Orbital: The Electronic Journal of Chemistry
journal homepage: www.orbital.ufms.br
e-ISSN 1984-6428

| Vol 11 | | No. 6 | | Special Issue November 2019 |

FULL PAPER

Data Preprocessing and Homogeneity: The Influence on Robustness and Modeling by PLS Via NIR of Fish Burgers

Caroline Marques*, Carla Cristina Lise, Vanderlei Aparecido de Lima, Marina Leite Mitterer-Daltoé

Graduate Program in Chemical and Biochemical Technology Processes, Chemistry Department, Federal University of Technology, Km 01, 85503-390 - Paraná, Pato Branco, Brazil.

Article history: Received: 18 September 2018; revised: 05 November 2019; accepted: 05 November 2019. Available online: 09 November 2019. DOI: http://dx.doi.org/10.17807/orbital.v11i6.1234

Abstract:

Fish burgers as new products require their shelf life investigated. Sensory results usually do not follow a homogeneous profile, as it measures human perception. Once the sensory and physicochemical monitoring of the shelf life takes time and considerable investment, the Near Infrared spectroscopy comes as a fast instrumental technique, which can access multiple parameters from the sample at the same time. In order to replace traditional methods improving mathematical modeling, the objective of this study is the estimation of the data preprocessing and homogeneity (Kolmogorov–Smirnov) influence in the quality parameters of Partial Least Squares modeling. Calibration and validation models were evaluated by means of correlation coefficient, Rank, robustness and Residual Prediction Deviation. All the preprocessing available on the software Opus Lab® were tested and compared. 72 readings/8 samples of refrigerated grass carp burgers originated the data regarding its water activity, rancid taste, pH and reactive substances of thiobarbituric acid results. The preprocessing methods accessible were Standard Normal Variate, Multiplicative Scatter Correction, 2nd derivative, 1st derivative, Straight Line Subtraction and Min/Max. Each chosen preprocessing generated a model with different parameters. The homogeneity of data proved to have a direct influence on the robustness, confirming the challenge to fit sensory results in Partial Least Squares prediction models. New possibilities to investigate meat products were shown.

Keywords: grass carp; hamburgers; NIR; PLS; predictive models; statistics

1. Introduction

Spectroscopy in the infrared region has gained notoriety as an analytical technique [1] for over a decade. This technique has already been approved by the Association of Analytical Communities (AOAC) as a technique for the analysis of moisture, fat, and protein in meat and meat products [2]. In recent years, the meat industry has been showing an increasing demand for fast quality control methods to keep up with the industrial pace. While physicochemical and sensory analyzes are usually time-consuming, the Near Infrared spectrometry (NIR) is fast and non-destructive. This technique requires a small amount of sample and, in addition, it is of high precision, accessing several properties of the product simultaneously, and also correlates the physicochemical characteristics with the sensorial ones through the statistical analysis of linear regression [2–9].

The development of a mathematical model in NIR, which relates the sensory and physicochemical information, could be used as a rapid method of quality analysis [10] which would optimize the quality assurance process. The NIR consists in the analysis of samples to provide analytical information directly after the reading, through absorption or emission of radiation, due to the variation of molecular energy associated to the vibrational transitions, by the absorption or emission of a photon, without electronic change [1]. Coupled to a computer and software there is scope for rapid decision-making on the quality of the sample if there is a calibrated and validated

*Corresponding author: E-mail: caroolmarques@gmail.com

model of the available product. However, due to the distance of the equipment from the production line, statistical misunderstandings, and some sampling errors, the NIR has been used only as a complement to the other analyzes [10]. Mathematical models that predict food quality, for example, are important to overcome the limitations found in the analyzes, such as time and apparatus sensitivity [11].

A multivariate calibration method commonly used for NIR data is the Partial Least Squares (PLS) regression. Within this analysis it is possible to choose parameters, including preprocessing (PS) of the data. However, caution is recommended when randomly selecting spectral regions, it is significant, but one can eliminate the regions that are related to the expected effect, and that only aid in multivariate calibration via PLS [12–14].

Choosing a suitable PS method can be a challenging task. The PS can remove noise, outliers, and unwanted spectral variation, improving the predictive model. The most used PS are 1st and 2nd derivatives (1st DER and 2nd DER), multiplicative scatter correction (MSC) and standard normal variate (SNV), in order to obtain the best data treatment [14, 15]. The literature discussing data distribution and its influence on NIR are scarce, Vukovinsky and Pfahler [16] mention the importance of data normality, not only its assumption, highlighting that outliers could arise when non-normal data are analyzed by NIR, and therefore the model would predict a change not accounted, thus it would not predict well. Data distribution is essential, and the distribution analyses are most assessed as a prior step, while Chen et al. [17] evaluated spectra wavelength distributions and both ways demonstrate the importance of normality for NIR.

Given this information, the objective of this study is the estimation of the data PS and homogeneity influence (normality by means of Kolmogorov–Smirnov) in the quality parameters of PLS modeling. The product analyzed is the grass carp burger, and the sensory parameter of interest measured in the present study is the rancid flavor, followed by physicochemical properties, water activity (a_w), content of thiobarbituric acid (TBARS) and pH.

2. Results and Discussion

To encourage fish consumption, fish burgers are a good strategy to increase the stability of the meat and practicability of preparation [18,19] and one of the main parameters of its quality is the lipid oxidation [20, 21], easily accessed through sensory analysis and TBARS [22]. The reference analysis for NIR were pH, TBARS, rancid flavor and water activity (a_w), during the 30 days of storage of the fish burger. The PLS models were validated through the test set and cross-validation, testing the best PS of data for each attribute analyzed, even though it is possible to choose no PS.

Regarding the pH analysis, with no PS, the best fit was the 1st DER + straight line subtraction SLS, correlation coefficient (R^2) of 95.12%, ratio of performance to deviation (RPD) 4.79 and Rank 7 (Table 1) in the test set and 84.97% of R^2 for calibration. The 1st DER PS calculates the DER of the spectrum, leaving the more pronounced signs in emphasis instead of the flatter bands, increasing the homogeneity of the signals [9, 23]. It is one of the best methods for removing baseline effects, highly used as pre-treatment of NIR data [15].

The R^2 represents the percentage of variability that was reproduced by the predicted model. Above 90% the R^2 is considered suitable for solids analysis in the NIR [23] and the R^2 of 80% is considered a limit of reliability when it comes to R^2 [10]. In addition, the Rank is a value from 0 to 10, representing the factors used by PLS. The lowest Rank values represent major changes in the spectrum, and values close to 10 express small spectral changes and may include noise from spectral lines. Ranks near to 5 are considered ideal for PLS models because it is centralized, capturing the most important changes [23, 24]. The pH best model achieved a worthy R^2, and the Rank 7 can be already affected by noise.

No PS improved the data correlation. The root mean square errors of prediction (RMSEP)/ root mean square errors of cross-validation (RMSECV) ratio was 0.92, indicating a robust model. Values closer to one (1.0), indicate greater robustness of the model [25]. The predictive quality of the model is also proven by the cross-validation error, RMSECV (0.0936), the lower the value, the better the correlation between the raw data and the predicted by the model [23]. This model with these characteristics can be considered accurate.

The RPD is the quotient of the standard deviation between true and predicted values of the model and is directly related to the variability explained by R^2. This value demonstrated in this case that the pH model had an adequate distribution. RPD values greater than 3.0 are considered good, greater than 8.0 are excellent and bellow 3.0 they are not trustworthy for quality control [23]. Given this fact, the model found for the pH showed aptitude for quality control of grass carp burgers with RDP near to 5.0. Data distribution is normal when analyzed by the Kolmogorov-Smirnov test (p> 0.20), corroborating the accuracy of the model.

Table 1. Best pH NIR results with and without PS.

Calibration			Cross-validation			Test Set validation		
R^2 (%)	RMSEC	RPD	R^2 (%)	RMSECV	RPD	R^2 (%)	RMSEP	RPD
			NO PS = [1st DER] + SLS – Rank 7					
84.97	*0.168*	*2.58*	*48.17*	*0.0936*	*1.39*	*95.12*	*0.0869*	*4.79*
			PS [SNV] + SLS – Rank 7					
83.35	0.176	2.45	42.91	0.299	1.32	77.16	0.187	2.09
			PS [MIN/MAX] + SLS – Rank 8					
83.61	0.177	2.47	24.89	0.344	1.15	90.64	0.12	3.30
			PS [1st DER] + 1st DER + MSC – Rank 5					
77.20	0.264	2.09	34.34	0.325	1.23	86.59	0.143	2.82
			PS [2nd DER] +1st DER – Rank 8					
87.18	0.158	2.79	17.18	0.365	1.12	72.05	0.207	2.01

Considering the best TBARS PLS model without any PS, the chosen correction was 1st DER + MSC. The R^2 found is 94.40%, RPD 4.34 and Rank 3 in the Test Set in addition of 55.49% of R^2 for calibration.

In an attempt to improve the R^2, all PS were applied and the best models are reported in Table 2. The PS SNV + 1st DER + MSC with Rank 2 improved the correlation and the RPD to 5.71, which demonstrates the model's ability to quality control. The calibration results did not present a high R^2 value and it can be attributed to the input data made of less than hundred readings. Data distribution was normal when analyzed by the Kolmogorov-Smirnov test (p> 0.20).

Table 2. Best PS NIR results applied to TBARS data compared to non-PS results.

Calibration			Cross-validation			Test Set validation		
R^2 (%)	RMSEC	RPD	R^2 (%)	RMSECV	RPD	R^2 (%)	RMSEP	RPD
			NO PS = [1st DER] + MSC – Rank 3					
55.49	0.158	1.5	35.33	0.182	1.24	94.40	0.0484	4.34
			PS [COE] + 2nd DER – Rank 2					
71.78	0.177	1.88	58.73	0.136	1.56	59.84	0.132	1.60
			PS [SNV or MSC] + 1st DER + MSC e PS[1st DER] + MSC – Rank 2*					
54.13	*0.157*	*1.48*	*28.5*	*0.062*	*1.18*	*94.70**	*0.0482*	*5.71*
			PS [2nd DER] + 1st DER + MSC – Rank 6					
57.11	0.158	1.53	18.53	0.203	1.11	90.36	0.0649	3.30

* PS that has improved PLS model performance.

The ratio RMSEP/RMSECV calculated was 0.77, indicating a model with intermediate robustness. A low value of RMSECV (0.062) confirms the predictive quality of the model. The value of 0.15 found for root mean square errors of calibration (RMSEC) corroborates results of [6] for FT-NIR with ground beef, which established 0.10 for this same parameter and 0.12 for RMSECV. Researchers [6] found that the 2nd DER PS better for TBARS data. The 2nd DER as the 1st DER are very effective to remove baseline offset and the 2nd DER additionally removes linear trend from a spectrum, polishing the data [15].

Combining the 1st DER with MSC and SNV, the base line offset was removed, multiplicative and/or additive scatter effects were compensated and a range scaling enable the dissimilar intensities of the samples to be compared. SNV centers and scales each individual spectrum [15,26]. Therefore, despite the low correlation in calibrations results, the normality of the data propitiated a good validation model, after the SNV PS addition to the 1st DER + MSC. SNV application also decreased the Rank to 2, what indicates only major changes being considered by the model, and perhaps some characteristic was ignored.

Regarding the results of rancid flavor, the 2nd DER without PS was the best fit, with 94.11% of R^2, RPD of 4.21 and Rank 5 (Figure 9) in the Test Set; and R^2 of 53.42% for calibration. All PSs were applied and the best ones are reported in Table 3, but none improved the correlation. The calibration results did not present a high R^2 value and it can be accredited to the input data made of less than hundred readings. We suggest a wider input data, containing more readings, to further investigations.

For both the TBARS and rancid taste models, the calibration involving 70% of the responses resulted in a relatively low R^2, which may have occurred because there was a high frequency of near zero values in the first days of analyses and a rapid increase in the values with subsequent stabilization. This fact leads to a greater accumulation of data at the beginning of the regression line, which decreased the R^2 for calibration.

Table 3. Best PS NIR results applied to rancid flavor data compared to non-PS results.

Calibration			**Cross-validation**			**Test Set validation**		
R^2 (%)	RMSEC	RPD	R^2 (%)	RMSEC	RPD	R^2 (%)	RMSEP	RPD
				NO PS = [2nd DER] – Rank 5				
53.42	*1.98*	*1.47*	*13.63*	*0.92*	*0.938*	*94.11*	*0.541*	*4.21*
				PS [SNV] + 2nd DER – Rank 5				
54.99	1.95	1.49	14.56	2.92	0.936	86.65	0.573	2.76
				PS [1st DER] + 2nd DER – Rank 7				
61.93	1.84	1.62	26.33	3.09	0.892	76.55	1.06	2.07
				PS [2nd DER] + 1st DER + SLS – Rank 7				
61.88	1.84	1.62	25.06	3.07	0.897	76.93	1.05	2.08
				PS [MSC] +1st DER– Rank 8				
61.86	1.86	1.62	85.17	3.74	0.736	73.59	0.824	1.95

The Kolmogorov-Smirnov normality test ($p < 0.05$), as shown by the histogram of Figure 1, did not confirmed normal distribution, due to the high frequency of values close to zero at the beginning of the analyses, that contributed to this fact. Data from sensory analysis assessing trained panels rarely demonstrate normal distribution, since the perception presents itself in a very homogeneous manner, without much variation.

Despite the RPD of 4.21 showing the quality of the model, and the Rank centralized (5) the best as possible, the ratio RMSEP/RMSECV of 0.58 indicates weak robustness. The predictive quality analyzed by the RMSECV obtained a higher value than the other models tested, of 0.92. Figure 1 represents the difference between a normal data distribution, and the non-normality. The calibration with all the values, the 72 readings, shows the influence of data agglomeration in certain points of the plot as the main reason for not obtaining robust models. Not even the PS 2nd DER, whose effects were mentioned above, or any other PS tested could improve robustness.

The a_w as the rancid flavor did not present normal distribution ($p < 0.01$). The elevate amount of similar values did not allow a suitable distribution. Table 4 shows the best results of all the PS attempts for a_w: 1st and 2nd DER, MIN/MAX and SNV. Four models of PLS were elaborated for a_w, in addition to the model generated without PS, which found a better fit in the SLS, with R^2 correlation of 70.02%, RPD of 1.92 and Rank 8 in the Test Set (external validation); besides a R^2 of 90.56% in the calibration.

Intermediate models were also generated when MIN/MAX and the 2nd DER was applied as PS. The 1st DER propitiated the model with the lowest R^2, 63.31%, and was therefore not suitable for the construction of predictive models for aw in grass carp burgers. The model with the PS MIN/MAX + SLS presented a R^2 of 85.06% for calibration, 73.09% for external validation and Rank 6, which was determined as the best model for predicting a_w of the fish burgers.

The PS improved the correlation of the validation data, so the model chosen for a_w was the one with PS [MIN / MAX] + SLS. The R^2 improved, and the Rank became more centralized (value of 6). In addition to the RPD for the calibration model, placed above 2, considered an adequate value [27] but not suitable for quality control. The ratio (RMSEP / RMSECV) of the PLS model for a_w was 0.65, not as adequate as pH and TBARS. The high frequency of similar responses to a_w contributed to low determination coefficients of the PLS modeling.

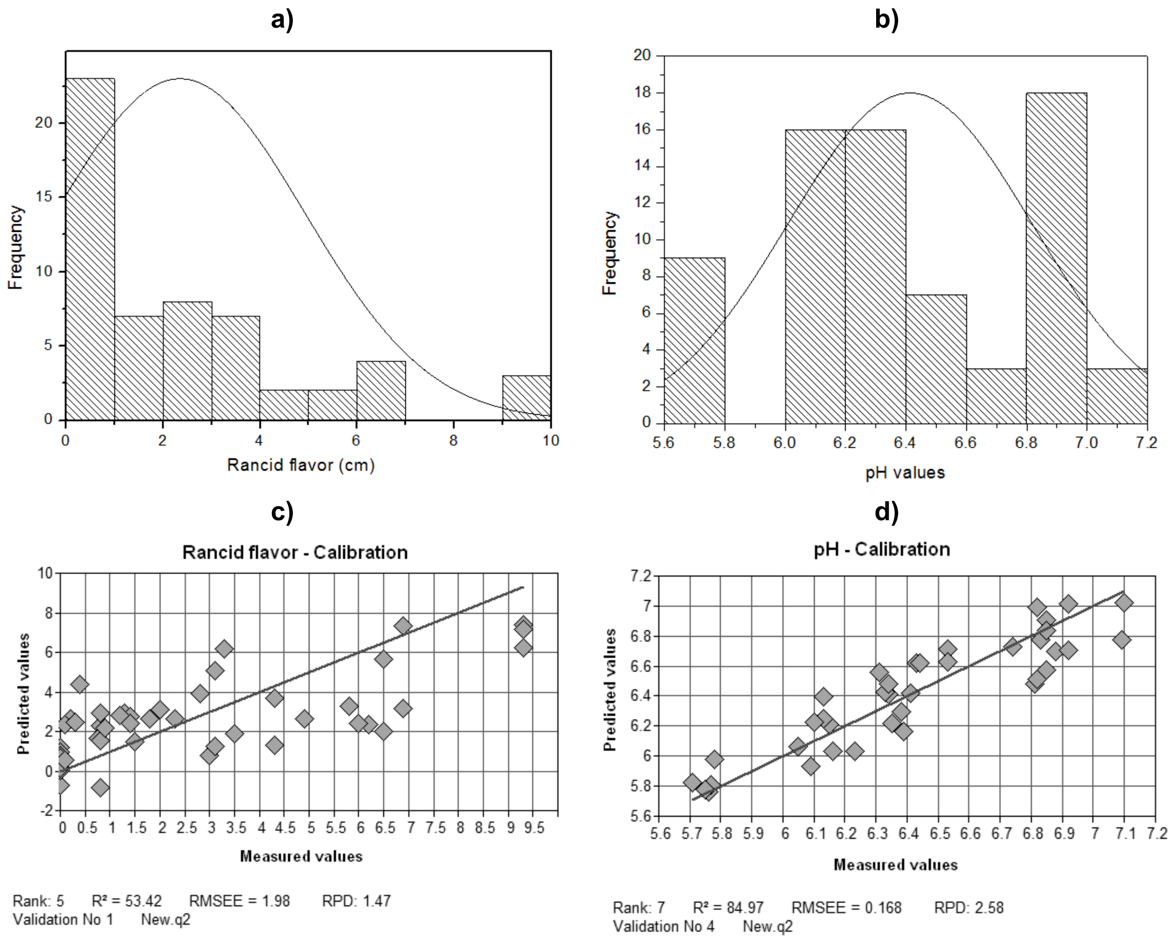

Figure 1. Histogram of the rancid flavor (a); the pH (b); scatterplots of the measured values versus the predicted ones from the rancid flavor (c) and the pH (d).

Table 4. Best PS NIR results applied to a_w data compared to non-PS results.

Calibration			Cross-validation			Test Set validation		
R² (%)	RMSEC	RPD	R² (%)	RMSEC	RPD	R² (%)	RMSEC	RPD
			NO PS = [SLS] – Rank 8					
90.56	0.00099	3.25	59.50	0.00189	1.59	70.02	0.00146	1.92
			PS [SNV] + *NO PS* – Rank 9					
93.71	0.00083	3.99	68.77	0.00167	1.79	72.96	0.00142	1.99
			PS [MIN/MAX] + SLS – Rank 6*					
85.06	*0.00123*	*2.59*	*45.06*	*0.0022*	*1.36*	*73.09**	*0.00142*	*1.93*
			PS [1st DER] + MIN/MAX – Rank 5					
66.22	0.00183	1.72	12.73	0.00277	1.07	63.31	0.00165	1.66
			PS [2nd DER] + SLS – Rank 6					
81.42	0.00138	2.32	37.2	0.00236	1.27	68.94	0.00152	2.00

* PS that has improved PLS model performance.

PS the data not always improve the correlations comparing with the default processing made by the software, although sometimes it helps only one of the three sets (calibration, cross-validation or test set), as we can see with the aw (Figure 2). Test set validation improved 3% and the outlier value is not considered as that anymore. Analyzing the data from rancid flavor and a_w, the non-normality hinders the PS action, what means that agglomerated values not always can be improved do fit in linear predictions. Despite the other parameters considered, the R^2 of cross-validation from the best fitting for rancid taste, is low (13.63) and only one PS improved that (PS [MSC] +1st DER), to a value of 85%, however, the test set R^2 decreased 20%. All these facts clarify that all PS methods must be tested over the data, in order to try any significant improvement that could enrich the predictive model.

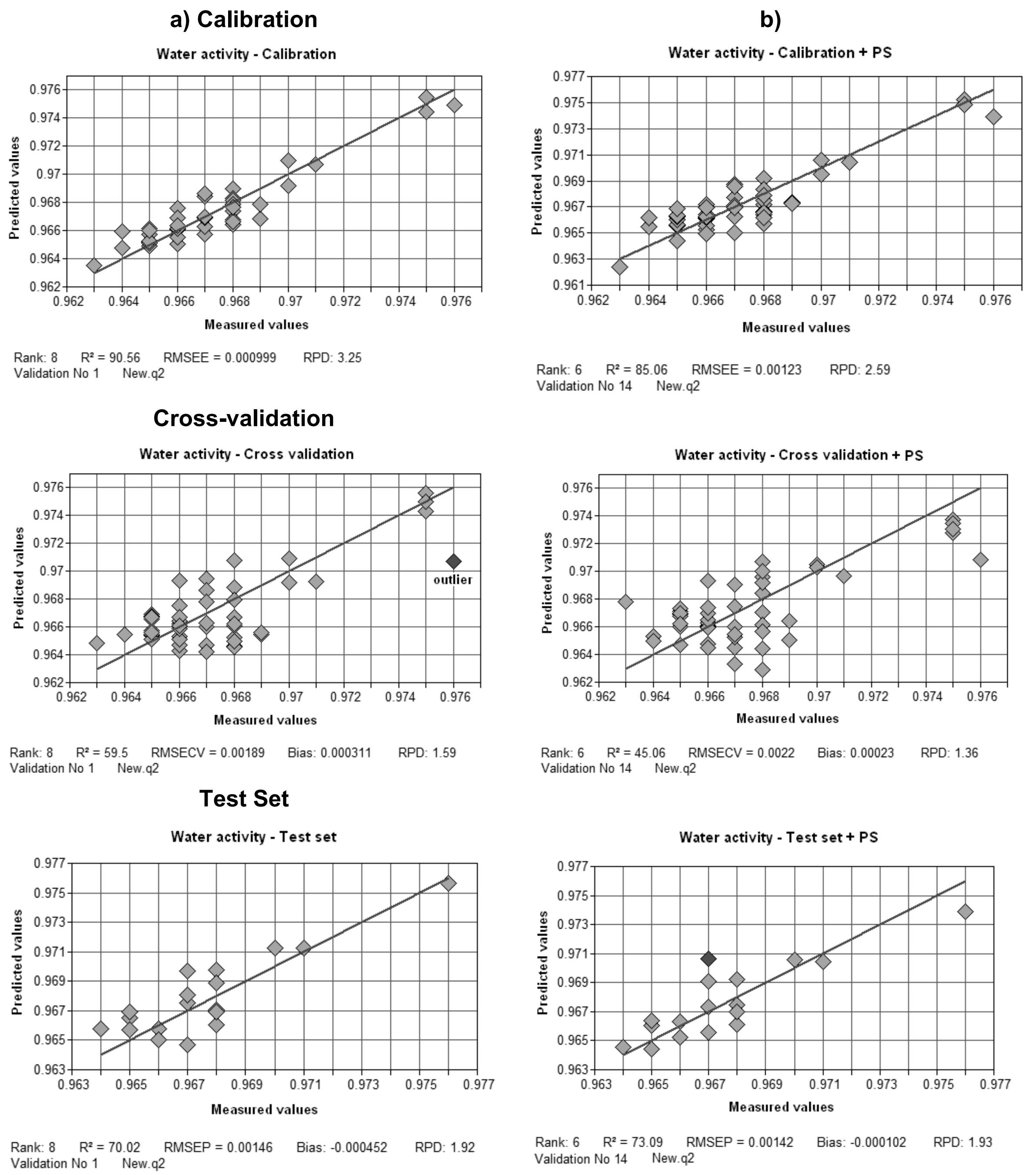

Figure 2. PS effect on aw fitting; regarding calibration, cross-validation and test set for NO PS (a) and the best PS process (b).

Chemometric analyses of NIR spectral data combined with sensory parameters represent a relatively recent procedure. Prieto et al. and Rødbotten et al. [2, 8] found a low correlation

(0.34-0.51%) between the sensory data for meat tenderness and the NIR spectra. Andrès et al. [10] also found a low correlation (0.13-0.38%) between NIR and the sensory analysis for mutton, but suggested that perhaps a more specific group of sensory characteristics could be applied, or that the samples be selected between the best and the worst results, allowing for greater correlations to be achieved. Liu [28] reported 0.50 and 0.58% of R^2 for chewiness and juiciness parameters, not adequate values for reliable models. Ripoll [29] obtained low correlations between NIR and the succulence and overall quality of steaks, and a good fit (R^2 = 0.97%) for softness.

However, good models and correlations have been found between NIR spectral data and the physical and chemical parameters of foods, such as the robust models found by way of TBARS and pH in the present study. Studies have reported that the intramuscular fat content showed a good correlation with the NIR spectral data of mutton [10], the beef pH value [4], the TBARS of ground meat [6], and the moisture content of beef [29], all with $R^2 > 0.84\%$.

As for the sensory parameters predicted by the NIR spectral data, [30, 31] obtained good calibration and validation models for the sensory attributes of coffee (acidity, body, overall quality, etc.), indicating the possibility of estimating the sensory results and monitoring their quality by NIR.

However, they did not follow through the shelf life to determine if NIR detected instrumentally the differences obtained in the sensory evaluation on the same scale. In addition, the models obtained and used by [31] provided Rank considered high (from 7 to 9) and errors not as low as expected, and [30] did not carry out an external validation.

When a person is applied as a measurement parameter, there are numerous differences in the results when compared to instrumental and physicochemical analyses. Each assessor has a perception threshold. Nevertheless, as Weber's, Fechner, and Stevens's laws of psychophysics suggest, when the stimulus is weak and near the threshold, the sensation and discrimination are also feeble [32]. This behavior was verified by the emergence of several zeros on the rancidity scale for the fresh fish burgers and after seven days of storage, compromising the generation of the PLS model, even with the exponential increase in rancidity shown in the sensory analysis and using TBARS.

In addition, the sensitivity of the apparatus may have only detected differences considered large ones, while the trained panel considered more subtle differences [10]. The solid relation found between NIR and sensory results is the rancid flavor and TBARS since they are connected to the burgers fat content and its changes during storage. Therefore, the general spectrum confirmed the detection of fatty acids, as it presented a peak in the overtone region, characteristic of carboxylic acids, present in fatty acids [23], exactly at 5250 cm^{-1} in the first overtone and 4900-4600 cm^{-1} in the combination band region. However, TBARS achieved a better model than rancid flavor, despite the obvious connection between them.

Besides the sensitivity of the apparatus, other factors have direct influence on NIR results. According to others researchers [33], the sampling presentation, statistical choices, the operator and environment (temperature, vibrations, light) can interfere on results. In the present study, all the efforts to control these factors were made. We kept the room temperature at 18°C, and employed the same previously trained operator for sampling, achieving a homogenous way of setting the samples inside the probe for solids, besides the careful analysis of the statistical possibilities as shown through the present work.

NIR calibration consists in a great amount of work, with many samples applied to achieve robust models. Nevertheless, after the calibration and validation, one can only analyze new samples and predict the results with the model. Further studies with grass carp burgers should use more samples (more than 100) to gather perhaps better R^2, RPD values and calibration models adequate for quality control. Another suggestion to future studies is the attempt to fit other sensory parameters into PLS regression, as the current literature and research still does not show solid results on this matter, especially none in fish burgers.

3. Material and Methods

Raw material and hamburger manufacture

The grass carp fishes were purchased from a project partnership with a local fish farmer. After the capture, the fish were percussive stunned in the head, slaughtered (marrow section followed by bleeding), peeled, gutted and filleted for transport (in Styrofoam boxes with ice). The burgers were prepared in the Food Technology Laboratory at UTFPR (N008) according to the formulation and procedure described by [34].

Physicochemical and sensory analysis

The pH of the burgers was measured in 25g of hamburger and 5mL of water in the benchtop equipment (TECNAL®) with the sensor coupled to a digital meter. The determination of TBARS followed [35] and the calculation was based on a malonaldehyde standard curve. The standard applied was the 1,1,3,3-Tetramethoxypropane 99%, Sigma-Aldrich®. The a_w was measured directly on the AquaLab instrument (Tecnal®), with a default of water 1.00. The physicochemical results are not shown at the present work. The sensory procedures are described by [34].

NIR evaluation and data processing

The NIR spectrum was collected by the spectrophotometer (FT-NIR MPA, Bruker Optics®, Ettlingen, Germany) in room temperature (25 ±1°C), 12500 to 3600 cm^{-1} of scanning and 16 cm^{-1} of resolution. On each day of analysis, nine burgers separated for sampling were read, resulting in 72 different samples for NIR reading. The predictive model considered firstly the Cross-validation (internal) - with 70% of the calibration samples (excluding one sample every 50 to ensure that all are validated) and the Test set (external) with the 30 % of the remaining samples. A random selection separated the samples.

The NIR OPUS Lab® 7.2 software calculated the PLS multivariate models. A calibration and a validation model were obtained for each chosen reference analysis, with or without PS. The Statistica® software 12.7 processed the data and graphically plotted the histograms. Kolmogorov-Smirnov (KS) determined the data distribution through the same software.

Selected merit figures assessed the accuracy of the models, giving basis for comparison. They were the R^2, RPD, RMSEC, RMSEP, and RMSECV. In addition, the ratio between RMSEP and RMSECV (RMSEP/RMSECV) was acquired to evaluate the robustness of the models [14,25]. All the results were placed in Tables for better understanding and visualization.

4. Conclusions

- Each chosen PS generated a model with different parameters. PS not always improve the quality parameters of the models, as accounted for pH and rancid flavor.
- pH proved to be the best data set, considering all the accuracy factors. RMSEP/RMSECV ratio (0.92), RPD suitable for quality control, R^2 (95%), corroborating the normality contribution.
- SNV improved validation correlation but decreased Rank, what could indicate some small characteristic being ignored in the TBARS model. Combining SNV with 1st DER and MSC, the RPD achieved potential for quality control.
- The homogeneity of data proved to have a direct influence on the robustness, confirming the challenge to fit sensory results as rancid flavor of fish burgers in PLS prediction models.
- Normality, the data distribution, affects directly on R^2 of the calibration models. Despite the reliable R^2 of the validation models, they consider only 30% of the data, and the high content of zeros or similar values in the distribution, influences all the PS regarding calibration, while validation results could make it seem an accurate model.
- In our study, we found good as not so suitable models to describe the pattern of fish hamburgers. We show in this work analyzes that can be performed on meat products.

Acknowledgments

This work was supported by the National Council for Scientific and Technological

Development – CNPq – Brazil (Universal Process nº 456102/2014-0). The authors thank The Federal University of Technology – Pato Branco (UTFPR), acknowledgements for the technical support provided.

References and Notes

[1] Lima, K. M. G.; Raimundo Jr. I. M.; Silva, A. M. S.; Pimentel, M. F. *Quim. Nova* **2009**, *32*, 1635. [Crossref]

[2] Prieto, N.; Roehe, R.; Lavín, P.; Batten, G.; Andrés, S. *Meat Science* **2009**, *83*, 175. [Crossref]

[3] Liu, Y.; Lyon, B. G.; Windham, W. R.; Lyon, C. E.; Savage, E. M. *Poult. Sci.* **2004**, *83*, 1467. [Crossref]

[4] Andrés, S.; Silva, A.; Soares-Pereira, A. L.; Martins, M.; Bruno-Soares, A. M.; Murray, I. *Meat Science* **2008**, *78*, 217. [Crossref]

[5] Pla, M.; Hernández, P.; Ariño, B.; Ramírez, J. A.; Díaz, I. *Food Chem.* **2007**, *100*, 165. [Crossref]

[6] Sinelli, N.; Limbo, S.; Torri, L.; Di Egidio, V.;'Casiraghi, E. *Meat Science* **2010**, *86*, 748. [Crossref]

[7] Rossato, R.; Prete, C. E. C.; De Castro, C.; Tomm, G. O.; Leite, R. S.; Mandarino, J. M. G.; De Araújo, P. M.; De Carvalho, C. G. P. *Pesqui. Agropecu. Bras.* **2013**, *48*, 1601. [Crossref]

[8] Rødbotten, R.; Nilsen, B. N.; Hildrum, K. I. *Food Chem.* **2000**, *69*, 427. [Crossref]

[9] Mazur, L.; De Oliveira, G. A.; Bicudo, M. O. P.; Ribani, R. H.; Nagata, N.; Peralta-Zamora, P. *Acta Sci., Technol.* **2014**, *36*, 369. [Crossref]

[10] Andrés, S.; Murray, I.; Navajas, E.; Fisher, V.; Lambe, N. R.; Bünger, L. *Meat Science* **2007**, *76*, 509. [Crossref]

[11] Koutsoumanis, K. *Appl. Environ. Microbiol.* 2001, 67, 1821. [Crossref]

[12] Chen, Q.; Zhao, J.; Liu, M.; Cai, J.; Liu, J. *J. Pharm. Biomed. Anal.* **2008**, *46*, 568. [Crossref]

[13] Cai, J.; Chen, Q.; Wan, X.; Zhao, J. *Food Chem.* **2011**, *126*, 1354. [Crossref]

[14] Skibsted, E. T. S.; Boelens, H. F. M.; Westerhuis, J. A.; Witte, D. T.; Smilde, A. K. *Appl. Spectrosc.* **2004**, *58*, 264. [Crossref]

[15] Gholizadeh, A.; Borůvka, L.; Saberioon, M. M.; Kozák, J.; Vašát, R.; Němeček, K. *Soil Water Res.* **2016**, *10*, 218. [Crossref]

[16] Vukovinsky, K. E.; Pfahler, L. B. *Pharmaceutical Technology.* 2014, *38.* Available: http://www.pharmtech.com/role-normal-data-distribution-pharmaceutical-development-and-manufacturing?pageID=2 (accessed August 19, 2018).

[17] Chen, Y.; Liu, Y.; Fei, T.; Jiang, Q.; Wang, J.; Shi, T. Statistical Understanding on the Pre-processing of VNIR Spectra Data from Soil Samples with Different Preparations, in: (ISSMTA2013), Fen Hu, Nanjing, Jiangsu, China, 2013: pp. 32–37. Available: http://www.isprs.org/proceedings/2013/ISSMTA2013/06.pdf (accessed August 19, 2018).

[18] Corbo, M. R.; Speranza, B.; Filippone, A.; Granatiero, S.; Conte, A.; Sinigaglia, M. Del Nobile, M. A. *Int. J. Food Microbiol.* **2008**, *127*, 261. [Crossref]

[19] Del Nobile, M. A.; Corbo, M. R.; Speranza, B.; Sinigaglia, M.; Conte, A.; Caroprese, M. *Int. J. Food Microbiol.* **2009**, *135*, 281. [Crossref]

[20] Soares, K. M. P.; Gonçalves, A. A. *Rev. Inst. Adolfo Lutz* **2012**, *71*, 1.

[21] Wu, T.; Mao, L. *Food Chem.* **2008**, *110*, 647. [Crossref]

[22] Campo, M. M.; Nute, G. R.; Hughes, S. I.; Enser, M.; Wood, J. D.; Richardson, R. I. *Meat Science* **2006**, *72*, 303. [Crossref]

[23] Conzen, J. P. Multivariate Calibration: A practical guide for developing methods in the quantitative analytical chemistry, Ettlingen: Bruker Optick GmbH, 2006.

[24] Ferreira, M. M. C. Quimiometria: conceitos, métodos e aplicações, Editora da Unicamp, 2015. [Crossref]

[25] Páscoa, R. N. M. J.; Magalhães, L. M.; Lopes, J. A. *Food Res. Int.* 2013, 51, 579. [Crossref]

[26] Verboven, S.; Hubert, M.; Goos, P. *J. Chemom.* **2012**, *26*, 282. [Crossref]

[27] Dal Zotto, R.; De Marchi, M.; Cecchinato, A.; Penasa, M.; Cassandro, M.; Carnier, P.; Gallo, L.; Bittante, G. *J. Dairy Sci.* **2008**, *91*, 4103. [Crossref]

[28] Liu, Y.; Lyon, B. G.; Windham, W. R.; Realini, C. E.; Pringle, T. D. D.; Duckett, S. *Meat Science* **2003**, *65*, 1107. [Crossref]

[29] Ripoll, G.; Albertón, P.; Panea, B.; Olleta, J. L.; Saludo, C. *Meat Science* **2008**, *80*, 697. [Crossref]

[30] Esteban-Díez, I.; González-Sáiz, J. M.; Pizarro, C. *Anal. Chim. Acta* 2004, 525, 171. [Crossref]

[31] Ribeiro, J. S.; Ferreira, M. M. C.; Salva, T. J. G. Talanta 2011, 83, 1352. [Crossref]

[32] Queiroz, M. I.; Treptow, R. O. Análise sensorial para avaliação da qualidade dos alimentos, Editora da FURG, Rio Grande, 2006.

[33] Huang, J.; Romero-Torres, S.; Moshgbar, M. Practical Considerations in Data Pre-treatment for NIR and Raman Spectroscopy, American Pharmaceutical Review. 2010. Available: https://www.americanpharmaceuticalreview.com/Featured-Articles/116330-Practical-Considerations-in-Data-Pre-treatment-for-NIR-and-Raman-Spectroscopy/ (accessed August 16, 2018).

[34] Marques, C.; Dos Reis, A. S.; Da Silva, L. D.; Carpes, S. T.; Mitterer-Daltoé, M. L. *Grasas Aceites* **2017**, *68*, 1. [Crossref]

[35] AOAC - Association Of Official Analytical Chemistry, Official Methods of Analysis, 17th ed., AOAC, Washington, D.C. USA, 2000.