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A NEW POSSIBILITY TO CONTROL THE MILLING PROCEDURE – USE OF NEAR INFRARED TECHNIQUE

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Abstract

Micro mill with small sample size (5–10 g) can substitute the lab grinders operating with some hundred grams of samples. The flour and semolina yields produced by micro mill procedure are significantly lower compared to macro method but their quality are good. A significant shift in mass distribution as well as in distribution of ash content of fractions was observed during micro mill procedure. The micro-scale separation resulted a higher ratio of endosperm in the bran fraction and low ash content flour. Near infrared (NIR) technique was able to indicate the differences between the fractions taken by macro or micro method. The applied NIR calibration and polar qualification system (PQS) methods were sensitive spectrum processing procedures in detection and evaluation of milling action both qualitatively and quantitatively.

Keywords: wheat milling; mass distribution; ash content; near infrared spectroscopy.

1. Introduction

The application of miniaturised, micro methods (reduced cost, time saving) is increasing in the analysis of cereal quality. Different small-scale test methods were developed recently for determination of physico-chemical, functional and rheological properties of wheat or wheat dough using miniaturised instruments with sophisticated sample preparation/handling and mechanics (RVA, 2 g mixograph, micro-Z-arm mixer, small-scale noodle maker, micro-baking method etc.). If the test methods can be scaled-down successfully the sample size can be reduced significantly. The small-scale methodologies can be used as basic research tools or as technology-supported measurements and can also be essential in the early selection for quality traits in breeding programs [1]. Micro methods can be very useful in the analysis of the effects of genetic modified (GM) materials and additives as well as in the investigation of model systems. Micro scale lab mill was developed recently for small-scale sample preparation providing flour and semolina samples from small amount of grain (5–10 g) in a reproducible and reliable way [2, 3].

The aim of present study was to compare the milling action carried out in a macro (QC–109) and a micro scale lab mill (FQC–2000) produced by Metefém Co. Ltd. Hungary [4]. The milling characteristics of instruments were analysed by near infrared spectroscopic, chemical and physical methods.

2. Materials and Methods

44 Hungarian hard, red, winter wheat samples (variety identical) were conditioned to 15% moisture content for 24 hours. The conditioned samples were milled parallel with macro lab mill (Metefém QC–109, 200 g sample) and with micro lab mill (Metefém FQC–2000, 10 g sample). The grist of macro mill and micro mill were separated with sieving into three fractions ($A > 315$ μm, $B = 215 - 315$ μm, $C < 215$ μm) and into four fractions ($a > 500$ μm, $b = 500 - 315$ μm, $c = 315 - 200$ μm, $d < 200$ μm), respectively. Mass distribution and ash content (modified ICC 104/1 method) of all fractions were tested. NIR spectra of fractions were scanned using the following spectroscopic conditions: NIRSystems 6500, 1100–2500 nm wavelength region, reflection, sample reduction accessory, NSAS 3.30 and PQS32 1.18 software packages.

3. Results and Discussion

Mass distribution histograms measured with macro and micro methods showed very different character. The macro method (*Fig. 1.a*) produced high flour yield (particle size < 215 mm) 60–70%, 10–15% semolina and approx. 25–35% bran fraction. The test of micro mill (*Fig. 1b*) showed broader mass distribution in fractions. The primary flour yield was only 15–25%, while the amount of semolina fraction was between 30–40%. The amount of bran fraction (which contains parts of bigger particles of semolina) was approx. 40–60%.

Results indicated the differences in milling action. Seeds were crashed in the macro mill with smaller milling angle (bigger roll diameter) and the relative depths of grooves were bigger than in case of micro mill. The shearing and crashing steps were intensive. In the micro mill the milling angle was relatively high, the milling surfaces were smaller compared to macro mill and the whole milling process was relatively fast, as a consequence the primary flour yield was lower. The efficiency of milling process was followed by the detection of ash content of fractions.

The ash content distribution indicated a great difference in ash content of different fractions taken by macro and micro mill. The macro mill (*Fig. 2.a*) 'separated' very clearly the bran fraction (ash content 3.2–5.5%) and produced a clear distinction in ash content in semolina (0.8–1.5%) and in flour (0.5–0.8 %) fractions. The ash contents of flour and semolina fractions produced by micro mill (*Fig. 2.b*) were between 0.4–0.8% and their distributions were very sharp. The bran fraction showed wide distribution in ash content but their absolute values (1.8–3.8%) are much lower compared to bran produced by macro mill.

Results indicated clearly that in the micro milling procedure the bran was not separated perfectly from endosperm parts, so its ash content was significantly lower compared to macro method. The observed differences in ash content distributions were due to differences in milling action indicating that the micro mill had significantly different efficiency in separation (fast crash, lower yield in flour)

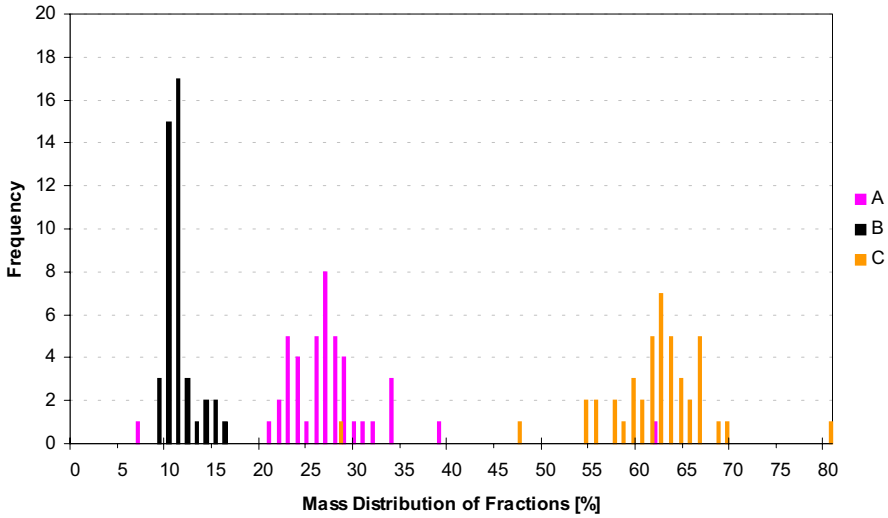

Fig. 1.a. Mass distribution of milled fractions using the macro mill (A: bran, B: semolina, C: flour)

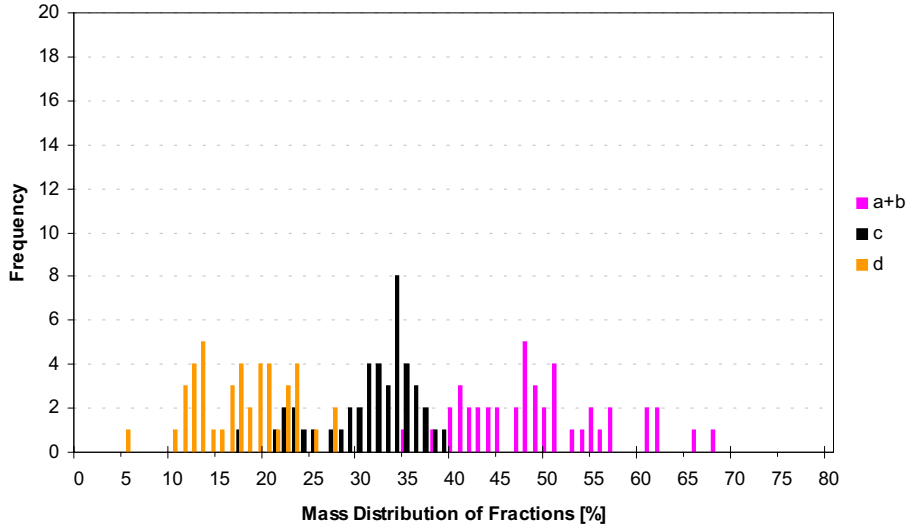

Fig. 1.b. Mass distribution of milled fractions using the micro mill (a+b: bran, c: semolina, d: flour)

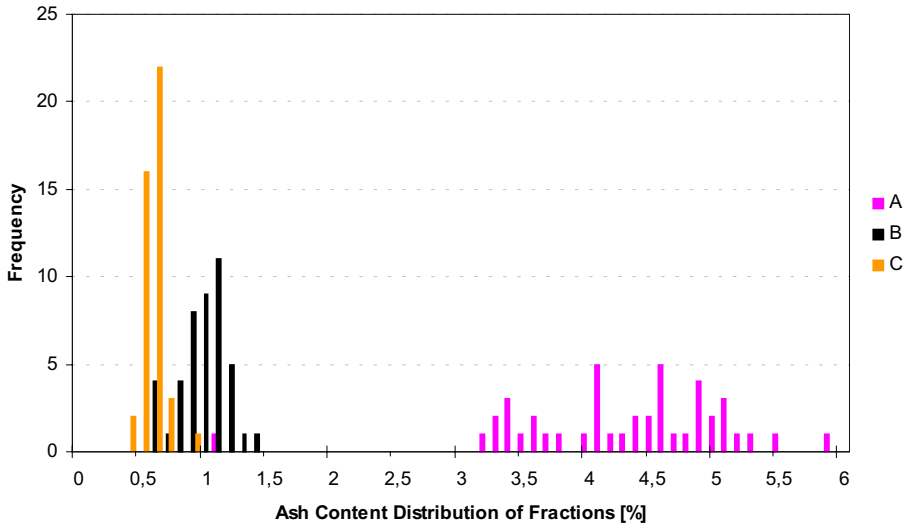

Fig. 2.a. Ash content distribution of milled fractions using the macro mill (A: bran, B: semolina, C: flour)

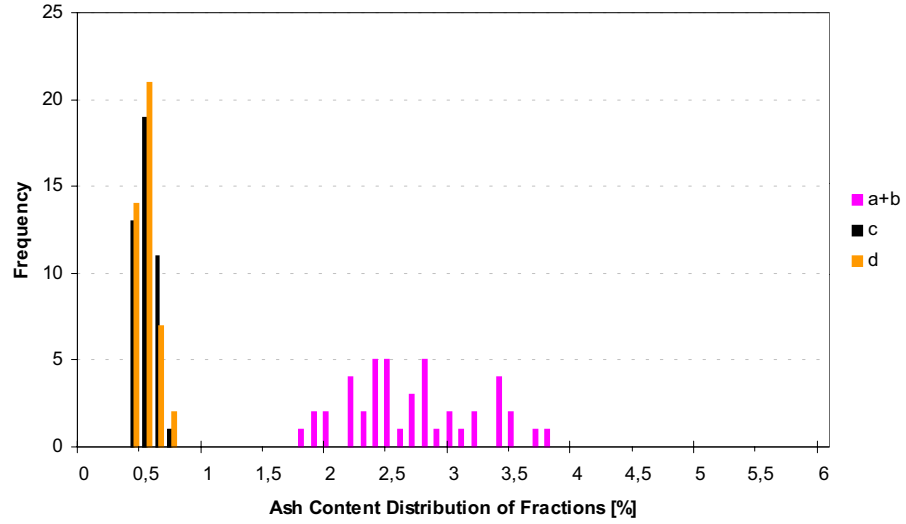

Fig. 2.b. Ash content distribution of milled fractions using the micro mill (a+b: bran, c: semolina, d: flour)

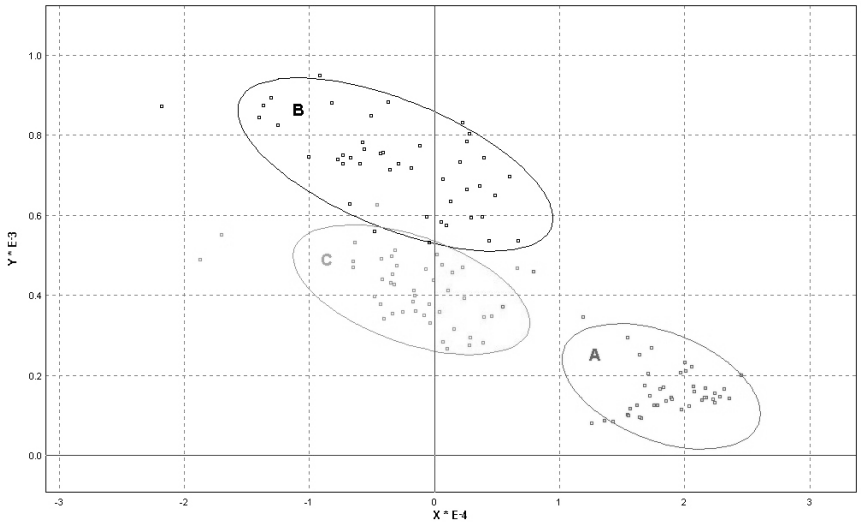

Fig. 3.a. Separation of fractions in 'quality space' calculated with PQS method (macro mill) (A: bran, B: semolina, C: flour)

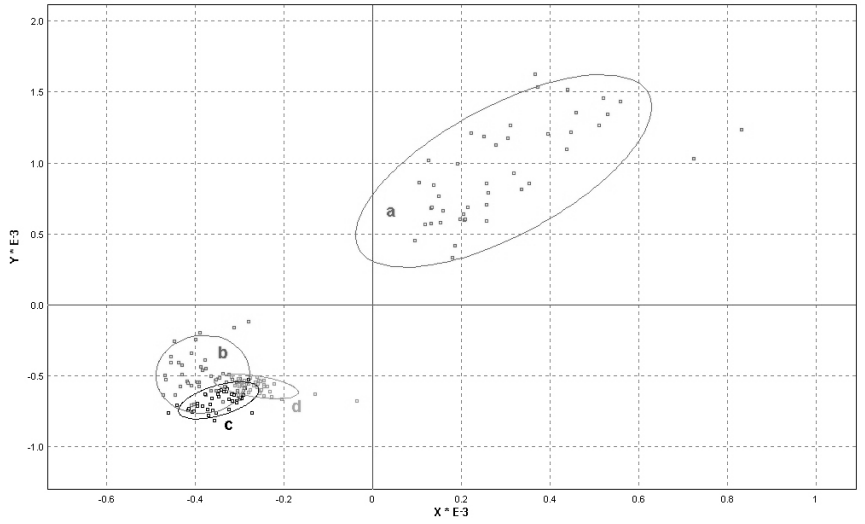

Fig. 3.b. Separation of fractions in 'quality space' calculated with PQS method (micro mill) (a+b: bran, c: semolina, d: flour)

compared to macro procedure. In spite of this 'crude' destruction of seed the micro mill produced good quality flour and semolina from 5 g sample. The separation of semolina from bran fraction can be improved and the efficiency of yield can be increased.

The separation and the determination of chemical composition of fractions were followed by near infrared spectroscopy. NIR spectra reflect the changes of chemical composition as well as the modification of particle size in fractions.

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

In order to avoid the particle size effect second derivative spectra were calculated (data not shown). In both cases (macro and micro mill) two regions of wavelength (between 1740–1770 nm and 2290–2340 nm) were observed with varying intensities of absorption. In the region 1740–1770 nm the bran fractions showed significant absorption bands the twin band of which indicated the high lipid content (approx. 4%) of wheat bran. In 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, hemicellulose components can be identified (bran fractions).

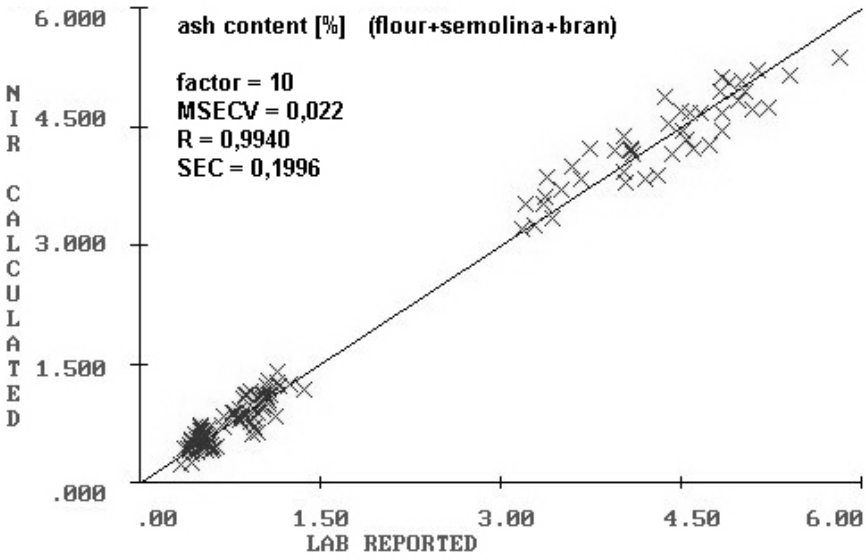

Fig. 4.a. Calibration equations developed for determination of ash content in fractions (macro mill)

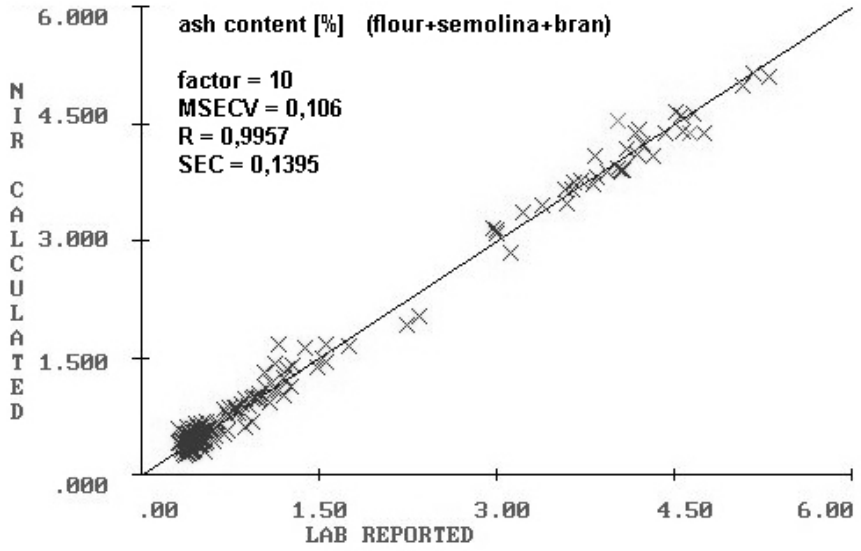

Fig. 4.b. Calibration equations developed for determination of ash content in fractions (micro mill)

The two most variable spectral regions (around 1700 and 2300 nm) were used for making distinction of the quality of fractions by polar qualification system method [5]. This method used to calculate the so-called quality points of materials and their distributions. The macro mill method (*Fig. 3.a*) provided three significantly separated fractions indicating the compositional differences between thm . In the case of micro mill (*Fig. 3.b*) only the bran fraction was clearly separated from the remaining other three ones. These results harmonized with the observations obtained for mass and ash distribution of fractions.

Calibration equations were developed for calculation of ash content of macro and micro mill fractions from NIR spectra. The standard error of cross validation (SECV) of equations developed for micro mill fractions (*Fig. 4.a*) was showing a 40% better accuracy (MSECV $= 0.106\%$, $R = 0.9957$) than the model for macro fractions (*Fig. 4.b*). Bran samples were separated clearly in both models.

4. Acknowledgement

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References

[1] GRAS, P. W. – O'BRIEN, L., *Cereal Chem.*, **69** (1992), p. 254.
[2] VARGA, J. – FODOR, D. – NÁNÁSI, J. – BÉKÉS, F. – SOUTHAN, M. – GRAS, P. – RATH, C. – SALGÓ, A. – TÖMÖSKÖZI, S., *Wheat Gluten*, RSC Publications, Cambridge (2000), p. 317.
[3] SALGÓ, A. – VARGA, J. – TÖMÖSKÖZI, S. – GRAS, P. – RATH, C. – BÉKÉS, F. – NÁNÁSI, J. – FODOR, D., *Cereals 2000*, Cereal Chemistry Division, Royal Australian Chemical Institute (2001), p. 35.
[4] FODOR, D. – VARGA, J – TÖMÖSKÖZI, S. – SALGÓ, A. – NÁNÁSI, J. – VERES, GY., *Hungarian Patent Application* (1999).
[5] KAFFKA, K. J. – GYARMATI, L. S., *J. Near Infrared Spectrosc.*, **6** (1998), p. A191.