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Watanabe, Lycio Shinji; Bovolenta, Yuri Renan; Junior, Vinicius Ricardo Acquaro; Barbin, Douglas Fernandes; Madeira, Tiago Bervelieri; Nixdorf, Suzana Lucy

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Investigação de métodos de pré-processamento em espectros NIR combinados com regressão multivariada para determinação de umidade em ovo em pó industrial

Lycio Shinji Watanabe

Universidade Estadual de Londrina, Brasil

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Yuri Renan Bovolenta

Universidade Estadual de Londrina, Brasil

Vinicio Ricardo Acquaro Junior

Universidade Estadual de Londrina, Brasil

Douglas Fernandes Barbin

Universidade Estadual de Campinas, Brasil

Tiago Bervelieri Madeira

Universidade Estadual de Londrina, Brasil

Suzana Lucy Nixdorf

Universidade Estadual de Londrina, Brasil

snixdorf@uel.br

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ABSTRACT:

High productivity and meantime perishability of *in natura* eggs, make powdered egg attractive for patisseries and pasta industries. Water reduction in 65%, extends shelf life from 1 to 12 months, preventing also *Salmonella*. Maximum powdered egg moisture allowed by Brazilian law is 6.0% (w w⁻¹). However, its determination by reference technique (oven at 105°C for 8 hour) is lengthy for processing industry. Therefore, the purpose of this study was to investigate the influence of several spectral pre-processing techniques in the application of near-infrared spectroscopy associated with chemometrics models for determination of moisture content in powdered egg, without the need of sample preparation and destruction, held at 0.5 min. Several pre-treatment techniques were evaluated to ensure spectral data reliability such as: standard normal variation; multiplicative scatter correction; smoothing and detrend. The principal component regression (PCR) and partial least squares (PLS) were evaluated with and without pre-treatment. The best results were observed in NIR/PLS model (49 samples), providing an adequate correlation (r) of 0.96, for cross-validation. Using 21 samples as prediction set, NIR/PLS showed relative error (RE) < 2.0%, compared to primary methods oven and thermobalance, indicating to be suitable for industrial quality control.

KEYWORDS: chemometrics, near-infrared spectroscopy (NIRS), thermobalance, oven reference method, calibration model, PLS.

RESUMO:

A alta produtividade e perecibilidade dos ovos *in natura* torna o ovo em pó atrativo para o uso em confeitorias e indústrias de massas. A redução em 65% de água estende a vida de prateleira do produto de 1 para 12 meses, prevenindo a *Salmonella*. Pela legislação brasileira, a umidade máxima permitida para o ovo desidratado é de 6% (m m⁻¹). Entretanto, sua determinação por técnica de referência (estufa a 105°C por 8h) demanda um tempo muito longo para seu uso em processo. Portanto, este

AUTHOR NOTES

snixdorf@uel.br

estudo teve como propósito avaliar a viabilidade da aplicação da espectroscopia do infravermelho próximo, associada a modelos quimiométricos na determinação da umidade do ovo em pó, feita em 0,5 min., sem a necessidade do preparo e nem destruição de amostra. Pré-tratamentos foram avaliados com intuito de aumentar a confiabilidade dos dados espectrais do modelo de calibração: variação de padrão normal; correção multiplicativa de sinal; e alisamentos. Regressões por componentes principais (PCR) e por mínimos quadrados parciais (PLS) foram avaliadas, juntamente com os pré-tratamentos. Os melhores resultados foram observados na modelagem PLS (49 espectros), apresentando um coeficiente de correlação (r) de 0,96 para a validação cruzada. O conjunto preditor (21 amostras) mostrou um erro relativo (ER) < 2,0% para o modelo NIR/PLS, comparado à estufa e à termobalança, mostrando-se adequado para o controle de qualidade industrial.

PALAVRAS-CHAVE: quimiometria, espectroscopia no infravermelho-próximo (NIR), termobalança, estufa - método de referência, modelo de calibração, PLS.

INTRODUCTION

Egg is composed of countless nutrients. It is rich in proteins and has been for a long time subject of nutritional studies involving amino acids (Tedesco, Barrado, Sanz, Tedesco, & De La Rose, 2006). However, water is the major component of chicken egg, making up for about 70% of its composition (Roe, Pinchen, Church, & Finglas, 2013). For this reason, if egg is not stored in appropriate conditions, osmotic changes may occur through its porous shell, causing contamination by microorganisms such as *Salmonella* (Barancelli, Martin, & Porto, 2012).

According to the *Instituto Brasileiro de Geografia e Estatística* (IBGE), Brazil produced 760885 dozen of chicken eggs in the 2nd quarter of 2016, increasing the production to 816103 in the 2nd quarter of 2017, an increase of 7.3% (*Instituto Brasileiro de Geografia e Estatística* [IBGE], 2017). Because of the high productivity and meantime high perishability of chicken eggs, industry seeks processing methods aiming at growth in production and exports, considering as an advantage the fact that the powdered egg has its shelf life increased when compared to fresh egg.

Processed egg is the final product of the whole fresh egg that undergoes some physical-chemical transformations, without changes in its protein source. In particular, powdered egg has been gaining importance in bakery and pasta processing industries due to its convenience of use and also for food safety (Baron et al., 2004). The high marketing price of powdered egg is balanced by transport losses reduction and shelf life increase. Powdered egg shelf life ranges from 6 to 12 months, while fresh egg shelf life is about 30 days if properly stored (Santos et al., 2009). According to Article 753 of the Rules of Industrial and Sanitary Inspection of Products of Animal Origin (RIISPOA), the maximum amount of moisture allowed in dehydrated egg is 6.0% by weight, reducing the probability of microbial growth and improving its quality (Brasil, 1952).

Moisture content of powdered egg can be determined by gravimetric difference after oven-drying at 105°C (Association of Official Analytical Chemists [AOAC], 2016), or by moisture analyzer (thermobalance). The last one is not considered an analytical reference method, but it is faster and more convenient compared to oven-drying. Traditionally, spectroscopic methods in the near infrared (NIR) range have been used as a fast, practical and non-destructive alternative method for moisture analysis and other compounds in pharmaceutical and food processing industries (Pasquini, 2003, Nagarajan, Singh, & Mehrotra, 2006).

This technique is growing as a processing analytical technology (PAT), because it brings some advantages, such as the possibility of spectral data acquisition for solid and liquid samples (with minimum or no sample pre-treatment); it provides physicochemical information of the sample (such as viscosity, water content, polymorphism); it predicts and determines multiple parameters through a single spectrum (Buckton, Yonemochi, Hammond, & Moffat, 1998, Blanco & Alacalá, 2006, Reh, Gerber, Prodolliet, & Vuataz, 2006, Nagarajan et al., 2006). This spectroscopic technique was associated with chemometrics, which is based on mathematical and statistical techniques to extract fundamental analytical data from analyzed samples (Pasquini, 2003).

This study aimed to assess the feasibility of using NIR spectroscopy in industrial quality control for powdered egg moisture, by several spectra pre-processing techniques and adjusted PLS calibration model, created from a primary data base determined by the moisture analyzer.

MATERIAL AND METHODS

Sample

Powdered egg samples were provided by an egg processing industry from Londrina region (Parana state, Brazil), totaling 70 samples stemmed from the same group, with different levels of moisture.

Reference method

The official reference method for food products moisture content determination is performed by oven-drying at 105°C until constant weight (AOAC, 2016). Results for determination of moisture content by oven-drying method were supplied by the egg processing industry. In addition, moisture was also determined using thermobalance (Ohaus MB45, Greifensee, Switzerland), with temperature adjusted to 105°C for 5 min. These analyses were carried out in triplicate, in the Laboratory of Food Science and Technology of the State University of Londrina (*Departamento de Ciências de Alimentos – DCTA/UEL*).

NIR spectroscopy

Sample spectra were collected using a spectrometer NIR (FOSS XDS™ Rapid Content Analyser, Hillerød, Denmark) from 400-2948 nm, with 2 nm interval. Each spectrum was collected in reflectance mode and converted to absorbance [$\log (1/R)$], as the average of 32 scans for each sample in 0.5 min. Forty nine (49) samples of whole powdered egg were analyzed for building of calibration models and twenty one (21) for validation. Samples (2.50 g) were placed inside the 'spinning', a quartz sample holder, pressed with a support to minimize the effect produced by sample particle size. These analyzes were performed at the Central of Multiuser Research Laboratories (*Central Multiusuária de Laboratórios de Pesquisa da UEL - CMLP*) at Laboratory to Support Agricultural Research at the State University of Londrina (*Laboratório de Apoio à Pesquisa Agropecuária - LAPA/UEL*).

Spectra pre-processing methods

In order to improve the efficiency of the multivariate models, spectral pre-processing techniques based on smoothing (moving average), detrend, multiplicative scattering correction (MSC) and standard normal variate (SNV) can be applied. Smoothing's techniques are mathematical tools that reduce the ratio signal/noise. Their main application can be represented by the least squares (Dardenne, Sinnaeve, & Baeten, 2000, Ramsay, Hooker, Campbell, & Cao, 2007). Detrend is applied to eliminate or minimize the effects of baseline and curvilinear displacement, using a polynomial model that the baseline is adjusted as a function of wavelength, showing the difference of each independent spectrum (Luypaert, Heuerding, De Jong, & Massart, 2002). MSCs can be applied, since the variations of the optical path or light scattering in the sample cause interference. This can be corrected compared to a reference spectrum obtained by the average spectrum of the samples (Naes, Isaksson, Fearn, & Davies, 2017). SNV is similar to MSC, but the reference is determined independently. Adjusted deviation for each sample spectrum is simply the ratio of the average of

all values for all variables, and the multiplicative setting is the standard deviation of the values of all variables. SNV improves the accuracy of prediction, but does not simplify and reduce systematic noise model (Naes et al., 2017).

Multivariate analysis methods

Sample spectra were recorded by using FOSS ISIscan software (version 3.5; Hillerød, Denmark). Quantitative NIR analysis models were developed using partial least squares regression (PLS) and principal component regression (PCR), which were done in the study herein by using FOSS Win ISI II software (version 1.5; Hillerød, Denmark), and their data compiled by Statistica software (Statsoft version 8.0, Tulsa, USA).

PLS modeling is a common technique because it relates to spectral information with reference data to obtain factors (or latent variables - LV). It is robust since the regression coefficients hardly change with the addition of new samples in the calibration set. This only strengthens the importance of reducing the experimental noise, linearity and non-linearities,

enabling the construction of models with a variable set larger than the number of samples. Typically, PLS provide suitable models with a smaller number of principal components compared to principal component regression (PCR) (Pasquini, 2003, Escandar, Damiani, Goicoechea, & Olivieri, 2006, Maluf, Pontarolo, Cordeiro, Nagata, & Peralta-Zamora, 2010).

PLS modeling removes information from the data set of spectral matrix (matrix X - independent values) to correlate them with the information taken from the reference set (Y matrix - dependent values). From linear combinations of the spectral data and reference data, the number of latent variables necessary to correlate the spectra and concentrations is calculated. These variables are used to construct the calibration model that offers the smallest differences among the reference values and the predicted values (Morgan, Faria, Ferrão, Bragagnolo, & Ferreira, 2008).

Validation parameters of the models

In order to create a calibration model, spectral information of 49 samples were randomly selected with their respective moisture content, analyzed by the reference technique. The remaining dataset composed of 21 samples were used for external validation. In internal validation, specifically cross-validation, part of the samples dataset is randomly selected for the calibration model, and the remaining samples are used for prediction. Alternatively, on external validation, 21 samples were selected and moisture content was predicted using the calibration model obtained (Ferreira, Antunes, Melgo, & Volpe, 1999).

With the purpose of assessing the efficiency of the models and calibration error, several parameters were calculated (Burns & Ciurczak, 2007):

a) Prediction residual error sum of squares (PRESS), represented by Equation 1:

$$PRESS = \sum_{i=1}^n (y_{exp} - y_{prev})^2 \quad (1)$$

where:

y_{exp} is the reference value and

y_{prev} the predicted value.

b) Root mean square error (RMSE), also called the root mean squared error of prediction (RMSEP), Equation 2:

$$RMSE = \sqrt{\frac{\sum_{i=1}^n (y_{exp} - y_{prev})^2}{n}} = \sqrt{\frac{PRESS}{n}} \quad (2)$$

where:

n is the number of samples analyzed.

c) Relative error percentage (RE) between the reference method and the spectral method (Skoog, West, Holler, & Crouch, 2014), Equation 3:

$$RE = \left[\frac{y_{exp} - y_{prev}}{y_{exp}} \right] 100\% \quad (3)$$

d) Correlation coefficient (r) between the estimated and experimental values of the reference method, Equation 4:

$$r = \sqrt{1 - \left(\frac{\sum_{i=1}^n (y_{exp} - y_{prev})^2}{\sum_{i=1}^n (y_{exp} - y_{med})^2} \right)} \quad (4)$$

where:

y_{med} is the mean of the experimental data.

Statistical comparison among the different methods for moisture content determination

Initially, the moisture content was measured by the reference method (oven) and then the thermobalance and the NIR/PLS model were used. ANOVA was applied to compare the three methods and the *Tukey* test for means comparison, all made by the Statistica software (Statsoft version 8.0, Tulsa, USA) at 5.0% level of significance.

RESULTS AND DISCUSSION

First, a comparison was made among the results obtained by oven and thermobalance methodologies, in which the samples were analyzed in triplicate ($n = 3$) for both methods, with the average of moisture content and its standard deviation are shown in Table 1. Results obtained by oven (AOAC, 2016), thermobalance and NIR/PLS method were compared by statistical analyses using Tukey and Student' t tests considering 5.0% level of significance.

No significant difference among the three methods ($p < 0.05$) was observed. Hence, moisture content measured by thermobalance method was used as the primary control/reference method for building the calibration model using NIR spectral data.

Pre-processing of NIR spectra

NIR spectra exhibited some noise, baseline distortion and/or some variation in optical path length occasioned by particle size (Figure 1a). Thus, it was necessary to investigate the application of spectral pre-processing detrend and smooth (moving average). In addition, in order to correct the effect of light scattering on the particle surfaces of the samples, standard normal variate (SNV) and multiplicative scatter correction (MSC) were respectively applied, all represented in Figure 1b and 1c, respectively.

TABLE 1.
Average of powdered egg moisture content ($n = 3$) and its standard deviation analyzed by oven, thermobalance and NIR/PLS methods.

Sample	Oven ¹ (%) (w w ⁻¹)	Thermobalance ² (%) (w w ⁻¹)	NIR/PLS ³ (%) (w w ⁻¹)	RE (1-2, 3) (%)	Significance (p)
1	4.70±0.07	4.67±0.01	4.71±0.01	0.86	
2	4.87±0.11	4.82±0.03	5.01±0.01	3.94	
3	4.94±0.06	4.92±0.03	4.98±0.17	1.12	
4	4.30±0.14	4.39±0.01	4.36±0.01	-0.80	
5	4.25±0.07	4.26±0.08	4.26±0.02	-0.00	
6	4.45±0.07	4.44±0.01	4.49±0.02	1.12	
7	4.55±0.07	4.62±0.03	4.48±0.02	-3.03	
8	4.50±0.00	4.61±0.16	4.70±0.03	1.84	
9	4.50±0.14	4.64±0.06	4.67±0.01	0.54	
10	4.35±0.07	4.32±0.03	4.33±0.02	0.68	
11	4.15±0.07	4.21±0.02	4.26±0.02	1.31	$p < 0.05$
12	4.65±0.07	4.75±0.07	4.73±0.03	-0.42	
13	5.05±0.07	5.27±0.03	5.19±0.01	-1.52	
14	5.11±0.07	5.28±0.14	5.23±0.03	-0.48	
15	4.95±0.07	5.09±0.01	5.19±0.01	1.86	
16	4.60±0.14	4.79±0.06	4.78±0.02	-0.10	
17	4.60±0.14	4.73±0.04	4.73±0.02	0.11	
18	5.28±0.03	5.24±0.02	5.08±0.03	-2.59	
19	5.12±0.03	5.19±0.01	5.19±0.01	0.10	
20	5.20±0.08	5.22±0.03	5.16±0.05	-1.15	
21	5.32±0.03	5.28±0.02	5.20±0.00	-1.60	

¹Oven method; ²Thermobalance method; ³NIR/PLS method; RE: relative error.

In order to enhance the resolution of high and low bands of water OH stretching, the 2nd derivative of 49 samples spectra was calculated as shown in Figure 2. The water characteristics band was observed at 1460 nm and its overtones appear at 950, 1010, 1470, 1790 and 1950 nm (Anbalagan, Mukundakumari, Murugesan, & Gunasekaran, 2009).

PCR models

PCR was performed in the range from 800-2000 nm, to cover the whole spectral variation based on powdered egg. Raw and pretreatment data with different methods were used to develop regression models with PCR. Results were shown in Table 2, in which it can be observed that PCR models developed on MSC showed better statistics compared with raw spectra and other pretreatment spectra for powdered egg samples. Smoothing pretreatment spectra could not improve results compared to raw spectra. PCR models developed with first and second pretreatment derivative take to low correlation factors. So, the best model for moisture forecast for powdered egg was the MSC treatment with r (0.9239), RMSEC (0.1012) and RMSEP (0.1044) (Luypaert, Heuerding, De Jong, & Massart, 2002).

TABLE 2.
Results of quantitative analysis performed for powdered egg samples with PCR.

Model	Pretreatment	r	RMSEC	RMSEP
1	None	0.90	0.11	0.12
2	MSC	0.92	0.10	0.10
3	SNV	0.92	0.10	0.11
4	1 st Derivative	0.91	0.11	0.12
5	2 nd Derivative	0.89	0.12	0.12

r: Correlation Coefficient; RMSEC: Root Mean Square Error of Calibration; RMSEP: Root Mean Squared Error of Prediction.

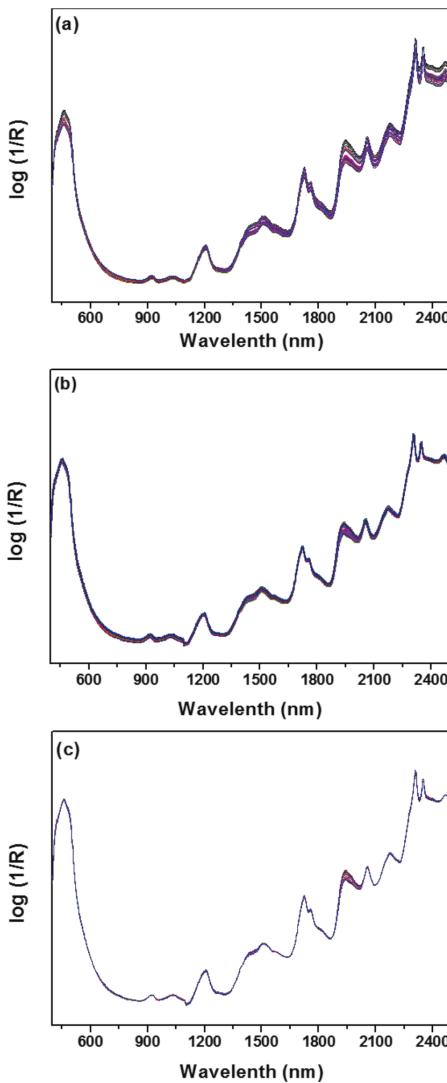


FIGURE 1.

NIR spectra of 49 samples of powdered egg used to build the calibration curve; (a) Spectra without treatment; (b) Spectra with detrend, SNV and smooth treatments; (c) Spectra with detrend, MSC and smooth treatments.

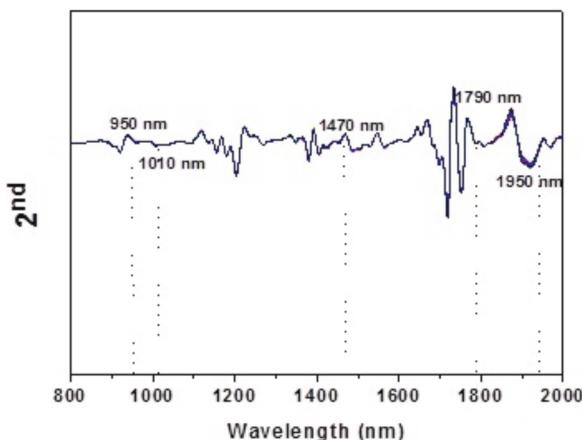


FIGURE 2.

2nd Derivative spectra of 49 powdered egg samples with zoom in bands of O-H bonds.

PLS models

PLS models for measuring powdered egg moisture levels, constructed using quality prediction models at 1000-2000 nm range of wavelengths are shown in Table 3. PLS models in determination of moisture seem feasible due to the high value of r ranged from 0.89 to 0.93. Correlation coefficient values do not vary to a great extent for first and second derivatives prediction, or by pretreatment by MSC, but they got a small improvement for raw spectra model. A slight decrease occurred in RMSEC and RMSEP for raw spectra model. So, the best values were obtained using PLS without any pretreatment with r , RMSEC and RMSEP of 0.93, 0.08 and 0.10, respectively.

TABLE 3.
Results of quantitative analysis performed for powdered egg moisture applying PLS, with best model indicated in bold.

Model	Pretreatment	r	RMSEC	RMSEP
1	None	0.93	0.08	0.10
2	MSC	0.92	0.10	0.11
3	SNV	0.92	0.10	0.11
4	1 st Derivative	0.92	0.09	0.11
5	2 nd Derivative	0.89	0.10	0.12

r: Correlation Coefficient; RMSEC: Root Mean Square Error of Calibration; RMSEP: Root Mean Squared Error of Prediction.

PLS calibration and internal validation

PLS regression was applied to the 49 powdered egg samples spectra calibration set with known moisture content determined by reference method. Four components and latent variables (LV) were obtained for cross-validation of the calibration model, in function of the coefficient of determination (R^2), as shown in Figure 3a and in function of the sum of the squares of the prediction errors (PRESS - Figure 3b) (Burns & Ciurczak, 2007).

Parameters calculated for PLS regression (Model 1 – Table 3) for calibration and internal validation or cross-validation, were compared with moisture content given by the control primary method (thermobalance) considered as a reference method (Figure 4). The parameters for model using all samples were: PRESS = 0.55; RMSE = 0.10; Average Relative Error = 1.78%; R^2 = 0.93 and r = 0.96.

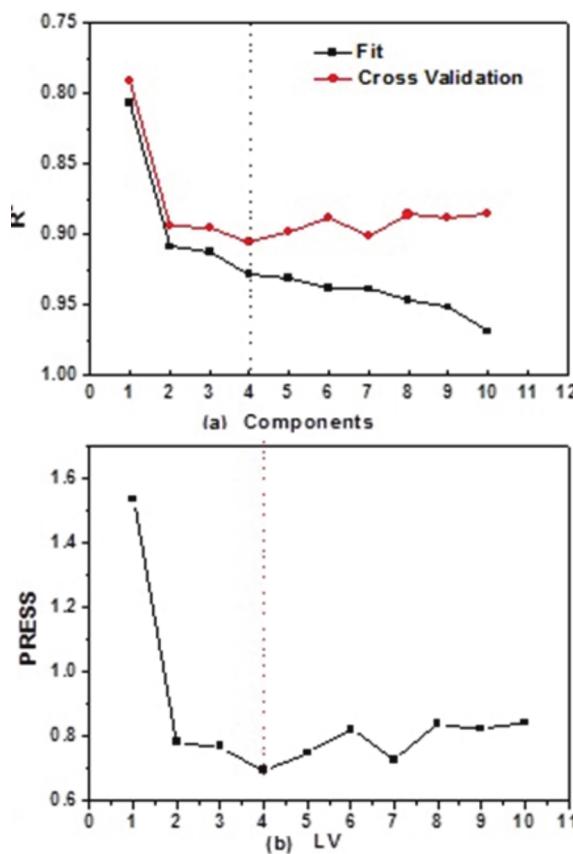


FIGURE 3.
Number of components and latent variables identification used in PLS modeling; (a) R^2 versus number of components; (b) PRESS versus LV.

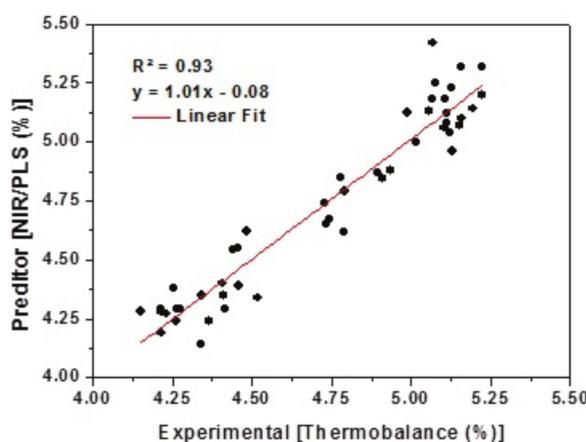


FIGURE 4.
Comparison of predicted by NIR and measured by thermobalance results for moisture content in powdered egg.

External validation of calibration model

Regarding external validation of NIR calibration model, spectra of 21 samples of powdered egg were randomly acquired in the spectrometer in triplicate ($n = 3$). Sample moisture average contents with

respective standard deviations are shown in Table 1. A comparison among the results obtained by control primary method (thermobalance) in relation to secondary NIR/PLS method, Student's *t* and Tukey's tests were used as descriptive statistics at 5.0% level of significance.

The *p* value smaller than 0.05 (Table 1) indicates no significant difference between moisture content measured by the two methods thermobalance and NIR/PLS at 5.0% level of significance. Furthermore, external validation parameters of dataset show that PRESS = 0.22; RMSE = 0.10; *r* = 0.96 and mean RE \sim 1.20% are lower than the analytical error of 5.0%. Figure 5 shows moisture content by prediction model done by NIR/PLS spectral method related to control content determined by thermobalance as reference method, with 92.0% of correlation ($R^2 = 0.92$).

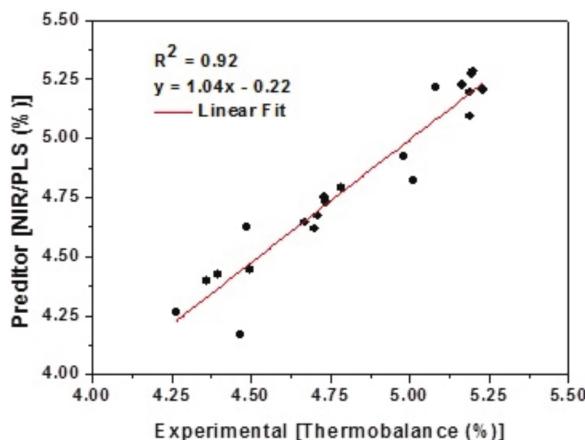


FIGURE 5.

Powdered egg moisture content determined by thermobalance (*X*) related to predicted moisture given content by NIR applying PLS model (*Y*) (Model 1 – Table 3) for 21 unknown sample set of external validation.

Similar moisture levels for egg powder analyzed by the secondary technique NIR/PLS compared with determined by two primary methods oven and thermobalance demonstrate the potential use for industrial with some advantages. First, the NIR (0.5 min for data spectra acquisition) associated with multivariate analysis decrease substantial the analytical time, saving up 4.5 min and over 8 hours, considering the thermobalance and AOAC methods respectively, without the need of sample preparation; it is environmental friendly and does not generate residues to be disposed. Second, it shows to be feasible by lowering operating cost, preserving samples, being a useful tool to introduce real time correction directly in on-line industrial processes, avoiding rework process by moisture out of specification.

CONCLUSION

After properly internal cross-validation and external calibration, using an adequate spectral pre-processing method, it was concluded that NIR/PLS is suitable and efficient for determining egg powder moisture content, to be directly applied to industrial process, guarantee same precision of reference method, although in a simple, fast, sustainable and reliable way.

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