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ORIGINAL ARTICLE

Plum pomaces as a potential source of dietary fibre: composition and antioxidant properties

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Abstract Plums because of their composition, especially of dietary fibre, sorbitol and polyphenols content, have positive influence on human health. Generally growing interest in cloudy juices production due to their prevalence compared to clear ones resulted in the appearance of plum cloudy juices on the market. Cloudy plum juice may be the attractive plum product, however during juice production some pomace appears, which is discarded or used for feeding animals most of all. This by product might be a source of valuable health-promoting compounds. The aim of this work was to characterize the composition and properties of pomaces of three cultivars obtained in pilot plant scale. The influence of drying parameters and cultivar on bioactive components and antioxidant activity were measured. Plum pomaces were characterized by 38–49% of total dietary fibre in d. m., with the share of soluble fraction from 7 to 13%. Energy value was from 202 to 240 kcal 100 g^{-1} d. m. Antioxidant activity was from 10 to 17.4 mikroM TEAC g^{-1} d.m. Cultivar and technology of drying had significant influence on polyphenols content of investigated plum pomaces. Considering their health-beneficial components: dietary fiber and polyphenols, plum pomaces can be used for production of dietary fiber preparations.

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Introduction

High content of dietary fibre and antioxidants makes plums an important component of a healthy diet. The taste of the fruits depends on cultivar and also on climatic and soil conditions. Although Poland is an important producer of plums, with 114 000 t in 2008 (STAT 2008), the intake of fresh fruits of plums with the diet is relatively low, because of short, limited to harvest season market availability. Plum fruits are processed into compotes, jams, marmalades, prunes, juices and alcohol beverages. However, drying is the oldest way of plum processing (Walkowiak-Tomczak et al. 2008). Dried prunes due to high dietary fibre, especially high pectin content give a quick feeling of satiety. Mild laxative effect, decrease of blood cholesterol and an ability to decrease osteoporosis during menopause are also attributed to dried prunes (Lucas et al. 2004). Plums are characterized by high amount of sugars with glucose consisting nearly 50%, then fructose, saccharose, and substantial content of sorbitol in some cultivars. They contain only small amounts of proteins and fats. Malic and citric are the main organic acids (Walkowiak-Tomczak et al. 2007; Usenik et al. 2009). Plums are a rich source of polyphenols, there are most of all flavonols, and caffeic acid derivatives: neochlorogenic acid, chlorogenic acid, cryptochlorogenic acid as well as anthocyans and flavonols (Balasundram et al. 2006; Chun et al. 2003; Dimitrios 2006; Nakatani et al. 2000; Stacewicz-Sapuntzakis et al. 2001; Tomas-Barberan et al. 2001; Walkowiak-Tomczak et al. 2008). The content of anthocyans depends on cultivar and is caused by size differences of fruits and the ratio of peel mass to flesh mass.

Polyphenols, anthocyans as well as antioxidant activity depends on cultivar, maturity as well as climatic and soil conditions (Kim et al. 2003a, b). High activity of polyphenol oxidase results in fast enzymatic browning of plum phenolic compounds during processing and influences the formation of favorable sensory quality of dried fruits (Łoś et al. 2000).

Dietary fibre, especially its soluble fraction (SDF) appears to have health-beneficial properties, like selectively lowering serum LDL cholesterol, improving glucose tolerance and promoting Bifidobacteria in gut (Das et al. 2011; Ahmed et al. 2011). Pomace a by-product of juice processing contains high quantity of dietary fibre, up to 64.5% in case of plum pomace, moreover considerable amounts of polyphenols thus should no longer be considered as a waste product but rather as a raw material for a production of diet supplements (Schieber et al. 2003; ADA 2008; Dikenam et al. 2004; Ramulu and Rao 2003; Lucas et al. 2004). However, the literature still lacks the data on plum pomace polyphenols composition.

The aim of the work was the extension of knowledge on composition of plum pomaces as a source of dietary fibre and phenolic components. The plum pomaces were obtained with ascorbic acid addition used in the process of juice production. The influence of drying process, air-drying or freeze-drying, was investigated as well.

Materials and methods

Materials

Fruits of plum (*Prunus x domestica* L.) of three cultivars 'Promis', 'Najbolja' and 'Dąbrowicka' were collected at picking maturity at Institute of Horticulture (IH), Skierniewice, Poland at 2008 season to be used in pilot plant scale trials carried out at IH according to technology developed within ISAFRUIT Project (Fastyn et al. 2010). Three plum cultivars: 'Dabrowicka', 'Promis' and 'Najbolia' were processed into cloudy juices. 500 mg kg^{-1} dose of ascorbic acid was used to overcome browning problems of the mash and juice during production. 10 kg of fruits were washed, then frozen and stored at −25 °C until processing. After equilibrating plums temperature to ca −7 °C they were pitted, heated up to 50 °C and disintegrated using perforated disc mill (BASIS 91/55, Fryma-Maschinen AG, Rheinfelden, Switzerland). Ascorbic acid in quantity 500 mg kg^{-1} was added continuously to the mash during grinding. The mash was depectinised using enzymes combination of pectin lyase and polygalacturonase in the dose of 100 g/t in proportion 2:1 Rohament PTE and Rohapect PL (AB Enzymes GmbH, Darmstadt Germany). Depectinisation was carried out at 50 °C for 1 h. After enzymatic treatment mash was pressed on hydraulic pack press (Bucher, Niederweningen, Switzerland). One part of the obtained press-cake was freeze-dried (Laboratory Freeze Dryer, Martin Christ Gefriertrocknungsanlagen GmbH, type 102241, Osterode AM Harz, Germany) and the other part was air-dried in a laboratory dryer (1.6 kW KC-100/200, WAMiE, Warszawa) at temperature below 70 °C. Dried material was grinded in IKA A11 basic grinder (IKA, Werke GmbH, StaufenGermany) and submitted to analysis. Yield of dried pomaces was 3–4 g on 100 g of fresh stoned fruits (fruits contained 8–10% of stones).

Analytical methods

Dry matter—AOAC 920.151; Ash in Fruits—AOAC 940.26, Fat (Ether Extract of Plants)—AOAC 930.09; Protein in Fruit products—AOAC 920.152; Acidity as citric acid-AOAC 942.15, Total Dietary Fiber in Foods (enzymatic gravimetric method) according to principle of AOAC Official Method 985.29; Insoluble Dietary Fibre in Food and Food products (enzymatic gravimetric method, phosphate buffer) according to principle of AOAC Official Method 991.42; Soluble Dietary Fibre in Food and Food products—was calculated as follows: SDF% = TDF − IDF (according to AOAC 985.29) (all AOAC methods according to AOAC 2005). Extraction of polyphenols was performed as previously described (Kołodziejczyk et al. 2007): 0.5 g of each sample was mixed with 4 ml of solvent [70% (v/v) methanol] and sonicated for 15 min. After centrifugation at 4,800*g* for 5 min, the supernatant was collected and the residue was re-extracted twice with 3 ml of the same solvent. The pooled extracts were made up to 10 ml. The methanolic extracts, containing most of the soluble polyphenols were analysed by HPLC. Extraction pattern was repeated on residue with 70% acetone for extraction of polymeric proanthocyanidins. Both extractions (methanol and acetone) were made in triplicates. Polyphenols HPLC analyses were carried out using a Dionex HPLC system with a Diode Array Detector (UVD340U, Dionex, Germering, Germany) equipped with Gemini 5 μC18 110A 250×4.6 mm (Phenomex, Torrance, CA, USA). The mobile phase consisted of 0.05% phosphoric acid (solvent A) and 0.05% phosphoric acid in acetonitrile (solvent B). A gradient was applied at flow rate of 1.25 ml min^{-1}. After stabilization for 5 min with 4% of B (v/v), a gradient of 4–15% of B from 0 to 12.5 min, then a gradient 15–40% of B from 12.5 to 42.4 min, then a gradient 40–50% of B from 42.4 to 51.8 min, then isocratic elution at 50% of B from 51.8 to 53.4 min, then gradient of 50–4% of B from 53.4 to 55.4 min was applied. Column temperature was kept at 25 °C. Chlorogenic acid, hyperoside, rutin and cyanidin glucoside (all from Extrasynthese, Genay, France) were used as standards to calculate the concentration of hydroxycinnamic acids, quercetin glycosides (excl. rutinoside), quercetin rutinoside and anthocyanins, respectively. Sum of proanthocyanidins were calculated as sum of results of vanillin test for methanol and acetone extracts. Sum of proanthocyanidins was measured according to method described by Nakamura et al. (2003). Antioxidant activity was measured by DPPH method and calculated as Trolox equivalent (TEAC) (Kim et al. 2002). Composition of sugars was determined by HPLC as described previously (Kołodziejczyk et al. 2009).

Statistical analysis

The effect of cultivar and type of drying on the content of the particular component was determined using two-way analysis of variance ANOVA, and significant differences between results were determined by Duncan's multiple range test. The differences were considered significant at $p<0.05$. Calculations were done using Statistica program ver.7 (StatSoft, Tulsa, USA).

Results and discussion

Nutritive and energy values of plum pomaces

Composition of air-dried or freeze-dried plum pomaces of three cultivars: 'Cacanska Najbolja', 'Dąbrowicka' and 'Promis' obtained in pilot plant scale is presented in Table 1. The content of TDF in dried plum pomaces from pilot plant was from 38 to 49% d. m., depending on cultivar with 'Dąbrowicka' and 'Najbolja' with the highest values. The content of TDF in apple pomaces, used the most often for production of dietary fibre preparations, obtained in similar conditions varied from 30 to 60% depending on cultivar (Kołodziejczyk et al. 2009), while data on dietary fibre content in industrial apple pomace is usually above 60% (Schieber et al. 2003). Mean SDF/TDF ratio in obtained pomaces was ca. 20% and as high share of soluble fibre is responsible for the decrease of cholesterol in blood (Lucas et al. 2004; Sierra et al. 2002) the most probably plum pomaces would have also similar influence on human digestive tract and plasma parameters. The content of available carbohydrates in these pomaces was 40–53%, the energetic value was from 208 to 246 kcal 100 g^{-1} d.m.

Significant differences in soluble dietary fibre and protein between pomaces obtained by different drying ways were found in two way ANOVA analysis (Table 1). Depending on drying process air-drying versus freeze-drying pomaces air-dried had a decreased level of soluble dietary fibre and increased of protein. In freeze-dried pomaces the contribution of soluble fibre fraction (SDF/TDF ratio) is significantly higher

Table 1 Nutritive and energy values of plum pomaces obtained from 'Dąbrowicka', 'Cacanska Najbolja', and 'Promis' cultivars submitted to air- or freeze-drying

		'Dąbrowicka'				'Cacanska Najbolja'				'Promis'				Cultivar	Drying	CxD
		AD±SD		FD±SD		AD±SD		FD±SD		AD±SD		FD±SD				
Protein	[%/d.m.]	8.2^{a}	0.2	6.3^{c}	0.2	6.8^{b}	0.2	6.6^{bc}	0.1	6.8^{b}	0.1	3.9^{d}	0.3	<0.05	<0.05	<0.05
Fat	[%/d.m.]	2.4^{b}	0.0	2.7^{a}	0.0	2.1^{c}	0.0	2.1^{c}	0.0	2.0^{d}	0.0	2.4^{b}	0.0	<0.05	<0.05	<0.05
Ash	[%/d.m.]	2.0^{c}	0.0	2.2^{b}	0.0	1.7^{d}	0.0	1.7^{d}	0.0	2.1^{bc}	0.0	2.5^{a}	0.0	<0.05	<0.05	<0.05
TDF	[%/d.m.]	46.0^{b}	0.7	49.3^{a}	0.1	47.9^{a}	1.0	47.7^{ab}	0.4	39.8^{c}	1.1	38.2^{c}	0.6	<0.05	0.27	<0.05
IDF	[%/d.m.]	36.1^{c}	0.5	36.2^{c}	0.4	39.3^{a}	0.5	37.8^{b}	0.2	32.2^{d}	0.8	31.8^{d}	0.3	<0.05	0.07	0.12
SDF	[%/d.m.]	9.9^{b}	0.3	13.1^{a}	0.3	8.6^{cd}	1.0	9.8^{bc}	0.2	7.6^{de}	0.5	6.5^{e}	0.3	<0.05	<0.05	<0.05
Energy value	[kcal 100 g^{-1} d.m.]	215.1		202.2		208.9		215.9		239.3		243.2				

AD air-dried, *FD* freeze-dried, *TDF* total dietary fibre, *IDF* insoluble dietary fibre, *SDF* soluble dietary fibre, *C* cultivar, *D* drying, values in rows marked with different letters are different at $\alpha \leq 0.05$, $n=3$

comparing to air-dried pomace. 'Promis' cultivar was characterized by the lowest TDF content.

Carbohydrates composition of plum pomaces

Plum pomaces were characterized by high content of glucose and fructose as well as relatively high content of sorbitol (Table 2). Analyzed pomaces contained from 16 to 22% of saccharides. Glucose stands up to 50%, fructose 20–40%, and sorbitol ca. 10% in d.m. of all sugars. High content of sorbitol in plum fruits is also attributed to mild laxative effect (Stacewicz-Sapuntzakis et al. 2001). As it was found in previous research (Fastyn et al. 2010) fruits of 'Promis' cultivar contained sorbitol on high level and in sensory analysis of juices 'Promis' cultivar had too violent laxative effect and therefore the cultivar should be excluded from a single variety juice production. Acidity of pomaces was from 1.4 to 5.3 g 100 g^{-1}. Pomace of 'Dąbrowicka' cultivar had the highest acidity among investigated cultivars.

Significant differences in saccharose, glucose, sorbitol and acids contents between pomaces obtained by different drying ways were found in two way ANOVA analysis (Table 2). Comparing air-drying versus freeze-drying, air-dried pomaces had a decreased level of saccharose caused by its hydrolysis and the leak of soluble substances, which occurs during drying at high temperatures. The content of sorbitol was higher with maturity in the following order: 'Dąbrowicka', 'Najbolia' and 'Promis'.

Characterization of polyphenols composition in plum pomaces

Sum of polyphenols calculated as total HPLC-determined polyphenols and procyanidins determined by vanillin test in plum pomaces was from 243 to 1,810 mg 100 g^{-1}. Pomaces of 'Najbojla' and 'Dąbrowicka' had similar quantities of total polyphenols (derivatives of hydroxycinnamic acids, quercetin glycosides and anthocyanins determined by HPLC) (Table 3). The poorest in above polyphenols were pomaces of 'Promis' cultivar. However, 'Promis' and 'Najbojla' cultivars were richer in proanthocyanidins. Calculated as above sum of polyphenols in 'Najbolia' and 'Promis' gives 2–3 times higher value, comparing to 'Dąbrowicka' cultivar. Two way ANOVA shows that all groups of polyphenols varied both with cultivar and a way of drying, except quercetin glycosides, which were stable both in freeze- and air-dryied pomaces.

In current literature there is little data on polyphenol composition of plum pomaces. The only available data shows the content and composition of polyphenols in plum fruits. In dried prunes 245 mg 100 g^{-1} in d.m., while in fresh plum fruits 56.3–259.3 mg 100 g^{-1} fresh matter (281.5–

Table 2 Carbohydrates composition of plum pomaces obtained from 'Dąbrowicka', 'Cacanska Najbolja', and 'Promis' cultivars submited to air- or freeze-drying

		'Dąbrowicka'				'Cacanska Najbolja'				'Promis'				Cultivar	Drying	CxD
		AD±SD		FD±SD		AD±SD		FD±SD		AD±SD		FD±SD				
Saccharose	[%/d.m.]	1.3c	0.2	3.0a	0.2	0.2d	0.0	1.9b	0.0	0.1d	0.0	3.0a	0.2	<0.05	<0.05	<0.05
Glucose	[%/d.m.]	8.3d	0.6	10.8c	0.3	9.1d	0.7	13.8a	0.4	6.3e	0.7	12.0b	0.1	<0.05	<0.05	<0.05
Fructose	[%/d.m.]	6.2ab	0.3	8.5b	0.2	4.5a	0.8	7.2ab	0.4	5.2a	0.3	5.6ab	0.3	<0.05	<0.05	<0.05
Sorbitol	[%/d.m.]	1.4c	0.0	1.2c	0.0	1.7c	0.1	2.1c	0.8	4.3b	0.0	6.7a	0.3	<0.05	<0.05	<0.05
Acidity[a]	[%/d.m.]	4.9b	0.1	5.3a	0.2	3.2c	0.0	3.1c	0.1	3.1c	0.1	2.4d	0.1	<0.05	0.08	<0.05

[a] Calculated as citric acid, *AD* air-dried, *FD* freeze-dried, *C* cultivar, *D* drying, values in rows marked with different letters are different at $\alpha \leq 0.05$, $n=3$

Table 3 Composition of polyphenols in plum pomaces obtained from 'Dąbrowicka', 'Cacanska Najbolja', and 'Promis' cultivars submitted to air- or freeze-drying

		'Dąbrowicka'				'Cacanska Najbolja'				'Promis'				Cultivar	Drying	CxD
		AD±SD		FD±SD		AD±SD		FD±SD		AD±SD		FD±SD				
HCA	[mg 100 g^{-1}]	7.7^{d}	0.5	16.5^{b}	1.1	10.8^{c}	0.1	31.2^{a}	0.2	16.0^{b}	0.6	9.5^{c}	0.9	<0.05	<0.05	<0.05
QG	[mg 100 g^{-1}]	21.8^{a}	1.0	21.7^{a}	0.6	19.6^{a}	1.3	19.7^{a}	0.9	12.6^{b}	1.3	11.9^{b}	0.3	<0.05	0.70	0.81
ANT	[mg 100 g^{-1}]	2.9^{c}	0.2	3.8^{bc}	0.5	4.1^{b}	0.6	12.9^{a}	0.6	4.1^{b}	0.1	4.8^{b}	0.2	<0.05	<0.05	<0.05
PO_{HPLC}	[mg 100 g^{-1}]	32.3^{c}	0.6	42.0^{b}	2.2	$34.5c^{d}$	1.8	63.8^{a}	0.5	32.7^{c}	0.7	25.7^{d}	0.3	<0.05	<0.05	<0.05
PRO_{VT}	[mg 100 g^{-1}]	210.8^{f}	14.1	644.2^{e}	5.6	1000.3^{c}	23.6	1512.8^{b}	10.7	886.0^{d}	5.4	1784.8^{a}	47.9	<0.05	<0.05	<0.05
$\Sigma\ PO_{HPLC} + PRO_{VT}$	[mg 100 g^{-1}]	243.1		686.2		1034.8		1576.6		918.7		1810.5		<0.05	<0.05	<0.05
Antioxidant activity	[mikroM TEAC g^{-1} d.m.].	9.9^{d}	0.5	10.0^{d}	0.7	13.7^{c}	1.0	17.4^{a}	0.1	15.2^{b}	0.1	15.5^{b}	0.7	<0.05	<0.05	<0.05

AD air-dried plum-pomace, *FD* freeze-dried plum pomace, *HCA* hydroxycinamic acids, *QG* quercetin glycosides, *ANT* anthocyans, PO_{HPLC} sum of polyphenols by HPLC, PRO_{VT} proanthocyanidins by vanilin test, $\Sigma\ PO_{HPLC} + PRO_{VT}$ sum of all polyphenols (by HPLC and by vanilin test), *C* cultivar, *D* drying, *TEAC* Trolox values in rows marked with different letters are different at $\alpha \leq 0.05$, $n=3$

1298.5 mg 100 g^{-1} in d.m. (Družić et al. 2007). The main groups of polyphenols were proanthocyanidins, hydroxycinamic acids, quercetin glycosides and anthocyanins. The same groups of polyphenols were previously found in plum fruits (Balasundram et al. 2006; Chun et al. 2003; Dimitrios 2006; Nakatani et al. 2000; Stacewicz-Sapuntzakis et al. 2001; Tomas-Barberan et al. 2001; Walkowiak-Tomczak et al. 2008; Cevallos-Casals et al. 2006; Donovan et al. 1998; Łoś et al. 2000; Usenik et al. 2009; Kim et al. 2003a, b).

Antioxidant capacity of plum pomaces

Antioxidant capacities of plum pomaces obtained in pilot plant scale were at the level of 9.9–17.4 mikroM TEAC g^{-1} d.m. with a mean value 13.9 mikroM TEAC g^{-1} d.m. (Table 3). The antioxidant capacity of plum pomaces depended on cultivar and a way of drying.

Mean antioxidant capacity of 'Dąbrowicka' cultivar pomace was statistically lower comparing to similar values for 'Promis' and 'Čačanska Najbolja' pomaces. The capacity of freeze-dried pomaces was statistically higher comparing to air-dried pomaces for 'Najbolja' cultivar. Reference data give the antioxidant capacity values of 84.0÷172.3 mikroM TEAC g^{-1} d.m. for fresh plums and 122.9÷151. mikroM TEAC g^{-1} d.m. for dried plums (Walkowiak-Tomczak et al. 2007, 2008). The values measured in analyzed pomaces were 10 times lower, what is linked with a transfer of most phenolics to juice during pressing. The antioxidant capacity was correlated with total polyphenol content (R=0.85). Comparing to previously measured antioxidant capacities of apples and black currants, plum and apple pomaces give similar values, while the value for black currant pomaces is 10 times higher (Sójka and Król 2009).

Conclusions

Plum pomaces obtained in pilot plant scale were characterized by 38–49% of total dietary fibre in d. m. with the share of soluble fraction from 7 to 13%. Considering the content of health-beneficial soluble fibre freeze-drying is preferred method, since air-drying reduces the share of soluble fraction. Cultivar and a manner of drying had significant influence on polyphenols content of investigated plum pomaces. Pomaces 'Dąbrowicka' and 'Najbolja' cultivars were characterized by the highest content of HPLC-determined polyphenols: 32–42, and 34–64 mg 100 g^{-1} d.m., respectively, while 'Najbolja' and 'Promis' by the highest content of proanthocyanidins: 1,000–1,513, and 886–1,785 mg 100 g^{-1} d.m, respectively. In case of polyphenols freeze-drying is preferred drying method as well, since it allows to preserve more heat-sensitive phenolics, however cheaper and more popular air-drying still makes it possible to obtain valuable product.

Looking at contents of dietary fibre and polyphenols freeze-dried pomace from ‘Cacanska Najbolia’ is most promising raw material.

Considering their health-beneficial components: dietary fiber and polyphenols, plum pomaces can be used for production of dietary fiber preparations with proven antioxidant activity.

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