

Thermal Analysis of the Essential Oil of *Aniba rosaeodora* Ducke by TGA and DSC

Análise Térmica do Óleo Essencial de *Aniba rosaeodora* Ducke por TG e DSC

Análisis térmico del aceite esencial de *Aniba rosaeodora* Ducke por TG y DSC

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Rogério de Mesquita Teles

ORCID: <https://orcid.org/0000-0001-9789-1847>

Federal Institute of Education Science and Technology of Maranhão, Brazil

E-mail: rogerioteles@ifma.edu.br

Victor Elias Mouchrek Filho

ORCID: <https://orcid.org/0000-0003-2855-7292>

Federal University of Maranhão, Brazil

E-mail: victor.mouchrek@ufma.br

Abstract

Thermal analysis comprises a group of techniques to measure a physical property of a sample subjected to controlled temperature programming, including thermogravimetry (TGA), derivative thermogravimetry (DTG) and differential scanning calorimetry (DSC). The species *Aniba duckei* Kostermans, synonymous with *Aniba rosaeodora* Ducke, occurs in the Amazon and is also known as rosewood. Essential oils are complex oily aromatic liquids obtained by extraction from highly volatile aromatic plant materials as secondary metabolites. This research aimed to chemically and thermally characterize the essential oil of *Aniba duckei*. Gas chromatography was used for the quantification, by the external standard method, of the main component. The thermal study of the oil and the linalool standard was carried out using DSC and TG-DTG. The essential oil yield was 1.93% and the analysis by GC-MS confirmed the majority presence of linalool, with a concentration of 89.34% in the oil. The technical analysis revealed some similarity between the curves of pure linalool and essential oil, which is attributed to the high content of linalool in the oil. The differences between the boiling points and enthalpies of the linalool and oil pattern are justified by the presence of minor components, their concentrations in the oil and because they are substances of different polarities, molecular masses and intermolecular forces. The techniques proved to be efficient to characterize the oil. The results of the thermal analysis of the essential oil of the *Aniba rosaeodora* plant species are unprecedented, enabling the determination of linalool in by DSC.

Keywords: Essential oil; *Aniba rosaeodora* Ducke; *Aniba duckei* Kostermans; Linalool; Thermal analysis.

Resumo

A análise térmica compreende um conjunto de técnicas para medir uma propriedade física de amostra submetida a programação de temperatura controlada, incluindo termogravimetria (TGA), termogravimetria derivada (DTG) e calorimetria exploratória diferencial (DSC). A espécie *Aniba duckei* Kostermans, sinônimo de *Aniba rosaeodora* Ducke, ocorre na Amazônia e é também conhecida como pau-rosa. Os óleos essenciais são líquidos aromáticos oleosos complexos obtidos por extração de materiais vegetais aromáticos altamente voláteis, como metabólitos secundários. Esta pesquisa teve como objetivo caracterizar química e termicamente o óleo essencial de *Aniba duckei*. A cromatografia gasosa foi utilizada para identificação e quantificação do componente principal, pelo método de padrão externo. O estudo térmico do óleo e do padrão linalol foi realizado utilizando DSC e TG-DTG. O rendimento de óleo essencial foi de 1,93% e a análise por GC-MS confirmou a presença majoritária de linalol, com concentração de 89,34% no óleo. A análise técnica revelou semelhança entre as curvas de linalol puro e as do óleo essencial, o que é atribuído ao alto teor de linalol no óleo. As diferenças entre os pontos de ebulição e entalpias do padrão do linalol e do óleo são justificadas pela presença de componentes minoritários, suas concentrações no óleo e por serem substâncias de diferentes polaridades, massas moleculares e forças intermoleculares. As técnicas se mostraram eficientes para caracterizar o óleo. Os resultados da análise térmica do óleo essencial da espécie vegetal *Aniba rosaeodora* são inéditos, possibilitando a determinação do linalol por DSC.

Palavras-chave: Óleo essencial; *Aniba rosaeodora* Ducke; *Aniba duckei* Kostermans; Linalol; Análise térmica.

Resumen

El análisis térmico comprende un grupo de técnicas para medir una propiedad física de una muestra sujeta a programación de temperatura controlada, que incluye termogravimetría (TGA), termogravimetría derivada (DTG) y calorimetría diferencial de barrido (DSC). La especie *Aniba duckei* Kostermans, sinónimo de *Aniba rosaeodora* Ducke, se encuentra en el Amazonas y también se conoce como palo de rosa. Los aceites esenciales son líquidos aromáticos aceitosos complejos obtenidos por extracción de materiales vegetales aromáticos altamente volátiles como metabolitos secundarios. Esta investigación tuvo como objetivo caracterizar química y térmicamente el aceite esencial

de *Aniba duckei*. Se utilizó cromatografía de gases para la cuantificación, por el método del patrón externo, del componente principal. El estudio térmico del aceite y del estándar de linalol se realizó mediante DSC y TG-DTG. El rendimiento de aceite esencial fue de 1,93% y el análisis por GC-MS confirmó la presencia mayoritaria de linalol, con una concentración de 89,34% en el aceite. El análisis técnico reveló cierta similitud entre las curvas del linalol puro y el aceite esencial, lo que se atribuye al alto contenido de linalol en el aceite. Las diferencias entre los puntos de ebullición y las entalpías del patrón del linalol y del aceite se justifican por la presencia de componentes minoritarios, sus concentraciones en el aceite y por tratarse de sustancias de diferente polaridad, masa molecular y fuerzas intermoleculares. Las técnicas demostraron ser eficientes para caracterizar el aceite. Los resultados del análisis térmico del aceite esencial de la especie vegetal *Aniba rosaeodora* no tienen precedentes, permitiendo la determinación de linalool por DSC.

Palabras clave: Aceite esencial; *Aniba rosaeodora* Ducke; *Aniba duckei* Kostermans; Linalol; Análisis térmico.

1. Introduction

Essencial oils

Brazilian biodiversity is one of the richest on the planet, with a high rate of biological endemism, dispersed in unique biomes (Maia *et al.*, 2009; Barata, 2012; Joly *et al.*, 2011). Natural products are produced by plants, as the specimens produce their own food and also have the ability to deal with adverse situations. In an irregular environment, they produce certain chemicals called secondary metabolites, which are used as an attack and defense mechanism (Nazir *et al.*, 2021).

Among these secondary metabolites are essential oils, which are volatile, lipophilic, odorous and liquid products extracted from different parts of aromatic plants, which have different and complex chemical composition and guarantee adaptive advantages to the plants in the environment in which they are inserted (Oussalah *et al.*, 2007; Hasani *et al.*, 2018; Tuğçe *et al.*, 2021). Essential oils are mainly composed of monoterpenes, sesquiterpenes and phenylpropanoids, metabolites that confer their organoleptic characteristics. Essential oils from various plant species are used in perfumery, cosmetics, food and as adjuvants in medicines, being considered important in commercial and monetary aspects (Bizzo *et al.*, 2009; Kreutz *et al.*, 2021).

The Vegetal Species *Aniba rosaeodora* Ducke

The botanical species *Aniba duckei* Kostermans, synonymous with *Aniba rosaeodora* Ducke (Maia, 2000; Teles *et al.*, 2018), of the *Lauraceae* family, commonly known as rosewood, was discovered in Brazil in Juriti Velho, in the state of Pará in 1925 (Correa *et al.*, 1975; Siani *et al.*, 2000). Its tree can reach up to 30 meters in height and its trunk 2 meters in diameter. The flowers are ferruginous and the fruit is a drupe, 2 to 3 cm long (Sampaio *et al.*, 2005). The extraction of native essences such as *Aniba rosaeodora*, began to consolidate during the Second World War (Vivan *et al.*, 2011).

Aniba rosaeodora essential oil is characterized by its strong odor, colorlessness and density lower than that of water, solubility in usual organic solvents and 70° GL alcohol, being linalool (3,7-dimethyl-oct-1,6-dien -3-ol) the major constituent. Linalol has wide application in several areas, being used as a starting point in important syntheses such as that of linanyl acetate and tested as an acaricide, bactericide and fungicide, sedative and anticonvulsant. A recent application for the oil is in aromatherapy. It is a product in great demand internationally for its use as a fixative in perfumes, for example the famous Chanel number 5 (Chaar, 2000; Sudam, 1971; Teles, Mouchrek Filho *et al.*, 2017).

Thermal analysis

Thermal Analysis is a group of techniques, in which a physical property of a substance and/or its reaction products is measured, while the sample is subjected to controlled temperature programming (Mackenzie, 1993; Ionashiro & Giolito, 2004).

Thermogravimetry (TGA) and Derived Thermogravimetry (DTG)

Thermogravimetry is a technique in which the mass variation (loss or gain) that occurs in the sample is monitored as a function of time (constant temperature) or as a function of temperature. The measurement is performed using an equipment called a thermobalance, which consists of a combination of an electronic microbalance coupled to an oven and a linear temperature programmer, allowing the continuous weighing of a sample as a function of temperature, as the sample is heated or cold (Santos *et al.*, 2004; Aslan *et al.*, 2018; Mallick *et al.*, 2018).

Derived thermogravimetry (DTG) is the first derivative of the thermogravimetric curve, that is, the derivative of mass variation in relation to time or temperature. The DTG curve presents the information in a clearer way, with the area being directly proportional to the mass variation, leading to the prompt determination of the peak temperature and indicating the initial and final temperatures of the process (Gonçalves, Teixeira & Teixeira, 2003).

Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) is the technique in which the difference in energy released or absorbed by the sample is measured, in relation to a thermally inert reference material, as a function of temperature, while the sample and the reference are subjected to a temperature programming (Bernal *et al.*, 2002).

When a material undergoes a change of physical state or chemical reaction, heat is released or absorbed. During the thermal event, the DSC measures change in the thermal energy of the sample. In general, phase transition, dehydration, reduction and some decomposition reactions produce endothermic effects, while crystallization, oxidation and some decomposition reactions produce exothermic effects (Dantas, 2006).

Thermal Analysis of Essential Oils

There are vast and diverse applications of thermal analysis in industrial activities, such as chemical, pharmaceutical, cosmetics, food and petrochemicals, among others. There is also the literature on thermal analysis of essential oils, for characterization, evaluation of their thermal behavior, stability and investigation of possible adulteration (Hazra *et al.*, 2002; Hazra *et al.*, 2004; Monteiro *et al.*, 2011; Cremasco *et al.*, 2011; Siqueira *et al.*, 2016).

However, there is no research in the literature on thermal analysis of essential oil of the plant species *Aniba rosaeodora* Ducke nor of species in its family – *Lauraceae*. Thus, the present work reports the research in which the chemical characterization and thermal analysis of the essential oil of the plant species *Aniba rosaeodora* Ducke and its major component – linalool – was carried out by Thermogravimetric Analysis (TGA) and by Differential Scanning Calorimetry (DSC).

2. Methodology

This research was developed in the Laboratory of Fuels and Materials (LACOM) located at Federal University of Paraiba (UFPB) in partnership with Laboratory of Researches and Tests of Fuels (LAPEC) at Federal University of Amazonas (UFAM) and Sao Carlos Institute of Chemistry, from University of Sao Paulo (USP).

Plant sample collection, extraction and chromatographic analysis of essential oil

Samples of leaves and twigs were collected from three *Aniba rosaeodora* Ducke trees cultivated in Ducke Forest Reserve, AM –Rodovia 010, km 26, Manaus, Amazonas, Brazil - 03°00'02" and 03°08'00" south latitude and 59°58'00" west longitude).

The essential oil was extracted from 30 grams of the sample with 300 mL of distilled water, by hydrodistillation, in a Clevenger system, and the oil was dried by percolation in anhydrous Na₂SO₄. The yield was calculated in the mass/mass ratio.

GC-MS essential oil analysis was performed on a Varian chromatograph, model 3900, using helium as carrier gas with flow in the column of 1 mL min⁻¹; Injector temperature: 270°C, split 1:50; capillary column (30m x 25 mm) by stationary phase VF-1ms (100% methylsiloxane 0.25 μm) and oven temperature programmed to 60°C and then increased to 220°C at a rate of 4°C min⁻¹ and then increased again to 260°C, this time at a rate of 1°C min⁻¹, with total running time of 100 minutes. For mass spectrometer, the manifold, ion trap and transfer line temperatures were set to 50, 190 and 200°C, respectively. 1.0 μL (automatic injector CP-8410) aliquots of the samples diluted were injected in proportion of 20 μL for 1.5 mL of hexane. Linalool was quantified by the external standard method, considering its high concentration in the samples.

For identification of the compounds the spectral databases of the spectral libraries NIST105, NIST21, WILEY139 and AMSDIS (Automated Mass Spectral De-convolution Mass & Identification System) software were used, as well as references (Teles *et al.*, 2018; Namara *et al.*, 2007; Adams, 2001). For linalool, confirmation was also by addition of standard.

Thermal analysis

The calorimetric curves were obtained in a Thermal Analyzer, brand TA INSTRUMENTS, model SDT 2920 through the non-isothermal method of analysis, at a heating ratio of 10 °C min⁻¹ and temperature range of 25-350 °C, in order to verify the thermal decomposition profile. The DSC (Differential Scanning Calorimetry) curves of the essential oil of *Aniba rosaeodora* Ducke and of the linalool standard, stored in closed aluminum (Al) sample holders, with and without holes, in atmospheres of air or nitrogen gas (N₂), were obtained with a heating ratio of 10 °C min⁻¹.

3. Results

From the essential oil chromatogram, the substances listed on Table 1 were identified, which include: the peak number in the order of elution, the retention time (t_{RET}) of each substance in the column (in minutes), the name of the substance identified, the percentage of normalized area (%A) which indicates the relative distribution of the compounds in the sample.

Table 1. Compounds identified in the essential oil of *Aniba roseadora* Ducke.

Peak	t _{RET}	Compound Name	%A
1	15,61	Limonene	0,52
2	15,71	1,8-Cineol	1,07
3	17,43	cis-linalool oxide	1,94
4	18,06	trans-linalool oxide	1,86
5	18,60	Linalool	89,34
6	21,88	α-Terpineol	3,06
7	28,26	α-copaene	0,89
8	31,74	α-Patchulene	0,77
9	32,02	Cariophyllene	0,55

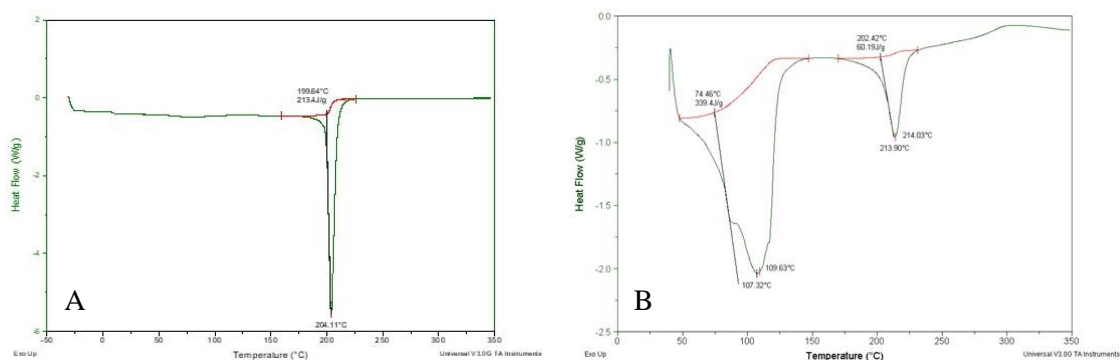
Source: Authors.

Thermal analysis

Fig. 1 shows the results of the DSC analysis for 6.50 mg of linalool standard sample (Figure 1A) and for 10.30 mg of *Aniba rosaeodora* Ducke essential oil (Figure 1B), both in an Al pan without a hole with air atmosphere.

The DSC curve for the linalool shows a single endothermic peak, with a peak temperature of 204.11°C and an enthalpy of 213.4 J g⁻¹. For the oil, there are two endothermic transitions, the first with a peak temperature of 107.32 °C and an enthalpy of 339.4 J g⁻¹, and the second with peak temperature of 213.90 °C and enthalpy of 60.19 J g⁻¹.

Figure 1. DSC curves, aluminum pan without a hole, with a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$, in air atmosphere for: A) linalool standard; and B) essential oil of *Aniba rosaedora* Ducke.

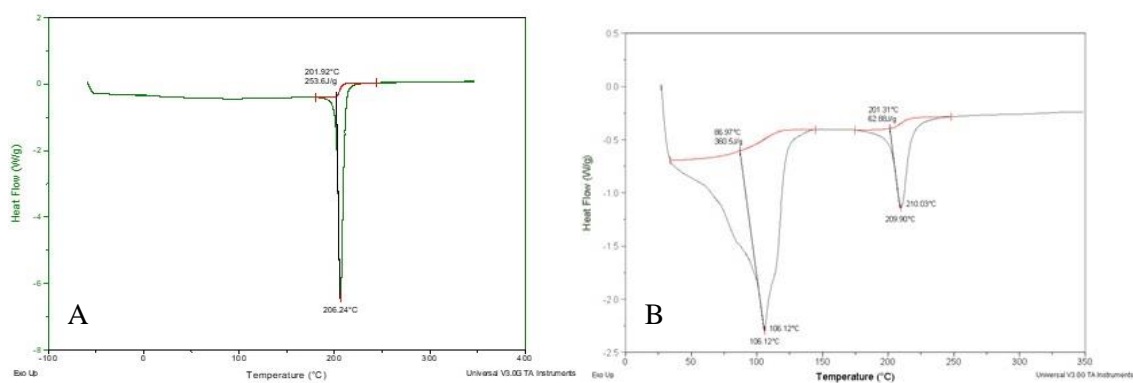


Source: Authors.

In nitrogen atmosphere, the DSC curve for 5.55 mg of the standard of linalool and the sample of 10.00 mg of essential oil of *Aniba rosaedora* resulted in graphics showed in Fig. 2, A e B, respectively.

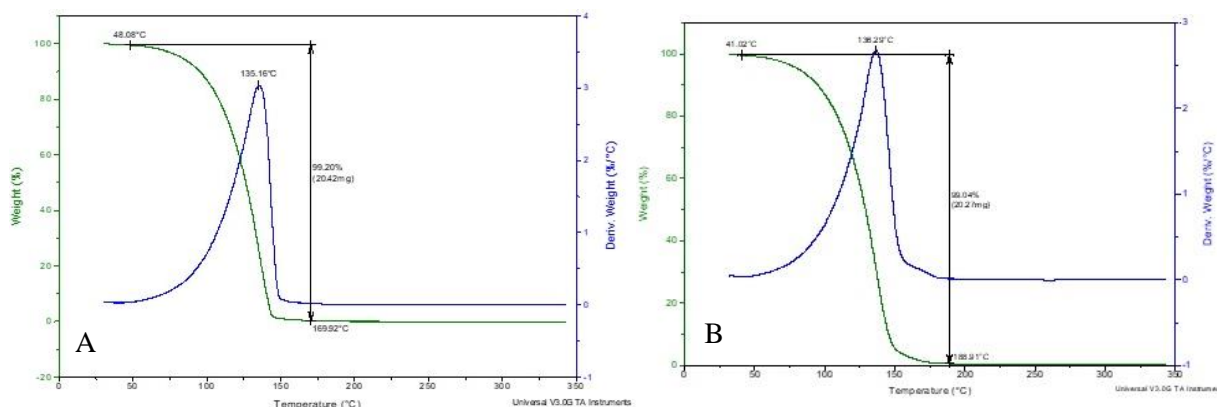
For the linalool standard, the DSC curve shows a single peak with a temperature of 206.24 °C and an enthalpy of 253.6 J g⁻¹. The sample of the essential oil of *Aniba rosaedora* Ducke showed two endothermic transitions, the first with peak temperature of 106.12 °C and enthalpy of 360.5 J g⁻¹, and the second with peak temperature of 209.90 °C and enthalpy of 62.88 J g⁻¹.

Figure 2. DSC curves, aluminum pan without a hole, with a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$, in nitrogen atmosphere for: A) linalool standard; and B) essential oil of *Aniba rosaedora* Ducke.



Source: Authors.

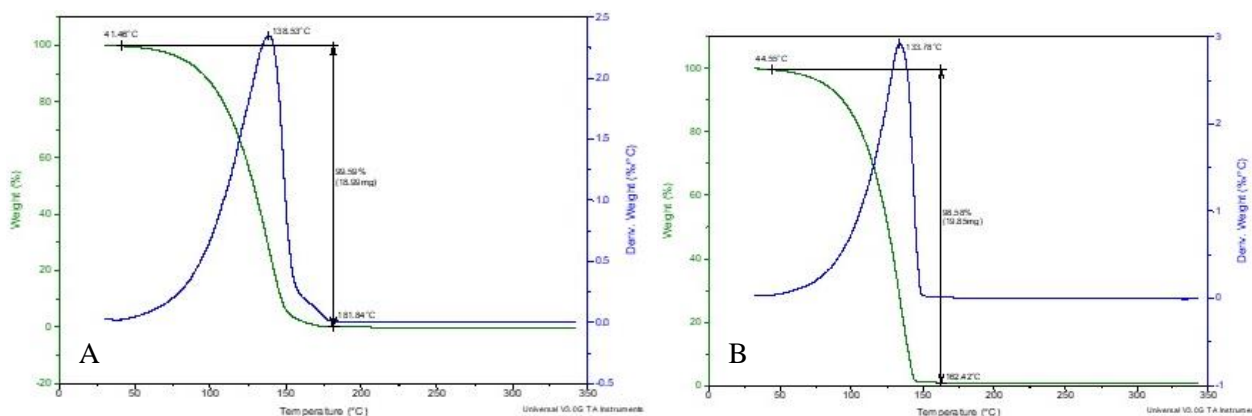
Figure 3. TG-DTG curves, aluminum pan without a hole, with a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$, in air atmosphere for: A) linalool standard; and B) essential oil of *Aniba rosaeodora* Ducke.



Source: Authors.

Figure 3 is the result of the TG-DTG analysis, in air atmosphere, for 20.58 mg of the standard (Figure 3A) and for 20.46 mg of essential oil of *Aniba rosaeodora* (Figure 3B). The TG-DTG curve of the Fig. 3A shows a single decomposition step observed between $48.08\text{ }^{\circ}\text{C}$ and $169.92\text{ }^{\circ}\text{C}$ with a mass loss of 99.20% as a consequence of the linalool volatilization process. For the essential oil (Fig. 3B) resulted else in a single endothermic transition between $41.02\text{ }^{\circ}\text{C}$ and $188.91\text{ }^{\circ}\text{C}$, with mass loss of 99.04%.

Figure 4. TG-DTG curves, aluminum pan without hole, with a heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$, in nitrogen atmosphere for: A) linalool; and B) essential oil of *Aniba rosaeodora* Ducke.

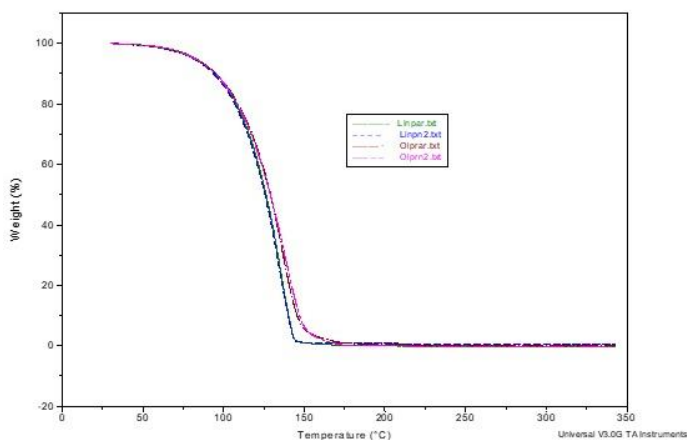


Source: Authors.

The TG-DTG curves shown on Figure 4 were obtained for 20.58 mg of the linalool standard (Figure 4A) and for 19.07 mg of the essential oil (Figure 4A), both in nitrogen atmosphere. It is possible to observe that the linalool volatilization is verified between $44.55\text{ }^{\circ}\text{C}$ and $162.42\text{ }^{\circ}\text{C}$ with a mass loss of 98.58% and the oil volatilization occurs between $41.46\text{ }^{\circ}\text{C}$ and $181.84\text{ }^{\circ}\text{C}$, with a mass loss of 99.5%.

The thermogravimetric curves (TG) of the essential oil of the plant species *Aniba rosaeodora* Ducke and the linalool standad, in different atmospheres, being: Linpar = linalool pattern in air atmosphere; Linpn2 = linalool standard in N_2 atmosphere; Olpar = essential oil in air atmosphere; and Olpn2 = essential oil in N_2 atmosphere.

Figure 5. Thermogravimetric curves (TG) of the essential oil of the plant species *Aniba rosaeodora* Ducke and the linalool standard.



Source: Authors.

4. Discussion

Comparing the values for the essential oil of the branches of the *Aniba rosaeodora* species with those in the literature, it is observed that there is a strong similarity. The substances identified from the chromatogram of the essential oil of *Aniba rosaeodora* Ducke showed that the major component is linalool, with 89.34%, which is also in agreement with the literature.

The DSC curves for the determination of standard linalool boiling temperatures in air and nitrogen show little variation in the peak temperatures and vaporization enthalpies of linalool. Lower values in an oxidizing atmosphere occur through the reaction of alcohol, forming less polar substances. The difference in peak temperatures and vaporization enthalpies verified in the DSC curves of the air atmosphere in essential oil samples and nitrogen gas is due to the presence of minor components in the oil, whereas the fact to this variation be small is justified by the high content of linalool in the essential oil.

The DSC curves obtained from the linalool standard demonstrated the decomposition of linalool and confirmed the absence of water of hydration. The DSC curves obtained with the essential oil, on the other hand, show an endothermic transition around 100 °C, which may show that the essential oil has water of hydration.

The TG-DTG curves in air (Fig. 3 and Fig. 4) showed a single decomposition step for both standard and essential oil, resulting from the volatilization process of the linalool or the oil. This analysis also showed that they both lost more than 99% of their mass. The oil starts to lose mass at a temperature lower than the standard temperature and ends up at a slightly higher temperature, which can be explained by the presence of minor components in the oil, as some are more volatile than linalool and others are less.

The difference between the temperature values of the essential oil and the standard is explained by the fact that linalool is the major component in the oil, with 89.34%. The temperature value attributed to the boiling temperature of linalool is similar to that found in the literature (Merk, 1996; Cavalheiros, Chaar, Breviglieri & Chierice, 2004).

The differences between the boiling points and enthalpies of the measured linalool and essential oil standard are justified by the presence of the minor components, their concentrations in the essential oil and their interactions. It should be considered that the oil has other substances of different polarities, molecular masses and intermolecular forces, which contribute to these differences.

5. Conclusion

The extraction yield of essential oil from *Aniba rosaedora* Ducke obtained was 1.87% (m/m), being major component the linalool (89.34%), followed by α -terpineol (3.06%).

The thermal analysis of essential oil, using TG-DTG e DSC, opened a new path for essential oil analysis. The results obtained were unprecedented for the essential oil of *Aniba rosaedora* Ducke, even allowing to suggest the quantitative determination of linalool by DSC.

As this is a new and efficient technique for determining the boiling temperatures of essential oils, further studies should be carried out in order to broaden its spectrum of scientific investigation of essential oils, as well as envisioning that the technique can be used in certification and quantification of these oils, considering that many of them with high economic value are frequently adulterated.

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