CHARACTERIZATION OF MILK BY INFRARED SPECTROSCOPY

Caracterização de leite por espectroscopia infra-vermelho

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SUMMARY

The present paper reports on the optical characterization of milk in order to evaluate its composition, by means of non invasive techniques, the FT-IR (Fourier Transform Infrared) and NIR (Near Infrared) absorption.. Samples were diluted with water and the fat density was found to decrease linearly with the increase of water.

Index terms: Milk; fat; infrared spectroscopy.

1 INTRODUCTION

The determination of the main constituents that compose the milk is important in the dairy food industry, for establishing the milk value, for consumers' information and for quality control. Fat and total protein content of milk, have economic importance because, in most countries, milk trade is based on these components and the somatic cell counts.

It has been an attempt for replacing traditional reference methods (like Kjeldahl for protein, Röse-Gottlieb or Mojonnier for fat, and polarimetry for lactose) in the last decades, by application of spectroscopy techniques, in order to save time and to avoid waste. Since the development of the first commercial mid-infrared (IR) spectrophotometer by Goulden (GOLDEN,1964) and the contributions of Biggs (BIGGS, 1967;1972), the quantitative IR spectroscopy to milk analysis has turned into a standard AOAC method (BIGGS, 1972). But this method is slower and more expensive if we compare with the relative recent technique, the near-infrared spectroscopy of foodstuffs. The advantages of NIR spectroscopy include a higher rapidity and a simultaneous, non-destructive measurement of a number of milk constituents and potential for on-line analysis (SATO et al., 1987; RODRIGUEZ-OTERO et al., 1997; TSENKOVA et al., 1999).

Since the milk is composed of mostly water the only difference between various types should be the concentration of fat, with the other constituents being practically the same.

The purpose of this work is to study fat concentration in milk samples and indicate a way easy, fast and cheap to determine this percentage.

2- MATERIALS AND METHODS

2.1 Fat Milk Dilution

Commercially available homogenized fat and nonfat milks were used. To study the relationship of fat concentration and absorbance spectra the fat milk was diluted in water at percentages presented in Table 1. All samples were stored at room temperature and were only gently shaken before measured. The samples are nearly of the same age and sold in tetrabricks[â].

To guarantee the fat influence in suspected wavelengths, the spectra of nonfat milk were evaluated to confirm the absence of variation in the second derivate of absorbance's curve at this wavelength. Second derivative is known to enhance small differences in infrared spectra. This procedure was performed by using commercial software ORIGIN[â].

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2.2 Near Infrared Spectroscopy in milk

The absorbance properties of milk in the infrared part of spectrum are determined by the presence of certain structural groups in milk such CH, -OH, -NH which are responsible for vibration spectra in near infrared part of spectrum. The main components of milk have specific bands: fat (2340, 2310, 2270, 1780, 1730, 1720 nm), casein (2790, 2340, 2310, 2100, 1980, 1820, 1780, 1730, 1720, 1680, 1450 nm) and lactose (2340, 2100,1820,1450 nm).

2.3 Near Infrared Spectra

Transmittance spectra of 0,5 mm thickness milk samples were obtained with a two-arms UV/Vis/NIR Spectrometer (Lambda 900, Perkin-Elmer) using a sample holder constructed from two clean microscope slides separated by a 0,5 mm thick material with an upside aperture to introduce the milk using a syringe's needle. Spectra were measured in the wavelength range from 700 to 2400 nm, at 2 nm intervals, with velocity of 250 nm per minute and were recorded in the linked computer as absorbance.

The background spectrum was scanned at the beginning of the measurement session with no sample. Spectra of the sample holder empty were scanned before and after the analysis of all samples.

2.4 Near Infrared Data Treatment

Data were analyzed by absorbance spectra and its second derivate transformation. We could compare our results with Tsenkova et al. (TSENKOVA et al., 1999; 2000; 2001), which had determined the characteristic absorption bands of fat, lactose and protein for raw milks. In this work we are only interested in fat absorption bands, and we used only the spectra range of fat with greater split, in second derivate transformation, between all samples.

3 RESULTS AND DISCUSSION

Due strong absorbance by groups O-H in water, two bands around 1448 and 1932 nm dominated the spectra. The characteristic absorption bands of fat, lactose and protein were very weak in comparison with this two water bands. It can be seen in Figure 1 the spectra of the fat milk without water dilution.

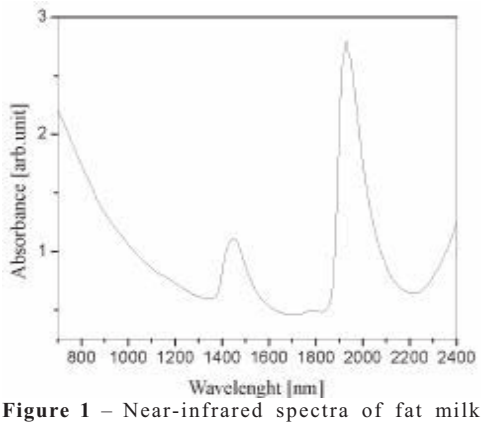

Figure 1 – Near-infrared spectra of fat milk (sample 1).

3.1 Fat Milk Dilution

In order to determine one of the bands of fat in the spectra we analyzed diluted samples. The spectrum baseline shifted downward with the decrease of fat as can be seen in Figure 2.

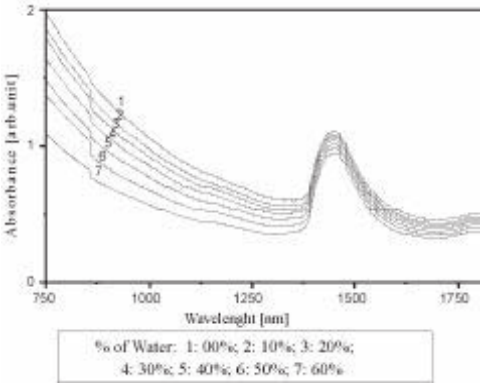

Figure 2 – Near-infrared spectra of milk samples diluted with water.

Table 1 – Percentage of water and fat milk at each examined sample.

Sample	Water %	Fat Milk %
1	00%	100%
2	10%	90%
3	20%	80%
4	30%	70%
5	40%	60%
6	50%	50%
7	60%	40%

To determine the characteristic absorption range of fat the computation of second derivates of spectrum was used to allow the resolution of overlapping peaks and removal of baseline variation. Figure 3 show the second derivate transformation of absorbance spectra of all seven samples of dilution analysis.

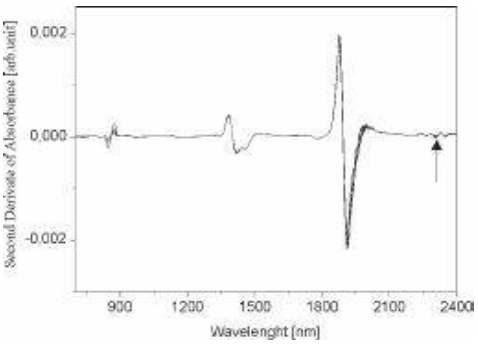

Figure 3 – Second derivate transformation of spectra milk samples.

Near 2308 nm can be noted the split of the different sample's spectra. To better visualization the region had been amplified in Figure 4, which shows a gradually upward shift whenever the percentage of water gets large. Is reasonable expect a linear dependence of fat concentration and absorbance value at the wavelength 2308 nm because the difference at each neighbor samples seem to be equal.

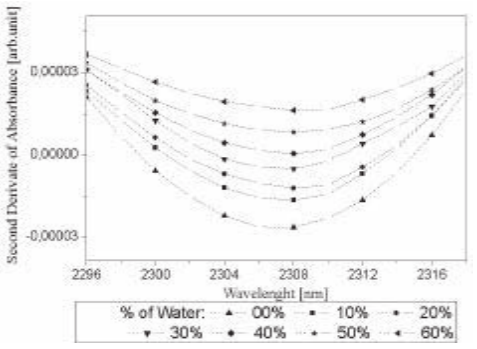

Figure 4 – Second derivate transformation of spectra milk samples near 2308nm.

3.2 Comparison between nonfat and fat milk

To confirm that the split in 2308 nm is originated by different fat concentration, we did the spectra of nonfat milk, which the only significant difference of fat milk is the absence of fat globules. On Figure 5 becomes clearly that nonfat milk doesn't have any variation in wavelength 2308nm and therefore the second derivate transformation peak in 2308nm is originated by fat different concentration of the seven samples of the dilution analysis.

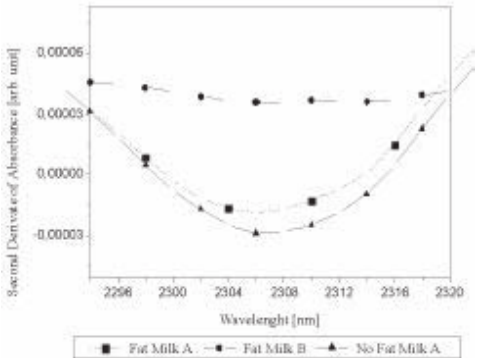

Figure 5 – Comparison of second derivate transformation of spectra milk samples, fat milks and nonfat milk, near 2308nm.

These results are entirely in agreement with Tsenkova et al. (TSENKOVA et al., 1999; 2000; 2001), which determined the region near 2308nm as one of the fat absorption bands.

3.3 Linear dependence of water dilution

The dependence of the absorbance value in the wavelength 2308nm by the dilution of milk with water is illustrated in Figure 6, which shows that this dependence is linear as expected.

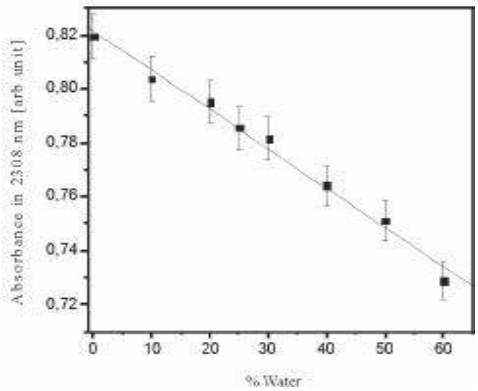

Figure 6 – Absorbance value of the milk samples in the wavelength 2308 nm.

This linear dependence is also seen between the area of the absorbance spectra and the dilution of milk. It is represented in Figure 7.

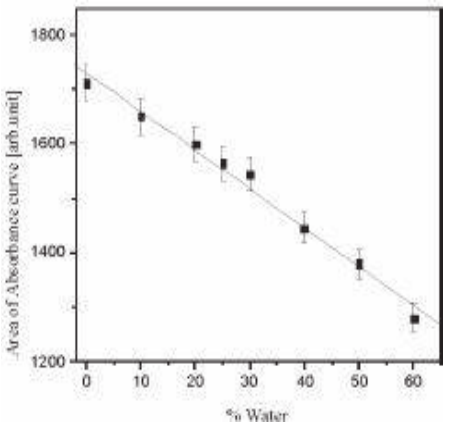

Figure 7 – Absorbance spectra's integral.

The angular coefficient of this straight line is greater than the one of linear fit by absorbance value in 2308 nm. This is the result of the addition of variations of the other components of milk such lactose and protein, and the existence of more than one characteristic region for each major component.

4 CONCLUSIONS

The conclusion that the wavelength 2308 nm can be use to determine the fat concentration of milk without other components influence is immediate. In addition the value of absorbance has linear dependence with the fat concentration in this wavelength. And the area under the absorbance's curves has linear dependence with the water dilution, what means that the other major components of milk decrease linearly with the water addition.

SUMÁRIO

O trabalho apresenta a caracterização óptica do leite com o objetivo de quantificar sua composição por meio de uma técnica não invasiva, a absorção infravermelha por transforada de Fourier (FT-IR). As amostras foram diluidas com água e a densidade de gordura apresentou diminuição linear com o aumento da diluição com água.

Termos para indexação: leite; gordura; absorção infravermelha.

5 ACKNOWLEDGEMENTS

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