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Quality Evaluation of Sugar Beet *(Beta vulgaris)* by Near-Infrared Spectroscopy

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The legal method (polarimetric measurement) for the determination of sucrose content and the wet chemical analysis for the quality control of sugar beet uses lead acetate. Because heavy metals are pollutants, the law could forbid their use in the future. Therefore, near-infrared spectroscopy (NIRS) was evaluated as a procedure to replace these methods. However, there are alternatives to lead clarification, such as the use of aluminum salts, which have been applied at many sugar companies. The real advantage of NIRS is in speed and ease of analysis. The aim of this study was to determine simultaneously the concentration of several components which define the industrial quality of beets. The first objective was the determination of sucrose content, which determines the sugar beet price. The standard error of prediction (SEP) was low: 0.11 g of sucrose/100 g of fresh beet. NIRS was also able to determine other beet quality parameters: brix, marc, glucose, nitrogen, sodium, potassium, sugar in molasses (i.e. sucrose in molasses), and juice purity. The results concerning brix, marc, sugar in molasses, and juice purity were satisfactory. NIRS accuracy was lower for the other parameters. Nevertheless, RPD (ratio standard deviation of concentration/SEP) and RER (ratio concentration range/SEP ratio) show that NIRS might be used for the sample screening on nitrogen, potassium, sodium, and glucose content.

KEYWORDS: NIRS; sugar beet; quality; multivariate regression; sucrose; nonsugar

1. INTRODUCTION

In the sugar beet factories, sucrose content is determined on receipt of the beets. The procedure for sampling and analyzing (*1*) beet is the same for all French factories and is defined by law (*2*): the beet is rasped, lead acetate is used for clarification, and the percentage of sucrose is determined by polarimetric measurement of juice. Moreover, in sugar refinery laboratories, several components are measured on this juice clarified by lead acetate: glucose, potassium, sodium, and nitrogen. These compounds, which are determined directly in sugar beets, allow the calculation of two industrial parameters: sugar in molasses and juice purity. Sugar in molasses is an estimation of the loss of sucrose (i.e. sucrose, which cannot be extracted) and the juice purity (estimated by the analysis of sugar beets) gives an estimation of the juice that will be obtained in the factory. These parameters and two others (brix and marc) define the industrial quality of sugar beets.

Several problems are raised by the use of lead acetate: lead is a pollutant and the government regulation of heavy metal uses is becoming more and more stringent. NIRS is a suitable replacement method and might be used in sugar factories, by beet seed producers, and by beet growers' laboratories. The most attractive features of NIRS are its speed, its low cost, and its environmentally friendly aspect (*3*).

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In previous articles, we demonstrated that near-infrared spectroscopy (NIRS) data can be used to solve classification problems and determine qualitative parameters such as geographical origin and disease resistance (*4*). The aim of this study was to determine sugar beet quality parameters by near-infrared spectroscopy. The NIRS quantitative analyze protocol was applied to determine several component contents, i.e. sucrose (S), brix, marc, glucose (Glu), sodium (Na), potassium (K), nitrogen (N), sugar in molasses (SM), and juice purity (JP).

2. MATERIALS AND METHODS

2.1. Sugar Beet Origins and Sample Preparation. More than 2700 sugar beet samples (**Table 1**) were collected from 15 sugar factories in different production areas of France. The samples of various quality and variety were collected several times during the period from 1999 to 2002 to create a large and robust database.

The protocol for sample preparation was the standard method used in sugar factories. Twenty kilograms of beets were washed to take the soil trace out. The top of the beet was removed. The root was sampled by use of a multiple rasp (Parmentière model, Azoir La Ferriere, France) to produce about 1 kg of fine brei. The sample was homogenized with an approved instrument (IUA model, Saint Quentin, France) for 7 s. Because of the oxidation and loss of moisture, the beet was analyzed by NIRS and by wet chemical analysis just after the preparation.

2.2. Wet Chemical Analysis (WCA). *2.2.1. Sucrose, Glucose, Nitrogen, Sodium, and Potassium Determination.* The sucrose (S), glucose (Glu), amino nitrogen (N), sodium (Na), and potassium (K) concentrations were determined for each sample. The samples were analyzed twice and mean values were used.

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Table 1. Wet Chemical Data

parameter	S[a]	brix[a]	marc[a]	JP[a]	SM[a]	N[a]	K[b]	Na[b]	Glu[a]
sample no.	2735	1980	416	2060	2060	2060	2060	2060	2060
minimum	14.38	17.00	3.48	91.31	0.87	0.21	2.15	0.02	0.01
maximum	20.62	26.24	5.33	96.98	2.45	2.33	6.25	1.48	0.34
mean	17.54	20.85	4.36	95.55	1.21	0.62	3.60	0.33	0.05
standard deviation	1.04	1.36	0.32	0.68	0.19	0.24	0.66	0.23	0.03

[a] Units: g/100 g. [b] Units: mmol·kg^{-1}.

For 26 g of sugar beet brei, a weight of 177 g of lead acetate solution was added. The solution was blended for 5 min and filtered on a simple filter paper (*5*). Wet chemical analyses (WCA) were done on clarified juice. The sucrose content was determined by a polarization measurement (*6*). Sodium and potassium rates were measured by flame-photometry (*7*). Amino nitrogen was estimated by the colorimetric method (*7*), using ninhydrine (Verbièse, Wasquehal, France). Glucose was determined by enzymatic test (GOD-PAP method, reference L94111) (*8*) provided by Hycel (Pouilly en Auxois, France). Glucose, nitrogen, sodium, and potassium were measured by an automatic continuous-flow instrument (LCA instruments, La Rochelle, France).

2.2.2. Brix. Brix is the percentage of dry matter in sugar beets. Sample preparation was different for the brix measurement. About 100 g of beet brei were weighted and centrifuged to produce a dark juice. This juice was filtered and analyzed by a refractometer (Mettler Toledo, Viroflay, Fance).

2.2.3. Other Industrial Parameters. (a) Marc. Marc is the sugar beet dry matter that is insoluble in water at 50 °C and in ethanol. A weight M (between 10 and 20 g) of beet brei was weighted. The sample was ground in distilled water (100 mL, 50 °C) and filtered. This step was done four times. Then the sample was washed with 100 mL of ethanol (50%). The sample was filtered and dried at 105 °C until the weight M' become stable. The value of marc was calculated as follows: marc (%) $= (M'/M) \times 100$.

(b) Sugar in Molasses (i.e. Sucrose in Molasses). The loss of sugar in molasses (SM) was determined with the contents of glucose, potassium, sodium, and nitrogen (measured in the sugar beets) by an empirical equation (*9*)

$$m_{SM}(\%) = a_1 m_{(K+Na)} + a_2 m_{nitrogen} + a_3 m_{glucose} + a_4$$

with the coefficients $a_1 = 0.14$, $a_2 = 0.25$, $a_3 = 3.3$, and $a_4 = 0.3$, and $m_{(K+Na)}$, $m_{nitrogen}$, and $m_{glucose}$ were respectively the content of potassium and sodium (mmol/kg of sample), the content of nitrogen (g/100 g of sample), and the content of glucose (g/100 g of sample).

(c) Juice Purity. The purity of the juice (JP) is the ratio (weight of sucrose/weight of dry matter) in the sugar factory juice. By analyzing sugar beet, we were able to predict by an empirical formula the purity of the juice obtained in the factory.

The formula was as follows (*9*)

$$JP = 99.36 - [14.27(m_{(K+Na)} + a_2 m_{nitrogen})/m_{sucrose}]$$

with $m_{sucrose}$ being the content of sucrose (g/100 g).

2.3. Near-Infrared Spectroscopy. A NIR reflectance spectrophotometer (model 6500, Foss NIRsystem, Silver Spring, MD) with a large cup (Natural product sample cup IH 0314P) containing 100 g of beet brei was used. During 1 min, the reference (ceramic) was scanned 10 times then the beet sample was scanned 20 times at a wavelength ranging from 400 to 2498 nm (**Figure 1**). The resolution, i.e., band-pass, was 10 nm and the spectrum is sampled every 2 nm. Between two samples, the cup was washed with distilled water at room temperature and dried. The washing and the drying steps took 2 min. Just after the NIR measurement, wet chemical analyses were realized.

2.4. Spectral Pretreatments. The NIR spectra pretreatments included standard normal variate (SNV), detrending (D) (*10*) algorithms, and the second derivative (*11*, *12*), the latter to enhance the spectral information and to reduce the baseline drift. Details concerning the choice of pretreatments were described in a previous work (*5*).

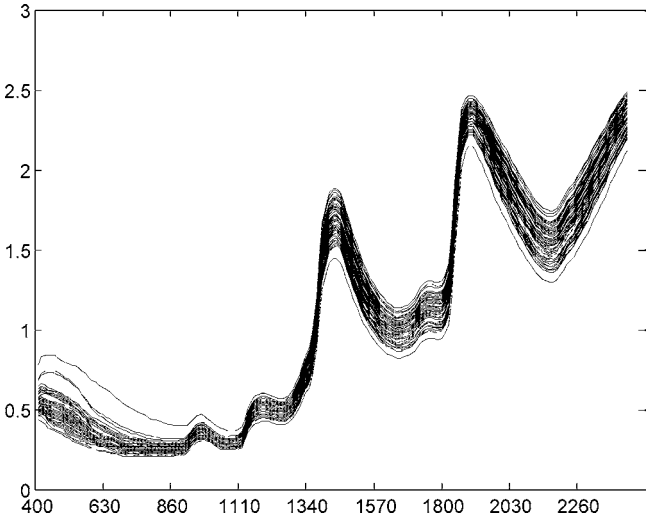

Figure 1. NIRS sugar beet spectra: *x*-axis, wavelengths; *y*-axis, absorbance; 400–750 nm is the visible range (variability due colors of the samples) and 750–2500 nm the NIR range. The water peaks are at 1450 and 1950 nm. The sucrose bands are described in **Figure 4**.

Table 2. Statistical Indicators for Calibration and Validation

parameters	S[b]	brix[b]	marc[b]	JP[b]	SM[b]	N[b]	K[c]	Na[c]	Glu[b]
				calibration					
sample no.	2210	1025	218	994	994	994	994	994	994
PLS terms	11	9	10	10	15	15	8	5	13
SEC[a]	0.09	0.17	0.11	0.29	0.07	0.10	0.38	0.12	0.01
R^2	0.99	0.98	0.91	0.79	0.75	0.74	0.58	0.42	0.49
				validation					
sample no.	525	955	198	1066	1066	1066	1066	1066	1066
SEP[a]	0.10	0.19	0.13	0.31	0.08	0.11	0.41	0.14	0.01
bias[a]	–0.01	0.00	0.00	0.03	–0.01	0.00	–0.01	–0.02	0.00
SEP(C)[a]	0.10	0.19	0.13	0.31	0.08	0.11	0.41	0.14	0.01
Slope	1.00	1.01	1.02	0.95	0.97	0.94	0.93	0.81	0.71
R^2	0.99	0.98	0.83	0.74	0.71	0.64	0.48	0.32	0.31
RPD	10.72	7.16	2.42	2.20	2.38	2.24	1.61	1.63	2.47
RER	62.40	48.63	14.02	18.23	19.75	20.19	10.05	10.43	27.33

[a] Unit of the component. [b] Units: g/100 g. [c] Unit s: mmol·kg^{-1}.

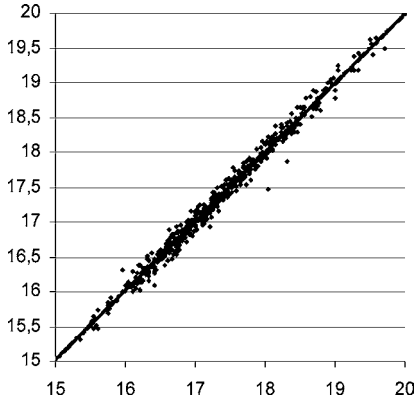

Figure 2. Validation results for the sucrose content: *x*-axis, lab values; *y*-axis, NIRS predicted values. The values are given in g/100 g.

2.5. Model Development and Statistical Indicators. The same protocol was applied for all the components. Two sample sets were prepared for calibration and validation. The samples were randomly distributed among the calibration and the validation sets. The data sets are described by **Table 2**.

The regression method was the modified partials least squares (*13*). The modification involved standardization of the residues after each iteration of the algorithm (*14*). Cross validation was used: the optimum number of terms for the calibration that minimized overfitting was based on the standard error of cross validation (SECV). The approach was as follows: 90% of the samples from the calibration set were used for

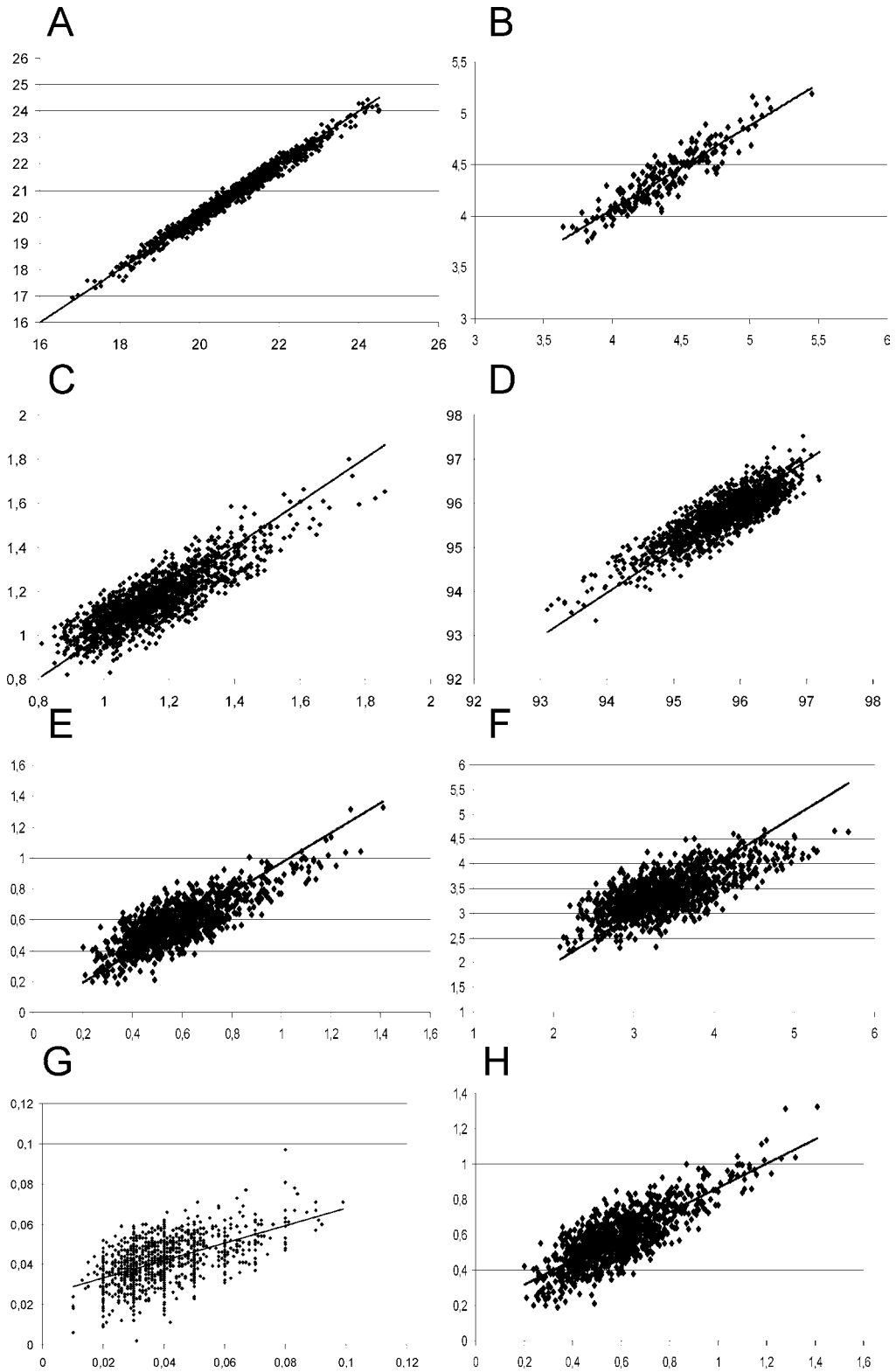

Figure 3. Validation results of the NIRS model for height parameters: *x*-axis, lab values; *y*-axis, NIRS predicted values; (A) brix, (B) marc, (C) juice purity (JP), (D) sugar in molasses (SM), (E) nitrogen, (F) potassium (K), (G) glucose (Glu), and (H) sodium (Na). The values are given in g/100 g except for sodium and potassium values which are given in mmol·kg^{-1}.

calibration and in the remaining 10% the standard error of prediction was calculated. This operation was done 10 times. Each time a different group was used for calibration and prediction. The final model was developed with the total samples of calibration set by using the number of factors with the lowest SECV (*15*).

The accuracy of a prediction model was evaluated by a low standard error of calibration (SEC), a low error of prediction (SEP), a low SEP with bias correction (SEP(C)), a high correlation coefficient (R^2), and a low bias. The units of SEP, SEP(C), and SEC were grams of sucrose per 100 g of beet (formulas: see ref 5).

Two other parameters (*16*) are used: the ratio (standard deviation of concentration/SEP), called RPD, and the ratio (concentration range/SEP), called RER.

2.6. Software and Hardware. Models were computed with Winisi (Infrasoft, Port Matilda, USA). The computer had an AMD Athlon processor (1.4 GHz) with 768 Mb of RAM.

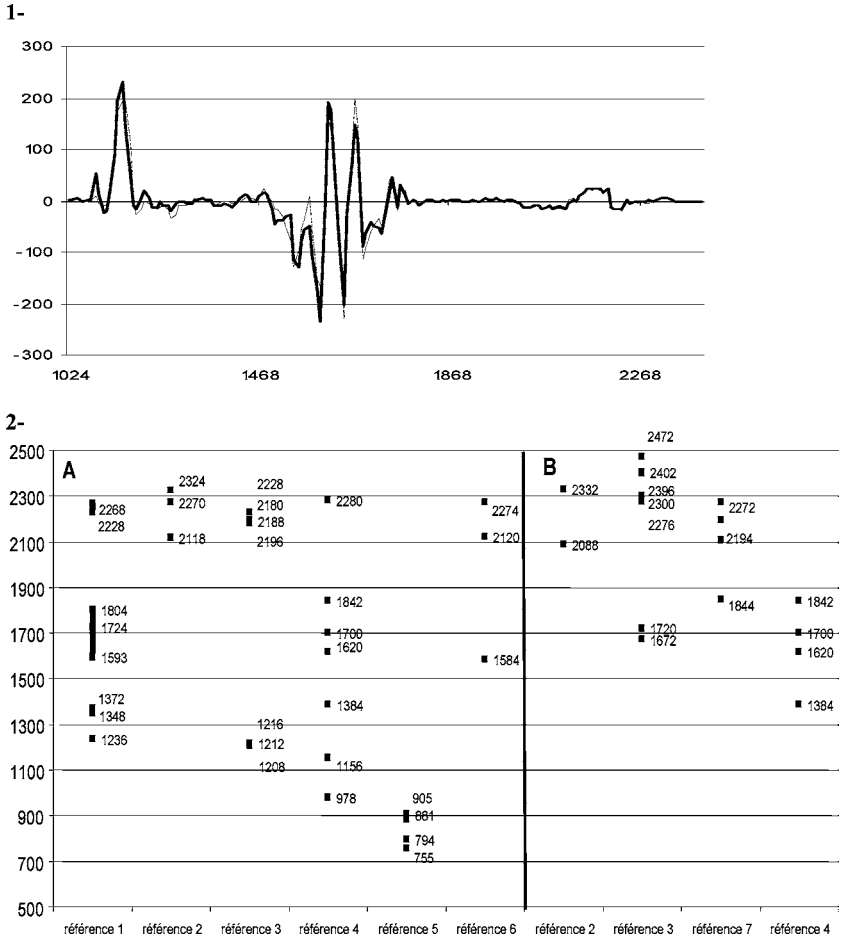

Figure 4. Regression coefficients for the sucrose model and comparison with previous studies. (1) Regression coefficients for the sucrose model: *x*-axis, wavelengths; *y*-axis, regression coefficients in gray for the model of 1999/2000 and in bold for the model of 1999/2002. (2) Comparison with previous studies for (A) wavelengths dues to sucrose and (B) wavelengths dues to total sugar: Reference 1, our results, sugar beet, reflectance; Reference 2, Robert et al. (*17*), aqueous solutions, transmittance; Reference 3, Cho et al. (*20*), apple, reflectance; Reference 4, Salgo et al. (*26*), sugar beet, reflectance: Reference 5, Miyamoto and Kitano (*18*), mandarin, transmittance; Reference 6: Rambla et al. (*19*), sugar beet, reflectance; Reference 7, Li et al. (*21*), orange juice, transmittance.

3. RESULTS AND DISCUSSION

3.1. WCA Data. Table 1 shows the number of samples, the mean, and the standard deviation of parameters. The standard deviation for the sucrose content or brix was relatively high. Nevertheless for SM, marc, N, Na, and glucose, the standard deviation was low. The concentrations of some components (like glucose) were quite constant even thought the samples had been collected and analyzed over a period of 4 years. However, the number of samples was sufficient to assume that our data sets were representative of the French sugar beets.

3.2. Calibration and Validation Results. The calibration and validation statistics are shown in **Table 2**. SEC and R^2 proved the validity of calibrations for sucrose, brix, and marc. Concerning JP, SM, and N, the value of R^2 in calibration was fair. However, the calibration results for K, Na, and Glucose were not satisfactory (low R^2).

Figures 2 and **3** represent the validation plot. As we see, NIRS was an accurate method to determine sucrose content and brix: SEP is low, R^2 is relatively high, and RPD is higher than 7 (**Table 2**). We can notice brix and sucrose are highly correlated because sucrose is 80% of the dried matter (brix) in sugar beet. This fact explains why if sucrose is well predicted, brix is accurately determined by NIRS too.

NIRS was able to predict values of marc, SM, JP. The statistical indicators R^2, RPD, and RER were higher than 0.7, 2, and 10, respectively.

The validation results for N, Glu, Na, and K were not satisfactory. Nevertheless, RPD values are between 1 and 3 and RER values were higher than 10. The calibration models might be used for the screening of sample. To have an accurate value, samples need to be analyzed by WCA.

RPD and RER are useful indicators. The RPD and RER ratio relates the SEP to the variance and range in the original reference data. The RPD should ideally be at least three and the RER at least ten (*16*). When the range and the standard deviation are low, the values for R^2 and RPD cannot be very high. Nevertheless, if the RER is higher than 10, it indicates that the model is able to predict the required concentration with an accuracy of at least one-tenth of the range. This accuracy can be considered as acceptable for certain applications.

We notice the model accuracy depends on the application. On one hand, sucrose content determination by NIRS needs to be accurate because the NIRS value will be used for the grower payment. On the other hand, the determination of the other parameters need not be as accurate as the sucrose content: these values are used to compare samples, to discriminate high from low concentrations.

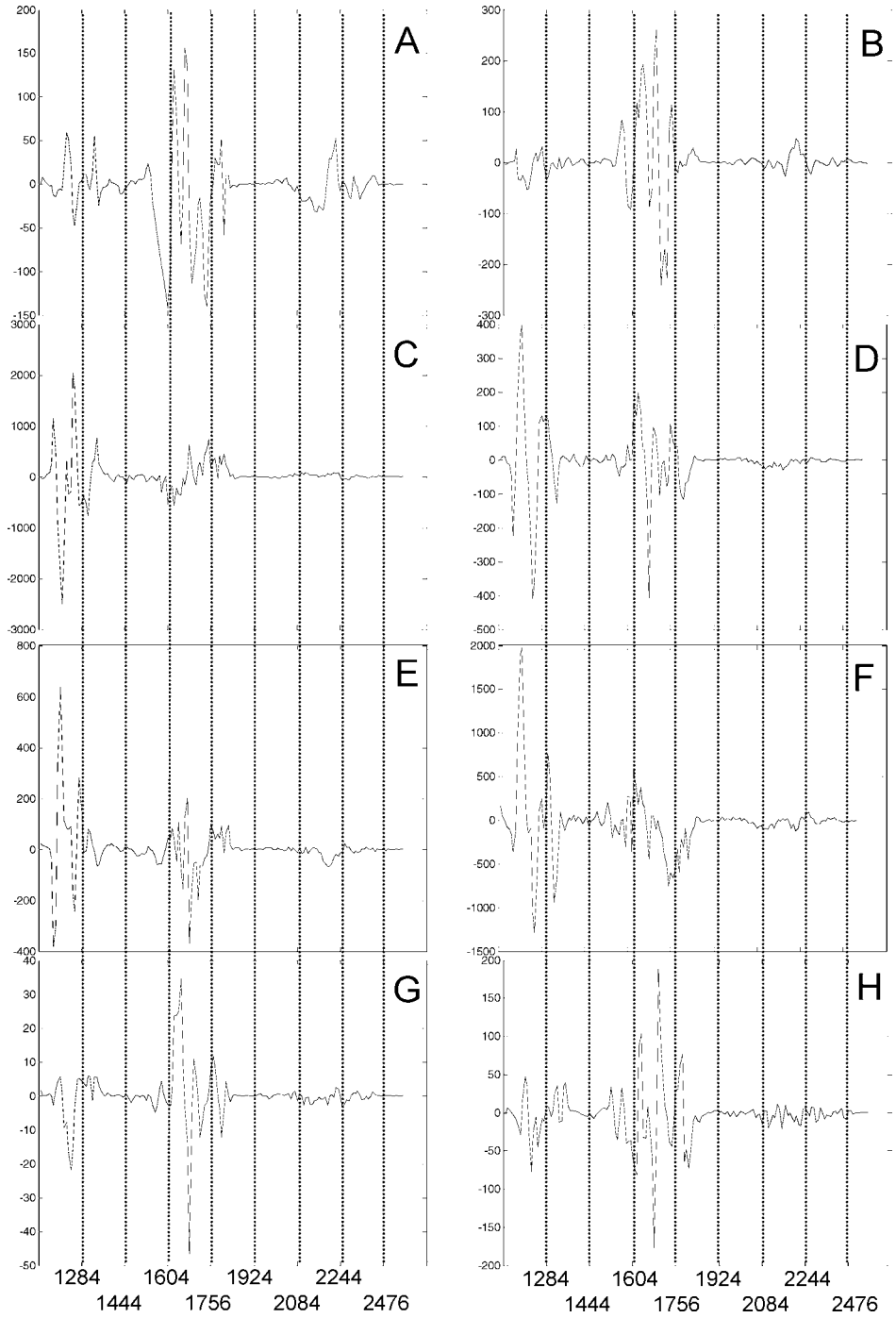

Figure 5. Regression coefficients for the height of other parameters: *x*-axis, wavelength (nm); *y*-axis, regression coefficient; (A) brix, (B) marc, (C) juice purity, (D) ,sugar in molasses, (E) nitrogen, (F) potassium, (G) glucose, and (H) sodium.

3.3. Model Coefficients and Wavelength Interpretation. **Figure 4** shows the key wavelengths used for the calibration equation to determine sucrose content and compare to the wavelengths used in previous study. The wavelengths are assigned to total sugar (*4*, *17*−*19*) or sucrose (*20*−*22*). Cadet and co-workers (*23*) determine the vibration bands dues to carbohydrate in the near-infrared spectral range: combination C−H elongation/C−C elongation and C−O elongation at 2500 nm; combination C−H elongation/CH_2 deformation at 2280−2330 nm; combination O−H elongation/ZOH deformation at 2100 nm; 1st overtone elongation at 1450 nm; 2nd overtone elongation at 1010−1030 nm; and 3rd overtone C−H elongation at 850−900 nm.

Figure 5 shows the wavelengths which are useful to determine the other quality parameters. We have noticed that some spectral ranges contain more significant information: between 1100 and 1300 nm, C−H second overtone; between 1300 and 1350 nm, C−H combination; between 1600 and 1800 nm, C−H first overtone; and between 2100 and 2300 nm, combination bands C−H + N−H and C−H + C−C.

Nevertheless, we noticed that the water bands (1450 and 1950 nm) have very low regression coefficients.

3.4. Discussion on Model Accuracy. Four hypotheses can explain the lack of fit of a model: (1) WCA are not enough accurate; (2) modeling methods introduce error; (3) near-infrared spectra do not contain enough chemical information; and (4)

Table 3. Accuracy Comparison of WCA and NIR Methods

parameters	S[b]	brix[b]	marc[b]	JP[b]	SM[b]	N[b]	K[c]	Na[c]	Glu[b]
mean of WCA values[a]	17.54	20.85	4.36	95.55	1.21	0.62	3.60	0.33	0.05
mean of NIR values [a]	17.55	20.85	4.36	95.52	1.22	0.62	3.61	0.35	0.05
WCA[a] repeatability	0.06	0.05	0.08	0.04	0.01	0.03	0.04	0.01	0.01
NIRS[a] repeatability	0.07	0.09	0.11	0.10	0.05	0.06	0.11	0.03	0.01
F-test value (significance at $\alpha = 5\%$)	1.36 (NS)	3.24 (S)	1.89 (S)	6.25 (S)	25 (S)	4 (S)	7.56 (S)	9 (S)	1 (NS)

[a] Units of the component. [b] Units: g/100 g. [c] Units: mmol·kg^{-1}. [d] S = significant F-test, NS = nonsignificant F-test with $\alpha = 5\%$.

concentrations are too low to be quantified by NIRS (under detection limits).

To verify the first hypothesis, the repeatability of WCA was calculated. Thirty samples were analyzed three times by WCA and the standard error of repeatability was computed (*24*). **Table 3** shows the WCA had a repeatability that is sufficient for this purpose. The last three hypotheses explain our results.

The NIRS repeatability is also determined by the measurement of 30 samples analyzed three times. The results are shown in **Table 3**: NIRS repeatability is low. However, WCA repeatability is equal or better than that for NIRS for all the components and F-tests (**Table 3**) underline the differences between the NIRS and WCA repeatability are significant with $\alpha = 5\%$ except for the sucrose and glucose content.

Modeling methods were optimized: several pretreatments were used and several regressions (linear and nonlinear) were applied (*4*). The results presented are the most accurate and we think the modeling error could not be reduced and the second hypothesis was rejected.

Glucose, sodium, and potassium are not determined accurately by NIRS. We can assume that near-infrared spectra do not contain enough information about K and Na. Ions do not have a near-infrared signature, they just modify the spectra of the other components by chemical interactions. Concerning the glucose, we can suppose the concentration is too low to be accurately determined by NIRS or the concentration range is too narrow to develop an accurate model.

3.5. NIRS in Sugar Factories. In this section, the uses of the quality parameters are discussed. The quality parameters are useful for the sugar factory and for the growers. As stated previously, sucrose content determines the grower payments. Moreover quality parameters may be used to estimate the production of a factory. The sugar factory produces crystallized sucrose, molasses, and beet pulp. Beet pulp is used in animal feed and molasses is used in distilleries. A ton of beet at 16 g/100 g of sucrose theoretically produces 130 kg of crystallized sucrose (called sugar), 18 kg of sugar (i.e. sucrose) in molasses (37.5 kg of molasses at 48% of sucrose), and 55 kg of beet pulp.

The production of crystallized sugar (m_{SP}) is calculated by a balance sheet (*25*): $m_{SP} = m_S - m_{SM} - m_{PP}$, with m_S being the beet sucrose content, m_{SM} the sugar in molasses content, and m_{PP} the sugar lost in the industrial process (all in kg).

m_S is measured at the beet reception, m_{PP} is considered a constant when the factory has a stationary state, and m_{SM} is calculated with an empirical equation. So the crystallized sucrose production in the sugar factory can be estimated by the sugar beet analysis.

The beet pulp production may be estimated by the marc values which represent the cellulose and the fiber contents. Moreover, the nitrogen content is an indicator of the content of the fertilizer used. If the nitrogen content in sugar beet is high, the grower should reduce the fertilizer quantities.

Table 4. Results of Previous Studies Concerning NIR Measurement of Sugar Beet Brei[a]

compds	ref	concn range	sample no. calibration/ validation	SEC	SEP	R^2 (validation)
brix	30	15–18.5	146/36	–	0.19	0.963
brix	26	–	75/75	0.24	0.27	0.96
sucrose	30	15–18.5	146/36	–	0.10	–
sucrose	37	13–19.6	1000/4500	0.19	0.20	–
sucrose	26	–	75/75	0.37	0.40	0.95
sucrose	28	–	175/75	–	0.25	–
nitrogen	27	–	146/36	–	1.70	0.79
nitrogen	28	–	175/75	–	3.95	–
sodium	28	–	175/75	–	2.22	–
potassium	28	–	175/75	–	4.19	–

[a] Units: g/100 g. A dash indicates a missing value.

Some studies deal with NIRS on sugar beet (*26*−*37*) brei but their results are less accurate than our study or the number of samples used for calibration is low or not representative of all sugar beet types. **Table 4** presents the results of a previous NIRS study on beet brei. The NIRS predictions were improved, compared with previous studies (*26*−*29*). It can be explained by our large database and by our NIRS and WCA protocol. However, the more recent De Bruijn paper reports a value for the standard error of prediction of 0.1 for the determination of sucrose in sugar beet extracts (*30*).

We notice NIRS is used in cane refineries to determine the water (*31, 32*), brix (*33*), glucose, fructose, sucrose, and lignin contents (*31*) in the cane. NIRS is also useful in the factory for the on-line control: NIRS is applied to determine on-line the brix and sucrose content of factory juices (*34*, *35*).

To conclude, this study shows the feasibility of NIR spectroscopy to determine sucrose content and the quality parameters of sugar beet. The best model is developed with MPLS regression and spectra modified by SNVD and second derivative (gap 8 and smooth 6). The standard error of prediction is 0.10 g of sucrose per 100 g of sugar beet. We should be satisfied by the accuracy of sucrose prediction. NIRS can also be used to evaluate the quality of sugar beet and to estimate the factory productions. The main advantages of NIRS are its speed, its low cost, and the environmentally friendly aspect.

ABBREVIATIONS USED

S, sucrose; JP, juice purity; SM, sugar in molasses; N, nitrogen; K, potassium; Na, sodium; Glu, glucose; WCA, wet chemical analyze; NIRS, near-infrared spectroscopy; SEP, standard error of prediction; SEC, standard error of calibration; SECV, standard error of cross validation; R^2, correlation coefficient; RER, ratio of the concentration range to SEP; RPD, ratio of the concentration standard deviation to SEP; SNV, standard normal variate; D, detrending; SNVD, standard normal variate and detrending.

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