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Effect of particle size and spectral sub-range within the UV-VIS-NIR range using diffuse reflectance spectra on multivariate models in evaluating the severity of fusariosis in ground wheat

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2 **Effect of particle size and spectral sub-range within the UV-VIS-NIR range**
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4 **using diffuse reflectance spectra on multivariate models in evaluating the**
5 **severity of fusariosis in ground wheat *)**
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Abstract

Control (crops grown in natural conditions) and *Fusarium* head blight (FHB) damaged (crops inoculated with *Fusarium culmorum* conidia) grain of four wheat cultivars was ground and sieved into three fractions of different particle size. A series of blended samples differing in content of damaged material were prepared within fractions and cultivars, and diffuse reflectance spectra recorded within the 200-2500 nm wavelength range. PLS models for the percentage of damaged material in blended samples were built for each of 12 series within different spectral ranges, and the root mean squared error of cross-validation (RMSECV) used for assessment of model performance. Errors using the models were lowest for the finest fraction independent of spectral range, however, their values depended on the cultivar. RMSECV for the finest fraction averaged over cultivars ranged from a little below 3.0 (when the UV sub range was used or participated with another one) to 8.1 % (when only the NIR sub-range was used). For the medium and coarse fractions, averaged errors showed the same tendency of dependence on the sub-range(s), however, with higher values that increased with an increase in particle size. In conclusion, within the different fractions of particle size and spectral ranges, the most sensitive to the presence of damaged material were models developed for the finest fraction and when the UV sub-range was used in modeling.

Key words: fusarium head blight, scab, wheat, reflectance spectroscopy.

Introduction

The quality of cereal grains, primarily their possible contamination with harmful substances, is of key importance for a safe human diet. An obvious strategy in delivering safe grain is the detection of possible contamination at the earliest possible stage in the “farm-to-table” chain of food production. One of the problems that arise here is the protection of grain, before its storage and/or processing, from contamination by any grain that has been infected with soil fungi, since developing fungi can produce harmful secondary metabolites (Mirocha et al. 2003). In countries with a temperate climate, the soil fungi most frequently infecting flowering cereals and then their grain are *Fusarium* spp. *Fusarium* spp. infecting cereals are known to cause a disease called *fusarium* head blight (FHB) (or scab, or fusariosis). FHB causes considerable economic loss (Johnsson et al. 2003) and often causes contamination of damaged grain with mycotoxins (Dexter and Nowicki 2003), first of all with deoxynivalenol (DON, vomitoxin). DON contaminated food and feed can cause severe health problems, both in humans and livestock (see Dexter and Nowicki 2003, and references therein). Regulations for DON content specify different limits for unprocessed wheat in different countries (see, e.g., Dexter and Nowicki 2003). These limits are mostly 1 mg/kg or higher (with the exception of Austria). There are reports of the occurrence of DON in cereal-based commodities on the market in many countries (Gareis et al. 2003). For these reasons considerable effort has been made to develop quick and cost effective methods that could be useful in the detection of fusariosis in cereal grain and that could replace tedious chemical methods, which are mostly based on chromatographic analysis (Mirocha et al. 2003). Of such methods the most popular ones seem to be those fully, or in part based on optical measurements. Quantitative evaluation of change in kernels colour and other visible symptoms supplemented with evaluations extended into near infrared region was found useful for detection of scab/DON damaged single kernels (Dowell et al. 1999, 2002, Delwiche and Kim 2000, Delwiche 2003, Delwiche and Hareland 2004, Polder et al. 2005), and can be also used for development of automatic systems for

1
2 single kernel classification (Pasikatan and Dowell 2001, Pearson et al. 2004, Dowell et al. 2006).
3
4 Another approach that can be useful in the detection of scab/DON damaged grain is based on
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6 optical measurements of ground grain followed with multivariate calibration (Abramović et al.
7
8 2007, Kos et al. 2002, 2003, 2004, Siuda et al. 2006, 2008). Errors of calibration models are a key
9
10 question in such an approach as they determine the ability of the method to detect fusariosis. Our
11
12 previous papers (Siuda et al. 2006, 2008) showed that the most promising PLS models for the fine
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14 fraction (particle size less than 0.180 mm) of ground winter wheat were the models based on spectra
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16 recorded within the UV range, or when the measurement covered additionally the VIS range. Errors
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18 of these models when applied to DON content resulted in a limit of detection below 1 ppm (Siuda
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20 et al. 2008). This paper aims to check whether that finding is valid when other fractions of ground
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22 winter wheat are used in diffuse reflectance measurements and to find which fraction ensures the
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24 lowest error (highest accuracy) in the calibration model.
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33 **Experimental**

34 **Material**

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36 Two lots of winter wheat (*Triticum aestivum* ssp. *vulgare*, cv. Turnia harvested in 2005) grain were
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38 obtained from two plots. One lot (referred to as control) was harvested from a plot where plants
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40 grew in natural conditions, while the other one (damaged) was from a plot where heads were
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42 inoculated with a suspension of *Fusarium culmorum* conidia ($1 \cdot 10^6 \cdot \text{ml}^{-1}$) during the flowering stage.
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44 The *F. culmorum* isolate used for inoculation was previously classified as DON chemotype on the
45
46 basis of the presence and functioning of *Tri7* and *Tri13* genes detected with a PCR assay. After
47
48 harvesting, control and inoculated lots were dried in air at room temperature to a moisture content
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50 of ca. 11% and then stored in a refrigerator at temperature 4°C. Grain from both categories was
51
52 ground (1.0 mm screen, rotor speed 7000 rpm, ZM1, Retsch, Haan, Germany) and sieved. Three
53
54 fractions of particle size were sieved: fine (<0.180mm), medium (>0.180 and <0.25mm) and coarse
55
56 (>0.25). Within the fractions control and damaged material was blended in different proportions
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1 ranging from 0% to 100% of damaged constituent in the mass of the sample. Essentially, the same
2 procedure for gaining and processing the material was repeated the following year (2006) with cvs.
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6
7 Tonacja, Satyna and Trend. Grain was ground with a ZM200 Retsch grinder (screen and rotor speed
8
9 the same as above), while the set of screens used for sieving was: fine (<0.18mm), medium (>0.180
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11 and <0.355mm) and coarse (>0.355 and <0.710mm). Sieving was done with a sieve shaker (AS20,
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13 Retsch) operating at maximum amplitude in a sequence (up to five) of 10 min time intervals,
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15 interrupted for manual manipulations necessary to prevent clogging of the material. As a result,
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17 three series of blended samples of different fractions, each consisted of ca. 30 samples were
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19 obtained for each analysed cultivar.
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26 Measurements

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28 A UV-VIS-NIR commercial spectrophotometer Cary 5000 (Varian Inc., Mulgrave, Australia) was
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30 used to record diffuse reflectance spectra in log(1/R) mode in the range 200 to 2500 nm, every 2
31
32 nm. For each sample, 5 scans at two crossed positions of the cuvette were taken and averaged. Each
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34 spectrum was scaled with reference to a base line spectrum recorded from the PTFE reference plate
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36 (a part of internal DRA accessory, Varian Inc.). An equal volume of approximately 1-ml of the
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38 material was taken from each blended sample and put into a quartz cuvette of diameter 17-mm
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40 equipped with a spring driven piston (a part of internal DRA accessory, Varian Inc.) that pressed the
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42 material. No special circumstances, such as air- and/or temperature conditioning in the laboratory
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44 during measurements were maintained.
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51 Multivariate calibration

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53 The PLS (Partial Least Squares) foundations have been broadly presented elsewhere (see eg. Geladi
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55 and Kowalski 1986, Vandeginste et al. 1997; 1998). Briefly PLS tries to find a relationship between
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57 the latent structure in spectra set and the latent structure in responses. It is carried out by finding
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59 specific directions in data space, the so-called latent variables (LV). In practical use of modelling by
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1
2 latent variables methods such as PLS, first of all, the number of significant latent variables
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4 (components) has to be determined for each calibration model. Cross-validation approach provides
5
6 a very reliable way for this (Wold 1978). A number of alternations is performed accordingly to
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8 some permutations schemes. Parameters used for assessment of PLS models are then the root mean
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10 square error of calibration (RMSEC) and cross-validation (RMSECV). It is commonly accepted that
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12 the number of LV giving the lowest RMSECV is used as an optimal. Besides other statistical
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14 parameters are used (eg. the coefficient of multiple determination (R^2), for more complete review
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16 see ASTM Standards 2000).
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23 Calibration models for the percentage of damaged material in blended samples (assumed as a
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25 measure of the severity of fusariosis) were built using commercial software PLS_Toolbox3.0
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27 (Eigenvector Research, Inc., Wenatchee, USA). Different pre-processing of spectra were tested,
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29 including MSC, SNV and differentiation. As the models based on differentiation (eleven-points
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31 Savitzky-Golay first derivative smoothing filter) and centring of the spectra give the best
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33 performance, these models were studied. Additional manipulations on spectra mentioned in the
34
35 following sections were performed with our own codes written in Matlab® software package
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37 (Mathworks Inc., Natick, MA, USA).
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45 **Results and discussion**

46 Calibration models for differing levels of severity of fusariosis within each cultivar and fraction
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48 were built in the UV, VIS and NIR spectral ranges and for all possible combinations. The results
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50 obtained are presented in Table 1. R^2 is not shown in the results of calibration, since it was always
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52 satisfactory (better than 0.9). Model parameters presented in Table 1 show noticeable differences
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54 when moving from one cultivar to another within a fraction, and from fraction-to-fraction within a
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56 cultivar. In order to make these results more easily visible, the RMSECV model errors were
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58 averaged over cultivars within each fraction and spectral range, and plotted in Figure 1. It can be
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1
2 seen from Figure 1 that independently of spectral range, model errors tend to increase considerably
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4 with an increase of particle size. This error enlargement is especially prominent when the UV range
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6 is included in the measurements and the NIR range excluded. The former finding can be supposed
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8 to be caused by changes in path length of diffusely reflected radiation with the change of mean
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10 particle size. This effect is well known in NIR reflectance spectroscopy (see, Osborne et al. 1993,
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12 Pasikatan et al. 2001). In some publications a tendency has been reported towards model errors
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14 being dependent on particle size that was qualitatively similar to that seen in Figure 1, despite the
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16 subject of their investigations being different from those reported here. Hareland (1994) used NIR
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18 reflectance spectroscopy to predict the percent volume of wheat flour particles of three size ranges:
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20 <10, 10-41 and 41-300 μm . The authors obtained an increase of both standard errors of cross-
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22 validation (0.26, 0.87 and 1.11) and errors of prediction (0.35, 1.17 and 1.13) with increasing
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24 particle size. Pasikatan et al. (2003) investigated the possibility of application of PLS models based
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26 on reflectance spectra (recorded in the 400-1700 nm wavelength range) to predict the granulation of
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28 wheat ground with an experimental roller mill. The authors found SEP equal to 3.53, 1.83, 1.43 and
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30 1.30 for the >1041, >375, >240 and >136 μm size fractions respectively. Kos et al. (2007) reported
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32 results on optimizing particle size for ATR-MIR measurements used as a tool for screening fungal
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34 infection and assessment of DON content in ground maize. The authors compared the repeatability
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36 of MIR spectra at several wavelengths for fractions of particle size >0.71, 0.71-0.50, 0.50-0.25,
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38 0.25-0.10 and <0.10 mm, and they found an increase of repeatability with decreasing particle size.
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40 The results presented here together with the results from the cited reports suggest that a general rule
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42 may be that an increase of errors in multivariate calibration models based on reflectance spectra of
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44 ground wheat follows from an increase in the mean particle size of analysed samples. In addition,
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46 our results suggest that this dependence holds its validity also in the spectral ranges other than NIR,
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48 in particular in the UV.
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As regards the improvement of the models observed when the UV range is included in modelling,

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2 this finding is in line with our previous results obtained for fine fraction only and a lower number of
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4 cultivars (Siuda et al. 2006; 2008). Therefore, considering the results presented here, there now
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6 exists better evidence to suggest that improved results can be obtained from the UV sub-range than
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8 from the two other sub-ranges considered. However, even though better results can be obtained
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10 from the two other sub-ranges considered. However, even though better results can be obtained
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12 when UV is merged with other spectral ranges, it is worth noting that the NIR range, which is the
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14 most commonly used range for the analysis of food and grain properties, provided results of
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16 noticeably poorer quality (unless it was merged with other sub ranges) especially in the case of
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18 fractions other than fine. Hence, one can state that inclusion of the UV sub-range improves the
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20 quality of PLS calibration. This observation remains in accordance with the fact that the main
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22 constituents contributing to the chemical composition of wheat have absorption bands located in the
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24 UV (for references see Siuda et al. 2008). Also, general properties of the spectra support this
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26 opinion about the importance of this spectral range. Exemplary spectra (shown for cultivar Tonacja
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28 only, since the general features of the spectra discussed below are similar for the other cultivars)
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30 presented in Figure 2a show vertical shifts caused by differences in particle size not only in the NIR
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32 range but also in the UV range. Plots presented in Figure 2b show that the variance in sets of spectra
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34 depends considerably on both the sub range of wavelength and on the particle size, nevertheless
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36 they illustrate the considerable amount of variance present in the UV range. In the NIR range, the
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38 variance due to the presence of spectral bands is superimposed on a monotonic background that
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40 contributes more strongly for coarser fractions. In the UV range, a strong contribution to variance
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42 from a background of perhaps non-monotonic character, can also be seen.
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52 This considerable contribution from the background to the variance present in sets of spectra seems
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54 to explain why PLS models based on differentiated spectra were found to be better than models
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56 based on non differentiated spectra (Siuda et al. 2006), since differentiation of the spectra reduces
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58 the variance coming from broad shaped features present in the structure of the spectra. Careful
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60 inspection of the curves which have been presented seems to suggest that after subtraction of the

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2 background, variance caused by changes in intensity of absorption bands could appear to be most
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4 prominent in the UV sub range, especially for fine fractions. Additional insight into the information
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6 that spectra contain in particular spectral sub ranges can be found from inspecting the values of the
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8 regression coefficient of PLS model. This coefficient, usually denoted as b , has a greater absolute
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10 value at wavelengths that contribute more to a calibration model. In Figure 3 are shown plots of
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12 absolute values of b versus wavelength for the best PLS models based on the whole spectral range
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14 for all 12 series of blended samples. Although the details of the superimposed plots cannot clearly
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16 be seen, the figure demonstrates that the largest absolute values of regression coefficients appear in
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18 the UV sub region. In order to evaluate quantitatively the importance of particular spectral ranges
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20 for PLS modelling, mean squared values of regression coefficients, defined as
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$$\langle b \rangle_{\text{range,cultivar,fraction}} = \sqrt{\frac{1}{N} \sum_{i=1}^N b_i^2},$$

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30 where b_i is the value of the coefficient at the i -th wavelength, and N is the number of wavelengths in
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32 the spectral range, were calculated for all cultivars, fractions and the UV, VIS and NIR spectral sub
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34 ranges. These values were next averaged over cultivars within fractions and ranges, and the results
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36 relevant for particular spectral sub ranges and fractions are presented in Figure 4. These values
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38 plotted against the errors of relevant PLS models presented in Figure 1 resulted in the data points
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40 shown in Figure 5. The data points in Figure 5 demonstrate regularity within a fraction: the larger is
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42 the mean correlation coefficient, the lower is the error of the model. This regularity also supports
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44 what has been stated above, i.e. that the UV sub-range can be considered as more informative than
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46 the other two.
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51 52 53 **Conclusions**

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55 In conclusion, the results presented suggest that the most promising approach for detecting
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57 fusariosis in winter wheat grain with diffuse reflectance spectroscopy seems to be that using of fine
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59 fractions of ground material and performing the measurements in the UV-VIS spectral range.
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However, as the measurements made solely in the UV range provide results of almost the same

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2 quality as those in the UV-VIS range, such measurements can be recognised as an even more
3
4 advantageous alternative because they need a narrower spectral range and less data to be processed.
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6 It is worth noting that the NIR spectral range, the most commonly used range in an evaluation of
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8 grain quality, when applied to an evaluation of the severity of fusariosis in ground wheat provides
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10 models of considerably poorer performance, even although the NIR range provides measurements
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12 from a number of wavelengths that much exceed the number in the UV.
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9 Figure 1. Averaged over cultivars the RMSECV model errors for all spectral ranges and fractions.
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14 Figure 2. a) Exemplary (for cv. Tonacja) spectra averaged within fractions of particle size, b)
15 variance in sets of spectra recorded from series of blended samples of different fractions (for cv.
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Tonacja).

Figure 3. Absolute values of regression coefficients in best PLS models for all cultivars and fractions.

Figure 4. Averaged over cultivars mean values of regression coefficients of PLS models for particular fractions and spectral sub ranges.

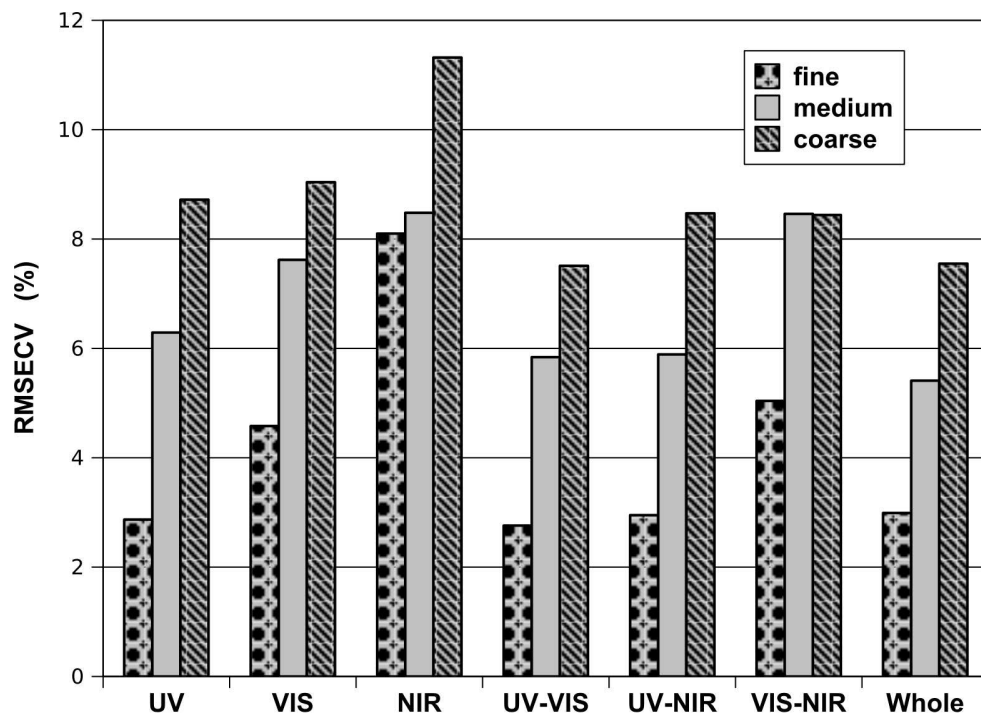
Figure 5. Plots of averaged over cultivars RMSECV of PLS models (cf. Figure 1) versus mean values of regression coefficient (cf. Figure 4) for fractions (diamonds for fine fraction, circles for medium and triangles for coarse) and spectral sub ranges (dark symbols for the UV, grey ones for VIS and empty for NIR). (Lines are just to guide the eye.)

Table 1. Calibration (RMSEC) and cross-validation (RMSECV) errors and number of latent variables of the best PLS models for all cultivars, spectral ranges and particle size fractions. Errors values are given as percentage of damaged material in blended samples.

| Cultivar | Range | Fine | | | Medium | | | Coarse | | |
|----------|---------|-------|--------|----|--------|--------|----|--------|--------|----|
| | | RMSEC | RMSECV | LV | RMSEC | RMSECV | LV | RMSEC | RMSECV | LV |
| TURNIA | UV | 1.77 | 2.52 | 4 | 2.63 | 4.34 | 5 | 3.12 | 6.42 | 7 |
| | VIS | 3.86 | 4.30 | 2 | 4.37 | 7.98 | 4 | 3.77 | 8.70 | 6 |
| | NIR | 4.95 | 8.10 | 5 | 5.45 | 7.44 | 3 | 1.09 | 3.10 | 6 |
| | UV-VIS | 1.45 | 2.43 | 5 | 2.54 | 4.41 | 5 | 1.75 | 5.07 | 8 |
| | VIS-NIR | 2.44 | 5.06 | 6 | 5.46 | 7.40 | 3 | 1.23 | 3.39 | 6 |
| | UV-NIR | 1.61 | 2.70 | 5 | 1.60 | 3.65 | 6 | 1.82 | 4.10 | 6 |
| | Whole | 1.56 | 2.76 | 5 | 1.37 | 3.55 | 6 | 0.99 | 3.80 | 6 |
| SATYNA | UV | 1.92 | 3.62 | 6 | 4.61 | 6.31 | 5 | 8.18 | 11.3 | 4 |
| | VIS | 3.04 | 3.98 | 3 | 4.52 | 5.48 | 3 | 5.02 | 6.41 | 4 |
| | NIR | 3.25 | 7.38 | 5 | 3.26 | 7.55 | 5 | 4.43 | 13.4 | 5 |
| | UV-VIS | 2.00 | 3.41 | 5 | 3.67 | 4.81 | 4 | 5.76 | 8.25 | 4 |
| | VIS-NIR | 1.87 | 3.97 | 5 | 4.06 | 6.14 | 4 | 5.96 | 7.11 | 2 |
| | UV-NIR | 1.21 | 3.72 | 8 | 3.52 | 6.29 | 6 | 8.39 | 12.4 | 4 |
| | Whole | 2.62 | 4.04 | 4 | 3.11 | 5.33 | 5 | 5.30 | 8.79 | 4 |

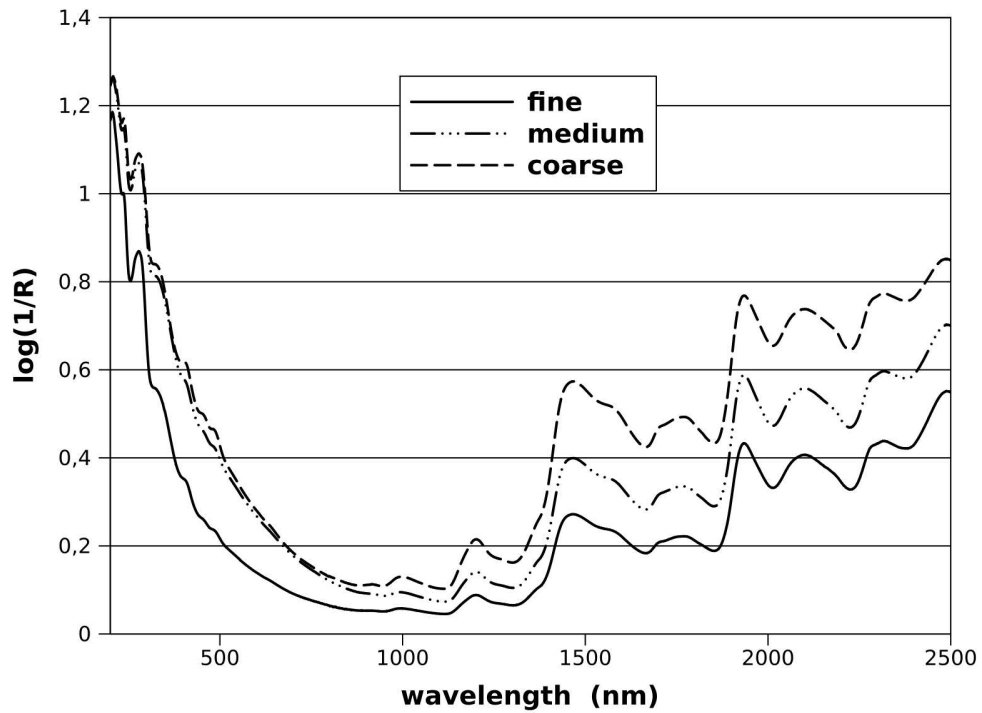
Table 1 – continued.

| | | | | | | | | | | |
|---------|---------|------|------|---|------|------|---|------|------|---|
| TONACJA | UV | 1.75 | 2.23 | 4 | 4.98 | 6.11 | 3 | 6.65 | 8.94 | 4 |
| | VIS | 5.32 | 6.09 | 2 | 5.88 | 10.6 | 7 | 6.84 | 10.4 | 5 |
| | NIR | 3.47 | 8.29 | 5 | 5.82 | 10.8 | 5 | 5.78 | 16.2 | 5 |
| | UV-VIS | 1.54 | 2.22 | 5 | 5.03 | 6.02 | 3 | 6.20 | 8.43 | 4 |
| | VIS-NIR | 2.24 | 5.56 | 6 | 4.94 | 10.1 | 6 | 5.24 | 10.8 | 5 |
| | UV-NIR | 1.38 | 2.14 | 6 | 2.82 | 4.62 | 5 | 6.01 | 10.3 | 5 |
| | Whole | 1.61 | 2.24 | 4 | 3.02 | 4.74 | 4 | 4.95 | 9.32 | 5 |
| TREND | UV | 1.72 | 3.11 | 7 | 6.30 | 8.42 | 4 | 5.24 | 8.26 | 6 |
| | VIS | 2.00 | 3.93 | 7 | 3.28 | 6.42 | 8 | 5.91 | 10.6 | 6 |
| | NIR | 2.64 | 8.63 | 6 | 3.20 | 8.15 | 6 | 5.42 | 12.6 | 5 |
| | UV-VIS | 1.66 | 2.97 | 6 | 6.21 | 8.13 | 4 | 6.39 | 8.30 | 3 |
| | VIS-NIR | 2.29 | 5.55 | 6 | 6.54 | 10.2 | 4 | 8.55 | 12.4 | 3 |
| | UV-NIR | 1.90 | 3.21 | 5 | 6.66 | 8.98 | 4 | 3.16 | 7.10 | 6 |
| | Whole | 1.59 | 2.90 | 6 | 6.50 | 8.03 | 3 | 6.03 | 8.30 | 3 |



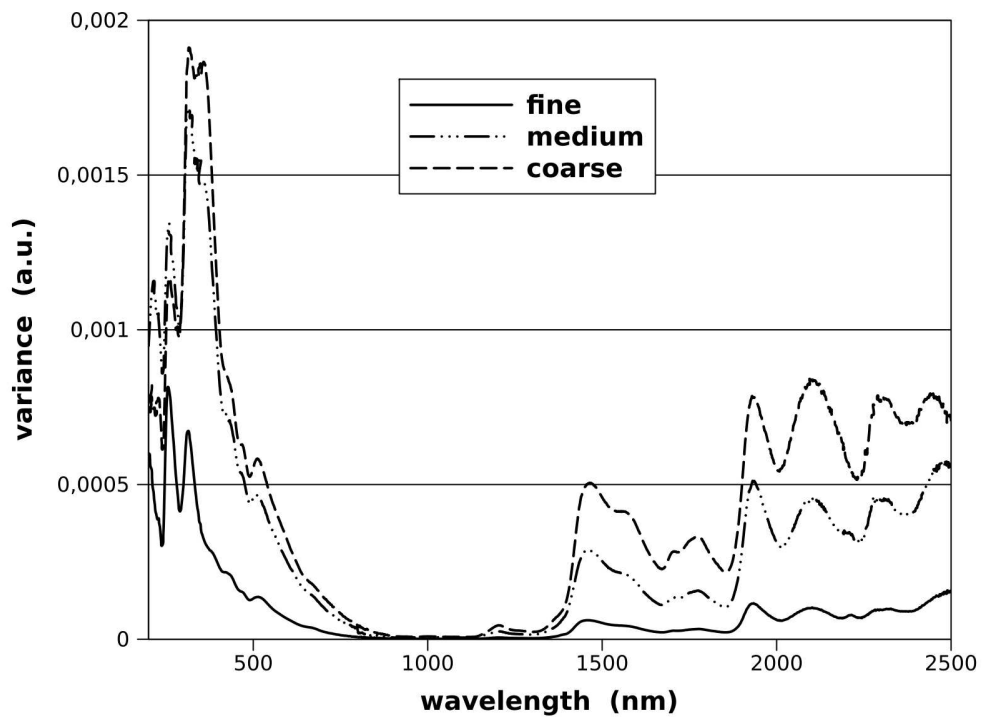
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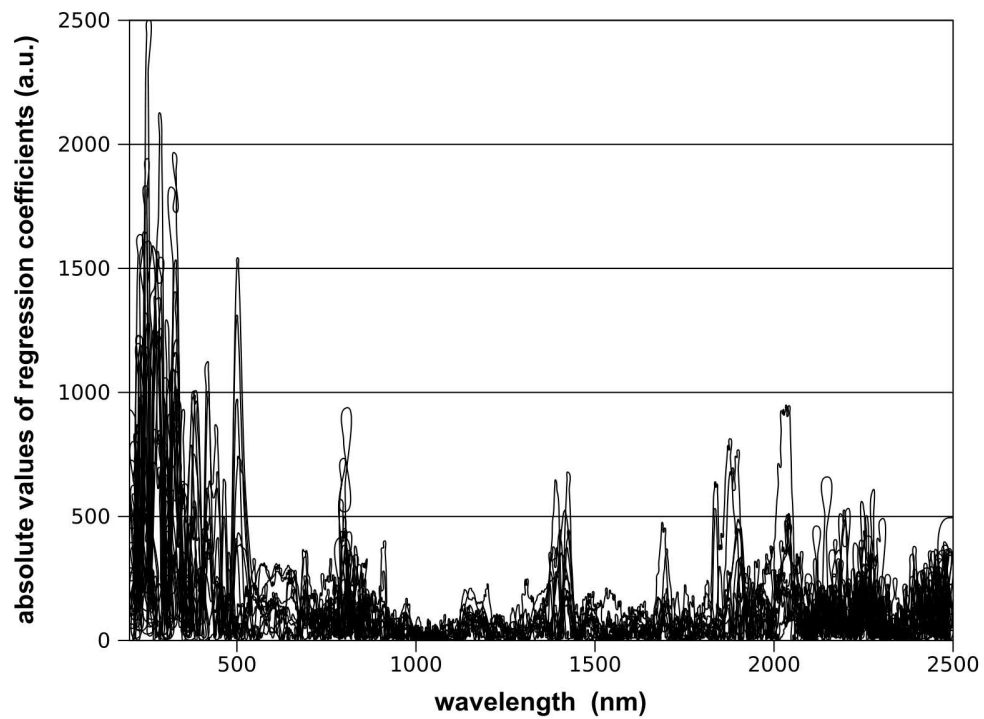
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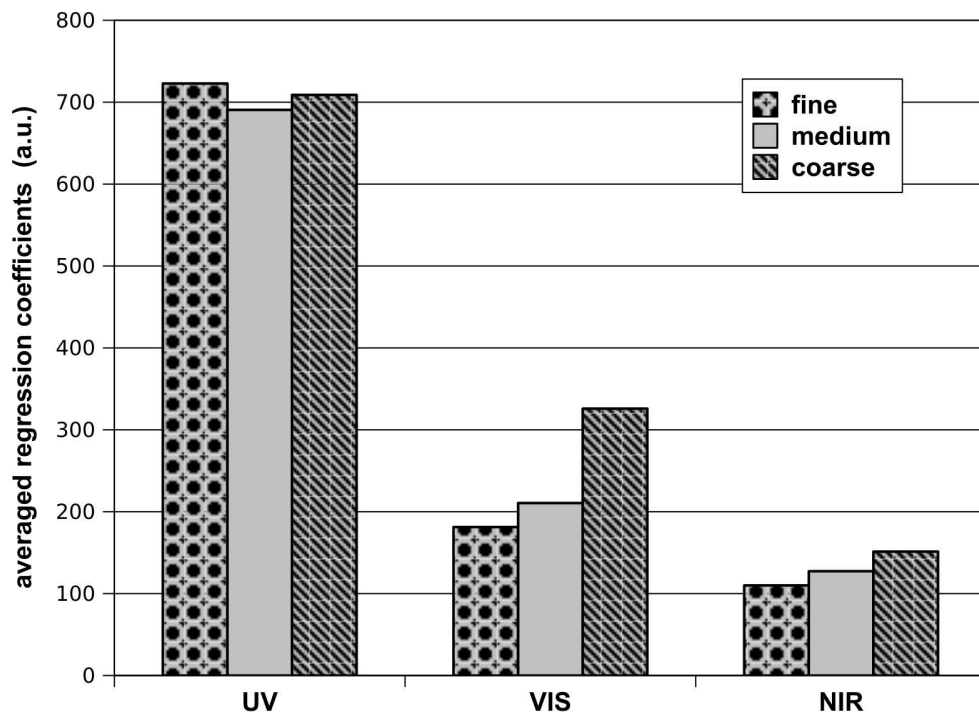
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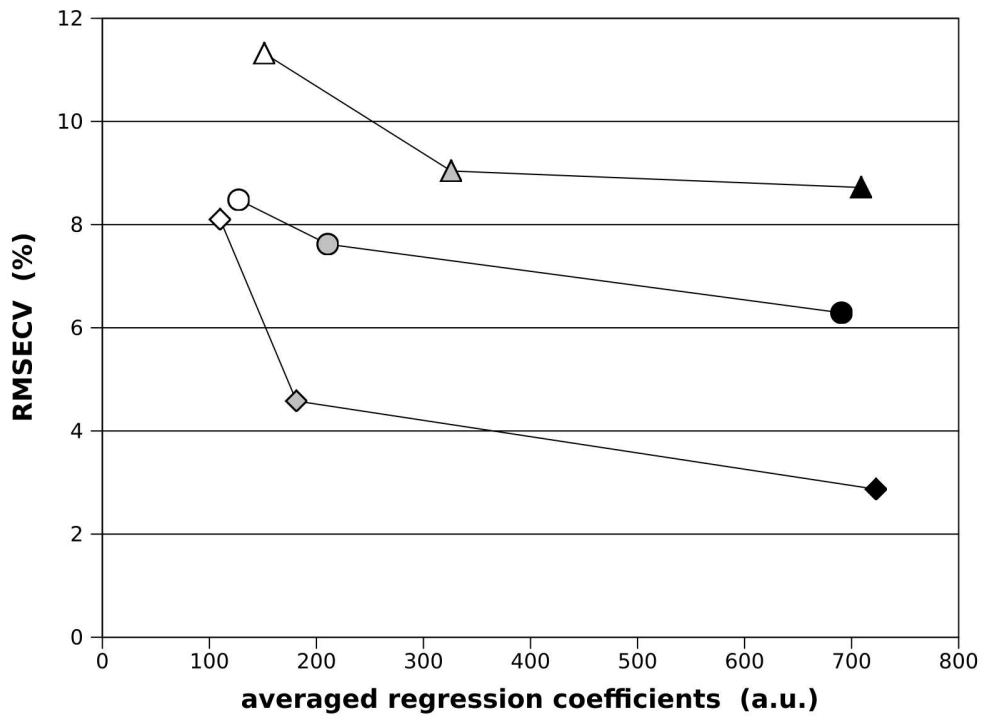
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