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OXIDATIVE PROPERTIES OF OILS EXTRACTED FROM FOUR OILSEEDS CONSUMED IN CÔTE D'IVOIRE: RICINODENDRON HEUDELOTII, CYPERUS ESCULENTUS, CITRULLUS COLOCYNTHIS AND IRVINGIA GABONENSIS

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ABSTRACT: *The many nutritional, pharmaceutical and industrial applications of oils extracted from plant seeds have in recent years generated growing scientific interest in the search for new unconventional oilseed sources. The needs of the oilseed market and the detection of new functionalities have aroused, at national level, an interest in scientific and technical research towards local plant oilseed sources, which are still under-exploited. The objective of this study was to extract the oils of Ricinodendron heudelotii, Cyperus esculentus, Citrullus colocynthis and Irvingia gabonensis and then to characterize them from a physico-chemical and nutritional point of view in order to evaluate their potential for use in the food, pharmaceutical and cosmetic industries. To do so, we evaluated their ability to resist oxidation through the measurement of oxidation indices. The results of this work showed a progressive increase in acidity and p-anisidine index, and a progressive decrease in peroxide value during storage at room temperature for 180 days. It appears that Ricinodendron heudelotii, Citrullus colocynthis, Cyperus esculentus and Irvingia gabonensis oils have chemical and biochemical potentials that could be exploited in the food, cosmetic and pharmaceutical fields.*

KEYWORDS: Vegetable oils, Oxidative properties, *Ricinodendron heudelotii, Citrullus colocynthis, Irvingia gabonensis, Cyperus esculentus*.

INTRODUCTION

According to the USDA [**1**], world vegetable oil production will reach 204 million tonnes, a 3% increase over 2017-2018. This increase in production reflects the interest and importance of vegetable oils, which are the main source of fats. Indeed, vegetable oils account for about 85% of the fats produced in the world [**2**]. These oils are generally extracted from fruits and seeds by two main extraction methods, namely the pressure extraction method and the organic solvent extraction method [**3**]. Biochemically, vegetable oils are mainly composed of triglycerides (95-98%) and minor substances including fat-soluble vitamins, pigments, phospholipids, phenolic compounds and free fatty acids [**4**].The qualitative and quantitative composition of these minor substances depends on the plant species, climatic conditions and extraction techniques [**5**].

The physical and chemical properties of vegetable oils determine their use in various fields such as the food, cosmetic, pharmaceutical and chemical industries. The food use of vegetable oils depends largely on the qualitative and quantitative composition of the constituent fatty acids. Fatty acids such as palmitic, stearic, oleic, linoleic, linolenic, linolenic, arachidonic, eicosapentaenoic and docosahexaenoic acid play a very important role in nutrition [**6**]. Oils rich in saturated fatty acids (SFAs) such as palm oil are used for frying, while those rich in polyunsaturated fatty acids (PUFAs) such as olive, soybean, sunflower oils are more suitable for seasoning in human food [**7, 8**].

In view of this interest in vegetable oils, the search for new multi-potential vegetable oils is nowadays of major scientific interest. With this in mind, we have carried out physico-chemical characterization studies on the oils extracted from the seeds of four oilseeds: *Ricinodendron heudelotii, Cyperus esculentus, Citrullus colocynthis* and *Irvingia gabonensis*, with a view to improving their technical value and their use in human food.

METHODOLOGY

Oil extraction

The oils subjected to the oxidation resistance test in this study were extracted in the laboratory by the Soxhlet method (AFNOR ISO 734-1, 2006) [**9**]. The oils were extracted from seeds of *Ricinodendron heudelotii*, seeds of *Citrullus colocynthis*, tubers of *Cyperus esculentus* and fines of *Irvingia gabonensis*.

Determining the tampering parameters

The UV-visible spectrum of the studied oils was carried out according to the Besbes method [**10**]. A mass of 0.1 g of oil was dissolved in 10 mL of n-hexane and the absorbance of the prepared solution was read at 200-600 nm against hexane using a PG Instruments T80+ spectrophotometer.

The oxidative properties of the oils were determined by monitoring the weathering parameters (acid value, peroxide value and anisidine value) during storage at a temperature of 25°C as described in the literature [**11, 12**].

Each vegetable oil extract was divided into 20 translucent glass tubes of 10 mL each. The tubes were then carefully sealed and stored at 25°C in the dark for six months. Weathering parameters such as acidity, peroxide value and anisidine value were determined every two weeks during storage.

Acid index

The acid index was determined according to the method described by AFNAR (NF ISO 1242: 1999 (T 75-103)) [**13**]. The acid index of a fat is the number of mg of potassium hydroxide (KOH) needed to neutralize the free fatty acids (FFA) contained in 1 g of fat. It measures the amount of FFA present in a fat. The principle consists of dissolving the fat in neutralized hot ethanol and then titrating the GLA present by means of a titrated solution of KOH in the presence of phenolphthalein as an indicator.

A quantity of 7 g of fat was weighed and placed in a balloon or conical flask. A volume of 50 to 70 ml of previously neutralized hot alcohol and 2 drops of colored indicator were added. This was titrated with potassium hydroxide (1N), stirring vigorously until a pink color persisted for 30 seconds. The result is expressed as follows:

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$$Ia = (V * 56{,}1 * N)/m$$

With:
V: volume of potash in ml; *N*: normality of potash solution; *m*: mass of test sample in g; *56, l g*: relative molecular mass of KOH; *Ia:* acid index. The acid index is expressed in mg KOH/g oil.
Peroxide index
The peroxide index was determined according to the method described by AFNOR (NF T 60-220) [**13**]. It is the quantity of peroxide present in the sample, expressed in milliequivalent of active oxygen contained in one kilogram of product, oxidizing potassium iodide with release of iodine. The principle is based on the titration of the iodine released by a solution of sodium thiosulphate Na_2S_2O.Approximately 1 g of oil was weighed into a vial to the nearest 0.001 g and mixed with 10 mL chloroform and stirred. 15 mL glacial acetic acid and 1 mL 10% potassium iodide (KI) were added. The mixture was stirred for 1 minute and allowed to stand for 5 minutes at 15 to 25°C in the dark. 75 mL dist. water was added followed by titration of the liberated iodine with 0.01 N sodium thiosulfate [C (Na_2S_3O)] solution with vigorous stirring and using the starch solution (1g/100 mL) as indicator. A blank test was performed simultaneously. The peroxide value in milliequivalent O_2/kg was calculated according to the equation:

$$Ip(\text{meqO2/kg d'huile}) = \frac{10 * (V - V0)}{m}$$

With
m: mass of test portion in grams; *V*: volume of sodium thiosulfate solution poured in mL; *V0*: volume of sodium thiosulfate solution for the blank test in mL.
Para-anisidine Index (p-anisidine)

The para-anisidine index was determined according to the method described by AFNOR (NF ISO 6885) [**13**]. The determination of the p-anisidine index is based on the following principle: in acetic acid medium, p-anisidine reacts with conjugated aldehydes resulting from the oxidation of lipids to form yellow compounds which absorb at 350 nm. The p-anisidine index is defined as 100 times the absorbance measured at 350 nm of a solution resulting from the reaction between 1 g of lipids and 100 mL of solvents and reagents. This method is more sensitive in the case of unsaturated aldehydes than saturated aldehydes because the coloured complex formed from unsaturated aldehydes absorbs more strongly at this wavelength. It allows products such as carbonyls, 2-alkenals and 2,4 dienals to be determined. This index correlates well with the amount of total volatile products [**14**] and is a reliable indicator of oxidative rancidity of lipids [**15**].

Totox Index
The Totox Index reflects the actual oxidation of the oils. It has been calculated according to the following formula IT = 2 IP + IpA (IT: Totox number; IP: peroxide number; IpA: p-anisidine number) [**16, 17**].

RESULTS

Evaluation of the UV-Visible spectrum

The UV-visible spectrum of vegetable oil extracts has been presented in **Figure 1** and consists of four superimposed curves, each with maximum absorbances at 240 nm and 300 nm. The maximum absorbance values obtained at 240 nm are 0.92, 0.90, 0.84 and 0.80 respectively for *Ricinodendron heudelotii, Citrullus colocynthis, Cyperus esculentus* and *Irvingia gabonensis* oils. At 300 nm these values are 0.87, 0.83, 0.78 and 0.74 respectively. The absorbance of the oils studied decreases rapidly from 0.70 to about 0.02 between 300 and 400 nm.

Evolution of the acid index during storage

The acidity of vegetable oil extracts has gradually increased to maximum values of 15.3% for *Ricinodendron heudelotii*, 16.2% for *Citrullus colocynthis*, 21.6% for *Cyperus esculentus* and 16.9% for *Irvingia gabonensis* from the 120th day of storage. Beyond thisday, the acidity values remained constant (**Figure 2**).

Evolution of the peroxide index during storage

The peroxide index of vegetable oil extracts decreases gradually and reaches limit values of 40.08 meqO2/kg for *Ricinodendron heudelotii*, 45.2 meqO2/kg for *Citrullus colocynthis*, 79.35 meqO2/kg for *Cyperus esculentus* and 14.1 meqO2/kg for *Irvingia gabonensis* from the 120th day of storage. Beyond this day, the peroxide index values tend to stabilize (**Figure 3**).

Evolution of the p-anisidine index during storage

The p-anisidine index of vegetable oil extracts increases progressively and reaches limit values of 400.14 for *Ricinodendron heudelotii*, 74.32 for *Citrullus colocynthis*, 46.86 for *Cyperus esculentus* and 3.16 for *Irvingia gabonensis* from the 150th day of storage. The oil extracted from the seeds of *Ricinodendron heudelotii* has the highest p-anisidine index. (**Figure 4**).

Evolution of the iodine index during storage

During storage, the iodine index of *Ricinodendron heudelotii, Citrullus colocynthis* and *Cyperus esculentus* oils decreased significantly while the iodine index of *Irvingia gabonensis* remained stable (**Figure 5**).

Evolution of oxidation indices during storage

The changes in the peroxide index, p-anisidine index and Totox index of *Ricinodendron heudelotii, Citrullus colocynthis, Cyperus esculentus* and *Irvingia gabonensis* oils after 180 days of storage at room temperature (25°C) are shown in **Figure 6**.Analysis of these different curves shows a growth of the p-anisidine curve for the four oils subjected to the oxidation resistance test from the sixteenth week onwards. *Citrullus colocynthis* oil shows the highest growth in p-anisidine index from week 16 onwards, while *Irvingia gabonensis* oil shows the greatest stability due to the consistency of its p-anisidine index.

DISCUSSION

Acidity, peroxide index and p-anisidine index are alteration parameters whose monitoring during storage makes it possible to highlight the phenomenon of rancidity of vegetable oils **[12, 18].** The progressive increase in acidity of *Ricinodendron heudelotii, Citrullus colocynthis, Cyperus esculentus* and *Irvingia gabonensis* oils during storage results from the hydrolysis of

the constituent triglycerides **[11].** As regards the peroxide index, its increase during storage is an indicator of the production of primary oxidation compounds such as hydroperoxides and epoxides in vegetable oils [**19, 20**]. On the other hand, the increase in p-anisidine index is an indicator of the formation of secondary oxidation compounds **[21].**

The recommended acidity limit value for edible vegetable oils of 3% [**22**] was reached after 30 days of storage for *Ricinodendron heudelotii* and *Cyperus esculentus* oils. For *Irvingia gabonensis* oil, this limit value was reached after 45 days of storage. For *Citrullus colocynthis* oil, this value was significantly exceeded only after two weeks of storage, which shows a rapid rancidity of this oil. Taking into account the limit value of the peroxide index (10 meqO2/kg) for edible vegetable oils [**23**], the rancidity process is detectable after 15 days of storage for *Ricinodendron heudelotii*, *Citrullus colocynthis* and *Cyperus esculentus* oils. In the case of *Irvingia gabonensis* oil, this phenomenon is detectable after 90 days of storage. Although these oils cannot be used for food purposes beyond the storage periods mentioned, they could nevertheless be used in the manufacture of derived products such as soaps. In addition, the shelf life of these oils could be extended by packaging them in appropriate containers at low temperatures or by the addition of antioxidants such as butylated hydroxyanisole [**12**].

The study of the oxidation resistance of *Ricinodendron heudelotii*, *Citrullus colocynthis*, *Cyperus esculentus* and *Irvingia gabonensis* oils is a means of measuring their oxidative stability under storage conditions. The increase in the peroxide index as a function of storage time at 25°C during the first phase of the oxidation process is due to the production of hydro-peroxides [**4**]. On the other hand, the decrease in the peroxide index during the second phase is related to the instability of the hydro-peroxides [**10**]. The oxidative stability study shows that the oil extracted from *Irvingia gabonensis* would be good for frying compared to the oils of *Ricinodendron heudelotii*, *Citrullus colocynthis* and *Cyperus esculentus*. This result could be explained by the low level of unsaturation of this oil [**24, 25**]. Under the same experimental conditions, the oil extracted from *Irvingia gabonensis* is more stable to oxidation than coriander and niger seed oils used as dietary supplements [**26**].

The decrease in the peroxide index correlates with the increase in the p-anisidine index. This reflects the conversion of primary oxidation products to secondary oxidation products [**27**]. The evolution of the p-anisidine index remains stable during the first eighteen weeks of storage. This reflects the oxidation stability of these oils [**28**]. The decrease in absorbance (from 0.7 to 0.02) between 300 and 400 nm gives the oils studied the advantage of being used in cosmetics, as ingredients in the formulation of anti-ultraviolet cosmetic products. Indeed, this wavelength range corresponds to ultraviolet A and B, whose absorption leads to cellular damage in the skin [**10**].The evolution of the oxidation indices during the storage of the oils of the study at room temperature, highlighted a time limit of two to three months for food use. These times could be extended under industrial conditions of conservation. The resistance of *Irvingia gabonensis* oil to oxidation is related to its high content of saturated fatty acids [**24, 25**]. Proper packaging away from oxygen in the air could extend the shelf life of the vegetable oil extracts in the study.

CONCLUSION

At the end of this study, it should be noted that acidity, peroxide index and p-anisidine index are parameters of alterability whose monitoring during storage can highlight the phenomenon of rancidity of vegetable oils. The peroxide index and the p-anisidine index seem to be good

indicators of the oxidation state and oxidative stability of the products. However, these elements remain to be confirmed in a more detailed study of product ageing. It appears that the oils of *Ricinodendron heudelotii*, *Citrullus colocynthis*, *Cyperus esculentus* and *Irvingia gabonensis* have chemical and biochemical potential that could be used in the food, cosmetic and pharmaceutical fields.

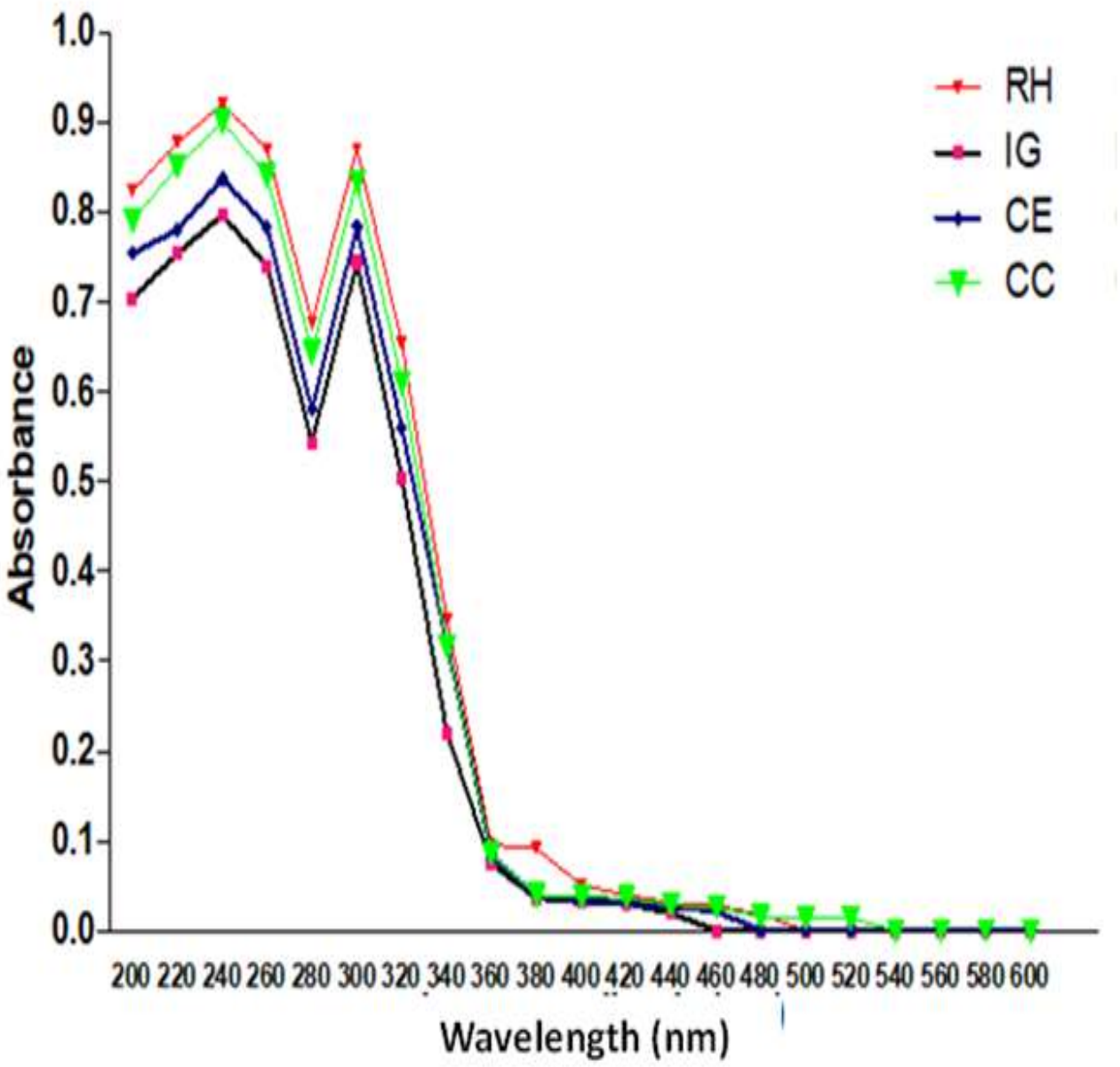

Figure 1: UV-visible spectrum of vegetable oil extracts
RH : *Ricinodendron heudelotii*, CC : *Citrullus colocynthis*, CE *: Cyperus esculentus*, IG :*Irvingia gabonensis.*

The UV-visible spectra were obtained by measuring the absorbance between 200 and 600 nm of vegetable oil extracts diluted 1% in hexane [**10**].

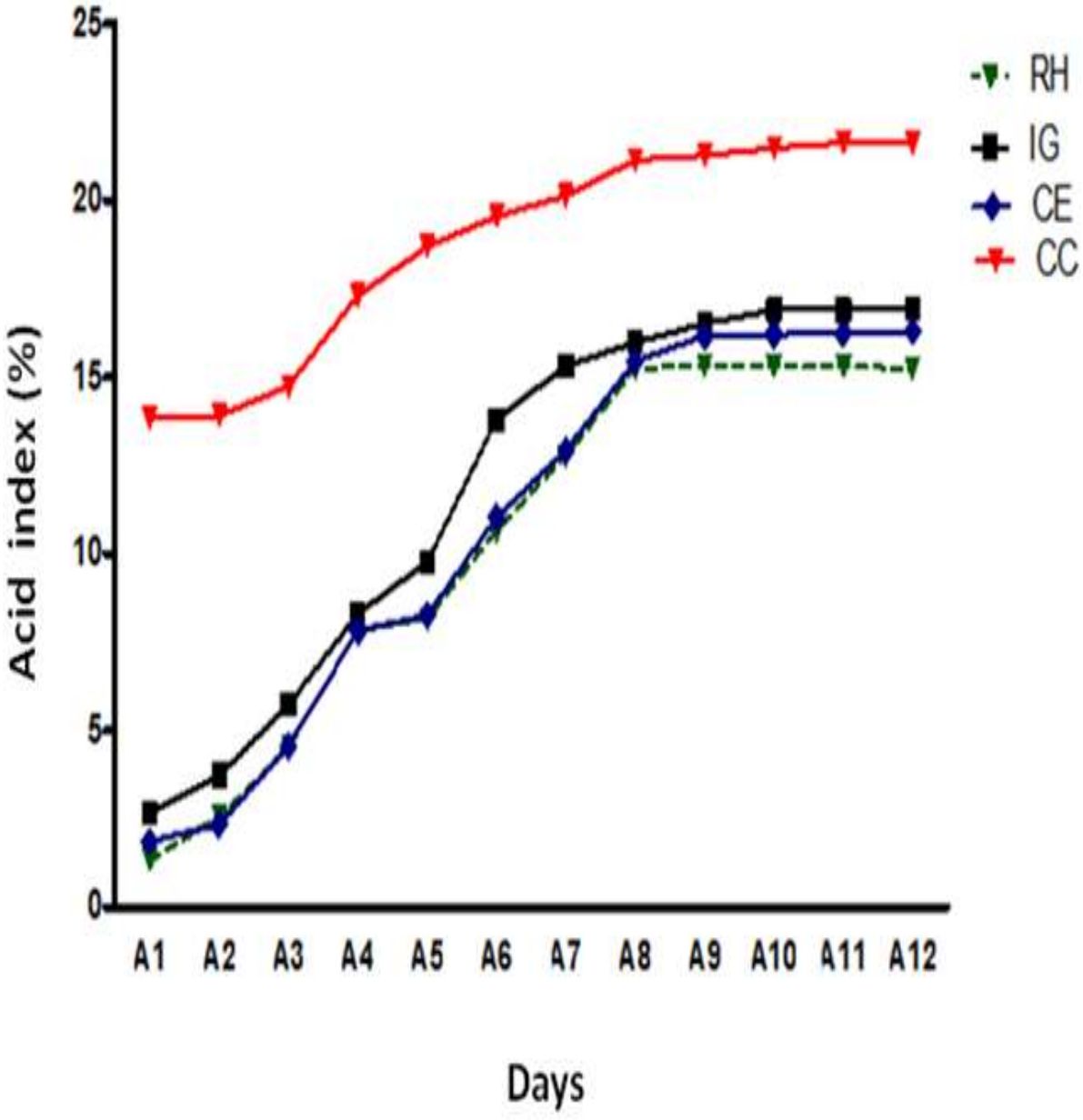

Figure 2: Variation in acidity of vegetable oil extracts during storage at room temperature (25°C).
RH : *Ricinodendron heudelotii*, CC : *Citrullus colocynthis*, CE *: Cyperus esculentus*, IG : *Irvingia gabonensis.*
The acidity of vegetable oil extracts stored at room temperature (25°C) in the dark for 180 days was measured fortnightly by triplicate tests to obtain the mean of the values affected by standard deviations [**11, 12**].

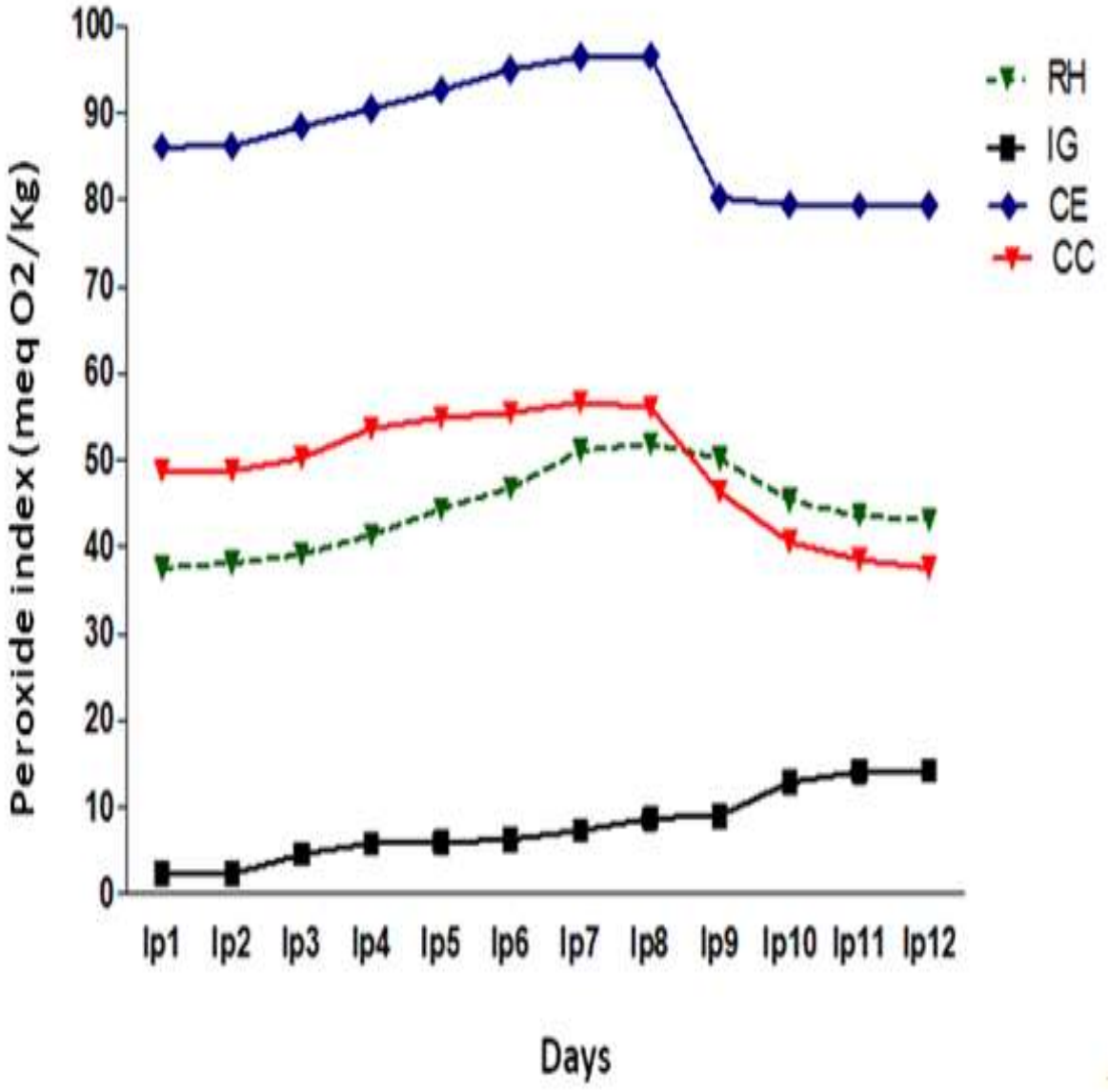

Figure 3: Variation of the peroxide value of vegetable oil extracts during storage at room temperature (25°C).
RH : *Ricinodendron heudelotii*, CC : *Citrullus colocynthis*, CE *: Cyperus esculentus*, IG : *Irvingia gabonensis.*

The peroxide index of vegetable oil extracts stored at room temperature (25°C) in the dark for 180 days was measured every two weeks by triple testing to obtain the mean of the values affected by standard deviations [**11, 12**].

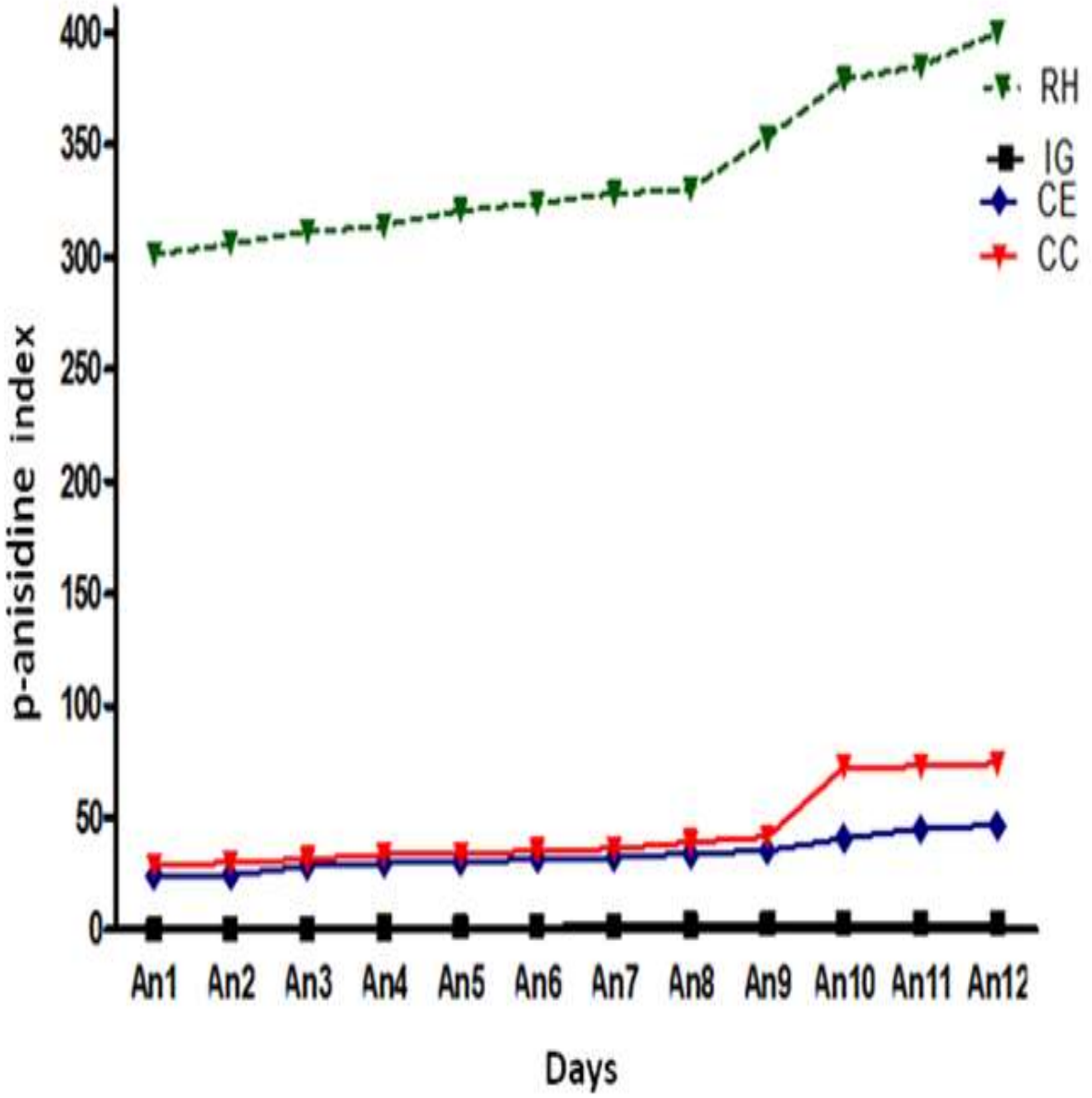

Figure 4: Variations in the p-anisidine index of vegetable oil extracts during storage at room temperature (25°C).
RH : *Ricinodendron heudelotii*, CC : *Citrullus colocynthis*, CE *: Cyperus esculentus*, IG : *Irvingia gabonensis.*

The p-anisidine value of vegetable oil extracts stored at room temperature (25°C) in the dark for 180 days was measured fortnightly in triplicate tests to obtain the mean of the values affected by standard deviations [**13**].

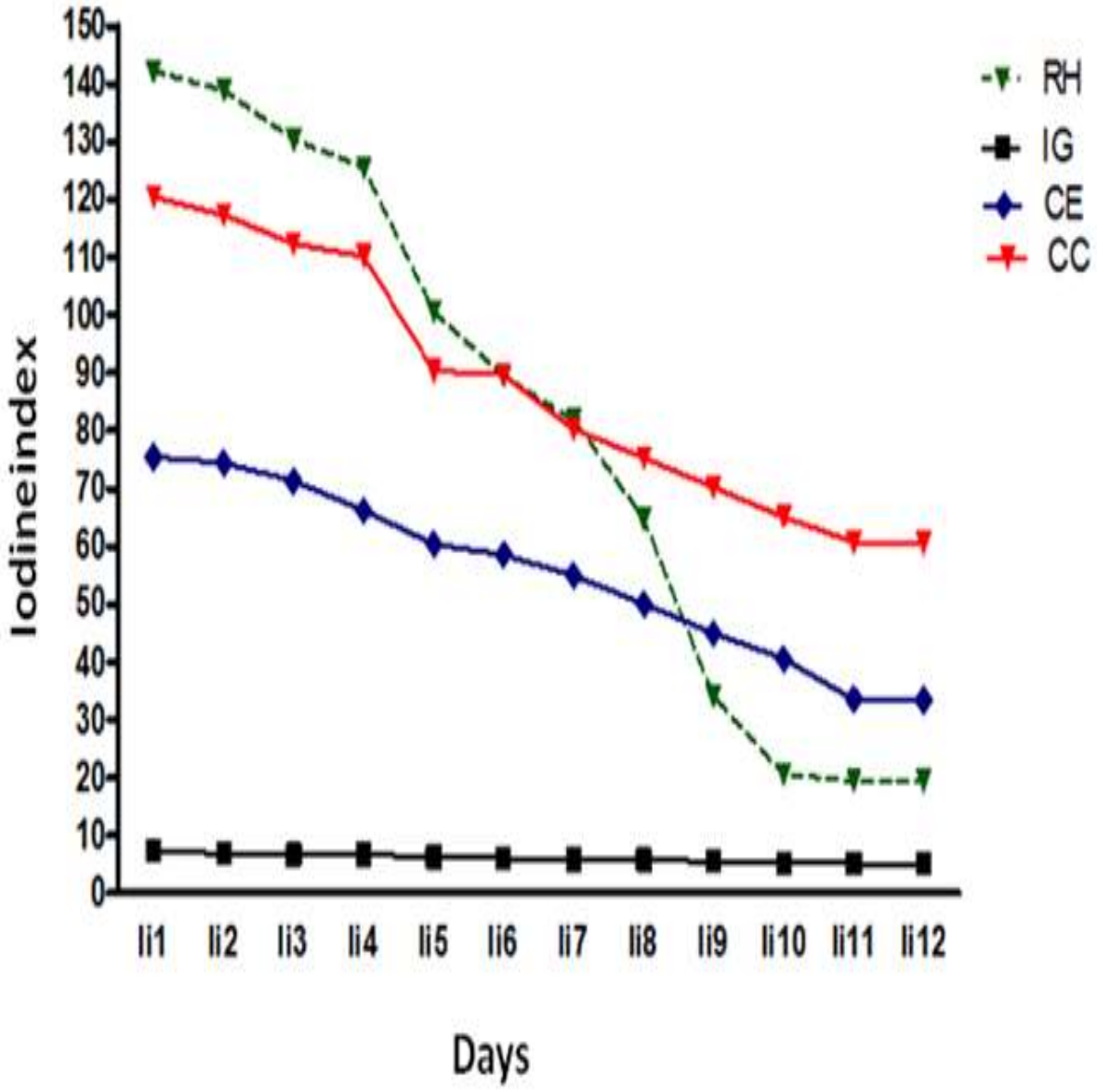

Figure 5: Variation of the iodine index of vegetable oil extracts during storage at room temperature (25°C).
RH : *Ricinodendron heudelotii*, CC : *Citrullus colocynthis*, CE *: Cyperus esculentus*, IG : *Irvingia gabonensis.*

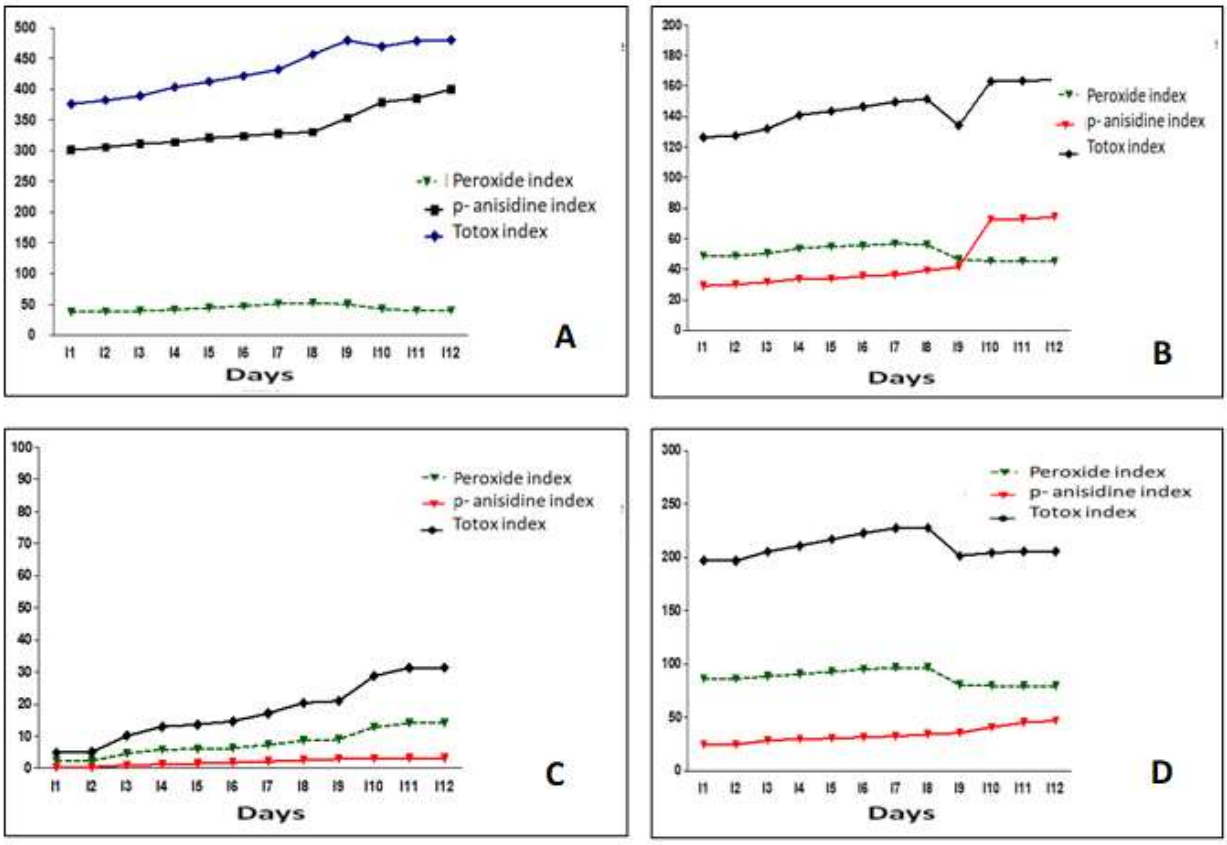

Figure 6: Variation in peroxide index, p-anisidine index and Totox index of vegetable oil extracts of *Ricinodendron heudelotii* (A), *Citrullus colocynthis* (B), *Cyperus esculentus* (C) and *Irvingia gabonensis* (D) during storage at room temperature (25°C).

Conflicts of interest

The authors declare that they have no conflicts of interest.

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