Determination Of The Moisture Content Of Gelatin Sample

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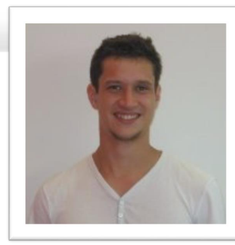


Introduction

Gelatin is a natural biopolymer obtained by denaturation and partial hydrolysis of collagen. The extraction process of gelatin is described in several steps; an acidic or basic pretreatment, hot extraction in aqueous phase, purification of the extract and then a drying step.

The loss of water generally increases the stability of biological materials, firstly by limiting endogenous chemical and enzymatic reactions and on the other hand by limiting the development of microorganisms. For the industry, the control of drying processes is essential, mainly because of the energy requirements of this stage, as well as the health risks associated with poor drying. Currently, the moisture content of the gelatin is determined by the loss on drying, which requires an analysis at 105°C. for 18 h.

The objective of this study is develop a fast and non-destructive tool, by near-infrared (NIR) reflectance spectroscopy, to evaluate the predictive capacity of moisture content of half finished industrial gelatin in the grain state (≈ 11.5% w/w) or powder (≈ 2.5% w/w). Determination of the moisture content in food is one of the first applications of NIR spectroscopie, it allows the realization of a measurement quickly and can be set up online or at-line. For this study, the gelatin samples were studied over a range of moisture of 1.6-13.9 (w/w), according to drying process (all samples, powder or grain). Different spectral pretreatments were studied and then compared to a multivariate calibration method, the Partial Least Square Regression (PLSr).

Materials and methods

Spectral acquisition :

The spectral acquisition is carried out with 100g of gelatin in a bulk at room temperature
Device : FTIR spectrometer (MPA, Bruker Optics, Ettlingen, Germany)
Module : Integrating Sphere
Range : 12500cm-1 – 3600cm-1
Resolution = 7,7 cm-1
Scan = 32 per sample
Scan velocity = 10 kHz

Preprocessing : RAW, RAW smooth (SavGol,1,15), Standard Normal variate (SNV), SNV Detrend, 1st derivative, 2nd derivative, multiplicative light scattering

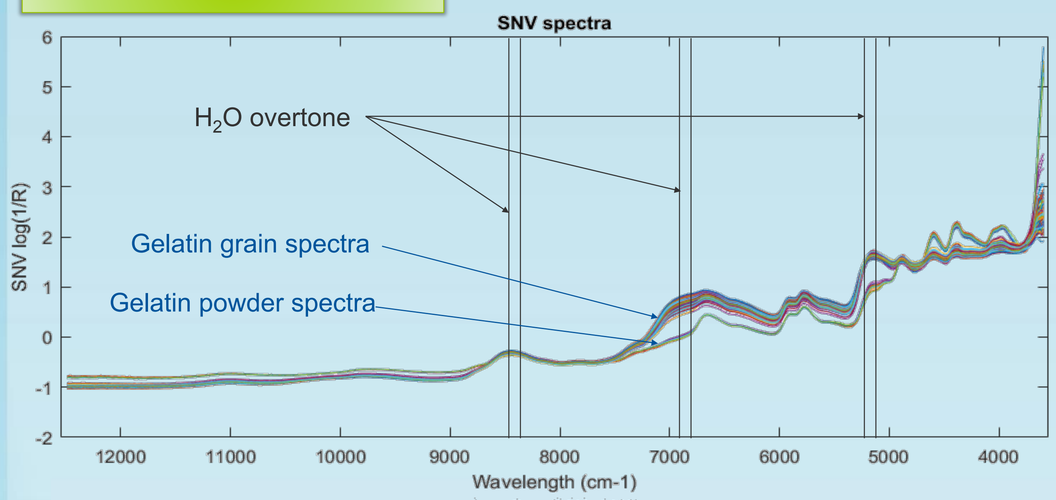
Data base :

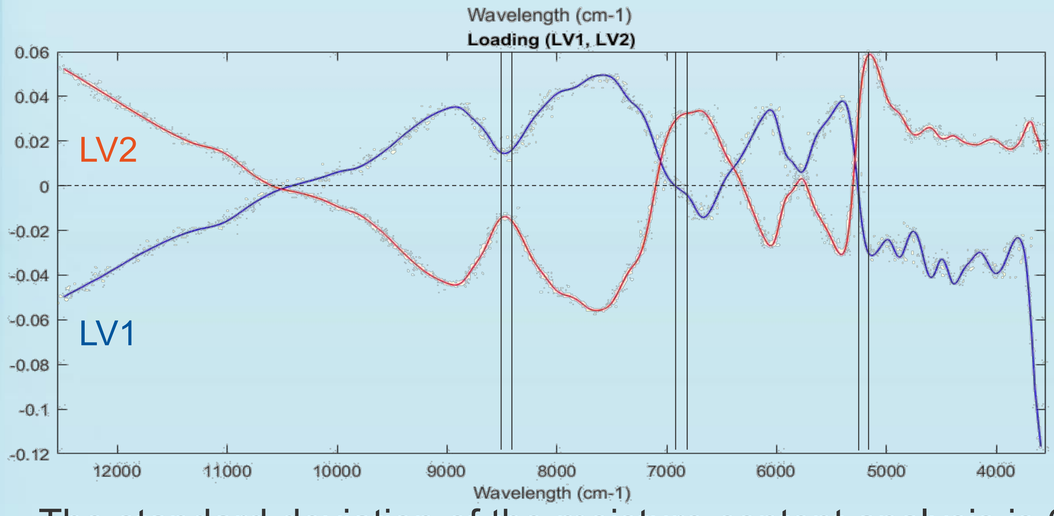
	Total (n)	Calibration (n)	Validation (n)
Full sample	153	123	30
Gelatin grain	136	109	26
Gelatin powder	18	15	3

Water content :
5 grams of gelatin are weighed in a crucible, them placed at 105°C for 18 h

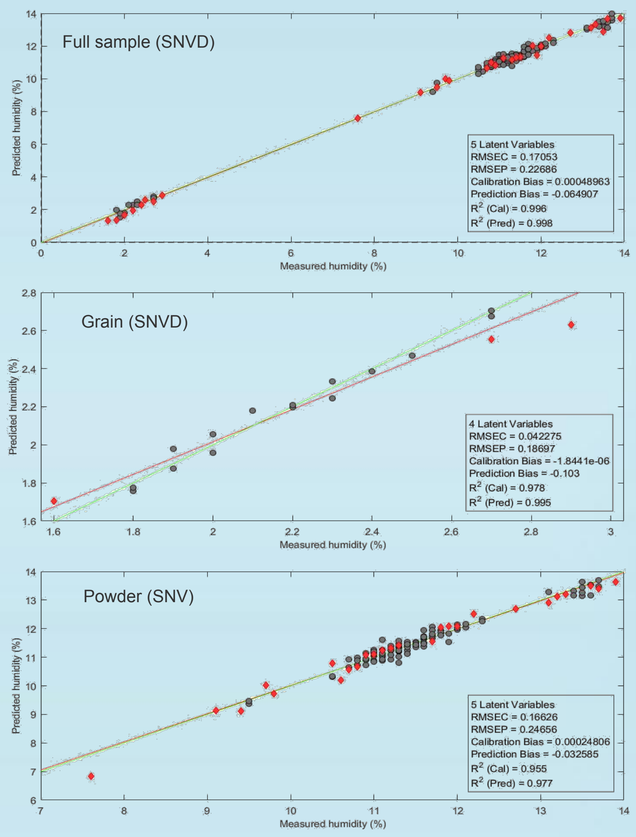
$$\%Dry\ matter = 100 * \frac{(m_{final} - m_{crucible})}{(m_{initial} - m_{crucible})}$$

Results





There are two distinct spectrum populations; gelatin grain spectra (≈ 11.5% w/w) and gelatin powder spectra (≈ 2.5% w/w). The granulometry is variable between the different samples, this can have a significant effect on light scattering. The best prediction model has been observed for SNV, SNVD and MSC pretreated spectra, because these transformations correct the light scattering effect. The NIR spectrum of the water comprises 4 characteristic absorption band, with absorption maxima at 5 150 cm^{-1}, 6 900 cm^{-1}, 8 400cm^{-1} and 10 300 cm^{-1}. By studying the loadings of the three databases, it is mainly the 5 150 cm^{-1} and , 6 900 cm^{-1} bands that influence the model. Usually theses bands are associated with free water.



The standard deviation of the moisture content analysis is 0.15% (w/w). For the prediction models; all the samples, gelatin grain and gelatin powder, the RMSEP values are respectively 0.2269, 0.2465 and 0.1869, with values of Ratio of standard error of Performance to standard Deviation (RPD) of 3.17, 2.51 et 13.0. For gelatin powder there is an overestimation of the model probably due to low sample number. These results clearly show a very good ability to predict the moisture of the gelatin.

Conclusion

- It is mainly the 5 150 cm^{-1} and , 6 900 cm^{-1} bands that react on moisture for gelatin samples
- The calibration results demonstrate a good ability to predict moisture content in gelatin samples. This can easily be set up for at-line analysis or even for online analysis
- The model can easily gain in robustness by enriching new reference samples

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