# Application of Near Infrared Spectroscopy and chemometrics for the characterisation of complex mixtures of food additives

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**FOOD ADDITIVE**

A chemical added to a particular food for a particular reason during processing or storage which could affect the characteristics of the food, or become part of the food.

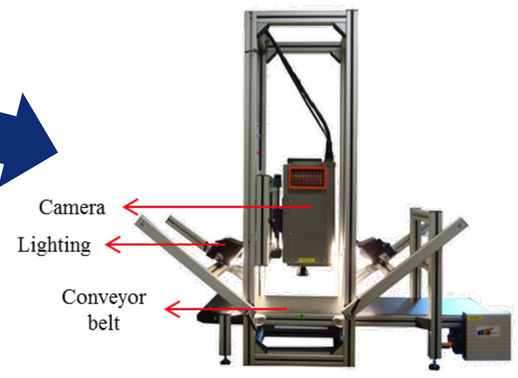
Certain ingredients present in complex blends of food products play an important role in the processing industry for adding texture, enhancing tasting, etc... However, controlling their proportions is essential both to asses the legal requirements for the safety of consumers and to avoid fraud in food labelling. These additives are often consisting of chemical compounds of very similar molecular structure. As a result, the discrimination of the different additives in mixtures is particularly challenging.

In the present work conventional Near Infrared Spectroscopy (NIRS) and Near Infrared Hyperspectral Imaging (NIR-HSI) have been used for the characterisation of complex blends of food additives. Artificial blends of food additives have been prepared in order to cover as much as possible the variability of possible mixtures found in actual blends on the market.

Partial Least Squares Regression (PLSR) (Geladi and Kowalski, 1986) has been tested in order to solve that issue. PLSR models have been constructed to assess the composition of each pure additive using the sample set of artificial blends.


conventional NIR spectrometer (FOSS XDS)

Ring cups containing blends of food additives for spectroscopic measurements.




NIR Hyperspectral Linescan Imaging System (BurgerMetrics).

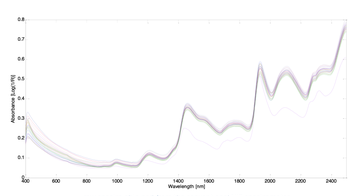
**Data preprocessing**
**Savitzky-Golay 1st derivative**
**(Window = 7 pts. ; Polynomial = 2nd order)**

| | | ADDITIVES | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | A | B | C | D | E | F | G | H |
| Calibration | Nb. blends | 57 | 52 | 51 | 54 | 54 | 53 | 66 | 63 |
| | $R^2$ | 0.98 | 0.96 | 0.99 | 0.95 | 0.94 | 0.97 | 0.57 | 0.96 |
| | RMSEC | 2.30 | 3.62 | 1.43 | 0.90 | 1.43 | 1.13 | 2.33 | 0.62 |
| | RMSECV | 2.62 | 3.94 | 2.14 | 1.84 | 2.21 | 1.80 | 2.84 | 0.84 |

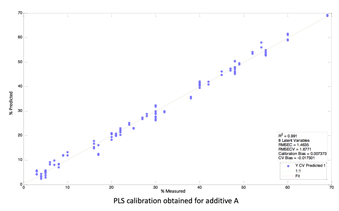
Example:

Additive "A" is present in 57 blends in various proportions. These 57 blends have been used to calibrate a PLSR model with $R^2$ of 0.979, RMSEC of 2.301 and RMSECV of 2.623.

The obtained regression plot for additive "A" is presented hereafter.




raw spectra obtained from the conventional NIR spectrometer




PLS calibration obtained for additive A

Additionally, for additives "A", "B" and "C" the data-set has been split into calibration and test sets in a way to cover the range of concentrations of the targeted additive. This data splitting is done in order to achieve an independent validation of the models as illustrated hereafter.

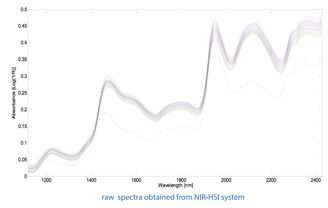
| | | ADDITIVES | | |
|---|---|---|---|---|
| | | A | B | C |
| Calibration (90% of the sample set) | Nb. blends | 52 | 47 | 46 |
| | $R^2$ | 0.98 | 0.96 | 0.99 |
| | RMSEC | 2.36 | 3.51 | 1.11 |
| | RMSECV | 2.73 | 3.88 | 1.88 |
| Independent validation (10% of the sample set) | RMSEP | 2.11 | 3.39 | 3.89 |

| | | ADDITIVES | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | A | B | C | D | E | F | G | H |
| Calibration | Nb. spectra | 57 | 52 | 51 | 54 | 54 | 53 | 66 | 63 |
| | $R^2$ | 0.93 | 0.93 | 0.88 | 0.23 | 0.78 | 0.64 | 0.49 | 0.96 |
| | RMSEC | 4.50 | 3.86 | 5.52 | 5.46 | 3.05 | 3.43 | 2.90 | 1.15 |
| | RMSECV | 5.65 | 4.14 | 6.72 | 7.13 | 4.11 | 4.50 | 3.54 | 1.53 |

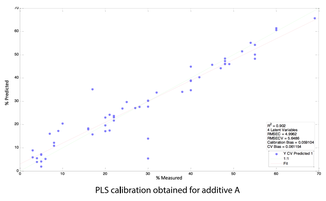
Example:

Additive "A" is present in 57 blends in various proportions. These 57 blends have been used to calibrate a PLSR model with $R^2$ of 0.934, RMSEC of 4.9962 and RMSECV of 5.6486.

The obtained regression plot for additive "A" is presented hereafter.




raw spectra obtained from NIR-HSI system




PLS calibration obtained for additive A

Additionally, for additives "A", "B" and "C" the data-set has been split into calibration and test sets in a way to cover the range of concentrations of the targeted additive. This data splitting is done in order to achieve an independent validation of the models as illustrated hereafter.

| | | ADDITIVES | | |
|---|---|---|---|---|
| | | A | B | C |
| Calibration (90% of the sample set) | Nb. blends | 52 | 47 | 46 |
| | $R^2$ | 5 | 2 | 4 |
| | RMSEC | 0.91 | 0.95 | 0.81 |
| | RMSECV | 4.21 | 3.84 | 5.98 |
| Independent validation (10% of the sample set) | RMSEP | 5.20 | 4.16 | 7.21 |

## Conclusions

For most of the additives, the PLSR models provided good results with $R^2$>0.8 and RMSCV between 2% and 7%. In addition, an internal validation has been done for three PLSR models and provided good results with RMSEP between 2% and 7%. In the present study conventional NIRS provided better results compared to NIR-HSI. However, NIR-HSI could be a useful technique combining the detection of contaminants with a quantitative analysis of the sample.

The obtained results showed that spectroscopic techniques such as NIRS and NIR-HSI combined with chemometrics are potentially useful in food industries for the characterisation of complex blends of food additives.

## References

Geladi, P. and B.R. Kowalski, "PLS Tutorial" Anal. Chim. Acta., 185(1), 1986.

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