# Application of Near Infrared Transmission for the Determination of Ash in Wheat Flour

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## Summary

Near infrared transmission (NIT) spectroscopy was investigated for applicability as a rapid screening tool to evaluate ash content in wheat flour. Ash content in flour samples was determined also by reference method. One hundred thirteen wheat flour samples with ash content of 0.367-0.964% were collected from the Belje,d.d., PC Mlin and PPK Valpovo, and used for this study. Infratec 1241 Grain Analyzer Flour Module that comes with ready-to-use calibrations, was used to obtain transmission spectra of wheat flour. All measured ash values obtained by NIT method were within calibration limits (0.00-1.10) of the instrument. Simple linear regression analyses showed high significant correlation between the NIT and the reference method (r=0.953). The NIT results were not accurate enough for all flour types when compared to the results obtained by the reference method. Only the results for flour types 400 and 550 indicate sufficient accuracy for flour type evaluation. Considering its advantages over standard methods, including its recalibration, NIT could represent a good tool for evaluation of all flour types in the future, as it is used for protein and moisture determination in wheat grain today.

## Key words

wheat flour, ash, near infrared transmission, regression

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## Introduction

Ash content is an important constituent for wheat flour quality and it is an indicator of flour purity. The ash is not evenly distributed throughout the kernel being more concentrated in the brain (6%) than in the endosperm portion (0.4%) of the grain. The milling process, even at the optimum conditions, can not completely separate the starchy endosperm from the bran, so a small amount of fine bran particles escapes separation and passes through to the flour streams contaminating them. Ash content indicates how completely and efficiently the endosperm has been separated from the bran. This measurement is critical to the flour milling process and is used to monitor the proper functioning of various milling stages (Singh et al., 1998; Mousia et al., 2004). Ash content refers to the mineral residue of flour, and is determined by oven method burning (ICC, 1990). It is standard but time-consuming method. A quick method for the ash determination has always been required by the milling industry.

Near infrared transmission (NIT) spectroscopy is an analytical technique that is rapid, requires very little labor and does not require chemicals or create chemical waste. The technique has extensive application for the analysis of constituents of agricultural crops (Williams, 1975; Delwiche, 1995; Delwiche et al.,1998; Jurković et al., 2003; Font et al., 2005, 2006; Kayes et al., 2005), feed (Aastveit and Marum, 1993; Locher et al., 2005) and food (Scotter, 1997; Cen and He, 2006). Near infrared spectroscopy analysis, like all other spectrophotometric methods, is an indirect method and must therefore be calibrated. The calibration is conducted using referent analytical values of the calibration samples, recording their spectrum and complex statistical evaluation of all data. The calibration is essential for the method efficiency; therefore, a large number of samples, comprising a broad range of values for the parameters of interest, should be used. The instrumentation used in near infrared spectroscopy can vary in method of radiation dispersion, wavelength range, detection system and chemo metric equation used to predict constituent concentration (Delwiche et al., 1998). NIT method measures only the fraction of electromagnetic radiation that passes through the sample. Although small, that fraction is sufficient for supplying information about the sample quality (Conradi, 2001). In the last few years Flour module was an optional equipment to the Infratec 1241 Grain Analyzer which has been developed for protein, moisture and ash content in fine milled flour and semolina.

The aim of this study was to test the application for determination of ash content in wheat flour using NIT instrument Infratec 1241 Grain Analyzer Flour Module with ready-to-use calibrations.

## Material and methods

One hundred thirteen wheat flour samples from regular manufacture were collected from mills: Belje,d.d., PC Mlin and PPK Valpovo. Ash content was determined using two different methods, the NIT method and the reference method. The NIT-method of ash was applied on Foss Tecator "Infratec 1241 Grain Analyzer Flour Module". For spectrophotometric methods, it is essential to have proper calibration for reliable analysis. The instrument utilizes a calibration provided by the manufacturer. The measurement is based on the fact that the main constituents in the grain absorb electromagnetic radiation in the spectrum area from 850 nm to 1050 nm. The monochromatic light beam is transmitted via an optical fiber to the sample cell, where it passes through the sample and reaches the detector. The detector signal is then amplified and further processed by the embedded computer. The Infratec 1241 Flour Module grain analyzer is very simple to operate. The sample cup is filled with flour at the filling station. An application model is chosen from the user menu after which the sample cups drops in the hoper. The instrument makes a reference scan before the conveyor start. The conveyor then transports the sample cup until it drops into the shaft. The lower shutter holds the sample cup right in front of detector where the scan is made. Results appear within a minute on the large display. They can be printed on an optional printer or can be dumped to an external computer for further data processing (Foss Tecator, 2002; 2002a).

According to the reference method (ICC, 1990) ash was defined as the quantity of mineral matter which remains as incombustible residue of the tested substance. Burning to ashes was carried out at 900 °C, and is completed when the cool residue was white or nearly white. The ash quantity in wheat flour expressed to dry matter and the moisture content of the flour has to be determined separately (ICC, 1976).

Several statistic parameters were used to test calibration and prediction equations: Standard error of prediction (SEP) - variability in the difference between predicted values and reference values; Coefficient of determination ($R^2$) - proportion of variability in the reference data accounted for by the regression equation; Correlation coefficient (r) - degree to which predicted and reference values are correlated; Bias (D) - the mean difference between the predicted and the reference values.

Statistical analyses were conducted using statistical-graphic system "Statistica" version 6.0 (StatSoft software).

## Results and discussion

The ash content measurements are still used in the milling industry worldwide as an indicator for flour purity.

**Table1.** Ash content (%) determined by reference and NIT method, and difference between the methods (bias)

| No. flour | Ash %/DM | | Bias | No. flour | Ash %/DM | | Bias | No. flour | Ash %/DM | | Bias |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Reference method | NIT method | | | Reference method | NIT method | | | Reference method | NIT method | |
| 1 | 0.367 | 0.397 | 0.030 | 39 | 0.543 | 0.553 | 0.010 | 77 | 0.767 | 0.727 | -0.040 |
| 2 | 0.384 | 0.412 | 0.028 | 40 | 0.544 | 0.583 | 0.039 | 78 | 0.773 | 0.817 | 0.044 |
| 3 | 0.394 | 0.390 | -0.004 | 41 | 0.548 | 0.560 | 0.012 | 79 | 0.786 | 0.733 | -0.053 |
| 4 | 0.394 | 0.410 | 0.016 | 42 | 0.548 | 0.583 | 0.035 | 80 | 0.790 | 0.730 | -0.060 |
| 5 | 0.403 | 0.428 | 0.025 | 43 | 0.555 | 0.553 | -0.002 | 81 | 0.804 | 0.702 | -0.102 |
| 6 | 0.431 | 0.412 | -0.019 | 44 | 0.555 | 0.587 | 0.032 | 82 | 0.806 | 0.725 | -0.081 |
| 7 | 0.450 | 0.490 | 0.040 | 45 | 0.557 | 0.480 | -0.077 | 83 | 0.818 | 0.743 | -0.075 |
| 8 | 0.468 | 0.503 | 0.035 | 46 | 0.558 | 0.517 | -0.041 | 84 | 0.821 | 0.790 | -0.031 |
| 9 | 0.480 | 0.563 | 0.083 | 47 | 0.559 | 0.553 | -0.006 | 85 | 0.824 | 0.753 | -0.071 |
| 10 | 0.485 | 0.470 | -0.015 | 48 | 0.559 | 0.567 | 0.008 | 86 | 0.825 | 0.783 | -0.042 |
| 11 | 0.485 | 0.543 | 0.058 | 49 | 0.559 | 0.563 | 0.004 | 87 | 0.836 | 0.753 | -0.083 |
| 12 | 0.487 | 0.480 | 0.007 | 50 | 0.561 | 0.530 | -0.031 | 88 | 0.837 | 0.757 | -0.080 |
| 13 | 0.489 | 0.560 | 0.071 | 51 | 0.562 | 0.580 | 0.018 | 89 | 0.840 | 0.740 | -0.100 |
| 14 | 0.490 | 0.410 | 0.080 | 52 | 0.563 | 0.587 | 0.024 | 90 | 0.841 | 0.817 | -0.024 |
| 15 | 0.493 | 0.533 | 0.040 | 53 | 0.564 | 0.537 | -0.027 | 91 | 0.842 | 0.730 | -0.112 |
| 16 | 0.495 | 0.570 | 0.075 | 54 | 0.566 | 0.550 | -0.016 | 92 | 0.857 | 0.783 | -0.074 |
| 17 | 0.496 | 0.553 | 0.057 | 55 | 0.568 | 0.563 | -0.005 | 93 | 0.858 | 0.850 | -0.008 |
| 18 | 0.496 | 0.553 | 0.057 | 56 | 0.570 | 0.572 | 0.002 | 94 | 0.860 | 0.802 | -0.058 |
| 19 | 0.497 | 0.570 | 0.073 | 57 | 0.571 | 0.575 | 0.004 | 95 | 0.863 | 0.890 | -0.027 |
| 20 | 0.498 | 0.567 | 0.069 | 58 | 0.571 | 0.553 | -0.018 | 96 | 0.872 | 0.813 | -0.059 |
| 21 | 0.498 | 0.550 | 0.052 | 59 | 0.572 | 0.572 | -0.000 | 97 | 0.877 | 0.767 | -0.110 |
| 22 | 0.502 | 0.530 | 0.028 | 60 | 0.574 | 0.572 | -0.002 | 98 | 0.878 | 0.860 | -0.018 |
| 23 | 0.503 | 0.560 | 0.057 | 61 | 0.574 | 0.537 | -0.037 | 99 | 0.878 | 0.847 | -0.031 |
| 24 | 0.505 | 0.567 | 0.062 | 62 | 0.574 | 0.570 | -0.004 | 100 | 0.880 | 0.827 | -0.053 |
| 25 | 0.505 | 0.560 | 0.055 | 63 | 0.581 | 0.585 | 0.004 | 101 | 0.890 | 0.847 | -0.043 |
| 26 | 0.505 | 0.510 | -0.005 | 64 | 0.582 | 0.575 | -0.007 | 102 | 0.890 | 0.785 | -0.105 |
| 27 | 0.506 | 0.560 | 0.054 | 65 | 0.582 | 0.597 | -0.015 | 103 | 0.890 | 0.840 | -0.050 |
| 28 | 0.513 | 0.573 | 0.060 | 66 | 0.587 | 0.580 | -0.007 | 104 | 0.891 | 0.910 | 0.019 |
| 29 | 0.519 | 0.567 | 0.048 | 67 | 0.600 | 0.582 | -0.018 | 105 | 0.898 | 0.857 | -0.041 |
| 30 | 0.524 | 0.480 | -0.044 | 68 | 0.606 | 0.560 | -0.046 | 106 | 0.909 | 0.870 | -0.039 |
| 31 | 0.527 | 0.557 | 0.030 | 69 | 0.632 | 0.540 | -0.092 | 107 | 0.913 | 0.890 | -0.023 |
| 32 | 0.529 | 0.527 | -0.002 | 70 | 0.638 | 0.590 | -0.048 | 108 | 0.918 | 0.847 | -0.071 |
| 33 | 0.530 | 0.577 | 0.047 | 71 | 0.640 | 0.540 | -0.100 | 109 | 0.938 | 0.883 | -0.055 |
| 34 | 0.536 | 0.543 | 0.007 | 72 | 0.648 | 0.520 | -0.128 | 110 | 0.938 | 0.815 | -0.123 |
| 35 | 0.537 | 0.560 | 0.023 | 73 | 0.680 | 0.620 | -0.060 | 111 | 0.951 | 0.892 | -0.059 |
| 36 | 0.539 | 0.547 | 0.008 | 74 | 0.709 | 0.685 | -0.024 | 112 | 0.961 | 0.917 | -0.044 |
| 37 | 0.539 | 0.530 | -0.009 | 75 | 0.718 | 0.677 | -0.041 | 113 | 0.964 | 0.857 | -0.107 |
| 38 | 0.541 | 0.550 | 0.009 | 76 | 0.733 | 0.520 | -0.213 | | | | |

However, currently, there is a tendency to replace the ash content analysis with other methods (Kim and Flores, 1999; Sing et al., 1998; Ugarčić-Hardi et al., 2001) being more rapid, sensitive and inexpensive, for the determination of the bran content in the flour. An alternative, not yet fully examined method for the ash measurement is near infrared transmission.

The ash content determined in an tested wheat flour sample by the reference and the NIT methods is presented in Table 1. The overall range of total ash content determined by the reference method varied from 0.367% to 0.964% on a dry weight basis. The ready-to use ash calibrations on Infratec Grain Analyzer Flour Module covers the complete range up to 1.10% ash content. All measured ash values obtained by NIT method were within calibration limits of the instrument and ranged from 0.397% to 0.917%. Figure 1 illustrates mean ash value and standard error of reference and NIT methods. Standard errors of ash value obtained by NIT (0.013) were lower than by reference method (0.016). The data presented in Table 1 were analyzed by t-test. T-test for dependent samples indicated that significant difference in mean ash value was obtained by reference and NIT methods ($p=0.003$). Value of bias ranged from -0.213% to 0.083% (Table 1), indicating that calibration had limited predictive ability.

Linear regression for the relationship between ash content obtained by the two methods is presented in Figure 2. The $R^2$ value (0,910) indicated that 91% of ash variability in flour samples was explained by equation (Fig 2). The correlation coefficient showed high positive significant correlation between investigated methods. A standard error of prediction (SEP) was 0.054 that is 8,37% of the mean

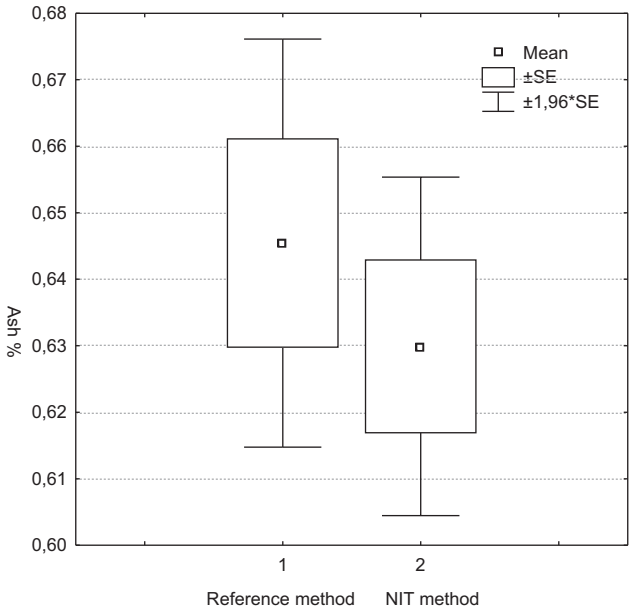


**Figure 1.**
Box- Whisker plot of ash content obtained by the reference and the NIT methods

reference value for ash content. This value was relatively high according to Williams (1987) who recommended that the SEP should not be more than 3% of the mean reference value for that analyte.

Wheat flour offered a range of special types of flour, different for their granulation, protein quantity and quality as well as mineral substance quantity. The quantity of ash helps classifying the flour in types. Assortment according to our regulation (NN 78, 2005) includes wheat flour of 400, 550, 700, 850, 1100 and 1600 type. Tested flour samples were distributed according to the regulation (Table 2). The number of samples represents types of flour, mostly utilized for end-usage. The results of the mean ash content for flour type, illustrated in Table 3 showed the difference in the ash content between reference and NIT method. Values of bias ranged from 0.017% to -0.083% indicated that tested calibration had sufficient accuracy only for flour type 400 and 550. These flour types had bias <0.020%. This difference refers to the upper limit for which duplicate measurements may differ in reference method. For other investigated flour samples, calibration was not confident. As presented in Figure 3, samples in range up to 0.600 % indicated that the Infratec instrument had a tendency to underestimate ash content. Therefore, according to ash content obtained by NIT method, samples did not inhere adequate flour types.

These results indicate that current calibration should be corrected (bias, slope and intercept corrections) on Infratec Grain Analyzer Flour module for ash content determination in wheat flour, aiming to obtain confident and accurate predicted value.

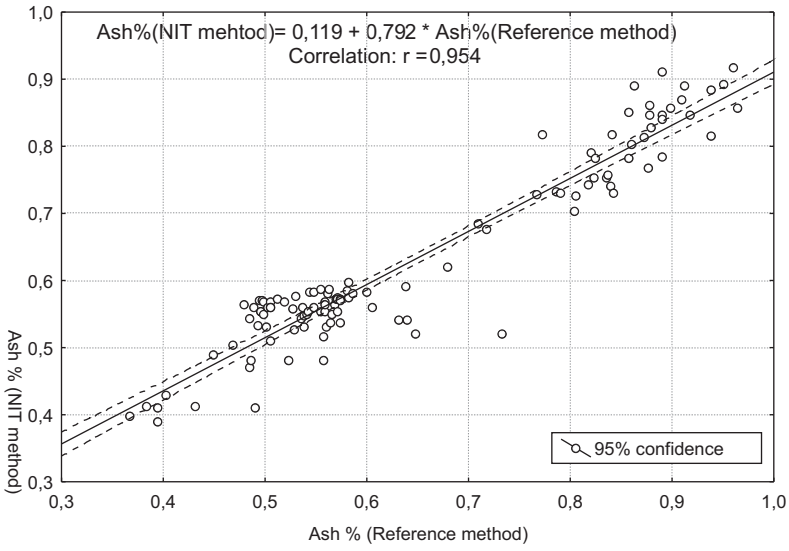


**Figure 2.**
Relationship between ash content obtained by the reference and the NIT methods

**Table 2.** Distribution of samples according to the regulation (Narodne Novine, 78/2005)

| Ash %/ DM (in samples) | Number | Wheat flour type | Ash %/ DM (regulation) |
|---|---|---|---|
| 0.367-0.450 | 7 | T-400 | to 0.45 |
| 0.451-0.600 | 60 | T-550 | 0.50 - 0.60 |
| 0.601-0.750 | 9 | T-700 | 0.65 - 0.75 |
| 0.751-0.900 | 29 | T-850 | 0.80 - 0.90 |
| 0.901-0.964 | 8 | T-1100 | 1.05 - 1.15 |

**Table 3.** Mean value of ash content for flour types and difference between the methods (Bias)

| Flour types | Ash %/DM | | Bias |
|---|---|---|---|
| | Reference method | NIT method | |
| T-400 | 0.403 | 0.420 | 0.017 |
| T-550 | 0.535 | 0.551 | 0.016 |
| T-700 | 0.667 | 0.584 | -0.083 |
| T-850 | 0.845 | 0.792 | -0.053 |
| T-1100 | 0.937 | 0.872 | -0.065 |
| All types | 0.645 | 0.630 | -0.015 |

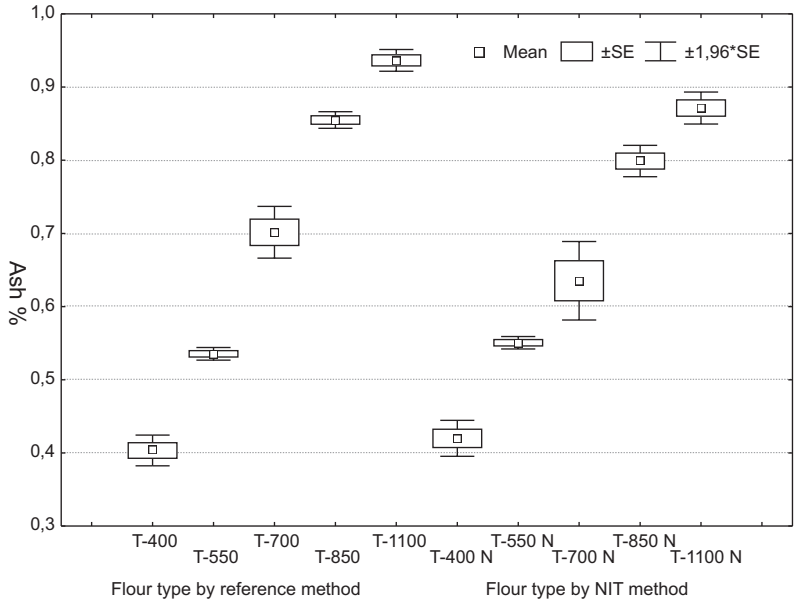


**Figure 3.**
Box-Whisker plot of ash content obtained by reference and NIT method in flour types

## Conclusion

The results of this study indicate that tested application for determination of ash content in wheat flour on NIT instrument Infratec 1241 Grain Analyzer Flour Module could be used as a nondestructive, accurate and rapid method for routine determination of ash in wheat flour types 400 and 550. Considering its advantages over standard methods, including its recalibration, NIT represents a good candidate for evaluation of all flour types in the future, as it is used for protein and moisture determination in wheat grain now days.

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