

[https://doi.org/10.52326/jes.utm.2025.32\(4\).08](https://doi.org/10.52326/jes.utm.2025.32(4).08)

UDC 637.146:634.8:663.26



EFFECT OF GRAPE SKIN INCORPORATION ON THE QUALITY OF A DAIRY-BASED FROZEN DESSERT

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Received: 12. 02. 2025

Accepted: 12. 28. 2025

Abstract. The valorisation of winemaking by-products is an important approach for the development of functional foods. This study investigated the effect of grape skin powder (GSP, 0–10%, *m/m*) incorporation on the quality of a laboratory-scale dairy-based frozen dessert prototype. Five formulations (0, 2.5, 5.0, 7.5 and 10% GSP) were evaluated in terms of physicochemical properties, oxidative stability, colour parameters, microbiological quality and sensory acceptability. Increasing GSP levels led to a progressive decrease in pH (6.72–5.62) and a slight increase in fat content (26.25–27.66%), while dry matter remained above 54%. Lipid oxidation during frozen storage was reduced in GSP-fortified samples, with peroxide values decreasing from 1.70 meq O₂/kg fat in the control to 1.36 meq O₂/kg fat at 10% GSP after 28 days. Pronounced colour changes were observed ($\Delta E^* = 37-55$), associated with grape skin anthocyanins. Functional enrichment was confirmed by increased total polyphenol content (up to 139 mg GAE/100 g) and antioxidant activity (up to 33.6%). Moderate GSP addition (2.5–5.0%) was associated with lower microbial counts, while higher levels introduced additional indigenous microflora. Sensory evaluation indicated good overall acceptability, with the 5% GSP formulation providing the most balanced profile. Overall, grape skin powder shows strong potential as a functional ingredient for dairy-based frozen desserts.

Keywords: *antioxidant activity, grape skin, dairy-based frozen dessert, microbiological quality.*

Rezumat. Valorificarea subproduselor din vinificație reprezintă o direcție importantă pentru dezvoltarea alimentelor funcționale. Prezentul studiu a investigat efectul incorporării pulberii de pieleță de struguri (GSP, 0–10%, *m/m*) asupra calității unui desert lactat congelat, obținut la scară de laborator. Cinci formulări (0, 2,5, 5,0, 7,5 și 10% GSP) au fost evaluate din punct de vedere al proprietăților fizico-chimice, stabilității oxidative, parametrilor de culoare, calității microbiologice și acceptabilității senzoriale. Creșterea nivelului de GSP a determinat o scădere progresivă a pH-ului (6,72–5,62) și o ușoară creștere a conținutului de grăsime (26,25–27,66%), în timp ce substanța uscată a rămas peste 54% pentru toate probele.

Oxidarea lipidică în timpul depozitării la congelare a fost redusă în probele fortificate cu GSP, valoarea peroxidică scăzând de la 1,70 meq O₂/kg grăsime în proba martor la 1,36 meq O₂/kg grăsime pentru 10% GSP după 28 de zile. S-au observat modificări cromatice pronunțate ($\Delta E^* = 37-55$), asociate cu antocianinele din pielea de struguri. Îmbogățirea funcțională a fost confirmată prin creșterea conținutului total de polifenoli (până la 139 mg GAE/100 g) și a activității antioxidante (până la 33,6%). Nivelurile moderate de GSP (2,5–5,0%) au fost asociate cu încărcături microbiene mai reduse, în timp ce adaosurile mai mari au introdus microfloră indigenă suplimentară. Evaluarea senzorială a indicat o bună acceptabilitate generală, formularea cu 5% GSP prezentând cel mai echilibrat profil. În ansamblu, pulberea de piele de struguri prezintă un potențial ridicat ca ingredient funcțional pentru deserturile lactate congelate.

Cuvinte cheie: *activitate antioxidantă, piele de struguri, desert lactat congelat, calitate microbiologică.*

1. Introduction

Until the end of the 20th century, increasing food production without improving the efficiency of food systems was the prevailing policy throughout the world. Nowadays, increasing demands for processed food has required certain strategies to minimize energy demands and economic costs, as well as to reduce food losses and waste [1]. The quantities of the latter are huge, and the figures are shocking. The Food and Agriculture Organization published a report in 2011 listing food losses and wastage around the world and reporting that the amount of food lost or wasted produced for human consumption amounts to 1.3 million tonnes globally each year [2]. The European Commission has also mentioned this significant problem, as in 2010 it indicated that 90 million tonnes of food waste is generated annually in the EU [3].

Grapes are recognized as a valuable plant-derived source of antioxidants, notably resveratrol, anthocyanins, catechins, and quercetin. The grapes contain skin, pulp, seeds and stems and all of the presented components may contain antioxidants. Mostly antioxidants are found in the skin, as are coloring substances [4]. Grapes also contain the polyphenol resveratrol, which is well known for its therapeutic effects. This substance is located in the skin of grapes [5]. It is known for its antioxidant properties and was found in red grapes in large amounts, which is why red grapes have more antioxidants than white grapes [6].

Most of the grapes harvested worldwide are used to make wine, while winemaking has been one of the most important industries in many countries [7]. Along with the growing global trend for wine consumption comes an increasing volume of accompanying by-products such as grape marc, seeds, skins, stems, leaves and wine lees. These by-products are an interesting source for further exploitation as they are rich in polyphenols, tannins, procyanidins, anthocyanins, stilbenes, dietary fibres, tartrates, essential oils, proteins, tocopherols, etc. [8]. In this context, they have the potential to be used as a source of ingredients that could be used in different applications in functional foods, dietary supplements, pharmaceuticals and cosmetics, as well as for agricultural and oenological purposes [9].

Due to the fact that grapes have valuable components that can be used to enrich any food product, this topic remains very relevant in our time and in our territory [10]. However, despite the well-known functional potential of grape skins, their application in frozen dairy products remains insufficiently explored, especially with regard to their technological impact,

oxidative stability, microbiological behaviour and sensory acceptance. Dairy-based frozen desserts represent one of the most popular dessert categories, widely consumed by individuals across all age groups and demographic segments. Various artificial colorants are used in their manufacture, such as tartrazine (E102), sunset yellow FCF (E110), Allura Red AC (E129), Brilliant Blue FCF (E133) and Fast Green FCF (E143), which are among the most commonly used colorants in dessert production. The use of natural colorants is a viable alternative to synthetic colorants, as they have superior biocompatibility, are free of cumulative toxic effects, and contribute to safer food products from a nutritional and functional standpoint. Therefore, the aim of this study was to evaluate the impact of grape skin powder (GSP) addition on the quality and functional properties of dairy-based frozen dessert, and to assess its potential as a natural ingredient for valorising winemaking by-products.

2. Materials and Methods

2.1 Materials

Grape pomace (Feteasca Neagră variety, Republic of Moldova) was obtained as a secondary by-product from a local wine producer. Prior to the experimental procedures, the pomace was stored at -20 ± 1 °C to preserve its chemical stability. Before processing, the grape skins were manually separated from seeds and remaining stem fragments. The grape skins were dried by convection at 60 ± 1 °C until reaching a final moisture content of $6.80 \pm 0.12\%$. The dried material was then ground using a coffee grinder (Bosch TSM6A013B, Bosch GmbH, Germany) and subsequently sieved through a 150 µm sieve to obtain a fine grape skin powder (GSP) suitable for incorporation into various food matrices. The resulting GSP was stored in airtight plastic bags at 4 ± 1 °C in a light-protected environment until further use.

Ingredients used for the dessert production included sweet cream (35% fat, JLC Ltd., Republic of Moldova), sugar and grape skin powder.

2.2 Method of Dairy-Based Frozen Dessert Prototype Preparation

The product developed in this study represents a laboratory-scale dairy-based frozen dessert prototype designed to evaluate the feasibility of grape skin powder incorporation in frozen dairy systems. The basic dairy-based frozen dessert formulation was adjusted to allow its enrichment with grape skin powder (GSP). Five variants were prepared by supplementing the formulation with different levels of GSP: 0% (control), 2.5%, 5.0%, 7.5% and 10.0%. The supplementation was performed by adding the corresponding amounts of GSP to the base recipe, while keeping the quantities of the base ingredients constant across all formulations, in order to avoid alterations in sweetness intensity and freezing point depression, which are key factors governing texture development and ice crystal formation in frozen dairy systems. Since no compensatory sweetening or structuring agents were applied, maintaining a constant sucrose level ensured that the observed effects could be primarily attributed to GSP incorporation.

Prior to processing, the bowl of the ice cream maker was placed in the freezer for at least 24 h, and all raw materials were kept under refrigerated conditions to ensure optimal freezing performance. Table 1 presents the ingredients and their respective proportions used in the preparation of the dairy-based frozen dessert formulations.

The liquid phase, consisting of sweet cream (35% fat) and sucrose (representing one-third of the total formulation mass), was transferred into the pre-chilled bowl of the ice cream machine (Cuisinart ICE31GE, USA).

Table 1

Formulation of Dairy-Based Frozen Dessert Samples with Constant Base Ingredients and Variable Grape Skin Powder Addition

Sample	Sweet cream, g	Sugar, g	Grape skin powder*, g
Control sample	75.0	25.0	0.0
2.5 % GSP	75.0	25.0	2.6
5.0 % GSP	75.0	25.0	5.3
7.5 % GSP	75.0	25.0	8.1
10.0 % GSP	75.0	25.0	11.1

*The amount of grape skin powder (GSP) was calculated to represent 2.5, 5.0, 7.5 and 10.0% (m/m) of the final product mass.

After gentle mixing, the required amount of GSP powder was incorporated, and homogenization was initiated using the electric ice cream maker for 40–50 min, until a soft cream consistency was achieved. Following production, all samples were stored in a freezer at -27 ± 1 °C until further physicochemical, microbiological and sensory analyses were carried out.

2.3 Physicochemical Analysis

The total solids content (DM) of grape skin and frozen dessert samples were determined gravimetrically by measuring the mass remaining after drying, according to AOAC Method 941.08. The moisture content was calculated as the complement of DM content and expressed as percentage of total sample mass. The pH of the samples was measured using a calibrated pH meter (TESTO 206-pH2, Pruszków, Poland), employing buffer solutions of pH 4.0 and 7.0. Measurements were performed by immersing the electrode directly into a homogenised sample previously macerated with distilled water, following AOAC (Method 945.10) recommendations. The total fat content (FC) of the frozen dessert samples was determined according to AOAC Method 952.06, using an ether extraction procedure. The method is based on solvent extraction of lipids with petroleum ether, followed by gravimetric determination of the extracted fat.

2.4. Acidity Index and Peroxide Value

Given the high fat content of the dairy-based frozen dessert prototype and its susceptibility to oxidation, both the acidity index (AI) and peroxide value (PV) were determined using AOAC (1997) official methods [11]. Milk fat was extracted from fresh and stored samples of the frozen dairy dessert prototype according to AOAC Method 960.32 in order to obtain the lipid fraction for oxidative stability analysis. Primary oxidation products were quantified by determining the peroxide value using AOAC Method Cd 8-53. The content of free fatty acids was assessed following AOAC Method Ca 5a-40. Analyses were carried out at different stages of processing and storage: (i) immediately after homogenization of the frozen dairy dessert mixture (day 0), and (ii) after freezing and packaging. During frozen storage, peroxide value and free fatty acid content were determined in all experimental replicates at 7, 14, 21, and 28 days to evaluate the evolution of lipid oxidation over time.

2.5. Colour Parameters

Colour parameters L^* , a^* , and b^* were determined using a chromameter (Minolta CR-400, Osaka, Japan), where L^* indicates lightness, a^* represents red–green values and b^* represents yellow–blue values. For all samples, the total colour difference (ΔE^*) was calculated according to Clydesdale [12].

2.6. Total Polyphenol Content (TPC)

2.6.1. Determination of Total Polyphenol Content in Grape Skin Powder

TPC of GSP was quantified using the Folin–Ciocalteu method, as described by Waterman and Mole, with minor adaptations [13]. Polyphenolic compounds were extracted using 50% (v/v) ethanol as solvent, at a solid-to-solvent ratio of 1:3 (w/v). Due to the high concentration of phenolic compounds in grape skin powder, the resulting extract was further diluted 1:10 prior to analysis. Absorbance was measured spectrophotometrically, and results were calculated based on a gallic acid calibration curve (0–10 mg/L; $y = 0.5056x + 0.0650$; $R^2 = 0.9977$). TPC was expressed as mg gallic acid equivalents per g of sample (mg GAE/g).

2.6.2. Determination of Total Polyphenol Content in Dessert Samples

For dairy-based frozen dessert prototype samples, a separate extraction procedure was applied in order to account for the complex food matrix and the high fat content. Samples were homogenized and polyphenolic compounds were extracted using 50% (v/v) ethanol, at a sample-to-solvent ratio of 1:3 (w/v). The mixture was subjected to centrifugation (≈ 5000 rpm, 10 min) to promote phase separation, and only the ethanolic supernatant was collected for subsequent analysis. This approach minimized lipid interference and ensured reliable spectrophotometric determination of phenolic compounds, in accordance with procedures reported for dairy and high-fat food matrices. The TPC of all samples was determined using the Folin–Ciocalteu method, based on the gallic acid calibration curve, and results were expressed as mg GAE/100 g of sample.

2.7. DPPH Antioxidant Activity (AA)

AA was evaluated using the DPPH radical scavenging assay and expressed as percentage of DPPH inhibition (%), in accordance with the method of Brand-Williams et al. [14]. The analysis was performed using the ethanolic extracts prepared for TPC determination, as described in Section 2.6.1 and 2.6.2, and results were expressed as percentage of DPPH radical inhibition.

2.8. Sensory Evaluation

Sensory evaluation of dairy-based frozen dessert prototype samples was performed by 37 non-smoking assessors aged 23–70 years. Panelists received preliminary training to familiarise themselves with the descriptive sensory attributes of frozen desserts and to ensure consistent evaluation, in accordance with ISO 6658:2017. A 9-point hedonic scale was used to assess consumer acceptability, where 1 = dislike extremely, 2 = dislike very much, 3 = dislike moderately, 4 = dislike slightly, 5 = neither like nor dislike, 6 = like slightly, 7 = like moderately, 8 = like very much, 9 = like extremely. Each assessor evaluated the samples individually and rated the following sensory quality indicators: appearance, colour, aroma, flavour, texture, melting behaviour, and overall acceptability. Samples were coded and served in a randomised order under controlled laboratory conditions.

2.9. Microbiological Analysis

Microbiological analyses of the dairy-based frozen dessert prototype fortified with grape skin powder (GSP) were performed in accordance with internationally recognized ISO standard methods and relevant European legislation [15, 16]. Samples were collected aseptically at defined storage intervals (0 and 28 days) and analyzed in triplicate. Prior to analysis, frozen samples stored at -27 ± 1 °C were thawed under refrigerated conditions (4 ± 1 °C).

The total viable count (TVC) was determined according to ISO 4833-1 [17], using plate count agar and incubation at 30 °C for 72 h, with results expressed as CFU/g. Yeasts and moulds were enumerated following ISO 21527-1 [18], using acidified yeast extract glucose chloramphenicol (YGC) agar, with incubation at 25 °C for 3–5 days.

The presence of *Listeria monocytogenes* was investigated according to ISO 11290-1 [19], and *Salmonella spp.* detection was carried out following ISO 6579-1 [20]. Results for pathogens were reported as presence or absence in 25 g of product.

Identification of microorganisms was supported by Gram staining, following the procedure described by Vijayakumar et al. [21].

2.10. Statistical Analysis

Statistical analysis was carried out using XLStat software (version 7.5.2, Addinsoft, Paris, France) integrated into Microsoft Excel. Differences between samples were assessed through one-way ANOVA, and mean separation was performed using Tukey's HSD post hoc test at a significance level of $p < 0.05$.

3. Results and Discussion

Grape skin powder obtained after convective drying preserved a high level of bioactive compounds, confirming its potential use as a functional food ingredient. The initial moisture content of fresh grape skins was $24.36 \pm 0.15\%$, which decreased to $6.80 \pm 0.12\%$ following standard drying procedures [22]. The dried grape skin powder exhibited an antioxidant activity of 87.9% and a TPC of 274.0 mg GAE/g, values consistent with those reported for polyphenol-rich grape by-products (Table 2).

Comparable values for TPC and antioxidant activity have been reported for grape skin and grape pomace derived from different *Vitis vinifera* varieties. Ky and Teissedre reported total polyphenol contents in grape skin extracts ranging from approximately 110 to 225 mg GAE/g DW, depending on grape variety and extraction solvent, with higher values obtained using hydroalcoholic extraction systems [23]. Similar trends were observed by Mostafa et al., who reported total phenolic contents of approximately 41.6 mg GAE/g DW and antioxidant activity of 10.9% for dried red grape skin powder obtained by convective drying, highlighting the strong influence of processing conditions and extraction efficiency on the measured bioactive levels [24].

Particularly relevant to the present study, Brezoiu et al. investigated polyphenolic extracts obtained from grape pomace and grape skin powder derived from *Feteasca Neagră* grapes, reporting high polyphenol concentrations and pronounced antioxidant potential in ethanolic extracts. In their study, total polyphenol contents reached up to 279.64 ± 4.52 mg GAE/g extract for grape pomace and approximately 212.21 ± 0.85 mg GAE/g extract for grape skin powder, accompanied by high DPPH radical scavenging capacity expressed as 105–137 mg TE/g vacuum dried extract [25].

In contrast, in the present study antioxidant activity was expressed as percentage of DPPH inhibition, as this unit is commonly used for comparative evaluation within a single experimental set.

The values obtained in the present study fall within or close to these reported ranges, confirming that convective drying followed by incorporation into dairy-based frozen desserts formulations preserved the bioactive potential of *Feteasca Neagră* grape skins.

Table 2

Physicochemical Characteristics and Bioactive Properties of Grape Skin Powder

Parameter	Value
Moisture content of fresh grape skins, %	24.36 ± 0.15
Moisture content of GSP, %	6.80 ± 0.12
Antioxidant activity (DPPH inhibition, %)	87.9 ± 0.6
Total polyphenol content, mg GAE/g	274.0 ± 1.3

GSP – grape skin powder; GAE - gallic acid equivalents; DPPH - 2,2-diphenyl-1-picrylhydrazyl.

The dairy-based frozen dessert prototype developed in this study was designed as a model system to evaluate the impact of grape skin powder (GSP) incorporation on composition-related quality parameters. Accordingly, the discussion that follows focuses on physicochemical indicators directly determined in the experimental samples, including dry matter, fat content and pH, which are essential for understanding the technological behaviour of the fortified frozen dessert.

3.1. Effect of GSP Incorporation on the Physicochemical Characteristics

The addition of grape skin powder (GSP) led to several coordinated modifications in the physicochemical attributes of the dairy-based frozen dessert prototype formulations, particularly in terms of dry matter, lipid content and acidity (Table 3). These parameters are closely interrelated and reflect both the composition of the ingredients and the functional behavior of grape-derived bioactive compounds in dairy matrices.

Table 3

Physicochemical Indicators of the Dairy-Based Frozen Dessert Prototypes Fortified with Grape Skin Powder

Examples	Dry matter, %	Fat content, %	pH
Control sample	55.38±0.35 ^a	26.25±0.01 ^a	6.72±0.12 ^b
2.5% GSP	55.23±0.16 ^a	26.61±0.09 ^a	6.66±0.06 ^b
5.0% GSP	55.09±0.09 ^a	26.83±0.03 ^{ab}	6.32±0.11 ^{ab}
7.5% GSP	54.94±0.18 ^a	27.02±0.06 ^{ab}	6.02±0.09 ^{ab}
10.0% GSP	54.80±0.11 ^a	27.66±0.08 ^b	5.62±0.01 ^a

GSP – grape skin powder. Values are expressed as mean ± standard deviation (n = 3). Values in the same column followed by different superscript letters (^{a-b}) are significantly different ($p < 0.05$), as determined by one-way ANOVA followed by Tukey's post hoc test.

Across all dairy-based frozen dessert prototype samples, the DM content exceeded 50%, values substantially higher than those typically reported for milk-based ice creams (28–39%) [26]. This is consistent with the high-fat raw material used (35% sweet cream), which naturally increases total solids. A slight gradual decrease in DM (55.38% - 54.80%) was observed with increasing GSP levels, likely due to the replacement of sucrose with fibre-rich plant material. The structural polysaccharides present in grape skins—known for their water-binding capacity—may influence the distribution of free vs. bound water, thereby subtly affecting total solids.

Fat content followed a similar pattern of moderate variation. Although GSP itself contains negligible lipid fractions, its incorporation modifies the solids balance and proportionally increases the relative contribution of cream fat in the mixture (26.25% - 27.66%).

The pH values decreased progressively with increasing amounts of GSP (from 6.72 in the control sample to 5.62 at 10% GSP), reflecting the intrinsic acidity of grape skins, which are rich in organic acids such as tartaric and malic acids. This reduction in pH was directly related to the level of GSP incorporation and contributed to perceptible changes in taste. At lower addition levels (2.5–5.0%), the moderate decrease in pH enhanced freshness and imparted a slightly acidic taste that was well accepted by panelists. In contrast, higher GSP concentrations (7.5–10.0%) intensified acidic and phenolic sensations, leading to more pronounced astringent notes that were reflected in slightly lower taste scores.

Overall, the results presented in Table 3 indicate that GSP can be incorporated into dairy-based frozen dessert prototype formulations without compromising physicochemical stability. Instead, its presence induces predictable and technologically manageable shifts related to the fibre composition and organic acids intrinsic to grape by-products. These observations are consistent with other studies incorporating plant-based powders into frozen dairy systems, supporting the feasibility of using grape skin powder as a functional fortifying ingredient.

3.2. Influence of GSP Addition on the Acidity Index and Peroxide Value

Given the high fat content of the sweet cream used (35%), the acidity index (AI) and peroxide value (PV) were monitored throughout frozen storage as indicators of hydrolytic and oxidative lipid degradation. Analyses were performed at 0, 7, 14, and 28 days of storage, and the results are summarized in Table 4.

Table 4

Evolution of Acidity Index and Peroxid Value Dairy-Based Frozen Dessert Prototype During Storage

Sample	Storage period, days				
	0	7	14	21	28
AI, mg KOH/g fat					
Control sample	0.10 ± 0.04 ^a	0.16 ± 0.08 ^a	0.17 ± 0.10 ^a	0.20 ± 0.09 ^a	0.21 ± 0.02 ^a
2.5% GSP	0.10 ± 0.02 ^a	0.14 ± 0.04 ^{ab}	0.16 ± 0.06 ^{ab}	0.17 ± 0.03 ^b	0.19 ± 0.03 ^b
5.0% GSP	0.10 ± 0.04 ^a	0.13 ± 0.06 ^b	0.13 ± 0.04 ^b	0.14 ± 0.01 ^c	0.16 ± 0.05 ^c
7.5% GSP	0.10 ± 0.03 ^a	0.11 ± 0.05 ^b	0.11 ± 0.06 ^c	0.13 ± 0.04 ^c	0.13 ± 0.03 ^c
10.0% GSP	0.10 ± 0.06 ^a	0.10 ± 0.02 ^b	0.10 ± 0.03 ^c	0.11 ± 0.06 ^c	0.12 ± 0.04 ^c
PV, meq O ₂ /kg fat					
Control sample	0.80 ± 0.02 ^a	1.30 ± 0.09 ^a	1.40 ± 0.06 ^a	1.60 ± 0.11 ^a	1.70 ± 0.07 ^a
2.5% GSP	0.80 ± 0.06 ^a	1.20 ± 0.08 ^b	1.25 ± 0.05 ^b	1.34 ± 0.07 ^b	1.49 ± 0.08 ^b
5.0% GSP	0.80 ± 0.03 ^a	1.20 ± 0.05 ^b	1.22 ± 0.03 ^b	1.30 ± 0.03 ^b	1.46 ± 0.02 ^b
7.5% GSP	0.80 ± 0.02 ^a	1.18 ± 0.04 ^b	1.39 ± 0.08 ^{ab}	1.40 ± 0.06 ^{ab}	1.41 ± 0.04 ^c
10.0% GSP	0.80 ± 0.03 ^a	1.11 ± 0.07 ^c	1.32 ± 0.10 ^b	1.36 ± 0.08 ^b	1.36 ± 0.09 ^c

Values are expressed as mean ± standard deviation (n = 3). Values in the same column followed by different superscript letters (^{a-c}) are significantly different ($p < 0.05$), as determined by one-way ANOVA followed by Tukey's post hoc test. GSP – grape skin powder; AI – acidity index; PV – peroxide value.

During frozen storage, both acidity index (AI) and peroxide value (PV) exhibited a time-dependent evolution; however, the magnitude and pattern of change differed among samples (Table 4). In the control sample, a continuous increase in AI (from 0.10 to 0.21 mg KOH/g fat) and PV (from 0.80 to 1.70 meq O₂/kg fat) was observed throughout the entire storage period, indicating progressive hydrolytic and oxidative lipid degradation.

In contrast, samples fortified with grape skin powder (GSP) showed a slower increase and, in some cases, a tendency toward stabilization after 7–14 days of storage. At higher GSP addition levels (7.5–10.0%), both AI and PV values exhibited only minor variations during prolonged storage and remained consistently lower than those of the control sample. This behaviour suggests that the incorporation of GSP mitigated lipid oxidation processes, which can be attributed to the presence of grape skin polyphenols with radical-scavenging and metal-chelating activity, as reported in the literature [27]. These results indicate that although AI and PV tended to increase during storage, the rate and extent of these changes were strongly dependent on GSP concentration, with higher incorporation levels contributing to improved oxidative stability of the dairy-based frozen dessert.

3.3. Influence of GSP Addition on Colour Parameters

The colour parameters of the dairy-based frozen dessert prototype samples fortified with GSP are presented in **Table 5**. The data clearly show that the addition of GSP had a strong and concentration-dependent effect on the chromatic attributes of the formulations.

Table 5

CIELab Colour Parameters of Dairy-Based Frozen Dessert Prototype Fortified with GSP

Sample	CIELab colour parameters			
	L*	a*	b*	ΔE^*
Control	79.59 ± 0.42 ^a	-0.45 ± 0.03 ^d	23.29 ± 0.28 ^a	-
2.5% GSP	49.51 ± 0.36 ^b	4.54 ± 0.05 ^b	1.68 ± 0.09 ^b	37.37 ± 0.51 ^d
5.0% GSP	34.04 ± 0.27 ^c	5.79 ± 0.07 ^a	-0.52 ± 0.06 ^c	51.77 ± 0.62 ^c
7.5% GSP	31.56 ± 0.22 ^d	4.39 ± 0.04 ^b	-0.42 ± 0.05 ^c	53.78 ± 0.58 ^b
10.0% GSP	31.50 ± 0.25 ^d	2.56 ± 0.06 ^c	-3.13 ± 0.08 ^d	54.94 ± 0.67 ^a

GSP - grape skin powder; L* - lightness (0 = black, 100 = white); a* - colour coordinate ranging from green (-) to red (+); b* - colour coordinate ranging from blue (-) to yellow (+); ΔE^* - total colour difference compared to the control sample. Values are expressed as mean ± standard deviation (n = 3). Values in the same column followed by different superscript letters (a–d) are significantly different ($p < 0.05$), as determined by one-way ANOVA followed by Tukey's post hoc test.

As observed, increasing the level of GSP resulted in a marked reduction of lightness (L*), with values dropping from 79.59 in the control sample to 31.50 in the 10% GSP frozen dessert. This effect is directly related to the high anthocyanin content of grape skins, which impart intense red–purple coloration to dairy matrices. Similar decreases in L values accompanied by shifts toward red/violet tones have been consistently reported in frozen dairy products fortified with grape skin or grape pomace derivatives rich in anthocyanins. Salem et al. (2014) demonstrated that the addition of red grape pomace extract to ice cream significantly reduced lightness and yellowness while increasing redness (a*), confirming the strong colouring capacity of grape-derived anthocyanins [28]. Comparable colour modifications were also reported by Nascimento et al., who observed statistically significant changes in CIELAB parameters following the incorporation of grape agro-industrial waste flour into ice cream formulations, without adversely affecting visual acceptability [29].

Further support for the anthocyanin-driven colour changes was provided by Rațu et al., who reported pronounced reductions in lightness and increased chromatic intensity in dairy-based frozen products enriched with grape pomace or grape skin derivatives [30]. In addition, studies on fermented dairy matrices, such as yogurt fortified with grape skin powder, have shown similar decreases in L values and increases in redness attributed to pH-

dependent anthocyanin stability and protein–polyphenol interactions [31]. The absence of a linear trend for a and b values in the present study can be explained by the pH sensitivity of anthocyanins and their interactions with milk proteins and water activity, which may affect pigment expression depending on the level of GSP incorporation.

By comparison, the grape skin powder used in the present study generated an equally strong and dose-dependent chromatic effect, with ΔE^* values exceeding 37–54 units, indicating visually perceptible and substantial differences from the control, comparable in magnitude to colour shifts reported in functional ice creams enriched with black carrot anthocyanins, grape marc or pomegranate peel. Taken together, these findings reinforce that grape skin powder operates effectively as a natural colourant, capable of imparting deep red–purple tones to both dairy and non-dairy frozen desserts.

3.4. Influence of Added GSP on the TPC and AA of Frozen Dessert Samples

The plant-based foods in our diet consist of primary and secondary ingredients produced by plants. Plants synthesize secondary metabolites such as terpenes, saponins, glycosides and polyphenolic compounds that are not present in all living things, as well as primary metabolites such as carbohydrates, proteins and fats to maintain their growth and vitality [32]. Most secondary metabolites are formed from polyphenols. Plants produce polyphenols as secondary metabolites to protect themselves and interact with other plant [33]. Although milk-based products are not considered major sources of polyphenols, previous studies have demonstrated the presence of measurable phenolic compounds in milk and dairy matrices, originating from animal feed, Maillard reaction products, and interactions with proteins [34]. Therefore, the detection of low baseline TPC values in control sample is consistent with literature data and does not invalidate the comparative assessment of GSP-fortified formulations [35,36]. The polyphenols in the studied samples were determined by applying the Folin Ciocalteu method, and based on the calibration curve for gallic acid. Thus, as a result, the data shown in Figure 1 were obtained.

Figure 1 shows a directly proportional relationship between the amount of added GSP and the TPC of the processed samples.

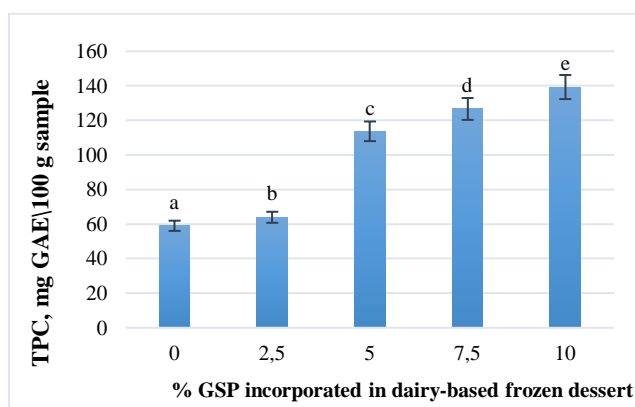


Figure 1. Total polyphenol content of dairy-based frozen dessert prototypes, mg GAE/100 g sample.

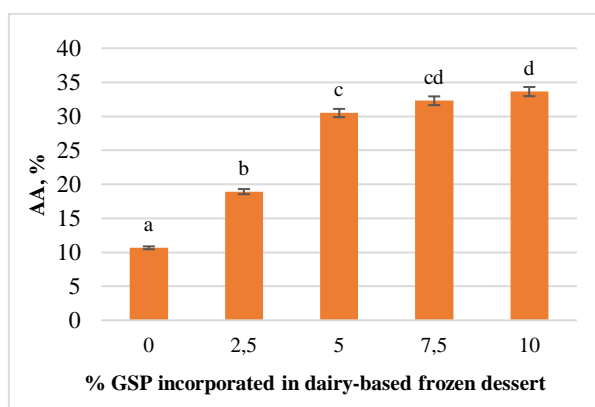


Figure 2. Antioxidant activity (% DPPH inhibition) of dairy-based frozen dessert prototypes.

Different letters above the bars indicate statistically significant differences ($p < 0.05$), as determined by one-way ANOVA followed by Tukey's post hoc test.

Thus, the highest polyphenol content was found in the samples with 7.5 and 10.0% GSP (126.61 and 139.29 mg GAE/100 g respectively). In the same context, based on the results

obtained from the data in Figure 2, we can say that, as a rule, the desserts with a higher amount of addition showed higher AA, % values than the other samples. The antioxidant activity of GSP dairy-based frozen dessert prototypes ranged from 18.92 to 33.63%, showing a directly proportional relationship with total phenolic content (TPC, mg GAE/100 g).

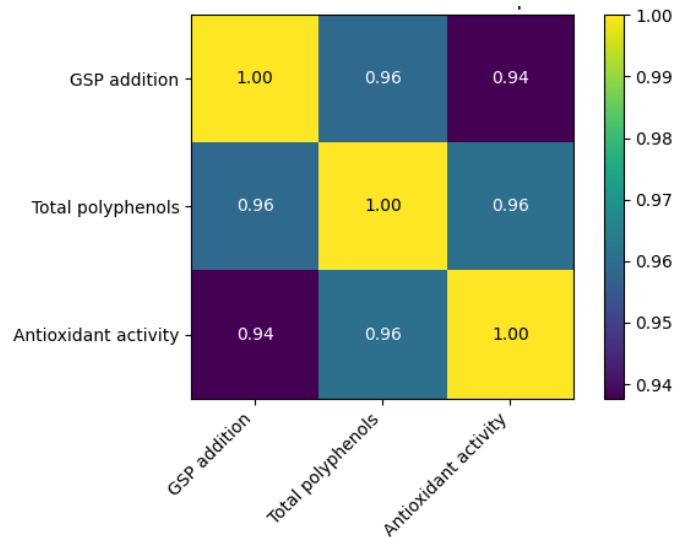


Figure 3. Pearson correlation heatmap illustrating the relationships between grape skin powder (GSP) addition level, total polyphenol content and antioxidant activity.

A very strong positive correlation was observed between total polyphenol content and antioxidant activity ($r = 0.96$), indicating that the increase in antioxidant capacity is closely associated with the higher polyphenol levels contributed by grape skin powder. In addition, GSP addition showed a strong positive correlation with both total polyphenol content ($r = 0.96$) and antioxidant activity ($r = 0.94$), confirming the dose-dependent functional contribution of grape skin powder (Figure 3). Comparable relationships have been reported for grape skin extracts, where Katalinić et al. observed a very strong linear association between total phenolic content and antioxidant capacity assessed by FRAP ($R^2 = 0.9456$, corresponding to $r \approx 0.97$), confirming that polyphenols are the principal contributors to the antioxidant potential of grape skin-derived materials [37].

3.5. Microbiological Quality of the Dairy-Based Frozen Dessert Prototype Fortified with Grape Skin Powder

The GSP incorporated into the dairy-based frozen dessert prototype was obtained through mild convective drying and grinding, without the application of sterilization treatments. As a result, this ingredient may act as a carrier of indigenous microflora typically associated with plant-derived raw materials, including bacteria, yeasts and moulds. Such microflora can survive processing and become detectable when favorable conditions, such as moisture availability during thawing and refrigerated storage, are present. Mould contamination is of particular relevance in plant-based ingredients due to the potential presence of mycotoxigenic species; therefore, monitoring microbial dynamics in fortified products represents an important aspect of product quality evaluation [38].

The microbiological quality of the dairy-based frozen dessert prototype fortified with different levels of GSP was evaluated during storage by determining total viable count, yeasts and moulds, Enterobacteriaceae, and the presence of selected pathogens, as presented in Table 6.

Microbiological analyses were performed after controlled thawing and refrigerated storage, as frozen storage at -27 ± 1 °C is not expected to support microbial growth but only microbial survival. On day 0, all formulations exhibited low total viable counts, ranging between approximately 1.2×10^2 and 2.3×10^2 CFU/g, indicating good initial hygienic quality of both the base formulation and the added grape skin powder. Yeasts and moulds were either below the detection limit or detected at very low levels, while *Listeria monocytogenes* and *Salmonella spp.* were absent in 25 g of product in all formulations.

Table 6

Qualitative Screening of Microbial Presence in the Dairy-Based Frozen Dessert Prototype Fortified with Grape Skin Powder During Refrigerated Storage after Thawing

Sample	Storage, days	Total viable count, CFU/g	Yeasts & moulds, CFU/g	<i>Listeria monocytogenes</i> (25 g)	<i>Salmonella spp.</i> (25 g)
Control	0	1.2×10^2	$<1.0 \times 10^1$	Absent	Absent
Control	28	4.5×10^3	2.1×10^2	Absent	Absent
2.5% GSP	0	1.4×10^2	$<1.0 \times 10^1$	Absent	Absent
2.5% GSP	28	3.2×10^3	1.6×10^2	Absent	Absent
5.0% GSP	0	1.6×10^2	$<1.0 \times 10^1$	Absent	Absent
5.0% GSP	28	2.8×10^3	1.3×10^2	Absent	Absent
7.5% GSP	0	1.9×10^2	1.1×10^1	Absent	Absent
7.5% GSP	28	5.8×10^3	3.6×10^2	Absent	Absent
10.0% GSP	0	2.3×10^2	1.5×10^1	Absent	Absent
10.0% GSP	28	7.4×10^3	4.8×10^2	Absent	Absent

During refrigerated storage after thawing, a gradual increase in total viable count and fungal populations was observed in all samples (Table 6). After 28 days, total viable counts increased to values ranging from approximately 2.8×10^3 CFU/g in the 5.0% GSP formulation to 7.4×10^3 CFU/g in the 10.0% GSP formulation. A similar trend was observed for yeasts and moulds, with counts remaining below 5.0×10^2 CFU/g even at the highest GSP addition level. Pathogenic microorganisms were not detected at any storage point. Formulations containing 2.5% and 5.0% GSP showed slightly lower microbial counts compared to the control during storage, particularly in terms of total viable count and yeasts and moulds. This behavior may be associated with the presence of grape skin polyphenols, which are known to exert antimicrobial effects against a broad range of microorganisms. In contrast, formulations containing 7.5% and 10.0% GSP exhibited higher microbial counts during prolonged storage, which may be attributed to the increased introduction of indigenous microflora and nutrients associated with higher amounts of grape skin powder, partially counterbalancing its antimicrobial potential.

3.6. Sensory Analysis

Sensory evaluation is one of the methods used to identify market acceptability especially in food or beverage products. It is useful for product development and improvement as the most important factor for a given market can be identified and improved. Influencing factors are essential for consumers to get the best product and for producers to develop and sell the best product [39].

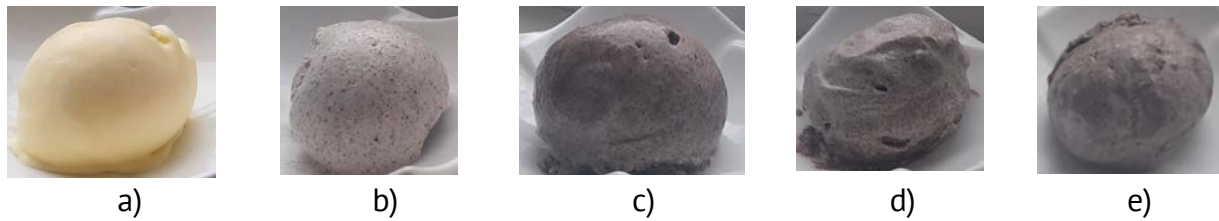


Figure 4. Visual appearance of dairy-based frozen dessert prototype samples fortified with different levels of grape skin powder (GSP): a) control; b) 2.5% GSP; c) 5.0% GSP; d) 7.5% GSP; e) 10.0% GSP.

Sensory attributes (Figure 5) in terms of colour, aroma, taste a texture and overall acceptability for dairy-based frozen dessert prototypes were determined after a period of 2 weeks after preparation. Sensory evaluation performed using the 9-point hedonic scale revealed that all samples, including those fortified with grape skin powder (GSP), obtained mean scores above 6.3, corresponding to the category “like slightly”. The distributions shown in the boxplots demonstrate that panellists generally evaluated the products favourably, with only a few lower ratings, all of which corresponded to the neutral point of the scale (5 = neither like nor dislike). This indicates that even for samples perceived as less optimal, the response was neutral rather than negative, suggesting room for further technological refinement but no sensory rejection.

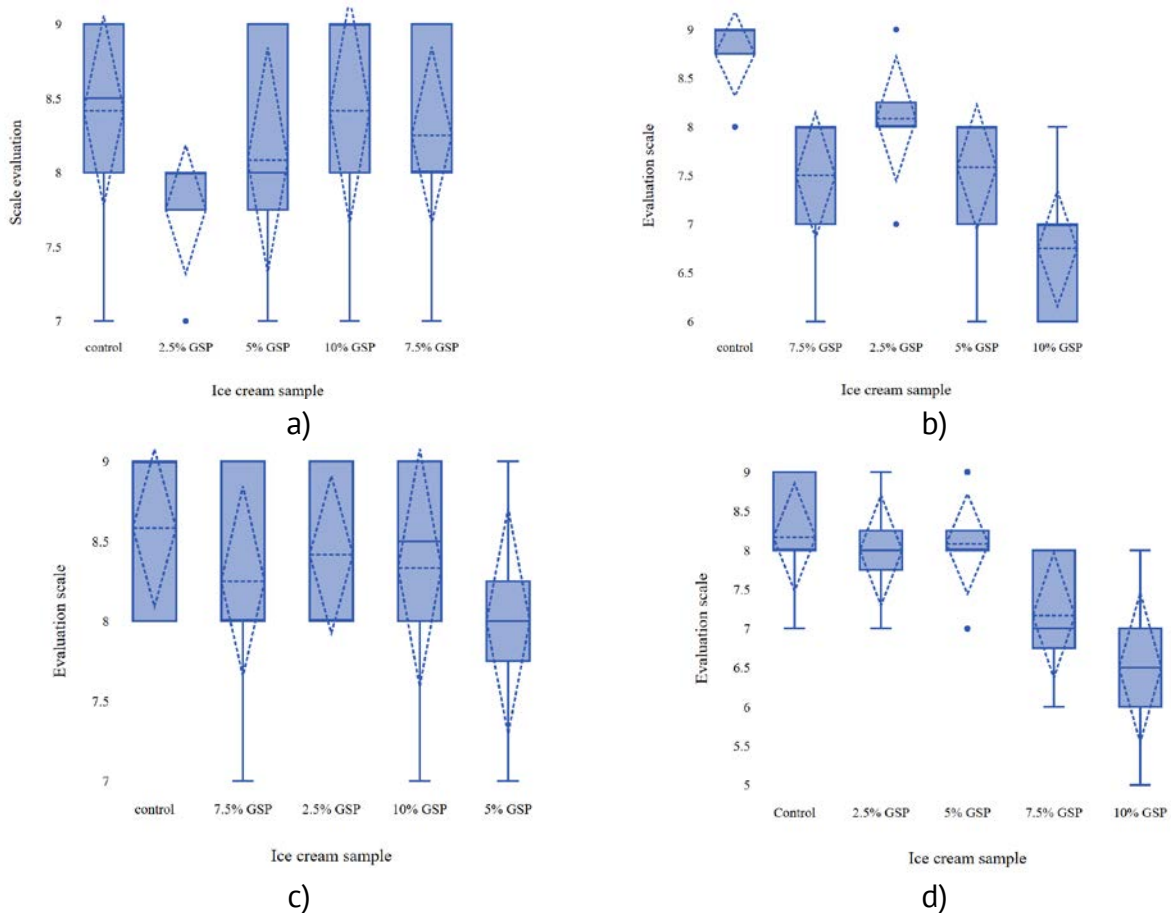


Figure 5. Sensory profile of dairy-based frozen dessert prototype samples: a) colour evaluation; b) texture evaluation; c) aroma evaluation; d) taste evaluation.

Across attributes, colour showed a clear upward trend in acceptability with increasing GSP concentration, reflecting the positive perception of the naturally intensified pigmentation provided by grape skin compounds. In contrast, texture exhibited the opposite tendency, with slightly lower and more dispersed scores at higher GSP levels, likely due to the perceptibility of fine grape skin particles. Still, even the 10% GSP sample maintained mean values around 6.3–6.5, confirming that texture remained within an acceptable range.

Aroma was consistently well appreciated across all samples, with compact distributions and high medians, indicating that the volatile compounds from grape skin integrated harmoniously into the frozen dessert matrix. Taste followed a similar pattern, with a slight decrease at higher GSP concentrations due to mild astringent or phenolic notes, yet maintaining mean values above 6 for all formulations.

Among all formulations, the 5% GSP sample clearly stood out as the most successful. It achieved the highest balance across attributes, with mean scores of 8 for colour, 7.6 for texture, 8 for aroma, and 8 for taste, corresponding to the categories “like very much” (score 8) and “like moderately” (score 7). These values highlight an optimal integration of GSP into the dairy-based frozen dessert prototype matrix, enhancing flavour and aroma while maintaining a pleasant texture and an appealing colour. Taken together, the results confirm that a 5% addition level offers the best compromise between functional enhancement and sensory acceptance.

4. Conclusions

This study demonstrates that grape skin powder, obtained from winemaking by-products, can be effectively incorporated into a dairy-based frozen dessert prototype, contributing to functional enrichment and improved oxidative stability. Increasing GSP levels induced predictable changes in product composition, including a moderate decrease in pH (6.72–5.62) and a slight increase in fat content (26.25–27.66%), without compromising overall physicochemical stability.

The addition of GSP significantly enhanced the functional profile of the product, as evidenced by higher total polyphenol content (up to 139 mg GAE/100 g) and increased antioxidant activity (up to 33.6%). These improvements were accompanied by enhanced resistance to lipid oxidation during frozen storage, with lower peroxide values compared to the control sample. Colour analysis confirmed a strong and dose-dependent chromatic effect ($\Delta E^* > 37$), highlighting the effectiveness of grape skin anthocyanins as natural colourants for frozen dairy systems.

Microbiological results indicated that moderate GSP incorporation levels (2.5–5.0%) were associated with lower microbial counts during storage, whereas higher levels (7.5–10.0%) introduced additional indigenous microflora from the plant material. Sensory evaluation revealed good consumer acceptance across all formulations, with the 5% GSP sample achieving the most favourable balance between colour, flavour, texture and overall acceptability.

Acknowledgments: The research was funded by State Project subprogram 020405 “Optimization of food processing technologies in the context of the circular bioeconomy and climate change”, running at Technical University of Moldova.

Conflicts of Interest: The authors declare no conflict of interest.

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Citation: Ruseva, O.; Covaliov, E.; Suhodol, N.; Deseatnicova, O.; Resitca, V. Effect of grape skin incorporation on the quality of a dairy-based frozen dessert. *Journal of Engineering Science.* 2025, XXXII (4), pp. 104–119. [https://doi.org/10.52326/jes.utm.2025.32\(4\).08](https://doi.org/10.52326/jes.utm.2025.32(4).08).

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