# ICE CREAM - FOOD MATRIX FOR FUNCTIONAL PRODUCT WITH GRAPE SKIN ADDITION 

## ÎNGHEȚATA - MATRICE ALIMENTARĂ PENTRU PRODUSE FUNCȚIONALE CU ADAOS DE PIELIȚĂ DE STRUGURI

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Rezumat: Strugurii recoltați la nivel mondial sunt utilizați în vinificație, iar odată cu dezvoltatea acestei industrii crește și volumul de subproduse, cum ar fi tescovină de struguri, semințe, etc. Aceste produse sunt bogate în polifenoli, taninuri, ș.a. și prezintă interes pentru exploatarea lor ulterioară. Scopul lucrării date este cercetarea posibilității utilizării pieliței de struguri în producerea înghețatei.

În rezultatul cercetărilor s-a stabilit că adaosul de pulbere din pielița strugurilor reduce viteza proceselor oxidative în inghețată, contribuind semnificativ la creșterea conținutului total de polifenoli (63,93-139,29 mg AG/mL extract) și activității antioxidante (33,63\% pentru proba 10,00 \% PS). Fortificarea inghețatei cu pulbere din pielița strugurilor influențează semnificativ culoarea inghețatei elaborate, diferența de culoare variind în limitele $\Delta E=37,37-54,94$.

Cuvinte cheie: pieliță de struguri, inghețată, polifenoli, activitate antioxidantă


#### Abstract

Grapes harvested worldwide are used in winemaking, and with the development of this industry, the volume of by-products, such as grape pomace, seeds, etc., is increasing. These products are rich in polyphenols, tannins, etc. and are of interest for their subsequent exploitation. The purpose of this paper is to investigate the possibility of using grape skin in the production of ice cream.

Research has shown that the addition of grape skin powder reduces the rate of oxidative processes in ice cream, significantly contributing to the increase in total polyphenols (63.93-139.29 mg AG/mL extract) and antioxidant activity ( $33.63 \%$ for test $10.00 \%$ PS). The fortification of the ice cream with grape skin powder significantly influences the color of the elaborate ice cream, the color difference varying in the limits $\Delta E=37.37$ - 54.94.


Keywords: grape skin, ice cream, polyphenols, antioxidant activity

## Introduction

Globally, food waste has become an increasingly recognized environmental issue in the last decade. The problem of food waste has become not only an ethical issue in a world where about 800 million people are starving, but the impact of food production that is then dumped on the environment can no longer be overlooked. As population and urbanization grow, more food is produced and more food is wasted. In addition, wasting food in an urban context has serious consequences for the environment and public health, which have a negative impact on human well-being and the environment.Food waste management should
follow certain policies based on the 3R concept, ie reduction, reuse and recycling [1]. Nowadays, reducing food waste is considered to be the highest priority of the economic, industrial and social sectors. Sometimes it is not economically or technologically possible. In this regard, food waste should be used as much as possible, as it can be used as a resource for many other processes, such as feed production or energy.

Most of the grapes harvested worldwide are used to make wine, with winemaking being one of the most important industries in many countries. Along with the growing global trend for wine consumption, there is a growing volume of accompanied by by-products, such as grape marc, seeds, hides, skins, stems, leaves and yeast. These by-products are an interesting source for further exploitation, as they are rich in polyphenols, tannins, procyanidins, anthocyanins, stilbenes, dietary fiber, tartrates, essential oils, proteins, tocopherols, etc. In this context, they have the potential to be used as a source of ingredients that could be used in various applications in functional foods, food supplements, pharmaceuticals and cosmetics, as well as for agricultural and oenological purposes.

In addition, grape marc, husks and seeds are the most promising wine by-products to become oenological additives to improve the chemical composition and sensory attributes of wines. The red color (anthocyanins and polymeric pigments) and the improvement of phenolic compounds appear as the simplest application with recognized efficacy.

Grape pomace consists mainly of skin, seeds and stems and represents about 20-25\% of the weight of crushed grapes for wine production. Grape seeds are rich in extractable phenolic antioxidants such as phenolic acid, flavonoids, procyanidins and resveratrol, while grape skins contain abundant anthocyanins. The health benefits of tescovine polyphenols have been of great interest to researchers, the food industry and the nutraceutical industry. In addition to phenolic antioxidants, tescovina also contains significant amounts of lipids, proteins, indigestible fiber and minerals. Grape seeds contain $13-19 \%$ oil, which is rich in essential fatty acids, about $11 \%$ protein, $60-70 \%$ indigestible carbohydrates and non-phenolic antioxidants such as tocopherols and beta-carotene [2].

Numerous studies have shown that polyphenols in grape seed have many health benefits, such as anti-mutagenic and anti-cancer activity, antioxidant and anti-inflammatory activity [3], prevention and delay of cardiovascular disease, increased lifespan and delayed appearance of markers. related to age.

Ice cream is rich in macronutrients, ie carbohydrates, fats, and proteins and some micronutrients, ie vitamins A, D, E and calcium mineral. However, commercially available ice cream is generally low in natural antioxidants such as vitamin C, pigments and phenols [4]. As for the Republic of Moldova, until the 1990s ice cream was made exclusively from natural products: milk, cream, butter and sugar. In addition, the factories used fruit, chocolate, vanilla and nuts. Today, our food industry has become like a chemical laboratory and almost all natural additives have been replaced with synthetic ones. Milk remained the basic ingredient, only now it is soy. The real milk fat, with its unique set of minerals, has been replaced with hydrolyzed vegetable fat - palm oil, coconut oil, not to mention all the stabilizers and emulsifiers present in a glass of ice cream.

These days there is a strong drive for health and there is a clear trend for consumers to buy healthier products. To meet this need, it is possible to produce a fatty product with a significantly reduced amount of saturated fatty acids without changing the quality of the ice cream [5]. For example, ice cream is now enriched with a number of components to improve its technological and functional state, for example, kiwi, grape juice concentrate and cane molasses, grape seed extract [6] and omega-3 fatty acids [7].

In the process of improving the dietary and functional properties of ice cream, various phytochemicals, vitamins, minerals, bioactive peptides, dietary fiber, probiotics, prebiotics, whey, various fatty acids and vegetables, spices are widely used. In addition, various components, such as fruits, wild fruits, vegetables, herbs, bee products (such as honey, pollen and propolis) and various sugar substitutes (vegetable sweeteners such as sugar alcohols and stevia) may be, also used with these components.

At the moment, there is a lack of publication on the enrichment of ice cream using grape by-products, which are in fact the result of their processing in order to obtain wine, juice, etc ..

Therefore, the objectives of this study were: (1) enrichment of ice cream with grape skin powder; (2)

- to determine the functional properties of fortified ice creams;
- and to determine the sensory properties of enriched ice creams.


## Research Methodology

## Materials

Grape pomace as a by-product of winemaking was supplied from a local producer. The skin was
dried separately at room temperature. The dried products were stored in plastic bags at $4{ }^{\circ} \mathrm{C}$ in a lightprotected environment. Grape skin in powder form was obtained by grinding and sifting.

The chemicals and solvents used in spectrophotometric and titrimetric analysis were of analytical grade and were purchased from Sigma Aldrich.

The ingredients for the preparation of ice cream, sucrose, cream, skim milk, skimmed milk powder, were food grade and were obtained from local suppliers.

## Methods

## Samples preparation

The ice cream samples were enriched with grape skin powder (GS). Five types of ice cream were prepared with the following combinations: Control (F1), enriched at $2.5 \%$ with GS, enriched at $5.0 \%$ with GS, enriched at $7.5 \%$ with GS and enriched at $10.0 \%$ with GS. The fortification of the ice cream was made at the expense of reducing the amount of sugar in samples 2.5 and $5.0 \% \mathrm{GS}$ and including and the amount of cream for samples 7.5 and $10.0 \%$ GS. In order to diversify the assortment, an attempt was made to make a sorbet, which in fact would be an alternative to ice cream for those who have adopted a vegan diet. In the case of sorbet, almond milk was used as a basic raw material (it was also enriched with grape skin powder (5\%)).

Initially, the ice cream vat was placed in the cold $\left(-18^{\circ} \mathrm{C}\right)$ for at least 24 hours. The raw materials were also kept in cold. Table 1 shows the raw materials and their proportions used in the manufacture of ice cream.

Table 1: Raw materials and proportions used in the manufacture of ice cream

| Sample | Cream, $\mathbf{g}$ | Almond milk, ml | Sugar, $\mathbf{g}$ | GS, g |
| :---: | :---: | :---: | :---: | :---: |
| Control | 300 | - | 100 |  |
| $2,5 \% \mathrm{GS}$ | 300 | - | 90 | 10 |
| $5,0 \% \mathrm{GS}$ | 300 | - | 80 | 20 |
| $7,5 \% \mathrm{GS}$ | 290 | - | 80 | 30 |
| $10,0 \% \mathrm{GS}$ | 280 | 400 | 80 | 40 |
| Sorbet | - |  | 80 | 20 |

The liquid phase represented by sweet cream (35\% fat, manufacturer Incomlac) and sugar ( $25 \%$ of the total mass) was poured into the ice cream machine tub. After a light mixing, the powder from the grape skin was added and the homogenization was started in the electric machine for 40-50 minutes, until the consistency of the soft ice cream was obtained.

## Methods for determining the ice cream physicochemical properties Dry matter

The dry matter content was determined by drying the samples in an oven (POL-EKO-APARATURA, Wodzislaw Śląski, Poland) at $105^{\circ} \mathrm{C}$ until a constant mass was established.

## pH

The pH of the ice cream mixture was determined using the pH meter Lab pH Meter inoLab pH 7110 SET 4.

## Color evaluation

Color parameters $L *, a *$, and $b *$ were determined using a chromometer (Minolta CR400, Osaka, Japan), $L *$ defines the brightness, $a *$ and $b *$ define red-green and blue-yellow, respectively. For the ice cream samples, the color difference $\Delta E$ was also determined.
Color differences can be calculated as the relative distance between two reference points in the CIELAB color space. This difference is usually expressed as delta $E(\Delta E)$ and is calculated by comparing the reference and sample $L^{*} a^{*} b^{*}$ values to indicate how far two colors are in a color space.

$$
\Delta E=\sqrt{\left(L_{\text {sample }}-L_{\text {control }}\right)^{2}+\left(a_{\text {sample }}-a_{\text {control }}\right)^{2}+\left(b_{\text {sample }}-b_{\text {control }}\right)^{2}}
$$

## Total polyphenol content

The total polyphenol content was determined using the Folin - Ciocalteu reagent, using gallic acid solutions as a standard. First, the hydroalcoholic extract (50\%) was prepared in a ratio of 1:10. Subsequently, for the spectrophotometric analysis of the total polyphenol content, 0.5 ml of distilled water and 0.5 ml of Folin-Ciocalteu solution are added to 0.5 ml of extract. After shaking the containers, 8 ml of Na 2 CO 3 are added to each. The obtained mixture after stirring was left for two hours in a dark place for the complete reaction of the polyphenols with the reagent Folin Ciocalteu. After two hours, the absorbance was read on the spectrophotometer, at a wavelength $\lambda=765 \mathrm{~nm}$.

## Antioxidant activity

The DPPH test was used to predict antioxidant activity by the mechanism by which antioxidants act to inhibit lipid oxidation, thus eliminating the DPPH radical and therefore determining the ability to capture free radicals. The free radical DPPH (1,1-diphenyl-2-picrylhydrazyl) is very stable, reacting with compounds that can donate hydrogen atoms and has a maximum UV-vis absorption at 515 nm .

## Sensory analysis

Quantitative descriptive sensory analysis was performed at the Department of Food and Nutrition, Technical University of Moldova. Fifteen non-smoking evaluators (women aged 23 to 70) were selected to assess the sensory attributes of ice cream. The evaluators were trained until they understood the descriptive sensory terms of ice cream and showed consistent cues. The taste descriptors were expressed as: tasty, sweet, sour, astringent, oxidized and unnatural flavour. The tasty attribute was used to assess the overall acceptability of the ice cream. The texture descriptors were named as: hard, coarse, gummy, icy, creamy and watery. The appearance was assessed by the intensity of the ice cream color. All attributes were evaluated on a 5 -point hedonic scale, where 0 was given for extremly dislike, 2 for imperceptible, 5 for extremlu like (the strong presence of ice cream related attributes).

## Result and Discussion

## Influence of grape skin powder addition on dry matter content and pH of ice cream

To begin with, the amount of dry matter and the pH were determined in the ice cream samples. The data obtained are presented in Table 2.

From the data presented we conclude that with the increase of the amount of vegetable addition, the amount of dry matter of the products also rises. The lowest value was recorded for the sorbet sample $(29.43 \%)$, much lower value than ice cream which was prepared with the same added content. The high dry matter content of the ice cream samples is largely due to the use of sweet cream with a fat content of $35 \%$, while almond milk (used to prepare sorbet) has a fat content of only $1.5 \%$. In the literature, there are studies in which lower values of dry matter content were obtained (35.84-38.18\% [4]; 28.7-38.3\% [8] 35.85$39.35 \%$ [9], these lower values can also be argued by the fact that milk, or cream with a lower amount of fat, was used as a raw material.

The incorporation of GS in the ice cream formulations caused differences in the pH and color of the ice cream. pH values decreased as the amount of GS increased in the ice cream. This fact may be due to the slightly acidic characteristics of the added GS. The pH of ice cream plays a critical role in milk proteins. A decrease in the pH of ice cream can lead to destabilization of casein micelles and coalescence of micelles. In the present study, the pH value of the control ice cream was 6.72 , while the lowest pH value (5.62) was observed for the ice creams enriched with GS $10 \%$. There was no coalescence of casein micelles at these pH levels during ice cream preparation. In the literature, the aggregation of casein micelles was observed at pH values of 4.6 and 4.9 when kiwi fruit was added to ice cream [10](Sun-Waterhouse et al., 2013) and when the pH of milk was between 5.5 and 5.0 [11].

Table 2: Dry matter content and pH of ice cream

| Sample | Dry matter, $\%$ | $\mathbf{p H}$ |
| :---: | :---: | :---: |


| Control | $55.65 \pm 1.12$ | $6.72 \pm 0.32$ |
| :---: | :---: | :---: |
| $2,5 \% \mathrm{GS}$ | $56.32 \pm 0.98$ | $6.66 \pm 0.17$ |
| $5,0 \% \mathrm{GS}$ | $57.45 \pm 1.34$ | $6.32 \pm 0.23$ |
| $7,5 \% \mathrm{GS}$ | $57.26 \pm 1.45$ | $6.02 \pm 0.56$ |
| $10,0 \% \mathrm{GS}$ | $55.16 \pm 0.67$ | $5.62 \pm 0.53$ |
| Sorbet | $29.43 \pm 0.76$ | $6.45 \pm 0.34$ |

## The influence of the addition of GS on the color parameters of the ice cream

Color is one of the most important qualities of food, and food coloring is a major additive, as it increases the acceptance and attractiveness of a food product. Natural colors have long faced a number of criticisms: they are more expensive, less stable, and less strong than their synthetic counterparts. Although the use of natural dyes in food is an ancient practice, it is gaining in importance due to the consumer's preference for natural products. The use of natural colors is seen as a sustainable and environmentally friendly process. The Joint FAO / WHO Committee of Experts on Food Additives has recommended that more attention be paid to methods of reducing synthetic food colors, and that the food industry's attention has been shifted to the potential of natural alternatives. Although most natural colors have a higher cost in use than the corresponding synthetic colors, this has not limited their use as dose levels, which are still relatively low. Natural colors are generally extracted from fruits, vegetables, seeds, roots and microorganisms. The color of the grape skin is determined by the accumulation of red pigments in plants called anthocyanins, respectively it has all the advantages to make it an excellent natural food coloring. As mentioned above, the color parameters of the ice cream and sorbet samples were assessed by applying the CieLab system. In the images below (Table 3) are the illustrative representations of the products, with the indication of their chromatic parameters.

Table 3: CieLab color parameters of ice cream samples

|  | Control | $2,5 \%$ GS | $5,0 \%$ GS | $7,5 \%$ GS | $10,0 \%$ GS | Sorbet $5,0 \%$ GS |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |
| L | $79.59 \pm 2.14$ | $49.51 \pm 1.45$ | $34.04 \pm 0.98$ | $31.56 \pm 0.78$ | $31.5 \pm 0.87$ | $12.13 \pm 0.12$ |
| a | $-0,45 \pm 0.04$ | $4.54 \pm 0.07$ | $5.79 \pm 0.25$ | $4.39 \pm 0.17$ | $2.56 \pm 0.08$ | $4.38 \pm 0.09$ |
| b | $23.29 \pm 0.32$ | $1.68 \pm 0.02$ | $-0.52 \pm 0.01$ | $-0.42 \pm 0.01$ | $-3.13 \pm 0.13$ | $-4.53 \pm 0.23$ |
| $\Delta \mathrm{E}$ | - | $37.37 \pm$ | $51.77 \pm$ | $53.78 \pm$ | $54.94 \pm 1.34$ | - |

From the analysis of the values presented for color parameters $L, a, b$, no relationship was established between the modification of parameters $a$ and $b$ and the addition of powder from grape skin. In contrast, the $L$-brightness parameter shows a decreasing trend with increasing GS content. Thus for the control sample the value of $L$ was 79.59 , and for the ice cream with $10 \%$ addition, it reaches the value of 31.50 . Also, in the case of sorbet (with $5 \%$ addition) the brightness reaches the value of 12.13 , this being probably caused by the fact that in the formulation of sorbet the fat content is lower and this medium (aqueous) is more suitable for solubilizing colored compounds. The results obtained demonstrated the possibility of using GS as a colorant in food compositions. Also, a directly proportional relationship was established between the amount of GS and the color difference $\Delta E(54.94)$, this reaching maximum values in the case of ice cream with $10 \%$ GS.

## The influence of the addition of GS on the total polyphenol content of ice cream

Polyphenols make up most of the components found in all vegetative organs, fruits and flowers. Plants produce polyphenols as secondary metabolites to protect themselves and interact with other plants. The results obtained for the investigated ice cream samples are shown in Figure 1.

Figure 1 shows a directly proportional relationship between the amount of GS added and the TPC of the ice cream samples. Thus, the highest polyphenol content was recorded in the samples with 7.5 and $10.0 \%$ GS ( 126.610 and $139.293 \mathrm{mg} \mathrm{AG} / \mathrm{mL}$ extract respectively). Even if the sorbet was prepared with the addition of $5 \%$ GS, in its case the TPC was lower ( $100,512 \mathrm{mg}$ AG / mL extract) than in the case of ice cream with the same amount of addition ( $113,683 \mathrm{mg} \mathrm{AG} / \mathrm{mL}$ extract), probably the almond milk contain some compounds that have prevent the manifestation of the polyphenols' properties.


Figure 1. Total polyphenol content of ice cream samples, mg AG/mL

## The influence of the addition of GS on the DPPH antioxidant activity of ice cream

Oxidative stress is the process of generating free radicals, including reactive oxygen species and nitrogen (ROS and RNS). These are inevitable consequences of the by-products of aerobic metabolism, which damage the structure and function of cellular organs. Elevated concentrations of ROS and RNS in metabolism are linked to the development of diseases, including cancer, cardiovascular and neurological diseases. Elimination or prevention of the generation of these free radicals is achieved through an effective anti-oxidative defense system that contains both enzymatic and non-enzymatic antioxidants. Researchers have discovered natural sources to prevent or reduce oxidative stress, which has attracted attention over the centuries.

Foods or plant components, such as polyphenols, can play an important role in preventing the generation of free radicals and are often found in fruits, vegetables, grains and marine organisms. Recent studies in phytochemistry highlight the need to include polyphenolic compounds in the diet due to their potential antioxidant activities and a wide range of health benefits. Grapes are widely available and are a good source of phenolic compounds that are associated with antioxidant activity. In general, grape residues, such as husks, seeds and stems, are potential sources of phenolic compounds and flavonoids. In the last century, there has been a dramatic increase in the identification of individual compounds with antioxidant activity in by-products of the grape industry. They clearly show that natural sources such as grapes are important in preventing oxidative stress. For this reason, researchers have begun to investigate analytical methods to evaluate the antioxidant activity and mechanisms associated with natural compounds. For this reason, researchers have begun to investigate analytical methods to evaluate the antioxidant activity and mechanisms associated with natural compounds.


Figure 2. DPPH antioxidant activity of ice cream samples, \%
The results obtained for the investigated ice cream samples are shown in Figure 2.
Based on the results obtained from the data in Figure 2, it can be stated that, as a rule, ethanolic ice cream extracts with a higher amount of addition showed higher AA values. The antioxidant activity varied in the limits of $18.92-33.63 \%$ for the ice cream samples with the addition of GS, and constituted $28.38 \%$ for the elaborated sorbet sample ( $5 \% \mathrm{PS}$ ). The direct proportional relationship between AA (\%) and TPC ( $\mathrm{mg} / \mathrm{mL}$ extract) can also be mentioned.

## Sensory analysis

The sensory attributes (figure 3) in terms of appearance, color, aroma, taste, texture and general acceptability of ice cream were determined after a period of 2 weeks after preparation. Sensory scores for ice cream revealed a significant decrease in the appearance of the sorbet sample (Figure 3) (accumulated score 4.42 out of 5).


Figure 3. Sensory profile of ice cream samples
The structure and consistency of the grape skin ice cream decreased significantly with the increase in the amount of GS ( 4.86 points for $2.5 \%$ GS and 4.00 points for $10.0 \% \mathrm{GS}$ ) for the sample. $10.00 \%$ GS stating that the powder particles feel too much. On the other hand, the sensory scores for the color of the samples showed an increasing tendency with the increase of the GS amount. Flavor scores did not vary in the samples, the aroma being associated with the smell of coffee or rum. It is worth mentioning that for the control test it was perceived as too sweet, while for the rest of the tests it was not so accentuated.

According to the accumulated score for overall acceptability ( 23.43 out of 25 ), the most successful sample is ice cream with $5 \%$ GS, however the other samples did not accumulate a score of less than 20 points, being included into the category "The product has a specific positive characteristic , quite contoured, but with very small defects", thus leaving room for further research on the technology of ice cream preparation and the method of incorporation of grape skin powder.

## Conclusions

In the context of current industrial development, and food waste resulting from the process of food processing, food waste should be capitalized as much as possible, as it can be used as a resource for many other processes, such as the production of dyes, additives with biological potential. assets, feed or energy. The paper explored the possibility of using grape skin in the production of ice cream. The technology for producing ice cream (sorbet) with the addition of grape skin was developed. he addition of grape skin powder significantly contributes to the increase of the total polyphenol content (63.93-139.29 mg AG / mL extract) and the antioxidant activity ( $33.63 \%$ for the $10.00 \%$ GS sample).

It has been established that the use of grape skin significantly influences the color of the processed ice cream, the color difference varying within the limits $\Delta \mathrm{E}=37.37-54.94$. As a result of the sensory analysis, all the samples received scores that put them in the following categories: The product has a specific positive characteristic, very well defined, no defects with a total acceptability of 4.50-4.88.

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