On line infrared measurements in food processing

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TITLE: ON LINE INFRARED MEASUREMENTS IN FOOD PROCESSING

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SUMMARY : Infrared spectrometry measurements were tried in the laboratory, in order to evaluate the feasability of the non destructive control of the apple sugar content on line, fruit by fruit and the piloting of the grape must sugar degradation during alcoholie fermentation. Sugar content obtained by reflectance through the epiderm of apples was predicted with a standard error of prediction (S.E.P.) of 6,8 g/l. The composition of grape musts in alcohol, sugars glycerin could be predicted with S.E.P of 0,15 vol %, 2,5 g/l and 0,25 g/l respectively.

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Abstract

Infrared spectrometry measurements were tried in the laboratory, in order to evaluate the feasability of the non destructive control of the apple sugar content on line, fruit by fruit, and the piloting of the grape must sugar degradation during alcoholic fermentation. Spectra data from apples were obtained by reflectance through the epiderm of fruits: sugar content in more than 320 apples was predicted with a standard error of prediction (S.E.P.) of 6.8 g/l and a coefficient of correlation of 0.96 .The composition of grape musts in alcohol, sugars, glycerin, during their fermentation could be predicted with a S.E.P. of 0.15 vol%, 2.5 g/l, 0.25 g/l respectively.

1. Introduction

The sugar content of the fruit flesh is characteristic of gustative quality. Presently, a destructive control is operated. Quality is rated by a refractive index measurement on juice: the soluble dry matter of the mature fruit is primarily made of sugars. On line measurements of sugar content, fruit by fruit, would enable to grade them according to their gustative quality. Consequently, these measurements would allow to warrant a minimal quality to the consumers.

The control of the temperature as a function of the sugar consumption, will allow to pilot better the alcoholic fermentation and predict the fermentation breakdowns.

R. GIANGIACOMO et al. predicted concentrations of individual sugars in dry mixtures by near-infrared reflectance spectroscopy with standard errors of prediction (S.E.P.) always better than 1.5% (1). G. G. DULL and R. GIANGIACOMO (2) carried on the determination of these sugars in aqueous solutions: the 95% confidence limits of the method for glucose, fructose and sucrose were 1.3, 1.0, 0.9% respectively. E.LANZA and B.W.LI (3) operated in transmission mode to predict the total sugar content of a variety of fruit juices. The best results were obtained when separated calibrations were used for each type of juice. For orange juice, S.E.P. was 0.25% (or 2.5 g/l).

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Several authors worked on finished wines. K. J. KAFFKA and K. H. NORRIS (4) made a first evaluation of infrared absorption technique for rapid determination of the composition wine. The composition data from chemical analysis were determined for 26 wine samples by linear regression analyses. S.E.P. was less than 0.1 vol% for alcohol, 2 g/l for sugar and 1 g/l for acid. M. T. CABANIS (5) confirmed these results for the alcohol content. The 99% confidence limits was 0.1 vol% and the reference method was the pycnometry with a precision of 0.02 vol%. J. C. BOUVIER (6) studied the effect of glycerin on the determination of the alcoholic degree measured by near infrared spectrometry

2. Sugar content by near infrared reflectance through the epiderm

2.1 Materials and methods

The data acquisitions have been performed on a set of 163 Golden Delicious apples, selected by MONOPRIX, a french supermarket company. Two near infrared spectra per apple were recorded, on two opposite zones at the equatorial level to take into account the eventual variation of the composition attributable to a difference in the sun lighting. The refractive index of the flesh under these opposite zones was used as reference. Therefore, these data corresponded to 326 samples.

The spectral data were collected using a Technicon Infraalyser 500, a grating spectrometer equipped with an integrating gold sphere. Data, ranged from 1100 nm to 2500 nm by 4 nm steps, were converted in log 1/R (R = reflectance data).

2.2 Results and discussions

Several methods of statistical analyses of the spectral data were tested. Some elementary spectra more or less typical of the main components (water, sugars,...) were extracted from the morphological analysis of the previous spectra. The principal components analyses and the factorial correlations analyses give new coordinates systems well adaptated to the description of these data. But the prediction was not appreciabily improved with regard to conventional techniques by multilinear regressions with 3, 4 or 5 wavelengths. The figure 1 illustrates the results of one of better regressions.

Sugar content was predicted with a standard error of prediction (S.E.P.) of 6.8 g/l and a coefficient of correlation of 0.96. 95% confidence limits was 1.4% or 14 g/l. Table 1 shows an attempt of fruit classifications in 4 gustative classes. These sortings were not perfect because some fruits were assigned to a classe immediately above or below down their "true" one. Meanwhile, more than 89% of fruits were correctly classified and only 5% were underclassified.

The OECD standards for fresh market aim at defining a small number of quality grades, in terms of size, colour and surface defects, with an error rate of 10%. The sugar assay of the fruit by diffuse infrared reflectance apparently allows to grade the fruit with the same accuracy.

3. Determination of main components of grape must during the fermentation by near infrared spectroscopy

3.1 Materials and methods.

About 15 must samples were taken during each fermentation. Samples were filtered to eliminate the yeast and stabilized with mustard oil. The enzymatic analyses were made by the enology laboratory of INRA at Narbonne. Accuracies of these reference analyses were estimated from 0.1 to 0.2 vol% for alcohol, 0.1 g/l for glycerin and, for each sugar, 0.2 g/l between 0 and 20 g/l content, from 1 to 3 g/l over 20g/l content.

Infrared transmittance measurements were acquired by an IRTF NICOLET 7199 monobeam spectrometer with Michelson interferometer. The sample were contained in an infrared glass cell with an optical path of one millimeter. Data were recorded as optical density (log Iw - log Is, where Iw is the light intensity transmitted by water and Is the one transmitted by the sample). Figure 2 represents the evolution of must infrared spectrum, between 4830 cm-1 (2070 nm) and 4200 nm (2380 nm), during a fermentation.

3.2 Results and discussion

The statistical analyses made on the data coming from two fermentations showed that it is possible to determine the major components of the fermenting musts with more than 0.99 correlative coefficient. Each individual sugar, fructose and glucose, was predicted with a S.E.P. of 1.5 g/l. This error was reduced to about 1 g/l for one fermentation. Alcohol and glycerol was predicted with a S.E.P. of 0.15 vol% and 0.25 g/l respectively. The error for alcohol was about 0,1 vol% for one fermentation.

The accuracies of the reference enzymatic methods used do not allow to know the real limits of the infrared one. The infrared method seems to be a little bit more accurate of other physical methods (flow of CO2, specific gravity, refractive index). But, at this moment, these performances are perhaps insufficient to use this sophisticated method in line. Further study has to be undertaken to determinate if the infrared method can measure, with a better accuracy, residual sugar content during the end of the fermentation and thus predict with certainty when it is finished.

4. Conclusion

The work in laboratory showed that it is possible to grade fruit in terms of gustative quality using the near infrared reflectance. But to use this technology on line, a sensor able to measure the sugar content, apple by apple, in real time (about 5 fruits by second) has to be developped. Probably at least two opposed measurements will have to be recorded by fruit to take into account the chemical heterogeneity of some fruits. The near infrared spectrometry gives a good prediction of the main components of fermenting musts, but its accuracy is perhaps not enough better than other physical methods, less expensive and sophisticated, unless futher study shows that a better prediction of the residual sugars, during the end of fermentations, could be obtain.

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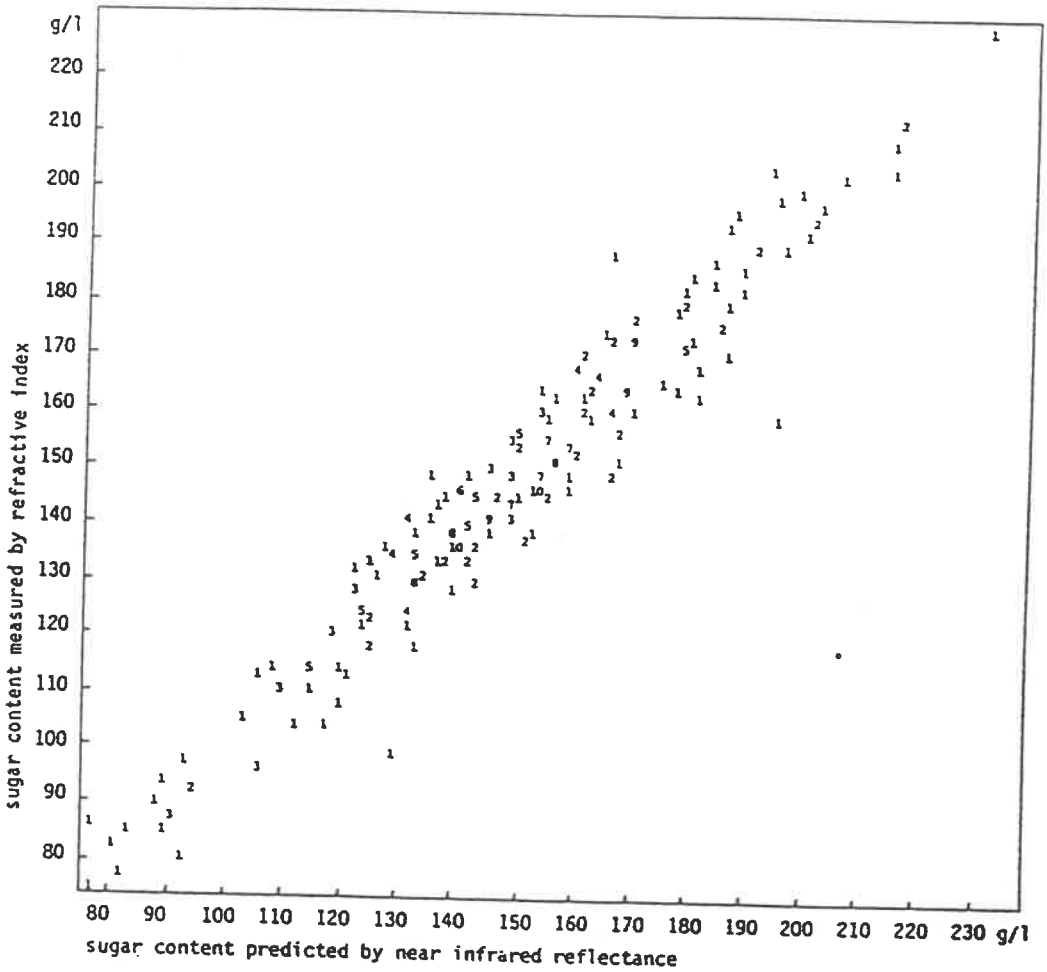

fig. 1 Prediction of the sugar content (refractive index) using near-infrared reflectance through the epiderm of apples (numbers in figure = numbers of samples in this position)

gustative classification using refractive index of juice		< 100 g/l	> 100 g/l < 130 g/l	> 130 g/l < 170 g/l	> 170 g/l	limits of classes
	4			6	78	> 170 g/l
	3		8	166	6	< 170 g/l > 130 g/l
	2	1	34	7		< 130 g/l > 100 g/l
	1	15	4			< 100 g/l
	CLASSES	1 bad fruits	2	3	4 better fruits	
		gustative classification predicted by near infrared reflectance				

table 1. attempt of gustative classification using near infrared reflectance (326 samples : Golden Delicious apples)

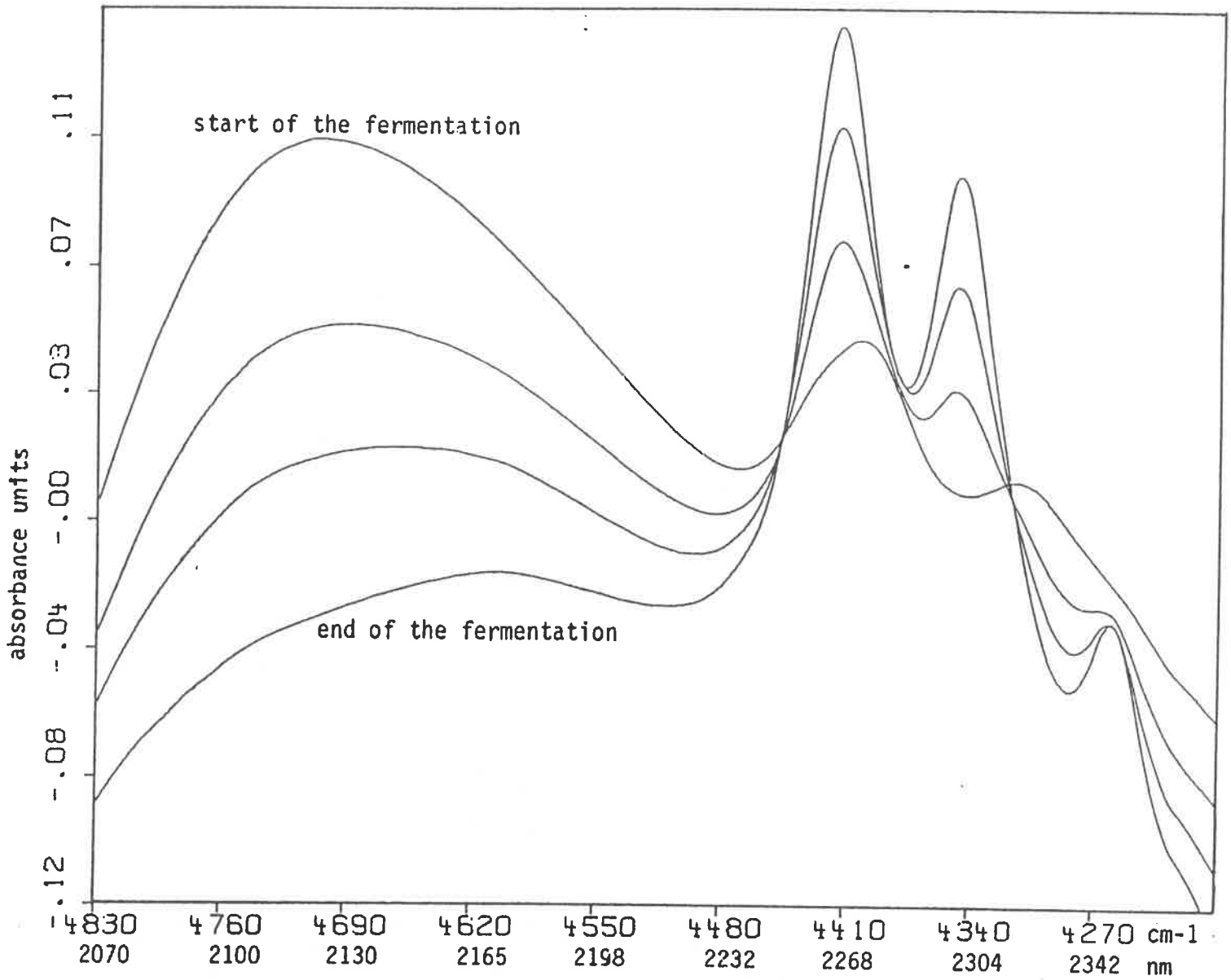

figure 2. Evolution of near infrared spectra, during a grape must fermentation