Original Article
Food Science and Technology

ISSN 0101-2061 (Print)
ISSN 1678-457X (Online)

DOI: https://doi.org/10.1590/fst.112322

Non-destructive determination of the oil content in peach palm (*Bactris gasipaes*) flour using NMR and NIR spectroscopies

Yves José de Souza SANTOS[1,2,3*], Natalia Alves CAMPOS[1], Gerson Balbueno BICCA[1], Gisele Teixeira de Souza SORA[1], Luis Fernando POLESI[1], Tania Maria ALBERTE[1], Poliana M. SANTOS[4], Rodrigo Henrique dos Santos GARCIA[5], Luiz Alberto COLNAGO[2], Gabrieli OLIVEIRA-FOLADOR[1]

Abstract

The oil from the fruit of peach palm or Pupunha (*Bactris gasipaes*) is an example of a material with low-cost and good antioxidant capacity. However, Conventional methods for measuring oil content are time-consuming, labor-intensive and use toxic chemicals. In this sense, the aim of this study was evaluated fast and non-destructive spectroscopy methods, such as Near Infrared (NIR) and Time-Domain Nuclear Magnetic Resonance (TD-NMR) (CPMG and ROSE pulse sequences), to quantify the oil content in pupunha flours collected in the amazon forest. For this, 93 samples were used and the results showed three distinct levels of oil in the samples: high, medium and low oil content. Furthermore, the determination coefficient (R^2) reached values of 0.92, 0.92 and 0.70 for NIR, TD-NMR (ROSE) and TD-NMR (CPMG), respectively. Therefore, the NIR and TD-NMR (ROSE) methods demonstrate a higher prediction efficiency, with the NIR achieving 100% classification of the samples.

Keywords: pupunha; Amazonian fruit; oil content; NIR; TD-RMN.

Practical Application: Quantification and characterization of the oil content present in Amazonian fruit by spectroscopic methods, which are included within the concept of green chemistry.

1 Introduction

Brazil has one of the largest plant biodiversity with more than 46.000 known species. Most of the native plants, that produce edible products, have been used only in the diet of the population in the regions where they naturally occur (Barreira et al., 2021). As a large portion of the population has neglected them, these plants are known as unconventional food plants (UFP) (Leterme et al., 2006). However, this scenario has been changing in recent years mainly due to consumer demand for healthier foods and by the industry, which is always looking for new sources of raw materials, as well as climate change, that can reduce the production of conventional plants (Lorenzo et al., 2021).

Peach palm (PP) or pupunha fruit (*Bactris gasipaes Kunth.*) is one of the UFP consumed in the Amazonia region (Andrade et al., 2003) and contains about 90% pulp and 10% seed. The PP pulp or its flour is rich in carbohydrates and oil and some studies have been implemented characterization of their nutritional values (Ferreira & Pena, 2003; Martínez-Girón et al., 2017; Pires et al., 2019; Santos et al., 2023). The importance of edible oils is linked to the fact that they provide energy, essential fatty acids and nutrients (Li et al., 2020), added to this, the lipid fraction of PP fruit is rich in monounsaturated fatty acids (max. 68.2%), which can help reduce total cholesterol (Yuyama et al., 2003) and has high concentration of carotenoids with good inhibition of oxidative processes (Santos et al., 2015), proving to be a good option for human consumption.

Santos et al. (2020) emphasize that the oil from pupunha fruit has a good quality, even when compared to the oil from other Amazonian fruits, and that it therefore diversifies the dietary sources of lipids, because they are sources of ω-3 (linolenic acid), 6 (linoleic acid), and 9 (oleic acid), and may have antioxidant action against some types of cancer, anti-inflammatory effect, and reduce some cardiovascular diseases. In addition, the authors related that the oil yield of the fruit it's on average 23.73% and, therefore, it's considered an oleaginous fruit. The production of linoleic acid in plants is higher than that of linolenic acid, but both are essential because neither can be synthesized in metabolism (Calder, 2017). Also, according to the authors, there is strong evidence that these fatty acids can partially inhibit many aspects of inflammation, oxidative stress and endothelial function. So there are daily consumption recommendations suggested by Food and Agriculture Organization of the United Nations (Burlingame et al., 2009).

Importantly, this plant is cultivated throughout the Amazon and has a very high genetic variability that consequently affecting variability in lipid content and composition (Santos et al., 2023). Traditionally the lipid content, whether in PP or in other

Received 29 Oct., 2022
Accepted 11 Dec., 2022
[1] *Departamento de Engenharia de Alimentos, Universidade Federal de Rondônia, Ariquemes, RO, Brasil*
[2] *Empresa Brasileira de Pesquisa Agropecuária – Embrapa Instrumentação, São Carlos, SP, Brasil*
[3] *Departamento de Engenharia de Alimentos, Faculdade de Zootecnia e Engenharia de Alimentos – FZEA, Universidade de São Paulo – USP, Pirassununga, SP, Brasil*
[4] *Departamento Acadêmico de Química e Biologia, Universidade Tecnológica Federal do Paraná, Curitiba, PR, Brasil*
[5] *Instituto de Química de São Carlos, Universidade de São Paulo, São Carlos, SP, Brasil*
*Corresponding author: yves.souza@usp.br

fruits, has been determined by extraction procedures that are usually slow and use chemical reagents that produce toxic waste (Hempel et al., 2014; Herch et al., 2014; Santos et al., 2020; Torres-Vargas et al., 2021; Zanqui et al., 2020). However, the use of spectroscopic methods, which do not make use of reagents such as Near Infrared (NIR) and Nuclear Magnetic Resonance (NMR), have not yet been reported in the literature to determine the oil content in PP flours.

NIR spectroscopy is a powerful non-destructive tool for quantitative and qualitative analyses and has been widely used in food science (Bizzani et al., 2017; Malegori et al., 2017; Oliveira et al., 2014; Silva et al., 2021; Zhou et al., 2023), chemistry (Fernandes et al., 2008), medicine (Trenfield et al., 2022), and industrial quality control processes (Blanco et al., 2007). TD-NMR has also been widely applied in food science (Bizzani et al., 2020; Machado et al., 2022; Qu et al., 2021; Wang et al., 2018) and several methods have been recognized by various international agencies, like the International Organization for Standardization (ISO) and International Union of Pure and Applied Chemistry (IUPAC) (Todt et al., 2006). Nevertheless, most applications of TD-NMR in food science have been developed in the last two decades with the introduction of new and more versatile hardware and software and new methods for signal acquisition and processing (Colnago et al., 2021).

The principal advantages of these methods are sensitivity, safety, non-invasiveness, inexpensive running costs, rapid and automated sample turnover (Santos et al., 2022; Silva et al., 2022), characteristics that, whether on an industrial or a labor scale, make the production and analysis of the products occur in an extremely optimized. Therefore, this study aimed to evaluate and validate fast and non-destructive NIR and TD-NMR methods to quantify and characterize the oil content in PP flour collected in the Brazilian north amazon forest.

2 Materials and methods

2.1 Selection and preparation of pupunha fruit samples

Peach palm fruits were collected in ideal maturation at Acre (09°58'29" S; 67°48'36" W), situated in the Amazon region in the north of Brazil, and they were inspected and sanitized in sodium hypochlorite solution (100 ppm) for 10 minutes. The fruit was pulped and then lyophilized (Liobras, Liotop L101, Brazil). For each flour sample ten fruits were used. After lyophilization, the samples were crushed and separated (the fine fraction from the gross fraction). The fine fraction samples were stored in packages for analysis. The coarse fraction, basically composed of fibers, was not used in this experiment.

2.2 NMR data acquisition

The TD-NMR analyses were performed in a 0.49 T (19.9 MHz for 1H) Minispec ND Mq-20 TD-NMR (Bruker, Germany) using a 10 mm probe at 25 °C. The TD-NMR signal was obtained with using Carr-Purcell-Meiboom-Gill (CPMG) and radiofrequency-optimized solid-echo (ROSE) pulse sequences (Colnago et al., 2021; Garcia et al., 2019). The CPMG decays were obtained with 90° and 180° of 2.4 and 4.8 μs, τ = 1 ms, 1500 echoes, recycle delay of 2 s and 16 scans. The ROSE sequence (Garcia et al., 2019) parameters were tp_1 = 10 μs, 90° pulse of 2.4 μs and echo time of 10 μs, recycle time of 2 s and 16 scans. The ROSE signals for rigid plus mobile and liquid components were measured at 10 and 50 μs, respectively.

The ^1H spectra of extracted oil were acquired in $CDCl_3$ using a 14.1 T Avance III NMR spectrometer (Bruker, Karlsruhe, Germany) using a 5 mm broadband probe. The chemical shift in ppm was reference to tetra methyl silane (TMS).

2.3 NIR spectra acquisition

The NIR spectra were obtained on a spectrophotometer Spectrum 100N FT-NIR (Perkin-Elmer, Norwalk, CT, USA) from an average of 32 scans in a spectral region between 10.000 and 4.000 cm^{-1}.

2.4 Reference fruit characterization

The oil content in ninety-three samples were determined by the Bligh and Dyer method (Bligh & Dyer, 1959). Initially, 1 g of sample was soaked in a mixture of 8 mL of chloroform, 16 mL methanol and 6.8 mL of distilled water. The solution was gently agitated for 30 minutes, with pause every ten minutes to remove the gas formed. After that, a further 8 mL of chloroform and 8 mL of 1.5% sodium sulfate solution were added and stirred for 3 minutes. Then, 10 mL of this solution was removed and transferred to falcon type test tube with 1 g of sodium sulfate. It was shaken for approximately 2 minutes and filtrated. Finally, 5 mL of the filtered solution was removed and placed in petri dishes, previously dried and weight.

The oil content was calculated following Equation 1.

$$Oil\ Content = \left(\left(\frac{DS}{M}\right)x3.2\right)x100 \quad (1)$$

Where: DS: dry sample mass (g); M: mass of sample used (g).

The analyses were performed in duplicate due to the low quantity of pulp material.

2.5 Multivariate data analysis

All data were processed in MATLAB software, v. R2021a (MathWorks, Natick, USA) version 8.9 (Eigenvector Technologies, Manson, USA) along with the PLS Toolbox. Before the analysis, NIR spectra were preprocessed by the first derivative and Savitzky-Golay polynomial filter with a 25-point window and mean centered. For the NMR data, mean centering was chosen as the only preprocessing method.

Initial multivariate data analysis was performed with Principal Component Analysis (PCA). The PCA was used as an exploratory analysis to visualize the sample distribution in the multivariate space and identify any natural clustering among to the samples.

Supervised classification models using partial least squares discriminant analysis (PLS-DA) was built to discriminate samples according to their lipid concentration. Initially, PLS-DA models were built for the discrimination of PP samples into three classes: low (1.8-6.8%), medium (7.0-9.9%) and high (10.6-15.1%) lipid concentration. The whole data set was split into training and test

sets, corresponding to two thirds and one third of the samples, respectively. The Kennard-Stone algorithm (Kennard & Stone, 1969) was applied for the selection of training samples separately in each class. The secondary models were built to discriminate the samples into two classes: low (1.8- 9.9%) and high (10.6-15.1%) lipid concentration. The 93 signals were also systematically separated into a training set of 69 samples (30 belonging to low concentration and 39 from high concentration) and a test set of 24 samples (11 belonging to low concentration and 13 from high concentration) using the algorithm previously mentioned. The assessment of the classification models was done using the classification rates.

The multivariate calibration method was developed based on partial least squares (PLS) regression. For the development of the PLS models the samples were divided into calibration (75%) and validation (25%) sets also applying the Kennard-Stone algorithm (Kennard & Stone, 1969). The number of latent variables (LV) was selected by using venetian blinds (with 10 splits) cross-validation. The regression models were evaluated using the Root Mean Square Error of Calibration (RMSEC), Root Mean Square Error of Cross validation (RMSECV), Root Mean Square Error of Prediction (RMSEP) and residual prediction deviation (RPD) (Magwaza et al., 2016; Santos et al., 2022; Tahir et al., 2016). The random *t*-test was employed to compare the predictive accuracy of models. Randomization tests (or permutation tests, as they are sometimes called) is a general distribution-free test for the equality of two distributions using paired data (van der Voet, 1994). A short MATLAB code for applying the randomization test is provided in Olivieri (2015).

3 Results and discussion

3.1 Characterization of the oil in peach palm flour

Figure 1 shows the oil content of the lyophilized fine fraction of 93 PP flour samples. The oil content varied from 1.8 to 14.1%, with the mean, standard deviation (δ) and coefficient of variation (CV = (δ/mean) x 100) equal 9.3%, 3.3 and 35.52%, respectively. These results indicate large oil content variability in the flours.

This result agrees with Santos et al. (2023) that observed a variation from 4.65 to 11.23 g/100 g in PP flour and Carvalho et al. (2013) that analyzed the PP flour from the state of Pará (east of the amazon forest in Brazil) and obtained about 11.56% of oil. This large variation in oil content is certainly due to variation in genetics, soils and environmental conditions, as observed by Santos et al. (2023) and Barbosa et al. (2020).

A recent review, showed that the oil from the pupunha fruit has very interesting properties: it is liquid at room temperature and less prone to oxidation when compared to other oils with a higher concentration of linoleic acid (Costa et al., 2022).

3.2 TD-NMR analysis

It was first investigated the use of the standard TD-NMR method for determination of oil content in dry samples [ISO 8292 (International Organization for Standardization, 1991)]. In this method, the amplitude of the FID at 50 μs, obtained after the 90° pulse, is proportional to the oil content and sample mass of the dry samples. The determination coefficient (R^2), obtained by the NMR intensity, that divide respective mass versus the oil

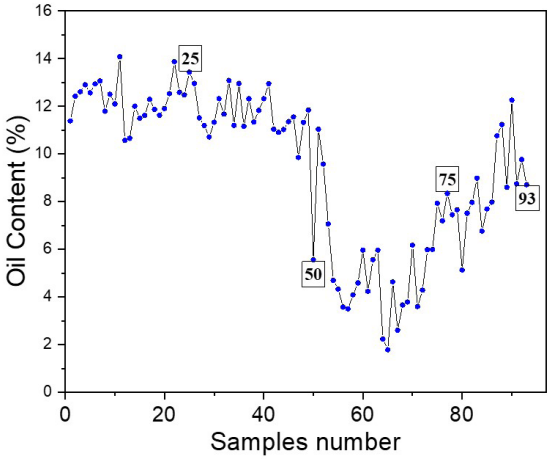

Figure 1. Distribution of samples in accordance with oil content*, in percentage, of the lyophilized fine fraction of 93 PP pulps (flour) samples. *High oil concentration zone: > 10% (represented by the position of sample 25), intermediate oil content zone: between 8 and 10% (represented by samples 75 and 93), low oil content zone: < 8% (represented by sample 50).

content of the PP flour samples (Figure 1) was $R^2 = 0.86$, which is lower than generally reported in the literature (Colnago et al., 2021). An explanation for this lower R^2 can be associated with the large variation in saturated/unsaturated fatty acid content observed in PP sample, that have been reported by several authors. Yuyama et al. (2003) analyzed by gas chromatography the fatty acid profile of PP oil from fruits collected in Brazilian central Amazonia area, and observed high variation in palmitic and oleic acids content. Palmitic acid varied from 24 to 42% and oleic acid from 43 to 61%. All the other fatty acids (palmitoleic, stearic, linoleic and linolenic) were in a much lower concentration. Santos et al. (2020) observed 36 and 50% values of palmitic and oleic acid in red PP oil, respectively.

In order to check the composition of the fatty acid content of the samples, ^{1}H-High resolution (HR) NMR spectra were obtained for several samples with high and low oil content. HR-NMR has been an alternative to gas chromatography for fast determination of fatty acid content, direct on the extracted oil, without any chemical reaction (Barison et al., 2010; Santos et al., 2017) and, based on the spectra, it was observed that samples with lower oil have high-saturated fatty acid content and samples with higher oil have higher unsaturated fatty acid content. Figure 2 shows the HR-NMR spectra of the oils extracted from a sample with high (red) and low (black) lipid content, with expansions of 2, 3, 4 and 8 signals.

As can be seen in Figure 2, the spectra obtained showed a typical profile of triacylglycerides (TAG). Peak 1, at approximately 0.9 ppm, is assigned to the terminal methyl group of fatty acid and peak 2 to the fatty acids methylene groups that are not close to carboxyl or double bonds carbons. Peak 3 is related to the carbon 3 methylene groups, while the peaks from 4 to 6 to the methylenes groups bonded to unsaturated carbons. Peak 7 can be assigned to hydrogens in carbons 1 and 3 of glycerol and peak 8 to hydrogens in double bonds carbons and to hydrogen in C2 of glycerol (small peak at 5.27 ppm) (Hama & Fitzsimmons-Thoss, 2022).

By comparing the HR-NMR spectra of the samples with low and high oil content (Figure 2) it is possible verify that the main differences are in the peaks 2, 4 and 8. The sample with low oil content has stronger signal 2, and weaker signals 4 and 8. Conversely, the sample with high oil content showed weaker signal 2 and stronger signals 4 and 8. These results indicate that the samples with low oil content have higher saturated fatty acids content than those with high oil content. These differences in fatty profile can explain the lower R^2 of the standard NMR method.

Another TD-NMR method to quantify oil content in PP flour was tested. The method is based on the ratio between the signal of solid and mobile hydrogens in PP pulps and does not need the sample mass. This method is based on a probe with very low dead time or based on a solid echo (SE) pulse sequence that refocuses the dipolar (Horn et al., 2011). Here it was used a new solid echo pulse sequence known as RK-ROSE (Garcia et al., 2019) which is much more efficient to refocus the solid signal than the standard solid echo (SE) sequence. In the ROSE sequence it was used the ratio between the echo intensity, related to the signal of solid plus liquid components, and the FID signal related only to the liquid component or oil.

Figure 3A shows the ROSE signals for PP flour samples with higher (12% blue), intermediate (7%, red) and low (4%, black) oil content. The determination coefficient between the ratio of the intensities of ROSE signals and the oil content for the 93 samples (Figure 1) was $R^2 = 0.7$. This value is lower than the one obtained by the standard method proposed in the ISO 10565 (International Organization for Standardization, 1998). This low R^2 value can also be related to the large fatty acid variation in the PP flour oils.

The oil quality in PP flour samples was analyzed by TD-NMR using the CPMG pulse sequence that yield an exponential decaying signal governed by the transverse relaxation time T_2. CPMG decay has been widely used as a fast and non-destructive TD-NMR method to determine the fatty acid content (Andrade et al., 2011; Santos et al., 2017). The CPMG decays of PP flour samples with higher (12% blue), intermediate (7%, red) and low (4%, black) oil content are showed in Figure 3B.

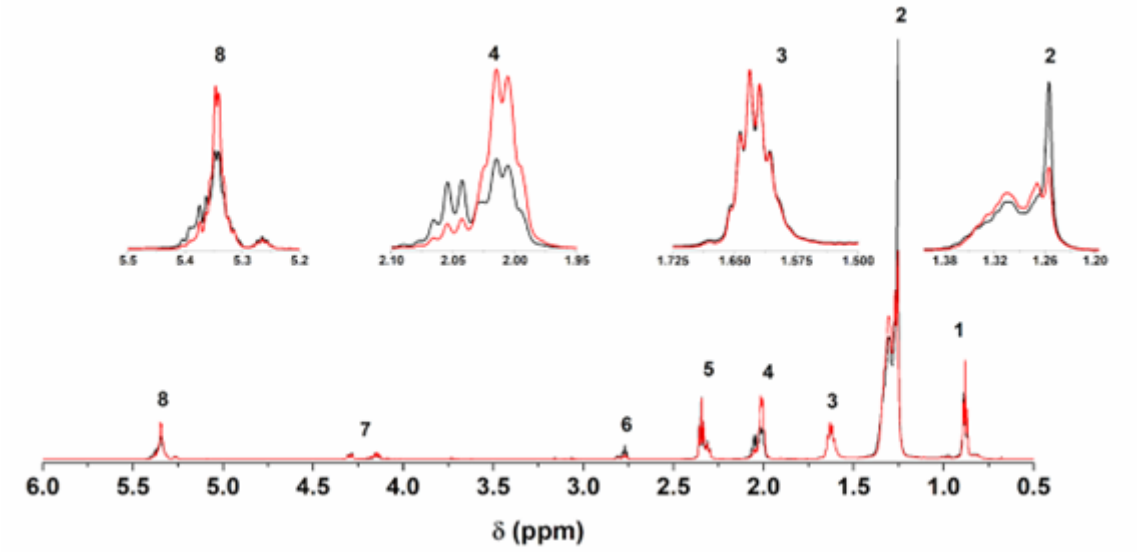

Figure 2. ^{1}H-NMR spectra of PP samples with high (red) and low (black) oil content with zoom for signals 2, 3, 4 and 8.

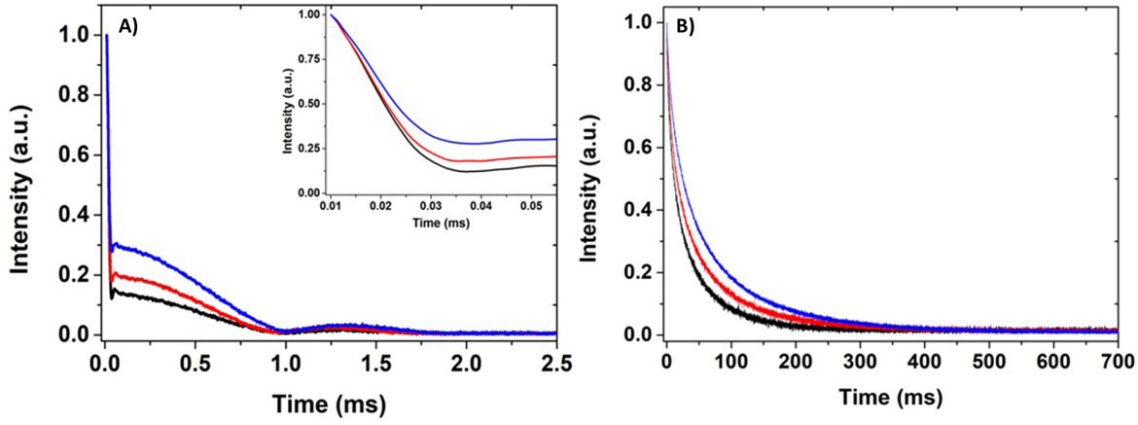

Figure 3. (A) ROSE and (B) CPMG signals of the 3 PP flour samples, with higher (12%, blue) intermediate (7%, red) and low (4%, black) oil content.

These results showed that the sample with higher oil content has longer decay than the samples with intermediate and low oil content. Therefore, it is possible to assume that the sample with low oil content has higher saturated fatty acids content than the sample with high oil content, as observed in the HR-NMR spectrum. The determination coefficient (R^2) between the T_2 values and oil content was 0.72 indicating the correlation the oil content and fatty acid profile.

3.3 Multivariate analyses

Due to the lowers R^2 values obtained with the univariate strategies, multivariate models were proposed by using TD-NMR (ROSE and CPMG) and NIR data.

Principal component analysis

Prior to the development of classification and quantification models, PCA models were constructed with NIR and TD-NMR data. The outcomes of the PCA (Figure 4) models reveal a trend grouping the samples according to the oil concentration. The scores plot of the NIR model (Figure 4A) showed that the second component (PC2) is the main responsible for this separation: PP samples with high lipids concentrations were placed on the positive side of PC2, whereas the samples with medium and low concentrations were in the negative side of PC2. On the other hand, looking the scores plot of the PCA models obtained with the TD-NMR data (Figure 4B-4C) can be observed that the samples are differentiated according to their value of PC1: In positive scores along the component, includes the samples with high oil concentration, while at negative values includes the samples with medium and low lipid content.

Classification of PP flour samples

After a preliminary exploratory analysis, PLS-DA models were developed for classification purposes. PLS-DA models obtained with spectroscopic techniques (NIR and TD-NMR) were examined by comparing the percentage of correct predictions.

As previously mentioned, in the first part of the present study, PLS-DA was used to distinguish PP flour samples into three classes: low, medium and high lipid concentration. Table 1 shows the results obtained of the correct classification rate for the test set and their arithmetic average, together with the number of latent variables (LVs) used in each model. This outcome suggests that the optimal PLS-DA model has been built with the NIR data it leads the highest average of correct classification (81.1%), corresponding to 5 samples of class 1 (over 6), 3 samples of class 2 (over 5) and 13 samples of class 3 (over 13) properly assigned. The PLS-DA model obtained with TD-NMR (CPMG pulse sequence) showed the worst performance, with an average of correct classification equal 41.1%, because the CPMG signal was not intended to measure the oil content but the variation of fatty acid profile.

Since the PLS-DA models obtained with TD-NMR showed an average of correct classification (below than 75%), further models were built to distinguish PP flour samples in two classes: low and high lipid concentration. Looking at the results in Table 2, it is evident that those models showed better classification ability than those mentioned above. NIR model showed 100.0% of samples correctly classified in both classes, confirming to give more accurate models than those built with TD-NMR data. However, the classification model obtained with the TD-NMR (ROSE pulse sequence) data, provided correct classification rates of 81.8% and 84.6% for Class 1 and Class 2, respectively.

Table 1. Validation results for the PLS-DA models on NIR, TD-NMR (ROSE pulse sequence) and TD-NMR (CPMG pulse sequence) for Class 1, 2 and 3.

Technique	LV	Correct classification (%)			
		Class 1	Class 2	Class 3	Average
NIR	3	83.3	60.0	100.0	81.1
TD-NMR (ROSE)	2	66.7	100.0	53.8	73.5
TD-NMR(CPMG)	2	0.0	40.0	83.3	41.1

Table 2. Validation results for the PLS-DA models on NIR, TD-NMR (ROSE pulse sequence) and TD-NMR (CPMG pulse sequence) for Class 1 and 2.

Technique	LV	% Correct classification		
		Class 1	Class 2	Average
NIR	3	100.0	100.0	100.0
TD-NMR (ROSE)	3	81.8	84.6	83.2
TD-NMR(CPMG)	3	72.7	84.6	78.7

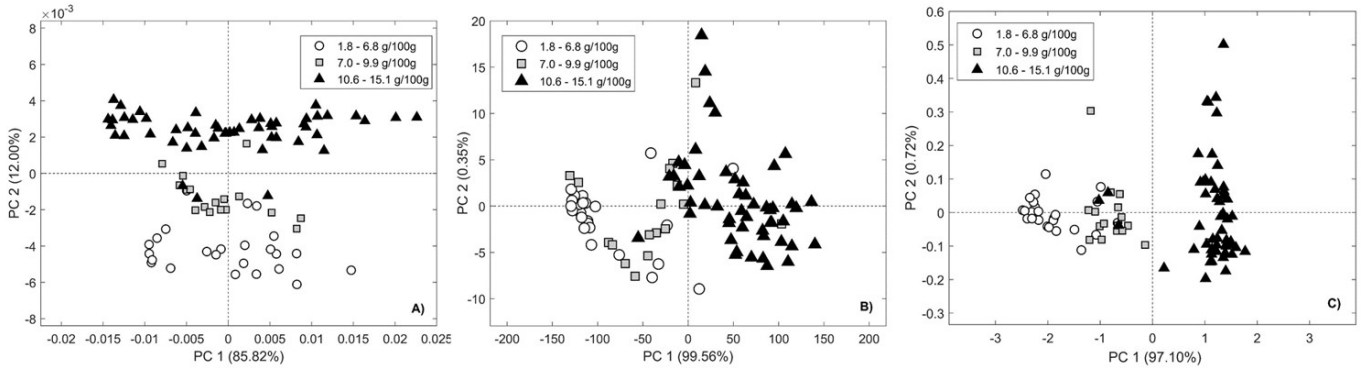

Figure 4. Scores plot of the models developed with (A) NIR spectra, (B) TD-NMR (CPMG pulse sequence) and (C) TD-NMR (ROSE pulse sequence).

3.4 Quantitative determination of lipid concentration

PLS models were developed in order to predict the concentration of lipid in the samples. The performances of the models obtained are summarized in Table 3. An initial comparison between results using NIR and TD-NMR (ROSE and CPMG pulse sequences) data suggests that the NIR model was slightly better, due to the lower RMSEP. However, the comparison of models should not be based only in the RMSE values. A suitable statistical test should be applied to assess whether the values are statistically different. In this study, we use the randomization test, suggested by van der Voet (1994) with a significance level of probability of 0.05. The result indicates that the RMSEP found by NIR is not statistically different from the one by TD-NMR (ROSE pulse sequence), since the probability values obtained are higher than the critical level of 0.05. Although, the difference between the RMSEP values of NIR and TD-NMR (CPMG pulse sequence) models was found to be significant ($p = 0.02$).

The PLS models were also evaluated based on the RPD values. According to the literature, good calibration models must have RPD values higher than 2.4, while models with RPD values between 2.4 and 1.5 are considered acceptable. Models with RPD lower than 1.5 are considered unusable (Fan et al., 2015). Considering the values presented in Table 3, RPD estimates were satisfactory for the NIR and TD-NMR (ROSE pulse sequence) models.

Table 3. The calibration and prediction results of PLS models for lipid in pulp pupunha.

REGRESSION PARAMETERS	NIR	TD-NMR (ROSE)	TD-NMR (CPMG)
Number of LV	4	2	2
RMSEC	0.96	1.00	1.39
RMSECV	1.11	1.19	1.54
RMSEP	1.39	1.54	1.88
R^2cal	0.92	0.92	0.70
R^2val	0.70	0.71	0.58
RPDcal	3.17	2.96	1.72
RDPval	1.97	1.78	1.46

RMSEC: Root Mean Square Error of Calibration; RMSECV: Root Mean Square Error of Cross validation; REMSEP: Root Mean Square Error of Prediction; RPD: Residual Prediction Deviation; cal: calibration group; val: validation group; R^2: determination coefficient.

Figure 5 presents the scatter plots showing reference versus predicted values by using NIR (Figure 5A), TD-NMR (CPMG) (Figure 5B) and TD-NMR (ROSE) (Figure 5C) data. It can be observed that the concentrations of all parameters analyzed are distributed along the adjusted regression line and the samples of the validation set are contained in the same range of calibration samples.

4 Conclusion

Therefore, the potential of TD-NMR and NIR techniques to quantify oil content in peach palm flour and classify the samples according to the oil content was demonstrated. According to statistical analysis, the PLS models obtained with NIR and TD-NMR (ROSE pulse sequence) data are not statistically different and the RPD values showed that those models are satisfactory.

The CPMG signal shows that the fatty acid varied in oil of peach palm flour been more and less saturated in flours low and high oil content, respectively.

Industrially, these noninvasive methodologies, considered innovative, represent a higher speed in the analysis of raw materials and, consequently, a higher rate of production. Therefore, they are extremely advantageous forms of analysis for industrial application and represent a great environmental advance. In addition, this work also opened perspectives about the quality of the fruit, since it was possible to relate the oil content to its quality and, therefore, such methodologies can help in the choice of the fruit according to the need and quality of the oil.

Acknowledgements

The authors would like to thank the Rondônia Research Foundation (FAPERO, grant 018/2016), São Paulo Research Foundation (FAPESP, grants 2019/13656-8; 2021/12694-3) National Council for Scientific and Technological Development (CNPq, grant 302866/2017-5; 307635/2021-0).

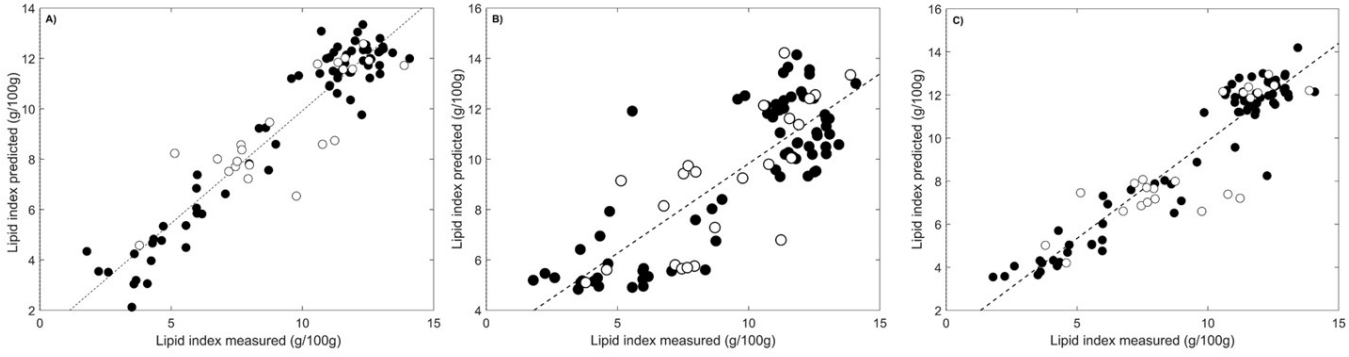

Figure 5. Plot of reference values versus predict values from (A) NIR, (B) TD-NMR (CPMG) and (C) TD-NMR (ROSE).

References

Andrade, F. D., Marchi, A. No., & Colnago, L. A. (2011). Qualitative analysis by online nuclear magnetic resonance using Carr-Purcell-Meiboom-Gill sequence with low refocusing flip angles. *Talanta*, 84(1), 84-88. http://dx.doi.org/10.1016/j.talanta.2010.12.033. PMid:21315902.

Andrade, J. S., Pantoja, L., & Maeda, R. N. (2003). Melhoria do rendimento e do processo de obtenção da bebida alcoólica de pupunha (Bactris gasipaes Kunth). *Food Science and Technology*, 23, 34-38. http://dx.doi.org/10.1590/S0101-20612003000400007.

Barbosa, A. P. P., Moraes, A. F., & Chisté, R. C. (2020). Physicochemical characterization and quantification of bioactive compounds of Antrocaryon amazonicum fruits cultivated in Brazilian Amazonia. *CyTA: Journal of Food*, 18(1), 616-623. http://dx.doi.org/10.1080/19476337.2020.1810129.

Barison, A., Silva, C. W. P., Campos, F. R., Simonelli, F., Lenz, C. A., & Ferreira, A. G. (2010). A simplemethodology for the determination of fatty acid composition in edible oils through ^{1}H NMR spectroscopy. *Magnetic Resonance in Chemistry*, 48(8), 642-650. https://doi.org/10.1002/mrc.2629. PMid:20589730.

Barreira, T. F., Paula, G. X. Fo., Priore, S. E., Santos, R. H. S., & Pinheiro-Sant'ana, H. M. (2021). Nutrient content in ora-pro-nóbis (Pereskia aculeata Mill.): unconventional vegetable of the Brazilian Atlantic Forest. *Food Science and Technology*, 41(Suppl. 1), 47-51. http://dx.doi.org/10.1590/fst.07920.

Bizzani, M., Flores, D. W. M., Colnago, L. A., & Ferreira, M. D. (2017). Non-invasive spectroscopic methods to estimate orange firmness, peel thickness, and total pectin content. *Microchemical Journal*, 133, 168-174. http://dx.doi.org/10.1016/j.microc.2017.03.039.

Bizzani, M., Flores, D. W. M., Moraes, T. B., Colnago, L. A., Ferreira, M. D., & Spoto, M. H. F. (2020). Non-invasive detection of internal flesh breakdown in intact Palmer mangoes using time-domain nuclear magnetic resonance relaxometry. *Microchemical Journal*, 158, 105208. http://dx.doi.org/10.1016/j.microc.2020.105208.

Blanco, M., Alcalá, M., Planells, J., & Mulero, R. (2007). Quality control of cosmetic mixtures by NIR spectroscopy. *Analytical and Bioanalytical Chemistry*, 389(5), 1577-1583. http://dx.doi.org/10.1007/s00216-007-1541-3. PMid:17805519.

Bligh, E. G., & Dyer, W. J. (1959). A rapid method of total lipid extraction and purification. *Canadian Journal of Biochemistry and Physiology*, 37(8), 911-917. http://dx.doi.org/10.1139/o59-099. PMid:13671378.

Burlingame, B., Nishida, C., Uauy, R., & Weisell, R. (2009). Fats and fatty acids in human nutrition: introduction. *Annals of Nutrition & Metabolism*, 55(1-3), 5-7. http://dx.doi.org/10.1159/000228993. PMid:19752533.

Calder, P. C. (2017). Omega-3 fatty acids and inflammatory processes: from molecules to man. *Biochemical Society Transactions*, 45(5), 1105-1115. http://dx.doi.org/10.1042/BST20160474. PMid:28900017.

Carvalho, A. V., Beckman, J. C., Maciel, R. A., & Farias, J. T. No. (2013). Características físicas e químicas de frutos de pupunheira no estado do Pará. *Revista Brasileira de Fruticultura*, 35(3), 763-768. http://dx.doi.org/10.1590/S0100-29452013000300013.

Colnago, L. A., Wiesman, Z., Pages, G., Musse, M., Monaretto, T., Windt, C. W., & Rondeau-Mouro, C. (2021). Low field, time domain NMR in the agriculture and agrifood sectors: an overview of applications in plants, foods and biofuels. *Journal of Magnetic Resonance*, 323, 106899. http://dx.doi.org/10.1016/j.jmr.2020.106899. PMid:33518175.

Costa, R. D. S., Rodrigues, A. M. C., & Silva, L. H. M. (2022). The fruit of peach palm (Bactris gasipaes) and its technological potential: an overview. *Food Science and Technology*, 42, e82721. http://dx.doi.org/10.1590/fst.82721.

Fan, S., Huang, W., Guo, Z., Zhang, B., & Zhao, C. (2015). Prediction of soluble solids content and firmness of pears using hyperspectral reflectance imaging. *Food Analytical Methods*, 8(8), 1936-1946. http://dx.doi.org/10.1007/s12161-014-0079-1.

Fernandes, H. L., Raimundo, I. M. Jr., Pasquini, C., & Rohwedder, J. J. R. (2008). Simultaneous determination of methanol and ethanol in gasoline using NIR spectroscopy: Effect of gasoline composition. *Talanta*, 75(3), 804-810. http://dx.doi.org/10.1016/j.talanta.2007.12.025. PMid:18585150.

Ferreira, C. D., & Pena, R. S. (2003). Comportamento higroscópico da farinha de pupunha (Bactris gasipaes). *Food Science and Technology*, 23(2), 251-255. http://dx.doi.org/10.1590/S0101-20612003000200025.

Garcia, R. H. S., Filgueiras, J. G., Azevedo, E. R., & Colnago, L. A. (2019). Power-optimized, time-reversal pulse sequence for a robust recovery of signals from rigid segments using time domain NMR. *Solid State Nuclear Magnetic Resonance*, 104, 101619. http://dx.doi.org/10.1016/j.ssnmr.2019.101619. PMid:31470338.

Hama, J. R., & Fitzsimmons-Thoss, V. (2022). Determination of unsaturated fatty acids composition in walnut (Juglans regia L.) Oil using NMR spectroscopy. *Food Analytical Methods*, 15(5), 1226-1236. http://dx.doi.org/10.1007/s12161-021-02203-0.

Hempel, J., Amrehn, E., Quesada, S., Esquivel, P., Jiménez, V. M., Heller, A., Carle, R., & Schweiggert, R. M. (2014). Lipid-dissolved γ-carotene, β-carotene, and lycopene in globular chromoplasts of peach palm (Bactris gasipaes Kunth) fruits. *Planta*, 240(5), 1037-1050. http://dx.doi.org/10.1007/s00425-014-2121-3. PMid:25023631.

Herch, W., Kallel, H., & Boukhchina, S. (2014). Physicochemical properties and antioxidant activity of Tunisian date palm (Phoenix dactylifera L.) oil as affected by different extraction methods. *Food Science and Technology*, 34(3), 464-470. http://dx.doi.org/10.1590/1678-457x.6360.

Horn, P. J., Neogi, P., Tombokan, X., Ghosh, S., Campbell, B. T., & Chapman, K. D. (2011). Simultaneous quantification of oil and protein in cottonseed by low-field time-domain nuclear magnetic resonance. *Journal of the American Oil Chemists' Society*, 88(10), 1521-1529. http://dx.doi.org/10.1007/s11746-011-1829-5.

International Organization for Standardization – ISO. (1991). *ISO 8292:1991: animal and vegetable fats and oils - determination of solid fat content - pulsed nuclear magnetic resonance method*. Geneva: ISO.

International Organization for Standardization – ISO. (1998). *ISO 10565:1998: oilseeds - simultaneous determination of oil and water contents — method using pulsed nuclear magnetic resonance spectrometry*. Geneva: ISO.

Kennard, R. W., & Stone, L. A. (1969). Computer aided design of experiments. *Technometrics*, 11(1), 137-148. http://dx.doi.org/10.1080/00401706.1969.10490666.

Leterme, P., Buldgen, A., Estrada, F., & Londoño, A. M. (2006). Mineral content of tropical fruits and unconventional foods of the Andes and the rain forest of Colombia. *Food Chemistry*, 95(4), 644-652. http://dx.doi.org/10.1016/j.foodchem.2005.02.003.

Li, X., Zhang, L., Zhang, Y., Wang, D., Wang, X., Yu, L., Zhang, W., & Li, P. (2020). Review of NIR spectroscopy methods for nondestructive quality analysis of oilseeds and edible oils. *Trends in Food Science & Technology*, 101, 172-181. http://dx.doi.org/10.1016/j.tifs.2020.05.002.

Lorenzo, N. D., Santos, O. V., & Lannes, S. C. S. (2021). Fatty acid composition, cardiovascular functionality, thermogravimetric-differential, calorimetric and spectroscopic behavior of pequi oil (Caryocar villosum Pers.). *Food Science and Technology*, 41(2), 524-529. http://dx.doi.org/10.1590/fst.16420.

Machado, G. O., Teixeira, G. G., Garcia, R. H. S., Moraes, T. B., Bona, E., Santos, P. M., & Colnago, L. A. (2022). Non-invasive method to predict the composition of requeijão cremoso directly in commercial packages using time domain NMR relaxometry and chemometrics. *Molecules*, 27(14), 4434. http://dx.doi.org/10.3390/molecules27144434. PMid:35889306.

Magwaza, L. S., Naidoo, S. I. M., Laurie, S. M., Laing, M. D., & Shimelis, H. (2016). Development of NIRS models for rapid quantification of protein content in sweetpotato [Ipomoea batatas (L.) LAM.]. *Lebensmittel-Wissenschaft + Technologie*, 72, 63-70. http://dx.doi.org/10.1016/j.lwt.2016.04.032.

Malegori, C., Marques, E. J. N., Freitas, S. T., Pimentel, M. F., Pasquini, C., & Casiraghi, E. (2017). Comparing the analytical performances of Micro-NIR and FT-NIR spectrometers in the evaluation of acerola fruit quality, using PLS and SVM regression algorithms. *Talanta*, 165, 112-116. http://dx.doi.org/10.1016/j.talanta.2016.12.035. PMid:28153229.

Martínez-Girón, J., Figueroa-Molano, A. M., & Ordóñez-Santos, L. E. (2017). Effect of the addition of peach palm (Bactris gasipaes) peel flour on the color and sensory properties of cakes. *Food Science and Technology*, 37(3), 418-424. http://dx.doi.org/10.1590/1678-457x.14916.

Oliveira, G. A., Bureau, S., Renard, C. M.-G. C., Pereira-Netto, A. B., & Castilhos, F. (2014). Comparison of NIRS approach for prediction of internal quality traits in three fruit species. *Food Chemistry*, 143, 223-230. http://dx.doi.org/10.1016/j.foodchem.2013.07.122. PMid:24054234.

Olivieri, A. C. (2015). Practical guidelines for reporting results in single- and multi-component analytical calibration: a tutorial. *Analytica Chimica Acta*, 868, 10-22. http://dx.doi.org/10.1016/j.aca.2015.01.017. PMid:25813230.

Pires, M. B., Amante, E. R., Lopes, A. S., Rodrigues, A. M. C., & Silva, L. H. M. (2019). Peach palm flour (Bactris gasipae KUNTH): potential application in the food industry. *Food Science and Technology*, 39(3), 613-619. http://dx.doi.org/10.1590/fst.34617.

Qu, Y. L., Xie, D. T., Hu, C. Y., Deng, H., & Meng, Y. H. (2021). Direct steam injection pretreatment improves microwave-assisted extraction yield for total flavonoids and myricetin from hovenia dulcis thunb. *Food Science and Technology*, 41(Suppl. 1), 334-342. http://dx.doi.org/10.1590/fst.13620.

Santos, M. F. G., Mamede, R. V. S., Rufino, M., Brito, E., & Alves, R. (2015). Amazonian native palm fruits as sources of antioxidant bioactive compounds. *Antioxidants*, 4(3), 591-602. http://dx.doi.org/10.3390/antiox4030591. PMid:26783846.

Santos, O. V., Soares, S. D., Dias, P. C. S., Duarte, S. P. A., Santos, M. P. L., & Nascimento, F. C. A. (2020). Chromatographic profile and bioactive compounds found in the composition of pupunha oil (Bactris gasipaes Kunth): implications for human health TT. *Revista de Nutrição*, 33, e190146. http://dx.doi.org/10.1590/1678-9805202033e190146.

Santos, P. M., Kock, F. V. C., Santos, M. S., Lobo, C. M. S., Carvalho, A. S., & Colnago, L. A. (2017). Non-invasive detection of adulterated olive oil in full bottles using time-domain NMR relaxometry. Journal of the Brazilian Chemical Society, 28(2), 385-390. http://dx.doi.org/10.5935/0103-5053.20160188.

Santos, Y. J. S., Facchinatto, W. M., Rochetti, A. L., Carvalho, R. A., Feunteun, S., Fukumasu, H., Morzel, M., Colnago, L. A., & Vanin, F. M. (2023). Systemic characterization of pupunha (Bactris gasipaes) flour with views of polyphenol content on cytotoxicity and protein in vitro digestion. *Food Chemistry*, 405(Pt A), 134888. http://dx.doi.org/10.1016/j.foodchem.2022.134888.

Santos, Y. J. S., Malegori, C., Colnago, L. A., & Vanin, F. M. (2022). Application on infrared spectroscopy for the analysis of total phenolic compounds in fruits. *Critical Reviews in Food Science and Nutrition*, 1-11. In press. http://dx.doi.org/10.1080/10408398.2022.2128036. PMid:36178354.

Silva, L. K. R., Jesus, J. C., Onelli, R. R. V., Conceição, D. G., Santos, L. S., & Ferrão, S. P. B. (2021). Discriminating Coalho cheese by origin through near and middle infrared spectroscopy and analytical measures. Discrimination of Coalho cheese origin. *International Journal of Dairy Technology*, 74(2), 393-403. http://dx.doi.org/10.1111/1471-0307.12767.

Silva, L. K. R., Santos, L. S., & Ferrão, S. P. B. (2022). Application of infrared spectroscopic techniques to cheese authentication: a review. *International Journal of Dairy Technology*, 75(3), 490-512. http://dx.doi.org/10.1111/1471-0307.12859.

Tahir, H. E., Xiaobo, Z., Tinting, S., Jiyong, S., & Mariod, A. A. (2016). Near-Infrared (NIR) spectroscopy for rapid measurement of antioxidant properties and discrimination of Sudanese honeys from different botanical origin. *Food Analytical Methods*, 9(9), 2631-2641. http://dx.doi.org/10.1007/s12161-016-0453-2.

Todt, H., Guthausen, G., Burk, W., Schmalbein, D., & Kamlowski, A. (2006). Water/moisture and fat analysis by time-domain NMR. *Food Chemistry*, 96(3), 436-440. http://dx.doi.org/10.1016/j.foodchem.2005.04.032.

Torres-Vargas, O. L., Luzardo-Ocampo, I., Hernandez-Becerra, E., & Rodríguez-García, M. E. (2021). Physicochemical characterization of unripe and ripe chontaduro (Bactris gasipaes Kunth) fruit flours and starches. *Starch*, 73(7-8), 2000242. http://dx.doi.org/10.1002/star.202000242.

Trenfield, S. J., Januskaite, P., Goyanes, A., Wilsdon, D., Rowland, M., Gaisford, S., & Basit, A. W. (2022). Prediction of solid-state form of SLS 3D printed medicines using NIR and Raman spectroscopy. *Pharmaceutics*, 14(3), 589. http://dx.doi.org/10.3390/pharmaceutics14030589. PMid:35335965.

van der Voet, H. (1994). Comparing the predictive accuracy of models using a simple randomization test. *Chemometrics and Intelligent Laboratory Systems*, 25(2), 313-323. http://dx.doi.org/10.1016/0169-7439(94)85050-X.

Wang, H., Wang, R., Song, Y., Kamal, T., Lv, Y., Zhu, B., Tao, X., & Tan, M. (2018). A fast and non-destructive LF-NMR and MRI method to discriminate adulterated shrimp. *Journal of Food Measurement and Characterization*, 12(2), 1340-1349. http://dx.doi.org/10.1007/s11694-018-9748-x.

Yuyama, L. K. O., Aguiar, J. P. L., Yuyama, K., Clement, C. R., Macedo, S. H. M., Fávaro, D. I. T., Afonso, C., Vasconcellos, M. B. A., Pimentel, S. A., Badolato, E. S. G., & Vannucchi, H. (2003). Chemical composition of the fruit mesocarp of three peach palm (Bactris gasipaes) populations grown in Central Amazonia, Brazil. *International Journal of Food Sciences and Nutrition*, 54(1), 49-56. http://dx.doi.org/10.1080/0963748031000061994. PMid:12701237.

Zanqui, A., Silva, C., Ressutte, J., Morais, D., Santos, J., Eberlin, M., Cardozo-Filho, L., Visentainer, J., Gomes, S., & Matsushita, M. (2020). Brazil nut oil extraction using subcritical *n*-Propane: advantages and chemical composition. *Journal of the Brazilian Chemical Society*, 31(3), 603-612. http://dx.doi.org/10.21577/0103-5053.20190225.

Zhou, M., Long, T., Zhao, Z., Chen, J., Wu, Q., Wang, Y., & Zou, Z. (2023). Honey quality detection based on near-infrared spectroscopy. *Food Science and Technology*, 43, e98822. http://dx.doi.org/10.1590/fst.98822.