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Wheat four quality evaluation from the baker's perspective: comparative assessment of 18 analytical methods

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Abstract In this study, we sourced 37 commercial flours from 14 mills based on 7 countries and analysed them with a total of 18 methods, generating 90 single analytical values for each flour. The 18 methods were chosen to cover the analytical practice of most European mills and bakery companies, as well as particle charge detection, GlutoPeak and solvent retention capacity as emerging methods. We investigated the relationship between the data from the individual methods, and performed a principal component analysis to describe the structure of the data set and identify the main underlying flour properties. Four principal components accounted for 64.8% of the total variance. They were interpreted as (PC1) starch gelatinization properties, (PC2) hydration properties, (PC3) dough resistance at variable water amount, and (PC4) dough strength at fxed water amount. From the emerging methods, solvent retention capacity (sodium carbonate and water) was highly correlated with PC2, while the GlutoPeak max torque was highly correlated with PC4.

Keywords Wheat flour · Principal component analysis · Rheology · Functional properties

Introduction

Both industrial and craft bakeries are facing the problem of variability of wheat four quality. This variability makes it impossible to bake over a longer period of time with constant

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recipes and constant process parameters. To be able to react pro-actively to four quality variations, bakery companies need reliable analytical methods that allow predicting the behaviour of four in production as well as the fnal bakery results. In practice, four analysis is usually performed in the mill; bakery companies rely on values provided on analytical certifcates, which typically include 5–10 parameters.

In this context, millers and bakers need to agree on a set of measurements that is practicable in daily production and delivers the most useful information. Interestingly, across Europe, the analytical practice difers depending on the countries. In UK and Ireland, for example, the level of damaged starch is considered to be of high importance for functionality. In France and Spain, AlveoLAB measurements are considered as a standard. In Germany, Farinogram measurements are common. One could argue that the reason for this diferent practice lies in diferent applications (local bread recipes), but a part of the explanation certainly lies in historical reasons, habits, and the continuous use of equipment already available in the mills.

Globalization of wheat commerce, modifcation of wheat quality related to climate change [[5,](#page-9-0) [22](#page-10-0)], evolution in bakery production practice (industrialisation, increasing use of freezing technology $[3, 9]$ $[3, 9]$ $[3, 9]$), change in consumption habits, and the availability of novel flour analysis methods are good reasons for questioning the four analytical practice.

In this study, our goal was to compare and better understand the information provided by diferent four analysis methods used across Europe, including both established and emerging methods. To this end, we analysed 37 commercial fours from 14 mills located in 7 European countries by 18 diferent methods, generating 90 analytical values for each four. The similarities and diferences of the data obtained by the diferent methods were evaluated by statistical techniques.

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Table [1](#page-1-0) gives an overview of the methods used in our study. Some methods quantify the amount of specifc four components (water, minerals, protein, acids, and damaged starch), while other characterize the functionality of gluten, starch, pentosans, and alpha-amylases. The table gives an evaluation of the level of skills required, the price category of the equipment, the workload per sample, and the analysis time.

Experimental

Raw materials

37 wheat flours (50 kg of each) were sourced from 14 European mills located in Belgium, France, Germany, Italy, The Netherlands, Spain, and the United Kingdom. The fours

substance concen-

Functionality assessment of were specifed regarding their ash content (0.50–0.65 after ICC 104), their protein content (min 11.0 after ICC 167), and their moisture content (max. 16.0 after ICC 110/1). In some countries, it is usual to add some substances to the flour in the mill during production (e.g., gluten, enzymes, malt flour, vitamins, and minerals). Our flours were packed without these additions at our demand. The only additive we accepted was ascorbic acid, which is used widely across Europe and was contained at concentrations between 0 and 25 ppm in the assessed fours.

Analytics

Price category Level of equipment

of skills

All 37 flours were analysed by a series of methods that were selected to refect the diversity of analytical approaches commonly used by millers and bakers across Europe. The corresponding ICC and AACC standards are mentioned in

> Approximate workload per sample

Approximate analysis time

Table 1 Comparison of the methods used in the study Method/device Standard Measurement of

Tables [1](#page-1-0) and [2.](#page-3-0) The selected methods included moisture, ash, protein, wet and dry gluten, and damaged starch content as well as pH and acidity. Furthermore, functionality assessment was performed with the established methods sedimentation value, falling number, Brabender (Duisburg, Germany) Farinograph, Extensograph and Micro Visco Amylograph, Chopin (Villeneuve-la-Garenne, France) Mixolab, AlveoLAB and Rheo F4, as well as Perten (Hägersten, Sweden) Glutomatic and Rapid Visco Analyser. Finally, the following emerging methods and devices were used: Brabender GlutoPeak, Chopin SRC-CHOPIN, and BTG (Eclépens, Switzerland) Mütek PCD-05. GlutoPeak is a gluten aggregation test, giving an indication of the gluten strength (aggregation energy GPG) and of the speed of aggregation (time of maximum torque GPT). Several authors reported correlations of the GlutoPeak values with Alveograph, Extensograph, and Farinograph values [[18,](#page-10-3) [10](#page-10-4)] and with the level of protein and gluten [[19\]](#page-10-5). Compared to those methods, the main advantages of GlutoPeak are the speed of analysis and the lower price of the device. With the SRC-CHOPIN based on the solvent retention capacity method [[14\]](#page-10-6), the functionality of gluten, pentosans and damaged starch, as well as four hydration can be investigated in one device. Particle charge detection investigates the electrical load of macroions in a flour suspension and represents a different, innovative approach to flour functionality $[21]$ $[21]$.

A code was assigned to each parameter, as shown in Table [2](#page-3-0). For each flour, all analytical investigations were performed within 2 weeks, to exclude as far as possible the infuence of quality change over time (as described in [\[8](#page-10-7)]) on the correlations observed. The acid ascorbic content ASA as declared by the mills was also included into the data set.

Data analysis

The Pearson's correlation coefficients relating the individual parameters were calculated, with the corresponding *P* values. The structure of the data set was further investigated by carrying out a principal component analysis with a Varimax rotation on four principal components. All calculations were performed using IBM SPSS Statistics version 22.

Results

Overview of data

Table [2](#page-3-0) gives an overview of the data obtained, with the minimum and maximum values observed for each parameter, the average, and the relative standard deviation. Some values were found to be slightly out of our specifcation, but we chose to keep the corresponding samples in the evaluation, as they represent the quality available on the market.

Observed correlations

Table [3](#page-6-0) gives all correlation coefficients with an absolute value superior or equal to 0.7 and a *P* value below 0.001, which were calculated between parameters generated by different methods. Table [4](#page-7-0) gives the results of the PCA, whose four frst principal components account for 64.8% of the variance in the data set. Figures [1](#page-8-0), [2](#page-8-1) show loadings plots of the single analytical parameters on the four principal components. In the following, the main relationships observed are described following the structure given by the PCA.

The frst principal component is related to rapid visco analyser values (peak viscosity VAM, holding viscosity VAY, setback viscosity VAS, and fnal viscosity VAV), micro visco amylograph values (viscosity at start of cooling MVD, viscosity at start of holding MVC, hot viscosity MVB, and cold viscosity MVE), Mixolab values (C4 torque X4D, C5 torque X5D, C4 time X4T, and stability XST), and the falling number FAN. Referring to Table [3A](#page-6-0), the rapid viso analyser values have strong and signifcant correlations both with the falling number FAN and the Mixolab C4 and C5 torque X4D, X5D and C4 time X4T.

The second principal component is related to solvent retention capacity values (calcium carbonate SRC, water SRW), the level of damaged starch DST, some AlveoLAB values (extensibility AVL, AVG, ratio AVR, and tenacity AVP), Mixolab hydration XHY, Rheo F4 gaseous release RGC and RGS, particle charge at 4 min of swelling P04, and the temperature at start of gelatinization from the micro visco amylograph MTA and the rapid visco analyser VAP. Referring to Table [3B](#page-6-0), the SRC water value SRW correlates with some PCD values (P04, P24), the damaged starch level DST, the Mixolab hydration value XHY, the AlveoLAB tenacity AVP and ratio AVR, and the pasting temperature VAP as measured by the rapid visco analyser. The solvent retention capacity calcium carbonate value SRC correlates with all the previous ones and the AlveoLAB AVL and AVG values. Next to this, the Mixolab hydration value XHY correlates with the Farinograph water absorption FWA as well as with all AlveoLAB values. The Farinograph water absorption FWA also correlates with the dough tenacity AVP as measured by the AlveoLAB.

The third principal component is almost solely related to some Extensograph values (resistance ER45, ER90, and ER135, resistance maximum EM45, EM90, and EM135, ratio resistance/extensibility EY45, EY90, and EY135, and ratio resistance maximum/extensibility ED45, ED90, and ED135), only with the amount of ascorbic acid ASA also having a high value on it (above the threshold of 0,7). Referring to Table [3C](#page-6-0), the amount of ascorbic acid ASA is related to the Extensograph ratio values ED90, ED135, and EY135.

The fourth principal component is related to further AlveoLAB values (baking strength AVW and index AVI),

Method/device	Standard		R Output	Code	Mean	Min	Max	rsd $(\%)$	Unit
Moisture	ICC 110/1	3	Moisture after 90 min	MOI	0.13	0.11	0.15	6.1	$%$ (dl)
Ash content	ICC 104/1	2	Ash in dry substance	ASH	0.57	0.44	0.70	10.0	$%$ (dl)
Protein content	ICC 105/2	2	Protein in dry substance	PRT	12.2	10.1	13.9	6.9	$%$ (dl)
Sedimentation value	ICC 116/1	2	Sedimentation value	SDV	34.3	24.8	53.4	16.2	mL
Brabender GlutoPeak	Setting:	2	Time of max torque	GPT	72.7	46.5	124.0	28.1	$\mathbf S$
	25 °C, 3000 rpm		Max Torque	GPM	81.2	67.0	91.5	8.9	BU (mu)
			Torque before maximum	GPB	34.3	23.5	44.0	16.4	BU (mu)
			Torque after maximum	GPA	60.6	46.5	75.5	9.3	BU (mu)
			Startup energy	GPS	277	148	366	18.0	GPE (mu)
			Aggregation energy	GPG	1916	1512	2329	9.7	GPE (mu)
Perten Glutomatic	ICC 155		2 Wet gluten	GMW	27.7	21.8	33.0	10.3	$%$ (dl)
			Dry gluten	GMD	10.6	7.9	13.3	10.5	$%$ (dl)
			Gluten index	GMI	84.4	62.6	94.4	9.9	$%$ (dl)
pH and acidity	ICC 145	$\overline{2}$	pH	PH	6.3	6.0	7.6	4.7	$-$ (dl)
			Acidity	ADT	0.97	0.80	1.21	10.6	acidity unit
Falling number	ICC 107/1	5	Falling number	FAN	356	277	419	11.4	$\bf S$
Perten		3	Peak viscosity	VAM	2227	1498	2910	15.0	RVU (mu)
Rapid Visco Analyser			Holding viscosity	VAY	1299	611	1776	25.0	RVU (mu)
			Breakdown viscosity	VAB	928	761	1186	10.5	RVU (mu)
			Final viscosity	VAV	2348	1303	3061	19.6	RVU (mu)
			Setback viscosity	VAS	1050	691	1337	13.9	RVU (mu)
			Peak time	VAT	6.11	5.56	6.38	3.2	$\bf S$
			Pasting temperature	VAP	59.9	58.1	62.5	1.9	$\rm ^{\circ}C$
			Peak temperature	VAX	95.1	95.0	95.2	0.1	$\rm ^{\circ}C$
Brabender Micro Visco Amylo- graph		2	Hot viscosity	MVB	633	316	1138	32.5	mPas
			Cold viscosity	MVE	1491	922	2531	24.8	mPas
			Temperature of start of gelatinisa- tion	MTA	59.2	54.7	62.7	3.2	$\rm ^{\circ}C$
			Temperature of max viscosity	MTB	89.1	83.3	91.7	2.6	$\rm ^{\circ}C$
			Viscosity at start of cooling	MVD	417	139	819	39.7	mPas
			Viscosity at start of holding	MVC	595	221	1123	38.3	mPas
			Difference Viscosity B-D	MVX	216	137	366	24.4	mPas
			Difference Viscosity E-D	MVY	1074	773	1712	20.7	mPas
Chopin SDmatic	ICC 172		1 Damaged starch (AI)	DST	94.4	93.2	95.9	0.8	$%AI$ (dl)
SRC-CHOPIN	AACC		1 SRC Water	${\mbox{SRW}}$	65.6	55.4	73.8	6.9	$\%$ (dl)
	56-11		SRC Sucrose	SRS	105	97	116	4.7	$%$ (dl)
			SRC Lactic Acid	SRL	124	97	156	8.9	$\%$ (dl)
			SRC Sodium Carbonate	SRC	83.1	68.2	94.1	7.3	$%$ (dl)
Mütek Particle Charge Analyser	$[21]$	2	Total charge after 4 min swelling	P ₀₄			-1.52 -2.70 -0.24 -35.5		$C.g^{-1}$
			Total charge after 24 min swelling	P ₂₄			-1.46 -2.66 -0.19 -36.1		$C.g^{-1}$
			Total charge after 44 min swelling	P44		-1.22 -2.51 0.05		-47.4	$C.g^{-1}$
			Total charge after 64 min swelling	P64		-0.84 -2.32 0.13		-70.7	$C.g^{-1}$
Brabender Farinograph	ICC 115/1		2 Water absorption 500 BE	FWA	58.1	50.9	61.7	4.0	$%$ (dl)
			Dough development time	FDD	2.03	1.36	4.63	28.0	min
			Stability	FST	9.80	2.83	22.17	42.9	min
			Dough softening	FDS	61.1	21.0	107.0	40.0	ВE
			Farino quality value	FQV	82.4	29.0	181.5	47.2	$-$ (dl)

Table 2 Methods used for four analytics in WP2 and results obtained with the 37 investigated fours (mean, min, max, and relative standard deviation)

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Table 2 (continued)

Table 2 (continued)

R number or repetitions, *dl* dimensionless, *mu* device manufacturer unit

the protein content PRT, the Extensograph extensibility at 45 min EX45 and the GlutoPeak max torque GPM. Referring to Table [3D](#page-6-0), the AlveoLAB baking strength AVW and index AVI correlate with the protein content PRT. The baking strength AVW also correlates with the GlutoPeak max torque GPM.

In addition to the structure given by the principal component analysis, Table [3](#page-6-0)D presents further interesting correlations. Indeed, the AlveoLAB baking strength AVW is also related to the Extensograph energy values EE45, EE90, and EE135, the GlutoPeak torque at maximum GPM, and torque after maximum GPA. The AlveoLAB index value AVI also correlates with the Extensograph energy and maximum values EE45, EE90, EE135, EM90, and EM135. Furthermore, the AlveoLAB tenacity AVP is related to the GlutoPeak start energy GPA, aggregation energy GPS, and torque after maximum GPG. Next to this, the SRC lactic acid value SRL correlates with the sedimentation value SDV. The Farinograph and Mixolab hydration values FWA and XHY correlate with the GlutoPeak start energy GPS, aggregation energy GPG, and torque after maximum GPA. Finally, the dry gluten GMD as determined with the Glutomatic correlates with the protein content PRT.

Other relationships are described in Table [3](#page-6-0)E: The Farinograph stability FST correlates with the AlveoLAB baking strength AVW, the Mixolab stability XST, and the Mixolab C2 torque X2D. The Farinograph dough-softening FDS correlates negatively with the Mixolab stability XST and the Mixolab C2 torque X2D. The Farinograph quality value FQV is related to the Mixolab stability XST, the Mixolab C2 torque X2D, and the Mixolab C1 time X1T.

Parameters without signifcant correlations

It is remarkable that the ash content ASH, the pH value PH, the acidity ADT, most of the Rheo F4 values, and the Gluten Index GMI and Wet Gluten GMW level were found to have no high and signifcant correlations with the other parameters measured. This indicates that these parameters are, to a certain extent, independent from the other four properties.

Discussion

Starch gelatinisation and viscosity

PC1 is related to starch gelatinisation and viscosity. The methods rapid visco analyser, micro visco amylograph, Mixolab, and falling number are all based on forming a starch gel by following a temperature cycle in the presence of water. The measured viscosities are mainly related to starch gelatinization, starch degradation by endogenous alpha-amylases and starch retrogradation. Similar correlations were reported by Defenbaugh and Walker [[6\]](#page-9-2) (RVA vs. micro visco amylograph) as well as Peña and Posadas-Romano [[20\]](#page-10-8) (Mixolab C4 and C5 vs. Falling number).

No relation with analytical values describing gluten was observed on PC1 (at the threshold of 0,7), suggesting that the viscosity values represented on this principal component are mainly infuenced by starch, not gluten.

In summary, a high score on PC1 indicates a high viscosity after gelatinization, which may be related to a low alpha-amylase activity. PC1 can be best assessed by the micro visco amylograph and the rapid visco analyser.

Table 4 Loading values of the single analytical parameters on the frst four principal components after Varimax rotation (values >0.7)

Water absorption and dough plasticity/elasticity at fxed water amount

PC2 describes water absorption and the dough plasticity/ elasticity ratio at fxed water amount. The level of damaged starch, as assessed by the SDmatic (DST) and the SRC sodium carbonate value (SRC), has a strong infuence on hydration, as assessed by the SRC water value (SRW), and the Mixolab and Farinograph hydration values (XHY, FWA). Similar results were obtained by Hammed et al. [\[11](#page-10-9)].

It is, furthermore, well understandable that the hydration is related to the plastic/elastic characteristic of the dough, as assessed with the AlveoLAB: the higher the level of water binding (by the starch but also by the gluten and the pentosans), the dryer the dough will be, which results in a lower extensibility (AVL, AVG), and a higher resistance to deformation (AVP), increasing the ratio P/L (AVR). This applies to the AlveoLAB measurement as it is performed with a fixed amount of water according to ICC 121, and is consistent with the results reported by Van Bockstaele et al. [\[23\]](#page-10-10) and Li et al. [[15\]](#page-10-11). The Extensograph measurement, on the contrary, is performed with a variable amount of water according to ICC 114/1, which probably explains why its values are not found on PC2.

The observed correlation of the RVA pasting temperature VAP with the SRC sodium carbonate (SRC) is in line the results of Barak et al. $[2]$ $[2]$ $[2]$, Yu et al. $[24]$ $[24]$ $[24]$, and Ma et al. [\[17\]](#page-10-13) stating a correlation with the level of damaged starch, and can be interpreted as an earlier pasting in case of better hydration of the starch granules.

It is interesting that the SRC water and sodium carbonate values (SRW, SRC) are related to the PCD total charge after 4 and 24 min (P04, P24). Longin et al. [[16](#page-10-14)] already observed this phenomenon on emmer fours. This suggests that a higher (negative) charge of the macroions in the dough leads to a higher water binding.

A high score on PC2 indicates a low water absorption, a low level of damaged starch, and a high dough extensibility at fxed water amount. PC2 can be well assessed by the measurement of the SRC values, the level of damaged starch, and the AlveoLAB extensibility.

Dough resistance at variable water amount

PC3 is related to dough resistance as measured with the Extensograph. PC3 shows that doughs that are standardized to a certain frmness (500 BU) by varying the amount of water in the Farinograph will still, after a resting time of 45, 90, and 135 min, have a diferent resistance to deformation (ER45, EM45, ER90, EM90, ER135, and EM135). As there is no relation of PC3 to the extensibility values, it is probable that the observed correlation with the ratio values EY45, ED45, EY90, ED90, EY135, and ED135 is only related to the resistance values.

Remarkably, the amount of ascorbic acid has a high correlation with the ratio values ED90, ED135, and EY135, which indicates that the addition of ascorbic acid favours elastic properties (resistance to deformation) at the expense of plastic properties (extensibility). This is consistent with the data of $[1, 7]$ $[1, 7]$ $[1, 7]$ $[1, 7]$.

A high score on PC3 indicates a high resistance to deformation of dough at variable water amount. It can be well assessed with the Extensograph and can be, to a certain extent, infuenced by the addition of ascorbic acid.

Fig. 1 Loadings plot of the single analytical parameters on PC1 and PC2. The methods that reached values higher than 0.7 on PC1 or PC2 are shown in a specifc color and mentioned in the legend

Fig. 2 Loadings plot of the single analytical parameters on PC3 and PC4. The methods that reached values higher than 0.7 on PC4 or PC4 are shown in a specifc color and mentioned in the legend

Dough strength at fxed water amount

PC4 describes dough strength, i.e., the combination of elastic and plastic properties, as measured by the AlveoLAB at fxed water amount. This property is related to the protein content and the GlutoPeak torque values (GPA, GPM). The latter is in line with the fndings of Marti et al. [\[19\]](#page-10-5), who developed correlation models to predict AlveoLAB values from GlutoPeak measurements.

A high score on PC4 indicates a high dough strength at fxed water amount. It can be well assessed with the AlveoLAB.

Other correlations

It is interesting that the AlveoLAB dough strength (AVW) is related to the Extensograph energy values (EE45, EE90, and EE135). Both values are based the same concept (combination of resistance and extensibility measured when stretching dough); despite the diferences between both measuring systems (infating a bubble of dough vs. stretching a dough piece) and the diferences of water amount (fxed vs. variable), the values are still correlated—unlike the other values determined by AlveoLAB and Extensograph.

The correlations found between the values measured by the Farinograph and the Mixolab confrm previous fndings, e.g., from [\[4,](#page-9-6) [13\]](#page-10-15). The correlation between the hydration values and the GlutoPeak values suggests that a stronger gluten will bind more water. This in line with the data of Marti et al. $[19]$ and Fu et al. $[10]$ $[10]$.

The absence of strong correlation between the level of gluten (wet and dry) and the measured functional values suggest that in our flour samples, the quality of the gluten was highly variable. This can be explained by our deliberate choice to use commercial flours from very various origins.

Conclusions

Our results highlighted the importance of the following properties in wheat four analysis: starch gelatinization, hydration, dough resistance at variable water amount, and dough strength at fxed water amount. These results may be of interest for millers and bakers when it comes to selecting methods appropriate to describe quality in an efective yet comprehensive manner. It seems indeed advisable to use at least one method from each of the four groups defned by the principal components. Several options are available regarding the choice of instruments. On the contrary, it probably makes less sense to use two methods which results are highly correlated to one another. It is to be noted that, as four processing companies will hardly invest in both an AlveoLAB and an Extensograph, the possibility to work with a single instrument at both fxed and variable water amount should be explored.

Similarly, we suggest that research teams may use our results to select the most appropriate setting of instruments to characterize their wheat fours—although this may result in a diferent selection of instruments than in processing companies, as the time of analysis may be less relevant.

The values assessed by the emerging methods SRC (as performed with the SRC-CHOPIN) and Brabender Gluto-Peak show to have high correlations with established methods. This suggests that these methods do not assess new properties of four, but rather represent an alternative to established methods—especially considering that they are faster and that they may be more cost effective.

As far as the baking performance is concerned, it is of course of interest to assess which of the investigated methods are mostly related to the properties of the fnal baked goods. Corresponding data will be published by our project team in a further article [\[12](#page-10-16)].

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Compliance with ethical standards

Confict of interest The authors declare that they have no confict of interest.

Compliance with ethics requirements This article does not contain any studies with human or animal subjects.

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