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Fruit candies enriched with grape skin powders: physicochemical properties

Carola Cappa, Vera Lavelli, Manuela Mariotti*

Department of Food, Environmental and Nutritional Sciences (DeFENS), Università degli Studi di Milano, via G. Celoria 2, 20133 Milan, Italy

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ABSTRACT

This study investigated the effect of addition of grape skins (GS) on a fruit candy having a gel-like structure. GS from Barbera, a red grape (*Vitis vinifera* L.) variety, were processed into powders through milling and sieving to obtain three fractions having different particle sizes. Three fruit candy types added with GS fractions and a reference candy (without GS) were produced and analysed during the dehydration process, mainly in terms of moisture, soluble solids, water activity, polyphenol contents, ferric reducing antioxidant power, colour and texture. The fortification with GS powders increased the anthocyanin, flavonol and procyanidin contents of the candies, resulting in an increased antioxidant activity, which remained stable during processing. Furthermore, the fibre-enriched candies exhibited good textural properties. In general, the addition of GS promoted the reduction of the processing time, the replacement of a significant amount of fruit puree with a low-cost and high-nutritional winemaking by-product, as well as the delivery of beneficial compounds, thus highlighting the high potentialities associated with the use of GS in the confectionery industry.

1. Introduction

Due to the increasing interest in the design of new foods containing optimal levels of fibre and bioactive compounds, along with the attention to the sustainability of the technologies adopted, by-products recovery has become a growing challenge. In particular, in Italy, the winemaking industry covers an important part of the national food industry and its by-products, namely grape pomace (skin and seeds), have a significant environmental impact. Consequently, the development of new foods incorporated with grape skins – a natural product that combines the presence of large amounts of dietary fibre (DF) and antioxidants (Saura-Calixto, 1998) – represents an interesting recovery strategy.

It has been demonstrated that a high fibre intake is associated with body weight control and reduced risks of cardiovascular diseases (Slavin, 2005; Thompson, 2000). In addition, grape phenolics have been clearly identified as being responsible for protection towards oxidative stress (Vislocky, & Fernandez, 2010) and protein glycation (Sri Harsha, Gardana, Simonetti, Spigno, & Lavelli, 2013).

Some food applications of grape pomace skins and extracts have already been investigated: Sáyago-Ayerdi, Brenes, and Goñi (2009) evidenced that grape antioxidant dietary fibre significantly improved the oxidative stability of raw and cooked chicken hamburgers; and Mildner-Szkudlarz, Zawirska-Wojtasiak, Szwengiel, and Pacynski (2011), as well as Mildner-Szkudlarz, Bajerska, Zawirska-Wojtasiak, and Górecka (2013), suggested the use of grape pomace as an alternative source of dietary fibre and phenolics in rye bread and wheat biscuits, respectively. Tseng and Zhao (2013) found that wine grape pomace could be utilized as a source of antioxidant dietary fibre even in yogurt and salad dressing. Nevertheless, there is scarce knowledge on addition of grape pomace derived ingredients on products having a gel-like structure such as fruit candies.

Fruit candies (frequently identified by the French name "*gelées*") are very simple gel-like systems, made of fruits (at least 45 g/100 g), sugars (about 55 g/100 g), pectin (as gelling agent) and organic acids. These products are part of the confectionery market and they

Abbreviations: ANOVA, one-way analysis of variance; B, blue; Ch, chlorogenic acid; Cmix, concentrated mixture; Cy-glc, cyanidin-3-O-glucoside; Dp-glc, delphinidin-3-O-glucoside; F1, force necessary to punch the surface of the candies; F2, force necessary to punch the bottom of the candies; FRAP, ferric reducing antioxidant power; G, green; GS, grape skins; J, fruit candy; K, kaempferol; L, large grape skin fraction; LSD, Least Significant Differences; M, medium grape skin fraction; Mix, mixture before concentration; Mv-glc, malvidin-3-O-glucoside; Mv-*pc*-glc, malvidin-*p*-coumaroyl-3-O-glucoside; Ph, phlorizin; Q, quercetin; Pn-glc, peonidin-3-O-glucoside; Pt-glc, petunidin-3-O-glucoside; Q-glc-3, quercetin-3-O-glucoside; Q-glnA, quercetin-3-O-glucuronide; R, red; S, small grape skin fraction; t0, freshly produced fruit candies; t23, fruit candies after dehydration for 23 h at 50 °C; t26, fruit candies after dehydration for 26 h at 50 °C.

* Corresponding author. Tel.: +39 (0)2 503 19186; fax: +39 (0)2 503 19190.
E-mail addresses: manuela.mariotti@unimi.it, mariotti.manu@gmail.com (M. Mariotti).

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refer to a large and heterogeneous group of consumers: from children to elderly people. Hence, their enrichment with fibre and phenolic compounds with antioxidant activity would be a successful way to deliver beneficial compounds for human health to a large number of people.

In addition, anthocyanins are natural colouring agents found in grape skins. Maier, Fromm, Schieber, Kammerer, and Carle (2009) supplemented gelatine and pectin gels with anthocyanin extracts obtained from grape pomace and demonstrated that anthocyanins could be a promising alternative to synthetic colourants, with an additional potential effect on consumer health (McCann et al., 2007).

On the other hand, to develop foods enriched with both antioxidant and fibre components, it is necessary to evaluate the effects of fibre incorporation in terms of texture, which is of relevant importance for gel-like products. DeMars and Ziegler (2001) studied the texture and structure of gelatine/pectin-based gummy candies applying a stretching test on the product. They measured the stress and strain at fracture, which is particularly important for gummy candies. Compression tests are also largely used, to measure the strength of gel-like systems, as reported by Cappa, Lucisano and Mariotti (2013), as well as by Marfil, Anhê, and Telis (2012), who studied the properties of starch-based gels and gelatine/corn starch-based gummy candies, respectively. Multiple puncture tests are also commonly applied when evaluating fruit marmalades or jams.

This study investigated the effect of the addition of grape skin powders – comprised essentially of fibres and antioxidants – on a gel structure, such as that of fruit candies, developing a confectionery product stable at room temperature. To produce different candies, the multifunctional ingredient was previously processed into powders having different particles sizes, which were easily dispersible into the food matrices. The texture, phenolic contents, antioxidant activity and colour of the various formulations obtained were then studied.

2. Materials and methods

2.1. Chemicals

Cyanidin-3-O-glucoside, delphinidin-3-O-glucoside, malvidin-3-O-glucoside, peonidin-3-O-glucoside, petunidin-3-O-glucoside, kaempferol, quercetin, quercetin-3-O-glucoside and quercetin-3-O-glucuronide were purchased from Polyphenols (Sandes, Norway). All other standards and chemicals were purchased from Sigma Aldrich (Milan, Italy).

2.2. Grape skin powders

Red grape skins (GS) of Barbera cultivar (*Vitis vinifera* L.), collected from wineries recovered in the Piedmont region, were sieved (with a 5 mm sieve) to separate the skins from the seeds, dehydrated at 50 °C, and finely milled by a rotor mill (Cross Beater Mill, Retsch GmbH, Germany) at room temperature. The powders thus obtained were sieved (50 g batches; 10 min; amplitude 8) by means of an analytical sieve shaker Octagon Digital (Endecotts Ltd., England), equipped with three certified sieves (openings: 125, 250 and 500 μm). The following GS powders fractions were collected: small ($S \leq 125$ μm), medium ($125 < M \leq 250$ μm) and large ($250 < L \leq 500$ μm). The three GS powders were immediately vacuum packed and stored at 4 °C until further use.

2.3. Characterization of GS powders

2.3.1. Moisture and fibre content

GS powders were characterized for moisture content, according to the Official Standard Method AACC 44-15A (2000), and for total dietary fibre (TDF), insoluble dietary fibre (IDF) and soluble dietary fibre (SDF) according to the 'Total Dietary Fibre Assay Procedure' (Megazyme International Ireland Ltd., Wicklow, Ireland). All these evaluations were performed at least in duplicate and results are expressed on dry weight basis (dw).

2.3.2. Polyphenol contents and ferric ion reducing antioxidant power (FRAP)

Phenolics were extracted from GS fractions with methanol/water (80:20, v/v) containing 0.03 mol/l HCl, at room temperature: 4 ml of the solvent was added to the sample (0.2 g of GS fractions) and the mixture was stirred for 2 h; the mixture was then centrifuged at 10,000*g* for 10 min, the supernatant recovered and the solid residue re-extracted using 2 ml of the same solvent for additional three times; the supernatants were then pooled and stored at −20 °C in a dark bottle. Extractions were performed in duplicate.

The phenolic content of the extracts was analysed using a model 600 HPLC pump coupled to a model 2996 photodiode array detector, operated by Empower Software (Waters, Vimodrone, Italy). A 2.6 μm Kinetex C_{18} column (250 × 4.6 mm; Phenomenex, Bologna, Italy) was used for the separation, at a flow-rate of 1.5 ml/min. The column was maintained at 40 °C. The separation was performed by means of a linear gradient elution. Eluents were: (A) 0.02 mol/l H_3PO_4; (B) acetonitrile. The gradient was as follows: from 6% B to 20% B in 18 min; from 20% B to 60% B in 7 min; from 60% B to 90% B in 19 min; 90% B for 10 min and then 6% B for 5 min. DAD analysis was carried out in the range of 200–600 nm. Phenolic compounds were identified by their UV–vis spectra and retention times and quantified by calibration curves built with external standards, namely, malvidin-3-O-glucoside at 520 nm for anthocyanins, quercetin-3-O-glucoside at 354 nm for flavonols, phlorizin at 280 nm for chalcones, and chlorogenic acid at 330 nm. Results were expressed as mg/kg dw.

Soluble proanthocyanidin content was analyzed according to the method of Porter, Hirstich, and Chan (1986). Briefly, 1 ml of the extract (diluted with methanol:water, 80:20, v/v containing 0.03 mol/l HCl) was added to 6 ml of *n*-butanol:HCl (95:5, v/v) and 0.2 ml of 0.04 mol/l $NH_4Fe(SO_4)_2.12H_2O$ in 2 mol/l HCl. Hydrolysis was carried out at 95 °C for 40 min. The reaction mixtures were cooled, and the absorbance was recorded at 550 nm with a Jasco UVDEC-610 spectrophotometer against a blank made as for the sample but incubated at room temperature. Proanthocyanidin amount was determined using 0.1736 (mg/ml) as conversion factor (Travaglia, Bordiga, Locatelli, Coïsson, & Arlorio, 2011) and expressed as g/kg dw.

The FRAP assay was performed according to the procedure of Benzie and Strain (1996). Briefly, FRAP reagent was prepared by adding 25 ml of 0.30 mol/l acetate buffer, pH 3.6; 2.5 ml of 0.01 mol/l 2,4,6-tripyridyl-*s*-triazine in 0.04 mol/l HCl and 2.5 ml of 0.02 mol/l $FeCl_3$. The reaction mixture contained 0.4 ml of the extracts (diluted with methanol:water 80:20:0.1, v/v containing 0.03 mol/l HCl) and 3 ml of FRAP reagent. The increase in absorbance at 593 nm was evaluated with a Jasco UVDEC-610 spectrophotometer after 4 min of incubation at 37 °C against a blank with no extract addition. A methanolic solution of $FeSO_4$ was used for calibration. Results were expressed as mmol Fe(II) sulphate equivalents/kg dw.

2.4. Fruit candy production

Four different candy recipes were considered: one without GS, as the reference (J), and three containing each of the L, M or S GS powder fraction, respectively. The flow-sheet of candy production is shown in Fig. 1, and it was defined after preliminary trials.