On-line determination of the target brown colour in caramel with Near Infra Red Spectroscopy

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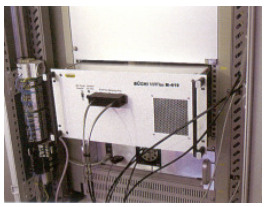
Introduction

Real time analysis, in the industrial plant, is getting more and more important to optimise production, getting a very high quality control of the final product, saving money and time.
The study is about an on-line application of Near Infrared Spectroscopy as a method to monitor industrial production process of food additives. The research scope was to monitor the production process of caramel, an additive which is normally used in many applications of food industry.

Materials and methods

The production tank has been modify for the connection of a dedicated on-line trasmittance stainless steel probe with zafire window and light path of 1mm.
The process spectrometer is a FT-NIR Büchi NIRFlex N-419 (spectral range 4.000-10.000 cm^{-1} and resolution of 12cm^{-1}) with Buchi N-420 Process Control Module to handle automatic mode analysis and the multiplex optic that in future will be implemented for a second line.
A pair of 20 mt. monofibres are used to connect the spectrometer to the probe in the tank. All spectra have been elaborated with NIRCal 4.21 a Chemometric Software by Buchi.
Reference analysis as been taken with a **XXX** colorimeter at 550 and 610 nm.



Spettrometer FT-NIR
Buchi Nirflex N-419

Trasmittance probe

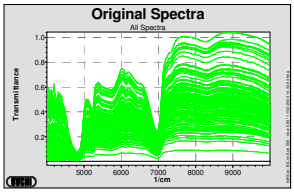
Application description

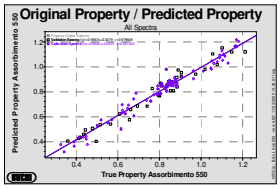
Caramel is produced from carbohydrates like glucose and saccharose mixing them with water into a thank during a controlled heat treatment. The tank has a little low pressure to take away water till the target colour of caramel is reach. At this time is no possible to take a sample to measure it with a colorimeter because at room temperature and pressure, the product became solid. It's only the operator experience that determines the end of this crucial step of the process, considering time, water extracted and looking at the intermediate product through a small windows placed in the top of the tank. Then a fixed quantity of new clean water is introduce in the tank and cool down. Finish this step, you have the final product (caramel) and it's now possible to open the tank and take a sample for quality control but if the target colour is not reach, you need to do a complete new production cycle.
Target colour of the final product is normally measured at 550 and 610 nm.

Production site

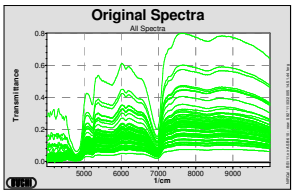
Result and discussion

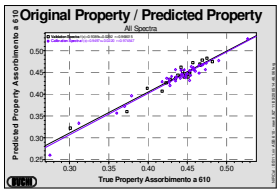
Two different caramel type were consider, one with the target of brown color measured at 610nm and the other at 550nm. The first set was done of 125 samples taken at the end of the critical step just before the water introduction. Reference value was add later after measurement of the final product color with the colorimeter at 610 mn. The second set consist of 65 spectras measured at 550 nm. In both case about 2/3 of the datas were used for calibrate the Spectrometer and 1/3 to validate the application. The result of calibrations are below.





Spectra and calibration curve at 550mn for the first set





Spectra and calibration curve at 610mn for the second set

Product Paramenter	Number of sample	Calibration range	Regression Coefficient for Calibration/validation set	SEE/SEEP
550mn	125	0.325-1.133	0.98/0.97	0.04/0.04
610nm	65	0.275-0.527	0.97/0.96	0.009/0.010

Calibration Results

Final product

Conclusion

During routine analysis, every minute, a spectra is measured by the spectrometer inside the tank with the calibration done. During the process, the "color" of the intermediate product is displayed and as soon as the target value of the correlated final product is reach, the new fixed quantity of clean water is introduced in the tank and the process is stop. In this way all production cycle will be inside specification and the production can be optimized.

This job shows how NIR technology can be very useful to control caramel production, avoiding out of target products, saving time and money. In the future, an other tank will be implemented with a new probes and a multiplex optic to control in sequence, with a single FT-NIR, also an other production line.