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Development of an instant drink rich in polyphenols from the flower of the majagua (*Talipariti elatum*).

Desarrollo de una bebida instantánea rica en polifenoles a partir de la flor de la majagua (Talipariti elatum).

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Abstract

The aim of this research was to develop a drink powder enriched with a concentrated extract of majagua flowers (*Talipariti elatum*). An experimental design was carried out using the numerical optimization method with an I-Optimal response surface design to generate a model mathematician who described the alterations of the response variables while considering the conditions of the process. Using the co-crystallization method, a dry granular material was obtained in a matrix of sucrose. The powder systems were characterized regarding the variables: humidity, total polyphenol content, cohesiveness, and acceptability; A sensory analysis was also carried out during the preparation of the system instant. The best responses were obtained for an extract value of 6.4% and estimated values of 1.85% humidity; 69.23 mg AGE/100 g; with a cohesiveness acceptable and a statistical convenience of 61%. The obtained powder was characterized by homogeneity, adequate fluidity, and high solubility capacity in watery systems. For the sensory evaluation, the most important descriptors were smell and taste. The ready-made system obtained excellent quality and sweet taste; it turned out to be light but pleasant to the palate, receiving a rating of “I like it” from some consumers.

Keywords. Instant beverage co-crystallization in sucrose; majagua flowers; polyphenols.

Resumen

El objetivo de esta investigación fue desarrollar una bebida en polvo enriquecida con un extracto concentrado de flores de majagua (*Talipariti elatum*). Se llevó a cabo un diseño experimental utilizando el método de optimización numérica con un diseño de superficie de respuesta I-Optimal para generar un modelo matemático que describiera las alteraciones de las variables de respuesta considerando las condiciones del proceso. Utilizando el método de co-cristalización, se obtuvo un material granular seco en una matriz de sacarosa. Los sistemas en polvo se caracterizaron en cuanto a las variables de humedad, contenido total de polifenoles, cohesividad y aceptabilidad; también se realizó un análisis sensorial durante la preparación del sistema instantáneo. Las mejores respuestas se obtuvieron para un valor de extracto del 6.4% y valores estimados de 1.85% de humedad; 69.23 mg AGE/100 g; con una cohesividad aceptable y una conveniencia estadística del 61%. El polvo obtenido se caracterizó por su homogeneidad, adecuada fluidez y alta capacidad de solubilidad en sistemas acuosos. En la evaluación sensorial, los descriptores más importantes fueron el olor y el sabor. El sistema listo para consumir obtuvo una excelente calidad y un sabor dulce; resultó ser ligero pero agradable al paladar, recibiendo una calificación de "me gusta" por parte de algunos consumidores.

Palabras clave. Bebida en polvo instantánea, co-cristalización en sacarosa, flores de majagua, polifenoles.

Introduction

In recent years, a large number of powdered beverages based on plant extracts have emerged, innovative products that set a new trend in the market and have experienced rapid growth over time. Powdered soft drinks or instant beverages are products consumed by both children and adults of all ages. The modern consumer demands products that have excellent organoleptic characteristics, provide health benefits and make up for the possible deficiencies that the current pace of life is causing[1]. Plant extracts are a rich and complex source of active ingredients with diverse chemical and physical properties, which makes them a viable alternative for the food industry.

The majagua flower (*Talipariti elatum*) is considered an important source of bioactive compounds such as phytosterols, phenols, organic acids, some of which have antioxidant properties and are considered to have others that are still being studied, always with the vision of improving human health; its phenolic content as gosipetrin-3'-Oglucoside and glycosylated derivatives of quercetin have demonstrated their antimicrobial action, neuroprotective and also against cancer[2].

The co-crystallization method is a microencapsulation process where two ingredients are incorporated into a porous conglomerate of sucrose microcrystals formed by spontaneous crystallization. It is a flexible and economical alternative, since it is a simple procedure; many products can be encapsulated using this technique, such as fruit juices or pulps, essential oils, flavorings, flavorings, among others [3].

The contribution of polyphenols in food provides great benefits to the health of the organism. Consumption, their effects are fundamentally a consequence of their antioxidant properties. They have vasodilatory effects; they are also capable of improving the lipid profile and attenuating the oxidation of low-density lipoproteins (LDL); they have clear anti-inflammatory effects and in turn are capable of modulating the processes of apoptosis in the vascular endothelium. Other multiple benefits they offer are: improving digestion and brain health, reducing the impact of diseases such as type 2 diabetes and even the appearance of certain types of cancer [4].

As additives are highly questioned, soft drinks have become an object of study, since among the ingredients the best known is sugar and not all of them contain it. In soft drinks we can find additives such as colorants, flavorings, clouding agents (to give the appearance of consistency), anti-humectants and anti-caking agents, acidulants, sweeteners and preservatives [1].

Other important additives to take into account are sweeteners, since they provide the sweet taste to the preparation. The mixtures of these are found in soft drinks and in some cases with sugar to reduce the nutritional value of the beverage. From the nutritional point of view, the calories they provide per serving of consumption correspond to carbohydrates from sugars [1].

Taking into account this background, the following research hypothesis was proposed: what concentration of majagua flower extract allows obtaining an instant beverage rich in polyphenols with acceptable quality using co-crystallization in sucrose?

In order to respond to the proposed hypothesis, the objective was to develop an instant beverage rich in polyphenols from the majagua flower (*Talipariti elatum*).

Materials and methods

The flowers of majagua (*Talipariti elatum*) used were collected manually, selecting in general those that presented the same characteristics of vegetative state, size, color, absence of spots, cracks and visible morphological alterations by fungi and parasites. The petals were separated from each other and from the pistil in each flower and were frozen at -32 ºC until their subsequent use.

Obtaining the extract

Extracts were prepared maintaining a mass/solvent ratio of 1:2, with a 78% hydroalcoholic mixture (acidified with 1% w/v citric acid) and extraction time 12 hours. The maceration was carried out in a sieve at 250 min-1 at room temperature. At the end of the extraction time, the resulting mixture was filtered under vacuum, discarding the solid residue. The hydroalcoholic extract was concentrated in a rotoevaporator Mod. R-114 rotoevaporator (BÜCHI, Switzerland) at 40 °C and 100 min-1 until a concentration of total solids between 10 and 11% was obtained.

Total solids, pH, and total phenolic content were determined in the extract [5].

Experimental design

To determine the maximum concentration of extract to be used in the experimental design of the instant beverage, 4 samples were prepared with different concentrations of concentrated majagua flower extract (0.5, 1, 1.5 and 2 mL per 100 mL of a beverage prepared by adding 150 µL of strawberry flavoring; 0.4 g of red colorant, 14 g of sucrose and 0.25 g of citric acid). Samples were evaluated using a 10-cm linear scale, structured every 2 cm with increasing intensity categories [6] of bitter aftertaste and overall quality with a scale from terrible to excellent according to quality categories [7]. The panel consisted of 5 trained tasters.

Design Expert 11.1.0.1 (Stad-Ease Inc., Minneapolis, USA) was used for the experimental design and processing of the results. The numerical optimization method used was the I-Optimal response surface design, which generated a mathematical model that described the alterations of the response variables taking into account the process conditions. The independent variable was the concentrated extract content (A). The response variables were total phenol content, moisture, cohesiveness and acceptability.

Obtaining powdered drinks

For each experimental run, sucrose syrups were prepared. First, 50 g of sugar was weighed and 17% (w/w) water was added, mixed in a 200 mL beaker in order to obtain a concentrated solution [8]. It was heated on an electric hotplate until a temperature of 130 °C was reached [9], which indicated the degree of supersaturation necessary for spontaneous crystallization to occur upon cooling. At that point, the syrup was removed from heating and the amount of extract specified in Table 1 to which strawberry aroma (14 µL/g sucrose) and 0.03% (w/w) red color had previously been added was quickly added. Subsequently, the mixture was subjected to constant stirring for 600 min-1 with the aid of a portable mechanical stirrer, equipped with straight metal blades 4 cm in diameter [8]. The stirring process was stopped when the formation of a granular solid material was observed. The granular material was placed in Petri dishes and then subjected to a drying process at 55 °C in an oven with forced air circulation for 24 hours. After this time, the samples were manually ground and sieved with a 1 mm sieve [3]. Finally, the sieved samples were mixed with citric acid (1.78%) and placed in a desiccator until the corresponding analyses were carried out.

For the quantification of total polyphenols, 0.5 g of the powdered beverage was taken and dissolved in 5 mL of distilled water. It was vortexed (IKA 3, Germany) for 3 minutes[5]. Determinations were performed as indicated in the previous section. The results were expressed as mg AGE/100 g of powdered beverage. The % retention was also determined taking into account the polyphenol content added at the beginning of the co-crystallization.

It was determined by indirect gravimetry (volatilization), by drying in a thermobalance (Sartorius Mod, MA-40, Germany) at 120 ºC to constant mass, with the purpose of separating the water from the powdered product. The Hausner index measures the flow properties of the powders. Values above 1.25 indicate powders with low flowability; values below 1.25 indicate free-flowing powders [10].

The data were analyzed using a one-way analysis of variance (ANOVA) using the SPSS computer program (IBM SPSS Statistics.25.) and the rank test was used Duncan multiples to compare the differences between the treatments evaluated. The significance level used was $p \leq 0.05$. The data obtained is presented as mean (standard deviation).

Results

Table 1 shows the characterization carried out on the majagua flower extract and afterwards. Having been concentrated in terms of the most important physical and chemical parameters.

Table 1. Physical and chemical indicators of the extract and concentrated extract of flowers of majagua (n = 3).

Indicators	**Mean (Standard deviation)**	
	Extract	Concentrated Extract
pH	3,67 (0,01)	2,81 (0,02)
Total Solids (%)	4,06 (0,34)	10,81 (0,54)
Total Polyphenol Content (mg AGE/ mL)	3,24 (0,13)	10,26 (0,16)

Figure 1 represents the results obtained to determine the allowable extract levels in the drink according to the evaluation of the tasters using the global intensity and quality scale.

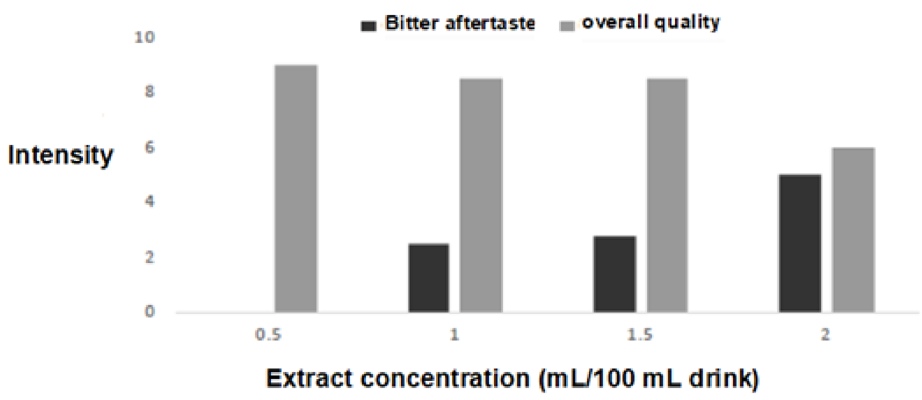

Figure 1. Evaluation of the maximum concentration of extract to be used in the formulation of the drinks.

Table 2 shows the formulations with an extract content higher than 8% (w/w) it was not possible to obtain powdered systems. The consistency obtained reflects the high levels of moisture and amorphous regions in the structures formed. To obtain a powdered product after drying, a granular consistency with a dry appearance must be produced during the co-crystallization process. Moisture increased with increasing extract concentration, being higher than 2 % for concentrations of 8 %. Moisture higher than 2 % caused an increase in the cohesiveness of the powders.

Table 3 shows the significance of the regression analysis of variance for the response variable moisture. It was observed that the linear model was significant at 95.0% confidence level. The R^2 statistic indicated that the fitted model explained 87.43% of the variability of moisture content in the formulated powders. The lack of fit F-value of 0.29 implies that the lack of fit is not linear for α=0.05. As shown in Figure 2, the co-crystallization process produced a dry product and only gentle drying at 55 ◦C was necessary to obtain a powder with free-flowing particles at humidities below 2 %. The equation obtained for the model shows that the average moisture content of all experimental runs is 2.12 % (intercept of the fitted equation).

Table 2. Results obtained for the experimental design matrix.

Experimental runs	Extract (%)	Consistency	Total polyphenol content (%)	Humidity (%)	Cohesiveness HI	Acceptance
		Creamy	-	-	-	-
1						
2	**12**	Dry granular	87,42 (1,75) a	2,24 (0,20) a	1,29 (0,01) a	4,96 (0 ,96) a
3	**8**					
4	**4**	Dry granular	42,97 (0,96) b	1,21 (0,13) d	1,18 (0,005) b	4,92 (0 ,98) a
5	**10**					
6	**8**					
7	**6**	Creamy	-	-	-	-
8	**8**					
9	**4**					
	4	Dry granular	85,87 (2,12) a	2,04 (0,22) ab	1,29 (0,01) a	4,81 (0,85) a
	8					
10						
		Dry granular	63,34 (0,96) c	1,92 (0,26) b	1,23 (0,008) c	3,35 (1,09) a

		Dry granular	86,65 (1,16) a	2,04 (0,09) ab	1,29 (0,01) a	4,42 (0,90) a
		Dry granular	41,28 (2,55) b	1,44 (0,12) cd	1,19 (0,007) b	4,35 (1,29) a
		Dry granular	43,11 (0,70) b	1,57 (0,11) c	1,20 (0,009) b	4,58 (1,10) a
		Dry granular	86,55 (1,82) a	2,07 (0,37) ab	1,28 (0,01) a	4,58 (1,33) a
11	**12**	Creamy	-	-	-	-

Table 3. Analysis of variance for the humidity present in the powdered drink.

Factor	**F-value**	**p-value**
Model	41,73	0,0007
Extract (A)	41,73	0,0007
Lack of fit	1,37	0,2951
R-squared	0,8743	
adjusted R-squared	0,8534	

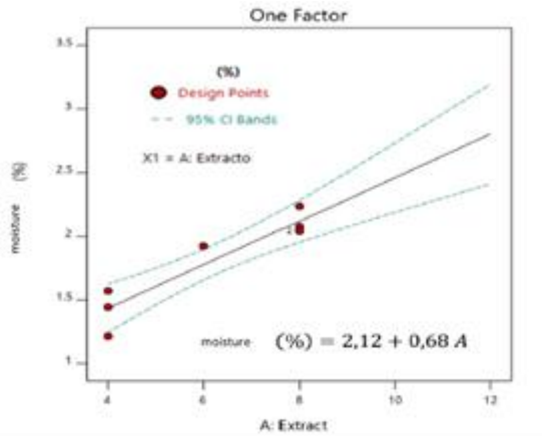

Figure 2. Estimated response surface for the humidity variable according to the fitted equation.

The total polyphenol content present in the co-crystallized products is closely related to the composition of the extract and the levels incorporated into the sucrose matrix. In this regard, Table 4 shows the significance of the regression analysis of variance for the response variable: polyphenol content.

Table 4. Analysis of variance for the CPT and cohesiveness of the analyzed powders.

Factor	Cohesiveness		Total polyphenol content	
	F-value	**p-value**	**F-value**	**p-value**
Model	480,63	<0,0001	4445,88	<0,0001
Extract (A)	480,63	<0,0001	4445,88	<0,0001
Lack of fit	3,61	0,1160	1,89	0,2272
R-squared	0,9877		0,9987	
adjusted R-squared	0,9856		0,9984	

Figure 3 a, shows the adjusted equation for the cohesivity response variable. On average, the powders obtained were not very cohesive (according to the intercept); an increase in the extract led to a slight increase in cohesivity. The formulated powders presented a high content of polyphenols (Figure 3 b), obtaining an average of 86.49 mg AGE/ 100 g of powdered beverage.

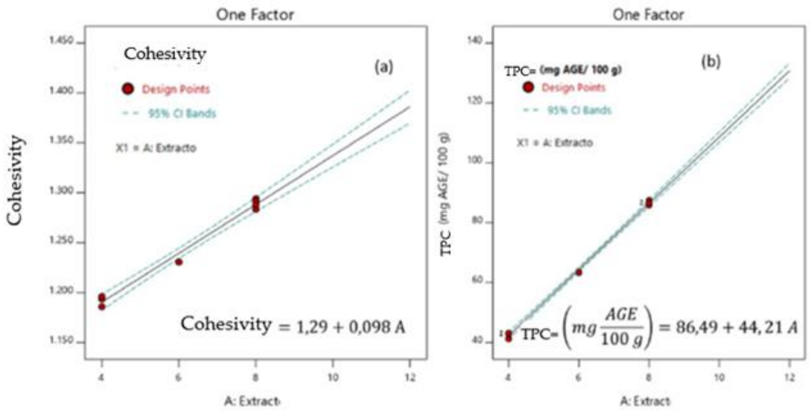

Figure 3. Estimated response surface for the cohesivity (a) and TPC (b) variables according to fitted equations.

For the numerical optimization of the process of obtaining the instant beverage, the intervals shown in Table 5 were used as restrictions. Under these restrictions, an optimum was obtained for an extract concentration of 6.4 % and estimated values of 1.85 % moisture; 69.23 mg AGE / 100 g with an acceptable cohesiveness (1.25) and a statistical convenience of 61 %.

Table 5. Constraints for the optimization of the co-crystallization process.

Parameters	Criteria	Lower limit	Upper limit
Extract (A)	En el rango	6	8
Moisture	En el rango	1,21	2,24

Total Phenol Content (TPC)	Maximizar	41,2762	87,4182
Cohesivity	En el rango	1,18	1,25

Discussion

The values corresponding to all the indicators increased when the concentration process was carried out except for the pH, which decreased, mainly conditioned by the addition of citric acid to the hydroalcoholic mixture. This is a desired value, since anthocyanins are more stable in this pH range (Table 1).

The total solids content provides relative information on the amount of non-volatile constituents present in the extract. The percentage (unconcentrated extract) corresponded to that reported [11]. The total solids content (4.06% TS) was slightly lower than reported [12] (5.57% TS), which could be mainly due to the extraction method, which in this case was maceration.

Regarding the content of total polyphenols (3.24 mg/mL), similar results were obtained to the study[13] (3.58 mg/mL) in a hydroalcoholic extract of *Talipariti elatum*; higher than that obtained[14], 2.3 mg/mL of polyphenols in a majagua flower extract for the formulation of a mayonnaise-type dressing and below 6.90 mg/mL of polyphenols obtained [12]. In the study of phenolic compounds present in the flowers of the species *Talipariti elatum* and quality control of its fluid extract.

In general, the difference in the results could be conditioned by the influence of extrinsic factors (characteristics of the ecosystem where the plant grows, harvesting season, among others) and intrinsic factors (ontogenic factors, phenological state, among others) on the plant material, that lead to variations in the content of metabolites and therefore in the total solid content.

As can be seen (Figure 1), up to the concentration of 1.5 mL of concentrated extract per 100 mL of drink, the perception of the bitter aftertaste turned out to be very light with an overall quality of very good according to the evaluated scale. In the case of the maximum concentration, the bitter aftertaste was light to moderate, influencing the overall quality to decrease considerably. From this result it is deduced that the maximum concentration of the extract to be evaluated should not be greater than 1.5.

Moisture increased with increasing extract concentration being higher than 2 % for concentrations of 8 % (Table 2). The conditions used for the extract in the sucrose matrix are similar to those proposed by several authors with different plant species. Deladino [15] was one of the first to attempt to encapsulate a phenolic extract by co-crystallization by studying the incorporation of yerba mate extract. He investigated whether the concentration of the extract affected the physicochemical properties of the co-crystallized product by adding 0.7 g of lyophilized extract to 100 g of sucrose.

Years later, they published an investigation on the encapsulation of marjoram extract by co-crystallization varying only the concentration of the extract. Three different concentrations were used; 10 mL of 3, 5 and 10 g of dried marjoram leaves dissolved in 100 mL of distilled water, to which 50 g of sucrose was added [16].

Other authors also encapsulated green tea extract by co-crystallization and concluded that the effect of extract concentration on encapsulation efficiency was significant, where extract concentration was used as a variable in the design [17].

The content of total polyphenols varied in correspondence with the levels of extract in the sucrose matrix reaching a concentration higher than 85 mg (AGE)/100 g of powdered beverage (dry basis). In this sense, the most important variable in terms of temperature effects on the polyphenolic compounds in the extract is the retention variable, which shows for all the concentrations evaluated. Retention values higher than 90 % were obtained taking into account the content added at the beginning of the process, being similar for all the concentrations analyzed ($p > 0.05$).

In the case of the acceptance variable, the results show that there were no significant differences between the observed responses. All the instant beverage formulations presented levels of liking between “I slightly like” and “I like” according to the scale used I like it” according to the scale used. Considering the potential health benefits of the beverage, the results achieved in sensory terms are very favorable and stimulate the consumption of the instant beverage.

The moisture content of powders (Table 3) can influence the particles and volume, properties, physical-chemical and biological stability, which consequently affects handling and processing operations[18]. Regarding humidity and water activity, they are two interrelated terms that are influenced by several factors, including water content, type of active compound and amorphous/crystalline ratio[19].

Similar moistures (Figure 2) were reported by several authors [16,20]; these values are favorable to avoid deterioration of the products during storage. Also in 2019 a study was reported [21,22] on co-crystallized samples of *Securigera securidaca* L. seed extracts with moisture contents of 0.08 and 0.14, respectively resulting much lower than those obtained in the present investigation.

It is observed (Table 4) that the linear model was significant at 95.0% confidence level, showing that there was a statistically significant relationship between the extract content in the formulations. The R statistic indicated that the fitted model explained more than 95% of the observed variability. The effect observed for the cohesiveness variable indicates that this parameter is of importance for obtaining an instant beverage with adequate flow properties. For both cases, the lack of fit was not significant with respect to pure error.

The sustained increase in polyphenols (Figure 3) is evidence that this is a method that offers an efficiency to cover the active ingredients in the extract presenting numerous advantages. Encapsulated food bioactive components play an important role in improving the efficacy of functional foods. With innovative approaches innovative approaches for the stabilization of food ingredients, properties and health benefits. The main advantages of encapsulation by co-crystallization are its simplicity and the fact that it enhances the physical properties of active ingredients (solubility, wettability, flowability and stability) and masks bitter tastes [23].

Considering that it was possible to obtain properly adjusted models [12], we proceeded to explore the described surface to find the combination of levels in the factors that result in an optimal value of the response, this was possible since the models found explain more than 70 % of the behavior in terms of the adjusted R^2 (Table 5).

Conclusions

The unconcentrated mahogany flower extract presented a value of 4.06% total solids and a polyphenol content of 3.24%; in the case of the concentrated extract, the value was 10.81% total solids and a total polyphenol content of 10.26%. The concentrated extract content directly influenced the moisture, total polyphenol content and cohesiveness of the powders. An optimal instant beverage was obtained with extract concentration values of 6.4 % and estimated moisture and total polyphenol content values of 1.85 % and 69.23 mg AGE / 100 g respectively, with an acceptable cohesiveness (1.25) and a statistical convenience of 61 %.

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

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