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Effect of grape pomace powder addition on chemical, nutritional and technological properties of cakes

Gjore Nakov [a,*], Andrea Brandolini [b], Alyssa Hidalgo [c], Nastia Ivanova [a], Viktorija Stamatovska [d], Ivan Dimov [e]

[a] *Department of Chemical, Food and Biotechnology, University of Ruse "Angel Kanchev" - Branch Razgrad, blv. Aprilsko vastanie 47, 7200, Razgrad, Bulgaria*
[b] *Consiglio per la ricerca in agricoltura e l'analisi dell'economia agraria - Centro di Ricerca Zootecnia e Acquacoltura (CREA-ZA), Via Forlani 3, 26866, S. Angelo Lodigiano (LO), Italy*
[c] *Department of Food, Environmental and Nutritional Sciences (DeFENS), Università degli Studi di Milano, via Celoria 2, 20133, Milano, Italy*
[d] *Faculty of Technology and Technical Sciences, "St. Kliment Ohridski" University of Bitola, Dimitar Vlahov bb, 1400, Veles, Macedonia*
[e] *Faculty of Technics and Technologies, Trakia University - Stara Zagora, blv. Graf Ignatiev 38, 8600, Yambol, Bulgaria*

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ABSTRACT

Aim of the research was to study the influence of grape (*Vitis vinifera*) pomace powder, a by-product of wine manufacturing, on chemical composition, nutritional properties and physical characteristics of cakes prepared replacing bread wheat flour with 4%, 6%, 8% and 10% grape pomace powder. The addition of growing quantities of grape pomace powder gradually increased ash, lipid, proteins, fibres, free phenolics, anthocyanins and total polyphenol content as well as antioxidant capacity (DPPH, FRAP), while decreased moisture and pH. The main phenolics provided by grape pomace were catechin, gallic acid, quercitin, protocatechuic acid, kaempferol and apigenin. The phenolic acids and flavonoids content increased from 4.1 mg/kg DM (control) to 26.4–60.9 mg/kg DM (cake with 4%–10% grape pomace powder). The colour coordinates L^* and a^* diminished, while b^* augmented. The cake containing 4% grape pomace powder showed the best sensory quality. The addition of grape pomace powder significantly improved the content in free phenolics, highly bioavailable, that are scarce in bread wheat, and thus the nutritional value of cakes without penalising their technological and sensorial attributes. Therefore, grape pomace powder utilisation will give foods with nutritionally enhanced properties; additionally, its utilisation will alleviate the ecological problems connected to its disposal.

1. Introduction

Every year the food industry produces million tons of waste, difficult to dispose and that could be conveniently utilised as source of high-quality protein, fibre, antioxidants, sugars, etc. Consequently, the industries and the researchers are keenly studying the potential and the best ways for the economical exploitation of by-products *per se* or as source of bioactive components for foods, pharmaceutical and cosmetic products, and many other goods.

Grape vine (*Vitis vinifera* L.) is widespread all over the world, with a 2016 total surface of 7.5 million hectares and a production of 7.8 million tonnes, about 50% destined to wine production (OIV, 2017). The wine

* Corresponding author.
E-mail address: gnakov@uni-ruse.bg (G. Nakov).

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industry generates substantial quantities of waste, such as grape marc, discarded clusters and seeds, sediments and lees; in fact, pomace represents about 20–30% of the original grape weight (Dwyer, Hosseinian, & Rod, 2014).

Large amounts of pomace are produced every year, and their disposal can have a major impact on the environment; most wineries normally use it as animal feed, transform it into compost, or discard it without pretreatments, often causing soil pollution and acidification because of the low pH of the waste, and oxygen depletion in soil and ground waters by tannins and other compounds (Devesa-Rey et al., 2011; Dwyer et al., 2014).

Grape pomace is a rich source of bioactive substances, especially polyphenols, proteins, lipids, soluble dietary fibre (SDF), insoluble dietary fibre (IDF) and minerals (Devesa-Rey et al., 2011; Mildner-Szkudlarz, Bajerska, Zawirska-Wojtasiak, & Górecka, 2012; Teixeira et al., 2014; Yu & Ahmedna, 2013). Hence, several innovative ways have been proposed for a sustainable reutilisation of grape pomace (Devesa-Rey et al., 2011). Within the bakery industry, grape by-products have been successfully used as additives in the production of bread (Mildner-Szkudlarz, Zawirska-Wojtasiak, Szwengiel, & Pacyński, 2011), biscuits (Mildner-Szkudlarz et al., 2012), cereal bars, pancakes and noodles (Rosales Soto, Brown, & Ross, 2012). Other promising products are the cakes which, depending on the method used for their preparation, are classified as foam (e.g. sponge cake) or batter (e.g. pound cake) cakes (Pycarelle et al., 2019; Wilderjans, Luyts, Brijs, & Delcour, 2013).

To the best of our knowledge, no information is available on the effect of grape pomace on cake properties. Therefore, aim of our research was to evaluate the influence of grape pomace addition on the chemical, nutritional (bioactive substances content), technological and sensorial characteristics of cakes. To this end, bread wheat flour and grape pomace powder (GPP), as well as cakes prepared with growing proportions (0%, 4%, 6%, 8% and 10%) of GPP were analysed.

2. Materials and methods

2.1. Materials

The bread wheat flour (T-550 quality) used for cake manufacturing was bought from Sofia Mel industry (Sofia, Bulgaria). The grape pomace powder (GPP) was produced from Muscat Hamburg grapes harvested in 2018 in the Razgrad region (43°32′00″ N, 26°31′00″ E), Centre-North of Bulgaria. After juice removal, the grape pomace was dried for 48 h at 60°C in a UFE 500 oven (Memmert GmbH, Schwabach, Germany), ground into powder with an IKA MF10 grinder (IKA®-Werke GmbH & Co. KG, Staufen, Germany) and stored at 4°C until analysis or cakes preparation. The other ingredients were from local shops in Razgrad (Bulgaria).

The cakes were produced in laboratory, using the methodology of Velioğlu, Güner, Velioğlu, and Çelikyurt (2017) with minor changes. Briefly: 220.0 g wheat flour, 62.5 g margarine and 175.0 g sugar were blended with a high-speed mixer (Stand Mixer ELITE STM-0248, Timetron Bulgaria, Sofia, Bulgaria) for 3 min; two eggs were added, and the stirring continued for 3 min more; afterwards, 100 mL whole milk and 50 mL sunflower oil were added, and the homogenization continued for 2 min. The enriched cakes were prepared by replacing the flour with 4, 6, 8 and 10% GPP; these percentages were chosen considering the existing information on leavened bakery products (Mildner-Szkudlarz et al., 2011). Finally, 5.3 g baking powder were added, and the batter mixed for 3 min. The cakes were formed in 11 × 2.5 cm moulds, baked at 170°C for 15 min in a BES351110M oven (AEG, Bulgaria), cooled to room temperature for 30 min and analysed within 24 h. Five types of cakes were produced: control (100% wheat flour) and enriched with 4%, 6%, 8% and 10% GPP; two sets of 10 cakes were prepared for each flour or mixture.

2.2. Methods

2.2.1. Chemical characteristics

The moisture and ash content of bread wheat flour, GPP and cakes were determined according to methods 6540:1980 and 5984:2002 (International Standard Organisation), the protein content was determined following the modified Lowry method, as described in Mæhre, Dalheim, Edvinsen, Elvevoll, and Jensen (2018), the lipid concentration was tested according to Soxhlet method 136 (ICC), the fibre content was assessed as in method 32–07.01 (AACC International) and the cakes pH was determined according to method 02–52.01 (AACC International). All the analyses were performed in double.

2.2.2. Phenolic composition

The phenolic compounds of grape pomace powder and cakes were extracted as described by Yilmaz, Brandolini, and Hidalgo (2015) with some variations. Briefly, exactly 1.0 g of sample were extracted three times with 15 mL of 80% methanol. The pooled extracts were evaporated under vacuum and nitrogen flux, resuspended in 2 mL 80% methanol solution, filtered with a 0.45 μm PTFE membrane and analysed by RP-HPLC following Hidalgo et al. (2019). The analysis was performed using a column Adamas® C18-AQ 5 μm 4.6 mm × 250 mm and a precolumn C18 5 μm 4.6 mm × 10 mm (Sepachrom SRL, Rho, Italy) thermostated at 30 °C; pump L-2130, column oven L-2300 and Diode Array Detector L2450 (Elite La Chrom, Hitachi, Tokyo, Japan). Gradient elution was performed using acetonitrile (A) and 1% (v/v) formic acid in water (B) mobile phases at flow rate 1.0 mL/min, following the gradient profile: 0–10 min from 10% to 25% A, 10–20 min linear rise up to 60% A, and from 20 to 30 min linear rise up to 70% A, followed by 10 min reverse to initial 10% A with 5 min of equilibration time.

For peak quantification, calibration curves were constructed using standards from Sigma-Aldrich (St. Louis, MO, USA) and recorded at 280 nm for catechin (2.0–99.2 mg/L), epicatechin (3.2–85.0 mg/L), gallic acid (2.2–101.6 mg/L), protocatechuic acid (0.8–27.3 mg/L) and tyrosol (3.9–98.2 mg/L); at 320 nm for apigenin (0.8–10.0 mg/L); at 360 nm for ellagic acid (1.3–19.2 mg/L), kaempferol (0.1–9.5 mg/L), myricetin (1.3–17.2 mg/L) and quercitin (1.2–21.6 mg/L). The calibration curves were linear in the concentration intervals assessed. The identity of the compound was confirmed by congruence of retention times and UV/Vis spectra with those of pure authentic standards. Unidentified peaks were quantified using the calibration curve of the compound with similar absorption spectrum and named as "phenolic derivative". The analyses were performed twice; the results are expressed as mg/kg on dry matter (DM) basis.

2.2.3. Anthocyanins content, total polyphenols content and antioxidant capacity

The total anthocyanins were extracted and measured by a spectrophotometric method (Abdel-Aal & Hucl, 1999) using a V650 spectrophotometer (Jasco, Japan). The results are expressed as mg/kg DM on the basis of the cyanidin 3-glucoside standard calibration curve. The total polyphenolic content (TPC) of bread wheat flour, GPP and cakes was assessed on 80% methanol extracts by the Folin-Ciocalteu method as described in Nakov, Brandolini, Ivanova, Stamatovska, and Dimov (2018) and was expressed as mg gallic acid equivalent (GAE)/g dry matter (DM). The antioxidant capacity was determined with the 2, 2-diphenyl-1-picrylhydrazyl (DPPH) radical, according to Nakov, Brandolini, et al. (2018), and by the ferric reducing antioxidant power (FRAP) with the OxiSelect™ Assay Kit (Cell Biolabs, Inc. San Diego, CA, USA). The DPPH and the FRAP results are presented as μmol Trolox equivalents (TE)/g DM.

2.2.4. Physical characteristics

2.2.4.1. Pasting properties. The pasting properties of the bread wheat flour and the GPP-enriched mixtures were tested using a Micro Visco-Amylo-Graph (Brabender OHG, Duisburg, Germany). The following parameters were determined: peak viscosity, breakdown, setback and final viscosity (in Centipoise), and pasting temperature (in °C). The measurements were performed in duplicate.

2.2.4.2. Dimensions and colour. Mean width and thickness (mm) of the cakes were calculated from five SC. The volume (cm^3) was determined using a Volscan Profiler (Stable Micro Systems Ltd. Godalming, Surrey, UK), on three samples. The colour, in the *CIE L* a* b** system, was scored with a Chroma Meter CR-400 colorimeter (Konica Minolta, Tokyo, Japan) on two sets of five random SC.

2.2.4.3. Textural characteristics. The textural properties of the cakes (hardness, springiness, cohesiveness and chewiness) were measured using a TA-XT2 Plus texture analyser (Stable Micro System Ltd. Godalming, Surrey, UK). After removing the upper part of the cake, the crumb texture profile was analysed using a 5 × 5 cm diameter probe. The initial force employed (5 g) was applied twice, with a 10 s delay between the two applications; the probe speed was 5 mm/s until a 50% deformation of the sample centre.

2.2.5. Sensory analysis

The sensory analysis of the five cake types was performed at University of Ruse “Angel Kanchev”, Branch Razgrad (Bulgaria). Twenty trained people participated to the sensory analysis, after providing an informed consent according to the guidelines on Ethics and Food-Related research defined by the European Union (Alfonsi et al., 2012). Appearance, texture, odour, aroma and taste were scored from 1 to 5, where 1 is extreme dislike and 5 is extreme like; an overall quality score was computed as average of the five traits evaluated.

2.3. Statistical analysis

One-way analysis of variance (ANOVA) and, when significant, Fisher’s Least Significant Difference test (LSD) at $p < 0.05$ were performed with the softwares XL STAT 2017 (Addinsoft Inc. Long Island City, NY, USA) and Office Excel 2013 (Microsoft, Redmond, WA, USA). Pearson’s linear correlation analysis was performed with the software Statgraphics® Centurion XVI (Statgraphics Technologies Inc. The Plains, VA, USA).

3. Results and discussion

3.1. Bread wheat flour and grape pomace powder composition

The flour and the GPP results are presented in Table 1. The bread wheat flour and grape pomace powder composition was different for all the characteristics. The GPP had lower humidity than the flour, but its ash, lipid, protein, soluble and insoluble fibre, were far higher. The GPP was rich in free phenolic acids, phenylethanoids and flavonoids (Table 2), not tested in the refined bread wheat flour where they are extremely scarce (Wang et al., 2013). Additionally, bread wheat flour and GPP differed for anthocyanin content (not detectable vs. 976 ± 7 mg/kg DM), TPC (1.00 ± 0.17 vs. 53.7 ± 0.2 mg GAE/g DM), DPPH antioxidant capacity (301 ± 2 vs. 3366 ± 2 μmol TE/g DM) and FRAP antioxidant capacity (1448 ± 11 vs. 9692 ± 23 μmol TE/g DM).

The composition of grape pomace, and hence of retrievable substances, is broadly influenced by grapes cultivar, maturity, climate, soil and processing conditions; nevertheless, the GPP results were within the variation reported by different authors (Antoniolli, Fontana, Piccoli, & Bottini., 2015; García-Lomillo & González-SanJosé, 2016; Llobera & Canellas, 2007; Xu, Burton, Kim, & Sismour, 2016; Yu & Ahmedna, 2013; Özkan, Sagdiç, Baydar, & Kurumahmutoglu, 2004).

3.2. Pasting parameters of the mixtures

Table 3 provides an overview of the pasting parameters of the bread wheat flour and of the GPP-enriched mixtures. The ANOVA (not presented) highlighted significant differences ($p \leq 0.05$) among samples for all pasting properties. The peak viscosity, setback, final viscosity and pasting temperature augmented with increasing percentages of GPP. The viscosity increase could be related to the high content in GPP of soluble dietary fibre, particularly pectin (Yu & Ahmedna, 2013) and/or to the abundant lipids of grape seeds, which may interact with other hydrophobic substances (e.g. gluten), thus increasing viscosity and delaying starch gelatinization (Mironeasa Codină, & Mironeasa, 2012; García-Lomillo & González-San José, 2016). Accordingly, in bread dough Mironeasa and Codină (2013) observed an increase in peak viscosity (but a decrease in pasting temperature) after replacing the flour with 2%–6% citrus fibre and attributed it to a quicker starch gelatinization because of the higher water content in the dough samples with greater fibre content. Similarly, in bread wheat enriched with up to 11% apple pomace powder (Masoodi, Chauhan, Tyagi, Kumbhar, and Kaur 2001) found a progressive increase in peak viscosity after substituting part of wheat flour with apple pomace and ascribed it to the gelling effect of pomace pectin.

3.3. Chemical characteristics of the cakes

The ANOVA (not shown) proved the existence of broad differences among cakes for all traits, as expected based on GPP composition. The results of the proximate composition are shown in Table 1.

The moisture content and pH of the control cakes were significantly higher ($p \leq 0.05$) than those of the GPP-enriched cakes and decreased rapidly with increasing GPP addition. The lower pH of the GPP-enriched cakes is probably related to the presence of organic acids in grape pomace (García-Lomillo & González-SanJosé, 2016). On the other hand, the moisture decrease seems directly linked to the inferior humidity of GPP.

The ash, lipid, protein and dietary fibre (soluble, insoluble and total)

Table 1
Mean values (±standard deviation) of pH, moisture (g/100 g) and chemical composition (g/100 g DM) of bread wheat flour, grape pomace powder (GPP) and five cakes prepared with increasing quantities of GPP.

	Wheat flour	GPP	0% GPP	4% GPP	6% GPP	8% GPP	10% GPP
pH			7.77 ± 0.05^{a}	6.28 ± 0.05^{b}	6.05 ± 0.08^{c}	5.74 ± 0.06^{d}	5.36 ± 0.03^{e}
Moisture	13.40 ± 0.08	4.04 ± 0.06	23.43 ± 0.04^{a}	21.19 ± 0.01^{b}	19.92 ± 0.04^{c}	19.89 ± 0.07^{cd}	19.52 ± 0.10^{d}
Ash	0.53 ± 0.03	6.51 ± 0.01	0.70 ± 0.02^{d}	0.82 ± 0.01^{c}	0.85 ± 0.01^{b}	0.87 ± 0.01^{ab}	0.89 ± 0.01^{a}
Lipids	1.39 ± 0.01	14.95 ± 0.06	22.06 ± 0.38^{c}	22.55 ± 0.04^{bc}	23.04 ± 0.40^{bc}	23.53 ± 0.41^{ab}	24.51 ± 0.42^{a}
Proteins	11.42 ± 0.02	15.48 ± 0.98	18.23 ± 0.04^{e}	18.84 ± 0.10^{d}	19.24 ± 0.06^{c}	19.54 ± 0.09^{b}	19.89 ± 0.12^{a}
Soluble dietary fibre	4.00 ± 0.10	6.24 ± 0.11	3.23 ± 0.02^{d}	3.45 ± 0.08^{c}	3.67 ± 0.02^{c}	3.75 ± 0.01^{b}	3.89 ± 0.02^{a}
Insoluble dietary fibre	2.48 ± 0.06	51.16 ± 1.32	3.51 ± 0.01^{e}	5.54 ± 0.01^{d}	6.55 ± 0.04^{c}	7.60 ± 0.01^{b}	8.61 ± 0.02^{a}
Total dietary fibre	6.48 ± 0.25	54.4 ± 1.46	6.73 ± 0.03^{e}	8.99 ± 0.09^{d}	10.21 ± 0.02^{c}	11.34 ± 0.05^{b}	12.49 ± 0.03^{a}

Cake values in the same row with different letters are significantly different ($p < 0.05$) following Fisher’s LSD test.

Table 2
Content of individual phenolics, total phenolic acids, total phenylethanoids and total flavonoids (mean ± standard deviation; mg/kg DM) of grape pomace powder (GPP) and five cakes prepared with increasing quantities of GPP; n.d.: not detected, i.e. lower than the detection limit.

	GPP	0%	4%	6%	8%	10%
Phenolic acids						
Gallic acid	40.1 ± 3.1	4.1 ± 0.7^{d}	4.7 ± 0.1^{cd}	5.6 ± 0.2^{bc}	6.4 ± 0.1^{ab}	7.4 ± 0.9^{a}
Protocatechuic acid	53.8 ± 33.9	n.d.	1.9 ± 0.1^{d}	2.6 ± 0.1^{c}	3.1 ± 0.1^{b}	4.2 ± 0.1^{a}
Ellagic acid	32.2 ± 3.1	n.d.	n.d.	n.d.	n.d.	n.d.
Phenylethanoids						
Tyrosol	74.3 ± 0.9	68.9 ± 5.0^{a}	64.5 ± 0.01^{ab}	60.3 ± 0.7^{bc}	57.9 ± 2.2^{c}	49.9 ± 0.4^{d}
Flavonoids						
Catechin	795.5 ± 63.4	n.d.^{e}	12.8 ± 2.2^{d}	19.1 ± 3.9^{c}	25.2 ± 1.0^{b}	32.0 ± 0.7^{a}
Catechin derivative	11.3 ± 1.5	n.d.^{e}	0.6 ± 0.1^{d}	0.8 ± 0.03^{c}	1.3 ± 0.01^{b}	1.9 ± 0.09^{a}
Catechin derivative	34.7 ± 9.3	n.d.^{e}	0.4 ± 0.03^{d}	0.7 ± 0.01^{c}	1.0 ± 0.08^{b}	1.5 ± 0.02^{a}
Catechin derivative	51.6 ± 2.5	n.d.^{e}	0.9 ± 0.01^{d}	1.1 ± 0.10^{c}	1.4 ± 0.03^{b}	1.6 ± 0.03^{a}
Catechin derivative	91.6 ± 3.5	n.d.^{d}	0.6 ± 0.02^{c}	1.0 ± 0.08^{b}	1.0 ± 0.22^{ab}	1.3 ± 0.06^{a}
Catechin derivative	70.9 ± 1.1	n.d.^{e}	0.1 ± 0.01^{d}	0.3 ± 0.03^{c}	0.7 ± 0.02^{b}	0.9 ± 0.06^{a}
Catechin derivative	13.0 ± 3.4	n.d.^{e}	0.7 ± 0.02^{d}	1.1 ± 0.01^{c}	1.5 ± 0.01^{b}	2.0 ± 0.02^{a}
Catechin derivative	25.7 ± 6.8	n.d.^{c}	0.6 ± 0.06^{b}	0.8 ± 0.10^{b}	1.0 ± 0.08^{a}	1.2 ± 0.01^{a}
Epicatechin derivative	224.5 ± 24.7	n.d.^{e}	2.0 ± 0.4^{d}	3.8 ± 0.1^{c}	4.9 ± 0.05^{b}	7.0 ± 0.2^{a}
Myricetin	8.4 ± 1.0	n.d.	n.d.	n.d.	n.d.	n.d.
Quercetin	56.3 ± 10.2	n.d.^{c}	2.3 ± 0.3^{b}	2.9 ± 0.4^{b}	3.6 ± 0.3^{a}	4.1 ± 0.04^{a}
Apigenin	38.9 ± 8.5	n.d.^{d}	0.8 ± 0.02^{c}	1.0 ± 0.06^{b}	1.3 ± 0.04^{a}	1.3 ± 0.12^{a}
Kaempferol	39.1 ± 10.9	n.d.^{e}	1.0 ± 0.04^{d}	1.2 ± 0.06^{c}	1.4 ± 0.11^{b}	1.7 ± 0.04^{a}
Phenolic acids	126.1 ± 10.9	4.1 ± 0.7^{d}	6.6 ± 0.2^{c}	18.2 ± 0.2^{b}	9.5 ± 0.1^{b}	11.6 ± 1.0^{a}
Phenylethanoids	74.3 ± 0.9	68.9 ± 5.0^{a}	64.5 ± 0.1^{ab}	60.3 ± 0.7^{bc}	57.9 ± 2.2^{c}	49.9 ± 0.4^{d}
Flavonoids	1461.5 ± 66.2	n.d.^{e}	33.0 ± 2.9^{d}	34.3 ± 5.2^{c}	44.4 ± 1.8^{b}	56.3 ± 0.6^{a}
Total	1661.9 ± 78.0	73.0 ± 5.7^{d}	94.1 ± 3.1^{cd}	102.9 ± 6.1^{bc}	111.7 ± 3.9^{ab}	117.7 ± 1.3^{a}

Cake values in the same row with different letters are significantly different ($p < 0.05$) following Fisher's LSD test.

Table 3
Pasting characteristics (mean ± standard deviation) of the five mixtures used for the preparation of cakes with increasing quantities of grape pomace powder (GPP).

GPP	Viscosity (centipoise)				Pasting temperature (°C)
	Peak	Breakdown	Setback	Final	
0%	1319 ± 8^{e}	688 ± 1^{b}	948 ± 1^{e}	1579 ± 4^{e}	61.1 ± 0.3^{a}
4%	1544 ± 3^{d}	689 ± 2^{b}	1024 ± 2^{d}	1879 ± 2^{d}	62.5 ± 0.4^{b}
6%	1671 ± 5^{c}	670 ± 3^{c}	1040 ± 3^{c}	2040 ± 7^{c}	62.5 ± 0.4^{b}
8%	1729 ± 2^{b}	622 ± 2^{d}	1065 ± 2^{b}	2173 ± 0^{b}	62.8 ± 0.4^{b}
10%	1858 ± 13^{a}	696 ± 3^{a}	1100 ± 1^{a}	2261 ± 3^{a}	63.0 ± 0.4^{b}

Values in the same column with different letters are significantly different ($p < 0.05$) following Fisher's LSD test.

concentrations increased almost linearly (r = 0.95–0.97) with augmenting percentages of GPP. The ash is constituted by the minerals, which in grape pomace may vary depending on cultivar, soil type, climate and vineyard management (Lachman et al., 2013; García-Lomillo & González-SanJosé, 2016), as well as type and duration of the maceration processes (García-Lomillo & González-San José, 2016). Nevertheless, the ash content in food products increases even with the addition of small quantities (2–15%) of grape pomace (Acun & Gül, 2014; Bender et al., 2017; Theagarajan, Malur Narayanaswamy, Dutta, Moses, & Chinnaswamy, 2019).

The lipids content of the cakes is mainly due to the presence of margarine, sunflower oil and whole milk among the ingredients, but the increase in the GPP-enriched cakes is a consequence of the abundant presence of lipids in grape seeds (Acun & Gül, 2014; García-Lomillo & González-San José, 2016), inasmuch that they are commonly used to manufacture grapeseed oil (Devesa-Rey et al., 2011), rich in unsaturated fatty acids (García-Lomillo & González-San José, 2016). Similarly, Acun and Gül (2014) and Theagarajan et al. (2019) recorded a significant rise of lipids content in their grape pomace-enriched biscuits; however, Bender et al. (2017) in muffins did not notice a similar trend.

The higher protein and dietary fibre content of the GPP-enriched cakes in comparison to the 100% bread wheat cakes is related both to the use of protein-rich ingredients (eggs and whole milk) in the preparation of the cakes and to a major concentration in grape pomace, especially in the case of the insoluble dietary fibre. Accordingly, Acun and Gül (2014) and Theagarajan et al. (2019) found that by raising the percentage of grape pomace, the quantity of proteins and total dietary fibre in biscuits increased; similar findings are reported by Bender et al. (2017) for muffins. These changes can have significant implications on the technological and textural characteristics of the products (Nakov, Stamatovska, et al., 2018): for example, during baking the proteins coagulate, reinforcing the structure of the gluten matrix. The dietary fibre, besides its contribution to the technological quality, has significant implications on several nutritional aspects (Slavin, 2008). The soluble dietary fibre (oligosaccharides, pectin, beta-glucans and gums) is degraded in the colon and finally digested by the bacteria; among other beneficial effects, it helps lowering blood cholesterol and controls blood sugar (Foschia, Peressini, Sensidoni, & Brennan., 2013). The insoluble dietary fibre (cellulose, hemicellulose, lignin, resistant starch) transits intact through the gastrointestinal tract and improves the peristalsis; additionally, is partially fermented in the large intestine and supports the growth of intestinal microflora, including probiotic bacteria (Foschia, Peressini, Sensidoni, & Brennan, 2013). Altogether, adequate total dietary fibre intakes contribute to the prevention of diseases like hypertension, diabetes and obesity (Anderson et al., 2009; Foschia et al., 2013).

3.4. Phenolic compounds content

The ANOVA for free phenols composition (not presented) showed significant differences (*p < 0.05*) between the control cakes and the samples enriched with GPP. The free phenolics identified in the GPP and in the cakes are reported in detail in Table 2. Catechin and catechin derivative compounds, the most abundant phenols in grape pomace powder (1094.3 mg/kg DM, i.e. 65.8% total), were absent in the control cake. Other flavonoids in GPP were, in decreasing order, epicatechin derivative, apigenin, kaempferol, myricetin and quercitin, while the phenolic acids recovered were protocatechuic acid, gallic acid and ellagic acid, and the only phenylethanoid detected was tyrosol. In the control cake only tyrosol, and in small quantity gallic acid, were found, probably coming from the other ingredients. In the GPP enriched cakes, all the phenolics detected in GPP were found (except ellagic acid and myricetin), with values increasing according to growing GPP concentrations; the only exception was tyrosol, abundant in the control and progressively diminishing in SCs. Hence, the total phenols concentration in the GPP-enriched cakes was significantly superior to the control cake, confirming the usefulness of this winery by-product for the nutritional improvement of bakery products. Even more relevant is that in wheat the bound polyphenols represent the most abundant fraction (77%), followed by conjugated (22%) and free (<0.5–1%) (Li, Shewry, & Ward, 2008). Bound phenolic acids are highly stable under heat treatments but have poor nutritional significance because of low bioaccessibility; the scarce free form, instead, is the most bioavailable and the least stable (Hidalgo, Brandolini, Čanadanović-Brunet, Ćetković, & Tumbas Šaponjac, 2018). Therefore, adding grape pomace powder, rich in free phenolics, will significantly improve the polyphenols composition of the cakes.

Nevertheless, when the phenolic content of the enriched cakes was compared to the theoretical values computed by mass balance, a relevant loss of these compounds during baking was noticed, going from 31.19 (4% SC) to 39.22 (6% SC) to 44.18 (8% SC) to 49.15 (10% SC). The loss was compound-dependent and showed that the flavonoids were extremely prone to degradation (on average, 61.5%, and similar at all concentrations), while phenolic acids (28.8%, similar across concentrations) and phenilethanoids (from 6.3 at 4% to 28.1 at 10% GPP) had better resistance to enzymatic and thermal degradation.

3.5. Anthocyanin, total polyphenols content, and antioxidant capacity

Fig. 1 depicts the increase in anthocyanins and TPC as well as in antioxidant capacity (DPPH and FRAP tests) from control cakes up to 10% GPP-enriched SC. The ANOVA (not shown) highlighted the existence of significant differences among the cakes for all these traits. The anthocyanins (Fig. 1A) were absent in the control and increased quickly with the grape pomace powder enrichment, reaching 26.4 g/kg DM at 10% GPP. Similarly, the control contained 19.56 ± 1.30 mg GAE/100 g DM TPC (Fig. 1B), but the addition of GPP rapidly increased that value to a maximum of 53.73 ± 0.75 mg GAE/100 g DM in cakes with 10% GPP.

Anthocyanins are abundant in red grapes, and therefore their increase in GPP-enriched products is not a surprise, as reported also by Theagarajan et al. (2019) for cookies.

A TPC increase following GPP addition was reported also in cookies by Acun and Gül (2014) (from 0.0 in the control to 75.1 g/kg GAE with 15% GPP), by Pasqualone et al. (2014) (from 440 in the control to 629 mg GAE/kg DM with ca 22% grape mark extract) and by Theagarajan et al. (2019) (from 3.41 to 4.03 mg GAE/g with 6% GPP), and by Hayta, Özugur, Etgü, and Seker (2014) in bread (from 35.4 in the control to 89.4 mg GAE/100 g DM in bread with 10% GPP). Therefore, the enrichment with grape pomace powder should allow to manufacture functional foods with improved nutritional properties. The phenolic substances have well-known biological activities (antioxidant, antimicrobial, etc.) (García-Lomillo & González-SanJosé, 2016); furthermore, they can react with superoxide anions, hydroxyl radicals and lipid peroxyl radicals (molecules responsible of lipid oxidation and products rancidity), thus acting as antioxidants and contributing to extend the shelf-life of foods (Fontana, Antoniolli, & Bottini, 2013; García-Lomillo & González-SanJosé, 2016).

In line with the TPC results, the enrichment with GPP led to a significant antioxidant capacity increase from 340 μmol TE/g (DPPH; Figs. 1C) and 1610 μmol TE/g (FRAP; Fig. 1D) in the control cakes (no GPP added) to 462 μmol TE/g (DPPH) and 2428 μg TE/g DM (FRAP) in the 10% GPP-added SC. Similarly, Acun and Gül (2014), Hayta et al. (2014), Pasqualone et al. (2014) and Theagarajan et al. (2019) found that grape pomace extracts significantly increased the antioxidant capacity of bakery products.

3.6. Physical characteristics

The ANOVA (not shown) highlighted significant differences ($p<0.05$) for all the traits assessed; Table 4 reports the information about the physical characteristics of the cakes produced without (control) or with 4%, 6%, 8% and 10% GPP. The width did not differ significantly among the samples up to 8% GPP addition and decreased slightly only in the 10%-enriched SC. The thickness, instead, decreased progressively from 34.1 mm (control) to 30.8 mm (10% GPP). Therefore, the volume

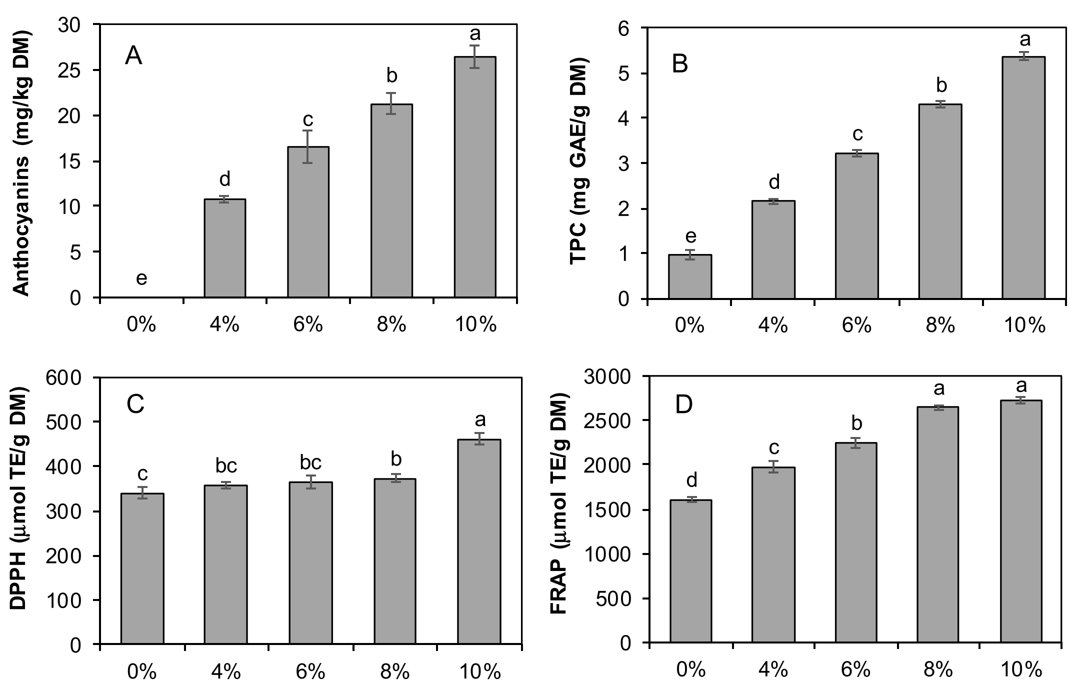

Fig. 1. Anthocyanin content (A), total polyphenol content (TPC) (B) and antioxidant capacity (C: DPPH and D: FRAP) of five sponge cakes prepared with increasing quantities (0%, 4%, 6%, 8% and 10%) of grape pomace powder. Error bars represent the standard deviation. Columns with different letters are significantly different ($p < 0.05$) following Fisher's LSD test.

Table 4
Physical and textural characteristics (mean ± standard deviation) of the five cakes, prepared with increasing quantities of grape pomace powder (GPP).

GPP	Width (mm)	Thickness (mm)	Volume (cm^3)	Colour			Texture			
				L^*	a^*	b^*	Hardness (N)	Springiness	Cohesiveness	Chewiness (N)
0%	57.25 ± 0.21[a]	34.05 ± 0.64[a]	128.00 ± 1.41[a]	58.17 ± 1.22[a]	43.11 ± 1.43[a]	71.29 ± 2.17[b]	35.48 ± 0.50[e]	0.84 ± 0.01[a]	0.58 ± 0.01[a]	17.19 ± 0.25[d]
4%	57.10 ± 0.57[a]	33.40 ± 0.42[ab]	127.00 ± 0.10[a]	55.32 ± 0.88[b]	35.61 ± 0.91[b]	72.15 ± 1.52[b]	44.15 ± 0.37[d]	0.83 ± 0.01[ab]	0.55 ± 0.01[b]	19.97 ± 0.97[c]
6%	56.80 ± 0.71[a]	32.20 ± 0.42[bc]	126.50 ± 0.71[a]	54.72 ± 0.28[b]	32.07 ± 0.62[c]	74.11 ± 0.10[a]	52.47 ± 0.69[c]	0.82 ± 0.01[b]	0.53 ± 0.01[b]	21.87 ± 0.78[b]
8%	56.50 ± 0.14[a]	31.05 ± 0.49[c]	123.50 ± 0.71[b]	53.60 ± 0.42[b]	31.26 ± 0.14[c]	74.18 ± 0.21[a]	57.35 ± 0.68[b]	0.79 ± 0.01[c]	0.53 ± 0.01[b]	22.95 ± 0.23[b]
10%	55.05 ± 0.21[b]	30.80 ± 0.71[c]	120.50 ± 0.71[c]	50.73 ± 0.52[c]	29.41 ± 1.33[d]	74.39 ± 1.41[a]	63.88 ± 0.09[a]	0.79 ± 0.01[c]	0.51 ± 0.01[c]	26.67 ± 0.11[a]

Values in the same column with different letters are significantly different ($p < 0.05$) following Fisher's LSD test.

did not change significantly from 0 to 6% GPP, but diminished sharply afterwards, down to 120.5 mm with 10% GPP. Acun and Gül (2014) did not record changes in width, thickness and spread ratio in biscuits enriched with up to 15% GPP, while Hayta et al. (2014) recorded a small, not significant decrease (from 1508 to 1425 mL) in leavened bread; on the other hand, Walker, Tseng, Cavender, Ross, and Zhao (2014) observed a substantial volume reduction in muffins with up to 20% GPP, and attributed it to the negative influence of GPP fibres which interfere with the dough structure and decrease CO_2 retention. The colour parameters L^*, a^*, b^* varied with the addition of GPP. The control cake was the brightest ($L^* = 58.17$), followed by the 4%, 6% and 8% GPP-enriched samples, which had similar luminosity, while the 10% GPP-added cakes was the darkest ($L^* = 50.73$). The a^* parameter diminished progressively from control (43.1) to 10% GPP-enriched cakes (29.4), indicating a decrease of redness. On the contrary, the b^* values were alike in the control and the 4% cake and increased in the other three samples, denoting a yellower tinge. These changes should be attributed to the darker colour of the GPP in comparison to the bread wheat flour and is observed also when other types of whole meals are employed. Our findings are in good agreement with the results of Acun and Gül (2014), Bender et al. (2017) and Hayta et al. (2014), who noticed lower L^* and a^* (and lower b*; Bender et al., 2017) in cookies, muffins and breadcrumbs, respectively, after adding grape pomace, while Pasqualone et al. (2014) recorded decreasing L^* and b^*, along with increasing a^*, in biscuits enriched with grape pomace extracts. These alternative behaviours are probably attributable to the different grape varieties and processes utilised in GPP preparation. Changes in CIE coordinates are common after the addition of different pomace to bread wheat products; for example, Tumbas Šaponjac et al. (2016) discovered some darkening in biscuits enriched with sour cherry pomace. In our cakes, the anthocyanin pigments present in the GPP played a major part in colour coordinates changes, but a significant effect on luminosity was probably exerted also by baking, because heating leads to the formation of dark Maillard reaction products as a consequence of reducing sugars (e.g. glucose and fructose, abundant in grape pomace) and amino acids presence. The different cakes are presented in Supplementary Fig. 1.

Hardness, springiness, cohesiveness and chewiness of the different SCs are shown in Table 4. The addition of GPP increased hardness (from 35.5 to 63.9 N) and chewiness (from 17.2 to 26.7 N), but slightly reduced springiness (from 0.84 to 0.79) and cohesiveness (from 0.58 to 0.51). Bender et al. (2017) and Hayta et al. (2014) observed similar changes in muffins and in bread wheats, respectively, enriched with up to 10% GPP. Similarly, Walker et al. (2014) described a reduction in springiness and an increase in firmness in GPP-enriched muffins, and attributed them to the reduced volume and increased density of the samples, due to the higher water absorption capacity of the fibre.

3.7. Sensory characteristics

Consumers appreciation of food products is largely based on visual and sensorial impression. Hence, for new products development a sensory analysis is necessary to test the acceptability of foods. Our analysis showed that the cake with 4% GPP got the highest evaluation marks (Fig. 2A) for all five the sensory parameters (appearance, texture, taste

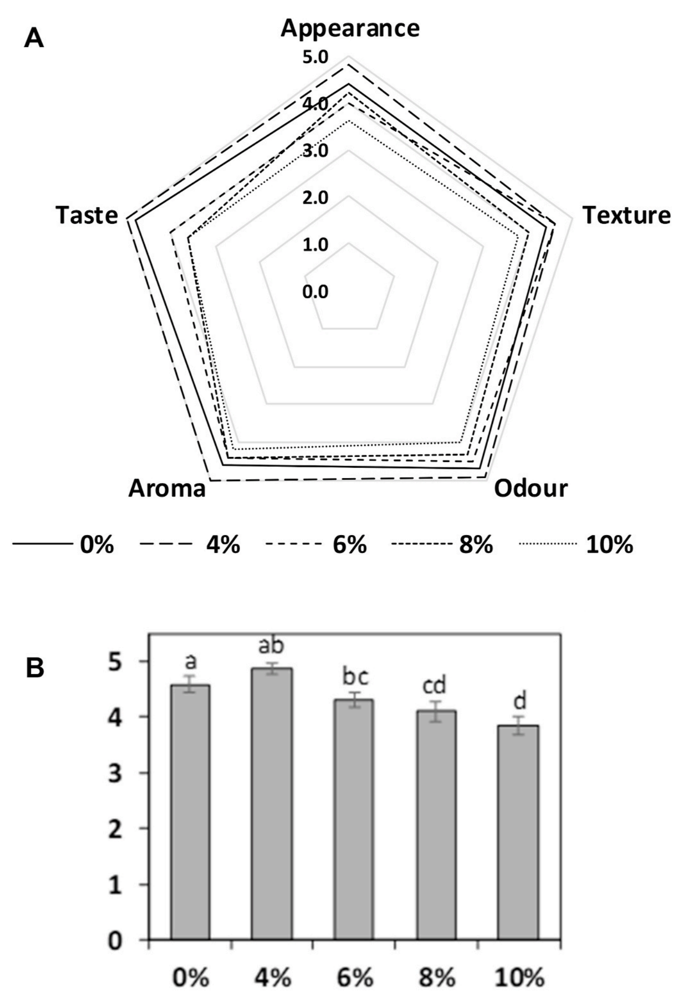

Fig. 2. Sensory characteristics (A) and overall sensorial quality (B) of five cakes prepared with increasing quantities (0%, 4%, 6%, 8% and 10%) of grape pomace powder. Scale from 1 (extreme dislike) to 5 (extreme like). Columns with different letters are significantly different ($p < 0.05$) following Fisher's LSD test.

and odour), while the cake with 6% GPP was outstanding for texture; the control was pleasing but received slightly lower scores. Hence, the best total evaluation in terms of sensory analysis was achieved by the cakes enriched with 4% GPP (Fig. 2B). Our observation that the addition of small quantities (4–6%) of grape pomace to food products improve the nutritional quality and impart better sensorial characteristics corroborates the findings of Acun and Gül (2014), Bender et al. (2017), Theagarajan et al. (2019), Hayta et al. (2014), Rosales Soto et al. (2012), and Walker et al. (2014) in different bakery products.

4. Conclusion

Cakes enriched with grape pomace powder have significantly higher ash, lipids, proteins, anthocyanins, polyphenols, dietary fibre and antioxidant capacity than the control. Particularly important is the increase in free phenolics, highly bioavailable and scarce in bread wheat. The addition of small quantities of grape pomace powder does not worsen the technological characteristics of the cakes and improves their sensory qualities. Therefore, the direct utilisation of grape pomace powder gives foods with nutritionally enhanced properties; additionally, its utilisation in food production will alleviate the ecological problems connected to its disposal.

Declaration of conflicting interests

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

CRediT authorship contribution statement

G.N., A.B., A.H., N.I., V.S. and I.D. conceived and planned the experiments. G.N., A.H. and I.D. carried out the experiments. G.N, N.I., A. S., V.S. and I.D. contributed to sample preparation. A.B, G.N. and A.H. contributed to the interpretation of the results. A.B. took the lead in writing the manuscript. All authors provided critical feedback and helped shape the research, analysis and manuscript.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2020.109950.

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