In-line measurement of high moisture products

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Introduction

There is increasing competition between food producing companies, especially among dairies. They have to improve their yields, because the raw material—raw milk—is expensive. Controlling the process by applying traditional methods, one is not able to perform process control on a solid basis. The reason is that one has to collect a sample at the production line and then bring the sample to the laboratory, which can be a couple of hundreds meters away, where the sample will be analyzed. Nearly all traditional methods are very time consuming. Let us take, as an example, the determination of the water content (inverse: solid content) in cottage cheese. The fast method needs half a hour to give the result of the water content. This method has poor precision parameters but is the only one which gives fast results. The reference method takes at least three hours before giving the water content. Using the fast method, the information about the water content can be returned back to the production line three quarters of an hour later. Considering the production of cottage cheese, the amount produced within this time could be somewhere between 1000 and 4000 kg having the wrong specification and causing either a total product or an economical loss.

There is some other problem associated with this kind of process control. In the meantime, the process could have changed so that the information is of no use or gives the wrong idea about how to influence the process. Therefore, one needs methods which give reliable results immediately. Near infrared (NIR) spectroscopy offers the possibility to solve this problem and to give accurate results on the basis of a solid calibration.

Material and method

A NIRSystems 6500 laboratory instrument has been used to determine the optimal optical path length/sample thickness, because a transmission measurement will be performed due to the high protein content of the product. The protein causes thick stationary layers so that a reflection measurement will only detect a nearly constant spectrum. To study further questions such as the influence of flow velocity, product damages and so on, the whole equipment has been set up in

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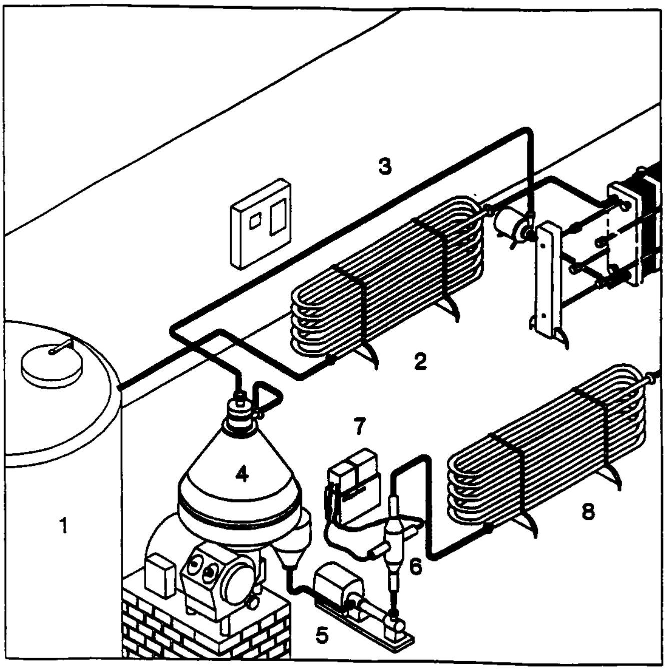

Figure 1. Schematic diagram of the production line for cottage cheese.

our pilot plant. The study has been performed on a type of cottage cheese which has a moisture content of approximately 82% water.

Figure 1 shows the production line for cottage cheese. The milk is coagulated in the tank (1), pumped through a heater (2) and feeding pipe (3) to the separator (4), where the milk is separated into whey and curd. A pump (5) brings the curd through the in-line device (6) and a cooler (8) to the packaging unit. The NIRSystems 6500 (7) is equipped with a transmission pair of fibers.

The investigation shows that a sample thickness of 8 mm can be measured in transmission mode if one uses the wavelength range from 700 to 1100 nm. A problem has been the calibration of the instrument due to the transmission device. Taking the spectra in an "off-line" transmission device and comparing with the spectra of the same material in an "in-line" transmission device, the spectra look different. Due to the high accuracy demand, the set-up has been modified to extract samples directly behind the in-line cell. This means, that a sample is taken during the measurement of the spectrum to determine a reference value (water content) as close as possible to the measured spectrum. Thirty spectra have been collected and averaged, which takes approximately 20 seconds. Partial least squares has been adopted as the calibration procedure.

Having extracted the optimal conditions, the set-up has been transferred to the production line of a dairy, which is similar to the one shown in the Figure 1. The only change has been that the NIRSystems 6500 has been replaced by the appropriate industrial instrument, namely the NIRSystems 5500. The characteristic of the instrument is slightly different due to the different fiber optics. The instrument does not have the same light intensity as the laboratory instrument causing a little reduction in optical path length.

Results and discussion

Only the results of the performance in the dairy will be considered. There has hardly been any influence due to the flow velocity of the product or different pressure in the product. Some of the spectra are taken during a stop of the pump (5), some during the start and some during full operation. If this would have been a problem, a coupling between the operational control of the pump and the spectrometer would have been the solution. There has been a slight increase in product pressure due to the decrease in the diameter of the pipe (in-line cell), which does not cause any damage to the product.

The calibration of the instrument has been performed in the dairy by collecting 800 spectra and reference values over a period of two months, so that production variations are included in

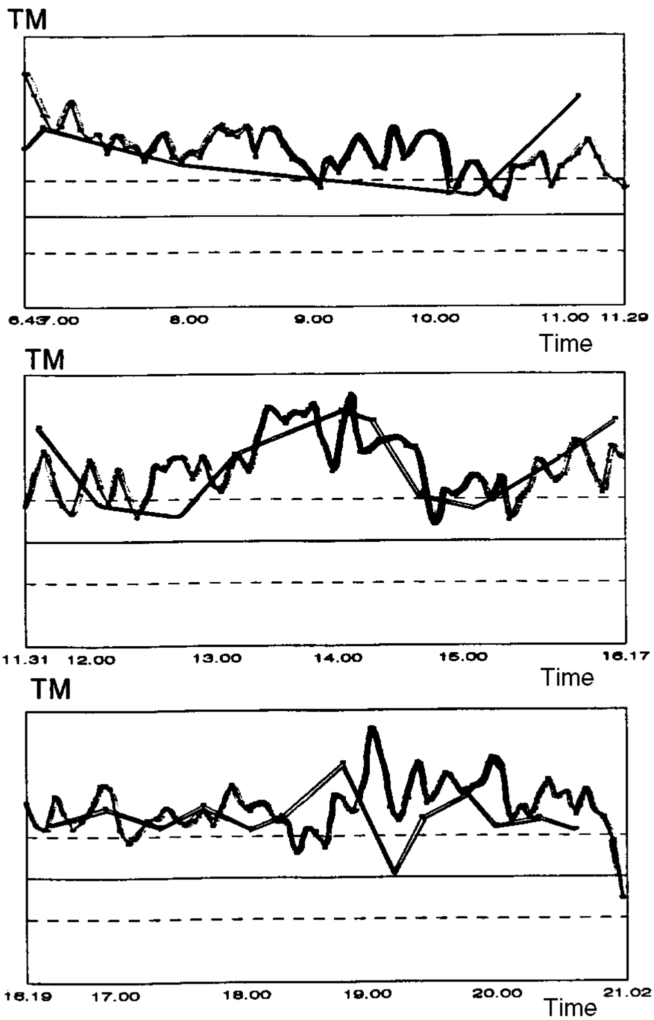

Figure 2. Results of the solid content (TM) measurements as a function of time.

the calibration. This does not mean that all variations are contained in the calibration because the recipe is changed according to the changes in the raw material, which happens twice a year and can be different from year to year.

Figure 2 displays the results of the solid content (TM) as a function of time over one production day for cottage cheese. The structured curve represents the NIR prediction, where a measurement has been performed every three minutes. The displayed results show the moving average over three measurements to smooth the curve. The flat line gives the results of the reference method, which is performed between every 20–90 minutes. The figure shows an ordinary, arbitrary production day.

Looking at the difference between NIR prediction and reference value, all differences fall within 0.25%. Taking into account the accuracy of the reference method, characterized by the precision parameter repeatability, which is 0.3%, one cannot do better than this, because the reference values can have errors of this order. The trend is well described. There are several reasons and explanations for the structure in the NIR results:

a) There is a continuous building and destruction of the layers at the windows of the in-line cell.
b) There could be some air bubbles in the curd causing changes in the absorbance.
c) There could be some product velocity or product pressure influence.
d) The separation process is not homogenous causing changes in particle size distributions as well as water content.

At the moment it is not clear whether the structures are real or artifacts. It is difficult to disentangle the various sources. Some will hopefully be understood in the next testing phase. Nevertheless, the results, taken in the production line of a dairy, are very promising.

One further remark: the in-line cell does not create any problems with respect to cleaning as well as the duration of continuous operation or microbiological questions. The cell has been designed especially for the food industry and is no limiting factor for the operation of a production line.

Summary and future work

Having determined the optimal parameters, the scale up from the pilot plant to the real process line has not created any problems. The NIR predictions and the reference values agree as much as possible. To ensure the proper behavior of the calibration and the instrument, it is necessary to arrange a continuous monitoring system for the calibration in the form of a control chart (see Figure 2) and for the instrument by performing internal tests on a regular time schedule.

The future work consists of three tasks:

i) to arrange a closed loop operation with the feeding pump [see (3) in Figure 1] to perform process control with respect to the water content on the basis of NIR predictions.
ii) to extend the in-line-measurement idea towards other production lines by using a multiplexer.
iii) to adapt the method to other products.

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