# Evaluation of the Properties of *Calotropis procera* Oil Aiming the Production of Biodiesel

**Lindeberg Ventura de Sousa\*, Adriana Paula Batista Santos, Luiz Di Souza, Anne Gabriella Dias Santos, Adilson Beatriz**

## Material and Methods

The seeds were collected, dehydrated (at 70ºC), selected and dried in an oven. The extraction of the oil was done with the Soxhlet method using hexane as the solvent. The yield was calculated using equation 1:

$$\text{Oil\%} = \text{OilM/SeedM} * 100 \text{ (Eq. 1)}$$

Where: **OilM** is the oil mass obtained and **SeedM** the seed mass used in the extraction. The ideal extraction time was determined experimentally.

The physicochemical properties determined in the oil, according to standard procedures [1 and 2], were acidity, free fatty acids, iodine, saponification, peroxide (determined volumetrically), and refractive indices (via a Quimis Q767D192 portable digital refraction meter). The content of water and sediments was obtained through the appropriate standard [3]. The kinematic viscosity was obtained through a TIC-17 RGT Saybolt Universal Viscometer according to the manufacturer's recommendations. The density was determined using a Pyrex glass pycnometer [4].

The thermal analyses were obtained in the temperature range of 30-800 ºC using an STA 449 F3-Jupiter Thermal Analyzer. The NMR data were obtained through a Bruker Avance DPX-300 device using frequencies of 300 MHz for $^1$H and 75 MHz for $^{13}$C. The internal reference was adjusted from the TMS signal and the solvent's residual hydrogen (CHCl$_3$) signals. The CDCl$_3$ volume is of about 0.7 mL, and the samples amount to about 15 mg.

The centesimal composition of polyunsaturated and monounsaturated fatty acids was determined by equations 2 and 3, respectively:

$$\text{Polyunsaturated\%} = \text{IAHI/6X100 (Eq. 2)}$$

$$\text{Monounsaturated\%} = \text{AHI} - \text{2(IHAI)/12X100 (Eq. 3)}$$

Where: **IAHI** = internal allylic hydrogens integration; **AHI** = allylic hydrogens integration. The centesimal composition of total unsaturated fatty acids (total unsaturated%) present in the oil can be determined by the simple sum of Polyunsaturated% and Monounsaturated%. The centesimal composition of saturated fatty acids is determined by equation 4:

$$\text{Saturated fatty acids \%} = 100 - \text{(Total Unsaturated\%). (Eq. 4).}$$

## References

[1]     American Oil Chemists Society. Official and Tentative Methods (AOCS). 3 Ed. Chicago: v.1, 1985.

[2]     Anvisa. Regulamento técnico para fixação de identidade e qualidade de óleos e gorduras vegetais. 1999. Available from: http://portal.anvisa.gov.br. Access July, 2014.

[3]     ANP – Agência Nacional do Petróleo, Gás Natural e Biocombustível. Resolução n.69 de dezembro de **2014**. Available from: http://nxt.anp.gov.br/nxt/gateway.dll/leg/resolucoes_anp/2014/dezembro/ranp%2069%20-%202014.xml. Access July, 2015.

[4]     Lima, L. S.; Silva, L. F. B.; Santo Filho, D. M. E.; Santos Júnior, J. J. P. S.; Rodrigues, C. R. C.; Pereira, R. G. Abordagem metrológica no estudo da variação da massa específica de biodiesel de sebo bovino com relação a temperatura, utilizando-se um picnômetro. In: Congresso Internacional de Metrologia Mecânica, 1, 2008, Rio de Janeiro. [Link]

**Figure 1S**. Seeds of *C. procera:* green (left) and mature (right).
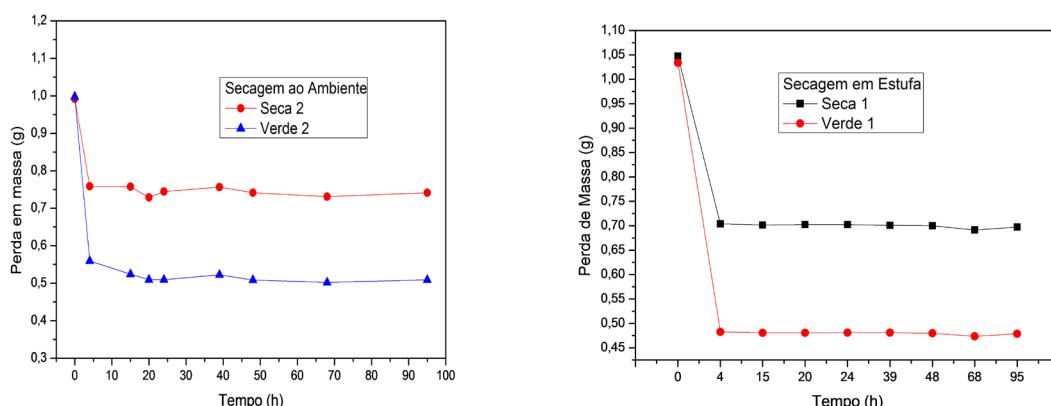


**Figure 2S**. Drying of *C. procera* seeds: in the environment (left) and in the greenhouse (right) at 70 ºC.
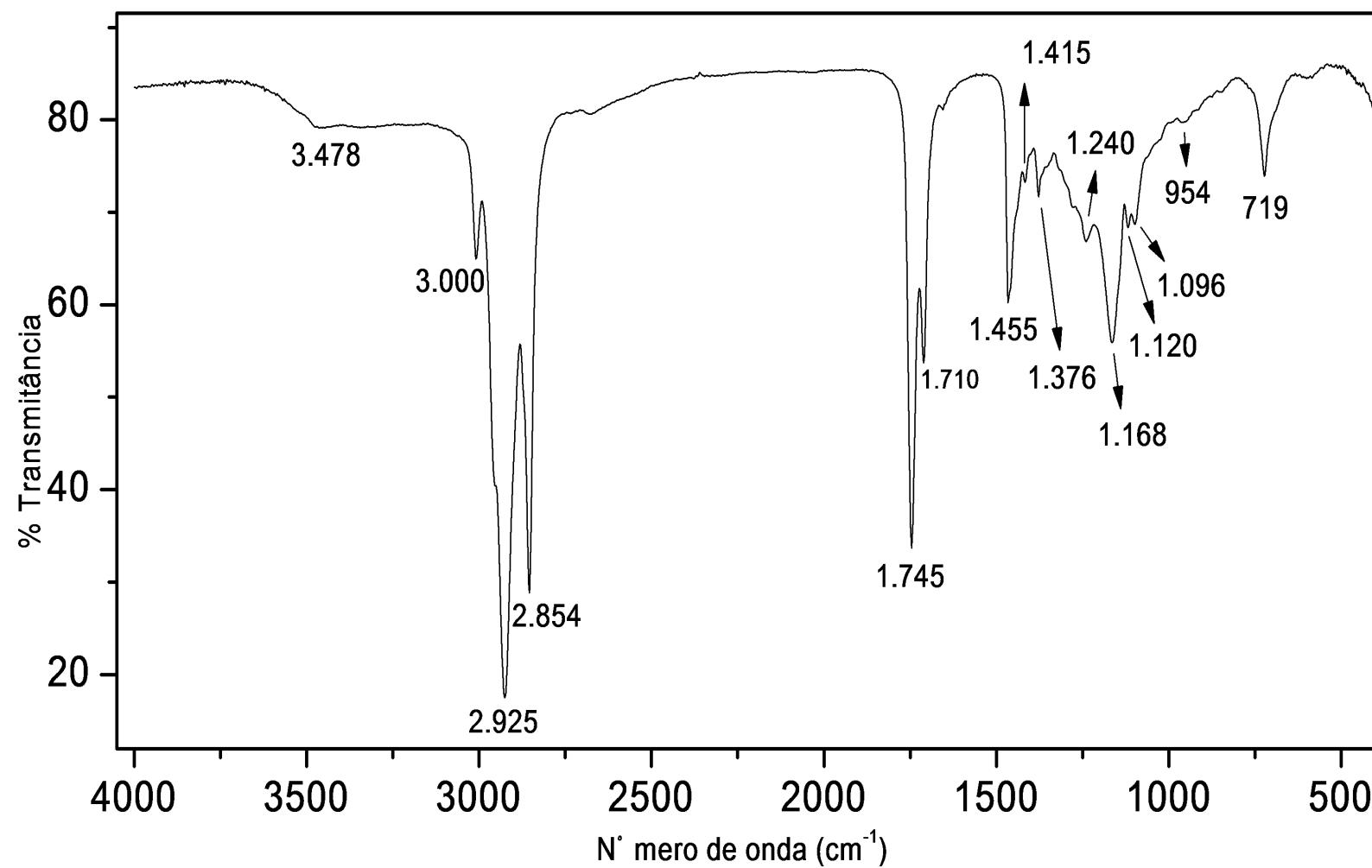
**Figure 3S**. FTIR spectrum of *C. Procera* oil.

| Acquisition Time (sec) | 6.8420 | Comment | 1H - CDCl3 AB-OCP-1 AB | | Date | 08 Jan 2015 13:45:34 |
|---|---|---|---|---|---|---|
| File Name | C:\Users\Administrador\Dropbox\Adilson\Lulu - UERN\AB-OCP-1\AB-OCP-1_001001r | | | | Frequency (MHz) | 300.13 |
| Nucleus | 1H | Number of Transients | 8 | Original Points Count 32768 | Points Count | 65536 |
| Pulse Sequence | zg | Solvent | CHLOROFORM-D | | Sweep Width (Hz) | 4789.27 |
| Temperature (degree C) | 27.000 | | | | | |
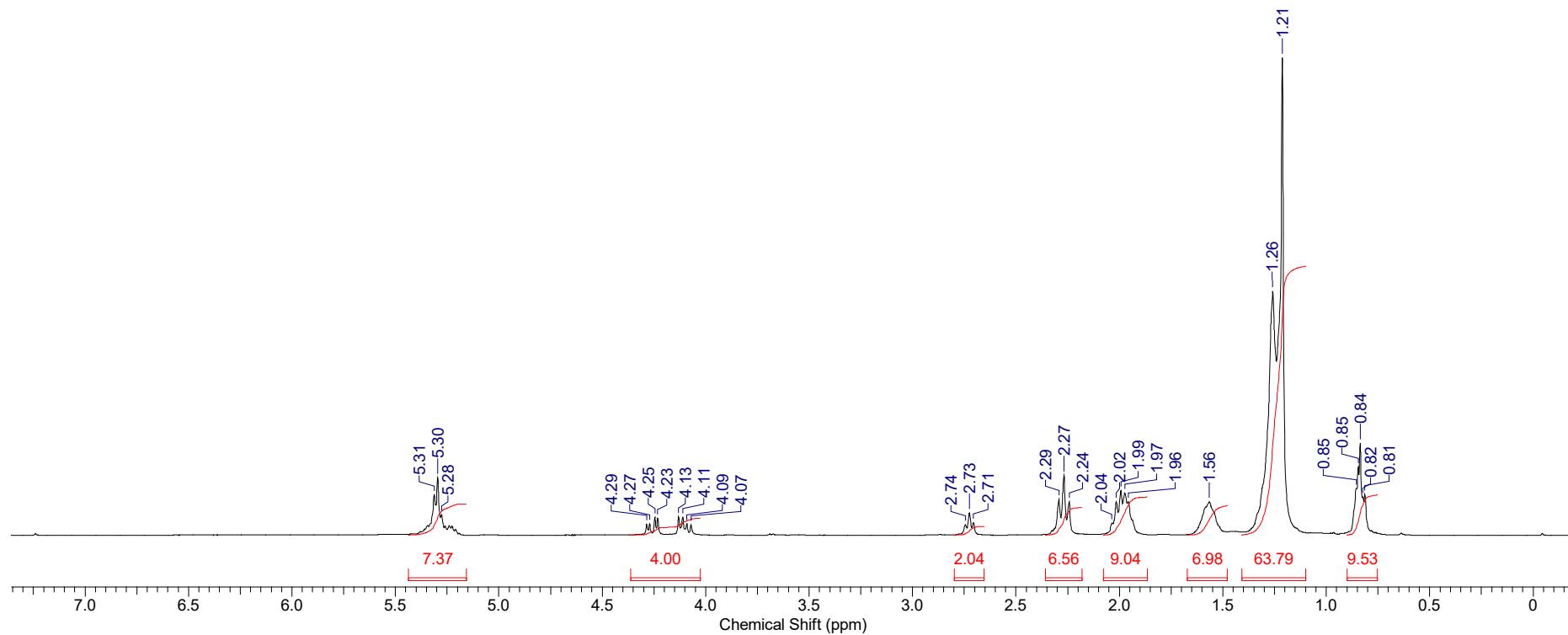


**Figure 4S**. $^1$H-NMR spectrum (300 MHz, CDCl$_3$) of *Calotropis procera* oil.

| Acquisition Time (sec) | 0.8700 | Comment | | 1H - CDCl3 AB-OCP-1 AB | | Date | 08 Jan 2015 13:53:20 |
|---|---|---|---|---|---|---|---|
| File Name | C:\Users\Adilbeat\Dropbox\Adilson\Lulu - UERN\AB-OCP-1\AB-OCP-1_003001r | | | | | Frequency (MHz) | 75.47 |
| Nucleus | 13C | Number of Transients | 371 | Original Points Count | 16384 | Points Count | 32768 |
| Pulse Sequence | zgpg30 | Solvent | | CHLOROFORM-D | | Sweep Width (Hz) | 18832.39 |
| Temperature (degree C) | 27.000 | | | | | | |
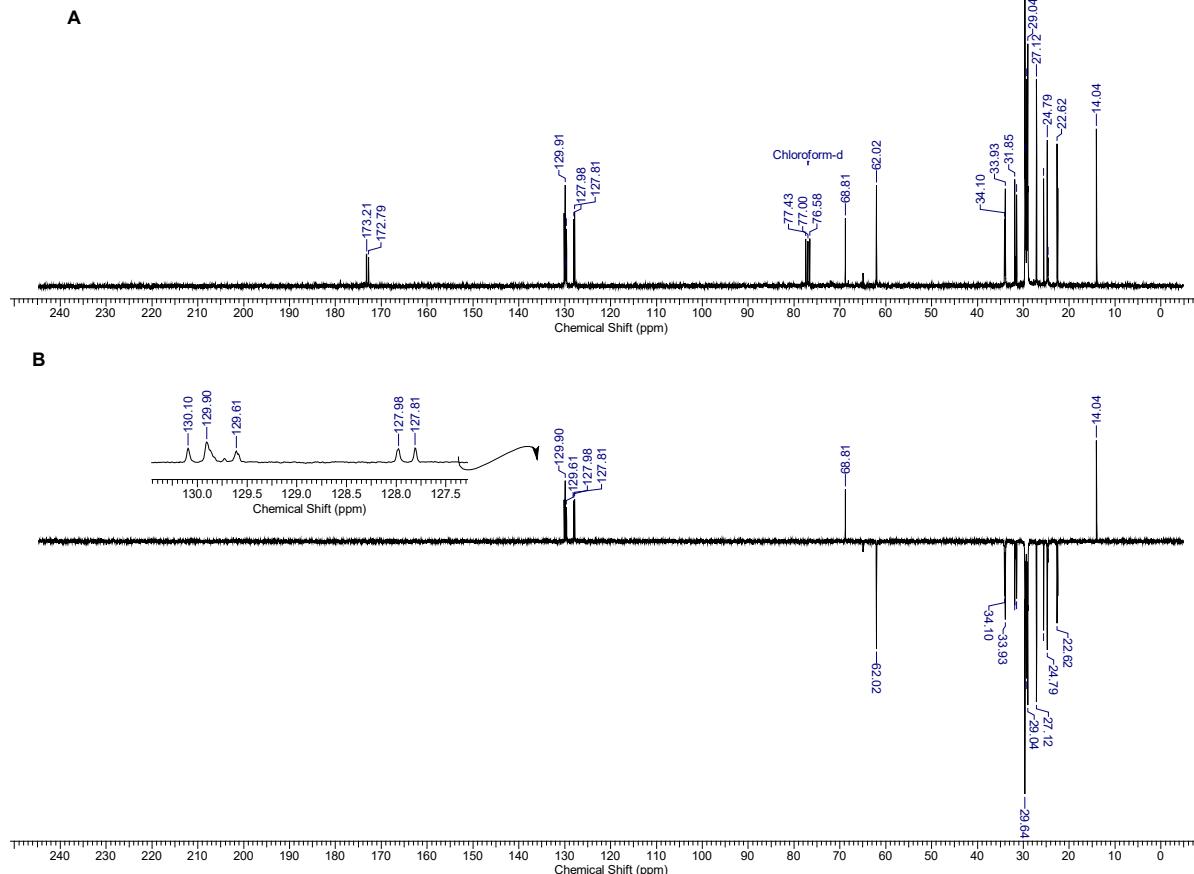


**Figure 5S**. A) $^{13}$C-NMR spectrum (75 MHz, CDCl$_3$) and B) DEPT-135 of *Calotropis procera* oil.