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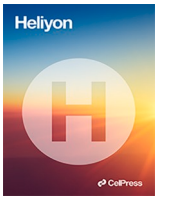

Preservation of white wine pomace by high hydrostatic pressure

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A B S T R A C T

The effect of different high hydrostatic pressure (HHP) treatments (400, 600 MPa for 1, 6 min) on white wine pomace was studied throughout storage conditions (270 days) at different temperature conditions (4° and 20 °C). The final use of this product would be as an ingredient for food products preservation. Microbiological, enzyme and physico-chemical parameters were evaluated after processing and during storage. HHP greatly reduced the microbial counts of treated pomace and allowed obtaining a safe product with a long shelf-life at 4 and 20 °C. The HHP treatment also preserved phenolic compounds content, however an important reduction of these compounds was found during storage since the polyphenol oxidase enzyme remained active after the treatment and during storage. Phenolic compounds were better preserved during storage at 4 °C than at 20 °C. The application of HHP at 600 MPa/6 min and the refrigeration of the treated pomace would allow obtaining a microbiologically safe pomace with high levels of phenolic compounds with a shelf-life of 90 days. The activity of the enzyme should be limited in future to ensure a long shelf-life of the processed pomace.

1. - Introduction

Winemaking is one of the most important agro-industrial activities in Spain, resulting in a strategic sector. The average annual production of wine and must is approximately 40 million hectoliters and Spain is the second largest exporter in volume worldwide with just over 2012 million liters in 2020 [1].

In the winemaking process, it is estimated that 25 % of the weight of the grapes is transformed into co-products/wastes [2,3], therefore, there is a large amount of material that represents a challenge and an opportunity for the sector. In general, co-products are known as those materials considered to be any non-main product obtained in a determined process and that may have certain applications or uses. The main co-product of winemaking is grape pomace, which accounts for 62 % of the total co-products [4].

Grape pomace (GP) is formed by seeds, skins, stems and remains of grape pulp. There are differences depending on whether it is red or white wine pomace, not only due to the different chemical composition of the grapes, but also because of the different processes involved. In the production of red wine, in order to transfer the phenolic substances that provide colour and other qualities, the grapes remain in contact with the juice during the fermentation process. During traditional white grape winemaking, seeds and skins are removed before fermentation and only grape juice (must) is fermented. Thus, this latter grape pomace generally has more sugars, more

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water and no alcohol [2,4].

Traditionally GP has been used to distil it for wine ethanol production. Other uses are for animal feed; directly added to agricultural soils to increase its content in organic matter, nitrogen and minerals; or it could be used for composting, forming part of cultivation substrates [4,5]. However, the high level of phenolic compounds of the pomace reduces the digestibility and causes phytotoxicity [6,7] Therefore, GP is a valuable co-product, whose high concentration of phenolic compounds may be a problem when used directly, but it is very interesting as a natural source of these compounds for the food industry due to its high content of phytochemicals.

GP has been proposed as a source of bioactive compounds that can be easily obtained from conventional solvent extraction, usually used at industrial scale, but other greener procedures are being sought [8,9]. An important drawback is that solid phase and solvent residues are generated in the extraction process. The conventional extraction process can be highly polluting for the environment, producing hazardous solvent and solid residues, and is therefore not in line with the principles of green chemistry. Therefore, it is necessary to look for alternatives such as the direct and integral use of GP, as an additive or ingredient, in different food sectors, such as the production of cereals, dairy and meat products [4,10,11]. GP is an unstable product that deteriorates rapidly if not properly preserved and it is also a seasonal product. The simplest processes for making pomace a safe product, drying, provide stability and safety, but can cause loss of thermolabile bioactive compounds [12,13]. The application of non-thermal technologies is considered as an alternative to adequately stabilize the by-products[14]. Similarly, the γ-irradiation treatments and addition of chemical preservatives have been studied for the stabilization of GP ([15]), however, this technology is not generally accepted by consumers.

High hydrostatic pressure (HHP) is a novel non-thermal technology, which is gaining attention as an environmentally sustainable and cost-effective technology for food product preservation [16]. In general, HHP allows a great preservation of bioactive compounds in vegetable products, especially compared to those treatments that require high temperatures [8]. HHP was applied to stabilize the grape before winemaking. The treatment reduced effectively microbial loads, which was beneficial for the use of starter cultures, and in addition, the treatment favored the extraction of phenolic compounds [17]. The application of HHP could be an interesting technology for the valorization of by-products from winery industry since it is a commercially accepted technology which would allow obtaining microbiologically safe products rich in bioactive compounds. Therefore, the main objective was to evaluate the effect of different high pressure treatments on white wine pomace (microbiological, enzyme and physical-chemical parameters) throughout storage at different conditions. The final use of this product would be as an ingredient for food products preservation. As far as we know, at the present, this is the first study that evaluates the effect of HHP on pomace. In addition, since this is a seasonal product the storage conditions of the processed product should be also evaluated.

2. Material and methods

2.1. Starting material

This study was performed with GP obtained from *Vitis vinifera* L. 'Cayetana', 'Pardina' and 'Montúa', traditional varieties grown in Extremadura region, provided by Santa Marta de los Barros cooperative (Badajoz, Exremadura, Spain) and harvested in 2020. The traditional white vinification was followed: grapes were destemmed and pressed and only the must was fermented.For the development of this study, 10 kg of GP were vacuum packaged in 1 kg-plastic bags (OptiDure™ ODA7005 plastic bags: oxygen permeability: 10 cm^3 m^{-2}, 24 h^{-1} and 0 % relative humidity) (Cryovac, Madrid, Spain). Vacuum packaging (−0.8 bar) was performed using Henkovac Proeco equipment (Henkovac International, Hertogenbosch, The Netherlands). Packages were stored at −80 °C until the experiment was performed.

The samples were prepared to develop the experiment. Firstly, the frozen pomace was ground in a Thermomix TM5 (Thermomix-Vorwerk, Germany) for 2.5 min at maximum speed, until obtaining a finely milled product, like a semi-solid purée. This milled GP was packed in 50 g vacuum bags (packages had the same composition as previously). A total of 150 bags were prepared and stored at −80 °C until the application of the HHP treatment.

2.2. High hydrostatic pressure (HHP) treatment and storage

The ground vacuum packaged GP was processed in a semi-industrial Hiperbaric equipment (6000/55, Hiperbaric, S.A., Burgos, Spain) with a vessel with a capacity of 55 L. Four different treatments were applied combining two pressure intensities (400 and 600 MPa) and two holding times (1 and 6 min). The initial temperature of the water was 16 °C. In order to use as control, some vacuum bags with milled GP were no treated. The different physical-chemical analysis were carried out on the following day of the application of HHP (day 1), and at four times over 9 months (day 30, day 90, day 180 and day 270), with storage at two different temperatures 4 °C and 20 °C. All samples were stored vacuum-packaged in darkness. 150 bags (samples) were analyzed: 3 replicates x 5 treatments x 5 sampling times x 2 temperatures. Each bag was individually analyzed. All analysis were carried out in the milled, vacuum packaged and treated (or not in control) grape pomace.

2.3. Proximate composition, pH and aw of the untreated pomace

In the initial, non-treated milled and vacuum packaged GP (control), and in the treated GP (3 independent vacuum packaged bags by sample), was also analyzed to evaluated the physico-chemical composition. pH was evaluated with a pHmeter (Hanna instrument) and water activity (Novasina Labmaster, Lachen, Switzerland). Moisture and protein were determined according to the AOAC [18], fat content was analyzed by Folch method [19]. Fiber content was determined according to the modified Southgate method [20].

2.3.1. Volatile compounds

The major volatile compounds volatile compounds from grape pomace were determined at day 1 (1 day after HHP). In the first step, 20 g of sample of grape pomace were mixed with 200 mL of water. This mixture was subjected to distillation in an oenological distiller (steam distillation system, DE 1626 GAB) to obtain 200 mL of distillate. A gas chromatograph (Hewlett Packard 6890) equipped with FID has been used for the analysis of aroma of major volatile compounds of the pomace distillate. Samples of 1 μL were injected into the gas chromatograph. The injector of the gas chromatograph was maintained at 250 °C and operated under split mode. Elution was achieved in a 60 m × 0.32 mm i.d. x 0.5-μm capillary INNOWAX column. The oven temperature program was as follows: 50 °C for 5 min, a linear ramp from 50 °C to 100 °C at 10 °C/min, and finally to 220 °C at a rate of 30 °C/min. Detection was by FID at a temperature of 250 °C. Identification was achieved by retention times of standars compounds. Quantitative data were obtained by interpolation of relative peak areas in the calibration graphs constructed by the analysis of mixtures containing known amounts of the analytes.

2.4. Microbiological analysis

For microbiological analysis, a 10 g sample of GP was aseptically weighted in a sterile plastic bag and homogenized with 90 mL of a sterile solution Peptone Water (Merck, Darmstadt, Germany) in a masticator blender (Stomacher 400 Circulator), 1/10 dilution. Serial 10-fold dilutions were prepared by mixing 1 mL of the previous dilution with 9 mL of sterile Peptone Water. Total viable counts were enumerated in Plate Count Agar (PCA; Merck) and incubated at 30 °C for 72 h; molds and yeasts were enumerated using CG Agar Base (Merck) with CGA Selective Supplement (Merck) and incubated at 25 °C for 4–5 days and *Enterobacteriaceae* (VRBG Agar, 37 °C, 24–48h). After incubation, plates with 30–300 colonies were counted. All microbial counts were expressed as colony-forming units (CFU) per g of sample (log CFU g^{-1}).

2.5. Instrumental color

The CIELAB color coordinates: lightness (L*), redness (a* red/green axis) and yellowness (b* yellow/blue axis) were determined using a Konica Minolta CM-5 spectrophotometer and one cuvette was used for readings (Konica Minolta, Tokyo, Japan) in reflectance. The color total difference (ΔE*) during storage was calculated ($\Delta E^* = ((L^*_1\text{-}L^*_2)^2 + (a^*_1\text{-}a^*_2)^2 + (b^*_1\text{-}b^*_2)^2)^{0.5}$.

ΔE processing compares color values of control-initial pomace with the pomace after HHP. *ΔE storage 1-30d* compares color values of initial pomace (day 1) with the pomace after treated at the same HHP conditions 30 days of storage. *ΔE storage 1-90d* compares color values of initial pomace (day 1) with the pomace (treated at the same HHP conditions after 90 days of storage. *ΔE storage 1-180d* compares color values of initial pomace (day 1) with the pomace treated at the same HHP conditions after 180 days of storage. *ΔE storage 1-270d* compares color values of initial pomace (day 1) with the pomace treated at the same HHP conditions after 270 days of storage.

2.5.1. - Phenolic compounds content (PCC) and polyphenol oxidase (PPO) enzyme activity

The total content of phenolic compounds was determined using the Folin–Ciocalteu reagent [21]. The results were expressed as mg of Gallic acid equivalent (GAE) per 100 g^{-1} of sample weight on wet base (WB).

The polyphenol oxidase (PPO) extracts and the enzymatic activity analysis were carried out as described by Terefe et al. [22]. Absorbance was measured at 420 nm and 25 °C for 3 min in a Thermo Scientific Evolution 201 UV–Vis spectrophotometer (Thermo Scientific™, Fisher Scientific SL, Madrid, Spain), in kinetic model. The results were expressed as a percentage of activity with respect to the control samples.

2.6. Statistical analysis

The assay was performed in triplicate (three bags per batch) and the mean values and their standard deviations (SD) were calculated. The one-way analysis of variance (ANOVA) was applied two times (Tables 2, 4 and 5) to know the effect of processing and time of storage using the statistical program SPSS 21.0 (SPSS Inc., Chicago, IL). When ANOVA detected significant differences between mean values, means were compared using Tukey's test ($p < 0.05$). In addition, the global effect of treatments applied was also evaluated by a three-way ANOVA to know the effect of the following factors: HHP, time of storage and temperature of storage (Table 3).

Table 1
Proximate composition (%), pH and Aw of the white wine pomace (mean±standard deviations).

	Mean		S.D.
Moisture	56.7	±	0.1
Fibre	27.8	±	0.8
Protein	3.2	±	0.4
Fat	1.6	±	0.1
pH	3.66	±	0.04
Aw	0.97	±	0.00

Table 2
Major volatile compounds in white grape wine pomace (concentration in mg kg^{-1}) after high hydrostatic pressure (1 day after processing).

	Control	400 MPa 1min	400 MPa 6min	600 MPa 1min	600 MPa 6min	p-value
Aldehydes						
Acetaldehyde	350.9b ± 42.3	374.8b ± 39.6	366.7b ± 25.7	402.4b ± 48.7	476.1a±45.7	***
Ethyl esters						
Ethyl acetate	49.8 ± 22.3	42.3 ± 12.8	55.3 ± 18.2	58.6 ± 13.9	37.5 ± 11.5	ns
Alcohols						
Methanol	734.7b ± 147.15	713.7BCE±117.7	518.7c±69.1	823.0b ± 141.1	1078.8a±80.3	**
1-Propanol	26.5b ± 4.8	27.8 ab ± 3.2	29.4 ab ± 3.9	31.5 ab ± 4.2	33.3a±2.7	*
2-Methyl-1-propanol	14.0 ± 7.5	15.1 ± 4.3	14.6 ± 3.4	17.2 ± 5.9	20.9 ± 5.7	ns
3-Methyl-1 butanol	72.1 ± 19.1	70.7 ± 9.9	69.0 ± 17.3	82.0 ± 8.1	79.1 ± 6.6	ns
2-Phenylethanol	26.7 ± 1.5	26.9 ± 0.9	25.9 ± 2.1	27.0 ± 0.6	27.9 ± 1.5	ns
Ethanol	22819.9 ± 4522.2	23962.7 ± 2983.2	24151.8 ± 2844.4	26571.0 ± 2998.8	28093.8 ± 2388.7	ns

Means values followed by different letters indicate the existence of significant differences between treatments by the Tukey test ($p < 0.05$). ns indicates no difference; * Significance at $p < 0.05$; ** Significance at $p < 0.01$; *** Significance at $p < 0.001$.

Table 3
Three-way analysis of the variance of the microbial counts, colour parameters, phenolic compounds content (PCC) and polyphenol oxidase enzyme activity (PPO).

	Probability						
	P1	P2	P3	P1xP2	P1xP3	P2x P3	P1xP2xP3
Mesophilic	***	***	***	***	***	***	***
Molds and Yeasts	***	***	***	***	***	***	***
Enterobacteriaceae	***	***	***	***	***	***	***
*CIE L**	***	***	***	**	ns	**	ns
*CIE a**	ns	***	***	ns	*	***	*
*CIE b**	ns	***	***	ns	ns	***	*
PCC	ns	***	***	ns	*	***	ns
PPO	ns	***	***	*	*	***	**

P1: P-value HHP (hydrostatic high pressure); P2: P-value temperature of storage; P3: P-value time of storage. PCC: Phenolic compounds content. PPO: Polyphenol oxydase activity.
ns (non-significant differences). *p < 0.05 **p < 0.01 ***p < 0.001.

3. - Results and discussion

3.1. Proximate composition and pH of initial GP

The characterization (Table 1) is a necessary step to know the potential use of this by-product for food processing, since the composition of each pomace is variable. The moisture of the pomace was similar to that found by other authors [23,24], although the literature also reported higher percentages than ours, such as 70 % [25]. In general, the white wine pomace has more moisture than the red wine pomace; and the variance in white wine pomace depends on the intensity of the crushing in each winery. The fiber is over the water, the main fraction of the wine pomace. It is made up of polysaccharides and lignin winemaking method, the red wine pomace tends to have more fiber than the white pomace [4,10]. The GP studied was within the range reported by other authors [10]. These data are difficult to compare, since the results of the proximate composition are often given on a dry basis or from pomace flour [26–29, 30], and sometimes only skins are used, without seeds or stalk remains [31,32]. Protein and fat presented values within the range reported by Antonić et al. [10]: 3.57–14.17 and 1.14–13.90 of grape pomace (on dry weight), respectively. It should be pointed out that the fat fraction comes from the seeds and is interesting for being rich in unsaturated fatty acids [4,10,28,30] and powerful antioxidants such as vitamin E [10,33].

Although the studies that analyze the Aw of pomace are scarce, (Taşeri et al. [24] obtained a value of 0.96 in pomace of Hamburg Muscat, a red grape variety. The low pH can also contribute to the microbiological stability of the product that, according to other studies, is around 3.5 [25], results similar to ours.

3.2. Effect of HHP on the major volatile compounds and ethanol of GP

The study of the changes in the volatile profile could be a useful tool to evaluate global variations in vegetable matrices and also could also evaluate new unexpected compounds that could be formed after processing [34]. Eight major volatile compounds were identified and quantified in the GPs (Table 2): six alcohols, one aldehyde and one ester. The most abundant compound isolated was ethanol and it was present in control and in treated pomace at similar levels. Only three volatile compounds were modified after HHP, acetaldehyde, methanol and 1-propanol. They significantly increased after HHP at 600 MPa/6 min (the most intense HHP conditions). In the case of methanol, one of the treatment (400 MPa/6min) also produced a significant reduction respect to the control pomace.

Table 4
Microbial counts (log colony forming units, CFU g^{-1}) of the high pressure treated white wine pomace stored at different temperatures.

	Temperature 4 °C						Temperature 20 °C					
	Control	400 MPa 1min	400 MPa 6min	600 MPa 1min	600 MPa 6min	*P-value*	Control	400 MPa 1min	400 MPa 6min	600 MPa 1min	600 MPa 6min	*P-value*
Mesophilics												
1d	4.1b[1]±0.1	3.4[12]±0.2	3.2[12]±0.1	3.2a[12]±0.1	2.8 ab[2]±0.8	*	4.1[1]±0.1	3.4c[12]±0.2	3.2d[12] ± 0.1	3.2b[12]±0.1	2.8b[2]±0.8	*
30d	3.3c±0.5	2.9 ± 0.4	3.0 ± 0.2	3.2a[12] ± 0.2	2.6b ±0.5	*ns*	6.1[12]±1.8	6.5a[1]±0.1	6.1a[12] ± 0.4	4.5a[12] ± 0.4	4.0a[2] ± 0.0	*
90d	5.8a[1]±0.5	2.8[2]±0.1	2.8[2] ± 0.3	<2b[3]	2.0b[3] ± 0.0	***	5.3[1] ± 0.2	5.4b[1] ± 0.2	4.4c[2] ± 0.1	2.0c[3] ± 0.0	2.0b[3] ± 0.0	***
180d	6.1a[1]±0.2	2.9[2]±0.2	3.1[2]±0.1	2.8a[2] ± 0.0	2.9 ab[2] ± 0.1	***	5.9[1] ± 0.0	5.2b[2] ± 0.1	4.7c[3] ± 0.2	2.0c[4] ± 0.0	2.0b[4] ± 0.0	***
270d	6.6a[1]±0.1	3.2[23]±0.1	3.5[23] ± 0.4	3.0a[3] ± 0.4	3.7a[2] ± 0.2	***	5.9[1] ± 0.1	5.2b[1] ± 0.3	5.3b[1] ± 0.2	2.5BCE[2] ± 0.9	2.4b[2] ± 0.3	**
P-storage	***	*	*ns*	***	**		*ns*	***	***	***	***	
Molds and yeasts												
1d	3.4 ab[1] ± 0.4	1.4b[2] ± 0.1	<1b[2]	<1b[2]	<1[2]	***	3.4b[1]±0.4	1.4c[2]±0.1	<1c[2]	<1c[2]	<1c[2]	***
30d	2.9b[1] ± 0.4	1.2b[2] ± 0.3	<1b[2]	<1b[2]	<1[2]	***	6.0a[12]±1.8	6.5a[1]±0.7	6.2a[12] ± 0.8	4.0b[2] ± 0.0	4.0a[2] ± 0.0	*
90d	3.1 ab[1]±0.1	<1b[2]	<1b[2]	<1b[2]	<1[2]	***	5.4a[2]±0.2	6.2a[1]±0.2	5.2 ab[2]±0.2	4.6a[3] ± 0.3	<2b[4]	***
180d	4.0a[1]±0.6	1.4b[2]±0.7	<1b[2]	<1b[2]	<1[2]	***	5.4a[1]±0.3	5.8 ab[1]±0.1	5.5 ab[1]±0.8	4.0b[2]±0.0	4.2a[2]±0.2	**
270d	3.4 ab[1]±0.3	2.5a[2]±0.4	2.3a[2]±0.3	2.2a[2]±0.4	1.3[3]±0.5	***	6.1a[1]±0.1	4.8b[12]±0.8	4.2b[2]±0.7	4.7a[12]±0.2	4.1a[2]±0.2	*
P-storage	*	**	***	***	*ns*		*	***	***	***	***	
Enterobacteriaceae												
1d	2.2b[1]±0.3	<1[2]	1.0[2]±0.0	1.0[2]±0.0	<1[2]	***	2.2b[1]±0.3	<1b[2]	1.0[2]±0.0	1.0[2]±0.0	<1[2]	***
30d	2.9a[1]±0.2	<1[2]	<1[2]	<1[2]	<1[2]	***	<1c	<1b	<1	<1	<1	*ns*
90d	2.1b[1]±0.4	<1[2]	<1[2]	<1[2]	<1[2]	***	3.0a[1]±0.1	2.9a[1]±0.3	1.1[2]±0.2	<1[2]	1.0[2]±0.0	***
180d	<1c	<1	<1	<1	<1	*ns*	3.2a[1]±0.1	1.0b[2]±0.0	1.0[2]±0.0	1.0[2]±0.0	1.0[2]±0.0	***
270d	<1c	<1	<1	<1	<1	*ns*	1.0c±0.0	1.0b ± 0.0	1.0 ± 0.0	1.0 ± 0.0	1.0 ± 0.0	*ns*
P-storage	***	*ns*	*ns*	*ns*	*ns*		***	***	*ns*	*ns*	*ns*	

ns. (non-significant differences). *p < 0.05 **p < 0.01 ***p < 0.001Different superscript numbers in the same row indicate significant differences in the Tukey test. Different letters in the same column indicate significant differences in the Tukey test.

Table 5
Instrumental colour parameters, phenolic compounds content (PCC, mg GAE 100g $^{-1}$ wet basis) and polyphenol oxidase enzyme (PPO, %) of the high pressure treated white wine pomace stored at different temperatures.

	Temperature 4 °C						Temperature 20 °C					
	Control	400 MPa 1 min	400 MPa 6 min	600 MPa 1 min	600 MPa 6 min	*P*	Control	400 MPa 1 min	400 MPa 6 min	600 MPa 1 min	600 MPa 6 min	*P*
L*												
1d	44.9 ± 1.5	43.5a±0.4	43.4 ± 0.9	44.0a ±0.4	44.1a ±0.3	ns	44.9a ±1.5	43.5 ± 0.3	43.4a ±0.9	44.0a ±0.3	44.1a ±0.2	ns
30d	43.3 ± 0.6	43.3a±0.4	43.1 ± 0.5	43.5 ab ± 0.3	43.7a ±0.5	ns	43.9 ab^{1}± 0.4	43.1^{12} ±0.7	42.0 ab^{2} ±0.3	42.4b^{2} ±0.6	42.3b^{2} ±0.4	*
90d	42.69 ± 0.7	41.9b ± 0.5	42.0 ± 0.7	42.1b ± 1.0	42.8b ± 0.6	ns	41.8BCE ±0.5	41.7 ± 0.5	41.6b ± 0.3	40.6c ±0.9	40.2d ± 0.5	*
180d	43.6 ± 0.4	42.3 ab ± 0.4	42.6 ± 1.0	42.8 ab ± 0.5	42.6b ± 0.2	ns	42.7abc^{1} ±0.5	42.6^{1}±0.7	42.2 ab^{12} ±0.4	41.1 ab^{2} ±0.4	41.2c^{2} ±0.5	*
270d	43.8^{1} ±0.3	42.1b^{2} ±0.5	42.5^{2} ±0.5	42.4 ab^{2} ±0.7	42.5b^{2} ±0.3	*	40.9c ±1.1	41.9 ± 1.1	41.1b ± 0.7	40.4c ±0.9	40.1d ± 0.3	ns
P	ns	**	ns	*	**		**	ns	**	***	***	
a*												
1d	7.6a ± 0.16	7.8a ± 0.2	8.00a ±0.2	7.8a ± 0.2	7.7a ± 0.1	ns	7.6a ± 0.16	7.8a±0.2	8.0a±0.2	7.8a±0.2	7.7a±0.1	ns
30d	7.6a ± 0.10	7.6 ab ± 0.1	7.54b ± 0.1	7.6a ± 0.2	7.5 ab ± 0.2	ns	6.8b ± 0.24	7.2b ± 0.1	6.7b ± 0.2	6.9b ± 0.2	7.0a±0.0	ns
90d	6.8b ± 0.21	7.0BCE ± 0.2	6.72c ± 0.2	6.9b ± 0.2	7.2b ± 0.0	ns	5.7c ± 0.40	5.3c ± 0.0	5.6c ± 0.3	5.7c ± 0.4	6.2b ± 0.6	ns
180d	6.8b ± 0.24	7.0BCE ± 0.2	6.96c ± 0.1	6.8b ± 0.3	6.6c ± 0.3	ns	5.7c ± 0.26	5.3c ± 0.4	5.4c ± 0.3	5.4c ± 0.3	5.5BCE ± 0.3	ns
270d	7.0b ± 0.13	6.7c ± 0.5	6.72c ± 0.1	6.9b ± 0.1	7.0BCE ± 0.2	ns	5.6c ± 0.35	5.7c ± 0.4	5.5c ± 0.4	5.6c ± 0.3	5.3c ± 0.1	ns
P	***	**	***	**	***		***	***	***	***	***	
b*												
1d	12.3a ± 0.46	12.7a ± 0.5	13.1 ± 0.6	12.5a ±0.6	12.4a ± 0.4	ns	12.3a ± 0.5	12.8a ± 0.5	13.0a ± 0.6	12.5a ± 0.6	12.4a ± 0.4	ns
30d	12.1a ± 0.34	12.0 ab ± 0.4	12.1b ± 0.1	11.9 ab ± 0.5	11.6 ab ± 0.6	ns	10.8a ± 0.5	11.5a ± 0.3	10.6b ± 0.3	11.0b ± 0.4	11.1b ± 0.1	ns
90d	10.3c ± 0.41	10.1c ± 0.5	10.0c ± 0.6	10.3c ± 0.6	11.1b ± 0.2	ns	8.9b ± 0.7	8.1b ± 0.1	8.7c ± 0.6	8.3c ± 0.6	8.5c ± 0.9	ns
180d	10.6BCE ± 0.39	11.0BCE ± 0.7	10.7c ± 0.3	10.7BCE ± 0.6	9.7c ± 0.7	ns	7.8b ± 0.5	8.0b ± 0.9	7.3c ± 0.7	7.67c ± 0.5	7.7cd ± 0.5	ns
270d	11.5 ab ± 0.51	10.5BCE ± 1.1	10.3c ± 0.2	10.7BCE ± 0.2	10.8BCE ± 0.4	ns	7.5b ± 1.0	8.3b ± 1.1	7.5c ± 0.8	7.2c ± 0.6	7.0d ± 0.3	ns
P	**	**	***	**	**		***	***	***	***	***	
PPC												
1d	457.4a ±23.5	463.5a ± 64.1	507.65a ± 67.1	506.45a ± 91.7	475.8a ± 111.6	*ns*	457.4a ± 23.5	463.5a ± 64.1	507.65a ± 67.1	506.45a ± 91.2	475.8a ± 111.6	*ns*
30d	354.9b^{2} ±30.5	394.8ab^{12} ± 51.0	456.9a^{1} ± 9.9	421.6a^{12} ± 6.8	357.6 ab^{2} ± 46.4	*	144.4b^{3} ± 57.2	211.5b^{23} ± 18.0	282.7b^{12} ± 60.4	315.3b^{1} ± 8.5	329.7b^{1} ± 27.4	**
90d	311.4b ± 15.2	270.5BCE ±83.4	332.4b ± 39.1	297.6b ± 43.9	373.8 ab ± 61.5	*ns*	137.9b^{1}± 10.3	139.8b^{1}± 34.6	104.9c^{12} ± 14.2	130.1c^{2} ± 10.7	77.6c^{2} ± 24.7	**
180d	219.7c ±28.3	275.7BCE ± 19.4	246.7b ± 85.3	243.1b ± 5.8	232.5b ± 21.7	*ns*	19.2c^{2} ± 10.8	29.0c^{2}± 11.3	44.2c^{2}± 50.0	93.9c^{1} ± 12.2	54.6c^{2} ± 3.7	***
270d	169.6c ±18.0	225.1c ±45.6	229.3b ± 15.0	222.1b ± 33.6	224.2b ± 18.7	*ns*	27.8c^{23} ±3.0	43.0c^{1} ±9.6	18.1c^{3} ±3.2	27.2c^{2} ±7.7	26.0c^{23} ±2.8	**
P	***	**	***	***	**		***	***	***	***	***	
PPO												
1d	100.0a ±46.5	81.6 ± 15.7	80.0 ± 7.1	64.3 ± 27.9	70.6 ± 21.6	*ns*	100.0a ±46.5	81.6a ±15.7	80a±7.1	64.3a ±27.9	70.6a ±21.6	ns

(*continued on next page*)

Table 5 (*continued*)

	Temperature 4 °C						Temperature 20 °C					
	Control	400 MPa 1 min	400 MPa 6 min	600 MPa 1 min	600 MPa 6 min	*P*	Control	400 MPa 1 min	400 MPa 6 min	600 MPa 1 min	600 MPa 6 min	*P*
90d	42.4b ± 18.7	37.6 ± 27.4	58.0 ± 9.5	46.3 ± 17.4	51.0 ± 27.3	*ns*	19.6b ± 1.4	18.8b ± 14.7	10.2b ± 7.6	13.3b ± 7.6	4.7b ± 0.0	ns
180d	26.7b ± 5.4	59.6 ± 47.1	58.8 ± 4.1	45.5 ± 15.7	54.9 ± 3.6	*ns*	0.0b ± 0.0	0.0b ± 0.0	0.0b ± 0.0	0.0b ± 0.0	0.0b ± 0.0	ns
270d	19.6b ± 6.8	49.4 ± 22.5	67.5 ± 19.0	50.2 ± 21.8	41.6 ± 16.5	*ns*	0.0b ± 0.0	0.0b ± 0.0	0.0b ± 0.0	0.0b ± 0.0	0.0b ± 0.0	ns
P	**	ns	ns	ns	ns		***	***	***	*	**	

ns. (non-significant differences). *p < 0.05 **p < 0.01 ***p < 0.001. Different superscript numbers in the same row indicate significant differences in the Tukey test. Different letters in the same column indicate significant differences in the Tukey test.

Cortés & Fernández [35],investigated the volatile compounds of distilled beverages obtained from different GPs and determined that ethanol and other longer-chain alcohols, esters, acids, and carbonyl compounds were the most abundant volatile compounds. Differences found between beverages were more quantitative than qualitative, and on other hand, these differences were caused by the quality of the raw material. In our case, the small differences found in the volatile composition of the samples should be caused by the HHP treatment. In this sense González-Cebrino et al. [36] reported that volatile compounds were well-preserved after HHP in plum purée at similar conditions as in this study. However, Cumplido-Laso et al. [37] reported unexpected changes in the volatile profile of pumpkin after HHP, probably due to the effect of the enzymatic activity, which could remain active after processing.

GP presented levels of ethanol which ranged between 2.4 and 2.8 % (w/w). GPs is generally stored in lots in an annex to the winery until it is processed, so spontaneous fermentation could appear in the original product at the beginning of storage. For that reason, volatile compounds derived from fermentation could also be isolated in the control GP. The levels of ethanol and other alcohols found in the GP (like methanol) were modified after HHP (increased or reduced), and these changes should be taken into account in case that the valorized product from GP was used as an ingredient for food manufacture. At high doses the valorized GP could affect the sensory characteristics of the final food, although probably the ingredient should be added at low doses due to its strong taste.

3.3. General effect of factors on GP

Table 3 provides a general overview of the effect of the different factors (processing, time of storage and temperature of storage) applied on the experimental results. The temperature and time of storage significantly affected ($p < 0.001$) all the studied parameters (microbiology, color, phenolics compounds content and enzyme activity), however, the HHP treatment significantly affected the counts of the different microorganisms and the colour parameter L*, but HHP did not affect the parameters CIE a* b*, PPC and PPO. All microbiological counts were affected by HHP treatments, the temperature and the time of storage, therefore, all factors could modify the shelf-life of the GP. Similarly, the colour parameters were affected by the temperature and the time of storage, which could reduce the appearance of the white wine pomace. However, the HHP treatment affected CIE L* parameter, but not CIE a* and b*. The PPC and the activity of the PPO enzyme were affected by the temperature and the time of storage, while the HHP treatment did not modify them.

Microorganisms' counts revealed significant three and two-way interactions among the three factors analyzed, which indicates that the effects analyzed shows interrelated relationships. Colour parameters, PCC and PPO presented significant interactions especially between the time and the temperature of storage. These factors are closely interconnected.

3.4. Effect of HHP and storage on the microbiology of GP

Counts of mesophilic aerobic microorganisms (Table 4) significantly lower after HHP at 600 MPa for 6 min than in the control (day 1); while the remaining treatments showed intermediate values. The counts of molds and yeasts and *Enterobacteriaceae* significantly decreased after any HHP conditions at day 1. The reductions of counts after processing were maintained at all sampling days during storage. *Enterobacteriaceae* was the most sensitive microbial group to HHP, and the most effective treatment was 600 MPa for 6 min, the most intense conditions of processing. In line with these results, the application of HHP was used to stabilize the grape before winemaking reducing effectively its microbial load, without affecting the valuable compounds of the product [17].

During refrigerated storage (4 °C), an increase in mesophilic counts and a decrease in *Enterobacteriaceae* counts were observed in the control group. However, at refrigeration the counts of the different HHP treatments fluctuated minimally, possibly because of competition between the different groups of microorganisms following the imbalance caused by the HHP treatment, which affects the microorganisms unevenly.

On the other hand, during the first storage period at 20 °C (day 1–30), the microbial counts of mesophilic aerobic (in HHP treated bags) and molds and yeasts increased (in control and HHP treated bags); however, after the first 30 days of storage (30–270 days), the counts in the HHP-treated bags decreased or stabilized, and at day 270, they were lower in the HHP-treated than in the control white wine pomace.

HHP show differences in effectiveness depending on pressure, time, and target microorganisms. The increase in mesophilic and molds and yeasts counts observed in storage at 20 °C during the first 30 days may be due to the presence of residual sugars in the

Table 6
Colour changes (ΔE) of the high pressure treated white wine pomace stored at different temperatures.

	Temperature 4 °C						Temperature 20 °C					
	Control	400 MPa 1 min	400 MPa 6 min	600 MPa 1 min	600 MPa 6 min	*P-value*	Control	400 MPa 1 min	400 MPa 6 min	600 MPa 1 min	600 MPa 6 min	*P-value*
ΔE processing	–	1.8 ± 1.4	2.2 ab ± 1.4	1.8 ab ± 1.0	1.4b ± 1.2	*ns*	–	1.8b ± 1.4	2.2c±1.4	1.8b ± 1.0	1.4b ± 1.2	*ns*
ΔE storage 1-30d	1.9 ± 1.8	1.0 ± 0.7	1.3b ± 0.2	0.8b ± 0.1	1.0b ± 0.4	*ns*	2.3b ± 1.3	1.6b ± 0.3	3.1BCE±0.5	2.4b ± 0.7	2.3b ± 0.3	*ns*
ΔE storage 1-90d	3.3 ± 1.6	3.2 ± 1.0	3.6a±1.1	3.2a±0.7	2.0 ab ± 0.2	*ns*	5.2 ab ± 1.5	5.6a±0.6	5.3 ab ± 0.9	5.9a±0.7	5.8a±1.3	*ns*
ΔE storage 1-180d	2.6 ± 0.6	2.2 ± 1.2	2.8 ab ± 0.3	2.4 ab ± 1.0	3.3a±0.6	*ns*	5.6 ab ± 1.2	5.5a±0.5	6.4a±1.0	6.2a±0.2	5.9a±0.5	*ns*
ΔE storage 1-270d	1.8 ± 1.5	2.9 ± 1.4	3.2 ab ± 0.3	2.6 ab ± 0.4	2.5 ab ± 0.5	*ns*	6.7a±1.5	5.2a±1.1	6.5a±1.1	6.8a±1.0	7.2a±0.2	*ns*
P-storage	*ns*	*ns*	*	*	*		*	***	***	***	***	

ns. (non-significant differences). *$p < 0.05$ **$p < 0.01$ ***$p < 0.001$. Different letters in the same column indicate significant differences in the Tukey test.

pomace [38]. The microorganisms would ferment these sugars until they are exhausted, resulting in stabilization of the microorganism's growth and even their decline. This would explain the first increase at the beginning storage period and the subsequent decline or stabilization after long storage times. In refrigerated storage, this fact is not observed since the fermenting microorganisms in the pomace would mainly be yeasts that have an optimum growth temperature of 25 °C [39].

From the point of view of the microbial stabilization of the plant material, the most suitable treatment is that of 600 MPa 6 min and storage at 4 °C. Although, at the end of storage at 20 °C the treated groups present acceptable counts, it should be considered that during the spontaneous fermentation that occurs mainly at first month of storage, and undesired metabolites (alcohol, …) can be produced and remain in the final product. Therefore low microbial counts in the pomace are desirable during all storage to avoid these changes and also to provide a safe ingredient that could be used for food industry.

3.5. Effect of HHP and storage on the instrumental colour of GP

Control white GP had values of L* = 44.9, a* = 7.6, b* = 12.3 and correspond to a red-brown visual colour (Table 5). The variations of the colour parameters that appeared during the study were mainly caused by temperature and storage time, while HHP did not affect colour parameters a* and b*, while it affected L* (Table 3). Few works have studied the instrumental colour of pomace. [26], obtained for the pomace of the red cultivar 'Isabel', a lightness value slightly higher than ours (L* = 47.15), and its colour coordinates (a* = 13.8 and b* = 4.81) which reflected the presence of anthocyanins, which are red pigments, characteristic of red-purple products.

HHP treatments did not cause significant changes on instrumental colour compared to the control at day 1. In general, during storage, changes occurred in all colour parameters with respect to their initial values, and these variations were more marked when the storage temperature was 20 °C. This could explain the interaction between pressure x temperature (Table 3).

During storage at 4 °C, lightness significantly decreased in samples treated at 400 MPa/1min and at 600 MPa/6 min. On the other hand, in general during storage at 20 °C, lightness decreased significantly in all samples, except for the 400 MPa 1 min. At refrigerated storage, a decrease in lightness was observed in packages treated at 400MPa/1min and at 600 MPa/1 and 6 min, however in control packages and those treated at 400MPa/6 min, the lightness remained unchanged. On the other hand, at 20 °C, a decrease in lightness was observed in all packages except in those treated at 400MPa/1min. Samples in which the luminosity was reduced during storage presented a darker colour at the end of storage.

CIE a* values were similar in control and treated samples at both temperatures and in all days of storage. CIE a* presented a significant decrease during storage, it was more marked when the temperature was 20 °C. The parameter CIE b* also presented a similar behavior, without differences between treatments and with a decrease in its value throughout storage, the greatest decreases were also at 20 °C. Reductions of a* and b*were related to less intensity of brown and yellow colours.

The colour differences (ΔE) globally show the changes in the instrumental colour with respect to a reference (Table 6). ΔE of processing compares colour changes of high pressure treated samples with respect to the control. Depending on the value of ΔE, the colour difference between the treated and untreated samples can be estimated as not noticeable (0–0.5), slightly noticeable (0.5–1.5), noticeable (1.5–3.0), well visible (3.0–6.0) and great (6.0–12.0) [8]. In this case, colour changes after HHP (ΔE processing) in white wine pomace were similar after all HHP treatments ($p > 0.05$), and they could be considered slightly noticeable or noticeable. The parameter ΔE storage compares changes during storage respect to the initial at the HHP conditions. All calculated ΔE storage were similar ($p > 0.05$) in control and all HHP treatments at the same temperature of storage at any sampling day. At 4 °C, ΔE values after processing and storage were similar in control and HHP at 400 MPa/1min (P-value >0.05 within the same column), however at 400 MPa/6min and 600 MPa/1min the highest value was found at 90 days of storage, and at 600 MPa/6min the highest value was found at 180 days of storage. At 20 °C, ΔE values processing/storage were similar in control (P-value >0.05 within the same column), however in the HHP treated samples the highest values were found at 90–180 days of storage. In general ΔE storage was higher at 20 °C than at 4 °C. Concretely, after 270 days of storage at 4 °C, the ΔE storage 1-270d ranged from 1.8 (control) to 3.2 (400 MPa/6min), so the colour changes at 4 °C would be estimated as noticeable. On the other hand, the changes of colour during storage at 20 °C ranged from 5.2 (400 MPa/1min) to 7.2 (600 MPa/6min) and they could be considered well visible and great. Therefore, processing changes could be considered insignificant compared to storage colour changes. In addition, the control pomace preserved at refrigeration conditions was which best maintained the original colour of milled pomace.

Changes in colour of vegetable products during storage are generally associated with the action of oxidative enzymes, like polyphenol oxidase or peroxidases which favor the development of dark-coloured pigments. This would explain the reductions of CIE L*, a* and b*. The milled pomace turned to dark brown colour. In other vegetable purée products treated by the HHP, colour changes during storage had been explained by the activity of the PPO enzyme after processing and during storage [36,40]. However, vegetable products generally do not reach such long storage times as pomace.

3.6. Effect of HHP and storage on the total phenolic compounds content and polyphenol oxidase activity of GP

The changes of phenolic compounds content (PCC) and polyphenol oxidase enzyme activity (PPO) were only affected by the temperature and the time of storage (Table 5), while the HHP treatment did not modify the initial contents.

On day 1, the control (457 mg GAE $100g^{-1}$ wb/1056 mg GAE $100g^{-1}$ dm) presented similar values as the HHP-treated samples. The literature reports variable contents of phenolic compounds, since they depend on the composition of the grapes and the wine-making process, with very wide ranges of variation [2,4,10,23]. On the other hand, the data found in the bibliography is sometimes difficult to compare, due to the great variability among pomace composition, analysis methods, sample moisture or whether the results are expressed on a dry or wet basis. Thus, (Llobera & Cañellas [27] obtained a value of 3490 mg GAE 100 g^{-1} dm, for a white grape

pomace, a value greater than ours. In contrast, Shirahigue et al. [41] obtained values of 430.6 and 522.2 mg GAE 100 g $^{-1}$ dm in pomace dried at 40 °C and ground, from the winemaking grapes of the cultivars 'Isabel' and 'Niagara', similar values as in our study. Another study analyzed the content of total phenolic compounds of five white wine pomace, which ranged between 1160 and 2670 mg GAE 100 g^{-1} dm, showing that the grape cultivar is important in the concentration of these compounds [31].

The degradation of phenolic compounds was greater as storage progresses and is more marked at temperatures of 20 °C than at 4 °C. When the storage was performed at 4 °C, the degradation of phenolic compounds was slightly lower in all the treated samples (44–49 % of retention at 270 d respect of the initial concentration) and in the control (37 % of retention at 270 d), although no significant differences were found in the PCC between control and treated pomace at the end of storage. The application of the most intense processing conditions of HHP (at 600 MPa/6 min) and the refrigeration of the treated pomace would allow obtaining a microbiologically safe pomace with high levels of phenolic compounds until the 90 days of storage. On the other hand, for storage at 20 °C, the degradation was very marked at day 30 and the most notable drop was in control, and continued throughout storage, dropping again markedly on day 180, with very low values that were maintained until the end of storage (day 270) and ranged between 3.5 and 9 % of their initial value. Therefore, it can be affirmed that phenolic compounds were better retained at refrigeration. Since the pomace is a seasonal product, it needs long times of storage to be available all the year. Long times of storage at room temperature produce a negative effect on pomace since more than the 90 % of the phenolic compounds are lost while refrigeration preserves better them.

In addition to fiber, the high concentration of phenolic compounds is very interesting in pomace, and both fractions have functional effects when incorporated into the diet [4,10]; therefore, the direct use of pomace in food preparation is considered a good alternative. The functional properties of phenolic compounds are very diverse, although the best known is that of acting as antioxidants, they also have antimicrobial and anti-inflammatory capacity, and their antioxidant capacity has been related to the prevention of certain degenerative diseases.

There are few studies on the effect of HHP on the concentration of polyphenols in the pomace. HHP has been shown to increase the extraction of anthocyanins from pomace [42–44], which can be an advantage when it is directly incorporated into a food. Corrales et al. [45] optimized the extraction of anthocyanins in pomace from red grape skins, achieving a significant increase when they applied HHP, regardless of the extraction temperature [17]. found that the application to grape berries of HHP at 200 MPa, prior to winemaking, improved extraction of anthocyanins. However, in our results this effect was not observed since PCC at day 1 were statistically similar control and treated pomace. Differences could be caused in the grape varieties, since those studies were carried out in red wine varieties, with high polyphenols content.

On the other hand, HHP treatments did not affect the activity of the polyphenol oxidase enzyme (PPO) (Tables 3 and 5) and similar percentages were found between control and treated pomace at all sampling times. When samples were stored at 20 °C, there was a drastic drop in PPO activity, and no activity was detected from the day 90 for all samples. At 4 °C, the enzymatic activity did not decrease during storage, except for the control. No significant differences were observed between the treated samples in PPO enzyme activity during storage. PPO is an undesirable enzyme in foods, as degrade the polyphenol compounds by oxidation and leads to browning processes that affect to the colour. The great reduction of the PPO activity at 20 °C would be explained by the great reduction of PCC at that temperature the principal substrate of the enzyme. In vegetables, PPO activity is usually controlled by the effect of the temperature, inactivating it at temperatures above 60 °C, or reducing the Aw of the products. PPO slightly decreased during refrigerated storage in grape juice treated by HHP [46]. Ranveer et al. [47] reported a decrease of PPO during 60 days of storage of dried grapes and the decrease was more marked at room temperature than at refrigeration. The causes of the decrease are not known, but probably the enzyme could be degraded or react with other components during storage.

Previous studies in purée of "Songold" Plum, "Grimson glove" plum and in pumpkin [36,48,49] reported no effect of HHP on the activity of the PPO and thus the enzyme would continue active during the storage of the product. In line with our results [50–52,], studied the effect of HHP on the stability of a nectarine/plum purée and suggested the need of application of a thermal blanching in HP-treated products to inactivate the PPO enzyme when it is not reduced after HHP application. Moreover, the addition of ascorbic acid to the vegetables processed by HHP also protects the degradation of PCC In this case, since HHP did not reduce the PPO enzyme, the application of a thermal blanching before HHP would be positive to preserve PCC of the pomace.

4. Conclusions

Hydrostatic high pressure maintained the bioactive compounds content in the white wine pomace after processing. However, an important reduction of phenolic compounds content was found during storage, especially at 20 °C. The polyphenol oxidase enzyme was not reduced after the high-pressure treatment and remained active during storage. In case that pomace was be applied as an ingredient (with antioxidant and/or antimicrobial activity) for the manufacture of other food products, it should have a long shelf-life (of around a year) since this a seasonal by-product. High hydrostatic pressure maintained the microbiological safety of the milled pomace for at least 9 months (at refrigeration and at room temperature). The application of high hydrostatic pressure at 600 MPa/6 min and the refrigeration of the treated pomace would allow obtaining a microbiologically safe pomace with high levels of phenolic compounds, although the reductions are important after 90 days of storage. However, the treatments did not reduce the polyphenol oxidase enzyme activity. Therefore, to obtain an ingredient from the pomace by high pressure processing, a pretreatment should be applied before processing, like a thermal blanching to ensure the inactivation of the enzyme and to preserve the bioactive compounds content during storage. In that case, high hydrostatic pressure would allow an integral re-utilization of the pomace. This would be a clean technology since no solvents are required and no waste products are generated in this new valorization process. Cost-benefits of high hydrostatic pressure application respect to other traditional treatments should be evaluated, although the economic cost of the treatment are being reduced each year due to its implementation at an industrial level.

Regarding the conditions to store the pomace ingredient, the storage at room temperature was not suitable (at least for long-term storages) since it produced great reductions of the phenolic compounds. Since the final valorized pomace should have microbiological safety, high bioactive compounds content and a long shelf-life, refrigerated storage should be recommended to preserve the phenolic compounds, the principal bioactive compounds in pomace.

CRediT authorship contribution statement

Rosario Ramírez: Conceptualization, Funding acquisition, Supervision, Writing – review & editing. **Jonathan Delgado:** Investigation, Methodology, Supervision. **Javier Rocha-Pimienta:** Formal analysis, Methodology, Writing – review & editing. **M. Esperanza Valdés:** Formal analysis, Methodology, Writing – review & editing. **María Jesús Martín-Mateos:** Data curation, Formal analysis, Methodology. **M. Concepción Ayuso-Yuste:** Investigation, Writing – original draft, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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References

[1] FEV, FEV, Retrieved from, 2022, https://www.fev.es/es/. Accessed June 2022FEV, 2022.
[2] C. Beres, G.N.S. Costa, I. Cabezudo, N.K. da Silva-James, A.S.C. Teles, A.P.G. Cruz, C. Mellinger-Silva, R.v. Tonon, L.M.C. Cabral, S.P. Freitas, Towards integral utilization of grape pomace from winemaking process: a review, in: Waste Management, vol. 68, Elsevier Ltd, 2017, pp. 581–594, https://doi.org/10.1016/j.wasman.2017.07.017.
[3] K. Dwyer, F. Hosseinian, M. Rod, The Market potential of grape waste alternatives, J. Food Res. 3 (2) (2014) 91, https://doi.org/10.5539/jfr.v3n2p91.
[4] J. García-Lomillo, M.L. González-SanJosé, Applications of wine pomace in the food industry: approaches and functions, Compr. Rev. Food Sci. Food Saf. 16 (1) (2017) 3–22, https://doi.org/10.1111/1541-4337.12238.
[5] M.M. Martínez Salgado, R. Ortega Blu, M. Janssens, P. Fincheira, Grape pomace compost as a source of organic matter: Evolution of quality parameters to evaluate maturity and stability, J. Clean. Prod. 216 (2019) 56–63, https://doi.org/10.1016/j.jclepro.2019.01.156.
[6] T. Baumgärtel, H. Kluth, K. Epperlein, M. Rodehutscord, A note on digestibility and energy value for sheep of different grape pomace, Small Rumin. Res. 67 (2–3) (2007) 302–306, https://doi.org/10.1016/j.smallrumres.2005.11.002.
[7] M.R. González-Centeno, Caracterización de los subproductos de la industria vitivinícola como fuente de fibra dietética y compuestos fenólicos. Uso de los ultrasonidos de potencia para la extracción de la fracción fenólica (Tesis de doctorado), UNIVERSITAT DE LES ILLES BALEARS Departamento de Química, 2013.
[8] F.J. Barba, Z. Zhu, M. Koubaa, A.S. Sant'Ana, V. Orlien, Green alternative methods for the extraction of antioxidant bioactive compounds from winery wastes and by-products: a review, Trends Food Sci. Technol. 49 (2016) 96–109, https://doi.org/10.1016/j.tifs.2016.01.006.
[9] D.P. Makris, Green extraction processes for the efficient recovery of bioactive polyphenols from wine industry solid wastes–Recent progress, Curr. Opin. Green Sustainable Chem. 13 (2018) 50–55, https://doi.org/10.1016/j.cogsc.2018.03.013.
[10] B. Antonić, S. Jančíková, D. Dordević, B. Tremlová, Grape pomace valorization: a systematic review and meta-analysis, Foods 9 (Issue 11) (2020), https://doi.org/10.3390/foods9111627. MDPI AG.
[11] C. Beres, G.N. Costa, I. Cabezudo, da Silva-James, A.S. Teles, A.P. Cruz, S.P. Freitas, Towards integral utilization of grape pomace from winemaking process: A review, Waste Manag 68 (2017) 581–594.
[12] A.M. Goula, K. Thymiatis, K. Kaderides, Valorization of grape pomace: drying behavior and ultrasound extraction of phenolics, Food Bioprod. Process. 100 (2016), https://doi.org/10.1016/j.fbp.2016.06.016.
[13] J.A. Larrauri, P. Rupérez, F. Saura-Calixto, Effect of drying temperature on the stability of polyphenols and antioxidant activity of red grape pomace peels, J. Agric. Food Chem. 45 (4) (1997), https://doi.org/10.1021/jf960282f.
[14] M. Cano-Lamadrid, F. Artés-Hernández, By-products revalorization with non-thermal treatments to enhance phytochemical compounds of fruit and vegetables derived products: a review, Foods 11 (Issue 1) (2022), https://doi.org/10.3390/foods11010059. MDPI.
[15] S. Augustine, V.B. Kudachikar, V. Vanajakshi, R. Ravi, Effect of combined preservation techniques on the stability and microbial quality and retention of anthocyanins in grape pomace stored at low temperature, J. Food Sci. Technol. 50 (2) (2013) 332–338, https://doi.org/10.1007/s13197-011-0325-0.
[16] F. Cacace, E. Bottani, A. Rizzi, G. Vignali, Evaluation of the economic and environmental sustainability of high pressure processing of foods, Innovative Food Sci. Emerging Technol. 60 (2020). https://doi.org/10.1016/j.ifset.2019.102281.
[17] A. Morata, I. Loira, R. Vejarano, M.A. Bañuelos, P.D. Sanz, L. Otero, J.A. Suárez-Lepe, Grape processing by high hydrostatic pressure: effect on microbial populations, phenol extraction and wine quality, Food Bioprocess Technol. 8 (2) (2015) 277–286, https://doi.org/10.1007/s11947-014-1405-8.
[18] AOAC, AOAC International, G.W.J. Latimer, Official methods of analysis of AOAC International, 2016, AOAC International, 2016 (20th ed.).
[19] J. Folch, M. Lees, G.H. Sloane Stanley, A simple method for the isolation and purification of total lipids from animal tissues, J. Biol. Chem. 226 (1957) 497–509.
[20] P.J. van Soest, R.H. Wine, Use of detergents in the analysis of fibrous feeds. IV. Determination of plant cell-wall constituents, J. AOAC Int. 50 (1) (1967), https://doi.org/10.1093/jaoac/50.1.50.
[21] V.L.A.G. Lima, E.A. Mélo, M.I.S. Maciel, F.G. Prazeres, R.S. Musser, D.E.S. Lima, Total phenolic and carotenoid contents in acerola genotypes harvested at three ripening stages, Food Chem. 4 (90) (2005) 565–568, https://doi.org/10.1016/J.FOODCHEM.2004.04.014.
[22] N.S. Terefe, K. Matthies, L. Simons, C. Versteeg, Combined high pressure-mild temperature processing for optimal retention of physical and nutritional quality of strawberries (Fragaria × ananassa), Innovative Food Sci. Emerging Technol. 10 (3) (2009) 297–307, https://doi.org/10.1016/j.ifset.2008.12.003.
[23] Q. Jin, J. O'Hair, A.C. Stewart, S.F. O'Keefe, A.P. Neilson, Y.T. Kim, M. McGuire, A. Lee, G. Wilder, H. Huang, Compositional characterization of different industrial white and red grape pomaces in Virginia and the potential valorization of the major components, Foods 8 (12) (2019), https://doi.org/10.3390/foods8120667.
[24] L. Taşeri, M. Aktaş, S. Şevik, M. Gülcü, G. Uysal Seçkin, B. Aktekeli, Determination of drying kinetics and quality parameters of grape pomace dried with a heat pump dryer, Food Chem. 260 (2018) 152–159, https://doi.org/10.1016/j.foodchem.2018.03.122.

[25] H. Javier, S.J. Ángel, G. Aida, G.M. del Carmen, M.M. de los Ángeles, Revalorization of grape marc waste from liqueur wine: biomethanization, J. Chem. Technol. Biotechnol. 94 (5) (2019), https://doi.org/10.1002/jctb.5909.
[26] L.P. Cilli, L.R.F. Contini, P. Sinnecker, P.S. Lopes, M.A. Andreo, C.R.P. Neiva, M.S. Nascimento, C.M.P. Yoshida, A.C. Venturini, Effects of grape pomace flour on quality parameters of salmon burger, J. Food Process. Preserv. 44 (2) (2020), https://doi.org/10.1111/jfpp.14329.
[27] A. Llobera, J. Cañellas, Antioxidant activity and dietary fibre of Prensal Blanc white grape (Vitis vinifera) by-products, Int. J. Food Sci. Technol. 43 (11) (2008), https://doi.org/10.1111/j.1365-2621.2008.01798.x.
[28] E.C. Sousa, A.M.A. Uchôa-Thomaz, J.O.B. Carioca, S.M. de Morais, A. de Lima, C.G. Martins, C.D. Alexandrino, P.A.T. Ferreira, A.L.M. Rodrigues, S.P. Rodrigues, J. do N. Silva, L.L. Rodrigues, Chemical composition and bioactive compounds of grape pomace (Vitis vinifera L.), Benitaka variety, grown in the semiarid region of Northeast Brazil, Food Sci. Technol. 34 (1) (2014), https://doi.org/10.1590/S0101-20612014000100020.
[29] M. Šporin, M. Avbelj, B. Kovač, S.S. Možina, Quality characteristics of wheat flour dough and bread containing grape pomace flour, Food Sci. Technol. Int. 24 (3) (2018), https://doi.org/10.1177/1082013217745398.
[30] C. Beres, G.N. Costa, I. Cabezudo, da Silva-James, A.S. Teles, A.P. Cruz, S.P. Freitas, Towards integral utilization of grape pomace from winemaking process: A review, Waste Manag 68 (2017) 581–594.
[31] Q. Deng, M.H. Penner, Y. Zhao, Chemical composition of dietary fiber and polyphenols of five different varieties of wine grape pomace skins, Food Res. Int. 44 (9) (2011) 2712–2720, https://doi.org/10.1016/j.foodres.2011.05.026.
[32] P. Rondeau, F. Gambier, F. Jolibert, N. Brosse, Compositions and chemical variability of grape pomaces from French vineyard, Ind. Crop. Prod. 43 (1) (2013) 251–254, https://doi.org/10.1016/j.indcrop.2012.06.053.
[33] N.G. Baydar, G. Özkan, E. Çetin, S.Characterization of grape seed and pomace oil extracts, Grasas y aceites 58 (1) (2007) 29–33.
[34] L. Vervoort, T. Grauwet, B.T. Kebede, I. van der Plancken, R. Timmermans, M. Hendrickx, A. van Loey, Headspace fingerprinting as an untargeted approach to compare novel and traditional processing technologies: a case-study on orange juice pasteurisation, Food Chem. 134 (4) (2012) 2303–2312, https://doi.org/10.1016/j.foodchem.2012.03.096.
[35] S. Cortés, E. Fernández, Differentiation of Spanish alcoholic drinks, orujo, obtained from red and white grape pomace distillation: volatile composition, Int. J. Food Prop. 14 (6) (2011) 1349–1357, https://doi.org/10.1080/10942911003654025.
[36] F. González-Cebrino, J. García-Parra, R. Ramírez, Aroma profile of a red plum purée processed by high hydrostatic pressure and analysed by SPME-GC/MS, Innovative Food Sci. Emerging Technol. 33 (2016) 108–114, https://doi.org/10.1016/j.ifset.2015.11.008.
[37] G. Cumplido-Laso, F. González-Cebrino, J.J. García-Parra, M.R. Ramírez, High Hydrostatic Pressure Processing of Pumpkin: Identification and Quantification of the Volatiles Profile, 2022.
[38] D. Canalejo, Z. Guadalupe, L. Martínez-Lapuente, B. Ayestarán, S. Pérez-Magariño, Optimization of a method to extract polysaccharides from white grape pomace by-products, Food Chem. 365 (2021), https://doi.org/10.1016/j.foodchem.2021.130445.
[39] F.J. Heredia, M.L. Escudero-Gilete, D. Hernanz, B. Gordillo, A.J. Meléndez-Martínez, I.M. Vicario, M.L. González-Miret, Influence of the refrigeration technique on the colour and phenolic composition of syrah red wines obtained by pre-fermentative cold maceration, Food Chem. 118 (2) (2010) 377–383, https://doi.org/10.1016/j.foodchem.2009.04.132.
[40] K.R. Gopal, A.M. Kalla, K. Srikanth, High pressure processing of fruits and vegetable products: a review, International Journal of Pure & Applied Bioscience 5 (5) (2017) 680–692, https://doi.org/10.18782/2320-7051.2930.
[41] L.D. Shirahigue, M. Plata-Oviedo, S.M. de Alencar, M.A. Bismara Regitano d'Arce, T.M. Ferreira de Souza Vieira, T.L. Cadorin Oldoni, C.J. Contreras-Castillo, Wine industry residue as antioxidant in cooked chicken meat, Int. J. Food Sci. Technol. 45 (5) (2010) 863–870, https://doi.org/10.1111/j.1365-2621.2010.02201.x.
[42] A.S. Cascaes Teles, D.W. Hidalgo Chávez, M.A. Zarur Coelho, A. Rosenthal, L.M. Fortes Gottschalk, R.V. Tonon, Combination of enzyme-assisted extraction and high hydrostatic pressure for phenolic compounds recovery from grape pomace, J. Food Eng. 288 (2020), https://doi.org/10.1016/j.jfoodeng.2020.110128.
[43] M. Corrales, S. Toepfl, P. Butz, D. Knorr, B. Tauscher, Extraction of anthocyanins from grape by-products assisted by ultrasonics, high hydrostatic pressure or pulsed electric fields: a comparison, Innovative Food Sci. Emerging Technol. 9 (1) (2008) 85–91, https://doi.org/10.1016/j.ifset.2007.06.002.
[44] P. Putnik, D. Bursać Kovačević, D. Ježek, I. Šustić, Z. Zorić, V. Dragović-Uzelac, High-pressure recovery of anthocyanins from grape skin pomace (Vitis vinifera cv. Teran) at moderate temperature, J. Food Process. Preserv. 42 (1) (2018), https://doi.org/10.1111/jfpp.13342.
[45] M. Corrales, A.F. García, P. Butz, B. Tauscher, Extraction of anthocyanins from grape skins assisted by high hydrostatic pressure, J. Food Eng. 90 (4) (2009) 415–421, https://doi.org/10.1016/j.jfoodeng.2008.07.003.
[46] D. Del Pozo-Insfran, A. Del Follo-Martinez, S.T. Talcott, C.H. Brenes, Stability of copigmented anthocyanins and ascorbic acid in muscadine grape juice processed by high hydrostatic pressure, J. Food Sci. 72 (4) (2007) S247–S253, https://doi.org/10.1111/j.1750-3841.2007.00316.x.
[47] R.C. Ranveer, V.N. Pawar, B.K. Sakhale, A. Sahoo, K.Effect of storage conditions on the residual polyphenol oxidase (PPO) activity of raisins, Int. J. Agric. Sci. 6 (1) (2010) 61–64.
[48] F. González-Cebrino, J. García-Parra, R. Contador, R. Tabla, R. Ramírez, Effect of high-pressure processing and thermal treatment on quality attributes and nutritional compounds of "songold" plum purée, J. Food Sci. 77 (8) (2012), https://doi.org/10.1111/j.1750-3841.2012.02799.x.
[49] F. González-Cebrino, R. Durán, J. Delgado-Adámez, R. Contador, R. Ramírez, Changes after high-pressure processing on physicochemical parameters, bioactive compounds, and polyphenol oxidase activity of red flesh and peel plum purée, Innovative Food Sci. Emerging Technol. 20 (2013) 34–41, https://doi.org/10.1016/j.ifset.2013.07.008.
[50] J. García-Parra, F. González-Cebrino, J. Delgado, M. Lozano, T. Hernández, R. Ramírez, Effect of thermal and high-pressure processing on the nutritional value and quality attributes of a nectarine purée with industrial origin during the refrigerated storage, J. Food Sci. 76 (4) (2011), https://doi.org/10.1111/j.1750-3841.2011.02129.x.
[51] J. García-Parra, F. González-Cebrino, J. Delgado-Adámez, R. Cava, O. Martín-Belloso, P. Élez-Martínez, R. Ramírez, Effect of high-hydrostatic pressure and moderate-intensity pulsed electric field on plum, Food Sci. Technol. Int. 24 (2) (2018) 145–160, https://doi.org/10.1177/1082013217735965.
[52] Baydar, et al., Characterization of grape seed and pomace oil extracts, Grasas Aceites 58 (1) (2007) 28–33, https://doi.org/10.3989/gya.2007.v58.i1.5.