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Quality control in the milling industry using near infrared transmittance spectroscopy

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Abstract

Flour quality control needs quick analytical tools for predicting rheological and chemical properties. In routine flour quality, wet chemistry analyses take more time. NIR technology allows us to obtain results in a few seconds. In this study commercial wheat flour samples were characterized in terms of protein, moisture, dry gluten, wet gluten, starch damage and ash contents. In addition, wheat flour dough rheological tests were assessed by farinograph and alveograph. Modified partial least squares analyses on NIR transmittance spectroscopy were developed for each constituent or property. Some NIR models obtained were accurate enough for screening end-use flour quality proposes purposes.

Keywords: Flour; Quality; NIR; Calibration; Scans

1. Introduction

The milling industry makes different wheat flour depending on the bread-making process. Nowadays, millers have flour blenders in order to do tailor made flour, because flour needs to be adapted to each bread or biscuit process. In that situation, many types of flour are made and much wet chemistry is needed to do quality control of end-use flour. Large mills make five or six principal flours, and depending on the type of bread or biscuits they could blend these flours in order to obtain the flour requirements.

The farinograph and alveograph have been used to help predict functional dough properties of wheat flour. These instruments are very useful for estimating important dough properties and can also be used to predict mixing requirements, water absorption and deformation energy.

The starch damage refers to starch granules that have been physically altered from their native granular form during milling process. These smaller particles hydrate more easily during dough preparation. Normally starch granules absorb one-third their weight in water; when damaged that increases to 2–3 times their weight. The level of starch damage therefore significantly affects both farinograph water absorption, and dough extensibility and resistance measured by alveograph (Chen & d'Appolonia, 1986). Damaged starch granules are very susceptible to attack by α-amylase enzymes, thus damage provides a further supply of sugars to the yeast during fermentation. Too much starch damage can result in poor loaf volume, heavy texture and coloured crust. Previous studies (Morgan & Williams, 1995; Osborne & Douglas, 1981; Osborne, Douglas, & Fearn, 1982) have shown how NIR technology can predict starch damage on wheat flour.

Knowing the protein content is not enough to characterize wheat flour, so it is useful to determine wet and dry gluten. Wet gluten is the insoluble protein fraction from flour protein content. Gluten proteins are essential for bread production because elasticity and extensibility are considered important in the bread making process. Researchers (Bolling & Zwingelberg, 1979, 1982; Norris, 1978; Schorch, 1983; Williams, 1979) were involved in establishing NIR reflectance for determining whole wheat and wheat flour protein. Moreover, proteins fractions (gliadin and glutenin) have been predicted using NIR techniques (Wesley et al., 2001).

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Ash content must be controlled in the milling industry because of legal regulations. On the other hand, ash content determines the purity of the flour, allowing for control of the milling process. Ash content using NIR has been previously reported (Bolling & Zwingelberg, 1979, 1982) showing good correlation.

Previous studies (Delwiche, Graybosch, & Perterson, 1998; Delwiche & Weaver, 1994; William, Saby El-Haramein, Ortiz-Fereira, & Srivastara, 1988) show NIR reflectance spectroscopy as an accurate technique for predicting bread making functional properties of wheat flour dough. Other researchers (Bolling & Zwingelberg, 1984; Posner & Wetzel, 1986) have described how online monitoring of flour mill streams by NIR can improve flour mill performance. NIR reflectance is widely used in the milling industry for measuring protein and moisture in flour or on whole wheat. However, NIR has the potential for measuring other components and physical properties.

Flour quality for a specific bread-making process cannot be described for one specific parameter. Bread-making industries, even retail bakers, are demanding progressively more physical and chemical parameters. As techniques have progressed, industries have required more parameters in order to predict flour behaviour. The moment the industry establishes quality standards, it demands flours with defined quality specifications including moisture, protein, wet gluten, dry gluten, and rheological analyses (farinograph, alveograph). This supposes that the flour industry must complete many physical and chemical analyses, with consequent economic and time-related costs. A technique that permits fast prediction of physical and chemical properties is therefore necessary. Many studies show how spectroscopy techniques could predict some physical and chemical properties, but some chemical and rheological wheat flour properties have not been studied using near infrared transmittance spectroscopy, although some whole wheat flour parameters have been predicted using NIR transmittance recently (Miralbés, 2003). The majority of the literature indicates how spectroscopy techniques permit the prediction of chemical and physical properties. The main objective of this research was the development of NIR models for predicting physical and chemical flour properties using near infrared transmittance spectroscopy.

2. Materials and methods

2.1. Samples

Commercial wheat flour samples from different mills were used. All wheat flour samples selected did not contain added enzymes or ascorbic acid.

2.2. Methods

Moisture, protein, wet gluten, dry gluten and ash contents were determined according to the approved AACC methods (AACC, 2000). Dough rheological properties were determined by alveograph and farinograph tests according to the approved AACC methods (AACC, 2000). The alveoragraph parameters registered were resistance of dough to deformation (P), the configuration ratio (P/L) and deformation energy (W). The farinograph parameters registered were absorption (ABS), stability (STA), degree of softening at a 20 min mixing (DDS20), degree of dough softening 12 min after reaching the dough development time (DDS12) and farinograph quality number (FQN).

The starch damage was determined according to the SDMatic procedure (Chopin, Triplette et Renauld, Paris, France) (Medcalf & Gilles, 1965).

The protein, moisture, wet gluten, dry gluten and ash content were measured on a duplicate of each flour sample. The protein and ash content were reported on an as-is moisture basis, whereas wet gluten, dry gluten and farinograph water absorption were reported as 14% moisture basis.

2.3. NIR hardware

A scanning monochromator Infratec 1241 Grain Analyzer (Foss Tecator) with flour module was used to measure NIR transmittance spectra from 850 to 1048.2 nm every 2 nm. The analysis was carried out using small ring cup cells. The NIR spectra were collected from flour.

2.4. Calibration and validation

Commercial spectral analysis software (WinISI III – ver. 1.50e) was used to collect and process the data and develop NIT models.

Prior to calibration, absorbance log $(1/T)$ spectra were transformed mathematically by standard normal variate and detrending (SNV + D) procedures, and transformed with first derivative processing (gap = 4; smoothing = 4, second smooth = 1). This math treatment and scatter correction was previously reported on whole wheat (Miralbés, 2003). Calibration was performed using modified partial least square (MPLS) regression available in WinISI. Full cross-validation was applied to optimise calibration models and detect outliers. With full cross-validation, each sample is removed one at a time from the sample set, a new calibration performed and a predicted score calculated for the sample removed. This procedure is repeated until every sample has been left out once. The optimal number of terms was determined by cross-validation of calibration samples. The outliers with a large residual (T value > 2.5

or H value > 10) were removed, and the calibration was performed again. The cycle of cross validation to eliminate outliers was done a maximum of two times.

The flour samples selected for calibration and validation were taken at random. Following completion of the calibration, the model was validated using an independent set of wheat flour samples. The performance of the model was determined by the following statistics: standard error of calibration (SEC), standard error of cross validation (SECV), standard error of performance (SEP), coefficient of determination (R^2), linear correlation coefficient (r) between reference values and values estimated by prediction models, and discrimination index (RPD = SD/SEP) (William & Soebering, 1993).

3. Results and analysis

The means, ranges and standard deviations of wheat flour for chemical parameters and physical properties are summarized in Table 1. Among the samples analysed for the different parameters, some of them were selected for calibration by the WinISI software, and the remaining samples were used for validation set (Table 2).

3.1. Chemical parameters

The statistical evaluation of calibration and validation of wheat flour for chemical parameters are summarized in Table 3. The models' performance for protein and moisture was excellent, showing $r^2 = 0.99$ and SEP = 0.14 and $r^2 = 0.99$ and SEP = 0.15, respectively. These findings are consistent with others authors working on reflectance mode (Delwiche et al., 1998; Osborne & Fearn, 1983; William, Norris, Gehrke, & Berstein, 1983). Protein and moisture are two constituents easily modelled by NIR. Wet gluten and dry gluten show good validation performance with $r^2 = 0.96$ and SEP = 0.86 (Fig. 1), and $r^2 = 0.99$ SEP = 0.22 (Fig. 2), respectively.

The success of the NIR models for wet gluten and dry gluten seems to be strongly dependent on the correlation to protein content, because about 80% of gluten is protein (Wrigley & Bietz, 1988).

Ash had high correlation with $R^2 = 0.98$ and high validation with $r^2 = 0.98$ and SEP = 0.024 on the range of 0.37–1.54% (Fig. 3). These results were consistent with other authors (Bolling & Zwingelberg, 1982) who obtained NIR calibrations of $r^2 = 0.99$ and SEP = 0.021 on the range of 0.37–0.80%. The ash content of flour is a traditional measure of fine bran particle contamination. Millers like to avoid having bran in their flour as it can have adverse effect on flour colour making it darker and causing specks in some end products. The results obtained (SEP = 0.024) appear to be acceptable compared with the precision of the standard method, which is between 0.01% and 0.03%.

Starch damage of a flour provides information on the flour's baking capability. SDMatic evaluates the starch damage rate of a flour, measuring the amount of iodine absorbed by starch granules in a solution at a temperature of 35 °C (9). Damaged starch absorbs much more water than intact starch granules, thus increasing the total water absorption of flour (Farrand, 1969; Tipples, Meredith, & Holas, 1978). NIR transmittance validation of starch damage with SDMatic procedure gave good performance validation with $r^2 = 0.94$ and SEP = 1.63 (Fig. 4). The first starch damage measurement using NIR was developed by Osborne and Douglas (1981). Starch damage was expressed as Farrand Units (FU) with a SEP = 3 in a range of 5–35. As grain hardness increases, the natural fracturing of the starch during the

Table 1
Summary of properties of wheat flour samples

Attribute[a]	n[b]	Mean	SD	Range
Protein (%)	296	11.96	1.65	7.86–18.08
Moisture (%)	296	14.66	0.65	11.9–16.99
Wet gluten (%, 14% mb)	341	24.73	3.63	17.7–36.76
Dry gluten (%, 14% mb)	341	8.75	1.38	6.18–13.06
DS (CDUc)	150	20.85	1.38	12.9–28.5
Ash (%)	170	0.68	0.26	0.37–1.54
ABS (%, 14% mb)	299	56.92	2.44	49.9–63.3
STA (min)	299	7.73	2.53	1.7–17.4
DDS20 (BU)	299	80	20.4	3–124
DDS12 (BU)	299	69	20	1–129
FQN	299	93	29.7	29–174
P (mm)	476	54	14.59	23–111
P/L	476	0.52	0.18	0.17–1.75
W (10^{-4} J)	476	207	66.7	44–448

[a] DS = damage starch (CDUc, Corrected Chopin Dubois Units), ABS = farinograph water absorption, STA = stability, DDS20 = degree of dough softening at 20 min mixing, DDS12 = degree of dough softening 12 min after development time, FQN = farinograph quality number, P = resistance of dough to deformation, P/L = ratio of deformation, W = deformation energy.

[b] n = number of wheat flour samples selected for each attribute.

Table 2
Means, ranges, standard deviations and number outliers of wheat flour parameters and physical properties of calibration and validation sets of NIR analysis

Attribute[a]	*n* Outliers[b]		*n*[c]	Mean	SD[d]	Range
Protein (%)	8	Cal	236	12	1.84	7.86–18.08
		Val	60	12.41	2.19	9–16.48
Moisture (%)	8	Cal	236	14.66	0.62	11.9–16.99
		Val	60	14.63	0.74	13.12–15.8
Wet gluten (%, 14% mb)	6	Cal	246	24.79	3.69	17.7–36.76
		Val	95	24.43	3.29	18.0–33.44
Dry gluten (%, 14% mb)	6	Cal	246	8.74	1.32	6.18–13.06
		Val	95	8.79	1.61	6.57–12.8
DS (CDUc)	4	Cal	116	20.76	3.68	12.9–28.5
		Val	34	20.38	3.56	13.7–28.3
Ash (%)	5	Cal	125	0.682	0.269	0.37–1.54
		Val	45	0.670	0.237	0.382–1.51
ABS (%, 14% mb)	6	Cal	200	57.4	2.44	49.9–63.3
		Val	99	56.33	2.38	50.2–62
STA (min)	6	Cal	200	7.71	2.6	1.7–17.4
		Val	99	7.75	2.28	3.3–17.2
DDS20 (BU)	6	Cal	200	81.6	22.69	3–124
		Val	99	76.11	25.42	5–110
DDS12 (BU)	6	Cal	200	67	22.3	1–129
		Val	99	71	23.2	30–115
FQN	6	Cal	200	88	30.5	29–174
		Val	99	95	32.1	33–165
P (mm)	14	Cal	358	55.04	13.08	23–111
		Val	118	54.3	17.99	26–104
P/*L*	14	Cal	358	0.52	0.17	0.17–1.75
		Val	118	0.53	0.23	0.18–1.56
W (10^{-4} J)	14	Cal	358	209	61.6	44–448
		Val	118	183	85.7	45–413

[a] DS = damage starch (CDUc, Corrected Chopin Dubois Units), ABS = farinograph water absorption, STA = stability, DDS20 = degree of dough softening at 20 min mixing, DDS12 = degree of dough softening 12 min after development time, FQN = farinograph quality number, *P* = resistance of dough to deformation, *P*/*L* = ratio of deformation, *W* = deformation energy.
[b] *n* Outliers = number of wheat flour samples removed from calibration.
[c] *n* = number of wheat flour samples selected for validation and calibration.
[d] *n* = Standard deviation.

Table 3
Results of NIR calibration and validation sets for wheat flour parameters and physical dough properties

Attribute[a]	Calibration			Validation		
	SEC[b]	SECV[c]	R^2	SEP[d]	r^2	RPD[e]
Protein (%)	0.11	0.12	0.99	0.14	0.99	15.6
Moisture (%)	0.12	0.13	0.99	0.15	0.99	4.9
Wet gluten (%, 14% mb)	0.66	0.66	0.97	0.86	0.96	3.8
Dry gluten (%, 14% mb)	0.17	0.17	0.98	0.22	0.99	7.3
DS (CDUc)	1.01	1.25	0.92	1.63	0.94	2.2
Ash (%)	0.023	0.021	0.98	0.024	0.98	9.9
ABS (%, 14% mb)	0.34	0.35	0.98	0.46	0.97	5.2
STA (min)	0.76	0.79	0.91	1.02	0.88	2.2
DDS20 (BU)	6.4	6.74	0.92	8.77	0.93	2.9
DDS12 (BU)	6.2	6.6	0.91	11	0.90	2.1
FQN	7.3	7.7	0.93	9.1	0.92	3.5
P (mm)	4.44	4.67	0.86	6.07	0.90	2.9
P/*L*	0.07	0.07	0.70	0.04	0.79	5.7
W (10^{-4} J)	15.6	16.5	0.92	21.5	0.95	4.0

[a] DS = damage starch (CDUc, Corrected Chopin Dubois Units), ABS = farinograph water absorption, STA = stability, DDS20 = degree of dough softening at 20 min mixing, DDS12 = degree of dough softening 12 min after development time, FQN = farinograph quality number, *P* = resistance of dough to deformation, *P*/*L* = ratio of deformation, *W* = deformation energy.
[b] Standard error of calibration.
[c] Standard error of cross validation.
[d] Standard error of performance.
[e] RPD = SD validation/SEP.

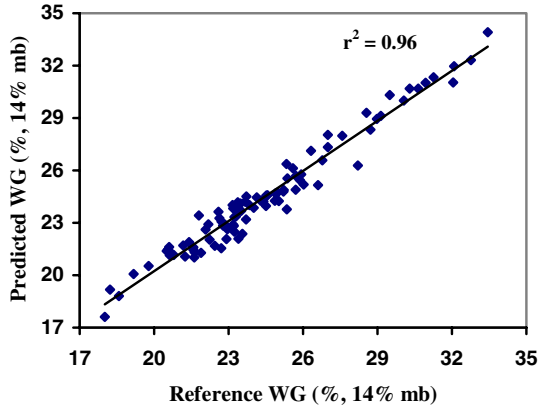

Fig. 1. Comparison of wet gluten content determined by prediction model and by reference method.

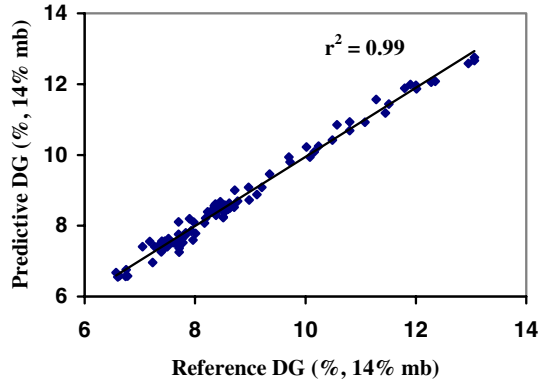

Fig. 2. Comparison of dry gluten content determined by prediction model and by reference method.

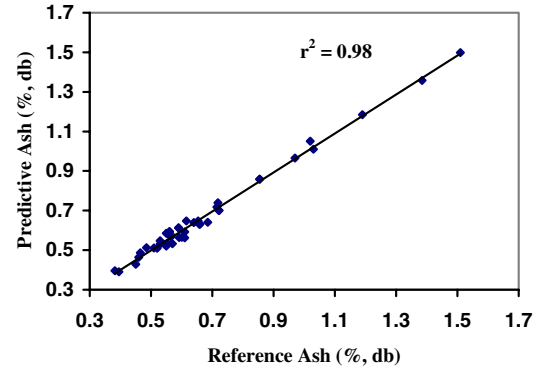

Fig. 3. Comparison of ash content determined by prediction model and by reference method.

milling process, resulting in a higher starch damage level. Excessive starch damage results in higher than require water absorption and this has a negative impact on dough properties and end product quality. Measuring the starch damage rate of flour could makes it possible to determine its baking capability in order to offset problems that may arise during the bread making process. According to the coefficient of determination and SEP value, starch damage could be used to monitoring

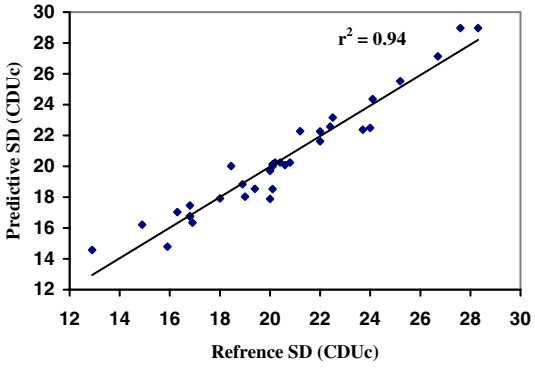

Fig. 4. Comparison of starch damage content determined by prediction model and by reference method.

the milling process, providing on-line results of the reduction system.

Other researchers (Morgan & Williams, 1995) using reflectance techniques obtained a coefficient of determination of 0.9 between the laboratory values and the near-infrared predicted values.

3.2. Farinograph parameters

The statistical evaluation of calibration and validation of wheat flour for farinograph properties are summarized in Table 3.

Water absorption is one of the most commonly used and widely accepted farinograph measurements. Protein content and starch damage are generally accepted as the major factors contributing to differences in farinograph absorption (Tipples et al., 1978). The hardness of the wheat kernel significantly influences the results of the milling process. Flour particles with greater starch damage lead to increased water absorption. Moreover, it is well known that water absorption increases with protein content. Thus, we would be expected to obtain good calibration for farinograph water absorption. As shown in Table 3, good validation sets were obtained for farinograph water absorption (ABS) with $r^2 = 0.97$ and SEP = 0.46 (Fig. 5).

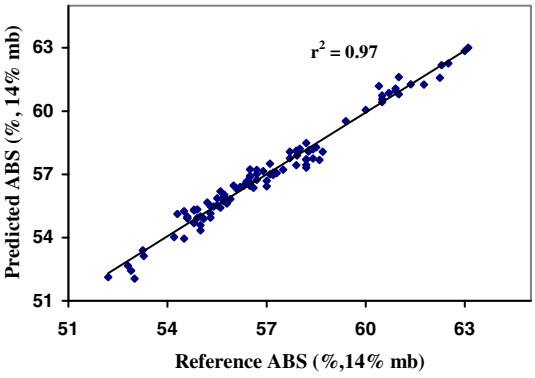

Fig. 5. Comparison of farinograph water absorption determined by prediction model and by reference method.

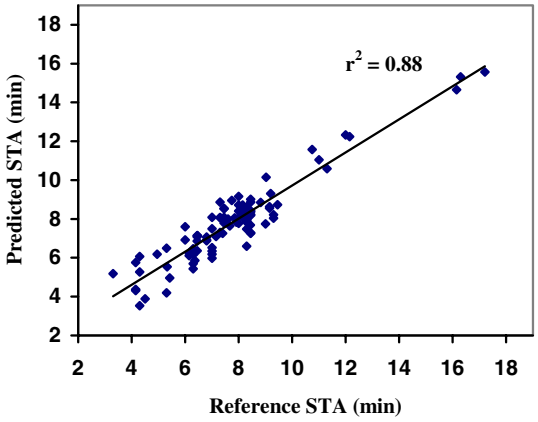

Fig. 6. Comparison of farinograph stability determined by prediction model and by reference method.

Degree of dough softening generally gives the rate of breakdown and strength of a flour: the higher the value, the weaker the flour. Thus, as increased, the protein decreased the degree of dough softening. Pfeifer, Vojnovich, and Anderson (1958) have shown how the farinograph profile increased as the protein content increased. Good validation sets were obtained for DDS20 and DDS12, with $r^2 = 0.93$; SEP = 8.7 and $r^2 = 0.90$; SEP = 11, respectively.

The farinograph stability gives some indication of the flour's tolerance for mixing. It is accepted that the stability increases as protein content increases. Wheat flour dough stability showed relativity high validation performance with $r^2 = 0.88$ and SEP = 1.0 (Fig. 6).

Farinograph quality number is a measure for the flour quality. Similar to the valorimeter number, FQN expresses the shape of the farinograph in a single number. Weak flour weakens early and quickly shows a low quality number, whereas strong flour weakens late and slowly shows a high farinograph quality number. The coefficient of determination was 0.92 with a SEP = 9.1.

Among farinograph parameters ABS shown the highest correlation ($r^2 = 0.97$) and lowest SEP (0.46) value. In the titration curve, a tolerance range of 480–520 FU is admissible. If the amount of water added exceeds 0.6%, the titration curve must be rejected and a new one must be recorded. Therefore, according to the precision method (0.6%), when a reference farinogram curve is needed NIR absorption (ABS) could be take in account preventing to perform the titration curve again.

3.3. Alveograph parameters

The statistical evaluation of calibration and validation sets of wheat flour for alveograph properties are summarized in Table 3.

Overpressure (P), commonly known as resistance of dough to deformation, is an indicator of dough resistance to deformation. Rasper, Hardy, and Fulcher (1985) show how P and W increased as the protein increased, but Aitken, Fischer, and Anderson (1944) revealed considerable differences among the flour samples with different amounts of protein. So, protein content is not the only factor affecting dough characteristics.

Model for deformation energy (W) showed good performance with SEP = 21.5 and $r^2 = 0.95$ for the validation set (Fig. 7). This relativity high validation was accurate for W range between 180 and 400, because SEP value is similar to the coefficient of variation (8%) of the alveograph method, whereas for a W range between 60 and 180, the SEP value was higher than the coefficient of variation.

Model for P/L showed bad performance with SEP = 0.04 and $r^2 = 0.79$ for validation set. However, the model for resistance of dough to deformation (P) was better than expected with SEP = 6.0 and $r^2 = 0.90$ for validation set (Fig. 8).

Among alveography parameters, only deformation energy (W) should be used for screening purposes, according the correlation and SEP values. The SEP value of 21 was relatively high when we intended to predict W

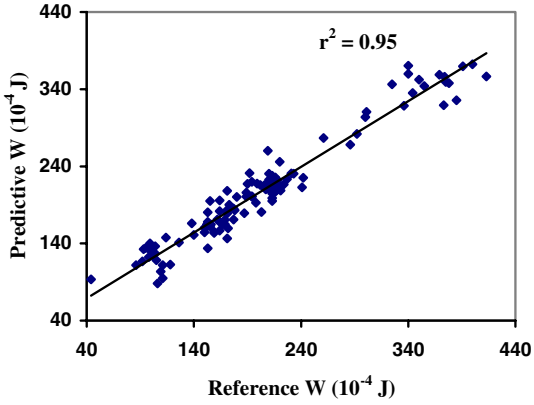

Fig. 7. Comparison of deformation energy determined by prediction model and by reference method.

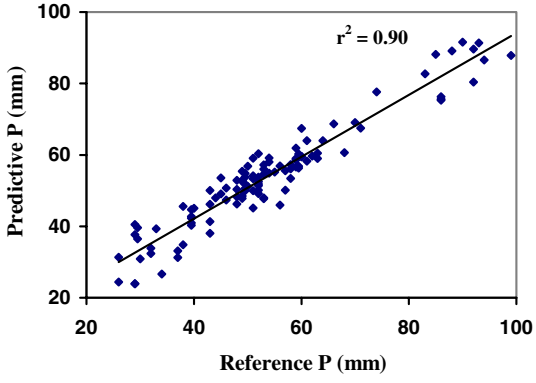

Fig. 8. Comparison of resistance of dough to deformation determined by prediction model and by reference method.

value between 40 and 140. However, for higher deformation energy, SEP value could be acceptable, according to the precision of standard method between laboratories (8%). The calibration equation for W joined to other suitably predicted parameters (protein, wet gluten, dry gluten, ash, starch damage and farinograph water absorption) could be used as discriminant criteria between different type of wheat flour.

A global calibration including these parameters could provide real-time results and delays corrective action.

4. Conclusions

Good NIR calibrations for a range of parameters related to flour bread making quality have been developed. Such calibrations will be of use to all who have an interest in the quality of wheat flour for particular processes whether they be millers or end-users. Because of the strong performance of the protein model, it was reasoned that successful NIR modelling of chemical constituents (wet gluten and dry gluten) and rheological properties (absorption, stability, degree of dough softening, resistance of dough to deformation and deformation energy) could arise as a result of correlations between them and protein content. The use of NIR has many advantages for those dealing with large numbers of samples due to the rapid nature of the technique and the ability to predict for a wide range of parameters from one assessment of a particular sample. In addition, only a small amount of material is required for analysis. Only one test could measure the most relevant flour constituents. Flour sample testing does not provide real-time results and delays corrective action. NIR transmittance can provide information about both physical and chemical characteristics of flour samples and have great potential for on-line quality control in the milling industry. Some models were accurate enough to apply them to estimating chemical or physical parameters by NIR transmittance spectroscopy in routine analysis.

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