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The use of spectroscopic measurements from full scale industrial production to achieve stable end product quality

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The present study was conducted to investigate different approaches to improve product quality in an industrial setting, by utilizing spectroscopic measurements for process optimization where data are obtained directly from full scale production. The approach taken is to utilise spectroscopic techniques as a fingerprint of uncontrollable raw material variation. The FTIR spectra were used to predict the end product characteristics along with the process parameters instead of predicting previously defined characteristics of the raw material. The present case under study is from cheese production aiming for reduced variation in the dry matter content of the final product. Fourier transform infrared spectra (FTIR) are available at the plant, but not fully utilized. The results from the study showed that the FTIR spectra do contain significant information relevant for the end product characteristics which is not yet utilized, suggesting that FTIR spectra can be used for process optimisation to improve the stability of the quality. Different strategies to incorporate the spectral data are presented, and the usefulness of full scale production for process optimisation is discussed. The present study also contains optimisation of the production based on data modelling of the raw material variation, the process settings and the product quality over a long time period.

1. Introduction

For most industrial production processes it is important to achieve stable end product quality. This is, however, usually a challenging task due to inherent variability of the raw material. One important strategy for solving this is to measure the quality of the raw material prior to the processing and adjust the processes accordingly. This strategy requires appropriate and rapid measurements reflecting the relevant properties of the raw material, and reliable knowledge on how the raw materials in combination with the process settings affect the end product quality. Spectroscopic techniques are rapid and have the potential to unravel variation in the raw material influencing the end product quality. Normally, spectroscopic techniques are used to predict the characteristics of the samples investigated, like for example the content and composition of fat and proteins. Here we investigate the possibility of using spectroscopic techniques to predict directly the end product quality along with process parameters to obtain a process control system where the process is adjusted according to the raw material characteristics. To obtain information that is as relevant as possible for the production to be optimized, we collected data directly from the full scale productions.

The case study is related to the production of a semi-hard Gouda-type cheese where the dry matter content of the final cheese is the response parameter in focus. Dry matter content was selected because it has a strong impact both on the sensory quality of cheese and the economy of the production. Being able to control the dry matter content and reduce its variability are therefore important aspects of all types of cheese production.

A number of studies have shown that the FTIR spectra are influenced by a large number of organic molecules in milk (Hewavitharana & vanBrakel, 1997; Etzion, Linker, Cogan, & Shmulevich, 2004). (Jørgensen & Næs (2004); Jørgensen, Segtnan, Tyhold, and Næs (2004) have successfully used FTIR to predict cheese fines lost in cheese production, showing that FTIR has the potential of revealing raw material variability affecting the dry matter content of cheese.

The data used in the present study are collected from a production plant which uses FTIR spectroscopy routinely to obtain information on fat and protein content of the milk. An interesting question is whether the FTIR spectra contain more information to be used for improving the process models beyond the prediction of fat and protein. If so, optimisation of future

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production could potentially be made by establishing a model (an equation) based on calibration data where spectra of raw material, process and product quality are known. Based on this the process could be adjusted according to the raw material to achieve more stable end products.

The focuses of the present publication will be on the strategies used for data collection as well as the data analysis. There are essentially two closely related modelling aspects that will be covered. The first one is modelling the dry matter content as a function of chemical measurements and process conditions. The other modelling considered is to show how FTIR spectrum of the raw material can be better utilized to improve the prediction ability and hence reduce the end product variability.

The strategies used are of a general nature and not specific for cheese production. Therefore, methodological issues and strategies will be highlighted more than issues directly related to cheese production. The paper will illustrate various challenges encountered for this type of situations, for instance the use of full scale productions for exploring variation, model quality evaluation, the incorporation of multivariate spectroscopic data together with process settings and spectroscopic pre-processing.

2. Material and methods

2.1. Material

All cheeses in the present study are taken from a commercial production of a semi-hard, Gouda-type cheese (at Tine Midt-Norge, Ørland, Norway). The plant typically produces a total of 18,000 kg cheese per day.

Fat and protein content and their ratio as predicted by FTIR, plus the dry matter content and information on the controllable process settings have been observed daily during production of cheese over a period of one and a half year covering a total of 297 samples, and the dry matter content was observed for another one and a half year for validation. The process settings included the time and temperature of each process step, and the volumes removed and the volumes of water added. Dry matter content of the cheese were analysed by drying powder of the cheese in 102 °C for 20 h. The fat and protein content and the process settings were used to model the dry matter content. Based on the results of the data modelling, changes in the production scheme were implemented sequentially to allow validation of each step. The effect of the changes in the production scheme was observed by the standard deviation in the dry matter content as observed in the time period between each new implementation.

Within the first one and a half year of data collection, FTIR was collected for a total of 58 cheese productions in four time intervals spread over time to cover seasonal variation in the raw material. Spectra were collected both on the raw material and on intermediate products, but only FTIR from the raw material is included in the present publication as that was found to be most useful. These data were used in order to illustrate how FTIR can be used to improve the prediction performance of the end product quality.

2.2. FTIR spectra

FTIR measurements were performed using a Milkoscan FT 120 FTIR spectrometer from Foss, Hillerød, Danmark on 58 productions over four time intervals. The instrument is based on pipelining fluid samples through a homogeniser and a temperature regulating unit to a cuvette with a fluid volume of 50 μm. Each sample was measured using FTIR technology in the spectral region from 925 to 5011 cm^{-1} consisting of a total of 436 variables. FTIR spectra were observed in room temperature.

Duplicate spectra of the same milk sample were recorded and the average spectra were used for further analysis. The transmittance spectra were linearized and converted to absorbance values. The FTIR spectra of milk contain both chemical information about the constituents of milk and physical information due to different light scatter effects and instrumental effects. Often, scatter effects are related to the texture of the investigated material. To be able to interpret chemical differences, and to distinguish the chemical effects from the physical effects we investigated the use of Extended Multiplicative Scatter Correction (EMSC) (See the supplementary material) (Kohler, Kirschner, Oust, & Martens, 2005; Kohler, Zimonja, Segtnan, & Martens, 2008; Martens, Nielsen, & Engelsen, 2003; and Martens & Stark, 1991). The EMSC is here used without the inclusion of spectra from known substances. After pre-processing by EMSC, the 2. derivatives of the spectra were calculated by a Savitzky-Golay algorithm using second order polynomial and 9 smoothing points (Martens & Stark, 1991; Martens et al., 2003). The pre-processing of the spectra was conducted using Unscrambler software package (version 9.2; CAMO A/S, Oslo, Norway).

The spectral regions from 1543 to 1717 cm^{-1} and 3000–3300 cm^{-1} were initially removed from the spectra since these regions mainly contain information from water. Furthermore, the whole region from 1825 to 2812 cm^{-1} was removed as there is no relevant information present in this region. This resulted in a spectrum consisting of 220 variables.

2.3. Modelling

The end product characteristics are here considered as functions of the process settings for the production and the raw material properties, giving the model:

$$Y = f(P, R) + \epsilon$$

where Y represents the end product properties, R the raw material measurements, P the controllable process settings; time and temperature of each process setting, as well as the volumes removed and added, and ϵ the random residuals not accounted for by the systematic part of the model. As soon as f is determined using an appropriate regression method and relevant process data, it can be used for the purpose of for instance feed forward optimisation based on measurements of the raw material properties. It should be noted that in this paper we use the symbols in the equation in such a way that they represent a "phenomenon" not a data table. When comes to actual modelling, they are replaced by matrices and the modelling is done according to the procedures to be discussed below.

When little knowledge is available on the relation between the input and the output variables, as in the present case, it is natural to use empirical models fitted by calibration data. Since data from a real world industrial production are expected to be noisy and complex, using as simple models as possible is important. Based on the multivariate nature of the data, we chose the multivariate linear regression method called Partial Least Squares Regression (PLSR) (Martens & Martens, 2001, (p. 445); Martens & Næs, 1989, (p. 419); Næs, Isaksson, Fearn and Davies, 2002, (p. 344) and Wold, Martens, & Wold, 1984) using Unscrambler software package (version 9.2; CAMO A/S, Oslo, Norway). In all cases the variables were standardised by dividing by their standard deviation as the variables are on different scales. For PLSR the main information among the regressor variables (raw material and process settings) which is important for describing the response variable is compressed down to a few new variables, called PLSR factors. These are estimated as linear functions of the original variables, to give in decreasing order