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# Physicochemical characterization of neem oil crude and epoxide from Senegal

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**Abstract**

The study is based on the physicochemical characterization of crude neem oil and five samples of the same neem oil epoxidised under different operating conditions. The results showed the correct course of the reaction with a considerable reduction of the ethylenic unsaturations of the oil whose iodine index varies from 063.50 $gI_2$/100g (crude oil) to 013.2$gI_2$/100g (epoxidized oil) corresponding to a molar ratio $CH_3COOH/H_2O_2/C=C$ : 1/1/1. These values correspond to an epoxy value of 75.54%. Furthermore, the clarity of the epoxidised oil decreases when the reaction temperature reaches 60.00 °C. Furthermore, minerals such as Cr (4.06-3.99), Se (0.23-0, 23), Ni (0.39-0.36), V (0.89-0.78), Fe (2.15-2.07), Zn (0.23-0.23), Mn (0.36-0.36), Co (0.38-0.31), Cu (1.21-1.12) and Mo (0.33-0.29), whose concentrations remain below 5g/$cm^3$. The small decrease in the content of some elements after the reaction could be due to handling errors or interactions of the metal catalysts during the oil epoxidation process. The work revealed that the reaction had no influence on the refractive index with a value of 01.46 which remained constant before and after the reaction. The statistical analysis revealed that the PCA axis1 contains 61.6% inertia against 17.2% of the axis2. In addition, the variance made it possible to visualize the distribution of individuals (epoxidised samples on either side of the negative direction of dimension 1 along dimension 2), in contrast to the crude oil HNNE. The latter remains the only individual in the positive space of the orientation of the two axes with a perfect correlation with axis1. The study showed that at 60 °C, the clarity of the oil decreases. The epoxidised oil requires washing to reduce the acidity. There is a fair amount of oxidation by-products in the initial state of the oil with K270 nm of 0.355. The oil retains its quality after the reaction with K232 nm and K270 nm values below the there should values of the standards with some acceptability due to the operating condition.

## 1. Introduction

Vegetable oils have long been the subject of research studies. In particular, some successful work has been done on neem oil for the production of bio-pesticide (Sane *et al.* 2018), synthesis of alkyl resins ( Jack 2016), study of antimicrobial activity (Jijakli *et al.* 2009) but also physicochemical and microbiological characterization (Adamou *et al.* 2020) among others.

However, the evolution of the physical and chemical parameters of the oil after the epoxidation reaction compared to the crude oil is still unknown. This is due to the lack of studies in this area. Indeed, in many epoxidised oils such as cottonseed oil (Zheng 2016), mahua oil (Coud *et al.*, 2006), soybean

oil (Vianello *et al.* 2018 ; Rahmah *et al.* 2017), jatropha oil (Goud *et al.* 2007), there is ambiguity about their states in relation to the experimental conditions. In 2016, (R. Jack 2016) conducted a comparative study of modified alkyd resins from epoxidised and crude neem oil without going into the physicochemical characterization of the epoxidised oil. In view of the interest aroused by this previous work, it is advisable to extend the study to better understand the nature of the oil after the epoxidation process. The latter is the result of a mixture between the oil, hydrogen peroxide (30%) and acetic acid (99.9%) as emphasised by authors such as (BALDE et al. 202 ; Milchert *et al.*, 2016 ; Coud, Pradhan, and Patwardhan 2006) during their research work.

As a result of this problem, it is advisable to proceed with a characterization using adapted physicochemical analysis techniques. To do this, we will start by studying the crude oil and finish with the epoxidised oil, then make a comparison in order to understand the variation of physical and chemical parameters according to the experimental conditions. Thus, we will basically rely on statistical applications such as principal component analysis (PCA) in the sense of having a real idea of the existing correlations between physical and chemical parameters. In this way, the links between variables and the similarities between individuals can be identified, and then perspectives can be developed in this new dynamic.

## 2. Material and methods

### *2.1. Material*

Hydrogen peroxide (30%), acetic acid (99.9%) were used in the proportions shown in **Table 2**. However, a constant mass of 200 g of neem oil was used in all experiments. The determination of the mineral elements was carried out using the Niton XL3S X-ray fluorescence spectrometer. Thus, the instruments listed in **Table 1** were used to measure the physicochemical parameters. In addition, the AW-meter (HygroPalm-HP23-AW-A, rotronic) was used to measure the activity of the crude oil (reference) and the epoxidised crude oil samples. The extinction coefficients K232 nm and K270 nm were determined according to the French standard NF T60-223 using a UV spectrophotometer (SPECORD 200 PLUS).

### *2.2. Methods*

The reaction is carried out according to Prileshev's in situ epoxidation protocol. However, the physicochemical characteristics of the oil are carried out according to the references in **Table 1**. Thus, the determination of the mineral elements is done according to the protocols written by (Malik *et al.* 2019 ; Palmer 2011 ; Robinson *et al.* 2012). The HCl/pyridine method used by (Bertho 2011) is used to determine the epoxy value of the oil after the reaction.

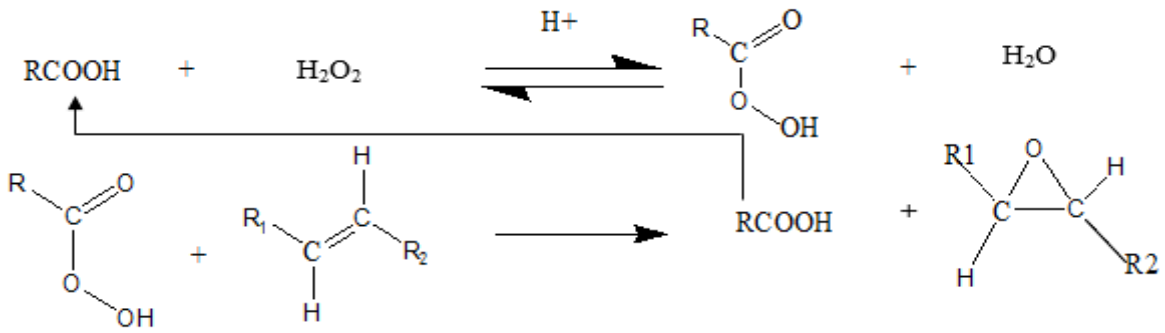



**Figure 1**. *Epoxidation reaction of the oil* (Coud *et al.*, 2006)

## 3. Results and discussion

The results presented are derived from the analysis of crude neem oil, otherwise known as non-epoxidised neem oil *(a)* and epoxidised or crude neem oil *(b)*. Thus, the physicochemical characteristics of the oil before and after the reaction are recorded in **Table 1** and **Table 2** respectively.


**Figure 2**. *Epoxidised (a) and crude neem oil samples (b)*

These five samples of epoxidised neem oil show different color appearance from each other *(a)* but also from the crude oil *(b)*. The operating conditions of the reaction are responsable for the change in color of these samples *(a)*.

### *3.1. Characterization of non epoxidised neem oil (HNNE)*

Some physical and chemical parameters of the non-epoxidised neem oil are measured and are recorder in **Table 1**.

**Table 1.** *Physicochemical parameters*

| Measured parameters | Values | Standards and equipment |
|---|---|---|
| a* | 002.05 | Colorimeter (CM-5, konica sensing Americas) |
| b* | 118.80 | Colorimeter (CM-5, konica sensing Americas) |
| L* | 080.78 | Colorimeter (CM-5, konica sensing Americas) |
| Unsaturated fatty acid content | 034.51 | NF EN ISO 12966-1 |
| Refraction value (nD, 25°C) | 001.46 | Refractometer(EXACTA-OPTECH,MODRMT) |
| Iodine (g $I_2$/100g) | 063.50 | Wij's |
| Acid value (mg KOH/g oil) | 007.47 | NFV 03-906 |
| Peroxide value (meq $O_2$/kg) | 000.80 | NF T 60-220 |
| Humidity (%) | 000.01 | NF V03-905 |
| Dry matter (%) | 099.99 | NF V03-905 |
| Density | 000.87 | AFNOR |
| Temperature | 027.30 | Thermometer |
| pH (25°C) | 006.65 | pH-meter |

*$L^*$: brightness represents the difference between light (where $L^* = 100$) and dark (where $L^* = 0$), $a^*$: represents the difference between green ($-a^*$) and red ($+a^*$), $b^*$: represents the difference between blue ($-b^*$) and yellow ($+b^*$).*

### *3.2. Characterization of epoxidised neem oil (ENO)*

Five samples from different experimental conditions were characterized. The study will help to understand the state of the epoxidised oil and to make the comparison with the crude oil. Therefore, several factors such as temperature, heating time and reagents are evolved in the epoxidation process of the oil.

### *3.2.1. Physicochemical parameters*

**Table 2** shows the operating conditions of five experiments with epoxidised neem oil and the results of the physicochemical parameters measured.

**Table 2.** *Operating conditions of the experiments and measured physicochemical parameters of the epoxidised oil*

| Parameters and reagents | | Eexp1 | Eexp2 | Eexp3 | Eexp4 | Eexp5 |
|---|---|---|---|---|---|---|
| Time (h) | Operating conditions | 03.00 | 07.00 | 03.00 | 03.00 | 03.00 |
| Temperature (°C) | | 45.00 | 45.00 | 60.00 | 60.00 | 60.00 |
| $H_2O_2$ (mole) | | 01.00 | 01.00 | 01.00 | 02.00 | 01.00 |
| Acetic Acid (mole) | | 01.00 | 01.00 | 01.00 | 01.00 | 02.00 |
| a* | | 02.03 | 01.08 | 02.97 | -02.55 | -00.60 |
| b* | | 90.24 | 90.33 | 79.30 | 59.60 | 61.01 |
| L* | | 74.77 | 79.42 | 65.13 | 79.22 | 75.18 |
| Acid value (mg KOH/g oil) | | 88.08 | 86.96 | 94.81 | 65.08 | 98.18 |
| Peroxide value (meq $O_2$/kg) | | 00.77 | 03.07 | 02.94 | 03.33 | 00.83 |
| Iodine value (g $I_2$/100g) | | 23.35 | 23.86 | 013.2 | 24.74 | 22.97 |
| Refraction value (25 °C) | | 01.46 | 01.46 | 01.46 | 01.46 | 01.46 |
| Temperature (°C) | | 26.40 | 26.50 | 26.00 | 26.60 | 26.30 |
| Density | | 00.95 | 00.92 | 00.92 | 00.94 | 00.91 |
| Dry matter (%) | | 93.00 | 94.34 | 87.76 | 90.32 | 96.39 |
| Humidity (%) | | 07.00 | 05.66 | 12.24 | 09.68 | 03.61 |
| pH (25 °C) | | 01.41 | 02.07 | 02.20 | 01.20 | 01.11 |

*E: sample, exp: experiment, L*: lightness represents the difference between light (where L*=100) and dark (where L*=0), a*: represents the difference between green (-a*) & red (+a*), b*: represents the difference between blue (-b*) & yellow (+b*)*

It can be seen that by varying the time, temperature and the amounts of $H_2O_2$ and acetic acid: in the first instance, we proceeded to vary the time from 03.00 to 07.00 h by fixing the other parameters. The above table shows us that a* decreases from 02.03 to 01.08 values much lower than that of HNNE. Reading this **Table 2**, it appears an increase for b* and L*. Indeed, the values obtained are lower than that of HNNE. When varying the temperature from 45.00 to 60.00 °C by fixing the other parameters, a* increase remains higher than that of HNNE, while b* and L* decrease and remain below that of HNNE. In Eexp3 and experiment 4, an increase in $H_2O_2$ from 01.00 to 02.00 moles by fixing the other parameters results in decreasing the values of a*, b* and increasing that of L*. In the following, we chose to increase the amount of acetic acid. The addition of 01.00 to 02.00 moles in experiment 3 and in experiment 5 results in increasing the values of a*, L*. On the other hand, that of b* decreases and lower than that of HNNE. It can be deduced that temperature is the factor responsible for the decrease in oil clarity. It also contributes to the increase in red coloration of the epoxidized neem oil compared to the crude oil ranging from 02.03 to 02.97. Thus, the yellow coloration characterized by b* decreased in all the experiments and lower than that of the crude oil. This varied according to the operating conditions. However, the consequent decrease in a* characteristic of the red colour is caused by the variation of 01.00 to 02.00 moles of acetic acid and hydrogen peroxide noted at low heating times. This results in a green shade of Eexp4 and Eexp5. The operating conditions in experiment 3 where the clarity decreased could justify the denaturation of the oil. In other words the conversion of the double bonds. The time and the reagents cause a decrease in the red colour of the oil apart from the temperature. The refractive index of the two oils remains unchanged. The same values are found in the work of (Adamou et al. 2020). The moisture content of the epoxidised samples increased significantly from 03.61 to 07.00%, which is much higher than the crude oil. The latter has no moisture compared to those of

Benin ranging from 20 to 49%. This could be due to the extraction conditions. In addition, the mixing of the oil with hydrogen peroxide and acetic acid consequently increased the acidity of the epoxidised oil with a low pH (Eexp1). However, with a longer reaction time, as shown in Eexp1 and Eexp2, the acidity decreases. This may be due to the reaction between hydrogen peroxide and acetic acid. The same observation applies to Eexp1 and Eexp3 when the temperature increases. The effect of the latter led to a considerable decrease in the iodine value from 063.50 (crude oil) to 013.2 g $I_2$/100g (epoxidised oil) under Eexp3. On the other hand, when hydrogen peroxide is varied from 01.00 to 02.00 moles, the acid number decreases. It increases when the amount of acetic acid increases from 01.00 to 02.00 moles. This implies that there is a minimum time for the hydrogen peroxide and acetic acid to react so that the acidity can decrease. The slight increase in temperature of the samples compared to room temperature may be the cause of the thermometer. Among the samples Eexp5 contains a lower pH. The consequence of this decrease could be due to the increase in the amount of acetic acid from 01.00 to 02.00 moles (Eexp4 and Eexp5). In addition, the high acid value of the samples is explained by the presence of acid in the samples. This is because the samples were not washed with water and this consequence can be observed in the pH.

### *3.2.2. Water activity and specific extinction coefficients*

The induced oxidation of neem oil through the epoxidation reaction was the subject of this diagnostic water activity and specific extinction coefficients (K232 nm ; K270 nm) of the crude oil (**Figure 2** (a)) as well as the epoxidised oil samples (**Figure 2** (b)) shown in **Figure 3**. This study aims to understand the presence of primary (K232 nm) and secondary (K270 nm) oxidation products and the influence of water. These two parameters not only allow us to evaluate the primary and secondary oxidation products but also to show the degree of exposure of the extracted oils to oxidation (Samba et al. 2022).

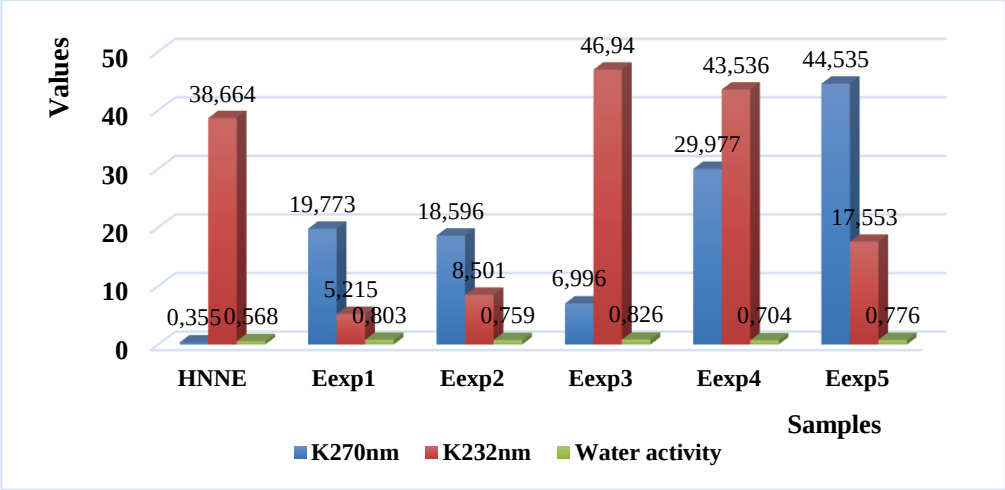



**Figure 3.** *Comparative study of water activity and specific extinction coefficients (K232 nm; K270 nm) of crude oil and epoxidised samples*

It can be seen in this **Figure 3** that the water activity equal to 0.568 of the crude oil (HNNE) is lower compared to the other samples whose values vary between 0.704 and 0.826. This justifies an absence of water in the crude oil. These results also confirm the difference in moisture content observed between the crude oil in **Table 1** and the samples in **Table 2** with values of 0.01% and (3.61 to 12.24%) respectively. All five samples have activities close to 1. This is due to the water generated during the reaction. The crude oil (HNNE) with a molar extinction coefficient (K270 nm) of 0.355 shows little or

no secondary oxidation products compared to the five samples with K270 nm coefficients between 6.996 and 44.535. According to (Monge and Oxydation n.d.) the non-volatile secondary oxidation compound are mainly monomeric oxidised triglycerides with at least one altered fatty acid bearing a hydroxyl, carbonyl or epoxide functional group. Thus, the high K270 nm values of the five samples could be due to their epoxidation. On the other hand, all the samples in this **Figure 3** show more or less important K232 nm molar extinction coefficients. The primary oxidation products of lipids are free radicals, hydroperoxides (Pratt, 2011) etc. The low content of K232 nm found in samples Eexp1 (5.215), Eexp2 (8.501) and Eexp5 (17.553) may be due to the maximum presence of secondary oxidation products found with respective values of 19.773; 18.596; 44.535. The values of these three samples except Eexp5 remain below the limit values of the specific extinction coefficients at 232 nm and 270 nm of the extracted oils fixed by the International Olive Council (IOC) and Codex Alimentarius 41 and 42(Samba et al. 2022) respectively. Samples Eexp3 and Eexp4 have K232 nm coefficients of 46.94 and 43.536 above the values of the two standards. So does Eexp5 with a K270 nm above the values of these standards. The rise in temperature from 45 to 60 °C in these last three experiments could be the origin of these variations. The oxidation of fats is linked to factors: physicochemical (temperature, oxygen, light), to the composition of the products and to the initial state of the raw materials (unsaturation of the lipids, transition metals, initial state of oxidation, oxidizing enzymes), technological (mechanical treatment: grinding, emulsion; thermal treatment: cooking, freezing), and to the conservation (duration) (Ag 2004). These two coefficients become more important when the temperature reaches 60 °C.

### *3.2.3. Effect of reagents on the iodine value*

**Figure 4** shows the evolution of the iodine value as a function of the variation of the reagent masses (acetic acid, hydrogen peroxide) according to the experiments carried out.

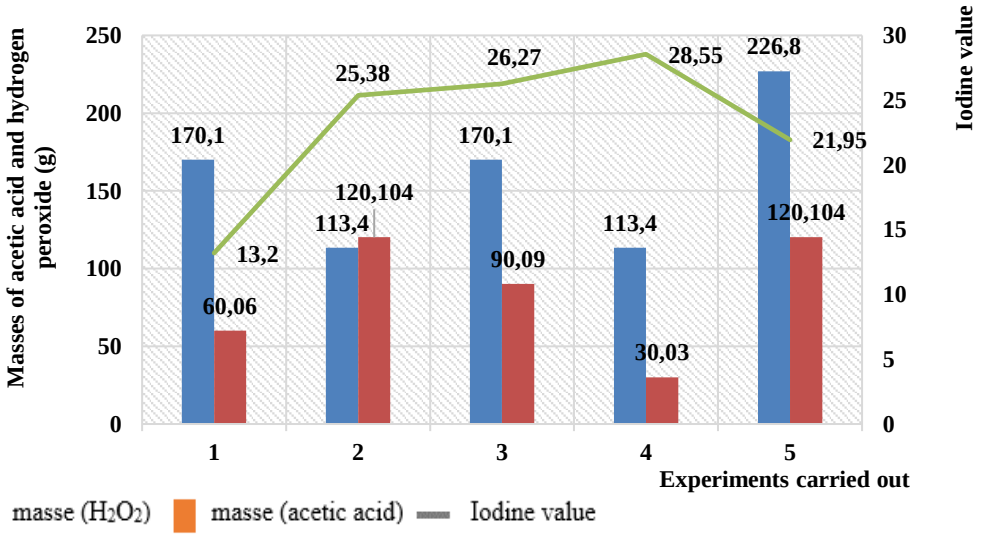



**Figure 4.** *Variation of the iodine value according to the masses of acetic acid and hydrogen peroxide in each experiment*

Eexp1, 3 and 4 show that when the mass of hydrogen peroxide is much higher than that of acetic acid, the iodine value increases. This index reaches its limit and decreases from 28.55 to 21.95 when the masses become too large between Eexp4 and Eexp5. Thus, there is a limit value for which the increase in the amount of material proportionally related to the mass of the reactants will only lead to a decrease in the iodine value or the unsaturations contained in the oil. Consequently, the first scenario,

i.e. Eexp1, 2, 3 and Eexp4, explains the normal course of the reaction between hydrogen peroxide and acetic acid to form per-formic acid (**Figure 1**). Thus, the drop in iodine value could be due to the action of the latter on the fatty substance (crude neem oil) resulting in the conversion of the unsaturations to give the epoxy and then the regeneration of the acetic acid according to the reaction in **Figure 1**. A lower iodine value is obtained in experiment 1 with respective mass quantities of hydrogen peroxide and acetic acid of 170.1 and 60.06 g. This will correspond to a maximum reduction of ethylenic double bonds with an iodine value of 013.2 compared to 063.50 g $I_2$/100 g of crude oil. This is equivalent to a molar ratio of $CH_3COOH/H_2O_2/C=C$: 1/1/1. This result is close to those obtained by (Milchert, Malarczyk-Matusiak, and Musik 2016 ; Maria Suzana Silva *et al*. 2015). This ratio corresponds to an epoxy value of 75.54%.

### *3.2.4. Composition of mineral elements*

This is carried out before and after the epoxidation reaction in order to understand their presence in the oil following the operating conditions. But also the effect that this oil could have on plants in terms of heavy metal absorption, as HNNE is used in many works as a bio-pesticide (Sane *et al.* 2018), synthesis of alkyd resins (Adamou et al. 2020), antibacterial activity (R. Jack 2016). The results of this analysis are shown in the **Figure 5** below.

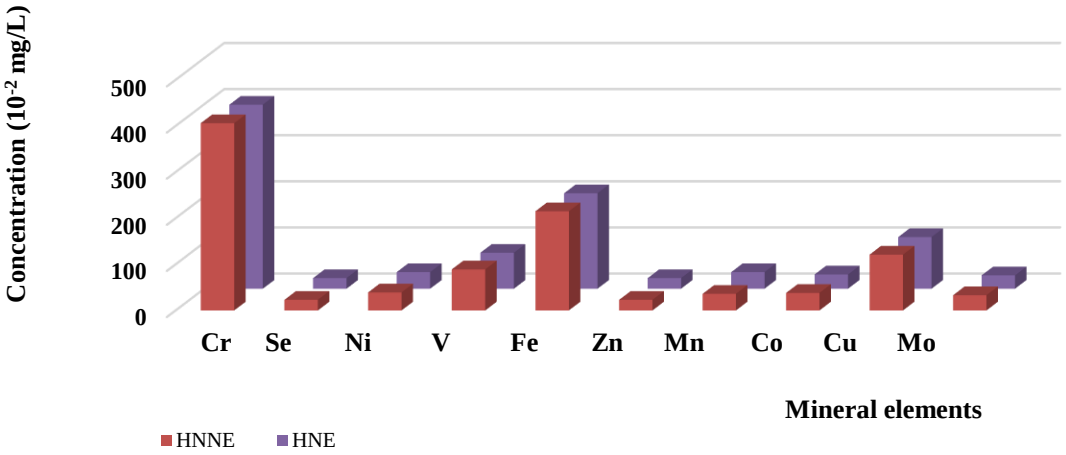



**Figure 5.** *Diagram comparing the concentration of mineral elements before and after the epoxidation reaction of the oil*

The data show that heavy metals are present in trace amounts in these oils, as the limit value recommended by the standard is 5 g/cm3. These values indicate that their presence in the oil is not influenced by any of the operating parameters (time, temperature). In fact, the small decrease in the concentration of these elements after the reaction could be due to handling errors or interactions of the metal catalysts on the epoxidation of the fatty substances, as in the cases of Cu, Cr, Co, Ni, Se, V, and Zn (Maria Suzana Silva *et al.* 2015 ; Louis and Strasbourg 2006). The oil in all these uses as bio-pesticide (Sane *et al.* 2018), bio-energy (Louis and Strasbourg 2006) and bio-fuel (ICDES 2010) cannot present any disadvantages on the soil compared to the minerals shown in **Figure 5**. Moreover, the leaves and bark contain respectively 3.4 and 4% of minerals (Zheng 2016), which are richer than the Senegalese neem seed oil. The presence of high levels is more frequently found in cattle and horses than in pigs (www.favv-afsca.be/denreesalimentaires/contaminants/metauxlourds). This is due to their life span and grazing. Food is the only route of exposure to heavy metals for humans. Humans may consume these minerals indirectly via animals because heavy metals accumulate mainly in the liver

and kidneys of animals (www.favv-afsca.be/denreesalimentaires/contaminants/metauxlourds) that consume the leaves or seeds.

## 4. Principal component analysis of crude and epoxidised neem oil

The amount of data collected in **Table 1** and **Table 2** is the subject of this principal component analysis (PCA). The aim is to synthesise this wealth of information on the physicochemical parameters of HNNE and HNE. This is done in order to better interpret the results, i.e. to identify the links between variables (physicochemical parameters) and the similarities between individuals (HNNE and HNE).

### *4.1. Two-dimensional representation of variables and individuals*

The interpretation of the factorial axes is done sequentially, for each axis (dimension) and each point cloud (circle).

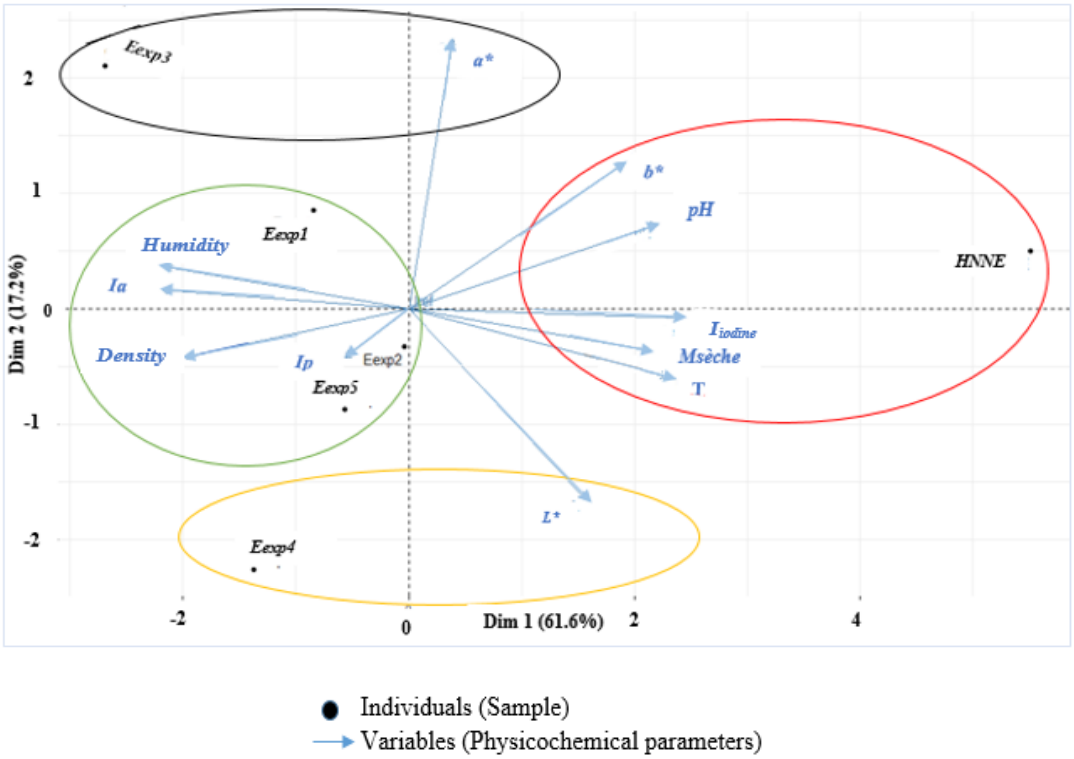



**Figure 6**. *Representation of dimension 1 (Dim-1), dimension 2 (Dim-2) individuals and variables*

**Figure 6** contains 61.6% inertia on the horizontal axis and 17.2% on the vertical axis. The variables contained in the red and green circles are well colored in the first dimension except for the peroxide index (PI). The interpretation of the latter cannot therefore be made with confidence. However, active oxygen is present in the samples as a result of the epoxidation reaction. This is generated by hydrogen peroxide as shown in Eexp4 of **Table 2**, where the variation from 01.00 to 02.00 moles of $H_2O_2$ causes a higher value of peroxide value 03.33 meq $O_2$/kg compared to crude oil 000.80 meq $O_2$/kg. However, this is below the three should value of 20 meq $O_2$/kg recommended by the standard (Des et al. 2004). The same observation can be made for the variables contained in the yellow and black circles in relation to the second dimension. The variable Iref (refractive index) is not correlated by the two dimensions and this is justified by its value which remains constant in all individuals. The individuals close to the axes and far from the center of the PCA mapping (**Figure 6**) are respectively (HNNE, Eexp3) and (Eexp4, Eexp3). The individuals Eexp2, Eexp3 and Eexp4 are better correlated to axis2 than axis1, unlike Eexp1, Eexp5 and HNNE which are well represented with

respect to the first axis. This can be explained by the variation in the values of the physicochemical parameters due to the operating conditions of each experiment. The iodine index is well correlated with the axis1 in the positive direction finding in the same circle as the individual HNNE (crude oil). This result shows that this oil is less saturated than the other individuals. This consequence is caused by the reaction of formic acid on the double bonds of triglycerides or free fatty acids to form epoxy. Thus, the water generated during the reaction as shown in **Figure 1** resulted in a moisture content that lies in the negative direction of the axis1 finding the individuals that underwent the epoxidation process. The individual Eexp3 is correlated on axis1 and axis2 in the negative and positive directions respectively. Its red color share and moisture content are significant compared to the other samples. In addition, individuals 2 and 4 have high clarity close to crude oil with a high clarity Eexp4.

### *4.2. Contributions of the variables*

**Figure 7** shows the percentage contributions of the variables at the axis level. Axis 1 represents (a) and axis 2 represents (b).

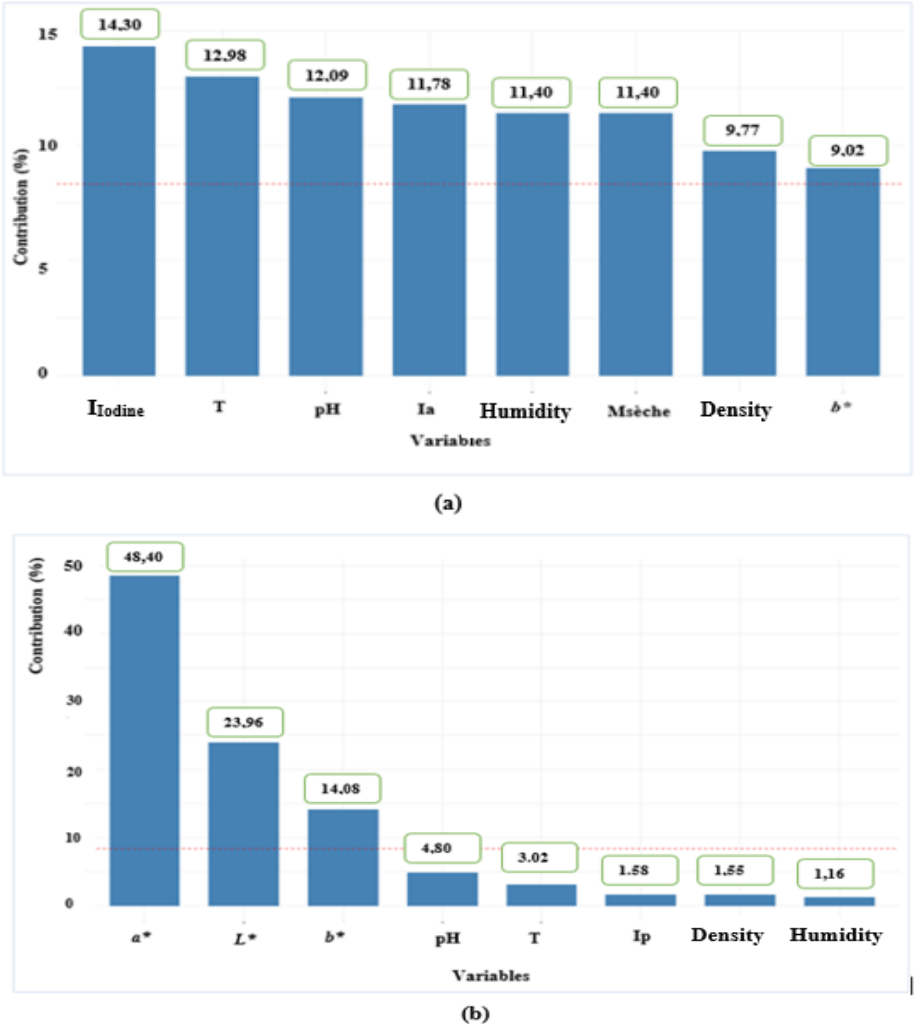
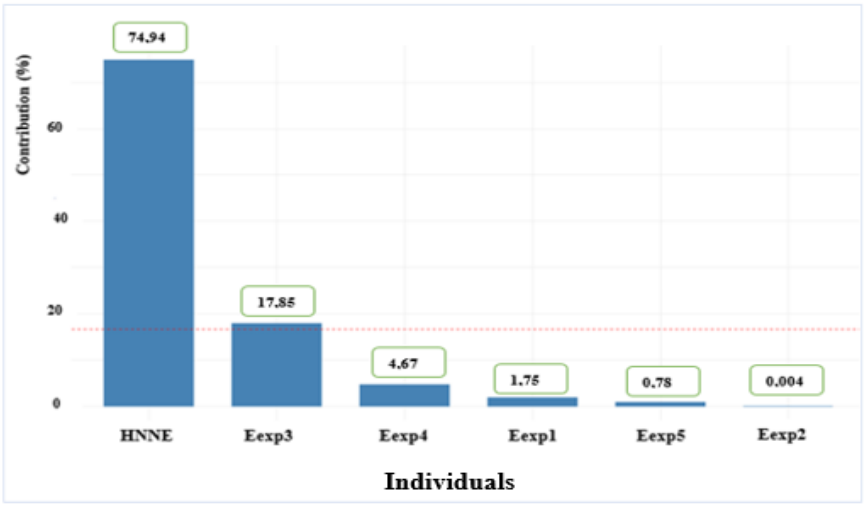



**Figure 7**. *Percentage contribution of variables on axis 1 (a) and 2 (b)*

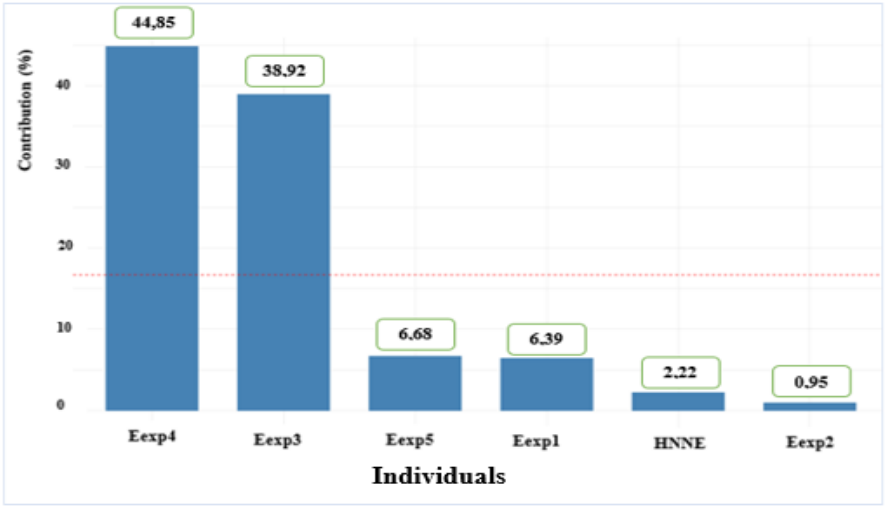
We notice that 8/8 of the variables in *(a)* have contributed well on axis1, i.e. 100% contribution. On axis 2 of *(b)*, only 3/8 of the variables (a*, L*, b*) contributed favourably, i.e. a rate of 37.5%. The figure shows that only the variable b* representing the shade of colour between yellow and blue contributes on both axis.

### *4.3. Contributions of individuals*

**Figure 8** represents the percentage contributions of individuals on the axis. Axis 1 represents (c) and axis 2 represents (d).




(c)




(d)

**Figure 8**. *Percentage contribution of individuals on axis 1 (c) and 2 (d)*

It can be seen that 2/6 of the individuals (Eexp3, HNNE), i.e. 33.33%, contribute better to axis 1. On the other hand, axis 2 is favoured by the contribution of 2/6 of the individuals (Eexp4; Eexp3) equivalent to 33.33%. In addition, we see that only the individual Eexp3 representing the shade of colour between red and green contributes to both axes at the same time. The last two **Figures** show that axis 1 is favoured by individuals HNNE and Eexp3 in terms of contribution. The same is true for the variables Iiodine, T, pH, Ia, humidity, Msèche, density and *b**. However, axis 2 is dominated by individuals Eexp4 and Eexp3. As well as the variables a*, L*, b*.

## Conclusion

In this work we have comparatively studied the physicochemical characterization of crude and epoxidized neem oil. A study that has not been published in the literature has revealed the state of the oil after the epoxidation process. The parameters measured at the end of the reaction varied to a greater or lesser extent. At the end of these analyses, the presence of secondary oxidation product of the epoxidised oil was noted, which is due to the epoxy groups with a rate of 75.5%. A decrease in

unsaturation was observed, causing a drop in the iodine value from 063.50 g $I_2$/100g (crude oil) to 013.2 g $I_2$/100g (epoxidised oil) corresponding to a molar ratio of $CH_3COOH/H_2O_2/C=C$ : 1/1/1. Furthermore, the study showed the presence of low concentrations of mineral elements in the oil and practically no variation before and after the reaction. In addition, the temperature remains the factor responsible for the decrease in the clarity of the epoxidised oil. Statistical analysis by means of PCA was able to process the large amount of information collected. This resulted in a good correlation of the majority of the variables in relation to axis 1. But also a dispatching of the individuals and variables is observed along all the directions of the mapping showing the relationship of each individual to the variables. The oil, apart from its physicochemical modification, keeps its qualities. Thus, its oxidation state remains within the range recommended by the International Olive Council (IOC) and the Codex Alimentarius. In the dynamics of this study, it would be more tangible to push the analyses by carrying out the infra-red to transform of fourrier and possibly the nuclear magnetic resonance with the aim of visualizing the functions and chemical bonds.

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## References

Adamou, Issa et al. 2020. "Effet Des Huiles de Pourghère (*Jatropha Curcas L.*) et de Neem (*Azadirachta Indica*) Sur Le Développement in Vitro Des Champignons Pathogènes Aspergillus Flavus, Aspergillus Niger, Curvularia Lunata, Fusarium Oxysporum et Rhizopus Oryzae." *International Journal of Biological and Chemical Sciences* 14(4): 1282–93.

BALDE, Ibrahima et al. 2022. "Vegetable Oils Epoxidation Mechanisms." *World Journal of Analytical Chemistry, Vol. 7, 2022, Pages 1-6* 7(1): 1–6. http://pubs.sciepub.com/wjac/7/1/1/index.html

Bertho, Julie. 2011. "Vieillissement Hygrothermique d'un Assemblage Acier Galvanisé/Adhésif Époxy: Évolution de La Tenue Mécanique En Fonction de l'état Physico-Chimique de l'adhésif." : 192.

Coud, Vaibhav V., Narayan C. Pradhan, and Anand V. Patwardhan. 2006. "Epoxidation of Karanja (Pongamia Glabra) Oil by H2O2." *JAOCS, Journal of the American Oil Chemists' Society* 83(7), 635–40.

Des, Etude et al. (2004). "Etude Des Caracteristiques Physico-Chimiques Des Huiles de Quelques Varietes Populations d'olive de La Region de Bejaïa." : 86–93.

Goud, Vaibhav V., Anand V. Patwardhan, Srikanta Dinda, and Narayan C. Pradhan. (2007). "Kinetics of Epoxidation of Jatropha Oil with Peroxyacetic and Peroxyformic Acid Catalysed by Acidic Ion Exchange Resin." *Chemical Engineering Science* 62(15), 4065–76.

ICDES. 2010. "Les Biocarburants En Afrique." *Institut de coopération économique et social*, 1–25.

Jack R. I. 2016. "Comparative Studies of Oil-Modified Alkyd Resins Synthesized from Epoxidized and Crude Neem Oil." *American Journal of Applied Chemistry* 4(4), 120.

Jijakli, H *et al.* (2009). "Etude Synergique Du Couplage Du Système Lactoperoxydase Avec d ' Autres Molécules Naturelles Actives Ayant Des Propriétés Antifongiques Pour l ' Amélioration de La Conservation En Frais Des Bananes Remerciements."

Louis, Universite, and Pasteur D E Strasbourg. (2006). "Vincent GEMBUS Organique ; Nouvelles Voies d ' Accès Au Tocophérol et." : 1–478.

Malik, Lateef Ahmad, Arshid Bashir, Aaliya Qureashi, and Altaf Hussain Pandith. (2019). "Detection and Removal of Heavy Metal Ions: A Review." *Environmental Chemistry Letters* 17(4), 1495–1521. https://doi.org/10.1007/s10311-019-00891-z.

Maria Suzana Silva et al. (2015). "Oil Epoxidation Approach for Passiflora Edulis Sims F . Flavicarpa Degener." *Journal of Latest Research in Science and Technology* 4(3), 46–52.

Milchert, Eugeniusz, Kornelia Malarczyk-Matusiak, and Marlena Musik. (2016). "Technological Aspects of Vegetable Oils Epoxidation in the Presence of Ion Exchange Resins: A Review." *Polish Journal of Chemical Technology* 18(3), 128–33.

Monge, Gaspard, and L Oxydation. "L ' OXYDATION DES CORPS GRAS Fiche Info :"

Palmer, Peter T. 2011. "Energy-Dispersive X-Ray Fluorescence Spectrometry: A Long Overdue Addition to the Chemistry Curriculum." *Journal of Chemical Education* 88(7), 868–72.

Pratt D.A., Tallman K.A., Porter N.A. (2011) Free radical oxidation of polyunsaturated lipids: New mechanistic insights and the development of peroxyl radical clocks. *Acc. Chem. Res.* 44(6), 458-67. doi: 10.1021/ar200024c.

Rahmah, M., N. Mohd Nurazzi, Farah Nordyana A. R., and Syed Anas S. M. (2017). "Effect of Epoxidised Soybean Oil Loading as Plasticiser on Physical, Mechanical and Thermal Properties of Polyvinylchloride." *IOP Conference Series: Materials Science and Engineering* 223(1).

Robinson, A. M., S. G. Harroun, J. Bergman, and C. L. Brosseau. 2012. "Portable Electrochemical Surface-Enhanced Raman Spectroscopy System for Routine Spectroelectrochemical Analysis." *Analytical Chemistry*, 84(3), 1760–64.

Samba, Balde et al. (2022). "Impact of Extraction on Biochemical Properties and Antioxidant Potential of <I>Momordica Charantia</I> L. Seeds' Oil." *Food and Nutrition Sciences* 13(02), 147–64.

Sane, Banna, Djibril Badiane, Momar Talla, and Ousmane Faye. 2018. "Évaluation de l ' Efficacité Biologique d ' Extrait de Neem ( Azadirachta Indica Juss .) Comme Alternatif Aux Pyréthrinoïdes Pour Le Contrôle Des Principaux Ravageurs Du Cotonnier ( Gossypium Hirsutum L .) Au Sénégal Evaluation of the Biological Efficienc." 12(February), 157–67.

Vianello, Chiara et al. (2018). "Study of Soybean Oil Epoxidation: Effects of Sulfuric Acid and the Mixing Program." *Industrial and Engineering Chemistry Research* 57(34), 11517–25.

Zheng, Jun Liu. 2016. "Développement d'un Procédé Pour l'époxydation et La Carbonatation Des Huiles Végétales : Application à l'huile de Coton." : 128.

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