematics with 28°C	mm^2/s	6.16	9.43
Index acid	mg KOH/g	3.28	0.89-3.70
Acidity	%	1.69	0.45-1.85
Index of saponification	mg KOH/g	152.40	168.50-185.83

The extracted oil has a pale yellow color with a density of 25°C of 0.95. The value of the density is high for an oil intended for use in a diesel engine. For the production of biodiesel, it would be desirable to reduce the value of the density. Nevertheless, this value of the density of castor oil obtained starting from the wild flora of Congo for the production of biodiesel is in the range of the actual values by Okechukwu et al. (2015) and Ramanamihaja (2006). Indeed, according to Al-Akraa and Islam (2022), the density of castor oil is approximately 0.96 to 15.56°C. This value is attributed to the presence of hydroxyl groups in the methyl ester of oleic acid in Ricinius, which gives him this dense character.

The castor oil extracted has an acid pH (pH = 5). The production of the biodiesel requires a minimum pH of 5,50. However, this value of pH can be increased by the reaction of cross-esterification to approach neutrality (pH=7) *according to* Bouhafs and Hadjaidji, 2017, by standard ASTM D6751,2024. The kinematic viscosity of extracted oil is 6.16 mm^2/s**.** The viscosity of this oil has a value close to that of the gas oil (Petro-diesel) which is about 6 mm^2/s at 40°C, but it remains lower than the average of the values of the raw castor oil obtained in the literature 9.43 mm^2/s. According to standard ASTM D6751, 2024, the oil intended for the production of the biodiésel requires a low viscosity. By applying the reaction of cross esterification, will make it possible to reduce the value of the oil viscosity to obtain a biodiésel quality with a viscosity included/understood ente 1.90 and 6.00 mm^2/s in conformity with standards ASTM D6751 and IN 14214 (Irish Standard, 2014).

The castor oil presents an acid value of 3.28 Mg KOH/g (high) when compared with an oil intended for the production of biodiesel whose value must be 2 mg KOH/g). This value of acid value remains lower than that found by *Hundie and Akuma, 2022* (6.4 mg KOH/g). The value of acid value indicates that the castor oil extracted presents a high quantity of Fatty Acids Free (CASE) and a hypothetical deterioration or oxidation of the oil. This high value of acid value could have an impact on the output of the biodiésel. Indeed, for certain authors, plus the value of the acid value of oil is raised, less is the output in biodiesel (Abdelouahed and Boulghiti, 2015. Moreover, according to Hundie and Akuma, (2022) affirmed in their work that to produce biodiesel, an acid value of oil must be lower than 2.5 mg KOH/g. It is to be noted that extracted oil is adapted for the production of biodiésel, indeed, the value of acid value remains included/understood in the beach of the values brought back in the literature.

The value of the acidity obtained from this castor oil is 1.69%, which is a good indication, because, it is lower than the minimal value which rises to 3%. Indeed, the quality of oil conditions the process of production of Esters of fatty acids (EAG) in order to respect the standards such as ASTM D 6751 or INTO 14214. An alkaline cross-esterification cannot succeed or will be inhibited if the content of CASE exceeds 3% of the weight (Lembrouk and Aichouba, 2012).

The index of saponification is a size with the length of the carbonaceous chains of the constituent fatty acids of oil; this value is large, plus the fatty acids are low-weight molecular, therefore, of short-chain carbonaceous (Djerifili, Gsassi, and Bakache, 2020). The value of the latter for this castor oil is about 152.40 mg KOH/g of oil. This value indicates that the fatty acids contained in this extracted oil present long carbonaceous chains. The latter is lower than the average of the values of the oils used for the production of biodiesel observed in the literature. The results obtained are in agreement with the work of Raharijaona (2015). For this last part, the castor oil extracted with hexane is composed of a linoleic majority of acid (70%) and palmitic acid (13%), which are acids with long chains. Physico-chemical castor oil characteristics extracted starting from seeds from the ricin wild flora of Congo for the production of biodiesel are close to those obtained by Al-Akraa and Islam, 2022 (Density = 0.96 with 15.56°C); Bouhafs and Hadjaidji in 2017 (pH = 5.5), Abdelouahed and Boulghiti in 2016 (IA = 2.81 mg KOH/g; IS = 117.11 mg KOH/g; Density = 0.91), Okechukwu and al. in 2015 (Density = 0.96; Kinematic viscosity = 6.48 mm²/g), and Ramanamihaja in 2006 (IA = 3.70 mg KOH/g; Density = 0.96).

CONCLUSION

The world demographic growth and the need to preserve planet generate the urgency to find new sources of energies clean in order to minimize the pollution of the environment and the wasting of the raw materials food. The goal this work was to contribute to the extraction and oil the physico-chemical characterization of seeds of *Ricinus communis L.* wild flora of Congo-Brazzaville for the production of the biodiesel. The results show that the output of oil extraction of *Ricinus communis* L of the wild flora of Congo east of 33.43%. This extracted oil is rich in fatty acids with long chains carbonaceous with an index of saponification of 152.40 mg KOH/g, has a pale yellow color, a density of 0.95 with 25°C and a low viscosity with 28°C, that is to say, 6.16 mm^2/s.It has a high index acid of 3.28 mg KOH/g when compared with an oil intended for the production of biodiesel (2 mg KOH/g). Oil is acid with pH= 5, lower than the oil pH intended for the production of the biodiésel to which the value must be close to 7. Thus, the ulitisation of this oil for the production of biodiesel requires the application of a process of cross-esterification upstream.

Author Contributions

All the authors contributed to the design and with the realization of this étude.La material preparation, the experimentation, the data acquisition were carried out by Elongo Bertrand Guelor, the outline of Nkounkou Tomodiatounga Domenica and Tatsy Nsimba. The first manuscript was written by Elongo Bertrand Guelor and all the authors presented their observations on preceding versions of the manuscript. All the authors read the final manuscript**.**

Ethical approval

Not applicable. No tests, measurements or experiments were performed on humans as part of this work.

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Conflicts of interest

The authors have no relevant financial or non-financial interests to disclose.

Consent for publication

Authors have agreed to submit it in its current form for consideration for publication in the journal

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