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Near-infrared Spectroscopy for Rapid Estimation of Dry Matter Content in Whole Unpeeled Potato Tubers

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Abstract

The dry matter is one of the main quality parameters of raw and processed potatoes. In the present study, the potential of utilizing high throughput commercially available NIR interactance systems for dry matter determination in whole unpeeled potato tubers is investigated. The performance of a 2D NIR interactance instrument was compared with that of a 1D NIR interactance instrument and a traditional underwater weight apparatus. A total of 114 tubers were assessed individually with both of the NIR instruments (760-1040 nm), the underwater weight and an external reference method (freeze drying). The 1D interactance instrument obtained better prediction results than what the 2D instrument could achieve (R^2=0.95, RMSECV=0.91, and R^2=0.83, RMSECV=1.65, respectively). The underwater weight obtained the highest explained variance (R^2=0.97), but the estimation was biased by approximately 1.5% (by weight). The poorer prediction performance of the 2D NIR interactance system can be partly explained by the lower penetration depths of the light compared to the 1D NIR interactance systems.

Keywords: near-infrared spectroscopy, potato, dry matter determination, on-line measurements

1. Introduction

On a global basis the potato (*Solanum tuberosum* L.) is grown with yields in millions of metric tons per year. The trend in potato consumption shows that the amounts used for processing has been steadily increasing over recent years (Loy, Riekert, & Steinhagen, 2011). A potato, however, is highly heterogeneous. During factory processing, unacceptably high variations in raw potato quality means that different processing parameters have to be periodically adjusted and optimised in order to obtain consistent end product quality and reduce waste quantities. The features most commonly assessed prior to industrial processing ofpotatoes include internal and external defects along with dry matter content and glucose levels. Dry matter is one of the variables required to calculate and estimate frying or cooking time, and hence the texture, color and acrylamide content of the end-product. Wrongly determined dry matter content may thus lead to an unacceptable quality of whole batches of processed potato products.

Traditionally, the preferred method for dry matter evaluation has been the underwater weight of the tubers. The relationship between specific gravity and dry matter has been investigated numerous times, and slightly different approximations of this relationship have been obtained for tubers of different growth years or geographic origins (Haase, 2003; Lunden, 1956; Nissen, 1955; Schippers, 1976). The accuracy of the approach is, however, disputed (Lunden, 1956; Nissen, 1967; Wilson & Lindsay, 1969), which is indicated by the large number of different approximations. It is also clear that this method is not suited for on-line measurements, and the technique is as such unable to assess the dry matter variation within a batch of potatoes. The need for better batch characterization along with the development of better technological solutions has led to a significant interest in methodology such as Near-Infrared (NIR) spectroscopy. In NIR spectroscopy, near-infrared light is absorbed at specific frequencies due to vibrations of molecules within the sample. The molecular vibrations are specific for the functional groups of each molecule and can therefore be used to quantify components such as water or starch. NIR spectroscopy is non-invasive and may provide high sampling rates, making the technique ideal for on-line assessment of food components.

The first reports concerning NIR spectroscopy for dry matter analysis of potatoes were published in the late 1980s (Dull, Birth, & Leffler, 1989). Dull et al. reported that NIR light in the region of 800-1100 nm in transmittance mode (light source and detector on opposite sides of the sample), could be used to determine dry matter in whole unpeeled potato tubers (Dull et al., 1989). As a proof of principle the authors reported correlations (R) of 0.9178 for whole unpeeled tubers, and 0.975 for thin-sliced tubers (Dull et al., 1989). Since then a number of feasibility studies have been published, most measuring mashed or sliced tubers, excludes the possibility of non-invasive measurements. Explained variances (R^2) between 0.58 and 0.99 for dry matter determination have been achieved with reflectance measurements, where mashed tubers as expected provided the highest explained variances (Brunt & Drost, 2010; Haase, 2011; Scanlon, Pritchard, & Adam, 1999). Studies have also concluded that even though feasible estimations of dry matter and starch could be obtained, the minor constituents such as glucose, crude protein and recoverable protein are difficult to predict (Fernandez-Ahumada et al., 2006; Haase, 2011; Scanlon et al., 1999). More recently, automated routines for dry matter determination have also been explored. Brunt and Dorst (Brunt & Drost, 2010; Brunt, Smits, & Holthuis, 2010) made a prototype off-line set up for determination of dry matter and starch. For this automated system the underwater weight approach was used for the dry matter, and NIR reflectance of mashed potato tubers were used for starch determination (Brunt et al., 2010). On-line procedures for estimation of dry matter and fat content in processed potato products have been described previously (Pedreschi, Segtnan, & Knutsen, 2010).

It is well known that the dry matter content is unevenly distributed throughout a potato tuber. Generally, the dry matter content is highest in the outer layer (parenchyma) and lowest in the middle layer of the tuber (pith). In addition, the gradient of dry matter content is not necessarily linear through the tuber or constant from one tuber to the next (Storey, 2007). Thus, in order to develop automated NIR instrumentation for analysis of whole unpeeled tubers, the sampling approach has to provide adequate sampling volumes. With a transmittance set-up this could be achieved, but the set-up would require instrumentation both above and below a conveyor belt and would thus be challenging to fit on-line. The reflectance approach presents obvious limitations due to reduced sampling volumes. The interactance approach, on the other hand, could potentially provide adequate sampling volumes, and the instrumentation could be easily situated above the conveyor belt. Subedi and Walsh recently published the first NIR interactance investigation on intact tubers (Subedi & Walsh, 2009). A system capable of measuring samples on a conveyer belt was also investigated, but only for sliced tubers. The authors concluded that short wave NIR (750-950 nm) in interactance mode was well suited for rapid assessment of potatoes. Using a high resolution laboratory instrument they achieved an explained variance (R^2) of 0.87 for whole unpeeled tubers. When they measured sliced tubers, the explained variance (R^2) increased to 0.92. For moving sliced tubers the explained variance (R^2) decreased down to 0.82.

Whereas Subedi and Walsh investigated NIR interactance approach for dry matter content assessment using a high-resolution laboratory instrument, there are no known studies where commercially available and industrially applicable instrumentation has been evaluated for the same purpose. Thus, in the present study, the potential of utilizing high throughput commercially available NIR interactance systems for dry matter content determination in whole unpeeled potato tubers is investigated. The instruments used in this study have previously been applied at-line and on-line for measuring main components in food systems like meat trimmings and fish fillets (ElMasry & Wold, 2008; J. P. Wold, O'Farrell, Hoy, & Tschudi, 2011). The instruments provide lower spectral resolution than reported in previous publications, hence enabling faster spectrum acquisition, higher signal to noise ratio (SNR) and higher sample volumes per time unit. The objectives of the study are three-fold: (1) To utilize 1D NIR interactance for stationary analysis of whole unpeeled potato tubers; (2) to utilize a commercially available 2D NIR interactance system to provide on-line estimation of whole unpeeled tubers; and (3) to compare the performance of the NIR instrumentation to the performance of the underwater weight. All measurements were conducted on single tubers in order to eliminate in-batch variation (Cole, 1975).

2. Materials and Methods

Potato tubers were acquired from a potato packaging facility (varieties Asterix, Folva and Celine) and a chips manufacturer (varieties Bruse and Saturna). For the main part of the experiment 20 tubers from each of the varieties with red skin (varieties Asterix, Bruse and Celine) and 30 tubers from each of the varieties with yellow skin (varieties Folva, Saturna) were selected. After cleaning in tap water the tubers were left overnight to dry and reach an even temperature (room temperature at 21°C). The tubers were selected so that they hada size distribution similar to that found in potato processing industry.

2.1 1D NIR Interactance Measurements

1D NIR interactance measurements were performed using a prototype VIS/NIR interactance instrument allowing a spectral resolution of 30 equally spaced channels in the VIS/NIR region (449-1040 nm) (Folkestad et al., 2008). The spectral acquisition time was set at two seconds. Two equal sized squares were illuminated by two 50 Watt halogen lamps (OSRAM, Augsburg, Germany), and backscattered light was collected through a collection tube. Measurements were done with contact between the sample and the collection tube, which effectively blocked all direct reflections from the sample surface. The 15 channels in the visible range (446-760 nm) were not used. Ten tubers (five with red skin and five with yellow skin) were used to estimate the penetration depth into the tuber of light from the 1D NIR interactance instrument. This was performed by recording a spectrum with a tuber placed first on a non-reflective (black plastic plate) and then on a reflective background (glossy metal plate). The tuber was then thinly sliced and measurement repeated. The distance where one could see a difference between the two surfaces was recorded as the light penetration depth. A series of 60 tubers were measured at three different places: stem, bud, and along the middle of the longitudinal axis. Data obtained were used to determine the best measuring position for NIR measurements on each tuber.

Each single tuber was measured three times on each side. Each tuber was rotated approximately 60^{o} between each measurement. Measurements were taken at the center of the longest axis. This position was shown to have a dry matter content closest to the average of the whole tuber (Pritchard & Scanlon, 1997).

2.2 2D NIR Interactance Measurements

Single tubers were scanned with the commercially available QV500 (QVision AS, Norway). The QV500 is equipped with 15 equally spaced channels in the NIR region (760-1040 nm), and is able to scan a conveyor belt moving with a speed of up to one ms^{-1}. The resolution perpendicular to the moving belt was 60 pixels distributed on a 50 cm wide area. Light was transmitted via a rotating mirror onto the potato. Backscattered light from the interior of the potato was collected in the spectrophotometer. The optical lenses were configured to block direct reflections, hence only light that had traversed the tuber was collected. As for the interactance instrument the light penetration depth was determined. A series of ten tubers (five with red and five with yellow skin) were measured with a non-reflective and a reflective surface underneath, with successively thinner samples. For the determination of dry matter content in intact tubers, each tuber was scanned once at each side. The distance from the detector to the conveyor belt was 31 cm.

2.3 Specific Gravity Measurements

After spectral acquisition tubers were suspended from a load cell (Tedea Huntleigh, Model no. 1004, accuracy of 0.017%) and the weight was measured in air and under water. The specific gravity (SG) was calculated based on equation 1.

$$SG = \frac{Weight\,in\,air}{Weight\,in\,air - Weight\,in\,water} \tag{1}$$

Utilizing equation 2 an estimate of the dry matter content was calculated (Lunden, 1956).

$$DM = 215.73(SG - 0.9825) \tag{2}$$

After measurement tubers were dried with tissue paper and placed in small plastic beakers.

2.3.1 Reference Analysis

Prior to freeze drying (Christ Gamma 1-16) tubers were carefully sliced into small pieces with a sharp knife and frozen to -40^{o}C. After freeze drying, remaining potato material was ground to a fine powder in a household blender (Moulinette, Moulinex, Italy). The remaining moisture in samples was determined in a moisture analyzer (Sartorius L420P/YTC01L Thermo Control). Total amount of dry matter content was calculated based on the weight before and after freeze drying.

2.4 Data Analysis

Data from 1D NIR interactance measurements were imported into Unscrambler X, V 10.1 statistical analysis software (CAMO PROCESS AS, Oslo, Norway). The average of six replicate measurements was calculated. Averaged spectra were standard normal variate (SNV) transformed (Barnes, Dhanoa, & Lister, 1989), and a regression model was developed by partial least squares regression (PLSR) (Martens & Næs, 1989), with dry matter percentage obtained by freeze drying as reference values (Wold, Martens, & Wold, 1983). The optimal number of PLSR factors was determined by full cross validation. The reference value, and the predicted value, of every sample were used to calculate the prediction error of the cross validated calibration model, expressed as the root mean square error of cross validation (RMSECV). The RMSECV value is defined by the following equation:

$$RMSECV = \sqrt{\frac{\sum_{i=1}^{I}(y_i - \hat{y}_i)^2}{I}} \quad (3)$$

Where i denotes the samples from 1 to I. y_i *and* $\hat{y}_i$ denotes the reference value and the predicted value, respectively.

For the data acquired by the 2D NIR interactance system, a segregation routine had to be applied in order to separate the potato tuber from the conveyor belt. This was performed by comparing the ratio between the absorption of the wavelengths of 800 and 980 nm. The threshold was set so that all pixels with a ratio above 0.04 were selected as potato tuber, and the center of the tuber was automatically calculated. An area of 1/10th of the total width was selected on each side of the center point, and 1/10th of the height above and below the center. The spectrum of each pixel within a selected area was extracted and averaged. This was performed by an in-house built MatLab routine (MatLab, V. 7.10, The Mathworks Inc., Natick, MA). All spectra were exported to Unscrambler software and the spectra from both sides of the tuber were averaged. The spectra were used in PLS regression towards the reference analysis without any pre-treatment. Regression was validated by full cross validation.

3. Results and Discussion

The potatoes used in this study were selected to give as large variation in both skin color and dry matter content as possible. The color of the potato flesh was yellow for all tubers, with slight variation in the shades of color. An overview of the dataset used for the experiment is provided in Table 1. The dataset ranges from 14.4% to 30.5% in dry matter content, which covers most of the natural variance found amongst potato tubers (Burton, 1989). The samples also provided a good distribution of dry matter content amongst the tubers within each variety. A total of six samples had to be removed from the data set due to abnormalities such as rot and internal cavities.

Table 1. An overview of the potato dataset used in this study

Variety	Skin Color	Min % DM	Average % DM	Max % DM	SD DM	n
Asterix	Red	18.0	20.1	21.8	1.0	19
Bruse	Red	21.4	26.4	30.5	1.9	19
Celine	Red	14.8	18.1	20.3	1.6	19
Folva	Yellow	14.4	16.6	19.7	1.4	29
Saturna	Yellow	22.2	24.4	27.0	1.4	28
Total	-	14.4	21.0	30.5	4.1	114

3.1 Specific Gravity

The specific gravity of a potato is easily transformed into an estimate of the dry matter content. Due to its feasibility and low cost this has been the method of choice for dry matter content determinations in industry since it was introduced. The underwater weight used in the current study was configured for evaluation of single potato tubers. The values obtained from specific gravity measurements are shown in Figure 1a. Estimated values had a very good explained variance with reference values (R^2=0.98). The RMSECV was estimated to be 0.65, but all measurements made with the underwater weight were approximately 1.5 weight-% higher than the reference value. This is clearly shown in Figure 1a and indicates that a consistent deviation is present in all measurements, or that the model used has a small offset.

As previously mentioned a number of different approximations exist for converting specific gravity to dry matter percentage. Five of the industrially utilized regression models are shown in Figure 1b, the uppermost one being that used in this study. This is the regression model used by the Norwegian potato industry. Apparently, a careful selection of regression models may give an accurate estimation without any significant bias.

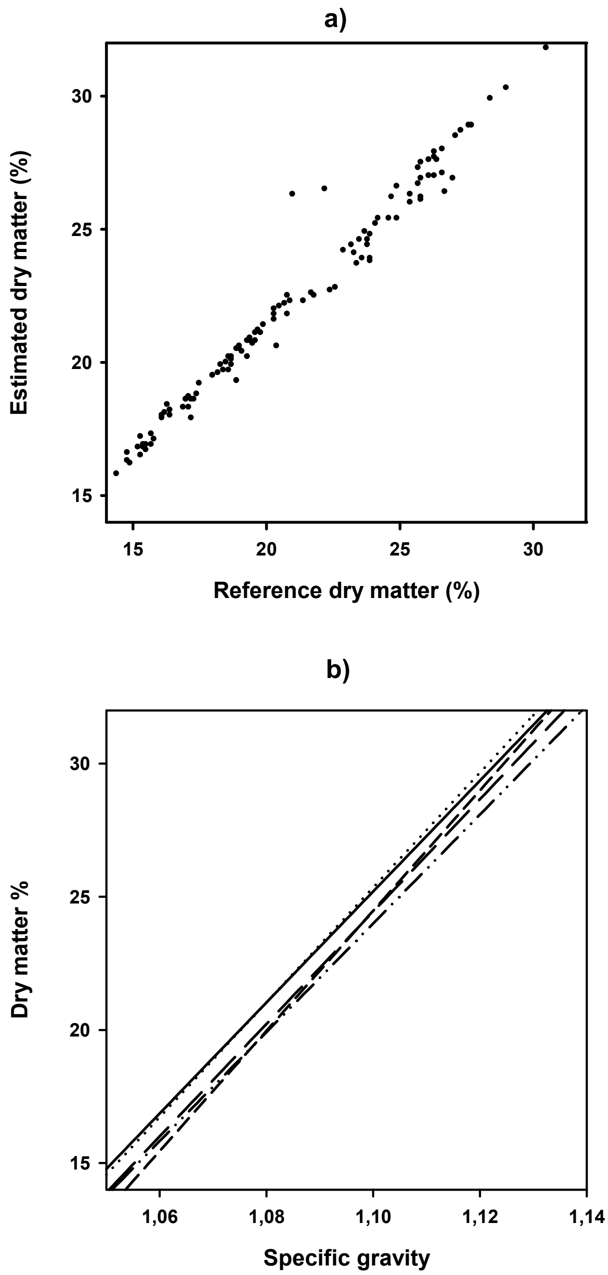


Figure 1. a) The dry matter values obtained from specific gravity measurements and the reference values obtained by freeze drying. b) Some of the most common regressions (Lunden, 1956) for converting underwater weight into dry matter content

3.2 1D NIR Interactance Measurements

The surface topography and especially heterogeneity of the potato tuber will clearly affect the NIR acquisition. To better understand how each sample affects the measurements, light penetration depths in 10 tubers were measured by 1D NIR interactance (data not shown). A penetration depth of up to 20 mm into unpeeled tubers could be achieved. An experiment measuring 60 tubers at three different places (stem, bud and middle) showed that measurements taken at the middle of the longitudinal axis of each tuber gave the highest explained variance (R^2) and the lowest RMSECV for estimation of dry matter content (data not shown). This is in accordance with the findings of Pritchard and Scanlon (Pritchard & Scanlon, 1997).

Figure 2a and 2b shows the absorption and the SNV corrected spectra, respectively, obtained from the 1D NIR interactance measurements. As one can see there is a large absorption band at 960 to 980 nm, which is typical for the second OH-water overtone (960-970 nm). Spectra were related to reference values using PLS regression. The performance of the developed model is provided in Figure 3a. PLSR regression provided a high explained variance value (R^2=0.95) and an RMSECV value of 0.91, with a model based on five PLSR factors. Regression coefficients (Figure 3b) show that the main positive contribution to the model is found in the area where the second overtone for the carbohydrate OH-stretch absorption band (901 nm) is known to appear (Williams & Norris, 1990). The second OH-water absorption overtone is known to appear at 960 nm, which seems to give a positive contribution to the dry matter content determination. The absorption in the region 960-980 nm may thus partly be ascribed to starch. The carbohydrate OH-stretch is known to give a strong absorption band at 979 nm, which may explain the positive contribution from the wavelengths in the region 960-980 nm (Williams & Norris, 1990).

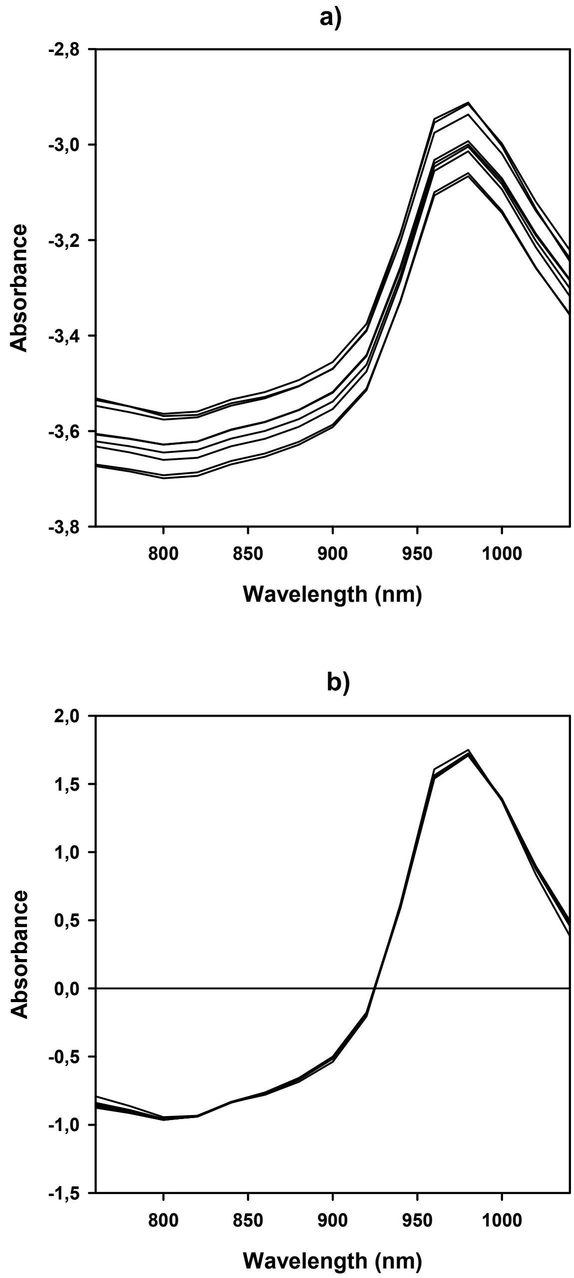
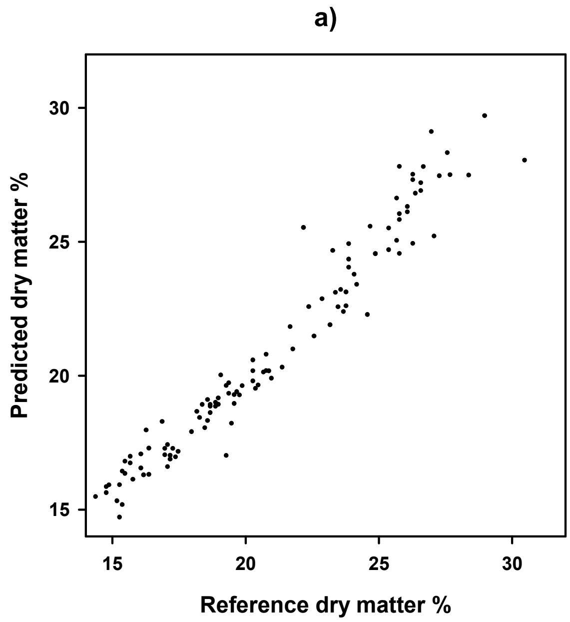


Figure 2. Absorbance a) and SNV-transformed spectra b) of whole unpeeled potato tubers acquired by 1D NIR interactance



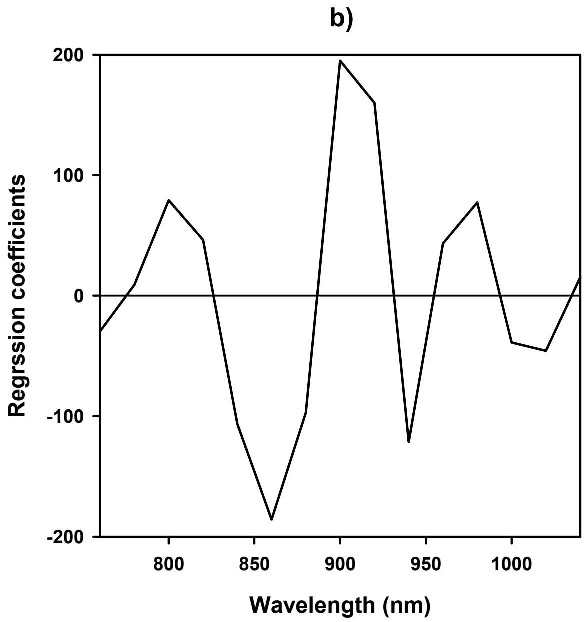


Figure 3. a) The relationship of predicted dry matter obtained by 1D NIR interactance against reference values obtained from freeze-drying. b) Regression coefficients as a function of wavelength obtained by PLS regression used to predict the dry matter in whole unpeeled potato tubers

3.3 2D NIR Interactance Measurements

In contrast to a 1D NIR interactance instrument, the 2D NIR interactance system creates an array of spectra in a short period of time, enabling it to monitor and assess an incoming stream of tubers in a factory. The 2D NIR interactance system obtained spectra similar to those shown for the 1D NIR interactance system. The 2D NIR interactance system could only measure 10 mm into unpeeled tubers. In contrast to 1D interactance measurements, 2D NIR interactance spectra were not SNV corrected prior to PLSR regression. This may indicate that the 2D NIR interactance system is less vulnerable to physical variation in e.g. sample surface. Figure 4a shows the regression result, providing an explained variance (R^2) of 0.83 and a RMSECV value of 1.65. The 2D NIR interactance system thus provided a significantly lower R-square value and a higher prediction error than what was obtained by 1D NIR interactance measurement. From Figure 4b one can see that the regression vector is based mainly in the same absorption bands as the model obtained with the 1D instrument.

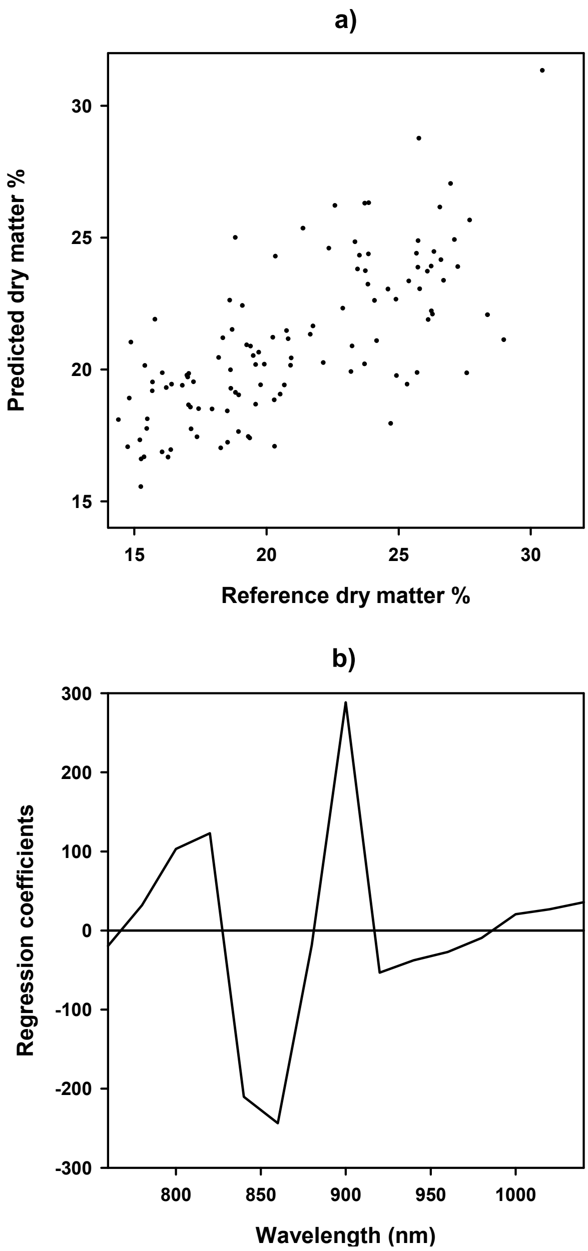


Figure 4. a) A scatter plot of the predicted dry matter values predicted by the 2D interactance instrument plotted against the reference values. b) The regression coefficients from the regression models used to predict the dry matter in whole unpeeled potato tubers

To be able to predict the dry matter content accurately, the 2D NIR interactance system has to distinguish between the sample and the conveyor belt. For the current study it was desirable to assess each tuber individually, but in an industrial application an average of the whole width of the conveyor can be determined, if necessary. The tubers used in this experiment varied in size. A single segregation routine was therefore not sufficient, and a double criterion was developed. After the first extraction the pixels were counted, and if the number of pixels exceeded a certain value the image was re-sampled with a new and higher threshold. This had a dual purpose: 1) to assure that the area used to extract spectra only contained potato matter and not parts of the conveyor belt; and 2) to ensure that an area with the same relative size, compared to the whole tuber, was selected for the sampling.

3.4 General Discussion

A summary of predictive performances of the three methods is shown in Table 2. Underwater weight is the method with largest deviation, but this is not truly comparable since an already existing regression model was used. The explained variance is still very good, and a careful selection of regression model might even further reduce the error.

Table 2. An overview of the R-square and RMSECV values obtained in the experiments

	R^2	RMSECV	Pre-treatment	number of PLSR factors	Average estimated value for all samples
1D Interactance measurements	0.95	0.91	SNV	5	21.0
2D Interactance measurements	0.83	1.68	none	9	21.0
Specific gravity	0.97	0.65	none	-	22.3
Reference value	-	-	-	-	21.0

One of the most important differences between the techniques investigated in this study is the ability to assess the variation of dry matter content in batches of potato tubers. The major drawback with the underwater weight is that it can only be used with small sample volumes. In the Norwegian potato industry the common sample size is 2 x 5 kg of tubers per 10 metric tons. The 1D NIR interactance system is not suited for assessment of samples on a conventional conveyor belt, but can be incorporated on a single line conveyor belt. The 1D instrument can be configured to record multiple spectra per second, and could hence monitor single tubers going down for instance a chute prior to sorting. The instrument used in the present study is a prototype, but a commercially available version exists (QPoint, QVision AS, Norway). The 2D NIR interactance system suffers from higher prediction error than the two other approaches, but the system can easily assess each tuber in the whole batch and calculate a batch mean. Then the user can decide whether the output should include both maximum, minimum, and average values, or just the latter. The higher prediction error is a typical tradeoff when one desires higher speed and larger throughput volumes. There will always be a trade off in each individual application whether the prediction accuracy, SNR or sampling speed is most important.

The dry matter content in potato tubers does not only vary within a batch, and there is also a considerable variation within each tuber (Storey, 2007). Light will travel a shorter distance relative to size, into a large tuber, than into a small one. Hence, it may be beneficial to adjust the regression model according to the actual size of the tuber, keeping in mind that the dry matter content is unevenly distributed within the tuber. For 2D NIR interactance measurements, if desirable one could include the same size threshold used for the segregation routine and say that tubers exceeding a specified size should be estimated by a different regression model. This model should thus to some degree compensate for the fact that the measurement is taken from the outermost layer of the largest tubers. In addition, light from the 2D NIR interactance instrument penetrated to a shallower depth into each tuber than the light from the interactance system, hence sampling a smaller part of the dry matter content gradient. This could explain some of the prediction error, especially when considering the large intra-tuber variation in dry matter content. Other factors that may have contributed to a high estimation error of the 2D NIR interactance system might include shorter measurement times and reduced SNR compared to the 1D system.

Pre-treatment of the data is an important part of data modeling, and SNV-transformation is one of the most frequently utilized techniques. In contrast to data obtained with a point measurement system, the 2D NIR interactance data did not undergo SNV-transformation before regression. Performing regression on SNV-transformed data yielded a model (data not shown) with slightly poorer performance than when using absorbance data. The reason for this was not investigated.

One issue not discussed so far is the sampling speed of the 2D NIR interactance system. The QV500 is rated to handle a conveyor belt speed up to one ms^{-1}, with a sampling width of 50 cm. This would accommodate the possibility of measuring large quantities in a short time frame. One drawback with both the 1D and the 2D NIR interactance systems is the low spectral resolution. The interactance system has the ability to measure 30 channels, 15 of these originating from the visible light region. The 2D NIR interactance system features the same 15 channels in the near infrared region. The visible part of the spectra was discarded since tubers of different color were used. The regression coefficients for the 1D and 2D NIR interactance systems (shown in Figure 3b and 4b, respectively) show that the second overtone of the OH stretch from carbohydrates dominates the regression coefficients. Since the regression model estimates dry matter content, this indicates that it can be difficult to estimate other tuber attributes, like starch content and composition, without a significant increase in spectral resolution of the measurement system.

4. Conclusion

A robust NIR-based calibration model is usually continuously developed to take into account all expected physical and chemical variation. For industrial purposes it may thus be beneficial to develop calibrations based on the varieties used on site, and leave other varieties out of the equation. The calibrations presented here should therefore not be seen as universal, and factors such as variety, surface topography, growth conditions and diseases may affect the prediction performances. The results, however, are still expected to provide realistic insight into the predictive performances of the commercial instrumentation investigated. This study shows the feasibility of using high speed industrially available 1D and 2D NIR interactance instrumentation for dry matter content assessment in single potato tubers. The performance of the 1D NIR interactance instrument exceeded the performance of a 2D NIR interactance instrument (R^2=0.95, RMSECV=0.91 and R^2=0.83, RMSECV=1.65, respectively). Better predictions could possibly be achieved by optimization of the light-configuration in order to obtain a largest possible penetration depth. Even though the 1D NIR instrument is primarily built for stationary use, it can easily be mounted and used on a single line conveyor belt or narrow chute.

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