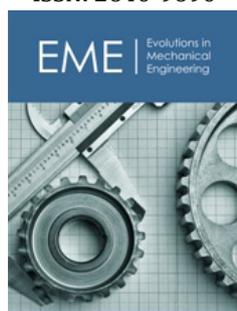


NMR Study of Cumbaru Oil to Apply as Mechanical Engineering Lubricant

Bathista ALBS*

Department of Education, Várzea Grande, Brazil

ISSN: 2640-9690



***Corresponding author:** Bathista ALBS,
Department of Education, Várzea Grande,
Brazil

Submission:  June 24, 2019

Published:  August 30, 2019

Volume 2 - Issue 5

How to cite this article: Bathista ALBS. NMR Study of Cumbaru Oil to Apply as Mechanical Engineering Lubricant. *Evolutions Mech Eng.*2(5). EME.000550.2019.
DOI: [10.31031/EME.2019.02.000550](https://doi.org/10.31031/EME.2019.02.000550)

Copyright@ Bathista ALBS, This article is distributed under the terms of the Creative Commons Attribution 4.0 International License, which permits unrestricted use and redistribution provided that the original author and source are credited.

Abstract

According to the great importance that seed oils has in relation to human health, cumbaru seed oil (DIPTERYX ALATA VOGEL) had its chemical structure and positional distribution of glycerol chains investigated by solution ^1H and ^{13}C NMR, as well as the natural degradation has been evaluated. The NMR techniques applied to the study of this oil showed that it is mainly constituted by oleic and linoleic glycerols with low content of saturated glycerols such as palmitic. Another important point was that this oil did not degraded or oxidated by itself or by light action quickly, it was stored at normal laboratory conditions during two years, and it was analyzed after that time and both ^1H and ^{13}C solution NMR spectra presented the same signals since this oil was extracted from the seed.

Keywords: Cumbaru oil; ^{13}C NMR; Chemical characterization

Introduction

Seeds are a great source of fiber, lignans, protein, fat, vitamins, minerals and oils. An extensive range of seed oil has high quality for nutritional uses, focusing on bulk nutritional oils. Seeds are also one of the best sources for the essential fatty acid alpha linolenic acid. Alpha linolenic acid is so important for bodybuilders because it “enhances insulin sensitivity within muscle cells.” This omega-3 fatty acid also has many other beneficial effects. Fruit seed from which oil is extracted, such as cumbaru have high oil content (30-40%). When the whole seeds are eaten, it is suspected that the oil portion is very slowly released and metabolized, preserving and enhancing both stable energy levels and favorable blood fat chemistry. The oils in oily seeds are an excellent energy source and the seed oil is also a rich source of linolenic acid [1-3].

Seed oils derived from tropical fruit can have a high commercial value, because of diversity uses such as pharmacological, cosmetics and food industries, which depends of its chemical composition. The content of linoleic and/or linolenic acids may promote aggregate values to those oils. According to the composition of seed oils they can be used in a wide variety of applications, from analgesic to digestive. The cumbaru fruit comes from Fabaceae family, its seed presents a high nutritional value with ~26% of proteins and can be eating crude or toasted [4,5]. The chemical components and properties of this fruit have a great importance in Brazil, especially in Mato Grosso State. Because of this different researcher have been developed and several studies to improve its production and application are in progress. The nutritional properties of the virgin oil have a good quality and it is frequently used for the people as tobacco aromatizing and also as anti-rheumatic [6]. According to those people speculations, the objective of this work is to study the chemical composition of cumbaru seed oil. For that reason, we have chosen nuclear magnetic resonance (NMR) employing ^1H and ^{13}C nuclei, as they are one of the best probes to generate response on this type of information [5-11]. Another purpose for choose NMR is based on the fact that no previous sample treatment like separation, esterification and other need to be done.

Experimental

NMR Spectroscopy

Cumbaru seed oil was extracted with acetone; this oil is yellow and has a sweet smell. The analyzes of ^{13}C NMR solution were recorded on VARIAN MERCURY 300 spectrometer, operating at 300MHz for ^1H and 75.4MHz for ^{13}C . The samples concentration for ^{13}C analyses

was about 20% of oil in 3.5ml of chloroform-d using a 10mm NMR tube at temperature of 26C, in the broadband proton decoupling mode. The quantitative ^{13}C NMR spectrum was acquired using 16K data point, spectral width 220ppm, acquisition time 1.8s, relaxation delay 6s, pulse width 90, no NOE effect and 25000 scans. High-resolution carbonyl spectrum was recorded with a 32K data point, spectral width 10ppm, acquisition time 21s, relaxation delay 5s and a 90 pulse [3]. For the ^1H NMR spectrum the sample concentration was about 2% of oil in 0.7ml of CDCl_3 using a 5mm NMR tube at 26C. The ^1H NMR spectrum was acquired using 16K data point, spectral width 12ppm, acquisition time 3.3s, relaxation delay 1s, pulse width 45 and 256 scans.

Result and Discussion

The assignments of partial ^1H spectrum of the cumbaru seed oil acquired after solvent extraction of the cumbaru fruit seed are listed in Table 1. From the assignments it can be seen that the oil is mainly constituted by triacylglycerols, with long aliphatic chains (Table 1). The same NMR study was carried out after it has been stored for six months, one year and two years. The results of both hydrogen and ^{13}C assignments were the same as it was done immediately the oil has been extracted from the seed. No difference on chemical shift assignments for the proton spectra after two years stored at room temperature was found, indicating that the sample did not change by itself along this time. The ^1H NMR spectrum of the cumbaru oil showed oxidation stability and don't have demonstrated the presence of minor components (Figure 1) (Table 2).

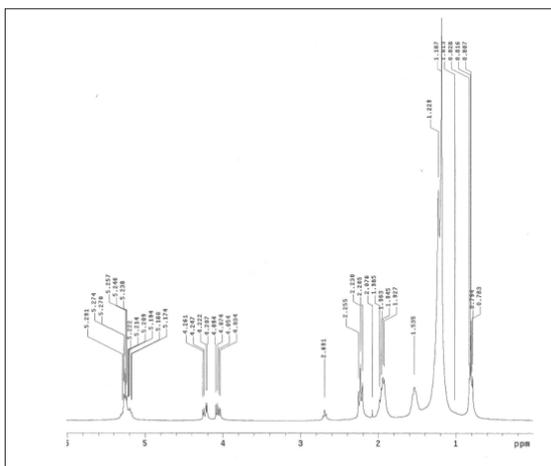


Figure 1: ^1H NMR spectrum of the cumbaru oil after two years of being extracted.

Table 1: Chemical shifts and hydrogen assignments of cumbaru oil, after solvent extraction.

(PPM)	Proton Assignments
0.8	$\text{CH}_3\text{CH}_2\text{CH}=\text{CH}$
1.20-1.24	$\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$
1.5	$(\text{CH}_2)_n$
1.9	$\text{CH}_2\text{CH}_2\text{COOH}$
2	CH
2.2	CH_2-COOH

2.7	CH_2
4.0-4.2	CH_2-OCOR
5.2	CH-OCOR
5.2	CH=CH

Table 2: Chemical shifts and hydrogen assignments cumbaru oil, after two years stored.

(PPM)	Proton Assignments
0.8	$\text{CH}_3\text{CH}_2\text{CH}=\text{CH}$
1.20-1.24	$\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$
1.5	$(\text{CH}_2)_n$
1.9	$\text{CH}_2\text{CH}_2\text{COOH}$
2	CH
2.2	CH_2-COOH
2.7	CH_2
4.0-4.2	CH_2-OCOR
5.2	CH-OCOR
5.3	CH=CH

Analyzing the ^{13}C NMR spectrum obtained at the same situations described before, we could have more information about the cumbaru oil. Figure 2 exhibits the ^{13}C NMR spectra of cumbaru seed oil after it has been extracted from the seed and the ^{13}C assignments for the oil is showed in Table 3. The same ^{13}C chemical shift values were determined for the cumbaru oil stored during two years after been extracted by solvent. Analyzing all ^{13}C spectra two values of methyl group was found one located at 13.7ppm and the other one at 13.8ppm. Two signals at 22.3 and 22.4ppm related to the methylene comes from long aliphatic chains were also detected. Those signals show that at least two distinct aliphatic chains, with different size, were observed from the NMR in this oil.

Table 3: Chemical shifts and ^{13}C assignments, for the cumbaru oil after solvent extraction

(PPM)	Carbon Assignments
172.7-172.3	C=O
129.7-129.3	CH=CH
127.7-127.5	CH=CH
68.6	CH-OH
61.7	CH_2-OH
33.8-33.6	CH_2
31.6-31.2	CH_2
30.4	CH
29.5-22.3	CH_2

In the Figure 2 a signal located at 30.6ppm was investigated by a technique named attached proton test (APT), which separates the different types of carbons. The methyl (CH_3) and CH were detected inverted and methylene carbons and no protonated carbons were normally detected. The signal at 30.0ppm was assigned as CH,

which is a carbon that probably presents a branch (Figure 3). This signal was also observed in the Figure 2 showing that the oil did not show any evidence of degradation or un-stability due to oxidation process. From the ^{13}C high-resolution carbonyl spectrum the positional distribution of the glycerols along the chains was determined. The relation of the integration area of the carbonyls located at 172.8ppm represents the substitution in sn-1,3 position and the carbonyls located at 172.4ppm shows the substitution in sn-2 position.

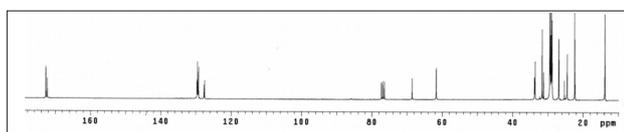


Figure 2: ^{13}C NMR spectrum of cumbaru seed oil after it has been extracted from the seed.

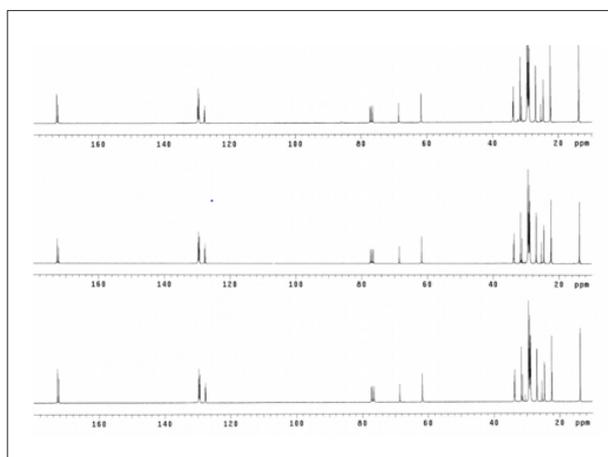


Figure 3: Comparison of ^{13}C NMR spectrum of the cumbaru oil:

1. After six month
2. Two years and
3. Immediately extracted.

Conclusion

According to the main purpose of this work, the NMR techniques were applied to the oil and the data obtained showed the main chemical compounds and the positional distribution on triacylglycerol chains. The most important conclusion was that this oil did not degraded by itself or by lights during the time studied, which is very good, for example, food and lubricant applications.

Acknowledgement

The author would like to thank you CNPq, CAPES and IFMT.

References

1. Moreto E, Fett R (1989) Óleos e gorduras vegetais, Florianópolis, UFSC, Brazil.
2. Gunstone FD (1993) Advances in Lipid Methodology. In: Christie WW (Ed.), Oily Press, Dundee, Scotland, UK, p.1.
3. Sacchi R, Patumi A, Fontanazza G, Barone P, Fiodiponti P, et al. (1996) A high field ^1H nuclear magnetic resonance study of the minor components in virgin olive oils. *J Am Oil Chem Soc* 73(6): 747-758.
4. Bellinelo LC (1996) *Biologia Vegetal*. Coleção Objetivo, São Paulo, Brazil, V. 4.
5. Ferreira RA, Soraya AB, Antonio CD, Marlene MM (1998) Morphological characterization of fruit, seed, seedling and mud DE dipteryx alata vogel-baru (*leguminosae papilionoideae*). *Cerne* 4(1): 073-087.
6. Sacchi R (2000) Applications of Magnetic Resonance in Food Science. Annals of the fifth international conference, Aveiro, Portugal, p. 120.
7. Sacchi R, Addeo F, Guidicianoi I, Paolillo L (1992) *Ital J Food Sci* 4: 117.
8. Wollenberg KF (1990) Quantitative high resolution ^{13}C nuclear magnetic resonance of the olefinic and carbonyl carbons of edible vegetable oils. *J Am Oil Chem Soc* 67(8): 487-494.
9. Sacchi R, Addeo F, Paolillo L (1997) ^1H and ^{13}C NMR of virgin olive oil. An overview. *Magnetic Resonance in Chemistry* 35(13): S133-S145.
10. Silva EO, Bathista ALBS, Tavares MIB, Filho NP, Nogueira JS (2005) ^{13}C NMR study of peach oil. *Journal of Food and Agriculture* 85(13): 2269-2272.
11. Nascimento AM, Tavares MIB (2007) Solid state NMR study of *couma utilis* seeds. *Int J of Polym Mat* 56(4): 115-125.

For possible submissions Click below:

Submit Article