# Evaluating thermal treatment of fresh egg pasta by near infrared spectroscopy

**Stefano Zardetto[a]– Paolo Miolo[b]**

[a]*Voltan Spa- Via Dosa 24 – 30300 Olmo di Martellago (VE) – Italia*

[b]*Büchi Italia S.R.L. – Palazzo 4, Str.4 – 20090 Assago (MI) - Italia*

## Introduction

Fresh egg pasta is a typical Italian product. Pasta is made by hydrating durum wheat with water and eggs, mixing it, kneading it and forming by sheeting-rolls to obtain the desired shape, and then pasteurised before packaged in gas-impermeable. A pasteurisation treatment is usually given to pasta to kill mould spores and spoilage microorganisms as part of the processing procedure. This thermal process however, determines phenomena of modification and reciprocal interactions on macromolecular level concerning proteins and starch which determine an improvement of pasta quality. These phenomena are evidenced with a colour modification,[1, 2] a decreasing of water activity, an increase of starch gelatinisation level, a modification of quantity of water absorbed in cooking phase [3, 4].

Besides the heat treatment can bring to negative modifications caused by Maillard reaction with a nutritional food decreasing as a result of biologically unavailability of amino compounds. The proper control of thermal processing conditions is crucial not only to ensure safety, but also to obtaining the wanted quality parameter.

The effectiveness of the thermal process is generally expressed in term of lethaliy and the lethal effect on pathogenic and spoilage organisms is due to the combined effects of duration and temperature 5. Total process lethality, designated by the symbol F. F, is the equivalent, in minutes at some given reference temperature, of all heat considered with respect to its capacity to destroy spores or vegetative cells of a particular organism. Since the F value is calculated with respect to a reference microorganisms characterized by the z and D parameter the F value determined for any process depends on the value of z employed in the calculation.

D is the time required at any temperature to destroy 90% of the vegetative cells of a given microorganism and z is the number of Celsius degrees necessary to cause 90 % reduction in the decimal reduction time D.

The reference temperature usually used in the pasteurisation process is 70 °C and a value of z equal to 10 (commonly observed for pathogenic microorganism vegetative cells) is normally adopted. It is customary to express F with a subscript denoting the temperature and a superscript of the z value for the microorganism being considered. Thus, $F_{70}^{10}$ is the common designation for the pasteurisation capacities of heat processes.

The objectives of this research were to use NIR diffuse reflectance spectroscopy for evaluating previous thermal treatment of fresh pasta and to determine the effect of different thermal treatment on the spectra.




Near Infrared Spectroscopy: Proceedings of the 11th International Conference.

www.nirpublications.com

## Materials and methods

### Evaluation of time-temperature treatments

The following relation (Bigelow method) was adopted to estimate $F_{70}^{10}$ value (pasteurising effect):

$$F_i = \int_{t1}^{t2} 10^{\frac{T-70°C}{Z}} dt \quad (1)$$

The temperature corresponding to each time interval can be obtained from the penetration of the heat curve and $F_i$ values defined by formula (1) can be calculated.

The value was measured by a data logger (EBI-125A EBRO-Germany). Temperatures recorded every 10 s and $F_{70}^{10}$ values of every treatment has been calculated.

Chen and Marks[6] reported a non linearity relationship between spectral data and F values and they have reported that base-10 logarithms had the best linear relationship with spectral data. Then the F values was been first transformed to base-10 logarithms.

### Samples and sample preparation

Experiments on samples of fresh pasta (thickness of pasta 0.90 mm) were carried out. Pasta has been produced using durum wheat with addition of 21% fresh pasteurised eggs. Final products showed a water activity values of 0.950±0.005, and a moisture content of 30.5%±0.351.

The pasta was pasteurized after the forming phase by means of damp heat The working pressure was 9120 Pa. At the end of the heating period the pasta was submitted to forced hot air for partial drying. Following this process, the samples were withdrawn. Tests have been made in different days and on samples with different batches.

Fresh pasta sample (n= 87, 1 cm thick) were acquired after the treatment. Circular sub-samples of 90 mm diameter were taken using a cutter and three separate slices per sample placed in a sample cell. Sample were stored in the NIR laboratory at a temperature of 22±1°C for at half hours prior to spectral measurements.

**Table 1. Statistics for the sample sets**

| | n | Mean | Min. | Max | SD |
|---|---|---|---|---|---|
| Calibration | 67 | 2,56 | 1,73 | 3,32 | 0,387 |
| Validation | 20 | 2,62 | 1,78 | 3,31 | 0,387 |

n= number of samples; SD= standard deviation

### Near infrared spectroscopy

NIR spectra were recorded using an NIRLAB N-200 (FT-NIR Buchi, Switzerland) in reflectance mode. Spectra were recorded in reflectance mode using an ISI ring cup. Spectral data were recorded from 1000 to 2500 nm at 2 nm intervals and saved as the 3 scans for each sample.

Measurements and chemometric interpretation of the NIR spectral data were performed using NIRCAL 4.21, chemometric software from Buchi (Switzerland). The calibration can be calculated and optimised with the patent pending “Calibration Wizard”.

The optimal factor number, the number of data pre-treatment and wavelengths calibration set, for each set of processing treatments, was automatically determined by the software. The raw

optical data were transformed into second derivatives using a 16 nm gap and height-point (16 nm) smoothing function.

Partial least squares (PLS) regression analysis was the regression method selected to establish the relationship between the $F_{70}^{10}$ value from its data logger method and NIR absorbance values with no outlier elimination. Model performance was measured as the coefficient of determination ($r^2$), the standard error of estimation (SEE), the standard error of performance (SEP), the slope of the linear regression of NIR predicted versus original, bias and the Q value. The Q-value is procedure of NIRCAL software and takes a row of statistical values into consideration and to give a mean a quality of the calibration. The higher the Q-value, the better the calibration. An ideal quality of the calibration has a value of 1.

## Results and discussion

A NIR model was obtained, using PLS regression, for the prediction of $F_{70}^{10}$ value in fresh pasta. A total of 87 samples were considered overall during the calibration development: using a randomised procedure 67 samples were assigned to the calibration set, where the remaining 20 samples constituted the validation set. The statistical results of calibration and validation for the $F_{70}^{10}$ parameter are summarized in Table 2.

**Table 2. Statistics for the prediction of $F_{70}^{10}$ value by Near-Infrared Spectroscopy**

| | n | Mean | SEE | $r^2$ | Range |
|---|---|---|---|---|---|
| Calibration | 67 | 2,56 | 0,160 | 0,910 | 1,73 - 3,32 |
| Validation | 20 | 2,62 | 0,183 | 0,884 | 1,78 - 3,31 |

n= number of samples; SEE= standard error of estimation; $r^2$ = coefficient of determination

The standard error calibration was 0,16 and regression coefficient of determination ($r^2$) 0,91. The validation samples were predicted with an SEP and r2 of 0,18 and 0,88 respectively. The bias was very small (0.02). The relationships between data logger and NIR spectroscopy predicted parameters ($r^2$) were high ($r^2$>0,90). This result is in good agreement with that reported by Chen and Marks[6] in the chicken patties. The second derivate absorption value in the 1000-1282 nm, 1514-1853 nm region and 2082-2276 nm region made positive contributions to the regression equation for $F_{70}^{10}$ value. The wavelengths in this region were used to develop the calibration model for thermal treatment estimate of fresh pasta. The model developed used four factors, which explained the spectral variation. PLS loading plots show the regression coefficients of each wavelength to the constituent of interest and can indicate which wavelengths are important in developing a model. Wavelengths of high variation in the loading plots can be associated with areas of spectrum of known chemical origin. Loading plots for four factor for the model developed indicate that vibration due to CH groups in wheat protein (1760, 1730, 1700 nm), OH groups in starch (2276 nm) and O-H/bend/hydrogen-bonded O-H stretch (2258 nm) are important in the model.[7, 8] Moreover, the loading plot v. wavelengths exhibits a negative correlation at 1678, 1740, 1850, 2206 and positive at 2238nm.

In summary, near infrared spectroscopy can be used for the rapid determination of $F_{70}^{10}$ value in fresh pasta. The monitoring of this parameter allows the control and the management of one of the critical control point of the process and to ensure safety and quality of the product. Examination of the wavelength of high variation in the PLS loadings suggest that the physical change due to heat treatment in fresh pasta can be evaluating by near spectroscopy. Future work should study if physical and chemical changes during heat treatment my be optimised by using NIR spectroscopy. In fact many studies have demonstrated the relationship between heat treatment change in the pasta

and its quality parameters[9, 2, 4] and this technique demonstrates the potential for control of heat treatment and thus may be used to ensure that high quality products are obtained.

## References

1. L. Cencic, C. Franca, M. Dalla Rosa. Studio su pasta fresca farcita (ravioli) a diverse attività dell'acqua. *Tecnica Molitoria* 5, 449-464 (1995)
2. M. Verrati. Studio sull'influenza di trattamenti termici operati in fase di processo su alcune caratteristiche chimico e chimico-fisiche di pasta fresca farcita. Tesi di laurea. Università degli studi di Bologna (2000).
3. E. Grazian. Studio di ottimizzazione del processo di essiccamento di paste fresche farcite. Tesi di laurea. Dipartimento di Scienze degli Alimenti, Università degli studi di Udine (1999).
4. S. Zardetto, S. Di Fresco, M. Dalla Rosa,. Effetto di trattamenti termici sulle caratteristiche chimico-fisiche della pasta. *Tecnica Molitoria* 2, 113-130 (2002).
5. C.R. Stumbo. Evaluation and equivalency of pasteurization processes, In Thermobacteriology in food processing. San Diego, California: Academic Press, Inc. pp. 223-234 (1973).
6. H. Chen, B.P. Marks. Evaluating previous thermal treatment of chicken patties by visible/near-infrared spectroscopy. *Journal of Food Science* 62, 753-780 (1997).
7. D.P. Law, R. Tkachuk. Near infrared diffuse reflectance spectra of wheat and wheat components. *Cereal Chemistry* 54(2), 256-265 (1977).
8. B.G. Osborne, T. Fearn, and P.H. Hindle. *Practical NIR spectroscopy with application in Food and Beverage*. Longman group UK Limited, England (1993).
9. L. Pasini. Studio sulla capacità di idratazione di sistemi amidacei a media e bassa umidità. Tesi di laurea. Università degli studi di Bologna (1998).