Near infrared spectroscopy—a tool for the evaluation of milling procedures

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Introduction

Small-scale test methods that offer cheaper and faster results are increasing in the analysis of cereal quality. Recently, a number of different small-scale tests have been developed for the determination of physico-chemical, functional and rheological properties of wheat or wheat dough. These use miniaturised instruments with sophisticated sample preparation/handling methods and mechanical systems (RVA, 2 g mixograph, micro-Z-arm mixer, small-scale noodle maker, micro-baking method etc.). If test methods can be successfully scaled-down, then the sample size can be reduced significantly. These small-scale methods can be used either as basic research tools or in support of technology, and can also be essential in the early selection for quality traits in breeding programmes.[1] Micro methods can be very useful in the analysis of the effects of genetically modified (GM) materials or additives, as well as in the investigation of model systems. Recently, a micro-scale lab mill was developed for small-scale sample preparation providing flour and semolina samples from small amounts of grain (5–10 g) in a reproducible and reliable way.[2,3]

The aim of this study was to compare the milling action of a macro (QC-109) mill with the action of a micro-scale lab mill (FQC-2000) produced by Metefém Co. Ltd, Hungary.[4] The milling characteristics of the instruments were analysed both by near infrared (NIR) spectroscopy and by chemical and physical methods.

Materials and methods

Forty-four samples of a single variety of Hungarian hard, red, winter wheat were conditioned to 15% moisture content for 24 hours. The conditioned samples were milled, in parallel, through a macro lab mill (Metefém QC 109, 200 g sample) and a micro lab mill (Metefém FQC-2000, 10 g sample). The grist from the macro mill was separated by sieving into three fractions based on particle size (a: > 315 μm, b: 315–215 μm, c: < 215 μm), while the grist from the micro mill was separated into the following fractions (a: > 500 μm, b: 500–315 μm, c: 315–200 μm, d: < 200 μm). The mass distribution and ash content (modified ICC 104/1 method) of all fractions were determined. NIR spectra were obtained using an NIRSystems 6500 (NIRSystems Inc., Silver Spring, MD, USA), fitted with a sample reduction accessory. Reflectance spectra covering the 1100–2500 nm wavelength region were collected using NSAS 3.30 (NIRSystems Inc., Silver Spring, MD, USA) and were processed using the PQS32 1.18 (Metrika R&D Co., Hungary) software package.

Results and discussion

Mass distribution histograms, measured with macro and micro methods, showed very different characteristics. The macro method [Figure1(a)] produced a high yield (60–70%) of flour (fraction c) with 10–15% semolina (fraction b) and approximately 25–35% bran (fraction a). The micro mill [Fig-

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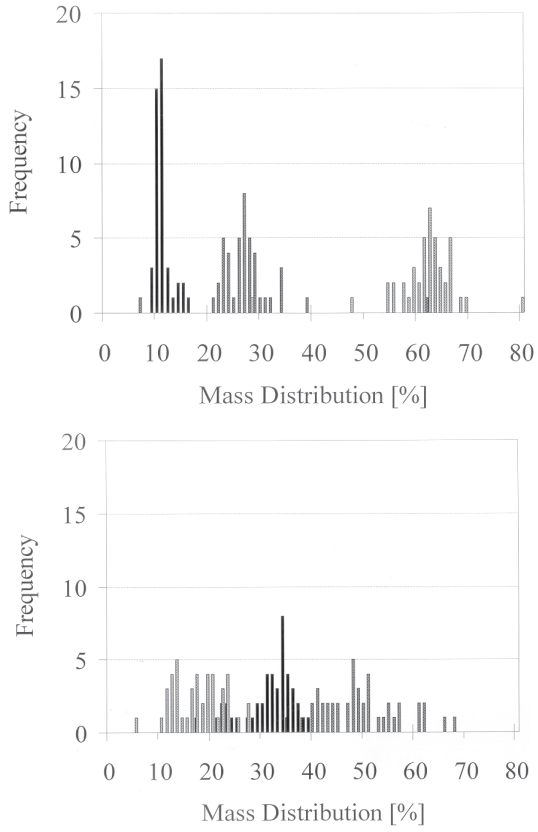

Figure 1. (a) Mass distribution of milled fractions using the macro mill and (b) mass distribution of milled fractions using the micro mill (dark grey = bran, black = semolina, light grey = flour).

ure 1(b)] showed a broader mass distribution in the fractions. The primary flour yield was only 15–25%, while the amount of semolina was between 30–40% and the amount of bran (which contains parts of bigger particles of semolina) was approximately 40–60%.

These results indicated differences in milling action. Seeds were crushed in the macro mill with a smaller milling angle (larger roll diameter) and the relative depth of grooves was bigger than with the micro mill. The shearing and crushing steps were more intensive. With the micro mill, the milling angle was relatively high, the milling surfaces were smaller and the whole milling process was relatively fast. As a consequence, the primary flour yield was lower. The efficiency of the milling process was tracked by the detection of the ash content of each fraction.

The distribution of ash content (Figure 2) indicated a high difference in ash for fractions produced by the macro and micro mills. The macro mill [Figure 2(a)] "separated" very clearly the bran fraction (ash content 3.2–5.5%) and produced a clear distinction in ash content between the semolina (0.8–1.5%) and flour (0.5–0.8%) fractions. The ash content of flour and semolina fractions produced by the micro mill [Figure 2(b)] were between 0.4–0.8% and their distributions were very sharp. The bran fraction showed a wide distribution in ash content but the absolute values (1.8–3.8%) are much lower compared with bran produced by the macro mill.

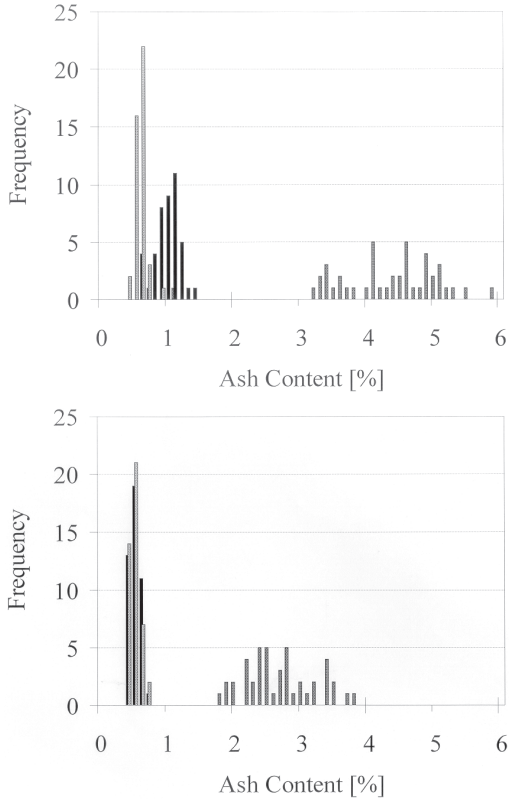

Figure 2. (a) Ash content distribution of milled fractions using the macro mill and (b) ash content distribution of milled fractions using the micro mill (dark grey = bran, black = semolina, light grey = flour).

These results clearly indicated that with the micro milling procedure the bran was not separated perfectly from the endosperm; as a result its ash content was significantly lower when compared with the macro method. The differences in the distribution of ash content were due to differences in milling action, indicating that the micro mill was significantly different in terms of efficiency of separation (rapid crushing, lower yield in flour) when compared with the macro procedure. In spite of this "crude" method, the micro mill produced good quality flour and semolina from a 5 g sample. The separation of semolina from the bran fraction can be improved and the efficiency of yield can also be increased.

The separation and chemical composition of fractions were detected by near infrared spectroscopy. NIR spectra were influenced by changes in chemical composition as well as the modification of particle size in different fractions.

Raw reflectance spectra of fractions obtained using the macro and micro mills were collected (data not shown). In the case of the micro mill, the spectra of semolina and bran fractions showed a higher variation, indicating the uncertain distribution (higher variance) of particle size.

In order to avoid the particle size effect, second derivative spectra were calculated (data not shown). In both cases (spectra from the macro and micro mills), two variable regions of the spectra

(wavelengths between 1740–1770 nm and 2290–2340 nm) were observed. In the 1740–1770 nm region, the bran fractions showed characteristic twin absorption bands relating to the high lipid content (approximately 4%) of wheat bran. In the wavelength region between 2290–2340 nm, two compositional changes were observed. At 2290 nm, the starch content of fractions can be followed (high in flour and semolina, low in bran). At 2340 nm, the cellulose and hemicellulose components can be identified (bran fractions).

The two most variable spectral regions (around 1700 and 2300 nm) were used by the polar qualification system (PQS) software[5] for making discriminant models for the quality of fractions. This method calculates "quality" points of materials and their distributions. The results of PQS analysis of fractions for macro and micro milling procedure were shown in Figures 3(a) and 3(b). The macro mill method [Figure 3(a)] provided three significantly separated fractions, indicating the compositional differences between fractions. In the case of the micro mill [Figure 3(b)], only the bran fraction was clearly separated from the other three fractions. These results matched the observations obtained for mass and ash distribution of fractions.

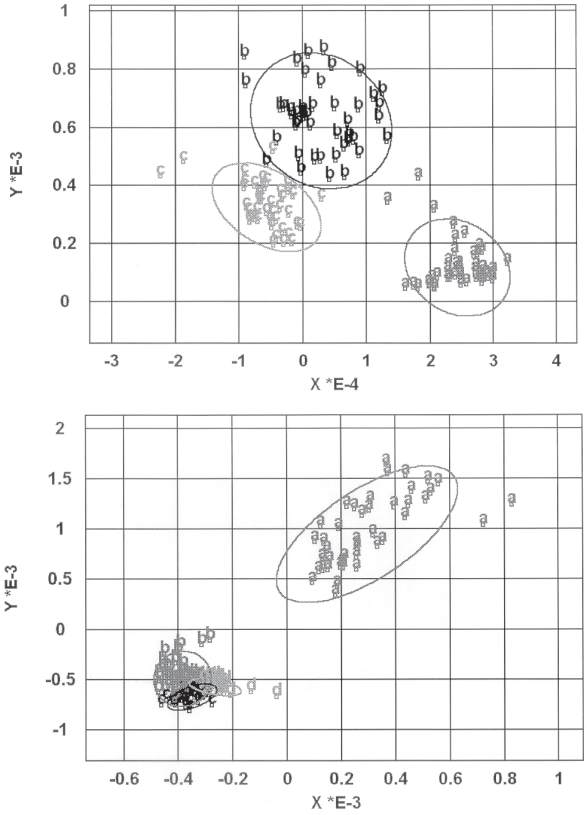

Figure 3. (a) Separation of fractions in "quality space" calculated using the PQS method (macro mill) and (b) separation of fractions in "quality space" calculated using the PQS method (micro mill) (dark grey = bran, black = semolina, light grey = flour).

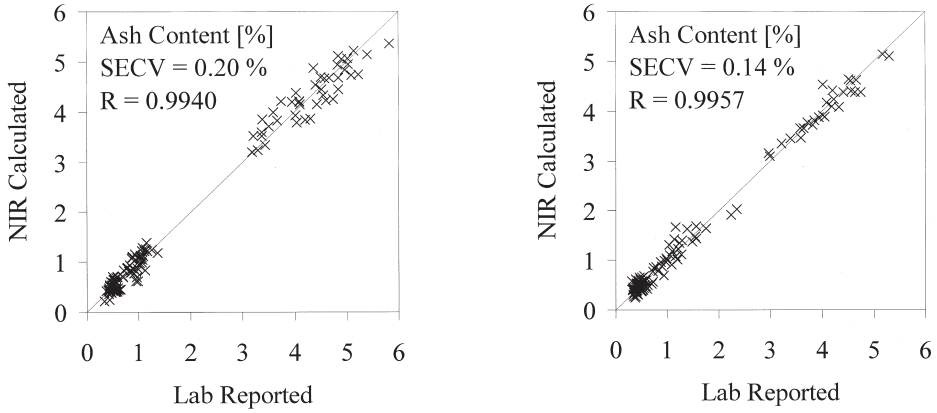

Figure 4. (a) Calibration equations developed for determination of ash content in fractions (macro mill) and (b) calibration equations developed for determination of ash content in fractions (micro mill).

Calibration equations were developed for the detection of ash content of macro and micro mill fractions from NIR spectra (Figure 4). The standard error of cross-validation (*SECV*) of equations developed for micro mill fractions showed a 40% better accuracy (*SECV* = 0.13%, *R* = 0.9957) when compared with the model for macro mill fractions. Bran samples were separated clearly in both models.

Conclusions

The micro mill, with its small sample size (5–10 g), can replace lab grinders requiring several hundred grams of sample. Compared with the macro method, the flour and semolina yields are significantly lower but the quality is good. NIR is a tool with sufficient sensitivity in the detection and evaluation of milling action for both qualitative and quantitative measurements.

Acknowledgements

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