On – line monitoring of dairy products with the use of NIR technology

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Abstract

The feasibility of the use of Near-Infrared Spectroscopy and multivariate calibration methods for the measurement of multiple constituents of complex mixtures has been shown for a variety of agricultural and food related products. This report presents the use of Near-Infrared Spectroscopy for on-line and at-line monitoring of dairy related products, such as milk, powder milk, butter, cheese, etc. The measurement includes parameters such as fat, protein, solids, salt, moisture, etc.

Key words: *Dairy products, monitoring, NIR Technology.*

Introduction

The use of Near Infrared (NIR) technology is rapidly being spread across the industry as a process control tool, due to the advantages that it offers. The increased yield, improved product quality and the lower product testing cost are some of the benefits that the industry has gained by the on-line or at-line use of the NIR technology.

This report describes the advantages that Near Infrared technology offers for one area of the food industry, specifically dairy products related industry.

Near Infrared Spectroscopy

Near Infrared Spectroscopy (Hildrum et al. 1992) is the technique where the spectral characteristics of samples are recorded in the Near Infrared range, typically 1200nm to 2400nm, of the electromagnetic spectrum. The sample is illuminated with a near infrared light source, and the reflected or transmitted light is recorded. The energy difference between the illuminating radiation and the reflected or transmitted radiation at each wavelength, constitutes the absorbance spectrum of the sample. The major absorbance features seen in the NIR region are due to combinations and overtones of fundamental vibrations that are seen in the Mid-Infrared region of the spectrum. These features include C-H, O-H, N-H, C=O, =C-H, COOH, as well as aromatic C-H bonds. Of course most of the composing elements of the dairy products include these bonds, which means that the composition of dairy products can be monitored with the use of NIR spectroscopy.

During the calibration process of the technique, multi-variable regression techniques (Burns and Ciurczak, 1992), such as Principal Component Regression (PCR), Partial Least Squares (PLS), and Multiple Linear Regression (MLR), are used to correlate the absorbance features of the spectra to the concentration values of the calibration samples. This correlation produces a calibration curve which can be used for the analysis of unknown samples.

The control of the NIR spectrometer, the data acquisition, as well as the above mentioned calculations for the analysis of the unknown samples, are curried by a computer system interfaced to the spectrometer. The results can then be used in one of two ways, 1) the results can be reported to the process control operator, who would be responsible to keep the process within the specified operational limits (open loop), or 2) the computer itself can generate a series of electrical signals which will automatically control the process (close loop).

Instrumentation

The instrumentation used for on-line installations and at-line installations, are usually quite different. Some of the differences are in the instruments themselves, but mainly the differences are in the way that the sampling of the product is done and the type of probes used. This section describes these differences.

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On - line

On-line installations are usually the most challenging ones. The instrument is placed close to the monitoried process requiring that the instrument withstands the harsh conditions of the process environment. Frequently, these conditions include extreme temperature conditions, high humidity, high dust levels, etc.

To withstand these conditions, the instrument itself should be completely shielded and have temperature control capabilities or it should be enclosed in a shielded and temperature controlled enclosure. Most on-line analysers provide these capabilities.

The sampling is done by a probe that is directly inserted into the process line, and the design of the probe should be such that the probe will have the ability to withstand the process conditions. In the case of dairy products the process conditions are usually not very severe, but the probe should also have the ability to withstand the conditions of the cleaning cycles used to clean and sanitize the process lines. These cycles frequently involve very strong and high temperature caustic solutions.

The probe should also have the ability to be removed from the process line, in order to be checked, cleaned if necessary, and referenced. The removal of the probe should be done without having to shut the process off, especially in continuous running processes. This can be accomplished by a retractable probe assembly, which either automatically or manually retracts the probe while at the same time seals the process line. The retracted probe can then be cleaned, checked and referenced before being reinserted in the line.

The on-line monitoring offers a great deal of advantages. Since the product is continuously being monitored, the process can continuously be adjusted to eliminate the production of off-spec products, which increases the yield and eliminates the cost of disposal or reprocessing of the products. The yield is also increased by not having to store the product while waiting for the lab analysis results, which often can take hours.

At – line

The requirements for an at-line NIR analyser installation are typically quite simple. The instrument is physically close to the monitoried process, but in a location that is easily accessible by an operator and usually heated and/or air-conditioned.

For at-line monitoring, an operator is required to grab a sample from the process line and bring it to the analyzer. Such a scenario offers a greater flexibility for the type of samples that can be measured. For example, samples such as powder milk, and processed cheese, can be monitored in conditions that would make it very difficult, if not impossible, to monitor otherwise.

The main advantage of the at-line monitoring is the great speed which the NIR analysis offers, allowing the measurement of multiple parameters and ingredients of the sample in only a few seconds. As stated above, it also allows the measuring of samples that are not easy to handle under normal conditions. The main disadvantage of the at-line monitoring versus the on-line monitoring is that the monitoring is not continuous, but still a sample has to be taken before the analysis can be done. Another disadvantage is the required training of the operators and, some times, the disposal of the samples.

Measurements

Typical measurements done on-line or at-line with NIR analysers in the dairy products industry include: percent fat, protein, and solids in milk, powder milk and cheese, as well as the percent moisture in powder milk.

The Standard Errors of Prediction for the above parameters come very close to the Standard Errors reported for the primary analytical methods used to analyse the calibration samples which are used during the calibration development. From the measurement point of view, the main advantages that the NIR method offers is high precision, (which is usually higher than that of the primary analytical method,) and speed (typically in the order of seconds for all of the above parameters).

For the purpose of this report, the calibration results obtained during the calibration development for the measurement of percent fat (%Fat), percent protein (%protein), and percent solids (%Solids) in processed milk will be discussed.

Fifteen calibration The analytical values for these ingredients of the fifteen samples are shown in Table 1. The samples were scanned samples of milk with varying amounts of fat, protein, and solids were used for this study. with an LT Industries Quantum 1200 Plus Near Infrared Analyzer, equipped with an NIR grating (1200nm to 2400nm) and a fibre optics cable with a 3mm path-length bubble shedding probe. The bubble-shedding probe is a special design, which eliminates the formation of bubbles at the tip of the probe, particularly in the case of viscous materials.Each of the collected spectra was the average of 30 complete scans between the 1200nm to 2400nm range, which were collected in approximately 20 seconds. The spectral activity of the analyzer was eliminated by dividing the collected spectra with a background spectrum (air). The collected spectra were converted to absorbance spectra by taking the negative logarithm (log1/x).

Tab.1. Analytical values of the calibration samples.

Sample	Fat(%)	Solids(%)	Protein(%)
1	3.8	21.1	3.5
2	1.8	24.3	6.0
3	0.9	23.8	4.8
4	0.2	25.9	6.8
5	4.3	19.9	2.5
6	0.2	25.1	5.9
7	1.6	22.6	3.8
8	3.0	23.1	5.4
9	3.0	21.4	3.4
10	1.1	27.2	9.1
11	2.3	25.1	7.4
12	1.3	24.4	5.9
13	3.4	20.6	2.6
14	3.7	21.8	4.4
15	0.1	27.7	9.0

Typical absorbance spectra can be seen in Figure 1.

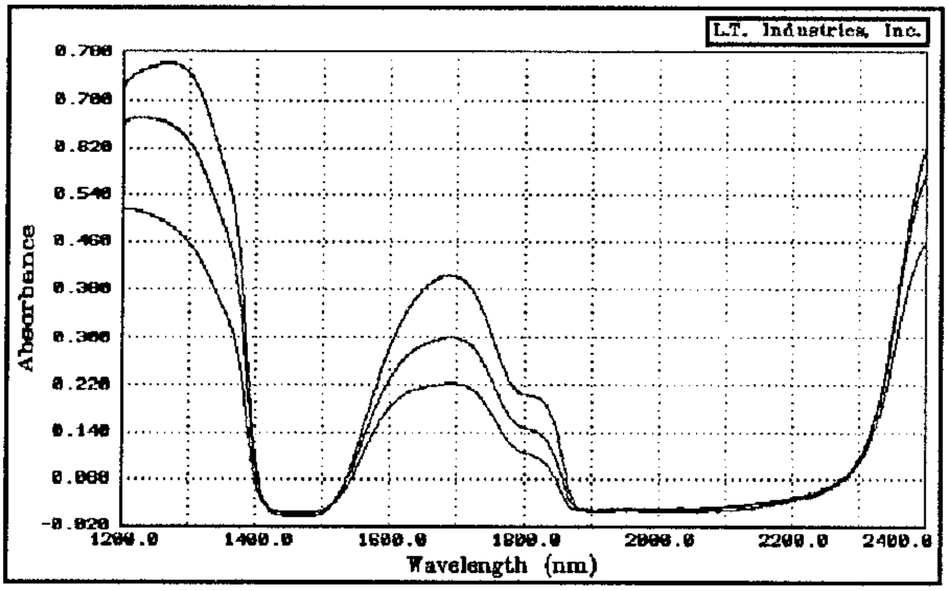

Fig.. 1. Typical absorbance spectra of the samples.

Also the first derivative of the spectra was generated (taking the derivative of the spectra removes the effects of baseline shifts). Typical first derivative spectra can be seen in Figure 2.

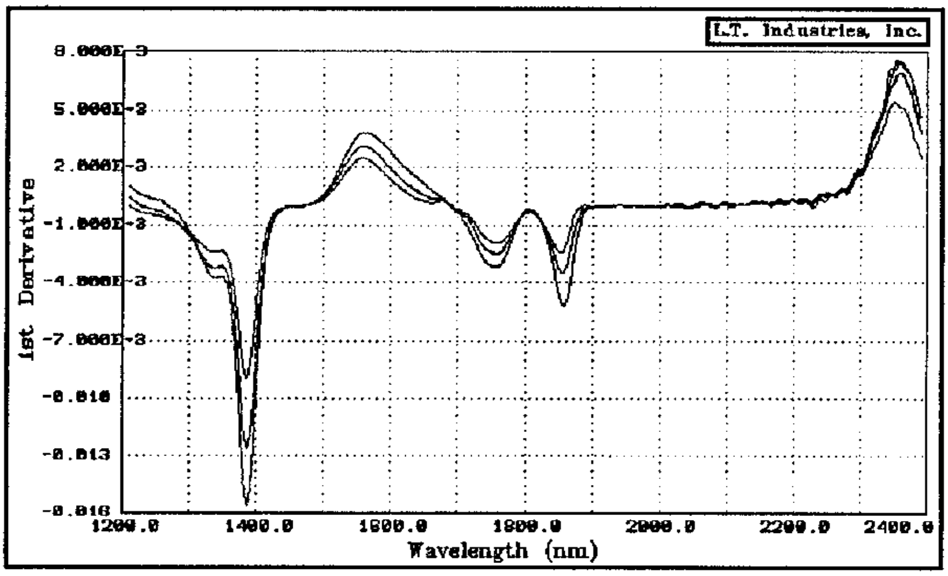

Fig. 2. Typical first Derivative spectra of the samples.

As it can be seen in the table 2, a very good correlation was found for all four ingredients (correlation coefficient values higher than 0.98), and the Standard Errors of Prediction, which match the Standard Error of the primary analytical method used to analyse the samples. Figures 3, 4, and 5, show graphs of the true analytical values for the calibration samples versus the values predicted by the NIR method. The graphs show the strong correlation for the three ingredients.

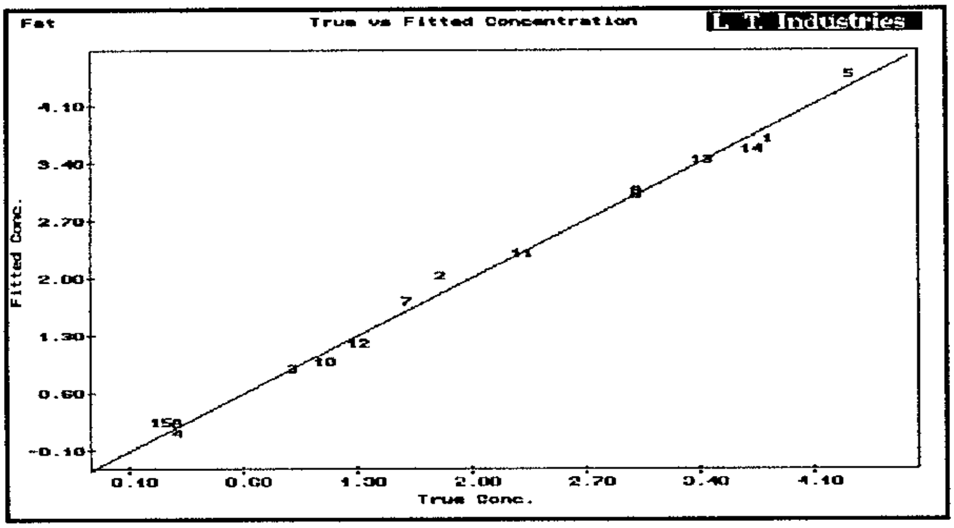

Fig. 3. Calibration curve (True vs. Fitted concentration) for Fat.

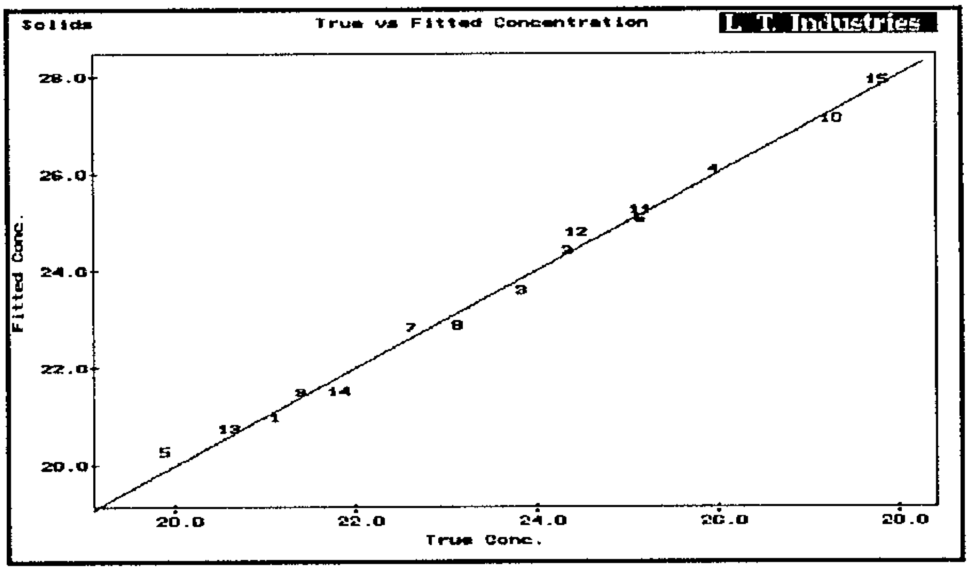

Fig. 4. Calibration curve (True vs. Fitted concentration) for Solids.

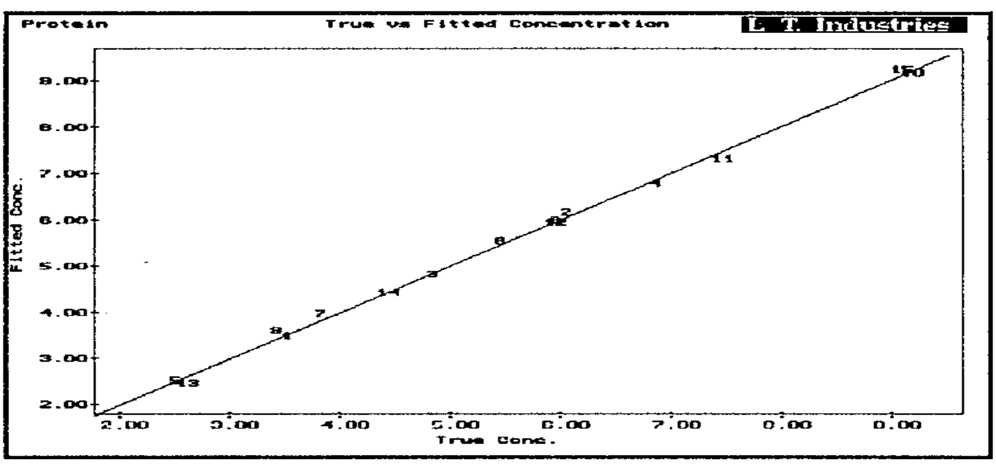

Fig. 5. Calibration curve (True vs. Fitted concentration) for Protein.

Conclusions

The on-line monitoring of the manufacturing process of dairy related products, as well as the at-line monitoring, is feasible with the use of Near Infrared technology. The technique allows the simultaneous measurement of several ingredients, such as Fat, Solids, Protein, Moisture, etc. and offers many advantages.

References

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