Welcome

Welcome to the 10th issue of Perten Science World.

When looking at the need for controlling quality, whether it is purchase of the raw material, process control in transformation or production, or when specifying the quality and characteristics of the final product, a wide range of methods and parameters may be considered. Often the control system required to fully determine the quality is a combination of testing functional properties and compositional analysis.

At Perten we regard both these areas important and we have the focus to develop and improve both.

Our interaction with different types of industries and applications sometimes gives rise to new possibilities in existing instruments. In this issue of Perten Science World you will find an example of this in the article describing how our dough rheology instrument doughLAB is used to test the production of cheese.

I hope you find this 10th issue of Perten Science World interesting, useful, and stimulating!

Martin Hallin,
Product Manager
Using the DA 7300 Digital Camera for Color Detection in Agri-Industry Processing

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Introduction
The DA 7300 in-line, Figure 1, incorporates a digital camera for real-time image analysis for speck detection, color measurements, and video streaming showing the product.

For optimal image analysis performance, the instrument is equipped with a unique, dedicated camera LED illumination system, Figure 3.

Continuous measurement stability is achieved through the use of an internal automatic reference used as white standard.

The color temperature of the illumination and camera gain per color channel is precisely controlled and ensures the measurement is stable over time and varying measurement conditions. The camera system is recording R (Red), G (Green), and B (Blue) color for all pixels, in the active color detection area. The color detection area is selectable, and is marked with a blue rectangle in the middle of the captured image field. The sensitivity of the pixels used for color measurements is factory normalized.

The DA 7300 in-line camera R, G, B values can be calibrated to present values in other color spaces and standards, e.g. L*, a*, b* color space, Hunter Lab color space or ICUMSA 420 RBU color. The instrument can be adjusted to match a specific reference spectrophotometer.
CIELAB color space

The CIELAB color space expresses colors in $L^*$, $a^*$, $b^*$ values in a way that approximates human vision, Figure 4. $L^*$, $a^*$, and $b^*$ values are mathematically defined from Tristimulus values denoted as $Y/Y_n$, $X/X_n$, $Z/Z_n$ that relates to how an average person sees color across the visible spectrum at a defined illumination and viewing angle. The $L^*$ coordinate in the CIELAB definition represents the lightness of the product, $a^*$ represents green-to-red and $b^*$ represents blue-to-yellow scale.

The DA 7300 in-line camera R, G, B measurements are calibrated to $Y/Y_n$, $X/X_n$, $Z/Z_n$ values. Then, the $Y/Y_n$, $X/X_n$, $Z/Z_n$ are converted through the defined equations to $L^*$, $a^*$, $b^*$, Figure 5.

For optimal color measurement performance, it is recommended that the calibrations are developed using similar products as those being measured and that the color range is covered.

$$L^* = 116(Y/Y_n)^{1/3} - 16$$
$$a^* = 500[(X/X_n)^{1/3} - (Y/Y_n)^{1/3}]$$
$$b^* = 200[(Y/Y_n)^{1/3} - (Z/Z_n)^{1/3}]$$

**Figure 4:** CIELAB color space and examples of $L^*a^*$ and $b^*$ values on three different semolina samples

**Figure 5:** Tristimulus values and how they relate to $L^*$, $a^*$ and $b^*$ values
In-process application examples

The color measurements can be used in combination with NIR measurements of chemical composition as valuable information in ensuring product quality and production within specifications, Figure 6.

Continuously displaying the color of the product to operators, and if they are within specifications, will allow for reduction of costly customer rejects.

The live camera display of the actual product stream gives additional visible information, Figure 7.

Application area examples are process steps in: durum semolina and flour milling, raw sugar processing and refining, dairy production, feed milling, oil seed processing, or corn wet and dry milling.

![Figure 6: DA 7300 Process Plus web operator interface with measurements and DA 7300 in-line camera image](image)

![Figure 7: DA 7300 captured images and live video](image)
The DA 7300 camera can be calibrated to match a specific reference spectrophotometer. Measurements can be on products ranging in color from dark to white. Figure 8 shows the colors displayed through \( L^*, a^* \) and \( b^* \) by the DA 7300 plotted in comparison to the reference spectrometer displayed color, making the high similarity visible.

**Figure 8:** The color measurement of Agri-products from the DA 7300 compared to the reference spectrophotometer
Producing Imitation Mozzarella Cheese Using the Perten doughLAB

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Introduction
Imitation mozzarella cheese (IMC) is a lower cost alternative for natural mozzarella cheese typically produced using vegetable oil, rennet casein, starch, emulsifying salts, and other dairy and non-dairy ingredients (Bachmann, 2001, Kapoor & Metzger, 2008). The functional properties of the IMC are influenced by the ingredients, equipment, and the manufacturing process (Hennelly, et al., 2005, Noronha, et al., 2008). Extensive research has been conducted to improve the functional and flavor properties of IMC. In most of these studies, IMC was manufactured in a pilot scale production twin-screw cooker with a batch size of 4.5 kg. However, in situations such as when experimental ingredients are being evaluated, a smaller batch size is desired. The doughLAB (Perten Instruments, Hägersten, Sweden) is a commercial heating and blending system used for dough rheology applications. Compared to a typical pilot scale twin-screw cooker, the doughLAB has a batch size of 600 g which is ideal for a lab scale evaluation of experimental ingredients.

Objective
The objective of this study was to compare IMC manufactured with the Perten doughLAB (PDL) to IMC made with a pilot scale Blentech twin-screw (BTS) cooker (cheese Therm cooker – Blentech Corporation, Santa Rosa, CA). The PDL utilizes a 600 g mixing bowl supported with a conventional Z-arm mixing. It also includes an automated software system to control the bowl temperature, mixing speed and to measure the torque value during the manufacturing process. This torque value can be used to monitor the formation of an emulsion during the manufacturing process. The BTS utilizes a batch size of 4.5 kg with an auger mixing system. The BTS cooker does not utilize any software to record the temperature profile and torque during the manufacturing process.

Materials and Methods
IMC Manufacture
Two different IMC formulations that had similar composition (16% protein, 24% fat and 50% final moisture) but utilized different ingredients were manufactured in triplicate in the BTS and the PDL. In the BTS cooker, the emulsifying salts and water were initially added to the cooker and then heated to 80°C. The fat blend was heated separately to 90°C in a microwave and then the remaining dry ingredients were added to the fat blend. Subsequently, the blend of fat and dry ingredients was transferred to the BTS cooker that contained the water and emulsifying salts and heated to 80°C and held for 5 minutes. The mixing speed was maintained at 140 rpm in the BTS during the entire manufacturing process. In the PDL the ingredient addition, blending procedure, and cooking temperature was the same as the BTS. However, the mixing speed in the PDL was 115 rpm instead of 140 rpm in the BTS. A lower mixing speed was used in the PDL based on the results of a preliminary study that indicated the PDL provided more intense mixing compared to a BTS. Additionally the cooking time in the PDL was determined based on the optimal torque value identified during initial trials of each formulation. A typical temperature, mixing speed, and torque profile obtained during the manufacture process is shown below in Figure 1 and a batch of IMC during the cooking process is shown in Figure 2.
Figure 1: The torque profile of cheese made in the PDL.

Figure 2: The different stages of the cheese manufacturing process: (a) at 0 min., (b) at 1 min., (c) at 2 min., and (d) at the end of cooking process.
Compositional analysis of imitation cheese
IMC samples were analyzed for fat using the modified Mojonner, method 989.05 (AOAC, 2000a), moisture content using a forced-draft oven (model OV-490A-2; Blue M, Blue Island, IL, method: 990.20 (AOAC, 2000b), and protein using a Kjeldahl analysis method 991.20 (AOAC, 2000c).

Functional analysis
Hardness of the cheese samples was determined using Texture Profile Analysis (TPA). In order to perform this test the imitation cheese samples were cut into cylinders with a 20 mm diameter and 20 mm height using a cork borer and cheese slicer. A double bite TPA compression test profile using a cylindrical (TA-4) probe was performed using a TA.XT2 Texture Analyzer (Texture Technologies Corp., Scarsdale, NY/Stable Microsystems, Godalming, UK). Three replicates of each sample were analyzed with a test speed of 2 mm/sec and the peak force at 50% compression was recorded as hardness.

Modified melt and stretch test
Cheese samples with a 38 mm diameter and 8 mm height samples were cut using a cork borer and cheese slicer. The samples were placed in a 15 x 100 mm glass petri dish, covered, and equilibrated to room temperature. After reaching room temperature, the petri dish was transferred to a forced draft oven at 232°C and baked in the center of the oven for 5 minutes. After removal from the oven, the sample was cooled for 30 seconds and was stretched by wedging a fork into the melted cheese disc and slowly lifting it upwards. The length of cheese stretch up to the breaking point (inches) was measured with a ruler and reported as cheese stretch. Melt appearance of the cheese was also observed for any visible browning and for visible free oil or water. The cheese melt was determined by measuring the diameter of the melted cheese spread.

Table 1: Melt and stretch results of two formulations manufactured in the BTS and PDL

<table>
<thead>
<tr>
<th>Property</th>
<th>Formula 1</th>
<th></th>
<th>Formula 2</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>BTS</td>
<td>PDL</td>
<td>BTS</td>
<td>PDL</td>
</tr>
<tr>
<td>Melt (mm)</td>
<td>17</td>
<td>15</td>
<td>24</td>
<td>24</td>
</tr>
<tr>
<td>Stretch (mm)</td>
<td>6</td>
<td>15</td>
<td>7</td>
<td>15</td>
</tr>
</tbody>
</table>

Statistical analysis
Method comparison statistics (mean comparison, graphical representation, repeatability) in Microsoft Excel were used to evaluate the agreement between the two methods. Samples were analyzed in triplicate and mean values were presented in the results.

Results and Discussion

Cooking time
The cooking time in the BTS was constant for all formulations (5 min.). However, the cooking time in the PDL was determined by identifying the optimal torque value for each formulation by conducting a preliminary trial. In the preliminary trial each formulation was cooked until a peak was observed in the torque profile. Subsequently, this peak value was used to determine the cook time used for each formulation and the cooking process was immediately stopped when the peak torque value was reached. The mean cooking time obtained in the PDL was 3.5 minutes for formula 1, and 2.0 minutes for formula 2. The shorter cooking time observed with formula 2 is believed to have been caused by differences in the protein source used for each formulation.

The melt and stretch results are shown below in Table 1. Similar melt results were found in the BTS and PDL for both formula 1 and formula 2. Additionally, the melt was higher in formula 2 than formula 1 in the PDL and BTS. The stretch results demonstrate that the IMC produced in the PDL had higher stretch compared to the IMC produced in the BTS for both formulations. The higher stretch with the PDL may be a result of the shorter cooking time used in the PDL. These results indicate that using the optimal cooking time may increase the stretch characteristics of IMC.
The TPA-hardness for each formulation is shown in Figure 3. The TPA-hardness was substantially different between the formulas. In formula 1 the hardness of the IMC produced in the BTS was similar to the PDL, whereas in formula 2 the PDL had a slightly lower TPA-hardness than the BTS. These results indicate that IMC with similar hardness can be produced with the PDL compared to the BTS.

Conclusions

The results of this study indicate that the Perten doughLAB can be used to produce 600 g batches of IMC that have functionality similar to IMC produced in a pilot scale twin-screw cooker. The PDL can also be used to identify the optimal cooking time by monitoring torque during the cooking process. In addition, the integrated software in the PDL provides graphs of torque, temperature profile, and mixing speed. This data can be used to monitor the cooking process and may be related to the functional characteristics of IMC.

References

AOAC. (2000a). Official Methods of Analysis. Fat in Milk, 989.05.
Predicting Baking Performance through Evaluation of Short-crust Dough

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Abstract
The three major components of short-crust dough are flour, sugar, and fat. Since high fat contents have been shown to have a major effect on the development of the gluten network, studying how these ingredients could affect the texture of the dough and the baked product became interesting. In addition, there are no existing methods on short-crust dough that allow predicting the characteristics of the baked product based on those of the dough. Therefore, the task was to study whether developing such a method is possible. In this paper, the amount of ingredients was varied and textural analyses were run on dough and baked samples. The results were analyzed using chemometrics and statistical tools and different graphs were plotted to visualize the relations between the variables and parameters. The analyses showed the margarine and egg amounts have a significant effect on the texture of the dough in terms of resilience, gumminess, and hardness. In addition, the hardness of the baked product has been shown to be positively correlated with the same parameters, which in turn can be controlled by the addition of fat and/or eggs according to requirements. Thus, prediction of baking performance based on dough characteristics is shown to be possible.

Keywords: short crust dough, rheology, textural characteristics, texture, shortening, margarine, texture analyzer, baking

Introduction
In daily life, at least at home, no more than basic knowledge is required to be able to produce bread and other bakery products. However, in order to improve such production, especially in industrial processing and applications, proper scientific understanding of the components and their interactions is a priority. Taking this to another level, it is valuable to be able to predict baking performance and the characteristics of the finished product based on those of the dough. This will help improve production in terms of quality and quantity, since time and materials will be saved. While methods for predicting bread-baking performance through dough evaluation already exist, no such methods have been developed for short crust dough. Therefore AAK suggested a study on the possibility of developing such a method.

Flour, sugar, and fat are the three major components of short crust cookies (Zucco, et al., 2011). Fat could be added as butter, shortening, or margarine. The high content of fat increases incorporation of air in the dough especially when subjected to a creaming stage. In addition, fat acts as a lubricant and competes with the aqueous phase and limits the gluten formation (Maache-Rezzoug, et al., 1998, Slade & Levine, 1994, Wade, 1990). However, during baking fat melts and together with sugar they increase the mobility of the dough and hence result in larger dough spread (Pareyta, et al., 2009). The dough components thus influence the dough rheology, dough making and handling, and baking quality that influence the rheology characteristics (Pedersen, et al., 2004). According to Bourne (1990) methods such as compression, bending-snapping, and puncture principle have been used to study rheological and textural properties of dough and baked products.

Materials and Methods

Materials
Wheat flour produced by Nord Mills (moisture content less than 15.5%; Kärnvetemjöl (ARTNR 140233)) granulated sugar, egg powder, and cold drinkable tap water. Four types of fat based on S100 were used in the experiments especially produced at AAK for this study: (1) shortening, (2) shortening with 2% emulsifier, (3) margarine, and (4) margarine with 2% emulsifier.
**Dough preparation**

*Mixing:* sugar, fat, and egg powder were mixed at low speed (60 rpm) in a mixer (Hobart N50 5-Quart Mixer) for 30 sec., scraped down, water was added, ingredients creamed for additional 30 sec., and the content was scraped down. The speed was increased to 124 rpm and creaming was allowed for 4 min. The cream was scraped down, flour was added, and the content was mixed at 60 rpm for 90 sec., the content was then scraped down, speed was increased to 60 rpm, and the content was mixed for 3 min. and 30 sec. Note that when margarine was used instead of shortening, in the first step the content was mixed for 60 sec. instead of 30, and no water was added since margarine consists of 20% water.

*Sheeting:* two rulers of the same height were placed on each side of a baking paper, and the sheeting was performed by allowing the edges of a rolling pin to roll on the rulers so that the dough would obtain a standardized height of 6 mm.

*Sampling:* a mold with a diameter of 42 mm was used to cut the samples. The excess dough surrounding the samples was carefully removed by lifting or pushing away. Using a knife, the baking paper was cut around the sample and a thin pie lifter was used to lift the samples with the baking paper and transfer onto a small petri dish. Thus sample deformation would be avoided and a standardized method would be obtained. The samples were then divided into three groups (baking, compression, and holding) composing five samples in each group.

*Baking:* the samples in the baking group were weighed and using a caliper the diameter was measured. A baking tray covered with baking paper was prepared and samples were lifted and tilted over (upside-down). The small piece of baking paper was gently removed to avoid deformation. The samples were baking for 20 min. at 180°C, and stored for at least two weeks before analyses.

**Color and textural measurements**

*Color measurements:* the color was measured using a color measuring instrument (CR-400 Chroma Meter, Konica Minolta).

*Textural measurements:* to determine the textural characteristics of the samples TexVol Texture Analyzer TVT-300XP was used. Three different methods were used, method (1) and (2) were used on dough and (3) on the baked product.

1. **Double compression:** double compression cycle with compression 4 mm over plate and 50 sec. pause between cycles. Probe P-Cy75A was used and the pre-test, test, and post-test speed was set to 0.2 mm/s.
2. **Hold until time:** a single cycle with compression 4 mm over plate and holding time 62 sec. Probe P-Cy75A was used and the pre-test, test, and post-test speed was set to 1.0 mm/s.
3. **Three point bend:** a single cycle that breaks the sample with a compression of 10%. Probe P-BP70A and rig R-TPBR were attached and the pre-test, test, and post-test speed was set to 1.0 mm/s.

**Statistics and chemometrics**

*Experimental design:* in order to detect the effect of the different ingredients as well as the interactions between them, a central composite design known as CCD was used. It consists of a complete factorial design combined with replicates in the centrum (Brereton, 2003). The variables were varied at 3 levels (-1 0 +1), which allow neglecting triplicates (Miller & Miller, 2010).

*Statistical analysis:* to analyze data obtained from the different measurements, different tools were used:

- **PCA:** consists of multivariate projections of the observations onto a two-dimensional plane. This enables visualization of the structure of the investigated data set and reveals the relationships between variables and observations as well as the relationships within the variables themselves (Brereton, 2003, Eriksson, et al., 2001).
- **PLS2:** a projection method as PCA that handles complex models and strongly correlated responses or parameters (Brereton, 2003, Eriksson, et al., 2001). This method was used to observe relations between variables and parameters as well as within parameters themselves.
- **ANOVA:** a tool used to study the significance of the differences between two or more vectors.
Results and Discussion

Effect of ingredients on characteristics of dough and baked product

PLS2 was used to analyze the effect of ingredients on the parameters. Figure 1 shows that the amount of margarine is negatively related to resilience (DCResilience), gumminess (DCGumminess), and hardness (DCForceA) obtained by double compression on dough in addition to the hardness of the final product (TPBForceA). This is due to the angle between their vectors being much larger than 90° and the considerable length of their vectors which are almost the same, giving a high scalar product (Brereton, 2003). This means that higher amounts of margarine result in decreasing the values of these parameters. Note that resilience is how much energy an object can absorb without causing permanent deformations (Campbell, 2008). Margarine seems also to be positively correlated with the adhesiveness, springiness, and stringiness of the dough, which means that higher margarine values result in higher elasticity and stickiness of the dough. This observation has also been stated by Lai and Lin (2006).

On the other hand, Figure 1 shows that shortening has no relation to DCGumminness and DCForceA as the angle between their vectors is 90°, yet, has a negative relation to DCResilience. What also can be seen in Figure 1 is that the amount of eggs is positively related to DCResilience, DCGumminness, DCForceA, and TPBForceA, but negatively correlated with DCAhhesiveness, DCSpringiness, and DCStringiness, which clearly is the opposite of margarine. Regarding brightness of the cookies (TPBBrightness), both eggs and margarine showed to be strongly related, however in an opposite manner, which is very logical since a high egg amount gives darker cookies due to the protein content in eggs reducing sugars which enhances Maillard reactions (Maillard, 1912).

These relations were studied further by calculating the standard error of estimating the different parameters by the ingredient values, and calculating the confidence interval for parameter estimation with 95% significance level. The results showed that shortening and margarine values could be used to predict all parameters since the zero was not included in the calculated confidence interval. Regarding egg powder, results showed that almost all parameters could be estimated except DCCohesiveness and DCChewiness. By this, it could be concluded that all the observations are true on 95% significance level.

Figure 1: Relation between input and output parameters

Relations between dough characteristics and those of the baked products

Figure 1 revealed that there seems to be a positive relation between TPBForceA, DCForceA, DCGumminness, DCResilience, DCChewiness, but negative relations to DCAhhesiveness, DCSpringiness, and DCStringiness. Therefore we used PCA to see if this observation could be supported. Initially, a cross validation was made (Figures 2 and 3) to analyze how many principal components should be considered to obtain a proper explanation of the data. The scree-plot (Figure 2) shows that two components are needed to describe the data, however, in the PRESS-plot (Figure 3) the slope increases between components 1 and 2 and then decreases to three components. Consequently, it was concluded that three components would be the best choice to describe the data, however since it is not possible to visualize the relation between the parameters in a 3D plot, the different principal components were plotted against each other in Figures 4 and 5.
ARTICLES

Figure 2: Scree-plot illustrating the number of components needed to describe the data

Figure 3: PRESS-plot illustrating the number of components needed to describe the data

Figure 4: Loading plot PC2 against PC1 with 59% degree of determination

When plotting PC2 against PC1 interesting results were obtained (Figure 4). This graphs shows that there are strong positive correlations between TPBForceA to DCRessilience, DCGumminess, and DCForceA, as well as negative relations to DCAdhesiveness, DCSpringiness, and DCSpringiness. However, since the explanation degree is 60%, PC3 against PC1 was considered to see if this pattern is still obvious.

In Figure 5, the relations above become clearer. Looking at the right side of the graph, the correlated parameters are ordered on a straight line over TPBForceA. In addition, the correlations seen in Figures 1 and 4 could still be seen in Figure 5 which is a good sign.

Conclusions

The amount of margarine added to short-crust dough has a negative effect on the hardness of the finished product, the gumminess, and the resilience of the dough. Meaning that increasing the amount of margarine would increase the ability of the cookie to deform upon application of force. However, this could be overcome by the addition of egg powder in appropriate amounts, since the egg powder has been shown to be negatively correlated with the parameters above. In addition, the hardness of the baked product is positively linked to the resilience, gumminess, and hardness of the dough. Since these results were obtained by three methods (PLS2, significance test, and PCA) and observed in three plots (Figures 1, 4, and 5), it is possible to say that measuring...
the resilience, gumminess, or hardness of the dough would allow the prediction of the characteristics of the baked cookie. Conclusively, in order to control the dough properties by balancing the amount of fat and eggs to the needs, it is possible to control the hardness of the baked product.

Acknowledgments
This work is done within the framework of a Masters thesis in Food Technology at the University of Lund, Sweden. Special thanks to Malin Sjöö, Ann-Charlotte Eliasson, Jörgen Andersson, and Jeanette Purhagen for the help they provided during this work.

References


Measuring Flour Using Novel Sample Packing and Presentation with an IM 9500 Near-Infrared Instrument – Calibration Development, Validation, and Transferability

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Introduction
Grains and seeds belong to one of the most important staple food categories in the world. They are used in a wide variety of products and processing industries including food, feed brewing, and vegetable oil production. The composition of seeds and grains is very important as farmers, elevators, and processors use the information for critical purchasing, processing, and formulating decisions. Grains are often segregated based on compositional differences to optimize price and maximize the value during processing.

The Inframatic 9500 (IM9500) is a grain analyzer based on industry standard Near-Infrared (NIR) technology transmittance technology. The instrument passes a monochromatic light beam through a sample. The sample interacts with the light dependent upon several factors, including molecular composition. The light passing through the sample is therefore modified in some form. A detector on the far side detects the transmitted light. The detector signal is amplified and processed. The instrument uses advanced chemometric calibrations to convert the signal into constituent results such as moisture, protein, oil, and ash. The IM9500 is also standardized to a National Institute of Standards and Technology (NIST) reference material for wavelength scale accuracy. It is officially approved for grain analysis in several countries (1-4).

In addition to measuring grains and oilseeds, many processors need to measure samples in powder and meal form. A new flour module was developed to enable flour measurements for millers and bakers using the IM 9500. Calibrations were developed for data collected using the flour module for moisture, protein, ash, and wet gluten in flour. The calibrations were further validated with independent samples. The results are presented in this paper.

Material and Methods

Sample presentation
Sample handling and presentation can affect results. For samples such as flour, packing a sample cell or cuvette differently can affect the density and therefore the amount of light transmitted through the sample. The new module and packing method are designed to minimize these differences. Multiple operators of varying degrees of training performed the measurements to include normal operator variation in the studies. The new flour module, Figures 1 and 2, was used for the measurements. The packing procedure of the module is described in a video (5).

Figure 1: Flour module and preparation kit

Figure 2: Flour module
**Calibration development**

Over 900 wheat flour samples from eight countries on three continents were used as a calibration set. The calibration set includes both hard and soft wheat flours. Note that reference data was not available for all samples on all parameters.

The samples were analyzed in replicate on several IM 9500s using multiple operators. In addition, samples were also sent to an external qualified analytical lab for reference analysis. These measurements were performed in duplicate. Data on samples at varying temperatures were collected to inoculate the calibration to temperature variation. The resulting data was combined and partial least squares (PLS) regression was used to generate the calibrations for moisture, protein, ash, wet gluten, and L*.

**Validation of the calibrations**

For the validation of the calibrations, 300 wheat flour samples from China were analyzed using the calibrations as described in the previous section for prediction. Each sample was analyzed in duplicate to measure re-pack reproducibility. The validation was made for moisture, ash, and wet gluten.

**Calibration transferability**

Thirty-three wheat flour samples from the USA were analyzed for test calibration transferability across five IM 9500 instruments. The sample cell was re-packed for each instrument. The comparison was performed using the ash results predicted by the globally developed calibration.

**Results**

**Calibration**

The summarized results from the calibration measurements are displayed in Table 1.

<table>
<thead>
<tr>
<th>Moisture</th>
<th>Protein</th>
<th>Ash</th>
<th>Wet Gluten</th>
<th>L*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>9.15</td>
<td>5.10</td>
<td>0.36</td>
<td>12.28</td>
</tr>
<tr>
<td>Max</td>
<td>15.59</td>
<td>21.30</td>
<td>0.89</td>
<td>52.70</td>
</tr>
<tr>
<td>Correlation</td>
<td>0.991</td>
<td>0.997</td>
<td>0.973</td>
<td>0.997</td>
</tr>
<tr>
<td>SECV</td>
<td>0.14</td>
<td>0.15</td>
<td>0.03</td>
<td>0.41</td>
</tr>
<tr>
<td>Sample (n)</td>
<td>902</td>
<td>557</td>
<td>822</td>
<td>537</td>
</tr>
</tbody>
</table>

As can be seen in Table 1, the ranges for the measured parameters are wide and encompass both hard and soft wheat flour. As demonstrated in Figures 3 and 4, the samples are well distributed across the ranges as well. The Lab to NIR correlations are better than 0.99 for moisture, protein, and wet gluten. Ash and L* have correlations of 0.97 and 0.96 respectively.

![Figure 3: Correlation for protein in wheat flour](image-url)
**Validation**

The validation of the calibrations was performed on moisture, ash, and wet gluten, and the results are presented in Table 2.

<table>
<thead>
<tr>
<th></th>
<th>Moisture</th>
<th>Ash</th>
<th>Wet Gluten</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>13.68</td>
<td>0.40</td>
<td>24.20</td>
</tr>
<tr>
<td>Max</td>
<td>15.27</td>
<td>0.84</td>
<td>43.30</td>
</tr>
<tr>
<td>Correlation</td>
<td>0.879</td>
<td>0.982</td>
<td>0.537</td>
</tr>
<tr>
<td>SEP</td>
<td>0.14</td>
<td>0.023</td>
<td>1.24</td>
</tr>
<tr>
<td>Repack SD</td>
<td>0.03</td>
<td>0.01</td>
<td>0.30</td>
</tr>
<tr>
<td>Bias</td>
<td>-0.20</td>
<td>0.03</td>
<td>1.28</td>
</tr>
<tr>
<td>Sample (n)</td>
<td>333</td>
<td>333</td>
<td>301</td>
</tr>
</tbody>
</table>

The validation samples were all within the ranges of the calibration samples.

Ash has the highest correlation followed by wet gluten and moisture. As can be seen in Table 2, the Repack Standard Deviation (Repack SD) indicates that packing of the flour module adds little error to the system.

**Transferability**

The transferability of the global calibrations was measured by using predicted results from five different IM 9500 instruments. The results for ash prediction from the five instruments are displayed in Table 3. All results are on dry basis.

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>0.386</td>
<td>0.577</td>
<td>0.391</td>
<td>0.384</td>
<td>0.374</td>
</tr>
<tr>
<td>Max</td>
<td>0.563</td>
<td>0.654</td>
<td>0.669</td>
<td>0.574</td>
<td>0.651</td>
</tr>
<tr>
<td>Correlation</td>
<td>NA</td>
<td>0.995</td>
<td>0.991</td>
<td>0.973</td>
<td>0.992</td>
</tr>
<tr>
<td>SD</td>
<td>NA</td>
<td>0.010</td>
<td>0.017</td>
<td>0.011</td>
<td>0.010</td>
</tr>
</tbody>
</table>

Instrument A was used as the reference instrument. The results from instrument B – E were plotted against the results from instrument A, Figure 5.

In Table 3 and Figure 5 it is clear that the correlation and performance amongst the five instruments is very good. The small standard deviations further demonstrate that the sample preparation and presentation can be performed by different operators with negligible effects on the results.
Conclusions
Stable calibrations for measuring flour samples using the IM 9500 equipped with the new flour module were developed. The calibrations were validated for moisture, ash, and wet gluten and effect of sample re-pack was tested. High lab to NIR correlations and very small standard deviations for the sample re-packs demonstrate the stability of the calibrations and sample presentation method. Minimal differences were observed between five instruments predicting the same samples. The calibration, validation, and transferability data indicate that the IM 9500 can measure flour accurately, for a wide range of flour sample types, and that the new flour packing and presentation method nearly removes all operator effects on the measurements.

References


3. Physikalisch-Technische Bundesanstalt (PTB), Approval number 11.26/13.01


Figure 5: Ash in flour prediction, instruments B – E versus instrument A
BVM


DA 7200

RVA


SKCS

TVT


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