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Impact of the biotechnological applications in the case of the citrus fruits flavours extraction on the quality of the juices, jellies and the medicinal candies

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Abstract
Currently, the assortments of the juices and jellies which are obtained by adding identical natural flavours or artificial flavours are large and have a high frequency of consumption at the children. In addition, candies as sugary products, but also medical candies, in the manufacturing recipes, flavours that give them the pleasant aromatic taste which is associated by the fruits. The pesticide residues were removed by immersing the fruits and keeping them in water 48 hours; at the peel fraction, it was measured the yield of it, then it was separated the aromas by extraction with alcoholic solutions with different concentrations. Following the cryo-concentration the flavor in the gelatin gel, flavored gels were introduced into cold liquid mixtures in the case of juices at 2-5ºC, in warm solid mixtures in the case of jellies at the 10ºC and medical candies at 20ºC. No flavor or color changes were obtained after the preparation of the citrus fruit extracts. Even the aroma remained persistent and the associated color. The biotechnological phases applied did not change the sensory or physical and chemical characteristics of the cryo-jellies obtained from citrus fruit extracts. It were prepared 14 samples from the concentrations of extracts by 1%, to 10% in one step and to the concentrations of 15% to 50% in the next step. To test the extracted flavours, fruit juices were used in two variants, the first variant with a concentration of 100% fruit juice and the second variant with a concentration of 50% with a dilution of 1:2. The results were the following: the yield of peel separation in the analysed citrus fruits decreases from 21.14% in mandarin to 4.24% in the grapefruit. The amount of flavour extracted after 3 days at the lime fruits the variation was from 62.6% (5% alcoholic sol.) to 27.8% (25% alcoholic sol). The cryo-jellies indicated an increasing concentration of the extract at lime, mandarin, orange, grapefruit and an average inflection point at the cryo-jellies of the orange (95%-43.27%), mandarins (95%-17.72%), lemon (77.4%-51.4%), lime (62.6%-27.8%) between the concentrations of 5-25%. Thus, initially the addition substances used to preserve citrus fruits were eliminated, in the case of some citrus fruits, such as the lemon, lime, orange, grapefruit and mandarin. In the second stage it was followed by extraction of flavouring substances, then concentration by freezing, impregnation on a gelatine backing and testing of oily citrus aroma on the juices or candies.
Key words: biotechnology vs aromatic character of citrus fruits

1. Introduction

In recent years, there has been increased interest in the use of natural dyes in various branches of industry, especially in the food industry, considering the harmful aspects (high degree of toxicity, as well as consumer health disorders) of synthetic additives. The results of research in the field have shown that natural dyes are appreciated due to their antioxidant properties, bioavailability, beneficial effects on health. Also, an important factor is the diversity of plant raw materials (plants, seeds, fruits and vegetables) from which natural dyes can be obtained, with emphasis also being placed on secondary raw materials, as well as environmentally friendly technological

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methods of production [1]. The fruit peel is an agricultural by-product and potential source to extract natural aroma compounds with low cost. In the past few decades, the extraction of plant aroma volatiles experienced a transition from traditional to modern technologies. The applications of fruit peel aroma extract in food, pharmaceutical and cosmetic industries are important for a lot of fields. This review provides comprehensive information for extraction and application of aroma compounds from fruit peels, which could facilitate the valorisation of the agricultural by-products and reduce environmental impacts [2].

The analysis of the citrus fruits after sensory characteristics is done according to specific indications of color, aroma, taste, which in the fruit becomes pleasant, sweet, acid, with the attenuation of astringency, when the pulp softens, the skin thins and is covered with a protective layer [3].

The fruit juice is one of the most valuable non-alcoholic beverages, characterized by a high content of carbohydrates, glucose, organic acids, minerals, vitamins, flavors and high nutritional value [4]. The profile of phenolic compounds in citrus fruits is given by the specific phenolic matrix that can be used to detect the species. Fruits contain two types of phenolic compounds, phenolic acids and flavonoids. The flavonoids are found mainly as glycosides, being represented by the flavanols, the flavones, the aurones, the chalcones, the flavanones and isoflavones [5].

A study investigated the impact of mosambi peel extract (MPE) on the stability of protein and textural properties of silver carp surimi during frozen storage at −20 °C. The findings demonstrated that surimi supplemented with 0.75% MPE exhibited improved protein solubility (PS), water-holding capacity (WHC), Ca^{2+}-ATPase activity, and sulfhydryl (SH) content compared to the control group treated with synthetic cryoprotectants. Furthermore, the MPE-treated surimi showed lower levels of total carbonyls and surface hydrophobicity, as well as higher overall acceptability throughout storage. MPE also effectively slowed the increase in total volatile basic nitrogen (TVBN) and the protein degradation index (PI). SDS-PAGE analysis revealed higher intensities of myosin heavy chain (MHC) and actin in the MPE-treated group, indicating reduced protein degradation over time. Additionally, the incorporation of MPE mitigated lipid oxidation, as evidenced by lower peroxide value (PV) and thiobarbituric acid (TBA) levels compared to the control. Fourier-transform infrared spectroscopy (FTIR) indicated greater stability of α-helix and β-sheet structures in the MPE-treated group, further correlating with enhanced gel and textural properties. These results suggest that 0.75% MPE could serve as a natural alternative to synthetic cryo-protectants, ensuring oxidative stability and preserving the quality of silver carp surimi during frozen storage while providing added bioactive benefits [5].

The odorous or aroma compounds from citrus fruits are determined by gas chromatography coupled with GC-MS mass spectrophotometry, which allow the separation and dosing of volatile odorous compounds characteristic of each species, but also the discovery of adulterations by the addition of analogous odorant substances. So, the analysis of the spatial structure of the flavor components proved that they are found in an enantiomeric type, which distinguishes them from the synthetic ones. Carotene pigments constitute a class of pigments well represented especially in the oranges and apricots [6]. More than 50 carotenoid compounds have been found in oranges, difficult to separate and dose each one [7].

In this experimental research it has proposed the extraction of the natural aromas from citrus fruits, orange, grapefruit, lime, lemon and mandarin, with alcoholic solutions, between 1% to 50%, which are more stable aromas over time and have the greatest flavoring power. The color of the aromatic extract obtained from citrus fruits was also monitored.

2. Material and methods

In this study, 5 citrus fruits, oranges, grapefruit, lemons, limes and tangerines, were analyzed, where were determined the peel yield, the percentage of flavor extracted from the solid fractions and the flavor was separated by cryo-concentration in gelatin gel. The obtained gels were later tested on juices, jellies and medical candies for natural flavoring.

2.1. Experimental technology: weighing, separation of the peel, maceration, extraction of aroma compounds with hydroalcoholic solutions, filtration, cryo-concentration in two stages (1. pre-cooling at -5ºC (duration 8 hours), stage 1, 2. crystallization-actual cooling -15ºC stage 2 (duration 16 hours)), Centrifugation to separate ice crystals, separation of concentrated juice, addition gelatin-homogenization and dispersion in concentrated juice, advanced cooling at -20ºC (7 days).

2.2. Preparation of the fresh citrus fruits. The citrus fruits studied were checked for pesticide content. In the case of the experimental samples, pesticide residues were removed by immersing the fruits in water and keeping them in water for 48 hours.

The peel's separation. In the initial phase, the samples to be analyzed, the citrus fruits, were weighed on the analytical balance. To highlight the peel fraction, it was separated and the peel weighed. The yield is the ratio between the mass of the peel and the mass of the pulp with the peel. The masses were weighed on the analytical balance to accurately establish the results.

Separation of the fractions. In the case of separating the solid fraction from the liquid fraction, the 5 sample citrus fruits that were experimentally analyzed were centrifuged. For centrifugation, a laboratory filter centrifuge was used, with a speed of 3000 rpm and an internal diameter of 400 mm, with automatic operation, including a program with the duration of filling (5 min.), filtering (20 min.) washing (15 min.), drying (20 min.), emptying (7 min.), cleaning (10 min.). The two fractions were separated by centrifugation, which were weighed separately. Through the results obtained experimentally after centrifugation, the liquid fraction was related to the total mass of the fruit, resulting in the separation yield of the juice – the liquid fraction. It was also possible to determine the percentage of solid fraction for each individual fruit.

2.3. Preparation of the alcoholic extracts. The hydroalcoholic extractive solutions are prepared using as a solvent ethyl alcohol of different concentrations. This is a more selective solvent than it is used on a large scale to obtain extractive solutions. The favorable for extraction is alcohol with a concentration of 30%-50%. This solution hydroalcoholic has extractive power and selective capacity bigger [12]. Ethanol extracts important groups of natural compounds (active principles, polyphenols, flavoring substances, etc.) and ensures the preservation, as well as the stability of extractive solutions. The working parameters for obtaining the hydroalcoholic extracts were: 1. the material - the quality conditions imposed for each fruit, 2. the extraction solvent - ethyl alcohol 30-50%, 3. the extraction-maceration method, 4. the extract concentration -1 -50% (g/ml alcohol), 5. extraction temperature -18-20ºC,
6. extraction time 3-7 days [8].

So, its were prepared 14 samples of alcoholic solutions with concentrations by 1%, 2%, 3%, 4%, 5%, 6%, 7%, 8%, 9%, 10% in one step and to the concentrations of 15%, 20%, 25%, 50% in the next step.

Maceration. For maceration, 14 hydro-alcoholic solutions from 1% to 50% were used for the 5 types of citrus fruits - orange, grapefruit, lime, lemon, tangerine. The maceration time was 3 days. During the maceration, quantities of peel and pulp were introduced in a ratio of 1:1 compared to the extraction level, the filtration was carried out using filter paper, under vacuum, with the vacuum trunk filtration system [8].

Cryo-concentration consisted in the concentration of a solute by freezing part of the contained water and removing the formed ice crystals. The temperature at which the operation is performed depends on the nature and concentration of the dissolved substances, the limit being conditioned by the eutectic point. The dependence between cooling temperatures until the appearance of ice crystals and the concentration of solutions such as citrus fruit juices, glucose, gelatin, is specific to the products. By cryo-concentration, pure ice crystals cannot be separated because a small amount of juice adheres to their surface, due to capillary forces. Losses can be 1-2% in the case of gels made from citrus juices and gelatin.In order to reduce juice losses from 5% to 1-2%, a slow cooling and a stepwise freezing were carried out. The formed ice crystals were separated from the mixtures by centrifugation at 3000 rpm as soon as possible after formation. The freezing method took place in 2 steps at decreasing temperatures and gradual removal of ice by centrifugation. In each step, a concentration of citrus juice with 6-10% dry matter was achieved. Finally, in the third phase, the citrus juice was mixed with the gelatin gel heated to 30ºC, to stabilize and maintain the sensory properties of freshness: appearance, color, taste, aroma, given by the volatile substances of the fruit [9].

2.4. Preparation of the juices, jellies and the medical candies. To test the extracted flavours, fruit juices were used in two variants, the first variant with a concentration of 60% fruit juice and the second variant with a concentration of 30% with a dilution of 1:2. Experimentally, the separation of mandarin, lime, lemon, orange and grapefruit aromas was sought. Along with the separation of aromas, there is also a significant coloring intensity, highlighted by the carotene pigments in the peel of the citrus fruits. In order to identify the stability of the aromas and the optimal intensity of extraction, different concentrations of peel in alcoholic solution were used, from 1% to 20%, even 25% and 50%, making 1 : ½ or 1 : 1 fractions extracts. Thus the matrix of alcoholic extracts of aromas obtained from citrus fruits comprises series of 14 samples from 1% to 10%, then 1% to 15%, 1% to 20%, 25%, 50% for each variety of citrus fruit. Thus, it was followed which is the optimal extraction ratio related to the extracted aroma, quantitatively establishing the doses of aroma associated with citrus fruits. Then, the optimal concentrations, with significant flavoring power, were impregnated in gelatin and the gels which were obtained, were preserved by cryo-concentration 7 days. Following the cryo-concentration process, the flavor did not change and the gelatin gel did not change its structure. These flavored gels were introduced into cold liquid mixtures in the case of juices at 2-5ºC, in warm solid mixtures in the case of jellies at the 10ºC and medical candies at 20ºC. No flavor or color changes were obtained after the preparation of the citrus fruit extracts. Even the aroma remains persistent and the associated color. The biotechnological phases applied did not change the sensory or physical and chemical characteristics of the cryo-jellies obtained from citrus fruit extracts.

To test the aromatic extracts, cryo jellies were used in two variants, the first variant with a concentration of 100% fruit juice and the second variant with a dilution of 1:2 (1 part juice: 2 parts water). The recipe for citrus juice was as follows: 100% citrus juice, water, flavours in the form of gelled cryo-concentrates or 50% citrus juice, 50% water, flavours in the form of gelled cryo-concentrates [10].

The recipes for the jellies used common manufacturing recipes (the recipe for the jellies includes-sugar, glucose, agar-agar, natural colouring, emulsifier, citrus flavours cryo-concentrated and gelled jellies, citric acid) where the identically natural flavours have been replaced with the innovative aromas obtained by cryo-concentration [10]. For medical candies, cryo-concentrated citrus flavours have been added to manufacturing recipes containing active anti-inflammatory principles, starch, glucose, sugar, citric acid, water and others [10].

3. Results and Discussion

The dynamic of the citrus peel yield and the amount of citrus flavour separated by extraction are presented in the figures 1-2.

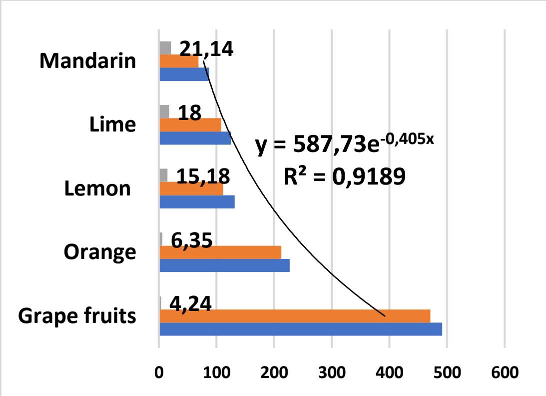


Figure 1 Dynamics of the citrus peel yield

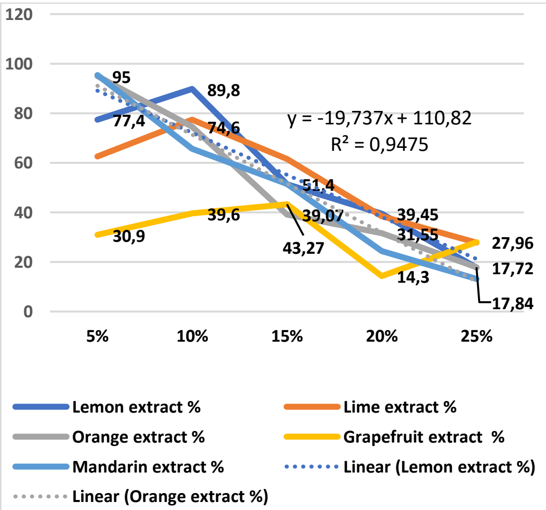


Figure 2 The amount of citrus flavour separated by extraction

It is observed that the yield of peel separation in the analysed citrus fruits decreases from 21.14% in mandarin to 4.24% in grapefruit. Thus, in small fruits the yield in the peel is the highest, as for example in mandarins and lemons, and in larger fruits such as grapefruit or orange the peel separation yield is 3-4 times lower. From this it follows that the extract will also be obtained efficiently and optimized when the amount of skin in the fruit is greater, compared to the situation where the amount of skin in the fruit is less. From a statistical point of view, the level of confidence of the experimental results is high $R^2 = 0.9189$ for the evolution of the peel fraction and $R^2 = 0.962$ for the separated peel yield, in the case of the studied citrus fruits, in relation to the separated percentage fractions. The amount of flavour extracted after 3 days varies decreasingly from 95% for mandarins at 5% alcohol solution to 17.72% with 25% alcohol solution. In lemon the variation is from 77.4% for 5% alcoholic solution, 51.4% for 10% alcoholic solution, 31.56% for 20% alcoholic solution and 17.82% for 25% alcoholic solution. At the lime fruits, the variation was from 62.6% at 5% alcoholic solution to 27.8% at 25% sol. alcoholic, and orange fruits the variation is from 95% for 5% alcoholic solution,43.27% for 25% alcoholic solution, 39.45% for 20% alcoholic solution and 17.72% for 25% alcoholic solution. The statistical distribution of the research results varies linearly according to a good probability $R^2= 0.9475$ both for the lemon extract and for the orange extract (Fig. 2).

The peel solid fractions remaining after extraction had a linear statistical distribution between a confidence level of $R^2 = 0.9912$ and a maximum confidence level of $R^2=0.9979$, the results having excellent scientific significance (Fig. 3). From matrix of samples it selected the optimal experimental results (table 1).

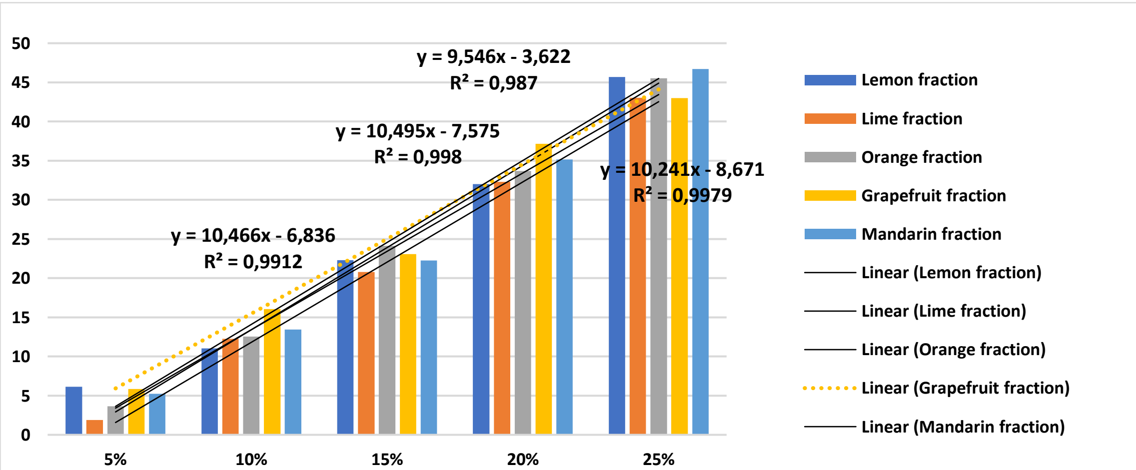


Figure 3 Amount of solid fraction remaining after extraction

Table 1 Comparative analysis of citrus extracts with cryo-jellies obtained from extracts by cryo-concentration

Citrus fruits samples Alcoholic solutions %	S1Mandarin samples, g/100ml		S2Grapefruit samples g/100ml		S3Orange samples g/100ml		S4Lime samples, g/100ml		S5Lemon samples g/100ml	
	extract fr. S1	Cryo jellyS1	extract fr. S2	Cryo jellyS2	extract fr. S3	Cryo jellyS3	extract fr. S4	Cryo jellyS4	extract fr. S5	Cryo JellyS5
1%	10.7	0.17	10.1	0.1	11.4	0.11	7.5	0.075	9.1	0.091
2%	11.4	0.14	15.3	0.1	12.65	0.12	8.1	0.080	14.3	0.143
3%	11.9	0.12	16.3	0.104	13.53	0.13	8.4	0.084	16.31	0.163
4%	12.61	0.106	17.5	0.107	16.53	0.16	8.35	0.085	17.72	0.173
5%	95.40	8.01	30.9	8.34	95.00	14.69	62.6	15.97	77.4	13.52
6%	87.38	8.93	32.6	3.26	89.78	8.978	61.3	5.99	80.97	8.097
7%	81.25	8.02	34.5	3.45	85.21	8.521	62.47	6.273	83.12	8.312
8%	74.38	7.4	36.14	3.84	81.90	8.190	64.82	6.321	85.71	8.539
9%	67.17	6.67	38.72	3.89	79.48	7.948	61.83	6.657	87.05	8.727
10%	65.70	11.12	39.6	9.07	74.6	13.5	77.4	14.85	89.8	12.82
15%	51.53	13.30	43.27	14.14	39.07	17.61	61.53	17.61	51.4	9.52
20%	24.35	9.94	14.3	5.92	31.55	15.09	38.45	15.09	39.45	13.74
25%	13.16	6.12	27.96	12.01	17.84	13.23	27.8	13.23	17.72	7.62
*50%	*NA	x	NA	x	NA	x	NA	x	NA	x

*The samples 14 with 50% absorbed all the solvent and no result was obtained on all citrus fruits tested, so this mixing ratio is not recommended.

The highest level of confidence was obtained in the extraction and cryo concentration of jellies with 25% alcohol solution, and the lowest level of confidence is observed at 15%

(table 2).
The dynamics of alcoholic citrus extracts (Fig. 4) have showed a high level of confidence R^2 = 0.8891 for 5% extracts of the lime, orange, lemon, grapefruit, mandarins / tangerine and a medium level of confidence R^2 = 0.5348 for 15% extracts of lime, orange, lime, grapefruit, mandarin.

Table 2 Confidence level of experimental results related to the difference between extracts and cryo jellies

Samples	Lime extract			Orange extract			Lemon extract			Grapefruit extract			Mandarin extract			
Alcoholic sol. %	Cryo jellies	Extracts	Differences, %	Cryo jellies	Extracts	Differences , %	Cryo jellies	Extracts	Differences, %	Cryo jellies	Extracts	Differences, %	Cryo jellies	Extracts	Differences, %	Pearson correlation
5%	7.94	8.13	2.34	6.54	6.64	1.51	7.43	8.87	16.23	4.17	4.37	4.58	4.77	4.87	2.053	0.8401
10%	7.16	7.74	7.49	7.46	7.96	6.28	6.32	8.89	28.91	3.96	3.99	0.75	6.57	6.77	2.954	0.8807
15%	8.67	8.67	9.23	6.31	7.86	19.72	4.56	7.71	40.86	6.34	6.94	8.65	7.73	8.01	3.496	0.6028
20%	7.11	7.11	7.69	4.46	6.31	29.32	6.79	7.98	14.91	2.86	3.16	9.49	4.87	5.14	5.253	0.8975
25%	6.5	6.5	6.95	1.72	4.46	61.43	3.05	4.33	29.56	5.99	6.99	14.31	3.29	4.51	27.051	0.9984

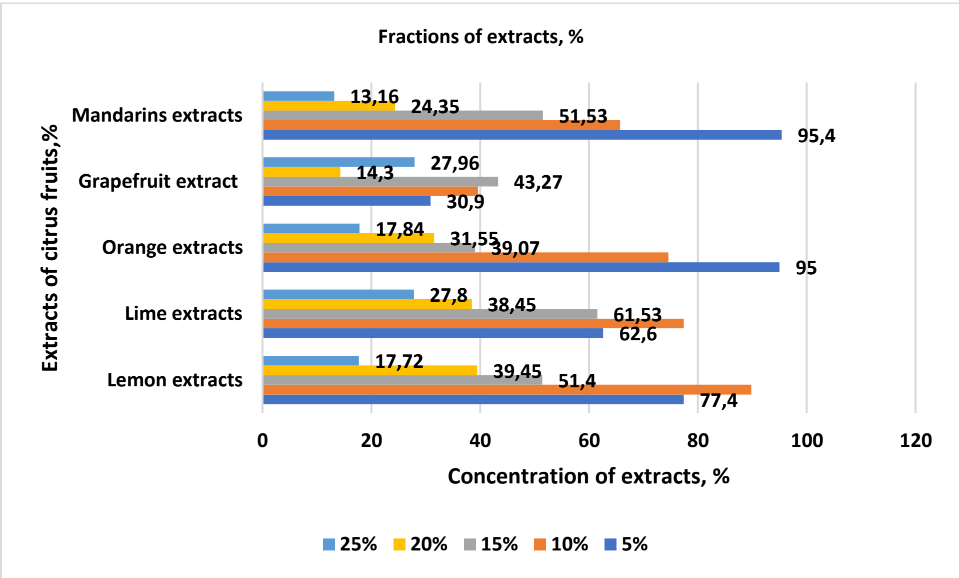
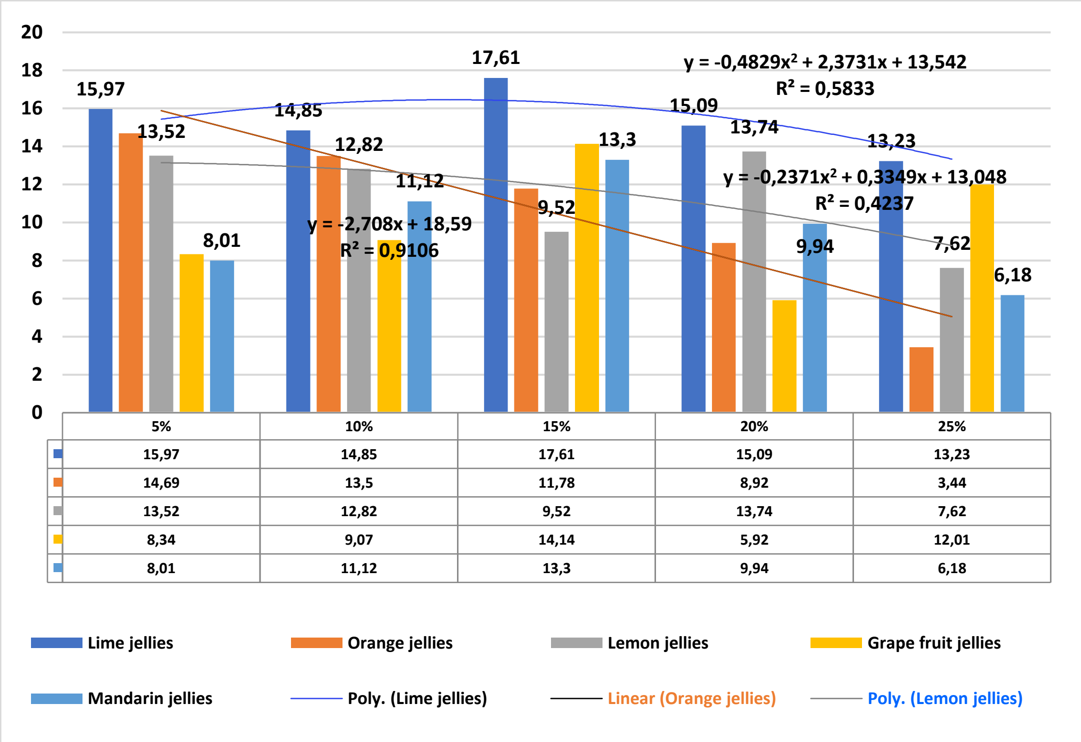


Figure 4 Dynamics of aromatic extracts obtained from the citrus extracts

Figure 5 shows the dynamics of the amount of gelled flavour for each the citrus. Statistically, the research results have a positive, a significant confidence level as follows: lime extract R^2 = 0.9542 very high level of confidence, for the mandarin's extract R^2 = 0.5833 above average level of confidence, lemon's extract obtained a medium level of confidence, R^2 = 0.4237. The oranges extract R^2 = 0.9106 has obtained the highest level of confidence and the grapefruit's extract R^2 = 0.583 has obtained the lowest level of confidence. The taste was weak and un specifically.
The figure 6 reflects by comparison the evolution of concentrated jellies and the cryo-jellies after cryo-concentration at different alcohol concentrations 5%, 10%, 15%, 20%, 25%. The significant flavour concentrations were obtained in the mandarins, oranges, lemon and lime jellies and cryo-jellies. Grapefruit jellies and cryo-jellies had had a weaker flavour and colouring power. The dynamics of the cryo-jellies indicate an increasing concentration of the extract in lime, mandarin, orange, grapefruit. The citrus concentrates had an average inflection point at: the cryo-jelly of the orange, mandarin, lemon, lime at a concentration of 15% and the maximum value was recorded at an alcohol concentration of 25%. The concentration of the extract increased 4-5 times at the 25% alcoholic solution.



	5%	10%	15%	20%	25%
Lime jellies	15,97	14,85	17,61	15,09	13,23
Orange jellies	14,69	13,5	11,78	8,92	3,44
Lemon jellies	13,52	12,82	9,52	13,74	7,62
Grape fruit jellies	8,34	9,07	14,14	5,92	12,01
Mandarin jellies	8,01	11,12	13,3	9,94	6,18

Figure 5 Dynamics of cryo-jelly amounts obtained from the citrus fruits extracts

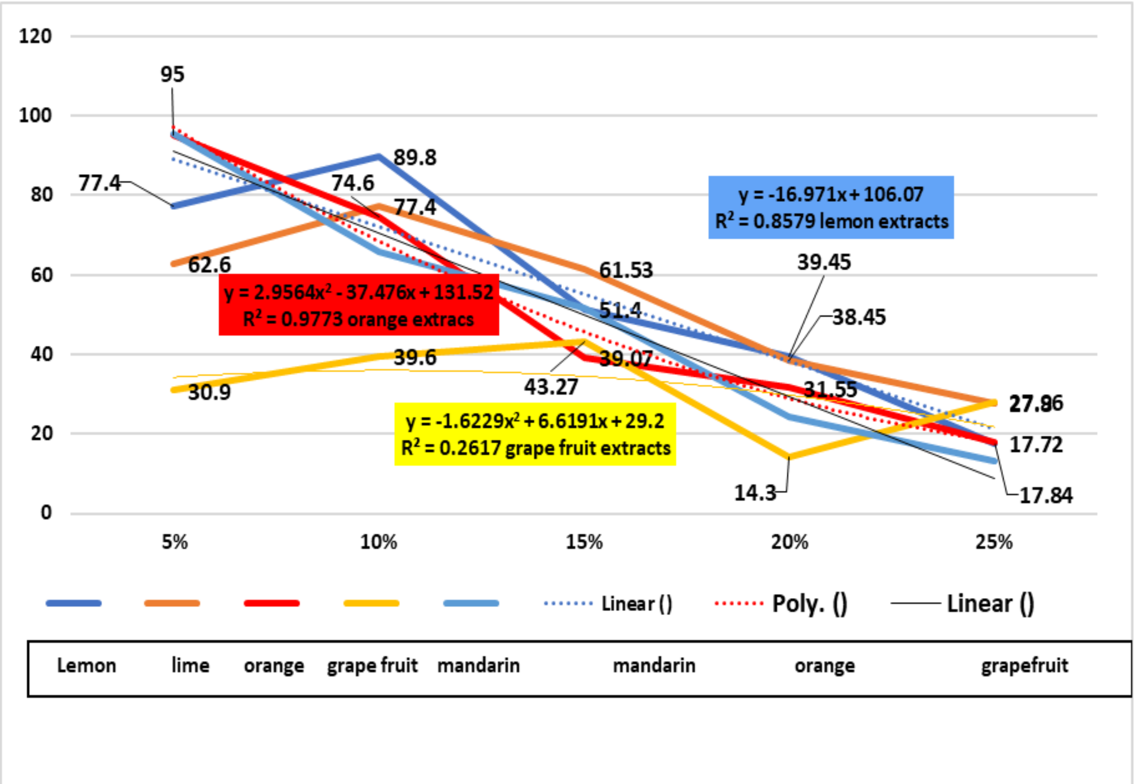


Figure 6 Dynamics of the cryo-jellies at the different concentrations

The stability of cryo-concentrated jellies obtained from citrus peel and their clarity as extracts are observed. The intensity of the color clearly defines the types of citrus fruits from which the flavors were extracted, which has a special biotechnological significance. This allows us to obtain cryo-jellies for flavoring, which at the same time will also have a high coloring power of juices, jellies or medicinal candies (Fig. 7). The mandarin cryo-jellies had the highest flavoring power, then oranges, lemons and limes. If we look at the cryo-jellies coloring, associated the power was the greatest at the mandarins and oranges, as can be seen in fig. 8.

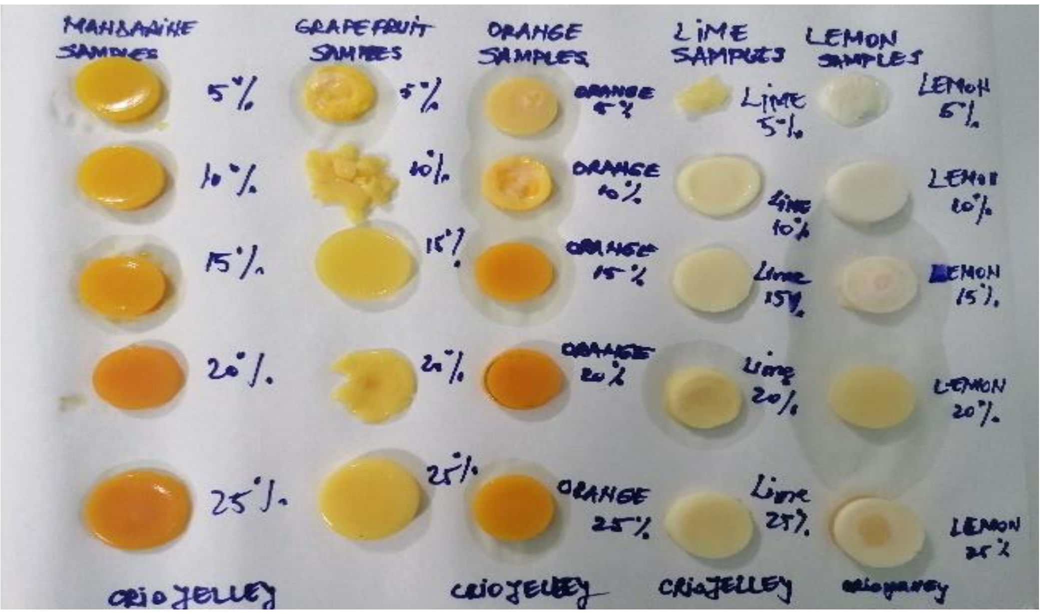


Figure 7 Images of aromatic cryo jellies obtained from the citrus fruits

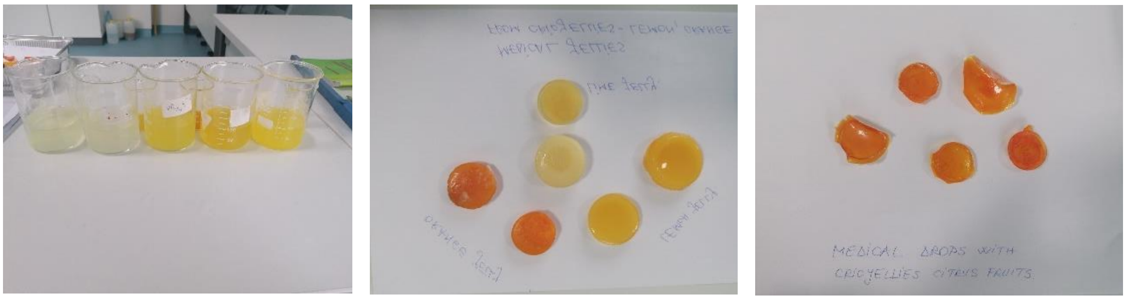


Figure 8 Image of juices, jellies and medical candies made from cryo-jellies

The medical candies were obtained with medical substances including in the recipes the aroma and color substances which were cryo-concentrated jellies and which resulted by extraction and cryo-concentration [10]. The mandarin extract was sweetened using the honey [11].

4. Conclusions

1. The mandarin, as small fruit, have an increased yield in the peel fraction, along with the lemon and lime, while the orange and grapefruit, being larger fruits, show that the peel fraction is lower.

2. The citrus peel flavour extracts have strong aromatic character and a significant colour intensity. Thus, the most colourful extract was the orange extract, followed by the mandarin extract, then the grapefruit extract with various shades of orange-yellow. The lemon and lime extracts have a weaker coloration, the most intense being the lemon extract. The colouring and flavouring intensity of the extracts have varied direct proportion to the amount of peel added.

3. Through the cryo-concentration of the extracts, the aim was to separate the water from the obtained aromas and the gelling of the extracts and it was a method of preserving the aroma fractions to avoid their oxidation over time and the loss of aroma and colour properties.

4. The flavour gels extracted from the citrus fruits obtained by cryo-concentration can be used to obtain recipes for the juices, the jellies or the medical candies/drops, thus replacing the practices of the classic technology of aromatization and acidulation by adding of the

identically natural compounds, similar to the natural compounds, which are surrogates or analogous substances.

5. The cryo-concentration and gelation of flavour extracts obtained from citrus peel revolutionizes the technique of obtaining and using flavours in various food products, thus reducing the practice of introducing excess food additives.

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