Colour Analysis and Discrimination of Laboratory Prepared Pasta by Means of Spectroscopic Methods

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Abstract

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For the CIE Lab colour profile determination of laboratory prepared pasta, two sample granulations and two spectral methods were tested. Pasta was manufactured progressively from semolina, common wheat, and corn flour. Sufficient colour spectra ranges were ensured by means of fortification with 9 non-traditional cereals in the first case, 8 natural colorants in the second one, and with 12 gluten-free pasta recipes in the last case. Both factors (i.e. granulation and spectral method) were proved as statistically significant by the cluster, variance and principal component analyses. In the comparison of the effects on the pasta composition and the spectral method, the latter demonstrated a stronger impact on the pasta colour profile measured.

Keywords: pasta; colour profile; granulation; spectral method; PCA

All over the world, food based on cereals as wheat or rice is explicitly the most frequently consumed stuff. In the case of wheat, the grain undergoes milling treatment with different types of flour as end-products adequately for their further use in food industry. With the exception of some regional habits, two botanical types of the Triticum genus are predominantly milled – *T. durum* and *T. aestivum*, the flours from which can be used for pasta manufacturing. Generally, wheat flour pasta is traditionally produced as eggless from semolina (durum wheat flour), and as eggs-containing from common flour by the recipe.

Flour colour is affected by different factors, firstly by milled kernel type and flour yield, and secondly by flour composition as ash, protein, pigment, and damaged starch contents (Miskelly 1984) corresponding to the tested flour stream and extraction rate (Oliver *et al.* 1992; Scanlon *et al.* 1993; Feillet *et al.* 2000; Hatcher *et al.* 2008). As dominant colour items can be highlighted the natural pigments of carotenoids or xanthophylls (Humphries *et al.* 2004; Fratianni *et al.* 2005) and protein molecular composition (Ohm *et al.* 2008). Besides that, the tint of cereal flour composites for pasta colour depends both on the flour botanical origin and mixing ratio (Hatcher *et al.* 2008; Zhu *et al.* 2009). The most frequently used crops for the pasta colour (and also nutritional) modification are amaranth and buckwheat (Rayas-Duarte *et al.* 1996; Zhu *et al.* 2008), corn (Jukić *et al.* 2007; Ugarčić-Hardi *et al.* 2007), and legumes as chickpea, soy, yellow or green pea, faba bean (Zhao *et al.* 2005; Chillo *et al.* 2008; Nasehi *et al.* 2009; Wood 2009; Petitot *et al.* 2010). To less used raw materials belong colour or archaic wheat types as amber, spelt, or diccocum wheat (Hatcher *et al.* 2008; Švec *et al.* 2008), barley or millet (Švec *et al.* 2008).

In the colour determination of the agriculture and food products, the influence of the used method and apparatus can not be omitted. The colorants ratio as carotene, lutein, or flavonoids in wheat flour mentioned above is usually evaluated by HPLC technique (Fratianni *et al*. 2005; Leenhardt *et al*. 2006), which is a time-consuming and more demanding procedure with the sample preparations than the use of visible/near-infrared reflectance spectrometers or colour spectrophotometers. A review of their applications in the field of agriculture and food commodities was published by McCaig (2002), who analysed 50 products including semolina, cornmeal, and wheat flour by means of both spectral techniques. The latter method operates mostly in the $L^*a^*b^*$ colour space (the Commission Internationale de l'Eclairage, CIE 1976) with necessary spectrophotometric equipment (usually the Minolta or Hunterlab models). In this space, L^* coordinate determines the white-black scale, while a^* and b^* the red-green and yellow-blue scales, respectively, from value 100 to 0 for the L^* and positive to the negative direction of the other axes. Some parameters are calculated consecutively, as the chroma C^*, hue h^*, the (Hunter) whiteness index WI, or the total colour difference ΔE. For the proper measurement, the standard illuminant and observer angle selection is also necessary (usually daylight illuminant D65 and 10°, respectively).

Besides the raw materials colour profile, the pasta preparation conditions such as the recipe, water temperature and its added amount (Nasehi *et al*. 2009; Petitot *et al*. 2010), pasta press or extruder used, dryer type, and operating parameters finalise the product colour (Jukić *et al*. 2007). Pasta colour can be analysed in the intact or milled states. The sample disintegration is considered to be one of key operations due to the reflectance dependence on the material granulation; maximal correspondence can be reached between the gained data and visual perception of the pasta shade (Acquistucci *et al*. 1993).

The objectives of this work were to verify the use of two spectral methods for CIE Lab pasta colour determination, especially for classic yellow types. In respect of this, the UV4 Unicam spectrophotometer and the Minolta 2500d colorimeter for pasta colour testing were employed. Due to the reflectance measurement, also two sample disintegration methods were developed and the possible influence on the CIE Lab results was explored.

MATERIAL AND METHODS

Flour samples. The composite pasta tested (Table 1) was based on three basic raw materials. Set A of the yellow pasta was prepared from semolina (industrial mill 1), set B of the colour pasta from semi-fine pasta flour (from common wheat; industrial mill 2), and set C of the gluten-free pasta from corn flour (industrial corn mill 3). For the substitution, non-traditional cereal fine flours (from spelt, spring waxy barley, and tritordeum) were granted by research trials. The sampe 4 was lupine flour. Non-treated millet, buckwheat, amaranth, and soy were bought on the Czech market and milled under laboratory conditions to fine flour. Natural colorant powders, sepia ink, potato starch, and gluten-free mixture were also purchased on the market.

Pasta preparation and sampling. All pasta types (standard, naturally coloured, and gluten-free) were prepared following the internal standardised method described earlier (Hrušková & Vítová 2007; Švec *et al*. 2008).

Pasta disintegration followed the recommendation of the spectrophotometer manuals used in terms of the required powder granulation, and it led to the development of two methods. The coarser milling followed by sifting was done by using the KM4 grinder (OZAP, Prague, Czech Republic) and 485 μm sieve as previously (Švec *et al*. 2008), and the finer one was performed with the oscillation ball mill MM 301 (Retsch, Haan, Germany). The ball mill setting was at 28 Hz for the disintegration frequency and 3 min for the milling time, when the final sample granulation was approx. 5 μm (Lhotáková 2010). The grinding jar with capacity of 10 g was filled with 10 elbow macaroni pieces, and a smaller one for 7 g was used empty (without a metal ball) as a counterbalance.

Colour measurement. The use of the spectrophotometer Minolta CM-2500d and the Spectra Magic software was described in the previous paper (Švec *et al*. 2008) when the pasta powder preparation was done according to the first method (milling and sifting through 485 μm sieve). In pasta colour determination with the help of the UV4 Unicam apparatus, the samples of both granulations were measured. The approved macro for the colour measurement in the Vision 32 software was set for total reflectance measurement in the range 380–880 nm with step of 2 nm and in 10 cycles. A laboratory spoonful of the sample (approx. 2–3 g) was placed into the aluminium small ring cup and tightened proportionately. The smoothness of the sample surface in the cup was checked visually, and than the colour measurement was realised (Lhotáková 2010).

Data treatment and statistical assessment. In the case of the Minolta spectrophotometer application, $L^*a^*b^*$ were obtained directly. For each sample, the average values were computed from 5 particular measurements. The UV4 spectrophotometer recorded the data in the CIE XYZ space, so the data were properly transformed into the CIE Lab space according to the known relations mentioned in scientific papers (Oliver *et al*. 1992; McCaig 2002). As the illuminant standard, daylight D65 and observer angle of 10° were selected for both spectral methods. The comparison of the granulation and spectral method effects on the pasta colour profile was accomplished in the STATISTICA 7.0 software. Two-way ANOVA, hierarchical cluster, and principal component analyses were applied to discriminate between the 2 samples granulations and the 2 spectral methods for pasta $L^*a^*b^*$ colour profile evaluation.

Table 1. Pasta sample composition

Recipe characteristic ingredient	Sample name	Pasta composition
A. Yellow pasta fortifed by non-traditional cereals		
Standard 1	Py00	1 kg of semolina, 1 egg, water (base 1)
Spelt	Py04	base 1 with 20% semolina replacement by fine flour from non-traditional cereals
Tritordeum	Py05	
Barley	Py06	
Millet	Py09	
Corn	Py10	
Buckwheat	Py11	
Soya	Py12	
Lupin	Py13	
B. Pasta dyed by natural colourants		
Standard 2	Pc01	1 kg of pasta semi-fine flour, 1 egg, water (base 2)
Carrot	Pc11	base 2 + colourant fine powder/ink as 5% of flour weight
Blueberry	Pc15	
Basil	Pc16	
Carob	Pc17	
Rowan	Pc18	
Sepia ink	Pc19	
Seaweed Wakame	Pc20	
Dried mushrooms	Pc21	
C. Gluten-free pasta		
Millet	PX1	corn flour (CF) + 1% of CMC + 20% of fine flour from non-traditional cereals, water
Quinoa	PX2	
Amaranth	PX3	
Soya	PX4	
Rice	PX5	
Buckweat	PX6	
Potato starch	Px1	CF + 5% of potato starch, water
Potato starch	Px2	CF + 10% of potato starch, water
Gluten-free mixture	Px3	CF + 10% of gluten-free mixture, water
Gluten-free mixture	Px4	CF + 15% of gluten-free mixture, water
Gluten-free mixture	Px5	CF + 30% of gluten-free mixture, water
Gluten-free mixture	Px6	CF + 50% of gluten-free mixture, water

RESULTS AND DISCUSSION

Samples composition

Generally, colour differences among 30 manufactured pasta samples were primarily based on the main recipe components – semolina, semi-fine wheat pasta, and corn flours. Through the basic flours yellowness, a relative similarity could be presumed between semolina and corn flour as well as the pasta sets prepared. Further variations were achieved by 20% semolina replacement (set A in Table 1), 5% semi-fine pasta flour dying (set B), and by nutritional modification of gluten-free type (set C). All tested modifications should extend the Czech pasta production and enhance both consumer's attractiveness and diet.

Sample granulation effect – yellow pasta set

The comparison of the sample set A data gained by both disintegration and spectral methods are given in Table 2. The contribution of the finer milling on the ball mill MM301 showed lower ranges in the natural pasta colour coordinate L^* (whiteness). The smaller particles in the pasta MM301-powders caused lower light absorption and thus more intensive spectral response than KM4-ones, contributing to the whiteness L^* attenuation. The observed differences were statistically provable as shown the dendrogram in Figure 1 – the samples are gathered according to the disintegration method. Regardless of that, both super-clusters in the $L^*a^*b^*$ colour space are located very close together – the Euclidean distances maximum was

Table 2. Effects of sample granulation and spectral method on the pasta colour profile – yellow pasta fortifed by non-traditional cereals

Mill	Spectrometer	Colour coordinate		
		L^* (min–max)	a^* (min–max)	b^* (min–max)
KM4	Minolta	82.48–89.80	1.19–2.58	14.76–25.16
KM4	UV4	95.47–96.71	0.59–1.08	0.70–2.10
MM301	UV4	97.40–98.00	0.01–0.63	0.51–1.49

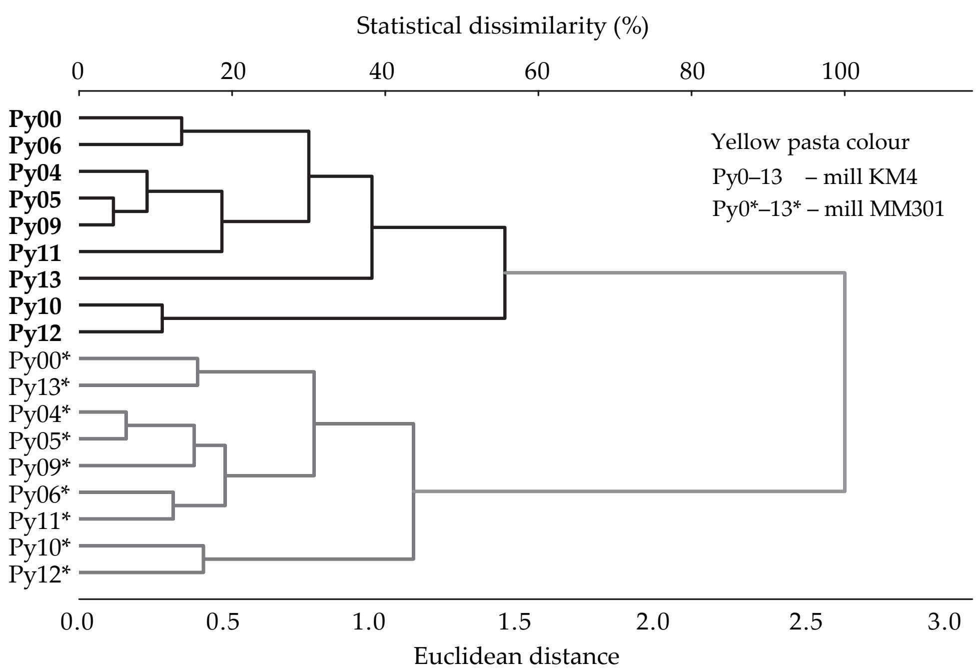

Figure 1. Effect of sample granulation on the yellow pasta colour profiles determined by hierarchical cluster analysis. Pasta colour profile – the UV4 spectral method

Table 3. Effects of spectral technique and sample composition on the pasta colour profile – yellow pasta fortifed by non-traditional cereals

Spectrometer	Colour coordinate		
	L^* (min–max)	a^* (min–max)	b^* (min–max)
Minolta	82.48–89.80	1.19–2.58	14.76–25.16
UV4	95.47–96.71	0.59–1.08	0.70–2.10
Minolta	45.72–91.60	–0.72–5.50	0.14–18.60
UV4	90.78–97.31	–0.36–0.79	–2.15–1.57
Minolta	84.07–92.15	1.36–3.67	17.01–32.27
UV4	96.21–97.42	0.89–1.29	2.33–3.65

Samples standard granulation – the KM4 mill method

lower than 3 units. Pasta spectral profiles closeness was found for the Py04, Py05, and Py09 samples (containing 20% of spelt, tritordeum, and millet, respectively) and in Py10-Py12 pair (pasta with 20% of corn and soy), independently on the sample granulation. For these samples colour profiles, the pasta composition factor is considered to be stronger than for the rest.

On the other hand, the Minolta spectral method at KM4 disintegration displayed multiple higher scatter compared to the UV4 spectrophotometer output, mainly in natural pasta colour coordinates L^* and b^* (whiteness and yellowness, respectively; Table 2). For the traditional yellow pasta and its nutritionally fortified variants, the combination of the coarser granulation and the Minolta colorimeter resulted in adequate distinction of the pasta tested.

Spectral method effect

In all three examined sets A, B, and C, lower L^* and reversely higher a^* and b^* values occurred in the colour profiles measured with the help of the Minolta spectrometer (Table 3). The data scatter was also three- to six-times higher than for $L^*a^*b^*$ values calculated from the UV4 reflectance spectra. Correlation analysis disregarding that verified the correspondence of the Minolta and the UV4 readings (Table 4), however, in the set A it was partially improvable due to its smaller extent.

The independence of the spectral method effect of the pasta sample composition was statistically proved by ANOVA (Table 5). The highest F-value for the spectrometer used was observed in the gluten-free pasta set C. With the exception of set C, both mentioned factors interaction was

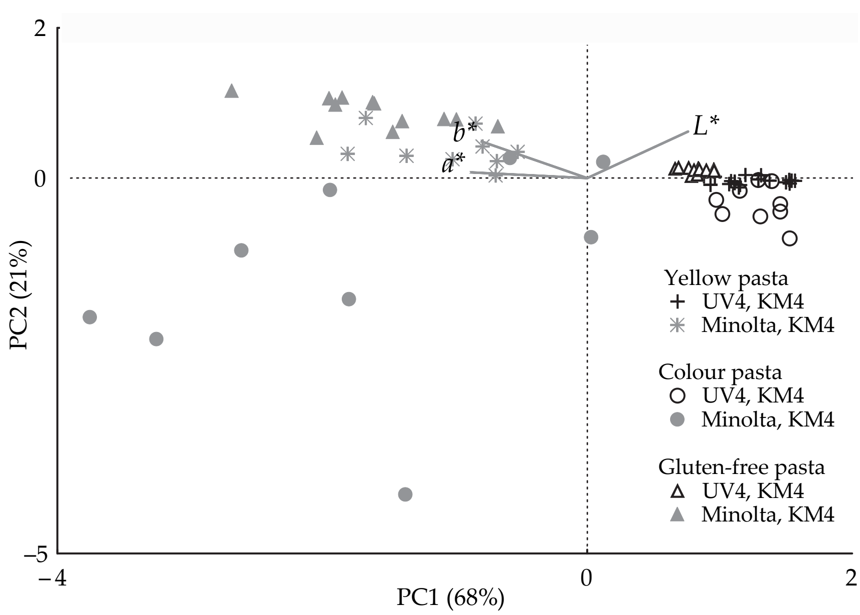

Figure 2. Effect of the spectral method on pasta colour profile determined by principal component analysis. Sample standard granulation - the KM4 mill method

Table 4. Correlaction analysis of the spectral method results correspondence

Colour coordinate†	L_2^*	a_2^*	b_2^*
A. Pasta with non-traditional cereals (N = 18, r = 0.467 at P = 95%; r = 0.589 at P = 99%)			
L_1^*	0.770**	–	–
a_1^*	–	0.365	–
b_1^*	–	–	0.355
B. Pasta with natural colourants (N = 18, r = 0.468 at P = 95%; r = 0.590 at P = 99%)			
L_1^*	0.987**	–	–
a_1^*	–	0.675**	–
b_1^*	–	–	0.872**
C. Gluten-free pasta with/without CMC (N = 24, r = 0.403 at P = 95%; r = 0.515 at P = 99%)			
L_1^*	0.875**	–	–
a_1^*	–	0.710**	–
b_1^*	–	–	0.869**

†lowercase numbers indicate pasta colour profile determined by: 1 – spectrometer Minolta, mill KM4; 2 – spectrometer UV4, mill KM4; **significant at P = 99%

statistically provable; with respect to F-values, its practical impact could be rated as negligible.

The samples mutual distance in $L^*a^*b^*$ space is documented in Figure 2, where the colour coordinates were transformed into the first two principal components (PC's). In the plane of the PC1 and the PC2, the samples are separated according to the spectrometer used; the Minolta items are negatively correlated with the PC1, and reversely for the UV4 ones. Eighty-one percent of the colour axis variability was explained by the first two PC's, 68% by the PC1, and 21% by the PC2 (Table 6). The main role could be noticed of the redness a^* on the samples distribution, which was sufficiently explained by the PC1 only (78%). Besides that, similar trends in the PC1-3 values for the L^* and the b^* coordinates confirmed their main importance in the pasta colour profiling.

Oliver *et al.* (1992) tested three spectrometer types (Micromatch 2000, Hunterlab D25-9SM, Minolta Chroma Meter 200) on the flour colour evaluation of 33 spring wheat cultivars, and found a sufficient agreement between all three method pairs for the L^* and the b^* characteristics (determination coefficients 0.957–0.987). For the a^*

Table 5. Analysis of variance of sample composition (nine cereals – A, nine natural colourants – B, twelve gluten-free pasta recipes – C), two spectrometers (D) and their interactions for pasta colour profile

Source of variation	Degree of freedom	F-value
A. Pasta with non-traditional cereals (hierarchical ANOVA)		
A	24	73*
D	3	7 138*
D × A	24	47*
B. Pasta with natural colourants		
B	24	476*
D	3	22 741*
D × B	24	289*
C. Gluten-free pasta with/without CMC		
C	33	48*
D	3	34 672*
D × C	33	32

*significant at P = 95%

Table 6. The portion (%) of explained variability by the first three principal components (PC's)

Colour coordinate	PC1	PC2	PC3
L^*	58**	38**	4
a^*	78**	2	20**
b^*	65**	25**	10**

**provable correlation between the colour coordinate and the proper PC (P = 99%)

values, mutual correspondence was the worst – the determination coefficients were 0.021, 0.284, and 0.153 in the pair of the Minolta-Micromatch, the Minolta-Hunterlab, and the Micromatch-Hunter readings, respectively. Similarly to our research, McCaig (2002) compared the colour of 50 food products estimated by 3 visible/near-infrared spectrometers and by 3 Minolta Chroma Meter models. The published results of these spectral methods comparison proved an excellent agreement for L^*, a^* and also b^* values – the range of regression coefficients was 0.94–0.99.

CONCLUSIONS

Colour visual perception is one of the dominant sensorial parameters of the pasta quality for consumers. Objectivised reflectance spectral methods allow precise direct or indirect determinations of the material colour and, consecutively, samples comparison, depending on the equipment used. As particle size affects the light absorption and reflection rate measured, individual optimal sample granulation is recommended for each colorimeter or spectrometer. In accordance with the aim to verify two spectral methods (Minolta CM-2500d and UV4 Unicam) suitability for pasta colour description, two disintegration methods were developed (particle size lower than 485 μm and approx. 5 μm).

In the pasta colour determination on the UV4 spectrophotometer, the tested pasta powder granulation of a distinct particle size significantly affected the reflectance spectra course and the CIE $L^*a^*b^*$ values as the final quality characteristics. A lower whiteness L^* as well as higher both yellowness b^* and redness a^* in the yellow pasta colour profiles were observed with a coarser powder. A light reflection rate was in that case lower, which allowed the dominance of the "colour" spectra wavelengths over single whiteness.

Statistical analysis confirmed the equability of the colour coordinates $L^*a^*b^*$ for pasta colour evaluation. Principal component analysis documented a predominance of the spectral method over the pasta composition similarly to the ANOVA test. Further, the investigated spectrometers were statistically most diverse in the red light component (a^*).

Summarised, the combination of coarser granulation and the Minolta colorimeter measurement seems to be the most effective process for pasta colour determination.

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