Applied Food Research 6 (2026) 101959

Contents lists available at ScienceDirect

Applied Food Research

journal homepage: www.elsevier.com/locate/afres

Valorization of agrifood waste: Optimizing bioactive extraction using accelerated solvent extraction with three solvent systems

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ARTICLE INFO

Keywords:
Broccoli (*Brassica oleracea* var. *italica*)
Almond (*Prunus dulcis* Mill.)
Grape (*Vitis vinifera* L.)
Pea (*Pisum sativum* L.)
Prickly pear (*Opuntia ficus-indica* L. Mill.)
Quince (*Cydonia oblonga* Mill.)
Antifungal activity

SUMMARY

The increasing demand for bioactive compounds from natural sources is focusing attention on agrifood by-products for applications beyond composting. A waste-to-value strategy, paired with energy-efficient extraction methods such as accelerated solvent extraction (ASE), represents an environmentally friendly approach contributing to sustainable development. The objectives of this research are aligned with these guidelines, studying by-products from the industrial processing of quince, broccoli, grapes, pea, prickly pear, and almond. The ASE process was optimized by varying the extraction solvent (ethanol, acetone and methanol) and the amount of sample used (0.5,1 and 1.5 g) in each extraction cycle (1, 2 and 3). Solvent type proved to have a significant impact on extraction yield, phenolic content, and both antioxidant and antifungal activity. Methanol was the most efficient solvent overall, and the highest yields were obtained from prickly pear peel (43.3 %) and broccoli discards (26.4 %). Epicatechin (16.8–38.6 mg/g extract), ellagic acid (3.5–6.4 mg/g extract), and catechin (2.6–3.4 mg/g extract) were the most abundant compounds identified. By-products from quince (QB) and grapes (stem and skin) exhibited the highest antioxidant activity (QB: 7.0; stem: 6.7; skin: 6.3 mmol TEAC/100 g extract) and total phenolic content (skin: 81.7; stem: 67.0 and QB: 31.6 mg GAE/g extract). Extracts were active against five mold strains, with some demonstrating superior activity against *Aspergillus puulaauensis* and *A. jensenii* over the standard, natamycin. Finally, extracts that combined the best antifungal and antioxidant properties were quantitatively ranked. Collectively, this research establishes a foundation for designing green preservatives and advances the circular economy *via* efficient resource valorization.

1. Introduction

The agrifood industry generates >190 million tons of by-products annually, accounting for approximately 30–50 % of total waste (including peels, shells, skin, cores, pomace, pulp, and damaged or unripe fruits and vegetables) (Zhu et al., 2023). This massive waste stream, originating across the entire supply chain, has a significant environmental and economic impact and many efforts are being made to identify high-value compounds present is this raw material that provides an innovative tuning of agricultural product utilizations (Sarker et al., 2024). In this context, agri-food by-products are increasingly recognized as rich sources of bioactive compounds and their antioxidant and antimicrobial properties have stimulated growing interest in exploiting it for food-related applications, particularly as natural preservatives (Rațu et al., 2023).

Currently, the main food preservation strategy is based on the use of chemically synthesized organic acids and their related salts (Gao et al., 2025). However, the prolonged utilization of synthetic preservatives may exert detrimental effects on consumer health and potentially contribute to the emergence of fungal resistance (Wang et al., 2024). Natural antimicrobial and antioxidant agents extracted from by-products represent an innovative and sustainable approach to combating these limitations. Moreover, in contrast to conventional preservatives, green alternatives are aligned with both consumer demand for clean label products and environmental sustainability (Lemoni et al., 2025). Plant extracts are among the most important sources of natural preservatives due to their richness in bioactive secondary metabolites such as phenolic compounds, flavonoids, terpenes, tannins and other phytochemicals, many of which possess demonstrated antimicrobial or antioxidant properties (Teneva & Denev, 2023). Additionally, different trials in which these extracts have been tested in real food matrices, including meat, seafood and dairy products, have shown

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https://doi.org/10.1016/j.afres.2026.101959
Received 7 November 2025; Received in revised form 31 March 2026; Accepted 1 April 2026
Available online 1 April 2026

promising results (Shah & Mir, 2022).

Polyphenols extracted from fruit and vegetable wastes are prime candidates for replacing synthetic alternatives (Socas-Rodríguez et al., 2021). Studies consistently show that many peels contain significantly higher concentrations of phenolic compounds than the edible pulp or seeds. For example, peels from papaya (*Carica papaya*), passion fruit (*Passiflora edulis*), and pomegranate (*Punica granatum*) contain roughly double phenolic compounds found in their internal tissues (Rifna et al., 2023). Moreover, papaya peel and leaf extracts commonly contain major flavonoids such as myricetin, quercetin, kaempferol, morin, apigenin, and luteolin. Similarly, this occurs in apple peels, which have 3 to 5 times more flavonoids and phenolic acids than the flesh, and in citrus peels whose phenolic contents together carotenoids are 10 to 15 times greater than edible portions (Matsuo et al., 2019). Quince (*Cydonia oblonga* Miller) peel also represents a notable source of polyphenolic antioxidants, containing approximately three times more phenolics than the pulp, which correlates with its stronger antioxidant and antimicrobial activities (Sonmez & Sahin, 2023). Furthermore, its flavonoid profile, particularly flavonol glycosides, is largely confined to the peel, while additional phenolics such as caffeic acid and procyanidins are present in the leaves (Silva et al., 2023).

Grape (*Vitis vinifera* Macabeo) stems, a lignocellulosic residue that accounts for approximately 5–7 % (*w/w*) of the total material processed, are removed upon destemming operations before vinification to prevent excessive astringency provided by proanthocyanidins, catechin derivatives, phenolics acids and flavonols (Dias-Costa et al., 2024). On the other hand, the peel and seed fractions present high concentrations of different phenolic acids, flavanols, flavonols, and stilbenes (Allcca-Alca et al., 2021). Resveratrol is an example of stilbene found in grape skin with high antioxidant potential (Yang et al., 2025). The aqueous extracts derived from of prickly pear (*Opuntia ficus indica*) peel have also exhibited higher antioxidant and anti-bacterial effect than the pulp and that has been related to the presence of phenolic compounds and flavonoids especially chlorogenic acid and hyperoside (Zourgui, 2021).

Tree nut by-products represent additional sources of high-value phytochemicals. Almond (*Prunus dulcis*) composition varies by cultivar but typically consists of 40–60 % hull, 20–33 % shell, and 15–31 % kernel with skin. Since only the kernel is the edible and economically valuable portion, >70 % of the almond fruit is generated as by-product or waste (Silva et al., 2025). Almond hulls contain triterpenoids, lactones, phenolic compounds, and tocopherols, while almond shells are rich in flavonoids and phenolic acids (Ben Khadher et al., 2023; Fabroni et al., 2023). These bioactive compounds contribute to the antioxidant, antimicrobial, and antidiabetic activities of almond by-products, making them increasingly attractive for applications in the food and pharmaceutical industries (Najari et al., 2022).

Beyond fruits, several vegetable by-products also constitute valuable sources of bioactive compounds. The stem of cruciferous vegetables (e.g., broccoli) is rich in hydroxycinnamic acids, especially *p*-coumaric, sinapic, and ferulic acids, which are frequently present in conjunction with sugar or other hydroxycinnamic acids (Yan et al., 2023). Chlorophylls, carotenoids and flavonols such as kaempferol and quercetin are accumulated in the leaves of broccoli (*Brassica oleracea*) in greater concentration than in the florets (García & Raghavan, 2022; Gudiño et al., 2024). Moreover, water-soluble extract from broccoli seeds has been shown to reduce fungal contamination in cereals (Thery et al., 2020). Likewise, pea (*Pisum sativum*) pods contain diverse phenolic profiles, including cinnamic and benzoic acid derivatives, as well as a broad spectrum of flavonoids such as flavanones, flavanols, flavonols, flavones, and isoflavones.

Accelerated Solvent Extraction (ASE) has emerged as a highly efficient alternative to conventional extraction techniques for recovering bioactive compounds from agri-food by-products, making it particularly relevant within the framework of waste valorization (Wang et al., 2022). ASE, a form of hot-pressurized liquid extraction (HPLE) (Rodríguez De Luna et al., 2020), is an automated technique that uses moderate-to-high temperature and pressure to extract target compounds from a sample contained within a vessel for short intervals, typically a few minutes (Tasfiyati et al., 2022). Compared with traditional extractions that typically require 8 to 10 h and hundreds of milliliters of solvent, ASE reduces processing times to 15 to 30 min while using up to tenfold less solvent. These conditions along with the use of organic solvents enhance solvent diffusivity, reduce viscosity, and weaken solute–matrix interactions, thereby improving mass transfer and solubilization of different compounds, especially phenolic acids, flavonoids, and other bioactive metabolites (Gil-Martín et al., 2022). However, the performance of the technique is strongly influenced by operating parameters, especially temperature, pressure, static cycle duration, and solvent polarity (Chamali et al., 2023). Optimizing these parameters has been shown to improve the recovery of valuable phytochemicals and the purity of the extract, reinforcing ASE as a powerful tool for advancing circular economy strategies (Zhang et al., 2022). This results in higher extraction yields, lower environmental impact, and greater sustainability, which are fundamental aspects in food waste recovery processes (Mogashane et al., 2024).

Due to these advantages, ASE was chosen to obtain extracts from seven common agrifood by-products. The primary objectives of this study were to: optimize the ASE parameters (sample amount, solvent type, and the number of ASE cycle); characterize the resulting extracts by identifying low molecular weight phenolic compounds *via* HPLC-DAD, quantifying their total phenolic content (TPC), and assessing their antioxidant and antifungal activities; and establish a ranking and selection matrix based on statistical analysis of the characterization data to identify the most promising by-product extracts for developing natural preservation applications.

2. Materials and methods

2.1. Agrifood wastes

The samples used in this study were obtained from seven different agrifood by-products. The skins and stems from grape (*Vitis vinifera* Macabeo) (GSK and GST, respectively) and almond (*Prunus dulcis*) hulls (AH) were provided by farmers from Valencia and Albacete (Spain), respectively. Broccoli (*Brassica oleracea*) discards (BD) and pea (*Pisum sativum*) pods (PP) were supplied by Ultracongelados Campoverde S.L. (Albacete, Spain), and quince (*Cydonia oblonga* Miller) by-products (QB) were supplied by Membrillo Emily S.L. (Murcia, Spain). Finally, prickly pear (*Opuntia ficus indica*) peel (PPP) was provided by the Università degli Studi di Palermo (Italy).

By-products were lyophilized in a freeze dryer (Sublimator 30 EKS, Tiel, Netherlands) for 48 h at −40 °C and 0.2 mbar. Lyophilized samples were ground using a Waring Commercial 7010 HS blender (New Hartford, CT) and sieved to obtain a powder with a particle size of 2–4 mm. Samples were subsequently stored under vacuum in polyethylene bags (22 × 30 cm, 90 μm) at room temperature in the dark until analysis.

2.2. Accelerated solvent extraction optimization

To determine the optimum extraction conditions, the combination of three variables was systematically studied: sample amount (0.5, 1, and 1.5 g), the number of ASE cycle (1, 2, and 3), and three different solvents (ethanol, acetone, and methanol). In total, 27 independent experiments were performed in three different extractions, and each extraction included three replicates of each sample. This procedure was applied to the seven by-products studied in this work. Extractions were performed using a BÜCHI E-914 Speed Extractor (Postfach, Switzerland) according to Rozalén et al. (2021) with minor modifications. For each extraction, the sample was homogenized with approximately 32 g of sand as a dispersing agent. This mixture was packed into an 80-mL stainless steel extraction cell, sandwiched between layers of sand (totaling approximately 37 g) at the top and the bottom, and sealed with cellulose acetate

filters (BÜCHI) to prevent particle leakage, ensuring a 1-cm headspace. The amount of solvent used per cell in each cycle was 60 ml. The fixed parameters for each cycle were set as follows: temperature at 40 °C and pressure at 100 bar, a 28-min static holding time, a 1-min heat-up, a 3-min discharge, a 2-min solvent flush, and a final 5-min N_2 flush. These conditions were established based on previous works seeking to obtain thermolabile bioactive compounds (Jara et al., 2022; Rozalén et al., 2021).

By-product extracts were collected in 250-mL vials and transferred into pre-weighed solvent evaporation flasks previously desiccated for 30 min. Solvent evaporation was then performed using a Multivapor BÜCHI P-6 parallel evaporator at 40 °C and 100 mbar. The flasks were then stored for 30 min in a desiccator before weighing. The extraction yield was then determined gravimetrically. All extraction and yield analysis procedures were performed in triplicate.

A univariate general linear model (GLM) was used to determine the interactions between the different variables and to identify the most efficient combination of these variables to achieve the maximum yield. The model used is represented by the Eq. (1):

$$Y = \mu + \alpha_i(Solvent) + \beta_j(Amount) + \gamma_k(Cycle) + (\alpha\beta)_{ij} + (\alpha\gamma)_{ik} + (\beta\gamma)_{jk} + (\alpha\beta\gamma)_{ijk} + \varepsilon \quad (1)$$

where Y indicates extraction yield, μ is the intercept, α_i, β_j and γ_k represent the main effects of the solvent type, sample amount and cycles number, respectively, $(\alpha\beta)_{ij}$, $(\alpha\gamma)_{ik}$, $(\beta\gamma)_{jk}$ and $(\alpha\beta\gamma)_{ijk}$ correspond to the interaction between solvent and amount, solvent and cycle, amount and cycle and solvent, amount and cycle, respectively, and ε is the random error term.

Ethanol was selected as the resuspension solvent due to its recognized GRAS (Generally Recognized as Safe) status and widespread use in food systems. Following preliminary solubility tests, it was established that the highest concentration at which all extracts were completely soluble was 10 mg/ml.Therefore, the dry matter resulting from each by-product was redissolved in ethanol at the same concentration (10 mg/mL) for subsequent analyses.

2.3. Determination of low molecular weight phenolic compounds by HPLC-DAD

Low molecular weight phenolic (LMWP) compounds were determined according to Cebrián-Tarancón et al. (2024). Extracts (10 mg/mL) were filtered through a 0.22 μm PVDF filter, and a volume of 20 μL was injected into an Agilent 1200 HPLC instrument (Palo Alto, CA) equipped with a Diode Array Detector (DAD, Agilent G1315D) coupled to an Agilent ChemStation (version B.03.01) data-processing station. Separation was performed in a reverse-phase ACE C18-PFP column (4.6 × 150 mm, 3 μm particle size) with an ACE Excel HPLC Pre-column Filter 1PK (0.5 μm particle size) at 30 °C. The solvents used were water/formic acid/acetonitrile (97.5:1.5:1v/v/v) as solvent A and acetonitrile/formic acid/solvent A (78.5:1.5:20 v/v/v) as solvent B. The elution gradient for solvent B was set up as follows: 0 min, 5 %; 8.40 min, 5 %; 12.50 min, 10 %; 19 min, 15 %; 29 min, 16 %; 30 min, 16.5 %; 34.80 min, 18 %; 37.20 min, 32 %; 42 min, 62 %; 52 min, 9 %; 54 min, 100 %; 56 min, 100 %; 60 min, 5 %; 65 min, 5 %. Compound detection was carried out by comparison with the corresponding UV–visible spectrum and retention time of the single pure standards (Sigma-Aldrich, Steinheim, Germany). The wavelength quantification of the analyzed compounds is outlined in greater detail by Cebrián-Tarancón et al. (2024). The pure compounds used as standards were: 10 phenolic acids (gallic acid, protocatechuic acid, 4-hydroxybenzoic acid, vanillic acid, syringic acid, ellagic acid, caffeic acid, p-coumaric acid, ferulic acid, and synapic acid), 8 flavonoids (catechin, (−)-epicatechin, epicatechin gallate, catechin gallate, epigallocatechin gallate, procyanidin B2, quercetin, and malvidin), 4 stilbenes (resveratrol, piceatannol, resveratrol-3-O-glucoside (piceid), and viniferin), 2 simple phenols (pyrogallol and pyrocatechol) and 2 phenolic aldehydes (coniferaldehyde and sinapaldehyde). Quantification was based on the calibration curves (R^2 = 0.92–0.99) of the respective standards at five different concentrations, measured *via* UV–visible signal. All analyses were made in duplicate before bottling.

2.4. Total phenolic content

The TPC was measured using the Folin-Ciocalteu method according to Codina et al. (2024). A volume of 150 μL of each sample was mixed with 750 μL of Folin-Ciocalteau reagent (Sigma-Aldrich) and kept at room temperature for 5 min. Subsequently, 600 μL of 0.7 N sodium carbonate (Sigma-Aldrich) was added to the mix, which was then incubated for 2 h in the dark at room temperature. Finally, the absorbance was measured at 765 nm in a UV–Vis GENESYS 150 spectrophotometer (Thermo Fisher Scientific, Waltham, MA) against distilled water. A standard curve with concentrations of gallic acid ranging from 0 to 100 μg/mL was constructed and the TPC of the extracts at 10 mg/mL was expressed as milligrams of gallic acid equivalent per gram of by-product extract (mg GAE /g extract). The samples were measured in triplicate.

2.5. Scavenging activity of DPPH

The 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical scavenging activity of the extracts at 10 mg/mL was determined by the method of Codina et al. (2024). A volume of 300 μL of each sample was mixed with 1.55 mL of DPPH (0.1 mM in ethanol) and incubated in the dark at room temperature for 30 min. The absorbance was then measured at 515 nm. A standard curve with concentrations of Trolox ranging from 0 to 0.3 mM was constructed and the results were expressed as mmol TEAC (Trolox equivalent antioxidant capacity) per 100 *g* of by-product extract (mmol TEAC /100 g extract). The samples were measured in triplicate.

2.6. Antifungal activity

The antifungal activity of the extracts (10 mg/mL) was measured against the following panel of mold strains provided by the University of Castilla- La Mancha and the University of León: *Penicillium nordicum* M32, *P. expansum* QZ1, *P. commune/biforme* 601, *P. crustosum* 1B01, *P. commune* 301, *Aspergillus jensenii* 501, and *A. puulaauensis* 1A05. All molds were isolated from cheeses factories (Ramos-Pereira et al., 2019; Muñoz-Tebar et al., 2021). Before the assay, mold strains were cultured for 7 d at 25 ± 1 °C, collected with Tween 20, suspended in sterile saline solution (0.85 % w/v), and diluted at 1–2 × 10^6 colony forming units (CFU)/mL. Then, 100 μL of each inoculum was spread onto the surface of potato dextrose agar (PDA) plates (Merck, Darmstadt, Germany). Wells (10 mm diameter) were cut into the agar, and 100 μL of the extracts were added into these wells in duplicate (Muñoz-Tebar et al., 2021). Plates were incubated for 7 d at 25 ± 1 °C and then examined for halos of growth inhibition. Plate images were captured with a Countermat flash instrument (IUL S.A., Barcelona, Spain) and the diameter of the inhibition zone was measured four times (in mm) using ImageJ v1.52a software. Natamycin (Sigma-Aldrich) was used as a positive inhibition control (0.2 mg/mL) according to various authors (Čutović et al., 2025; Duy et al., 2019; Teixeira & O'Keefe, 2019). Moreover, ethanol was included as a negative control in all assays, and its inhibition values were used to correct the inhibition percentages of the extracts, ensuring that no solvent derived effect influenced the final antifungal results. The growth inhibition as a percentage was calculated with the Eq. (2):

$$\text{Inhibition } (\%) = [(Ac - Af)/Ac] \times 100 \quad (2)$$

where Ac is the growth zone of the negative control and Af is the growth

zone of the extract. All analyses were conducted in duplicate. Fungal growth inhibition (%) relative to the natamycin control was also calculated.

2.7. Ranking of the agrifood by-products using a score calculation

A score was attributed to each variable considered: TPC (mg GAE/g extract), antioxidant activity (mmol TEAC/100 g extract), number of inhibited strains (n° strains) and antifungal activity (% inhibition relative to natamycin) according to the results of a statistical analysis (SPSS software, IBM SPSS Statistics version 25) in which the values were grouped in quartiles. Based on this analysis, four groups were generated in the case of the TPC, antioxidant activity and number of inhibited strains. The scores were calculated as follows: "1″ values included in the first percentile (1 ≤ p25), "2″ values between the first and second percentile (p25 < 2 ≤ p50), "3″ values between the second and third percentile (p50 < 3 ≤ p75), and "4″ values above third percentile (4 ≥ p75). Regarding antifungal activity relative to natamycin, five groups were generated considering the above scoring system, adding one more group and attributing "5″ to the by-products with antifungal activity relative to natamycin higher than 100 % (5 ≥ 100 %). To calculate the final score of each extract, the weighted value corresponding to relative inhibition was multiplied by three and the remaining terms were added according to the Eq. (3). Based on the score obtained, the extracts were classified as "excellent", "good", "average", and "bad" following the same statistical criteria by quartiles used in the previous analysis.

$$\text{Final score} = \text{TPC} + \text{antioxidant activity} + \text{n}^{\circ}\text{ strains} + 3*\text{relative inhibition} \quad (3)$$

2.8. Statistical analysis

The results were statistically analyzed by a univariate GLM (General Lineal Model) using SPSS software. The data were statistically reported

Table 1
Univariate general linear model analysis of parameters influencing extraction yield.

Parameter	Broccoli discard (BD)					Almond hull (AH)				
	Sum of squares	df[a]	F	p	Partial η^2	Sum of squares	df	F	p	Partial η^2
Model	3796.10	26	80.20	<0.001	-	479.62	26	47.98	<0.001	-
Intercept	3392.72	1	1863.675	<0.001	-	384.94	1	1001.31	<0.001	-
Solvent	866.51	2	237.99	<0.001	0.95	194.99	2	253.60	<0.001	0.95
Sample Amount	5.31	2	1.46	0.250	0.10	0.83	2	1.08	0.354	0.07
Cycle	2233.68	2	613.50	<0.001	0.98	191.09	2	248.53	<0001	0.95
Solvent* Sample Amount	12.81	4	1.76	0.166	0.21	0.58	4	0.38	0.824	0.05
Solvent*Cycle	639.24	4	87.79	<0.001	0.93	84.62	4	55.03	<0.001	0.89
Sample Amount*Cycle	17.29	4	2.37	0.077	0.26	3.97	4	2.58	0.06	0.28
Solvent*Sample Amount*Cycle	21.27	8	1.46	0.218	0.30	3.54	8	1.15	0.362	0.25
		R^2= 0.987		Adjusted R^2= 0.975			R^2= 0.979		Adjusted R^2= 0.958	
	Grape stem (GST)					Grape skin (GSK)				
Parameter	Sum of squares	df	F	p	Partial η^2	Sum of squares	df	F	p	Partial η^2
Model	827.16	26	33.72	<0.001	-	2323.14	26	316.38	<0.001	-
Intercept	949.80	1	1006.68	<0.001	-	1906.34	1	6750.02	<0.001	-
Solvent	254.79	2	135.02	<0.001	0.91	289.62	2	144.81	<0.001	0.97
Sample Amount	0.07	2	0.04	0.963	0.00	1.90	2	0.95	0.05	0.20
Cycle	411.79	2	218.23	<0001	0.94	1352.15	2	676.07	<0001	0.99
Solvent* Sample Amount	4.11	4	1.09	0.382	0.14	2.70	4	0.68	0.076	0.26
Solvent* Cycle	150.49	4	39.88	<0.001	0.86	672.53	4	168.13	<0.001	0.99
Sample Amount* Cycle	1.92	4	0.51	0.73	0.07	2.69	4	0.67	0.077	0.26
Solvent* Sample Amount* Cycle	3.99	8	0.53	0.824	0.14	1.55	8	0.19	0.699	0.17
		R^2= 0.970		Adjusted R^2= 0.941			R^2= 0.997		Adjusted R^2= 0.994	
	Pea pod (PP)					Prickly pear peel (PPP)				
Parameter	Sum of squares	df	F	p	Partial η^2	Sum of squares	df	F	p	Partial η^2
Model	1994.07	26	138.77	<0.001	-	11,223.14	26	3.44	<0.001	-
Intercept	1447.46	1	2618.94	<0.001	-	8011.75	1	63.83	<0.001	-
Solvent	385.12	2	348.40	<0.001	0.96	3353.12	2	13.36	<0.001	0.50
Sample Amount	2.77	2	2.50	0.101	0.16	305.04	2	1.22	0.312	0.08
Cycle	1103.70	2	998.48	<0.001	0.99	1741.19	2	6.94	0.004	0.34
Solvent* Sample Amount	4.91	4	2.22	0.93	0.25	490.92	4	0.98	0.436	0.13
Solvent*Cycle	491.76	4	222.44	<0.001	0.97	3785.46	4	7.54	<0.001	0.53
Sample Amount*Cycle	1.83	4	0.83	0.518	0.11	429.19	4	0.85	0.503	0.11
Solvent*Sample Amount*Cycle	3.98	8	0.90	0.53	0.21	1118.21	8	1.11	0.385	0.25
		R^2= 0.993		Adjusted R^2= 0.985			R^2= 0.768		Adjusted R^2= 0.545	
	Quince By-products (QB)									
Parameter	Sum of squares	df	F	p	Partial η^2					
Model	1886.19	26	77.60	<0.001	-					
Intercept	1629.50	1	1742.33	<0.001	-					
Solvent	166.97	2	89.27	<0.001	0.87					
Sample Amount	0.84	2	0.45	0.644	0.03					
Cycle	1439.24	2	769.45	<0.001	0.98					
Solvent* Sample Amount	7.31	4	1.95	0.13	0.22					
Solvent*Cycle	254.03	4	67.90	<0.001	0.91					
Sample Amount*Cycle	2.43	4	0.65	0.632	0.09					
Solvent*Sample Amount*Cycle	16.05	8	2.15	0.066	0.39					
			R^2= 0.987			Adjusted R^2= 0.974				

[a] df: Degrees of freedom

as average and standard deviation. Differences among means were analyzed by Tukey's test using a confidence level of 95 %.

3. Results and discussion

3.1. Optimization of ASE conditions

The yields obtained for each by-product by varying the extraction solvent (ethanol, acetone and methanol) and the amount of sample used (0.5,1 and 1.5 g) in each cycle (1, 2 and 3) are shown in the Supplementary Table 1. A univariate GLM was employed to analyze the relationship between the extraction yield and the following independent variables: sample amount, cycle number and solvent type. Furthermore, the model was used to investigate the interactions among these independent variables. This analysis was performed separately for each sample (BD, AH, GST, PP, GSK, PPP and QB), and the detailed results of the resulting models are presented in Table 1. The coefficient of determination (R^2) values, used to verify the adequacy of the generated model, were close to unity, and the difference between R^2 and adjusted R^2 was consistently <0.2. These findings suggest a good fit for the data. Moreover, the consistently high F-values ($F > 1$) and remarkably low p-values ($p < 0.001$) indicate that the overall model constructed for each sample was highly significant and appropriate for representing the experimental data.

The effect size of each factor was quantified using partial η^2, which along with the p-value, enables the determination of both the magnitude and statistical significance of a specific factor's influence on the model. As detailed in Table 1, the factors "solvent" and "cycle" were highly significant ($p < 0.001$) and demonstrated a large effect size (partial $\eta^2 > 0.5$) across all sample types. The sole exception was the PPP (prickly pear peel) sample, where the "cycle" remained significant but exhibited only a moderate effect size. Conversely, the sample amount (grams of sample used) was not statistically significant and showed a negligible effect size in every case. This finding indicates that increasing or decreasing the initial mass of the sample does not have a significant impact on the final extraction yield. Regarding factor interactions, the only one found to be statistically significant and to have a large effect size was the solvent*cycle interaction. Fig. 1 graphically presents the extraction yields for each by-product, focusing only on the two factors whose influence was found to be significant (solvent and cycle number). These data are crucial for determining the most efficient extraction conditions for each sample, as the required number of extraction cycles to achieve the maximum yield directly impacts the time and economic resources involved in the process. The differences between by-products within the same solvent have also been analyzed (superscripts a-f). Although this analysis does not contribute directly to the optimization of the extraction method, given that extraction yield is inherently a matrix-dependent variable, it provides valuable contextual information regarding the relative efficiency of each by-product and offers a broader perspective on how each matrix behaves under identical extraction conditions. As shown in Fig. 1, the maximum yield was obtained with the first cycle in all the cases, with the results being significantly higher than those found in the second and third cycle. Specifically, the BD by-product showed the highest ethanolic extraction efficiency in the first cycle (19.2 %). In general, the results indicated that the solvent was the most determining factor for the extraction yield, with the most efficient solvent being methanol, followed by ethanol and acetone. Within methanol extractions, PPP (43.3 %) followed by BD (26.4 %) were significantly more efficient than the other by-products. For acetone, which generally yielded the lowest results, BD (5.3 %), GSK (5.5 %), and QB (6.3 %) obtained the highest values. These results are likely due to the polarity of the solvents (methanol > ethanol > acetone). Generally, polar protic solvents, such as methanol and ethanol, are more efficient in plant extractions due to their ability to dissolve polar compounds and penetrate the cell matrix (Lee et al., 2024). However, lipophilic phenols (e.g., ferulic acid or tocopherol) and some flavonoids (e.g., quercetin or kaempferol) may have a greater affinity for acetone. Consequently, the TPC values for samples extracted with acetone could potentially be higher than those extracted with ethanol or methanol (El Mannoubi, 2023).

The role of the solvent in extraction yield is a well-established topic in the literature. Rahman et al. (2022) indicated that broccoli by-products extracted with methanol using a maceration technique showed the highest yield (18.5 %), followed by ethanol (16.0 %) and a mixture of acetone, water, and acetic acid (15.3 %). The results of the methanolic and ethanolic extracts derived from broccoli in the present study were slightly higher than those reported by Rahman et al. (2022). Consistent with the general trend, the yield in our study decreased as solvent polarity decreased (methanol: 26.4 %; ethanol: 19.2 %; acetone: 5.3 %). In separate work (El Mannoubi, 2023), the extraction yields of deseeded *Opuntia ficus indica* fruit were calculated using maceration with 80 % v/v solutions of acetone, methanol, and ethanol. In that study, the yields obtained by the protic solvents were substantially higher than those from acetone (acetone: 4.7 %; methanol: 40.9 %; ethanol: 57.7 %).

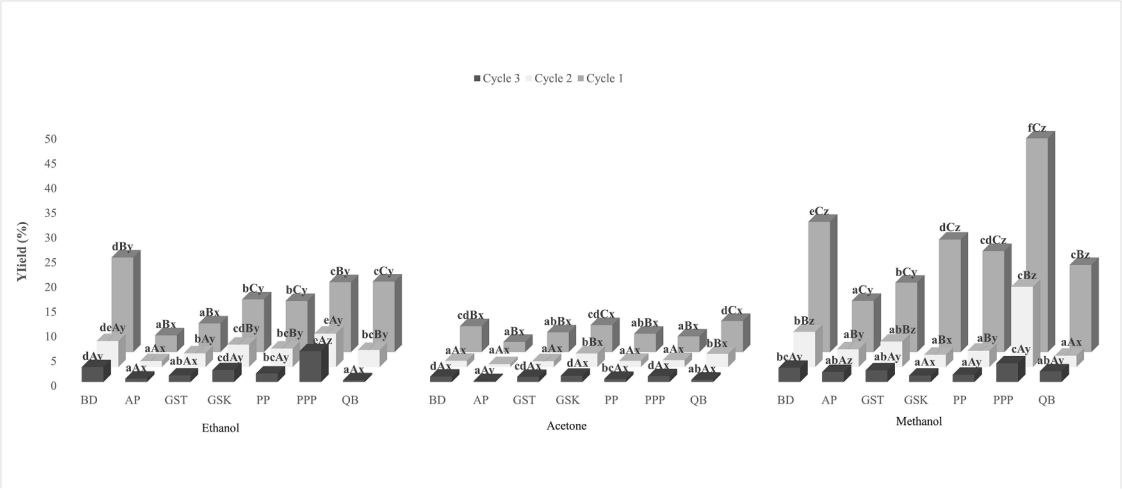

Fig. 1. Superscript letters (a–f) indicate significant differences ($p < 0.05$) between different by-products extracted with the same solvent, superscript letters (A–C) indicate significant differences ($p < 0.05$) between cycles in the same extraction of a by-product and superscript letters (x–z) indicate significant differences ($p < 0.05$) within an agrifood by-product extracted with three different solvents. BD: broccoli discard; AH: almond hull; GST: grape stem; PP: pea pod; GSK: grape skin; PPP: prickly pear peel; QB: quince by-products.

These findings align with the results obtained from PPP in the present study (acetone: 3.2 %; ethanol: 14.2 %; methanol: 43.3 %).

3.2. Determination of low molecular weight phenolic compounds by HPLC-DAD

The chemical composition of each extract was analyzed using HPLC-DAD. The quantification of individual compounds expressed as mg/g of extract provides valuable insight into both extract purity and the efficiency of compound recovery (Mariga et al., 2026). Higher concentrations of target compounds in the extract generally indicate greater selectivity of the extraction parameters, particularly solvent polarity, temperature, and cycle number, which determine not only total yield but also the enrichment of target bioactive molecules (Castillo-Correa et al., 2025). The analysis led to the identification of 13 compounds, primarily consisting of phenolic acids and flavonoids (Table 2). The identified benzoic acids included vanillic acid, ellagic acid, and gallic acid. Vanillic acid was only detected in the PP extract, and in a low concentration (0.2–0.6 mg/g extract). By contrast, ellagic acid was identified in four extracts (AH, GST, GSK, and PPP) in high concentrations (3.5–6.4 mg/g extract). Gallic acid was frequent across different samples (BD, AH, GST, PP, and GSK) but was less abundant than ellagic acid, with GSK methanol showing the highest concentration of this compound (2 mg/g extract). Cinnamic acid derivatives, including synapic acid (in BD), coumaric acid (in AH and PP), and caffeic acid (in BD and GST), were also present, but their concentrations were generally low compared with the other major compounds. We observed a clear partitioning of flavonoid types based on solvent polarity. Although present in low concentration (0.1–0.5 mg/g extract), quercetin was detected in all samples except BD and AH. The highest concentration of quercetin was found in the PPP acetone extract. This observation is consistent with the greater affinity of some aglycone flavonoids for aprotic solvents such as acetone, likely due to the hydrophobic nature of these compounds (Chebil et al., 2007; El Mannoubi, 2023). This behavior–higher concentration in acetone than in protic solvents–was also noticeable for viniferine in GST (acetone: 0.7; ethanol: 0.2; methanol: 0.2 mg/g extract) and GSK (acetone: 0.5; ethanol: 0.2; methanol: 0.2 mg/g extract). Conversely, other flavonoids, such as catechins, are more soluble in polar solvents due to their accessible hydroxyl groups, which make them good hydrogen donors (Chaves et al., 2020). For this reason, catechin was significantly more soluble in methanol than in ethanol or acetone in the case of GSK (methanol: 3.4; ethanol: 2.6; acetone: 2.7 mg/g extract) or was detected exclusively in methanol in the case of GST (1.4 mg/g extract). Epicatechin, the *cis* epimer of catechin, was the compound detected in the highest overall concentration in the BD sample (methanol: 38.6; ethanol: 16.8 and acetone: 6.1 mg/g extract), which is consistent with previous reports (Dębski et al., 2021; Kim et al., 2022). The only flavanol where the concentration in methanol was lower than in ethanol was epicatechin gallate detected in PPP (ethanol: 0.7, acetone: 0.1 and methanol: 0.1 mg/g extract). This difference could be attributed to its more lipophilic nature, which originates from the ester linkage to a gallic acid group (Sevillano et al., 2013). Chlorogenic acid, identified in GST (0.1–0.2 mg/g extract) and QB (0.2–0.5 mg/g extract), belongs to the group of caffeoylquinic acids formed by an ester linkage between caffeic acid and quinic acid. These compounds are widely reported for their antioxidant and antimicrobial properties (Kabir et al., 2014; Nguyen et al., 2024). The main compound identified by Othman et al. (2022) in the ethanolic extract of quince peel (ethanol 80 %) was *cis*-5-*O*-caffeoylquinic acid (0.5 mg/g extract). This concentration was similar to the total chlorogenic acid obtained in the present work using ethanol (0.3 mg/g extract) and methanol (0.5 mg/g extract).

3.3. Total phenolic content

Samples extracted with acetone showed the highest TPC values and the differences between solvents were statistically significant in the case of BD, GST, PPP, and QB (Table 3). Although methanol is the most common solvent employed for phenolic compound extraction (Gudiño et al., 2024), the TPC values of samples extracted with acetone are typically higher than those extracted with ethanol or methanol due to the abundance of lipophilic phenols (El Mannoubi, 2023). The grape by-products exhibited the greatest TPC values (58.5–81.7 and 56.2–78.8 mg GAE/g extract for GSK and GST, respectively). These results are higher than those reported by Li et al. (2019) in ethanolic extracts of grape skin under different conditions (12.0–17.1 mg GAE/g extract) and also higher than those reported by Dias-Costa et al. (2024) in methanolic extracts of stems of various grape varieties (26.1–50.0 mg GAE/g extract). The TPC values exhibited by AH (ethanol:14.7; acetone: 17.9; methanol: 22.0 mg GAE/g extract) were within the range of the results

Table 2
Concentration of low molecular weight phenolic compounds (mg/g extract) in the different agrifood by-products extracted with three different solvents.

	By-products	Solvents		
		Ethanol	Acetone	Methanol
Vanillic acid	PP	0.45 ± 0.01 ^{b}	0.16 ± 0.01 ^{a}	0.55 ± 0.01 ^{c}
Ellagic acid	AH	4.04 ± 0.00 ^{a}	4.07 ± 0.01 ^{b}	4.16 ± 0.00 ^{c}
	GST	5.09 ± 0.01 ^{b}	4.47 ± 0.07 ^{a}	6.41± 0.06 ^{c}
	GSK	5.34 ± 0.07 ^{b}	4.17 ± 0.03 ^{a}	6.09 ± 0.09 ^{c}
	PPP	3.61± 0.03 ^{b}	3.85 ± 0.07 ^{c}	3.53 ± 0.01 ^{a}
Gallic acid	BD	0.37 ± 0.02 ^{b}	nd ^{a}	0.64 ± 0.03 ^{c}
	AH	0.22 ± 0.03 ^{ab}	0.13 ± 0.06 ^{a}	0.36 ± 0.05 ^{b}
	GST	1.07± 0.05 ^{b}	0.45 ± 0.01 ^{a}	2.00 ± 0.04 ^{c}
	PP	0.14 ± 0.01 ^{a}	0.11± 0.01 ^{a}	0.26 ± 0.02 ^{b}
	GSK	0.67 ± 0.00	0.43 ± 0.01	1.18 ± 0.33
Synapic acid	BD	0.30± 0.01 ^{b}	0.06 ± 0.00 ^{a}	0.55 ± 0.02 ^{c}
Coumaric acid	AH	0.51 ± 0.00 ^{b}	1.11± 0.00 ^{c}	0.21 ± 0.00 ^{a}
	PP	0.07 ± 0.00 ^{a}	0.12± 0.01 ^{b}	0.08 ± 0.00 ^{a}
t-Caffeic acid	BD	0.49 ± 0.04 ^{c}	nd ^{a}	0.16 ± 0.00 ^{b}
	GST	1.27 ± 0.00 ^{b}	nd ^{a}	1.26 ± 0.03 ^{b}
Quercetin	GST	0.12 ± 0.00	0.13 ± 0.00	0.13 ± 0.00
	PP	0.01 ± 0.00 ^{a}	0.13 ± 0.00 ^{c}	0.11 ± 0.00 ^{b}
	GSK	0.17 ± 0.00 ^{c}	0.16± 0.00 ^{b}	0.15 ± 0.00 ^{a}
	PPP	0.17 ± 0.00 ^{a}	0.45 ± 0.00 ^{b}	0.13 ± 0.00 ^{a}
	QB	0.22 ± 0.00 ^{b}	0.18 ± 0.00 ^{a}	0.26 ± 0.00 ^{c}
Viniferine	GST	0.19 ± 0.00 ^{a}	0.65 ± 0.01 ^{b}	0.19 ± 0.01 ^{a}
	GSK	0.17 ± 0.01 ^{a}	0.46± 0.13 ^{b}	0.18± 0.03 ^{a}
Catechin	GST	nd ^{a}	nd ^{a}	1.42 ± 0.03 ^{b}
	GSK	2.63 ± 0.04 ^{a}	2.78 ± 0.03 ^{a}	3.43 ± 0.01 ^{b}
Epicatechin	BD	16.77 ± 3.00 ^{b}	6.13 ± 0.35 ^{a}	38.56 ± 0.87 ^{c}
Epigallocatechin	BD	0.62 ± 0.01 ^{b}	0.17± 0.00 ^{a}	1.00 ± 0.02 ^{c}
Epicatechin gallate	PPP	0.68 ± 0.00 ^{b}	0.13 ± 0.00 ^{a}	0.10± 0.00 ^{a}
Chlorogenic acid	GST	0.09 ± 0.00 ^{a}	0.10 ± 0.00 ^{a}	0.19 ± 0.00 ^{b}
	QB	0.34 ± 0.01 ^{b}	0.18 ± 0.00 ^{a}	0.47 ± 0.01 ^{c}

[1]BD: broccoli discard; AH: almond hull; GST: grape stem; PP: pea pod; GSK: grape skin; PPP: prickly pear peel; QB: quince by-products. $^{a–b}$ in the same row indicates significant differences ($p < 0.05$) between solvents; nd: non detected.

Table 3
Total phenolic content expressed as mg GAE/g extract of seven agrifood by-products extracted with three different solvents.

Sample	Solvent		
	Ethanol	Acetone	Methanol
BD	19.77 ± 4.46 ^{ax}	25.97 ± 2.65 ^{ay}	18.95 ± 3.17 ^{ax}
AH	14.67 ± 7.18 ^{a}	17.88 ± 1.65 ^{a}	22.04 ± 4.56 ^{a}
GST	58.49 ± 6.21 ^{bx}	67.00 ± 4.14 ^{by}	56.23 ± 3.85 ^{bx}
GSK	81.69 ± 5.50 ^{c}	78.79 ± 10.21 ^{b}	76.46 ± 9.24 ^{c}
PP	13.62 ± 2.27 ^{a}	17.60 ± 1.48 ^{a}	14.84 ± 3.52 ^{a}
PPP	14.86 ± 0.70 ^{ax}	23.08 ± 3.78 ^{ay}	17.80 ± 1.30 ^{ax}
QB	26.78 ± 2.68 ^{ax}	31.59 ± 3.52 ^{ay}	25.67 ± 5.06 ^{bx}

[1]BD: broccoli discard; AH: almond hull; GST: grape stem; PP: pea pod; GSK: grape skin; PPP: prickly pear peel; QB: quince by-products.
$^{a-c}$ superscripts indicate significant differences ($p < 0.05$) within by-products extracted with the same solvent.
$^{x-y}$ superscripts indicate significant differences ($p < 0.05$) within by-products extracted with different solvents.

shown by Ben-Khadher et al. (2023) and by Sfahlan et al. (2009) in hydromethanolic extracts (13.0 and 35.9 mg GAE/g extract, respectively), were higher than that obtained by Valdés et al. (2022) in hydroethanolic extracts (6.3 mg GAE/g extract), and were lower than those extracted with acetone by Meshkini (2016), whose reported concentration was 78.1 mg GAE/g extract.

The TPC results of BD described in the present study (19.8–26.0 mg GAE/g extract) were higher than those obtained in broccoli stem (224.2 μg GAE/g extract) through microwave-assisted extraction (García & Raghavan, 2022), and those from traditional extraction of broccoli crop surpluses (3.0 mg GAE/g extract) with hydroalcoholic ethanol (Villaño et al., 2023). The increased efficiency of HPLE techniques such as ASE over other extraction techniques has established it as the reference method for the scaling of plant-waste valorization (Gil-Martín et al., 2022) and the high TPC yields obtained in this work support this trend.

3.4. Scavenging activity of DPPH

The influence of the solvent on the antioxidant activity of each by-product, as well as the activity of the samples within each solvent, was analyzed and statistically compared (Table 4). In the case of GST and GSK, methanolic (6.7 and 6.1 mmol TEAC/100 g extract, respectively) and ethanolic (6.3 and 6.3 mmol TEAC/100 g extract, respectively) extracts exhibited higher DPPH inhibition than those extracted with acetone (5.1 and 5.0 mmol TEAC/100 g extract, respectively). By contrast, the PPP acetone extract had significantly more antioxidant activity (5.1 mmol TEAC/100 g extract) than the ethanol (3.1 TEAC/100 g extract) and methanol (3.3 mmol TEAC/100 g extract) extracts. The

Table 4
Scavenging activity of DPPH expressed as mmol TEAC/100 g extract of seven agrifood by-products extracted with three different solvents.

Sample	Solvent		
	Ethanol	Acetone	Methanol
BD	4.75 ± 0.61 ^{abc}	2.98 ± 0.82 ^{a}	4.57 ± 0.31 ^{b}
AH	4.03 ± 1.38 ^{ab}	3.71 ± 0.85 ^{a}	6.65 ± 0.28 ^{d}
GST	6.31 ± 0.55 ^{bcxy}	5.09 ± 0.16 ^{bcx}	6.68 ± 0.16 ^{dy}
GSK	6.26 ± 0.08 ^{bcy}	5.01 ± 0.20 ^{bcx}	6.12 ± 0.12 ^{cdy}
PP	4.24 ± 0.04 ^{ab}	3.35 ± 1.04 ^{a}	4.97 ± 0.55 ^{bc}
PPP	3.12 ± 0.13 ^{ax}	5.12 ± 0.23 ^{bcy}	3.29 ± 0.25 ^{ax}
QB	6.97 ± 0.06 ^{c}	6.48 ± 0.22 ^{c}	6.95 ± 0.21 ^{d}

[1]BD: broccoli discard; AH: almond hull; GST: grape stem; PP: pea pod; GSK: grape skin; PPP: prickly pear peel; QB: quince by-products.
$^{a-c}$superscripts indicate significant differences ($p < 0.05$) within by-products extracted with the same solvent.
$^{x-y}$superscripts indicate significant differences ($p < 0.05$) within by-products extracted with different solvents.

by-products with the lowest antioxidant activity were PPP in ethanol and methanol (3.1 and 3.3 mmol TEAC/100 g extract, respectively) and BD, PP, and AH in acetone (3.0, 3.4, and 3.7 mmol TEAC/100 g extract, respectively). QB was the most antioxidant by-product in the three solvents (6.5–7.0 mmol TEAC/100 g extract), followed by GST (5.1–6.7 mmol TEAC/100 g extract) and GSK (5.0–6.3 mmol TEAC/100 g extract). The high antioxidant ability of QB is likely related to the high concentration of quercetin and chlorogenic acid detected by HPLC (Table 2). Quercetin is renowned as one of the flavonoids with the greatest radical-scavenging ability (Aghababaei & Hadidi, 2023) and chlorogenic acid, a hydroxycinnamic acid, has proven antioxidant activity across various assays (DPPH, ABTS, and FRAP tests) (Huang et al., 2023). Both of these compounds are consistently identified as predominant compounds in QB in numerous studies, where different authors associate them with the high TPC and DPPH inhibition values exhibited by these samples (Fattouch et al., 2007; Silva et al., 2023; Maghsoudlou et al., 2019). As in the present work (Table 2), these compounds were also identified in grape by-products by Nile et al. (2013) in skin and by Esparza et al. (2020) in stem, where high TPC and antioxidant values were also reported.

3.5. Antifungal activity

The antifungal activity of the different sample extracts against diverse mold strains was analyzed and statistically compared, examining the influence of the solvent within each by-product and the activity of the samples within each solvent (Table 5). Natamycin activity also was measured to compare the results with a reference antibiotic used in dairy industry. In general, neither the extracts (2.6 %−7.2 %) nor natamycin (2.9 %−12.8 %) showed high antifungal activity, indicating that these mold strains are highly resistant. Indeed, the extracts were not active against two of the seven strains tested (*P. expansum,* QZ1 and *P. commune/biforme,* 601). All the ethanolic extracts were active against 1B01 (*P. crustosum*) with PPP and PP showing the highest values (4.7 % and 4.6 %, respectively). In the acetone extractions, BD (7.4 %) was the most active. Methanolic extracts from AH, GST, PP, and GSK showed similar activity against this strain. All extracts derived from GST and QB showed antifungal activity against 301 (*P. commune)* and no significant differences were observed among the solvents used for these extractions. Contrastingly, BD, GSK and PPP extracts were not active against this strain. The highest antifungal activity against M32 (*P. nordicum*) was shown by the methanolic extracts (GST followed by PP), and the only by-product extracted with acetone with the ability to inhibit this mold was PP (3.0 %). Notably, while natamycin showed maximum inhibition against M32 (12.8 %), the activity of this antibiotic against the *Aspergillus* strains tested (*A. puulaauensis,* 1A05 and *A. jensenii,* 501) was the lowest (2.9 % and 3.1 %, respectively). All the extracts with activity against 1A05 (*A. puulaauensis*) showed a greater ability to inhibit this mold than natamycin (2.9 %). For the ethanolic extracts, all samples were active, with no significant differences found between them. Among the acetone samples, AH, PP, and PPP showed activity, with AH acetone being the most effective (6.9 %). The activity against *A. jensenii* 501 was similar to that of natamycin in the case of AH ethanol (2.9 %) together with GST and PPP acetone (2.6 % and 2.9 %, respectively). The remaining active extracts (GST and QB in ethanol, and AH, GST, PP, and QB in methanol) showed higher inhibition than the antibiotic, with AH methanol exhibiting more than double the activity (6.5 %). These results clearly indicate that the antifungal activity of the extracts is highly dependent on the solvent chosen and the specific mold strain. Furthermore, in several key instances, the tested extracts demonstrated activity that was equal to or greater than that of natamycin, an authorized antibiotic used in the food industry.

The search for antimicrobial properties in agrifood by-products has been widely explored by other authors, providing strong evidence of their potential. Previous work by Fattouch et al. (2007) associated the activity of QB against pathogens such as *Staphylococcus aureus,*

Table 5
Comparison of fungal inhibition (%) between by-product extracts (10 mg/ml) and between solvents within the same agrifood by-product.

Sample	*P. crustosum* (1B01)			*P. commune* (301)			*P.nordicum* (M32)		
	Ethanol	Acetone	Methanol	Ethanol	Acetone	Methanol	Ethanol	Acetone	Methanol
BD	3.96 ± 0.14 ^{ab}	7.36 ± 1.57 ^{b}	nd	nd	nd	nd	nd	nd	nd
AH	3.23 ± 0.04 ^{ab}	2.37 ± 0.41 ^{a}	4.20 ± 1.28	nd	3.60 ± 0.80	2.75 ± 1.17	5.13 ± 1.44	nd	3.17 ± 0.75
GST	3.87 ± 0.48 ^{ab}	3.34 ± 0.30 ^{a}	4.84 ± 0.99	3.79 ± 0.19	4.09 ± 0.15	5.51 ± 1.79	5.21 ± 0.38	nd	7.08 ± 2.85
GSK	3.53 ± 0.14 ^{ab}	nd	3.21 ± 0.38	nd	nd	nd	nd	nd	nd
PP	4.59 ± 0.74 ^{b}	4.56 ± 0.92 ^{ab}	2.98 ± 0.50	nd	2.92 ± 1.15	nd	nd	3.01 ± 1.41	6.69 ± 2.01
PPP	4.67 ± 0.82 ^{b}	nd	nd	nd	nd	nd	4.40 ± 2.75	nd	nd
QB	2.58 ± 0.14 ^{a}	nd	nd	4.23 ± 1.45	3.49 ± 0.66	4.41 ± 1.38	nd	nd	nd
Natamycin	10.82 ± 1.76			12.77 ± 1.29			12.81 ± 0.05		

Sample	*A. puulaauensis* (1A05)			*A. jensenii* (501)		
	Ethanol	Acetone	Methanol	Ethanol	Acetone	Methanol
BD	5.08 ± 2.31	nd	3.12 ± 0.16	nd	nd	nd
AH	3.63 ± 1.72	6.93 ± 2.77	nd	2.85 ± 0.19 ^{x}	nd	6.49 ± 0.34 ^{y}
GST	5.06 ± 0.78	nd	nd	3.91 ± 0.86	2.63 ± 0.71	4.00 ± 0.28 ^{a}
GSK	6.94 ± 1.74	nd	4.03 ± 0.71	nd	nd	nd
PP	3.56 ± 2.66	4.83 ± 0.01	3.10 ± 0.29	nd	nd	4.19 ± 0.69 ^{a}
PPP	3.02 ± 0.64	3.61 ± 0.70	nd	nd	2.90 ± 0.92	nd
QB	5.63 ± 1.55	nd	3.28 ± 0.19	4.11 ± 0.19 ^{y}	nd	3.65 ± 0.20 ^{ax}
Natamycin	2.88 ± 1.29			3.11 ± 1.68		

[1]BD: broccoli discard; AH: almond hull; GST: grape stem; PP: pea pod; GSK: grape skin; PPP: prickly pear peel; QB: quince by-products. NS: no significant. nd: no detected.
$^{a-b}$ superscripts indicate significant differences ($p < 0.05$) between by-products extracted with the same solvent.
$^{x-y}$ superscripts indicate significant differences ($p < 0.05$) within by-products extracted with different solvents.
* Natamycin: Used as a positive control.

Pseudomonas aeruginosa, Escherichia coli, and *Candida albicans* with their high concentration of chlorogenic acid, present in the peel and pulp of this fruit. Similarly, Thery et al., 2020 identified a napin-like protein in aqueous extracts of broccoli seeds capable of inhibiting cereal contaminants such as *Fusarium culmorum, Aspergillus niger* and *Penicillium expansum*. Furthermore, studies on prickly pear residues and AH have demonstrated their efficacy against various phytopathogenic fungi (Alqurashi et al., 2022; Coronado-Contreras et al., 2023; (Mozaffari et al., 2024). All these results underscore the significant potential of agrifood waste as a source of antimicrobial agents.

3.6. Ranking the by-products using a scoring system

To evaluate and compare the bioactivity of each by-product as a function of the solvent employed, a scoring system was developed, primarily aimed at classifying their potential as an antifungal preservative. The score integrated metrics for TPC, antioxidant activity, and antifungal activity. The aim was to classify them based on their potential

Table 6
Ranking of the agrifood by-products extracted with ethanol, acetone, and methanol using score calculation based on different tests made in this research.

Extract	Antioxidant activity (mmol TEAC /100 g)	TPC (mg GAE /g extract)	N° molds inhibited	Relative inhibition (%)	Antioxidant activity*	TPC *	N° molds inhibited *	Relative inhibition (%) *	Final Score *	Classification
BDe	4.75	19.77	2	106.44	2	2	1	5	20	excellent
BDa	2.98	25.97	1	68.16	1	3	1	2	11	bad
BDm	4.57	18.95	1	108.33	2	2	1	5	20	excellent
AHe	4.03	14.67	4	71.90	2	1	3	2	12	average
AHa	3.71	17.88	3	96.89	1	2	2	4	17	good
AHm	6.65	22.04	4	73.41	4	2	3	3	18	good
GSTe	6.31	58.49	5	81.54	3	4	4	3	20	excellent
GSTa	5.09	67.00	3	49.15	3	4	2	1	12	average
GSTm	6.68	56.23	4	67.94	4	3	3	2	16	good
GSKe	6.26	81.69	2	136.84	3	4	1	5	23	excellent
GSKa	5.01	78.79	1	139.88	2	4	1	5	22	excellent
GSKm	6.12	76.46	1	29.66	3	4	1	1	11	bad
PPe	4.24	13.62	2	83.02	2	1	1	3	13	average
PPa	3.35	17.60	4	64.05	1	1	3	2	11	bad
PPm	4.97	14.84	4	84.69	2	1	3	3	15	average
PPPe	3.12	14.86	3	60.74	1	1	2	2	10	bad
PPPa	5.12	23.08	2	109.43	3	3	1	5	22	excellent
PPPm	3.29	17.80	0	0.00	1	2	0	0	3	bad
QBe	6.97	26.78	4	96.18	4	3	3	4	22	excellent
QBa	6.48	31.59	1	27.31	4	3	1	1	11	bad
QBm	6.96	25.67	3	88.55	4	3	2	3	18	good

[1]BD: broccoli discard; AH: almond hull; GST: grape stem; PP: pea pod; GSK: grape skin; PPP: prickly pear peel; QB: quince by-products. e: ethanol; a: acetone; m: methanol.
* Item: pondered according to the equation from chapter 2.8. The quartile-based ranges for each parameter were: Antioxidant activity ($x \leq 3.87$, **1**; $3.87 < x \leq 5.01$, **2**; $5.01 < x \leq 6.39$, **3**; $6.39 < x \leq 6.97$, **4**); TPC ($x \leq 17.697$, **1;** $17.697 < x \leq 23.08$, **2**; $23.08 < x \leq 57.359$, **3**; $57.359 < x \leq 81.69$, **4**); N° molds inhibited ($x = 0$, **0**; $0 < x \leq 2$, **1;** $2 < x \leq 3$, **2**; $3 < x \leq 4$, **3;;** $4 < x \leq 5$, **4**); Relative inhibition (%) ($x = 0$, 0; **0** $< x \leq 54.94$, **1**; $54.94 < x \leq 71.9$, **2**; $71.9 < x \leq 92.36$, **3**; $92.36 < x \leq 100$, **4**; $x > 100$, **5**); Final score ($x \leq 11$, **"bad"**; $11 < x \leq 15$, **"average"**; $15 < x \leq 19$, **"good"**; $19 < x \leq 23$, **"excellent"**).

as antifungal preservatives. For this specific goal, the antifungal activity was weighted more heavily: the inhibition percentage relative to natamycin was multiplied by three, giving it higher relevance in the final score. The final scores led to the classification of the extracts (Table 6). The extracts with the highest final scores ($x \geq 20$, "excellent") were BDe, BDm, GSTe, GSKe, GSKa, PPPa and QBe. The following in the ranking were those designated as "good" ($15 \leq x \leq 19$) and were AHa, AHm, GSTm and QBm. The remaining samples were classified as "average" and "bad". Although four of the seven by-products with the highest scores were obtained using ethanol as the extraction solvent, no clear pattern emerged linking a specific solvent type to overall bioactivity. However, the ranking strongly suggests superior intrinsic bioactivity in certain by-products regardless of the solvent. Specifically, the three extracts derived from GSK and those obtained by methanolic and ethanolic extraction of BD achieved scores above 20 and ranked among the top-performing samples. These results suggest that both by-products possess strong potential as antimicrobial and antioxidant agents.

The richness of bioactive compounds and documented benefits found in broccoli and grape residues have driven extensive industrial application research. Specifically, the phenolic and polyphenolic compounds from grape by-products are being explored for cosmetical formulations, demonstrating anti-aging properties and photoprotective capacity against UV and visible light (Mulinacci et al., 2019). Furthermore, extracts from vinicultural waste, high in proanthocyanidins, have been incorporated into topical products active against fungi causing mucocutaneous infections, with some preparations currently undergoing registration for human and veterinary use (Simonetti et al., 2020). Similarly, extracts derived from broccoli by-products–often obtained *via* methods such as supercritical fluid extraction with ethanol–have shown promise as food preservatives, extending the shelf life of items such as pasta and fish burgers (Angiolillo, Spinelli, Conte et al., 2019, 2019). Key compounds in these extracts, such as glucosinolates, also exhibit activity against various phytopathogenic fungi, highlighting their potential as natural pesticides (Eugui et al., 2023). Moving forward, the primary industrial focus is on standardizing and reproducing the extraction processes to ensure a safe, consistent, and low-cost supply of these natural preservatives (Simonetti et al., 2020).

The establishment of a ranking based on a scoring equation in which different parameters were pondered according to the intended application represents a practical and objective decision-making tool. This approach enables the integration of multiple relevant criteria into a single comparative framework, facilitating the identification of the most suitable by-products for the targeted use. Furthermore, this selection matrix is not limited to the present study but can be extrapolated to other research contexts in which samples are characterized through multiple properties and require prioritization according to defined application-driven criteria.

4. Conclusions

In this study, the extraction of seven agrifood by-products were carried out by accelerated solvent extraction method. Extraction optimization revealed that the solvent was the most determining factor for the yield, with the most efficient solvent being methanol, followed by ethanol. The extraction solvent also had a significant impact on the concentration of phenolic compounds, the specific phenolic profile, and the resulting antifungal activity of each sample. Based on a comprehensive evaluation of the TPC, antioxidant activity, and antifungal performance, a final ranking identified seven extracts with the most favorable characteristics as antimicrobial and antioxidant agents: three derived from grape by-products, two from broccoli discards, one from prickly pear peel, and one from quince by-products. This scoring system provided a versatile and transferable methodology to support the selection of these by-products based on their potential application.

Scaling up to an industrial level would be straightforward, as the technologies for extraction and subsequent purification are widely available in the industry. Furthermore, the use of food-grade solvents such as ethanol further enhances safety and ensures compliance with regulatory requirements in food application. Once dry after ethanol removal, the resulting product would have a low microbial load due to the production method and could be formulated by adding directly or by using appropriate food-grade carriers to ensure homogeneous incorporation into complex food matrices. A simple redissolution protocol would be established for industrial use and the product could be applied as an additional step during the formulation or blending stage of functional food production. Therefore, this research provides a robust foundation for the design of green preservatives by an environmentally friendly method, contributing directly to sustainable development. Moreover, the results of this study contribute to the principles of circular economy and zero-waste policies by promoting the valorization of natural resources.

Funding

This work and the predoctoral contract of M.C. Codina was supported by the Spanish Ministry of Science and Innovation and by the Grant PID2021–122145OR-C22 funded by MCIN/AEI/ 10.13039/ 501100011033 and by "ERDF A way of making Europe" and by the 2022-GRIN-34,372 project co-financed by the Universidad de Castilla-La Mancha and the European Regional Development Fund. E. Aranda (2023-INVGO-11,949 founded by the Investigo Program funded by JCCM, PRTR and Next Generation EU program).

Ethical statement - studies in humans and animals

This work does not include studies on humans or animals.

CRediT authorship contribution statement

M.C. Codina: Writing – original draft, Methodology, Investigation, Formal analysis. **Riccardo Gannuscio:** Investigation, Formal analysis. **A. Molina:** Writing – review & editing, Resources, Funding acquisition. **M. Carmona:** Writing – review & editing, Supervision, Formal analysis. **M.I. Berruga:** Writing – review & editing, Supervision, Resources, Project administration, Investigation, Funding acquisition, Conceptualization.

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.

Acknowledgements

The authors also thank Rafael Molina (Membrillo Emily S.L.) and Irene García (Ultracongelados Campo Verde S.L.) for the supply of the by-products, Dr. Cebrián (ETSIAMB-UCLM) for her support with SBSE-GC–MS and Elena Aranda Peco for their technical assistance in the laboratory. We thank Dr. Kenneth McCreath for English editing

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.afres.2026.101959.

Data availability

Data will be made available on request.

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