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Monitoring of heat-treated wheat milling fractions by near infrared spectroscopic method

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RESEARCH ARTICLE

Abstract

Near infrared (NIR) technology is used effectively in the quality control field of cereal science and technology. The aim of this study is to elaborate on the usage of the near-infrared method for testing purposes, as well as on the recognition of the heat-treatment effects in the case of wheat milling fractions and changes in quality in these fractions. The heat treatment processes are being applied to increase the shelf life properties of products or to change the physical/rheological properties of goods. The wheat products and fractions examined in this study had been produced under industrial conditions The following products have been analysed: Hungarian wheat fraction (WF), Hungarian cake flour (CF) and aleurone-rich wheat flour (ARF). These basic flours were originated and produced by Gyermelyi Corp. flour mill. After that, the entire heat treatment process of WF, CF, ARF flour fractions were achieved and completed in cooperation of Bühler AG and Budapest University of Technology and Economics. The samples were collected based on the heat treatments, and involved hydrothermal and dry-thermal treated samples as well as untreated samples. The changes in the main chemical components (such as starch and protein) were analysed with dispersive spectrophotometers, using visible and NIR regions of the electromagnetic radiation with regards to the heat treatments. Close correlation has been established between the data of spectroscopic measurement techniques processed by various chemometric methods (e.g. principal component analysis, cluster analysis) and the types of treatments that were used. Not only differences caused by the milling technology and the heat treatment settings have been clearly observed, but also differences between the dry-thermal and the hydrothermal treatment. During this task, it became obvious that the NIR methods can detect the deviation in parameters of the heat treatments.

Keywords: hydrothermal-treated and dry-treated, wheat flour, NIR, PCA, CA

1. Introduction

Wheat is one of the most important cereals from the aspect of human nutrition, because the grain crops contain 60-70% carbohydrate, are easy to store and process and have a wide range of use (Cauvain, 2003; Lásztity, 1999). The average composition of a wheat grain is 71.9% starch, 12.2% protein, 1.9% non-starchy carbohydrate and 1.7% ash, respectively (Lásztity, 1999). Common wheat (*Triticum aestivum*) is generally classified as winter and spring wheat, and is used in different types of milling products in Hungary, such as the Hungarian wheat flour (WF) and the Hungarian cake flour (CF), as well as the newly developed aleurone-rich wheat flour (ARF) (Bagdi *et al.*, 2014). 'Pannon Wheat', which constituted the samples during our study, is a certification mark (i.e. one that gives legal assurance regarding the quality or other characteristics of certain products) that certifies the homogeneity and the excellent baking characteristics of the product and guarantees that it had produced under controlled conditions (Centre for Agricultural Research, 2011). The main parameters of the wheat that can be measured by infrared spectrophotometers are moisture, protein, starch, wet gluten, ash content and rheological parameters (Miralbés, 2003). The development of the on-line near infrared (NIR) technique allows for the real-time monitoring of quality. On-line NIR devices allow for the detection and improvement of grain and cereal product quality on all levels ranging from the raw materials (i.e. grain

and flour) to finished goods (i.e. bread, extrudates, etc.) (Evans *et al.*, 1999; Pojić *et al.*, 2012; Vigni *et al.*, 2009). The heat treatment process generally enhances the safety of the food. Heat treatments methods could be applied to realise physical and rheological modifications in wheat flour, or to enable longer shelf-life. There are two kinds of basic heat treatment processes, dry-thermal and hydrothermal. These can be distinguished based on the presence or absence of moisture (Lehtinen *et al.*, 2003). The dry heat treatment causes significant changes in the physico-chemical properties of the starch, without destroying its granule structure (Chiu *et al.*, 1999; Chung *et al.*, 2007). Despite this, the hydrothermal heat treatment has been employed for a long time for food preparations as well as chemical and structural modifications leading to the generation of desired coloured and flavoured compounds (Friedman, 1996). The results showed that the heat treatment of flour decreased the gluten extensibility and partial gelatinisation of the observed starch granules. The NIR technique had been successfully applied in the examination of various types of heat-treated foods, such as heated fish and shellfish meats as well as carrots (Uddin *et al.*, 2002). The NIR method has proved to be useful in the detection of heated foods (Uddin *et al.*, 2006). There are several reports about examining flour mixtures, but these prefer to observe only one kind of bakery products or raw materials. In contrast, the main aim of our study is to monitor different fractions of flour, which were carried out simultaneously to highlight the variability of the different kinds of products based on the different types of heat treatment effects. The main aim of this study is to monitor the chemical and physical/rheological characteristics of milling fractions using the NIR technique in an industrial environment, measuring the variability of the different kinds of products per the heat treatment settings.

2. Materials and methods

Samples

The *T. aestivum* wheat origin milling fractions (WF, CF and ARF) were produced by the Gyermelyi Corp. flour-mill (Gyermely, Hungary), the producing of these three flours were realised in there. After that, the entire heat treatment process of WF, CF, ARF flour fractions were achieved and completed in cooperation of Bühler AG and Budapest University of Technology and Economics. The allied wheat milling fractions that were examined (i.e. 9×3 samples 'Pannon Standard' fractions), were the CF and the standard WF. In addition, an ARF, which is a newly-developed experimental wheat flour, was also produced and developed at Gyermelyi Corp., Hungary with the cooperation of the Department of Applied Biotechnology and Food Science at the Budapest University of Technology and Economics, Hungary and the Bühler AG company in Switzerland (Bagdi *et al.*, 2014). 'Pannon Wheat', is a certification mark (i.e. one that gives legal assurance regarding the quality or other characteristics of certain products), that certifies the homogeneity and the excellent baking characteristics of the product and guarantees that it had been produced under controlled conditions (Centre for Agricultural Research, 2011). The compositions of the samples are summarised in Table 1.

The samples were collected based on two aspects, dry-thermal treatment and hydrothermal treatment. First, during the dry-thermal treatment, the samples were heated up using thermo pneumatics and held at a temperature of 100 °C for 12 minutes. During the hydrothermal process, the flours were heated up with steam, i.e. 5 l/h steam for 5 minutes and they were dried with the thermo pneumatics. At the end of these processes, the moisture content of the products was adjusted to 5% at the dry-thermal treatment, and to 10% at the hydrothermal treatment, respectively. The parameters of the treatments are listed in Table 2. All samples had a permanent moisture content (approximately 10% based on Bagdi *et al.*, 2014 and Sayaslan *et al.*, 2006) during these experiments in the case of each treatment.

Reference materials

The reference materials originated from Sigma Chemical Co. (St Louis, MO, USA) and were gluten from wheat and unmodified wheat starch. The chemical composition of the flour samples, such as ash, protein, starch, fibre and, crude fat content of the untreated CF, standard WF and the ARF were measured in accordance with international standard methods (AACC, 1999; ICC, 1996).

Table 1. Compositions of the examined wheat flour (Bagdi *et al.*, 2014; Bucsella *et al.*, 2016).

Composition	Hungarian wheat flour % dry weight basis	Hungarian cake flour % dry weight basis	Aleurone-rich wheat flour g/100 g
Ash	1.7	0.6	3.8
Protein	12.2	11.4	26.6
Starch	71.9	69.4	47.6
Total dietary fibre	1.9	4.1	17.5
Crude fat	1.9	1.5	4.2

Table 2. Parameters of applied methods in the case of different types of thermal treatments (Bucsella *et al.*, 2016, with kind permission of Elsevier).[1]

Types of milling product	Abbreviation	Steam moisture (l/h)	Retention time (min)	Temperature of product (°C)
Untreated CF	CF	–	–	–
Untreated WF	WF	–	–	–
Untreated ARF	ARF	–	–	–
Dry-thermal treated WF	Dth WF	0.0	12.0	102.0
Dry-thermal treated WF	Dth WF	0.0	12.0	90.0
Dry-thermal treated WF	Dth WF	0.0	6.0	90.0
Dry-thermal treated WF	Dth WF	0.0	12.0	101.0
Dry-thermal treated CF	Dth CF	0.0	15.0	107.0
Dry-thermal treated CF	Dth CF	0.0	8.0	106.0
Dry-thermal treated CF	Dth CF	0.0	12.0	101.0
Hydrothermal-treated WF	Hyd WF	5.0	5.0	96.0
Hydrothermal-treated CF	Hyd CF	5.0	5.0	96.0
Hydrothermal-treated WF	Hyd WF	10.0	5.0	96.0
Hydrothermal-treated CF	Hyd CF	10.0	5.0	95.0
Hydrothermal-treated WF	Hyd WF	20.0	5.0	95.0
Hydrothermal-treated CF	Hyd CF	20.0	5.0	96.0
Dry-thermal treated ARF	Dth ARF	0.0	12.0	101.0
Hydrothermal-treated ARF	Hyd ARF	5.0	5.0	96.0
Hydrothermal-treated ARF	Hyd ARF	10.0	5.0	96.0
Hydrothermal-treated ARF	Hyd ARF	20.0	5.0	96.0

[1] CF = Hungarian cake flour; WF = Hungarian wheat flour; ARF = aleurone-rich wheat flour.

Spectroscopic measurements

Samples and reference materials were scanned using dispersive instrument to collecting the raw spectra. Three independent scans were recorded from each spectral sample, and the means of these replicates were used in subsequent calculations. The dispersive NIR instrument – NIR Systems 6500 monochromator system (Foss-NIR Systems, Silver Spring, MD, USA) fitted with a rapid content analyser and micro-sample cup equipped with threaded back. Samples were scanned from 400 nm to 2,498 nm in reflectance mode (R mode: Si detector (400-1,098 nm) and PbS detector (1,100-2,498 nm)). Data were collected every 2 nm (1,050 data points per spectrum).

Data processing

Spectral and reference data were processed using Vision 3.20 (Minneapolis, MN, USA), Microsoft Excel 2013 (Microsoft Corporation, Redmond, WA, USA), and Statistica 11 (StatSoft, Inc., Tulsa, OK, USA) software packages. The D2OD 8/0 nm gap-segment (Hopkins, 2001; Norris, 1983) setting in the case of each spectra and after these derivatives spectra were applied principal component analysis (PCA) method (Martens and Næs, 1991; Wold *et al.*, 1987) and cluster analysis (CA) method (Heise and Winzen, 2002), respectively.

3. Results

During this study, the results of the heat-treated wheat fractions were systemised per the different mathematical treatments, and the changes in main protein peaks in case of heat treatments and samples. The systems had a permanent moisture content during these experiments in the case of all treatments, so the changes of the water peaks were irrelevant and were not influenced by the heat treatments. Despite this, the proteins reacted very sensitively in the case of all kinds of heat treatments.

Raw spectra

The separation of the raw spectra in the case of the untreated samples (WF, CF and ARF) was detected in the visible range (400-780 nm) (data not shown). The deviations of the treated fractions (i.e. hydrothermal and dry-thermal treated Hungarian CF, WF and ARF) had already appeared in the case of the raw spectra in visible as well as NIR ranges (Figure 1). The huge baseline shift between the ARF fraction and the other two fractions were caused by two factors: the deviation of the treated ARF from the treated

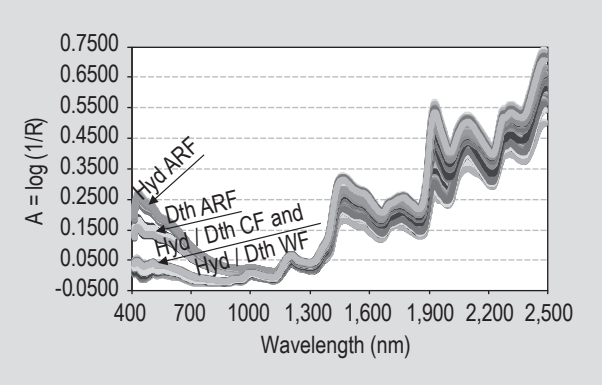

Figure 1. Raw spectra of visible-near infrared (400-2,500 nm) in the case of hydrothermal and dry-thermal treated samples. Hyd = hydrothermal treated; Dth = dry-thermal treated; WF = Hungarian wheat flour; CF = Hungarian cake flour; ARF = aleurone-rich wheat flour.

WF and the CF fractions derive from the difference in chemical and physical properties (Table 1). In the case of the ARF fraction, the colour causes the main variability in the visible range, because it has a higher aleurone amount than the WF and CF fractions.

D2OD spectra

After the evaluation of the raw spectra, the second order derivative of spectra (D2OD) was calculated with 8/0 nm gap-segment setting in order to retain the sensitivity and to get more informative, optimal signals about the milling fractions per the treatments. During these experiments based on the present literature, the peaks of the D2OD 8/0 nm spectra could be identified. According to Heise and Winzen (2002), the most informative protein ranges were highlighted, such as 2,040-2,080 nm (protein peak value is 2,055 nm), 2,160-2,190 nm (protein peak value is 2,180 nm) and 1,960-2,000 nm (protein peak value is 1,980 nm), respectively. Regarding biochemistry interferences, different kinds of vibrations were identified. In the case of 2,055 nm, the vibration is amide II/A, which has the highest intensity between the protein bands. The amide I/III combination belongs to the 2,180 nm peak, and the asymmetric N-H stretch and amide II combination is due to the value of 1,980 nm (Gergely and Salgó, 2007; Shenk *et al.*, 2007).

D2OD spectra in the first protein peak 2,055 nm

Afterwards, the D2OD 8/0 nm spectra, which belongs to the highlighted protein ranges per the effects of the heat treatments, was evaluated first in the case of the 2,055 nm protein peak. As shown in Figure 2, the deviations of the hydrothermal treated, dry-thermal treated and the untreated fractions appeared in the range of 2,040-2,080 nm. In the case of the WF and CF fractions by the main protein peak of this range (i.e. 2,055 nm), the separations could be observed between the three kinds of treatments (Figure 2A and 2B). Checking the ARF fraction, whose chemical composition differs the most from the rest of the two fractions (i.e. WF, CF), the protein content is significantly higher than the other two fractions (Table 1). In the case of the ARF fraction, shifts between the hydrothermal and dry-thermal treatment have appeared by 2,055 nm (Figure 2C). According to the three treatments by the WF and CF fractions, the total separations could be observed based on the D2OD 8/0 nm spectra (Figure 2A and 2B). In the case of the WF, CF and ARF fractions, differences have been detected concerning quality and quantity.

Subsequently, the D2OD 8/0 nm spectra was used to compose a score plot of PCA for the formerly highlighted range, i.e. 2,040-2,080 nm (Figure 2D-F). The score values of the first principal component (PC 1) and the third principal component (PC 3) showed the main deviations between the WF fractions, per the heat treatments. In the case of the dry-thermal treated WF fraction, the deviations between the different types of dry-thermal treated samples have already been detected (Figure 2D). In the case of the CF and ARF fractions, the differences in the settings of hydrothermal treated samples have also appeared, besides the dry-thermal treated and untreated samples (Figure 2E and 2F). In both hydrothermal treated CF and ARF per the highest steam moisture content (Table 2), the samples separated well from the rest of the samples, so the protein structure is changed based on the most effective settings and the first and second principal component (PC1 and PC2), respectively.

D2OD spectra in second protein peak 2,180 nm

The other main protein peak of the examined range was around 2,180 nm (i.e. 2,160-2,190 nm). The deviations of the hydrothermal treated, dry-thermal treated and untreated fractions of WF, CF and ARF were not separated clearly in this range (Figure 3). Checking the 2,180 nm peaks in the case of all examined samples, baseline shifts have been experienced firs in a quantitative sense (Figure 3A-C). In the case of the WF and CF, in D2OD 8/0 nm spectra there were no significant deviations between the hydrothermal treated and untreated samples, therefore in this range, the different settings of hydrothermal heat treatment did not clearly appear by the spectra. The applied PCA method is able to detect variances between the different types of dry-thermal and hydrothermal treated samples based on PC1 and PC2 in the case of WF fraction (Figure 3D).

Despite this, the hydrothermal treated, dry-thermal treated and untreated CF fractions were not separated clearly per the heat treatments by the PCA score plots as shown in the Figure 3E. But the different settings of the hydrothermal treated samples showed up in the case of the ARF fraction by PCA score plot (Figure 3F).

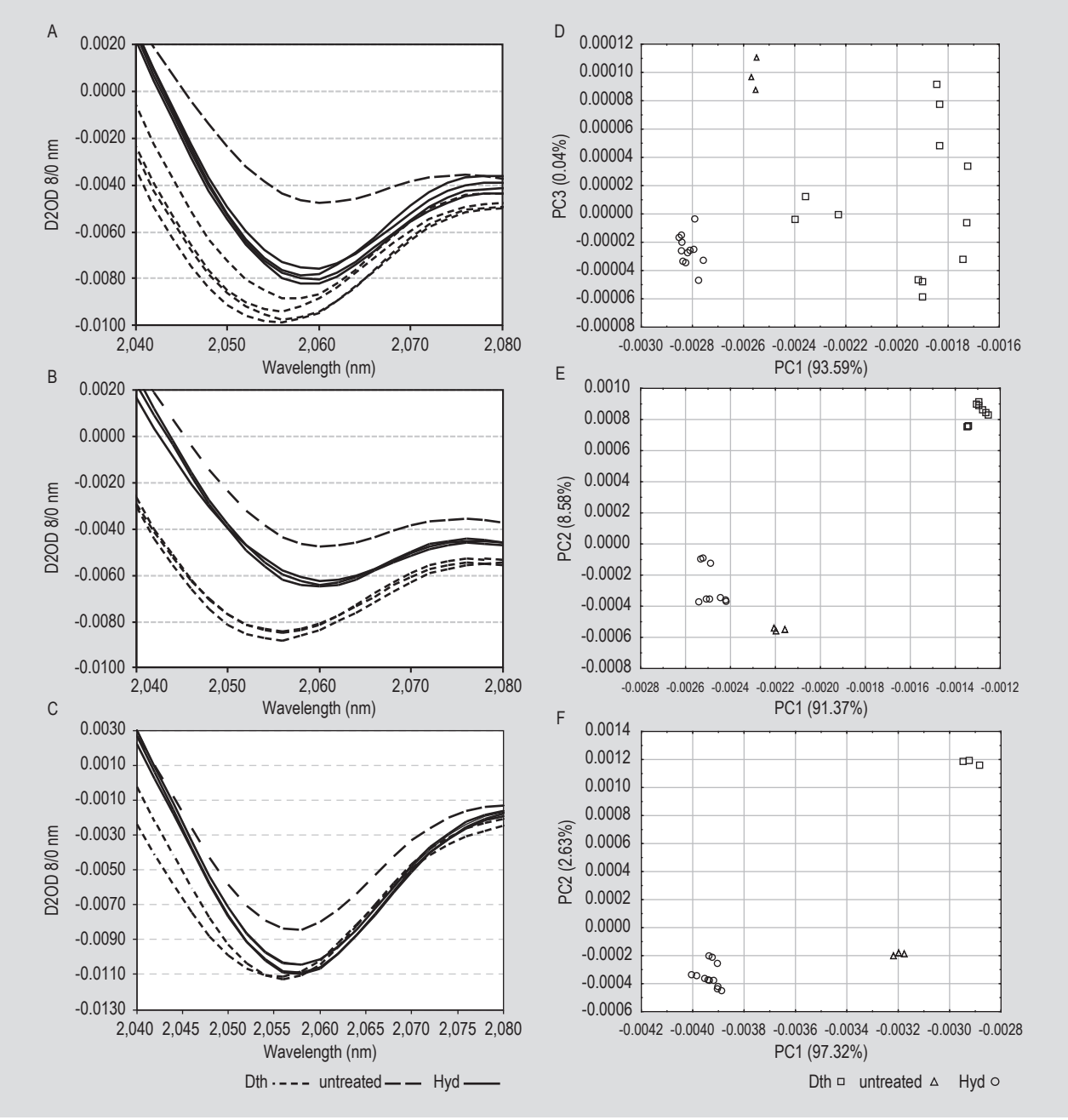

Figure 2. The D2OD 8/0 nm spectra and the principal component analysis score plots in the case of Hungarian wheat flour (A-D), Hungarian cake flour (B-E) and aleurone-rich wheat flour (C-F) samples in reference to the untreated, dry-thermal treated (Dth) and hydrothermal treated (Hyd) samples in the highlighted range of 2,040-2,080 nm. PC = principal component.

This fraction has higher protein content on the score plot, this could be observed mainly in separations based on the heat effects. In the case of all samples, changes caused by the different treatments could be monitored by the NIR spectroscopic method. This non-destructive method was able to detect the deviations between the samples even if there was no major difference in the composition of the fractions using the same dry-thermal and hydrothermal treatment settings. The Pearson correlation values (r) were calculated between the first three loadings of PCA of the total range and the D2OD 8/0 nm NIR spectra of pure reference materials in the case of the dry-thermal and hydrothermal treatments. The starch correlated with the loadings of PC 1 of hydrothermal-treated samples (r=0.977) and the loadings of PC 3 with protein (r=0.549), respectively. In the case of the dry-thermal treated samples, the loadings of PC 1 had a high (r=0.969) correlation with starch and the loadings of PC 3 with protein (r=0.583).

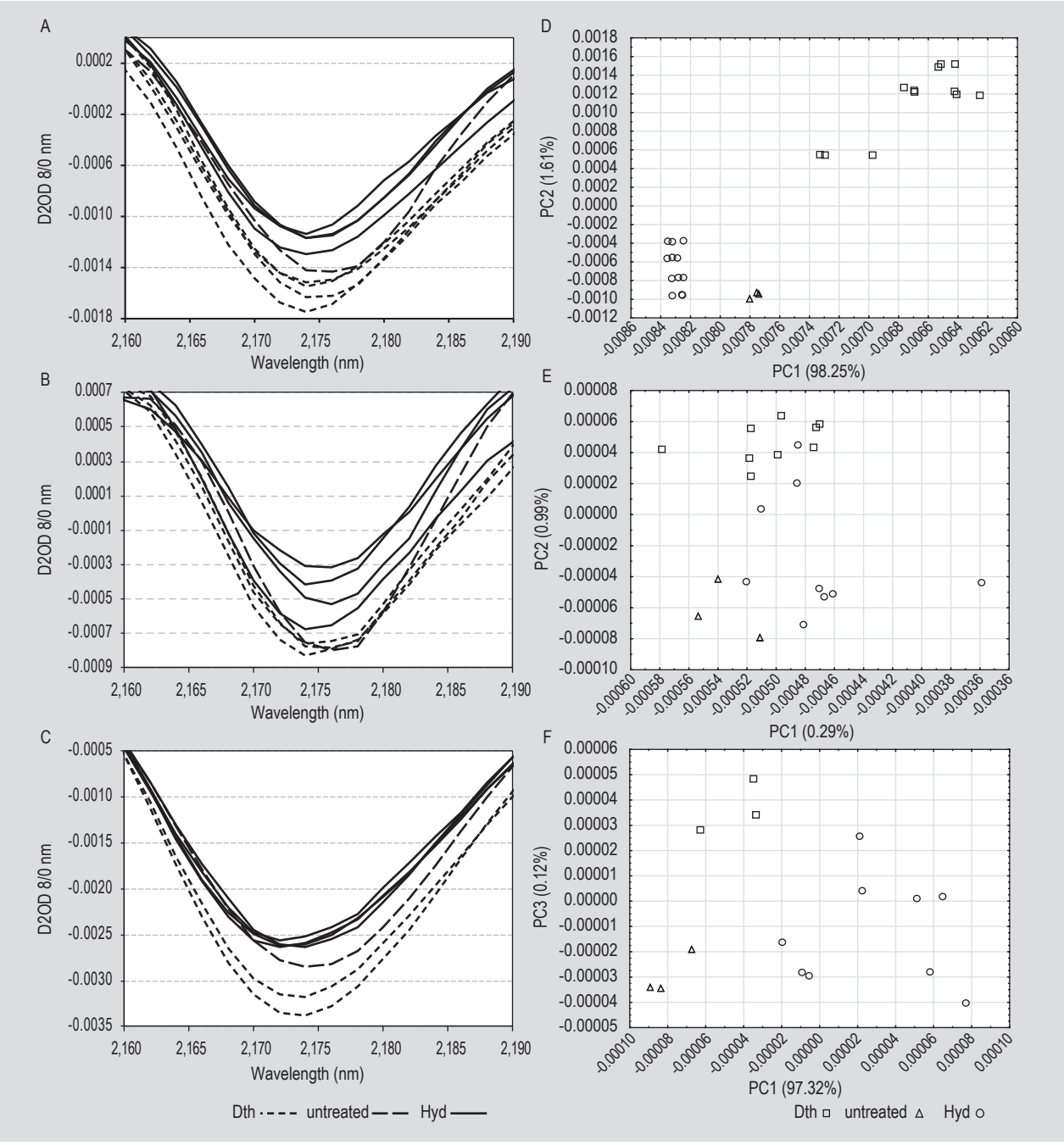

Figure 3. The D2OD 8/0 nm spectra and the principal component analysis score plots in the case of Hungarian wheat flour (A-D), Hungarian cake flour (B-E) and aleurone-rich wheat flour (C-F) samples in reference to the untreated, dry-thermal treated (Dth) and hydrothermal treated (Hyd) samples in the highlighted range of 2,160-2,190 nm. PC = principal component.

Cluster analysis dendrogram

On the CA dendrogram (Figure 4), all fractions were separated clearly; first, the ARF fraction in the case of the range of 1,100-2,500 nm. Between the CF and the WF fraction, the separation could be observed primarily per the treatments, and not per the type of fractions, owing to their chemical similarity. On the other hand, in the case of the CF and WF fractions, the CA method was able to detect the differences between these two fractions, within the dry-thermal treated and hydrothermal-treated samples.

The separations of the heat treatments of the fractions in the highlighted protein range have appeared as shown in Figure 5. In the 2,040-2,080 nm range, the treatments of the ARF fraction are separated at a very high level, and the initial differences between the untreated and treated ARF fractions are shown in Figure 5C. In the case of the other two

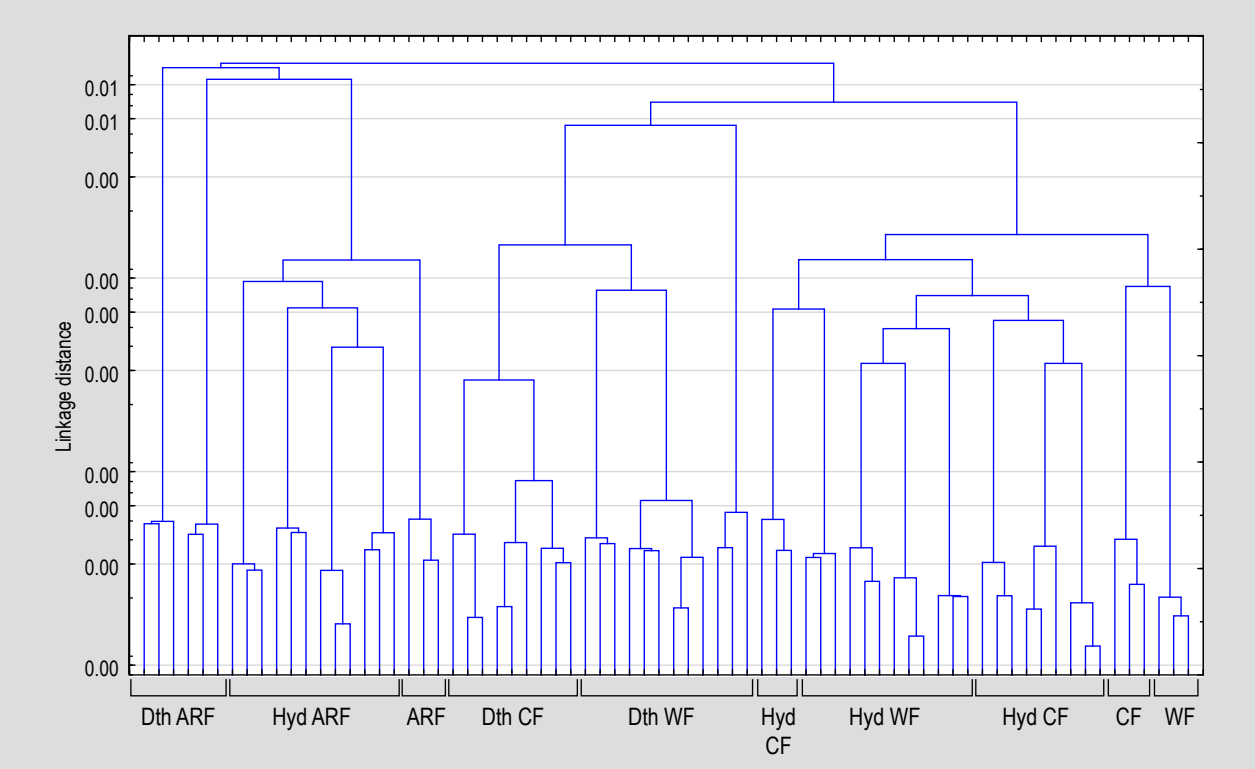

Figure 4. The cluster analysis dendrogram in the case of Hungarian wheat flour (WF), Hungarian cake flour (CF) and aleurone-rich wheat flour (ARF) samples in reference to the untreated, dry-thermal treated (Dth) and hydrothermal treated (Hyd) samples in the range of 1,100-2,500 nm.

fractions, the WF were separated soon (Figure 5A), but the CF fractions were separated later than the other fractions (Figure 5B). In the case of the 2,160-2,190 nm range, the same pattern could be observed per the separation of the fractions, but this range is less sensitive compared to the 2,040-2,080 nm range (Figure 5D,E,F).

4. Discussion and conclusions

During the evaluation of the milling fractions, not only the differences between the fractions were examined, but also the changes concerning the dry and wet heat treatments in the highlighted protein ranges. The heat treatments are very important for increasing the shelf-life of products, so this method is very useful for the industry. The differences between the products were represented on the D2OD spectra and PCA score plots after each treatment in the case of 2,040-2,080 nm range. Regarding the WF and CF fractions by the main protein peak of this range, separations could be observed between the three kinds of treatments. In the case of the ARF fraction, whose chemical composition differs the most from the rest of the two fractions, shifts have been appeared between the hydrothermal and dry-thermal treated ARF by 2,055 nm. Per the three treatments by the WF and CF fractions, the total separations could be observed based on the D2OD 8/0 nm spectra and PCA plots and CA dendrogram, too. In the case of the WF, CF and ARF fractions, differences have been detected in the quality and quantity sense. During this task, it became apparent that the NIR methods are able to detect the dissimilarities between the different types of heat treatments within one fraction with different sensitivity. The flour samples were monitored in this study based on the heat treatments with NIR equipment. This is a novel step in beginning to realise the on-line technique during the heat treated milling process. The use of the on-line technique is leading to a distinctive improvement in the quality of the product, because this technique allows for an immediate intervention during the manufacturing process.

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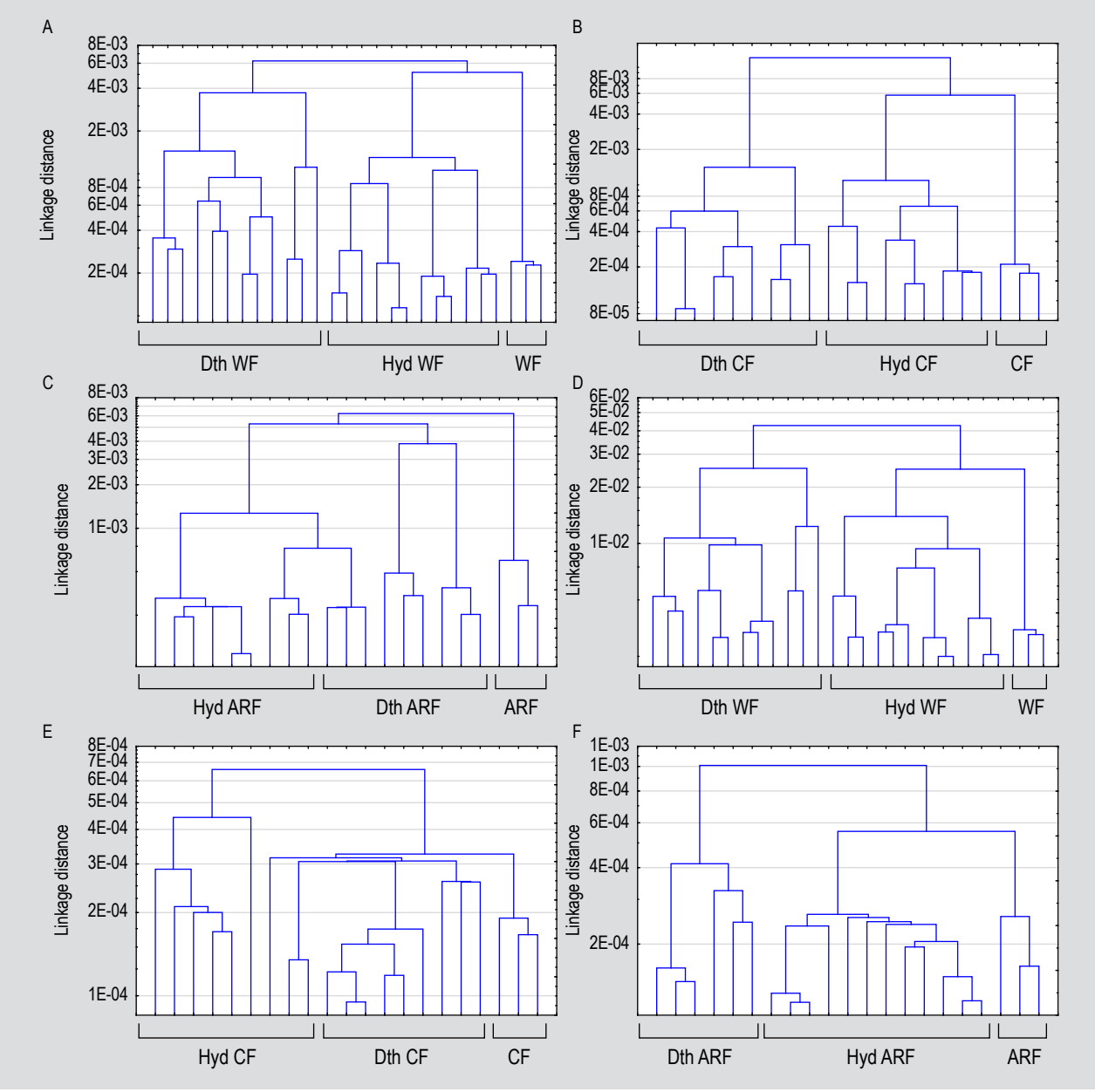

Figure 5. The cluster analysis dendrogram in the case of Hungarian wheat flour (WF) (A-D), Hungarian cake flour (CF) (B-E) and aleurone-rich wheat flour (ARF) (C-F) samples in reference to the untreated, dry-thermal treated (Dth) and hydrothermal treated (Hyd) samples in the range of 2,040-2,080 nm (A-C) and 2,160-2,190 nm (D-F).

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