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Sustainable mitigation of acrylamide formation during Maillard reaction using grape pomace extracts obtained by green pressurized liquid extraction

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Acrylamide (AA) is a food neo-contaminant classified as a probable human carcinogen formed during the Maillard reaction. Natural (poly)phenolic extracts have emerged as promising mitigation strategies to inhibit AA formation. In this study, pressurized liquid extraction (PLE) conditions were optimized to obtain grape pomace extracts with maximum AA mitigation. Temperature (50–180 °C) and ethanol percentage (0–100% v/v) were optimized using a central composite design, finding an optimal response at 180 °C and 21% ethanol, with which AA formation was decreased by 33.36 ± 3.31% in the asparagine–glucose (ASN–GLC) model that emulates the Maillard reaction. This extract (0.1–0.8%) was also assayed in a bread model, reducing AA formation by 41.25 ± 9.36% when used at 0.4% w/w. Epicatechin, gallic acid, and catechin were the predominant (poly)phenols, and their individual effect on AA formation showed reductions from 35.46 ± 1.30% to 42.83 ± 10.94% in the ASN–GLC model. These findings confirm that PLE grape pomace extracts can successfully mitigate AA formation mainly through catechin, epicatechin, and gallic acid.

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Sustainability spotlight

More than 10 million tons of grape pomace are generated worldwide each year as a major by-product of the winemaking industry. Although grape pomace constitutes a valuable source of bioactive compounds with significant potential for valorization, it is frequently underutilized and often regarded as waste. In line with Sustainable Development Goal (SDG) 12 (Responsible Consumption and Production), the present study reports a practical example of a circular economy strategy to grape pomace. Through the implementation of a sustainable extraction process based on an advanced green technology (supporting SDG 9, Industry, Innovation and Infrastructure), this abundant winery by-product was successfully valorized into a high-value functional ingredient rich in (poly)phenols, which was succesfully mitigated acrylamide formation in food systems, contributing to SDG 3 (Good Health and Well-Being).

1 Introduction

The Maillard reaction is a non-enzymatic browning process that happens during food thermal processing.[1] The initial reaction occurs between reducing sugars and amino groups present in amino acids, peptides, and proteins.[2] The Maillard reaction is highly desirable because produces flavors, aromas, and colors in typical foods like bread, coffee, potato chips, *etc.* However, in the presence of asparagine, it also generates neo-contaminants such as acrylamide (AA),[3] which is classified by the International Agency for Research on Cancer as a probable human carcinogen (Group 2A), and it has also been associated with other adverse consequences like neurotoxic and genotoxic effects.[4] Natural products might play an important role in reducing AA content, particularly the antioxidant compounds, which, through the reaction with α-dicarbonyl compounds, decelerate the chemical reaction responsible for AA formation.[5] Additionally, antioxidants can block the reaction's propagation through the interaction between the quinone ring and free amino groups, thereby avoiding the formation of intermediates such as Amadori products. These intermediates are readily oxidized, generating free radicals and reactive carbonyl compounds such as glyoxal and methylglyoxal, intensifying the Maillard reaction.[2] Likewise, antioxidants can bind to these intermediates and prevent the formation of Strecker aldehydes, which also

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contribute to generate AA.[6] Antioxidants can also participate *via* Michael reaction with the already formed AA, binding to its carbon–carbon (C=C) double bond through nucleophilic groups, facilitating its neutralization.[6] (Poly)phenols are widely recognized for their antioxidant properties, closely related to their chemical structure, particularly to the number of hydroxyl groups and the degree of glycosylation.[7] Due to these structural characteristics, they can act as free radical scavengers and hydrogen donors, interacting with Maillard reactive intermediates, decreasing the AA formation.[8] Some extracts rich in (poly)phenol compounds obtained from matrices such as tea,[9] coffee,[10] olive leaves, potato peels, pomegranate peels, and lemon peels,[11] and olive by-products[12] have been evaluated as natural strategies to reduce the AA formation in thermally processed foods, producing relative decreases in its formation. Grape pomace is also a rich source of bioactive compounds, particularly (poly)phenols.[13,14] Xu *et al.*[15] reported the mitigating effect of Muscadine grape seeds and skin extracts on AA formation. The reduction observed with both extracts was higher than 60% and 90% in Maillard and potato chip models, respectively. The study also described the inhibitory effect dependence on the type of (poly)phenol. It is well-known that the extraction method significantly affects the type and quantity of (poly)phenols obtained, and both are strongly influenced by the extraction mechanism, and parameters like time, temperature, type of solvent, *etc.*[16,17] Therefore, optimizing the extraction conditions *via* design of experiments is essential to achieve the best results; particularly relevant is the choice of the response variables. In the present work, a unique approach was used, defining AA formation as the critical response variable. Amongst the most used designs of experiment, a central composite design (CCD) was selected given its high efficiency with a reduced number of experiments.[18] CCD has been utilized for optimizing several analytical processes, such as (bio)chemical reactions[19,20] and bioactive molecules extraction.[21,22] The optimal (poly)phenol extraction yield should be achieved using green extraction conditions. In this context, it is of high interest to move forward from conventional extraction techniques like maceration, agitation, and reflux extraction to advanced and sustainable extraction technologies such as pressurized liquid extraction (PLE), supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), and ultrasound-assisted extraction (UAE) that present several advantages such as higher yields, reduced extraction time, and lower solvent volumes.[23] Among them, PLE emerges as a robust and useful alternative because it is an environmentally friendly technique, uses small volumes of green and GRAS solvents like water, and delivers faster extractions due to an enhanced mass transfer rate and higher matrix penetration.[24] PLE operates under high pressure and temperature, keeping the solvent in the liquid state even above its normal boiling point. Under subcritical conditions, the dielectric constant of water decreases, enabling the solubilization of less polar compounds behaving like an organic solvent.[25] Since temperature and solvent type significantly affect extraction efficiency and selectivity, they should be optimized simultaneously. Increasing temperature improves extraction by reducing the viscosity and surface tension of the solvent. This facilitates solvent diffusion and mass transfer into the plant matrix, resulting in higher extraction yields.[26] Additionally, the polarity of the solvent determines the type of (poly)phenol compound recovered.[27] Water favors the extraction of highly polar compounds (phenolic acids and anthocyanins); while methanol and ethanol extract a wider range of (poly)phenols (phenolic acids and flavonoids); on the other hand, aqueous ethanol improves the recovery of compounds of intermediate polarity (flavan-3-ols, flavonol glycosides); and solvents such as acetone favor the extraction of less polar compound (flavanols).[28–30] Owing to these advantages, PLE has been widely used to extract antioxidant compounds from wine residues and other natural product sources.[31,32] The objective of this work was to optimize the PLE of (poly)phenols from grape pomace to maximize its mitigation effect on AA formation in an asparagine–glucose (ASN–GLC) model and then confirm it on a bread model system. Additionally, the role of individual phenolic compounds on AA mitigation in the ASN–GLC model was also assessed based on the (poly)phenolic profile of the extracts, offering a more integrated understanding of grape pomace as a natural source of bioactive molecules for reducing the formation of food neo-contaminants.

2 Material and methods experimental

2.1 Raw material, reagents, chemicals, and standards

Cabernet Sauvignon grape pomace (skins, seeds, and pulp residues) was provided by Viña Chillán (6°46′44.0″S 72° 13′18.1″W). The pomace was first dried at 40 °C for 6 h in a Binder (Tuttlingen, Germany) oven to remove ethanol and then freeze-dried for 72 h at −55 °C using a Martin Christ (Osterode am Harz, Germany) Alpha 1–2 LD plus freeze dryer. After drying, samples were milled into a fine powder using a laboratory grinder and stored protected from light at −20 °C in sealed polyethylene bags. Ethanol, acetonitrile (MS-grade), formic acid (p.a. & MS-grade), Folin-Ciocalteu reagent, 2,2-diphenyl-1-picrylhydrazyl (DPPH), 2,2′-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), gallic acid, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox), D-(+)-glucose monohydrate, L-asparagine monohydrate, AA, and AA-$^{13}C_3$ standards were purchased from Merck (Darmstadt, Germany). Polyvinylidene difluoride (PVDF) 13 mm syringe filters (0.22 μm) were obtained from Whatman (NJ, USA). Ultrapure water (18 MΩ cm 25 °C) was produced using a PURIST Ultrapure Water System.

2.2 Pressurized liquid extraction (PLE)

(Poly)phenol compounds were extracted following the procedure described by Peterssen-Fonseca *et al.*[24] using a Dionex (Sunnyvale, CA, USA) ASE-200 pressurized liquid extractor equipped with 11 mL stainless steel extraction cells containing a cellulose filter at the base. Extractions were carried out at different temperatures and ethanol percentages in water according to the experimental design described in 2.3. For each extraction, 1 g of dry sample was mixed with 2 g of sea sand (dispersing agent) and loaded into the cell. Following

Table 1 Experimental runs for a central composite design showing independent variables and experimental data for TPC, DPPH, ABTS and AA generated (AAG) responses to optimize PLE conditions[a]

Runs	Temp (°C)	Ethanol (%)	Yield* (%)	TPC^+ (mg GAE per g EDW)	$DPPH^+$ (μmol TE per g EDW)	$ABTS^+$ (μmol TE per g EDW)	AAG^+ (%)
1	50	100	12.73 ± 0.87	154.94 ± 17.84	627.60 ± 49.69	2174.46 ± 113.97	163.95 ± 4.34
2	180	0	56.30 ± 1.10	169.34 ± 11.68	676.69 ± 18.27	1722.71 ± 43.34	62.94 ± 2.42
3	50	0	19.85 ± 1.97	67.23 ± 1.72	477.23 ± 63.58	1227.84 ± 4.82	90.89 ± 2.82
4	115	100	19.30 ± 0.44	210.19 ± 22.40	704.60 ± 16.80	1745,41 ± 81.86	118.47 ± 8.46
5	50	50	15.33 ± 0.06	246.86 ± 30.00	978.98 ± 16.08	2332.23 ± 115.57	95.33 ± 8.51
6	115	50	33.40 ± 6.11	220.56 ± 14.57	874.60 ± 90.62	2092.73 ± 332.27	100.78 ± 11.98
7	115	0	27.22 ± 0.09	147.52 ± 19.50	618.30 ± 5.85	1465.06 ± 202.25	88.50 ± 0.56
8	115	50	34.19 ± 1.00	249.24 ± 24.99	924.20 ± 29.23	2544.48 ± 219.91	86.45 ± 0.27
9	115	50	34.20 ± 0.66	241.78 ± 25.93	906.12 ± 48.96	1991.72 ± 77.05	78.51 ± 0.20
10	180	100	27.71 ± 2.14	241.75 ± 20.68	882.86 ± 30.69	2323.14 ± 83.47	105.86 ± 7.80
11	180	50	54.78 ± 1.41	251.60 ± 19.19	896.30 ± 39.46	2369.68 ± 165.33	87.73 ± 6.61

[a] Results are expressed as mean ± standard deviation ($n^* = 2$, $n^+ \geq 4$). EDW: extract dry weight.

extraction, solvents were removed under a nitrogen stream using a TurboVap LV evaporation system (Caliper Life Sciences, MA, USA), and the resulting residues were freeze-dried as described in Section 2.1. Extraction yield (%) was calculated as the ratio between the mass of dried extract obtained by PLE and the initial sample mass (grape pomace powder), using the following equation:

$$\text{Extraction yield} = \frac{\text{weight of dried extract}}{\text{weight of dried grape pomace}} \times 100$$

2.3 Design of experiment: face-centered CCD

Based on preliminary analysis and previously published studies, a face-centered CCD with two independent factors was established to optimize the PLE conditions, *i.e.*, ethanol percentage in water (0 to 100% v/v) and temperature (50 to 180 °C). The experimental plan consisted of 11 runs, including three central points (Table 1); each one was performed in duplicate ($n = 2$) in random order to minimize the effects of uncontrolled factors. All measures were performed at least in triplicate ($n = 3$). The response variables were extraction yield, total phenolic content, antioxidant capacity, and the percentage of AA generated in the ASN–GLC model system, which emulates the Maillard reaction. Response surface methodology (RSM) based on a CCD was established and analyzed using JMP 16 (JMP Statistical Discovery LLC, Cary, NC, USA) at a significance level of $\alpha = 0.05$.

2.4 Total phenolic content (TPC)

TPC of grape pomace extracts was determined using the Folin–Ciocalteu method described by Fernández-Martínez *et al.*[33] with slight modifications. Briefly, 10 μL of extract or standard were mixed with 600 μL of ultrapure water and 50 μL of undiluted Folin–Ciocalteu reagent. After 1 min reaction, 150 μL of 20% (w/v) sodium carbonate and 190 μL of ultrapure water were added to adjust the final volume to 1 mL. The mixture was incubated in darkness at 25 °C for 2 h, after which 300 μL were transferred to a 96-well microplate. Absorbance was measured at 760 nm and 25 °C using a BioTek (Winooski, Vermont, USA) PowerWave XS microplate reader. TPC was calculated based on a gallic acid calibration curve (0.03–2 mg mL^{-1}) and expressed as milligrams of gallic acid equivalents (GAE) per gram of extract dry weight (EDW).

2.5 Antioxidant capacity

The antioxidant capacity was determined through DPPH and ABTS methods to evaluate both mechanisms, *i.e.*, hydrogen atom transfer and/or electron transfer reactions.[34] DPPH assay was carried out following the method described by Carrasco-Sandoval *et al.*[35] with slight modifications. Briefly, 30 μL of extract or standard were mixed with 970 μL of DPPH solution (60 μM) and incubated in darkness at 25 °C for 30 min. After incubation, 300 μL were transferred to a 96-well microplate, and the absorbance was measured at 517 nm using a PowerWave XS microplate reader. Trolox (0.2–2 μM) was used as the standard,

and the results were expressed as μmol Trolox equivalents (TE) per gram of EDW. ABTS assay was carried out following the method reported by Poblete *et al.*[36] with minor modifications. Briefly, the $ABTS^{+}$ radical cation was generated by reacting ABTS with potassium persulfate and incubating the solution in the dark for 12–16 h. The solution was then diluted to an absorbance of *ca.* 0.70 at 734 nm. Ten microliters of extract or standard were mixed with 990 μL of $ABTS^{+}$ solution and incubated in darkness at 25 °C for 30 min. After incubation, 300 μL were transferred to a microplate, and the absorbance was measured at 734 nm. Results were expressed as μmol TE per gram of EDW using Trolox calibration (0.2 to 2 μM).

2.6 Phenolic profile determination by liquid chromatography-mass spectrometry

Grape pomace analysis was performed based on the method reported by Peterssen-Fonseca *et al.*[37] using a Waters (Milford, MA, USA) I-Class system coupled to Xevo TQD triple quadrupole mass spectrometer. Chromatography was carried out in a Phenomenex (Torrance, CA, USA) Kinetex XB-C_{18} column (150 mm × 4.6 mm, 3.5 μm) maintained at 30 °C using a mobile phase composed of ultrapure water (A) and acetonitrile (B), both acidified with 0.1% v/v formic acid (all solvents were MS-grade) at a flow rate of 0.5 mL min^{-1} with the following gradient: 0.1–2 min 20% B; 2–6 min 20 to 30% B, 6–15 min 30 to 70% B, 15–20 min 70 to 20% B and 20–23 min 20–20% (column conditioning). Mass analyzer was operated in ESI (−) mode with the following parameters: capillary voltage, 2 kV; sampling cone, 30 V; source temperature, 120 °C; desolvation temperature, 400 °C; cone gas (N_2), 5 L h; desolvation gas (N_2), 600 L h^{-1}; and, collision energy ramp from 20 to 30 V. Full Scan mode (*m*/*z* 100–1200) was used for qualitative analysis and selected (Multiple) Reaction Monitoring (SRM or MRM) mode for quantitative determination using the following *m*/*z* transitions: gallic acid (169 → 125); protocatechuic acid (153 → 109); epicatechin and catechin (289 → 123), quercetin 3-O-glucuronide (477 → 301); and quercetin (301 → 151). Data acquisition and processing were carried out using MassLynx V4.2 software (Waters).

2.7 Effect of grape pomace extract on AA formation in ASP-GLC model

The effect on AA formation of each extraction condition from the face-centered CCD experimental plan was evaluated using an asparagine (ASN)-glucose (GLC) model system according to the methodology described by Ou *et al.*[38] The model system was established considering a maximum AA formation when an equimolar ASN : GLC ratio is mixed.[39] Equimolar solutions (0.2 M) of ASN and GLC were prepared in ultrapure water, and 1 mL of each solution was transferred into a 20 mL glass vial, to which 500 μL of each extract obtained *via* PLE at a concentration of 10 mg mL^{-1} were added. Blank sample (positive control) was prepared following the same procedure but adding 500 μL of ultrapure water instead of (poly)phenol extract. All vials were hermetically sealed and subjected to thermal treatment in a Memmert (Schwabach, Germany) oven at 160 °C for 60 minutes. The same approach was used to determine the mitigation effect of epicatechin, gallic acid, and catechin on AA formation. Five hundred microliters of each standard solution (1 mg mL^{-1}) were added for a final concentration of 200 μg mL^{-1} (ppm) per vial. After heating, vials were cooled to room temperature and then filtered (0.22 μm), properly diluted, and analyzed by liquid chromatography coupled to mass spectrometry (LC-MS/MS).

2.8 Effect of grape pomace extract on AA formation in bread model

Breads were prepared according to the formulation reported by Pedreschi *et al.*,[40] with slight modifications. Briefly, 30 g of dough was prepared by mixing flour (19.00 g, 63.33% w/w), vegetable shortening (0.95 g, 3.16% w/w), salt (0.38 g, 1.25% w/w), powdered milk (0.11 g, 0.38% w/w), yeast (0.38 g, 1.25% w/w), sugar (0.09 g, 0.31% w/w), and warm water (9.09 g, 30.32% w/w) at 30 °C. The dough was manually kneaded for 15 min and fermented for 30 min. Different extract concentrations (0.05% (16 mg), 0.10% (32 mg), 0.20% (64 mg), 0.40% (128 mg), and 0.80% (256 mg) w/w), previously dissolved in 1 mL of the extraction solvent, were incorporated into 30 g of dough, followed by an additional kneading step of 15 min. Control group (blank) was prepared using the same procedure as for the treated samples but adding 1 mL of the extraction solvent instead of (poly)phenol extract. Once the extract was homogeneously incorporated into the dough, circular pieces of 4.4 cm in diameter were cut using a circular mold and baked in a conventional electric oven at 200 °C for 20 min. AA was extracted from dried and defatted samples (250 mg), previously spiked with 83.33 μL of internal standard solution (1.2 μg mL^{-1} DMAA), using 916.67 μL of ultrapure water: methanol (8 : 2 v/v) mixture acidified with 0.125% formic acid, applying ultrasound-assisted extraction (UAE) for 30 min at 240 W. Samples were then centrifuged at 4427×*g* for 15 min at 4 °C, filtered (0.22 μm), and analyzed by LC-MS/MS.

2.9 AA quantification by LC-MS/MS

AA quantification was performed following the method proposed by Patiño-Arias *et al.*[41] using a Thermo Scientific (San Jose, CA, USA) Accela liquid chromatograph coupled to a TSQ Quantum triple quadrupole mass analyzer. Separation was carried out on an Agilent (Palo Alto, Ca, USA) XDB-C_{18} column (150 × 4.6 mm, 5 μm) set at 30 °C, with a mobile phase composed of ultrapure water (A) and acetonitrile (B), both acidified with 0.1% v/v formic acid (all solvents were MS-grade) employing the following gradient program at a flow rate of 0.4 mL min^{-1}: 0.1 min, 5% B; 0.1–10 min, 5–50% B; 10–15 min, 50–30% B; 15–17 min, 30–5% B; and 17–20 min, 5–5% B (column conditioning). MS analysis was carried out in selected (multiple) reaction monitoring (SRM or MRM) mode operated in ESI (+) mode using the following parameters: capillary voltage, 4.5 kV; source desolvation temperature, 200 °C; sheath (N_2) pressure, 50 arbitrary units (AU); auxiliary gas (N_2) pressure, 5 AU; collision energy, 9 V for *m*/*z* transitions 72 → 55 (AA), and 12 V for 75 → 58 (AA-13C_3 (AAd), internal standard), with a collision gas (Ar) pressure of 1.70 mTorr. Data acquisition and processing

were carried out using the Xcalibur software (Thermo Scientific).

2.10 Statistical analysis

Data were analyzed using descriptive statistics, including mean, standard deviation (SD), and relative standard deviation (RSD). Calibration curves were obtained by linear regression. Variance and mean comparisons were performed using ANOVA with or without Welch correction, followed by Dunnett's T3 post-hoc test. All statistical analyses were conducted at a significance level of $\alpha = 0.05$ using Prism 8.0 software (GraphPad, San Diego, CA, USA).

3 Results and discussion

3.1 Optimization of PLE conditions

The experimental runs from face-centered CCD were analyzed to maximize the response variables extraction yield, total phenolic content, and antioxidant capacity and to minimize the AA generated in the ASN–GLC model system. According to ANOVA results, both factors, temperature (linear $p = 0.0005$) and ethanol percentage (linear $p = 0.0128$ and quadratic $p = 0.0396$), showed a significant effect on extraction yields. Under the optimal conditions, *i.e.*, 180 °C and 11% v/v ethanol, the predicted value (56.18% w/w) closely matched the highest observed value at 180 °C across the 0–50% v/v ethanol range (56.30–54.78%) (Table 1). The positive effect of higher temperatures on PLE yields has been previously reported and is attributable to a combination of elevated temperature and pressure that maintains the solvent in a liquid state above its boiling point, enhancing diffusivity, reducing viscosity, facilitating matrix penetration, and promoting analyte diffusion and dissolution.[42,43] High temperatures also induce cell disruption, facilitating solute–solvent interaction, increasing the release of bioactive molecules to the medium.[44] Overall, all these effects contribute to higher mass transfer rates compared with conventional solid–liquid extraction conditions. The observed ethanol effect agrees with the results reported by Nieto *et al.*[45] Regarding TPC, ANOVA results indicated that both factors, temperature (linear $p = 0.0366$) and ethanol percentage (linear $p = 0.0225$ and quadratic $p = 0.0077$), had a significant effect. As shown in Fig. 1, TPC values increase with higher temperature, particularly when the solvent contained between 50 and 80% ethanol. This behavior may be attributed to the reduction of the solvent dielectric constant at PLE high temperatures, resulting in greater extraction of semipolar compounds, which leads to a higher TPC value.[46] Additionally, ethanol facilitates intermolecular interactions with (poly)phenols, through hydrogen bonding with the functional groups,[47] as it was demonstrated by density functional theory (DFT) based on the interaction of ethanol and flavonoids.[48] (Poly)phenols can be present associated with cell wall components through non-covalent interactions, mainly hydrogen bonds and hydrophobic interactions.[49] The presence of ethanol can weaken these phenolic–matrix associations, as experimentally demonstrated by Le Bourvellec *et al.*[50] In agreement with our results, Allcca-Alca *et al.*[51] reported similar results describing the positive effect of ethanol in the total recovery of phenolic acids from grape skins and seeds. At the optimal conditions, 180 °C and 61% v/v of ethanol, TPC showed a predicted value of 274.89 mg GAE per gram of EDW, which is in the same range than the highest values observed experimentally at 180 °C and 50% v/v of ethanol (251.60 ± 19.19 mg GAE per gram of EDW) and comparable to the values reported by Otero-Pareja *et al.*[52] for PLE of (poly)phenols from Merlot grape pomace (254.60 ± 2.60 mg GAE per g EDW). Antioxidant capacity determined by DPPH and ABTS methods revealed similar trends despite some differences in magnitude (Fig. 1). DPPH inhibition was not linearly affected by temperature ($p = 0.1366$) and ethanol percentage ($p = 0.0887$), but the quadratic effect of ethanol percentage showed a significant effect ($p = 0.0050$). For ABTS, temperature did not significantly influence the results ($p = 0.3003$), while ethanol percentage

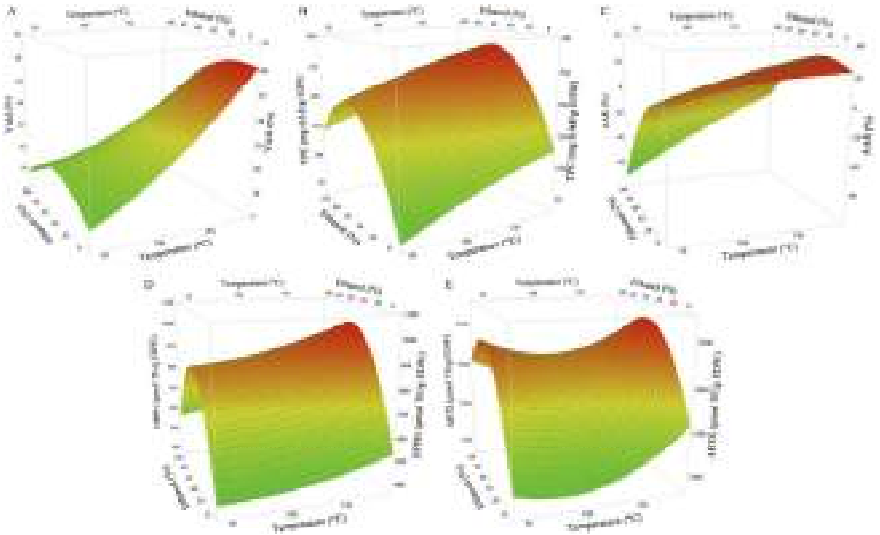

Fig. 1 Response surface plots for extraction yields (A), TPC (B), AA reduction (C), DPPH (D), and ABTS (E).

Table 2 Main (poly)phenol compounds present in grape pomace extracts obtained by optimized PLE[a]

t_R (min)	Compounds	Parent ion $[M–H]^-$ (m/z)	Fragments (m/z)	CID (V)	Concentration (μg g^{-1} EDW)
3.35	Gallic acid	169.03	125.09	−20	1640.82 ± 155.46
4.09	Protocatechuic acid	152.79	108.95	−25	771.84 ± 20.31
4.46	Epicatechin	288.98	123.20	−30	3905.04 ± 250.02
5.09	Catechin	288.27	122.94	−30	1850.78 ± 266.25
7.52	Quercetin-3-O-Glucuronide[b]	477.00	301.24	−30	681.17 ± 32.20
11.46	Quercetin	300.97	151.23	−30	329.27 ± 37.39

[a] EDW: extract dry weight. [b] μg QE per g EDW.

showed a significant linear ($p = 0.0269$) and quadratic ($p = 0.0156$) effect. These findings are consistent with previous studies, highlighting the significant effect of ethanol concentration on antioxidant capacity in PLE extracts from grape by-products.[45] Optimal PLE conditions for antioxidant capacity were identified at 180 °C and ∼60% v/v of ethanol for both DPPH (59% v/v) and ABTS (60% v/v). These PLE conditions are similar to those reported by Rajchman *et al.*[31] for the extraction of antioxidant compounds from Tannat pomace. The extract's mitigation effect on AA formation in the ASN–GLC model system was linearly affected by the factors temperature ($p = 0.0382$) and ethanol percentage ($p = 0.0073$), without quadratic effect of temperature ($p = 0.7679$) and ethanol ($p = 0.1494$). This dependence may be associated with the release of a specific type of (poly)phenol that directly mitigates AA formation. Allcca-Alca *et al.*[51] reported the predominant presence of gallic acid in grape pomace PLE extracts of *Vitis vinifera*. This high content could be explained by the high temperatures used in PLE, which induce the hydrolysis of polymers like hydrolysable tannins through the cleavage of galloyl linkages, releasing gallic acid units to the medium.[53] As shown in Table 1, the AA formation in the presence of 10 mg mL^{-1} of extract ranged from 62.94 ± 2.42% (12.18 ± 0.47 μg mL^{-1}) to 163.95 ± 4.34% (31.76 ± 0.84 μg mL^{-1}), considering 100% of AA formation the amount generated in the model system without extract addition. Based on the primary objective of this study, AA formation was defined as the priority response variable, selecting the following optimal conditions: 180 °C and 21% v/v ethanol, with which a predicted AA content of 71% should be obtained (29% reduction). The experimental results matched the predicted value, observing an AA content of 66.64 ± 6.61% (17.42 ± 0.63 μg mL^{-1}), corresponding to a reduction of 33.36 ± 3.31%. The calculated error between predicted and experimental value was 4.30%, and the model demonstrated acceptable statistical performance with a lack-of-fit value of 0.38, R^2 of 0.86, and R^2 adjusted of 0.73.

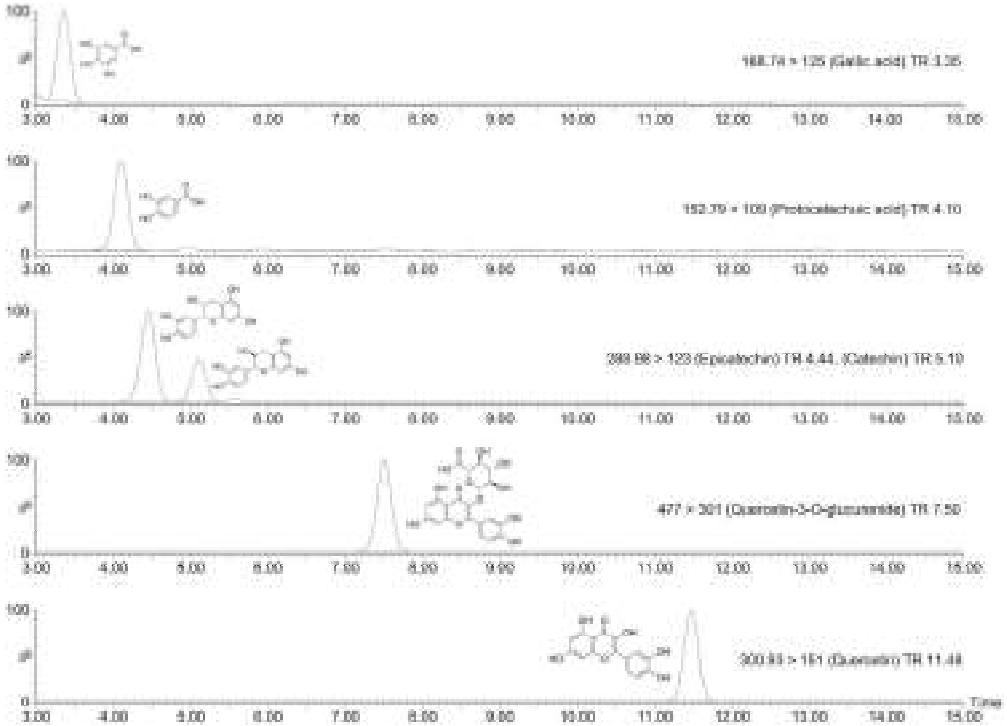

Fig. 2 LC-MS/MS (MRM) chromatograms in multiple reaction monitoring mode showing the (poly)phenols studied in grape pomace extract. Detailed peaks information is shown in Table 2.

3.2 Characterization of the phenolic profile

As shown in Table 2, four flavonoids (epicatechin, catechin, quercetin, and quercetin-3-O-glucoside) and two phenolic acids (gallic acid and protocatechuic acid) were identified in the optimal grape pomace extract. The identification of these phenolic compounds was based on comparison of retention times and mass spectral data (Fig. 2). Quantification was carried out using individual calibration curves, except for quercetin-3-O-glucuronide, which was quantified using the quercetin calibration and reported as quercetin equivalents (QE). Mass analysis in MRM mode identified epicatechin, gallic acid, and catechin as the predominant compounds (>1000 μg g^{-1} EDW); for that reason, they were selected for individual evaluation of their AA mitigation potential in the ASN–GLC model system.

3.3 Mitigation effect of epicatechin, gallic acid, and catechin on AA formation

As described in Section 2.7, the mitigation effect of each (poly)phenol was individually evaluated in the ASN–GLC model system. Fig. 3 shows that all assayed (poly)phenols significantly reduced AA formation ($p < 0.05$), without significant differences among them ($p > 0.05$). Catechin produced the greatest reduction (42.83 ± 10.94%), followed by gallic acid (41.67 ± 6.13%) and epicatechin (35.46 ± 1.30%). The reduction observed with gallic acid is similar to the ones reported by Zhu *et al.*[54] using the same ASN–GLC model system (47%). Catechin and epicatechin possess *o*-dihydroxyl (catechol) moieties that stabilize phenoxyl radicals through intramolecular hydrogen bonding, thereby enhancing radical scavenging activity and preventing the propagation processes that lead to AA formation.[55] In agreement with our results, (poly)phenols with multiple OH substituents in the ortho position, such as catechin and epicatechin, have been shown to have an inhibitory effect on AA formation in ASN–GLC model systems.[56,57] Peterson and Totlani[58] suggested that this kind of (poly)phenol reacts with carbonyl compounds like methylglyoxal, decelerating the Maillard reactions and the subsequent AA formation. This was further supported by Yin *et al.*,[2] who related the inhibitory effect of epicatechin to its interaction with Millard intermediates *via* reaction with radicals, carbohydrates, and amino acids. Although catechin and epicatechin are diastereomers sharing the same number and location hydroxyl groups (five –OH groups, including the 3′,4′ catechol -B ring), they differ in the relative configuration of the C2 and C3 chiral centers (2,3-*trans* in catechin and 2,3-*cis* in epicatechin). This difference in three-dimensional orientation could contribute to the slightly higher inhibitory capacity observed for catechin by modifying the spatial arrangement and accessibility of its functional groups, including the catechol, to reactive carbonyl species involved in AA formation.[59] Gallic acid, on the other hand, has a pyrogallol ring with three adjacent phenolic hydroxyl groups, which makes it a nucleophile capable of reacting by aromatic electrophilic substitution with dicarbonyl intermediates of the Maillard pathway.[60] However, its entrapment capacity is lower than that of free pyrogallol, since the carboxyl group modifies the electron distribution of the ring (positions C4 and C6), reducing its reactivity with electrophilic species. This shows that not only the number and position of the hydroxyl groups, but also the nature of the additional substituents of the aromatic ring, conditioning the phenolic reactivity with Maillard reaction intermediates.[61] The present results also corroborate the findings of Xu *et al.*[15] about the positive impact of grape pomace on AA formation reduction, as well as the effectiveness of catechin, epicatechin, and gallic acid to mitigate its generation. Our results confirmed that the reductive effect of phenolic compounds over AA is not necessarily related to the total content, but rather to the specific type and structural profile of compounds present in the extract.[62] Interestingly, some extracts promoted AA formation in the ASN–GLC model, to determine if differences on the (poly)phenol composition could be related to the divergent effect observed on AA formation, representative experimental runs, *i.e.*, 1, 2, and 6, were analyzed. According to the results, quercetin emerges as a key phenolic compound because it seems to be directly associated with the promoting effect on AA formation. Experimental PLE conditions applied on run 1 and 6 produced extracts with significantly higher quercetin concentrations compared with the runs 2 and at the optimal conditions. Even when runs 1 and 6 exhibited similar quercetin concentrations, their effects on AA formation were significantly different (164% *vs.* 101%). This disparity could be explained by the presence of other (poly)phenol compounds, particularly epicatechin and catechin, that counteract the quercetin's promoting effect on AA formation. To prove this hypothesis, the effect of quercetin (200 μg mL^{-1}) over AA formation was assayed on the ASN–GLC model applying the same protocol used for the other phenolic compounds. Results demonstrated the quercetin promoting effect on AA formation, increasing by 48.78 ± 5.37% the produced amount in the ASN–GLC model. Thus, the observed behavior on AA formation is the result of the presence of (poly)phenol compounds with promoting and mitigating effects, noticing clear counteract effects between quercetin and catechins. These results are

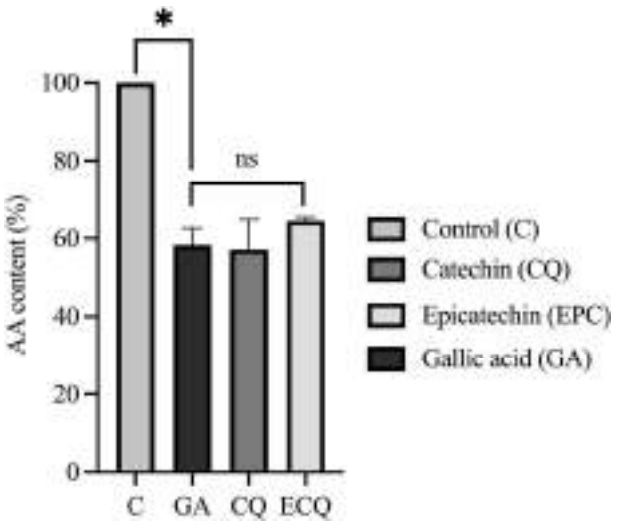

Fig. 3 Effect of major (poly)phenols from PLE grape pomace extract on AA formation in the ASN–GLC model system (*: $p < 0.05$).

consistent with the findings reported by Huang *et al.*,[63] who evaluated the effects of various flavonoids, including flavones, flavonols, and isoflavones, on AA formation. Authors observed that quercetin showed the strongest promoting effect increasing up to 179% the AA formation at a concentration of 1 mmol L^{-1} (302 ppm).

3.4 Mitigation effect of grape pomace extract on AA formation in bread model

As shown in Fig. 4, the AA content decreased progressively as the extract concentration increased. To visualize the inhibitory effect of grape pomace extract, the AA content was expressed as the percentage of AA formed in the presence of different extract concentrations with respect to the control (without extract addition, set at 100%). All extract levels (0.05–0.8% w/w) produced a statistically significant reduction of AA content ($p < 0.05$), with a minimum value of 58.75% observed at 0.4% w/w of extract addition (128 mg extract per 30 g of bread) without significant differences from 0.2% to 0.8% w/w. The two lower extract concentrations (0.05%–16 mg extract and 0.1%–32 mg extract) produced a similar decrease in AA content to *ca.* 88% ($p < 0.05$). The three larger concentrations (0.2%–64 mg, 0.4%–128 mg, and 0.8%–256 mg) had a greater and significant impact, reducing AA formation to approximately 59% ($p < 0.05$). A dose-dependent decrease in AA content was observed with increasing extract concentration up to 0.4% w/w. This attenuated response to higher extract concentrations could be attributed to the viscoelastic nature of fermented dough, in which the gluten network acts as a physical (and maybe chemical) barrier, limiting the diffusion and homogeneous distribution of (poly) phenols.[64] Uneven distribution will result in zones with effective inhibition and zones where (poly)phenol interaction is insufficient. This explanation remains hypothetical, as no experimental evidence was obtained on the spatial distribution or diffusion of (poly)phenols within the mass matrix in the present study. Alternative or complementary mechanisms—such as saturation of reactive phenolic sites, extract-induced changes in mass rheology, or dilution/interaction effects with other formulation components—cannot be ruled out. Future studies incorporating spatial distribution analyses of (poly) phenols within the dough/bread matrix are needed to confirm the proposed physical barrier mechanism. The reduction of 41.25 ± 9.36% in AA formation observed in this work is comparable to the values reported by Pedreschi *et al.*[40] using 750 mg kg^{-1} Tara pod (*Caesalpinia spinosa*) extract in bread. Jing *et al.*[65] also described similar results using buckwheat extracts with reductions of 47% and 27% in AA formation in ASN–GLC and bread models, respectively. In contrast, Onacik-Gür *et al.*[9] did not observe a significant reduction in AA formation when green tea extract was applied to rye breads. These contrasting results demonstrate that the mitigation effect of (poly)phenols on AA formation is conditioned by multiple factors, including the extraction method, type of (poly)phenol, concentration, degree of homogenization and incorporation into the food matrix, and reaction conditions/production.

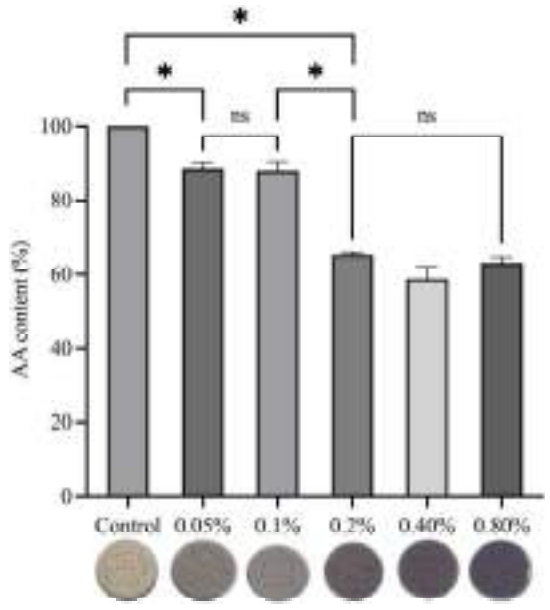

Fig. 4 Effect of different PLE grape pomace extract concentrations on AA formation in bread model (*: $p < 0.05$).

4 Conclusions

The present study demonstrated that grape pomace, a widely available agro-industrial byproduct, is a sustainable and effective source of bioactive compounds capable of mitigating AA formation in thermally processed foods. The usefulness of the PLE technique for obtaining grape pomace extract in a green and sustainable process with a significant inhibitory effect on AA formation during the Maillard reaction was also proved. The PLE conditions were optimized to maximize the responses: yield, TPC, antioxidant capacity, and mitigation of AA formation. The latter showed different optimal conditions, highlighting the importance of using AA formation as the response variable rather than relating the inhibitory effect solely to the TPC. The phenolic profile of the optimized extract was characterized, observing the presence of six main compounds, among which epicatechin, catechin, and gallic acid were identified as the predominant species and the primary contributors to AA mitigation. This proved the importance of establishing the (poly)phenol profile alongside TPC and antioxidant capacity. The application of the optimized grape pomace extract in real bread production validated its practical potential, beyond its successful inhibitory activity on the ASN–GLC model system that mimics the Maillard reaction. These results are comparable to other natural mitigation strategies reported in the literature, supporting the feasibility of grape pomace extracts as clean-label food additives. Overall, the integration of green extraction technology with agro-industrial waste valorization offers a promising and scalable strategy for reducing AA in baked foods like bread, contributing to both food safety and circular economy principles. Future research should explore the sensory impact of the extract, its stability under different processing conditions, and its efficacy across a broader range of food matrices.

Author contributions

Lina Patiño-Arias: investigation, methodology, writing – original draft. Mikaela Rajchman: formal analysis and investigation. Lidia Montero: formal analysis. María Salome Mariotti-Celis: methodology and supervision. Karem Henriquez-Aedo: funding acquisition and resources. Miguel Herrero: supervision and funding acquisition, writing – review & editing. Mario Aranda: conceptualization, writing – review & editing, project administration.

Conflicts of interest

There are no conflicts to declare.

Data availability

Data will be available on request

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