Analytical data for plum paste as a tool for evaluation of plum paste authenticity

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Summary
Plum (*Prunus domestica* L.) is a nutritionally and technologically important fruit. In the Czech Republic, there has been a long tradition of growing plum trees and manufacturing plum-based products. Plum paste is traditionally produced by boiling for a long period of time and contains only plums. In this study, we measured chemical composition and colour of sets of samples of plum paste focusing mainly on the following markers: soluble solids, ash, organic acids (malic, citric, shikimic), saccharides (glucose, fructose, saccharose, sorbitol), minerals (potassium, calcium, magnesium, sodium, phosphorus), phloridzin, rutin, 5-hydroxymethylfurfural, titratable acidity and total amino acid content. Plum paste was found to be a nutritionally rich source of saccharides (from 469.0 g·kg^{-1} to 571.5 g·kg^{-1}), organic acids (from 9.32 g·kg^{-1} to 19.58 g·kg^{-1} expressed in grams of malic acid) and minerals (from 2.98 g·kg^{-1} to 9.26 g·kg^{-1} expressed as ash), in particular potassium (from 1232 mg·kg^{-1} to 4299 mg·kg^{-1}). From a nutritional point of view, plum paste contains a very advantageous ratio of sodium to potassium (K/Na = 49.3). The aim of the study was to evaluate the chemical composition and quality of plum paste in order to fill in the missing data in food composition tables, and to facilitate authentication of plum products.

Keywords
Prunus domestica L.; chemometry; colour; phloridzin; adulteration

Plum (*Prunus domestica* L.) is a nutritionally and technologically important fruit for direct consumption and for production of various products, in particular the traditional ones. The origin and taxonomy of *Prunus domestica* is still controversial [1]. Correct taxonomic classification could, however, be provided by innovative methods such as analysis of allozyme polymorphism or analysis of chloroplast DNA variation [2]. Based on geographic distribution, plum can be divided into three groups: European – Asian, North American and Oriental [3].

The European – Asian group includes *P. domestica* L. (Greengages, Prunes, Yellow Eggs, Imperatries, Lombards), *P. domestica* subsp. *insititia* Bailey (Damsons, Bullaces, Mirabelles, St. Julians) and *P. cerasifera* Ehrt. (Myrobalans, common wild plums). Hend [4] classifies European plum into four groups: *Prunus domestica* L., *P. cerasifera* Erth., *P. spinosa* L. and *P. insititia*. In the Czech Republic, the most common type of plum are dark blue to indigo-coloured European – Asian plums belonging to *P. domestica* L., which are not differentiated and are all called "švestka" in Czech. They all have oval shape, yellow-green flesh and dark blue to indigo-coloured skin. Other plums have oval or round shape and purple, yellow or red skin. Plums are consumed fresh and are also used for home and industrial production of various traditional products, which are typical for Central and Eastern Europe [5]. The most important plum products are dried plums, plum brandy, plum paste and plum jam [6]. Plum brandy ("slivovice" in Czech) is probably the most popular plum product followed by plum paste (called "povidla" in the Czech Republic, "lekvár" in Slovakia and Hungary, and "das Powidl" or "Pflaumenmuss" in Austria and Germany). In English-speaking countries, plum paste can be called plum butter, plum jam or high fruit content jam. Plum paste is widely used in Central and Eastern Europe where it is used as a base for preparation of a wide range of

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traditional dishes and cakes, such as various sweet and sour sauces for meat dishes or cake fillings. Plum paste is traditionally produced by long term boiling of plums without any other additives, until the final product is concentrated to such an extent as to be stable during storage at ambient temperature. Its low water activity and high acidity are sufficient barriers against microbial spoilage. The product has distinctive characteristic sensory properties, such as dark colour (brownish black), tough and glossy texture and specific pure fruit taste. The brownish black colour of this product is due to intensive Maillard reactions. Nowadays, plum paste is produced industrially, using various additives (sugar/saccharose, citric acid). The volume of plum production in Central Europe has decreased during the last decades due to sharka viral disease caused by plum pox virus [7]. The plum paste has therefore also been produced from imported plums, usually dried or in the form of various intermediate products. Very often, other plum varieties and other types of *Prunus* fruit different than *Prunus domestica* are used as well. The shortage of plums is sometimes overcome by adulteration of plum paste by non-declared addition of apples or apple intermediate products. Even though plum paste producers are required to indicate its fruit content on the label, it can still be difficult to authenticate such products. Commonly used markers such as formol number, saccharide profiles or composition of acids, are affected by the long-term boiling and by the fact that various stone fruits other than plums may be added in the product, and these may differ in the contents of authenticity markers. The available sources of chemometric data are inconsistent because of unclear classification of various plum varieties. Comprehensive review of authenticity of fruit products is available [8]. Our study focuses on chemical composition and quality of plum paste in order to fill in missing data in food composition tables and to facilitate authentication of plum products.

MATERIALS AND METHODS

Samples

Twenty-one plum pastes were analysed. All samples were purchased at the Czech market or directly from the Czech producers Nova Sobotka (Sobotka, Czech Republic) and Hamé (Kunovice, Czech Republic). The declared contents of plums (grams of plums used per 100 g of final product) in the plum paste samples were P18: 140 g, P5: 160 g, P19: 165 g, P9, P13: 180 g, other samples: 170 g. All samples were declared to be made from plums as the only fruit. In all samples, sugar addition was declared. All samples were produced in 2010 or 2011. The country of origin of the samples was Czech Republic, Poland or Germany.

Methods

Soluble solids (expressed in degrees Brix) were determined using AOAC Official Method 970.59 [9]. Ash was determined by AOAC Official Method 940.26 [10]. Phosphorus was determined by AOAC Official Method 995.11 Phosphorus (total) in food [11]. Potassium, sodium, magnesium and calcium were determined by isotachophoresis according to Kvasnicka [12]. Titratable acidity was determined by AOAC Official Method 942.15 [13]. Total amino acid concentration (formol number) was determined according to the EN 1133:1994 [14]. Saccharose, glucose, fructose and sorbitol were determined according to Opatova [15]. 5-Hydroxymethylfurfural (5-HMF) determination was performed following the method described by Hidalgo [16]. Colour determination was performed in the CIE $L^*a^*b^*$ (L^* lightness, a^* redness, b^* yellowness) colour space following the method described by Rajchl [17]. Colour measurements were carried out using a Minolta CM-2600d spectrophotometer (Minolta, Osaka, Japan). Measurements were carried out in the specular component included (SCI) mode using a 10° standard observer and illuminant D65. Before analysis, the instrument was calibrated on a white standard tile ($L^* = 98.82$; $a^* = -0.18$; $b^* = -0.31$).

Determination of phloridzin and rutin

Phloridzin (phloridzin dihydrate, 99%) and rutin (trihydrate, > 95%) standards were purchased from Sigma-Aldrich (Prague, Czech Republic). Phosphoric acid was purchased from Lachema (Brno, Czech Republic), acetonitrile and methanol from Merck (Prague, Czech Republic). Demineralized water (Milli-Q quality; Millipore, Prague, Czech Republic) was used for preparation of a buffer, the mobile phase, standard solutions and for extraction of samples. HPLC analyses were performed on the Dionex HPLC instrument including a P680 pump, the Ultimate 3000 photodiode array detector and ASI 100 autosampler controlled by Chromeleon 6.80 software package (Dionex, Camberley, United Kingdom). Chromatographic separation of phloridzin was carried out on Eclipse XBD C8 column (150 × 4.6 mm, 5 μm; Agilent Technologies, Santa Clara, California, USA). The column temperature was kept at 30 °C. The mobile phase consisted of 0.1% phosphoric acid in water (solvent A) and 0.1% phos-

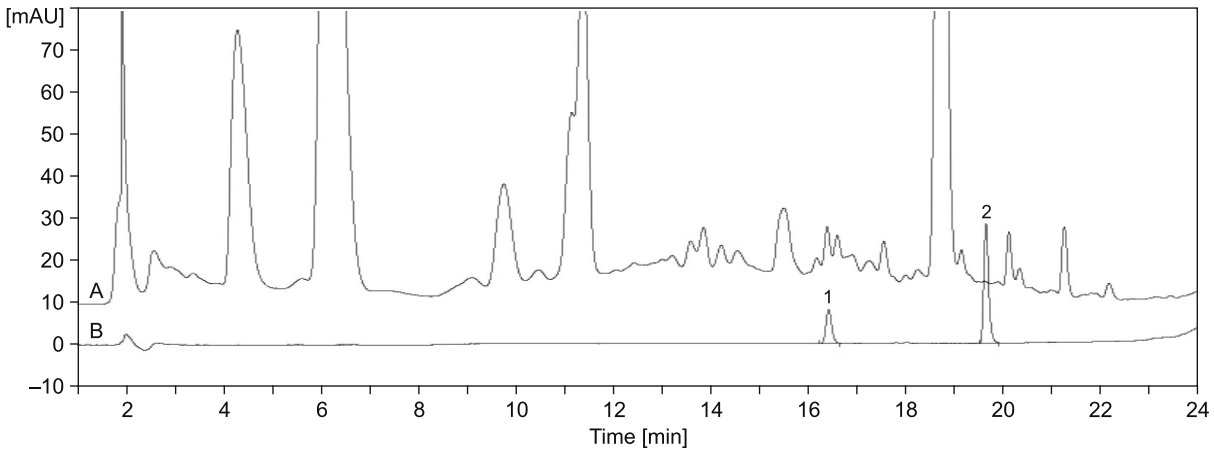

Fig. 1. Chromatograms of the plum paste extract and standard.

A – plum paste extract, B – standard.
Retention times: 1 – rutin, 16.425 min; 2 – phloridzin, 19.658 min.

phoric acid in acetonitrile (solvent B). A linear gradient profile of the mobile phase was applied at a flow rate of 1 ml·min^{-1}. The elution conditions were: 0–5 min, 5% B isocratic; 5–15 min, linear gradient 5–20% B; 15–20 min, linear gradient 20–40% B; 20–28 min, linear gradient 40–80% B; 28–35 min, 5% B isocratic. The detection was carried out at wavelengths of 254, 280, 328 and 350 nm. One analysis took 35 min. The quantitative analysis was based on the external standard method. A homogenized sample (5 g) was weighed into a 50 ml volumetric flask and 80% methanol (45 ml) was added. The sample was extracted in ultrasonic bath at 25 °C for 1 h. Subsequently, the content was cooled down to 20 °C and made up to volume with 80% methanol. The sample was filteres through a membrane filter (pore size 0.45 μm) and the filtrate was analysed for phloridzin and rutin contents. The basic parameters of HPLC assay were, for phloridzin, linearity: 0–100 mg·l^{-1}, repeatability: 3.24% (n = 7, concentration level 50 mg·l^{-1}), recovery: 96.74% (concentration level 8 mg·l^{-1}), limit of detection: 0.38 mg·kg^{-1}, limit of quantification 0.190 mg·kg^{-1} and, for rutin, linearity: 0–100 mg·l^{-1}, repeatability: 2.24% (n = 7, concentration level 50 mg·l^{-1}), recovery: 96.31% (concentration level 8 mg·l^{-1}), limit of detection: 0.043 mg·kg^{-1}, limit of quantification 0.215 mg·kg^{-1}. The chromatograms of plum paste extract and standards are presented in Fig. 1.

Determination of organic acids

Shikimic acid (> 99%) and potassium phosphate monobasic (KH_2PO_4) were purchased from Sigma-Aldrich, malic acid (DL, > 99%) was purchased from Carl Roth (Karlsruhe, Germany) and citric acid (monohydrate, > 99,9%) was purchased from Lachner (Neratovice, Czech Republic). Phosphoric acid was purchased from Lachema. Demineralized water (Milli-Q quality) was used for preparation of a buffer, for the mobile phase, standard solutions and for extraction of samples. Chromatographic separation of organic acids was carried out on Phenomenex Luna C18(2), particle size 5 μm, pore size 100 Å, 250 × 4.6 mm and Phenomenex Luna C18(2), particle size 10 μm, pore size 100 Å, 250 × 4.6 mm (Phenomenex; Torrance, California, USA). The column temperature was kept at 30 °C. The mobile phase consisted of 0.02 mol·l^{-1} potassium phosphate monobasic (KH_2PO_4) in water, the pH being equilibrated on 2.5 by phosphoric acid. The detection was carried out at wavelengths of 210, 254 and 280 nm. One analysis took 30 minutes. The quantitative analysis was based on the external standard method. A homogenised sample (5 g) was weighted into a 100-ml volumetric flask and demineralised water (80 ml) was added. The sample was extracted in ultrasonic bath at 25 °C for ten minutes. Then the content was cooled down to 20 °C and made up to volume with demineralized water. The filtrate (0.45 μm filter) was analysed for organic acids content. The basic parameters of HPLC assay were determined. The parameters of HPLC assay were: malic acid: linearity: 0–1 500 mg·l^{-1}, repeatability: 7.43% (n = 5, concentration level 250 mg·l^{-1}), recovery: 95% (concentration level 250 mg·l^{-1}), limit of detection: 5.53 mg·kg^{-1} and limit of quantification was 27.65 mg·kg^{-1}, shikimic acid: linearity: 0–1 100 mg·l^{-1}, repeatability: 2.29% (n = 5,

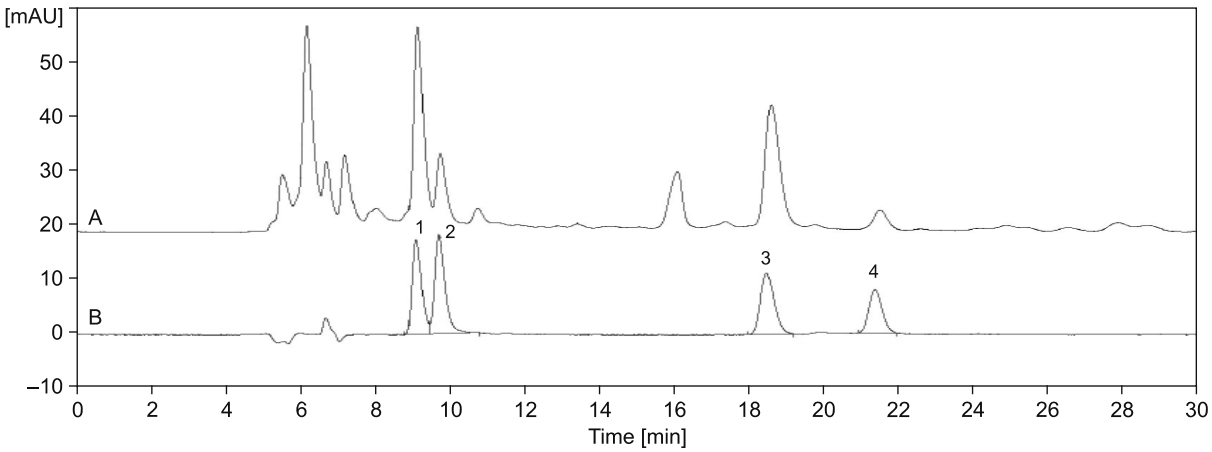

Fig. 2. Chromatograms of the plum paste extract and standard.

A – plum paste extract, B – standard.
Retention times: 1 – malic acid, 9.087 min; 2 – shikimic acid, 9.691 min; 3 – citric acid, 18.459 min; 4 – the peak with retention time 21.355 min in the standard chromatogram is fumaric acid, an impurity in the malic acid standard.

concentration level 250 mg·l^{-1}), recovery: 97% (concentration level 250 mg·l^{-1}), limit of detection: 0.55 mg·kg^{-1} and limit of quantification was 2.75 mg·kg^{-1}, citric acid: linearity: 0–1 100 mg·l^{-1}, repeatability: 4.43% (n = 5, concentration level 250 mg·l^{-1}), recovery: 99% (concentration level 250 mg·l^{-1}), limit of detection: 14.96 mg·kg^{-1} and limit of quantification was 74.80 mg·kg^{-1}. The chromatograms of plum paste extract and standards are given in Fig. 2.

Statistical analysis

The tests were carried out in triplicate for each sample and mean values ± standard deviation (SD) are reported. Differences at $p < 0.05$ were considered to be significant. All statistical analyses were performed using Statistica 8.0 (StatSoft CR; Prague, Czech Republic) and Excel 2010 (Microsoft; Prague, Czech Republic).

RESULTS AND DISCUSSION

Summarized results for all the samples and the selected samples that were not suspected of adulteration are given in Tab. 1. It is shown that plum paste is a rich source of saccharides (saccharose, glucose, fructose) and sorbitol. High content of soluble solids is mainly caused by sugar being added, but also by high level of thickening of the plum paste. Citric acid and malic acid are present in plum paste in relatively high contents, which roughly correspond to titratable acidity. Citric acid is added to plum paste during production as an acidulant and therefore the content of citric acid in plum paste is higher than would correspond to plum fruit. Formol number indicates the contents of free amino acids in the product. This parameter is commonly used as one of the markers for assessing authenticity of fruit and vegetable products.

Tab. 1 shows that plum paste is a nutritionally rich source of minerals (in particular potassium). Colour measurements indicate that plum paste is very dark (parameter L ranged from 25.51 to 28.22), which is due to enzymatic oxidation by polyphenol oxidase and due to the content of Maillard reaction products. Anthocyanins (present mostly in plum skins) are greatly degraded by heat treatment [18, 19]. Interpretation of parameters a^* and b^* is difficult. The 5-HMF content and L parameter can be used as markers for evaluating the technology used. As long as plum paste is traditionally produced by long-term boiling in an open pot, these parameters are expected to correlate with the duration of boiling. The contents of 5-HMF in the samples of plum paste were not lower than 19.5 mg·kg^{-1} (mean 194.3 mg·kg^{-1}) and parameter L not higher than 28.22 (mean 26.74).

Principal component analysis was applied to the data matrix. Fig. 3 shows the 2-D scatterplot obtained for the first and the second principal components of the plum paste products. These two principal components accounted for 60% of the total variance. The set of samples could be divided into 3 groups (grey ellipses). The samples suspected of adulteration (P18, P9, P3 and P4) were roughly separated from other samples (circles). Fig. 3 demonstrates that the samples con-

taining phloridzin (adulterated samples) were difficult to recognize by measurement of many standard markers. Phloridzin is the only robust marker for estimation of apple content in fruit products (such as fruit spreads, jams).

The plot of weights of components (the orthogonal projection of variables in a two principal components space – correlation circle) is shown in Fig. 4. The distribution of the variables in Fig. 4 does not allow identification of the main components. The graph shows the correlation between markers, meaning that when the variables are closer together, the correlation is stronger.

Saccharides and citric acid are added to jam as additives and thus, together with the titration acidity and soluble solids, their contents are affected by the recipe. A significant positive correlation was identified between ash and the contents of potassium and phosphorus. A weak correlation was identified between ash and the contents of calcium, magnesium and sodium. Apparent correlation was also identified between the colour and the content of phloridzin, which supports the assumption that addition of apples to plum paste may affect its colour as apples brown very quickly due to enzymatic oxidation.

Plum paste samples P3, P9, P18 and P21 contained phloridzin, which indicates that they were adulterated with apples. The content of phloridzin at a level of 1 mg·kg^{-1} corresponds approximately to the addition of 30 g of apples to a kilogram of products. According to our calculations, P3 sample contained 417 g·kg^{-1} of apples, P9 contained 483 g·kg^{-1} of apples,

Tab. 1. Chemical composition of plum paste samples.

Marker	Unit	All samples					Unadulterated samples (P3, P4, P9, P18, P21 excluded from the list)				
		Minimum	Maximum	Mean	Median	SD	Minimum	Maximum	Mean	Median	SD
Soluble solids*	[°Bx]	49.3	66.3	58.7	59.6	3.8	49.3	66.3	59.3	60.3	3.5
Titratable acidity*	[g·kg^{-1}]	9.32	18.61	13.07	12.54	2.61	9.32	18.39	13.03	12.63	2.53
Formol number	[ml·kg^{-1}]	95	294	193	192	49	95	259	194	192	43
Saccharose*	[g·kg^{-1}]	47.6	265.2	103.7	81.5	54.2	48.2	265.2	108.7	81.2	59.2
Glucose*	[g·kg^{-1}]	138.8	254.5	204.0	207.4	25.0	138.8	254.6	202.2	206.6	26.2
Fructose*	[g·kg^{-1}]	90.9	246.1	191.1	217.4	43.1	90.9	246.1	188.8	217.4	46.6
Sorbitol	[g·kg^{-1}]	3.0	43.8	25.1	22.2	11.9	16.8	43.8	29.1	30.5	9.2
Ash	[g·kg^{-1}]	2.98	9.26	6.50	6.65	1.13	5.91	9.26	6.80	6.75	0.76
Phosphorus	[mg·kg^{-1}]	129.4	327.2	251.5	268.2	50.3	208.7	327.2	266.8	280.2	37.2
Potassium	[mg·kg^{-1}]	1232	4299	2798	2713	597	2393	4299	2914	2746	501
Sodium	[mg·kg^{-1}]	21.4	83.6	52.9	53.5	20.3	21.4	83.6	57.0	59.8	18.1
Magnesium	[mg·kg^{-1}]	90.7	242.2	171.4	174.8	33.8	90.7	242.2	173.1	177.1	33.4
Calcium	[mg·kg^{-1}]	3.7	74.5	35.3	32.4	18.3	3.7	74.5	36.9	36.3	18.7
Malic acid	[g·kg^{-1}]	1.70	15.89	7.26	8.42	4.70	1.70	15.89	6.91	5.74	4.75
Citric acid*	[g·kg^{-1}]	1.16	9.53	5.05	5.00	2.43	1.16	9.53	4.95	4.66	2.17
Shikimic acid	[mg·kg^{-1}]	26.4	77.7	63.4	68.0	13.1	53.5	77.7	67.5	69.3	7.3
5-hydroxymethylfurfural	[mg·kg^{-1}]	19.5	385.9	194.3	212.5	83.5	19.5	341.0	195.8	213.6	73.2
Phloridzin	[mg·kg^{-1}]	n.d.	16.1	2.4	0.0	5.0	n.d.	n.d.	n.d.	n.d.	n.d.
Rutin	[mg·kg^{-1}]	n.d.	83.7	33.4	27.7	27.6	n.d.	83.7	34.7	36.7	30.3
Colour *L**		25.51	28.22	26.74	26.68	0.82	25.57	27.76	26.63	26.43	0.76
Colour *a**		1.06	5.37	2.52	2.09	1.07	1.40	5.37	2.40	2.01	0.95
Colour *b**		0.68	3.27	1.81	1.62	0.73	0.77	2.95	1.68	1.62	0.56

SD – standard deviation, n.d. – not detected.
Titratable acidity is expressed in grams of malic acid. Formol number is expressed in millilitres of 0.1 mol·l^{-1} NaOH per kilogram. The markers signed by * are affected by the used recipe.

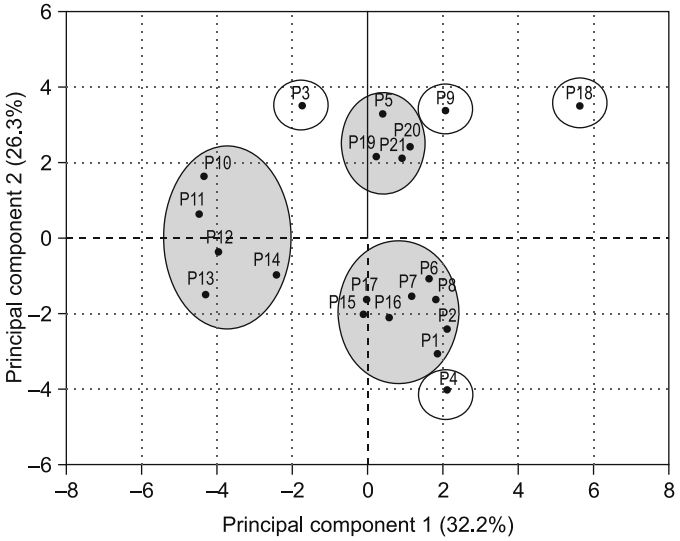

Fig. 3. Principal component analysis – 2-D scatterplot obtained for the first and the second principal components of the plum pastes.

The set of samples is divided into 3 groups (grey ellipses). The samples suspected of adulteration (P18, P9, P3 and P4) are marked by circles.

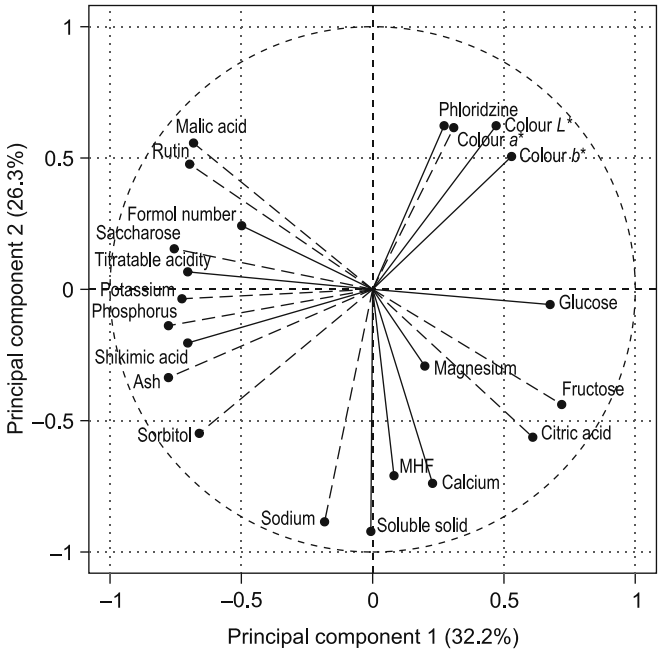

Fig. 4. Principal components analysis – plot of the weights of components, graphical representation of the structure and correlation of variables (measured analytes).

P18 306 g·kg^{-1} of apples and P21 285 g·kg^{-1} of apples. P4 plum paste sample was suspected of adulteration due to a significantly lower content of ash, as well as due to formol number and phosphorus content that did not correspond to the data published for plums [20]. A significantly lower content of sorbitol, which is a characteristic compound in plums, was observed in P3 and P9 samples. For these reasons, plum paste samples P3, P4, P9, P18 and P21 were excluded from the list of samples, and average values of the remaining samples (unadulterated) are reported in the right column of Tab. 1. The results in Tab. 1 show that exclusion of the suspected samples significantly decreased variability (expressed as standard deviation).

CONCLUSION

The plum paste is a nutritionally rich source of saccharides, organic acids and minerals (in particular potassium). From a nutritional point of view, plum paste contains a very advantageous ratio of sodium to potassium (K/Na = 49.3). On the other hand, plum paste contains relatively high amounts of a potentially toxic saccharide degradation product 5-HMF due to the extensive thermal treatment. Due to the high saccharide content, plum paste is a high energy food and consumption should be included into the calculation of personal daily energy intake [21]. Addition of apples to plum paste was detected in four samples, which lacked information on content of apples on the label.

Acknowledgements

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