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PHYSICOCHEMICAL CHARACTERIZATION OF FRUIT FILLINGS FOR BAKERY PRODUCTS

CARACTERIZACIÓN FISICOQUÍMICA DE RELLENOS FRUTALES PARA PRODUCTOS HORNEABLES

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ABSTRACT

Fruit fillings are an understudied product. Their design and formulation must take into account a series of factors inherent to their applications, including stability during thermal treatments such as baking and freezing, during which their quality must remain intact. Throughout this study, a physicochemical characterization was carried out on different baking fruit fillings prepared by a Caucana company, including Guava Fillings FRG1, FRG2, and FRG3 (different formulations), Blackberry Filling (RCZ), and Chocolate Filling (RCCH) using standard methodology. The physicochemical characterization of the fillings revealed data on their moisture content, pH, soluble solids, total solids, ether extract, and ash content. Among the results collected during this analysis, the soluble solids and pH contents found for the FRG1, FRG2, FRG3, and RCZ samples are highlighted, as these values can be compared with the supplier's requirements(FAMESA 404,FAMESA 425), with values from 74.8% to 79.6% for soluble solids and for pH from 3,84 to 4,22. It was determined that the pH of the FRG1, FRG2, and FRG3 fillings is above the established range according to the FAMESA Technical Sheet, as are the soluble solids contents, which are below the expected range. As for the Blackberry Filling, it meets the desired ranges according to the Technical Sheet for both soluble solids and pH.

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Keywords: Bakeable Fruit fillings; Blackberry; Guava; Chocolate*;* Physicochemical characterization,

RESUMEN

Los rellenos de frutas son un producto poco estudiado. Su diseño y formulación deben tener en cuenta una serie de factores inherentes a sus aplicaciones, incluida la estabilidad durante los tratamientos térmicos como el horneado y la congelación, durante los cuales debe mantenerse intacta su calidad. A lo largo de este estudio, se realizó una caracterización fisicoquímica de diferentes rellenos frutales horneables preparados por una empresa Caucana, entre los cuales se encontraban Rellenos de Guayaba FRG1, FRG2 y FRG3 (diferentes formulaciones), Relleno de Zarzamora (RCZ), y Relleno de Chocolate (RCCH) por implementación de la metodología estándar. La caracterización físico-química de los rellenos arrojó datos sobre sus contenidos de humedad, pH, sólidos solubles, sólidos totales, extracto etéreo y ceniza. De los resultados recopilados a lo largo de estos análisis, se destacaron especialmente los contenidos de sólidos solubles y pH encontrados para las muestras FRG1, FRG2, FRG3 y RCZ, por ser los valores que se pueden comparar con los requerimientos del proveedor (FAMESA 404, FAMESA 425), valores desde 74,8% hasta 79,6% para sólidos solubles y de pH desde 3,84 hasta 4,22. Se determinó que los pH de los rellenos FGR1, FRG2 y FRG3, se encuentran por encima del rango establecido por la Ficha Técnica FAMESA, al igual que los contenidos de sólidos solubles, los cuales se encuentran por debajo del rango esperado. En cuanto al relleno de Zarzamora, este cumple con la Ficha Técnica tanto para sólidos solubles como para pH.

Palabras claves: Caracterización fisicoquímica; Chocolate; Guayaba; Rellenos Frutales Horneables; Zarzamora.

INTRODUCTION

The production of fruit fillings is one of the fastest growing sectors in the food industry. These have been consumed for centuries as bakery and confectionery products, but have recently gained notoriety due to the existing demand for increasingly healthy and/or innovative products. However, fruit fillings are a little studied product and very few works address this topic in the scientific literature (Agudelo *et al*., 2014).

Fruit fillings have been used for centuries as ingredients for a great diversity of bakery and confectionery products, being currently, one of the infallible products in the so-called "snacks", boosting the expansion of the production of bakery products with the incorporation of different fruit-based fillings (Cropotova *et al*., 2016). These are characterized by maintaining their physicochemical and organoleptic properties after the baking process: the aroma and flavor of the fruit, texture, preventing the formation of cracks and avoiding syneresis (leakage of liquids), so the appearance and qualities of the filling and the final product are not significantly impaired by heating (Janna & Svetlana, 2012).

Growing consumer demand for healthier food products and an increasing demand for food due to the global population growth have forced the scientific community and the food industry to create new strategies for more sustainable food production with lower wastage rates. As a result, Carcelli *et al*, (2022), proposed the use of corn-based fiber syrup as a sugar substitute in fruit fillings; Young *et al,* (2003), developed a new polyuronan blend made of alginate and pectin, whose synergistic effect allows a stable texture and consistency of fruit fillings during the cookie baking process; Agudelo *et al,* (2014) examined the thermal stability and syneresis of the tapioca starch-pectin model. However, no reports of studies implementing sustainable production of fruit fillers were found, except Cropotova *et al*, (2016) who propose a way for the integration of some principles of circular economy, such as reuse and recycling with the food industry, in other words, they propose to reuse the solid residue left after the grinding and pressing of fruits, as raw material for the production of fruit fillers.

The food industry is increasingly more demanding with fruit fillings in terms of their thermostable and water retention properties; currently, the baking industry faces high oven temperatures, ranging around 180-200°C, therefore, it is required that the creams, toppings and fruit fillings used are thermostable and therefore remain in the dough sample during baking (Agudelo *et al.,* 2014).

The baking stability can be improved by incorporating a polysaccharide that provides higher temperature resistance. Thermal stability of these products would be an inherent property of their structures or could be acquired by using some special structure stabilizers such as: starch, gelatin, xanthan, pectin, carrageenan and other hydrocolloids (Janna & Svetlana, 2012). Nevertheless, the functionality of these compounds is largely dependent on the total soluble solids content and the contribution made by the fruit (Cropotova *et al.,* 2017).

In order to favor heat treatment it is important to take into account two basic operations in the formulation of thermostable fruit fillings: in first place, thermal processing during preparation, and oven heating during the baking process. Upon heating, especially during the baking process, the fruit filling composition undergoes several degradation processes, such as melting of sugars, caramelization, partial hydrolysis of polysaccharides and/or breaking of their glycosidic bonds, which leads to undesirable changes in the textural and sensory properties of the fillings. The addition of polysaccharides, as mentioned above, can increase the thermostability of fruit fillings by increasing the thermal degradation temperature, depending on the type of polysaccharide and its concentration. The addition of polysaccharides would also help reduce glass transition temperatures and syneresis of the fillings, which results in manufacturers storing these fillings in the freezer either alone or in a frozen, pre-baked bakery product (Cropotova *et al*., 2016).

Meanwhile, lard and coconut oil are important technological ingredients for confectionery products, mainly those related to chocolate, due to their unique physical and chemical properties, which influence the various physical properties of confectionery fillings, such as melting and crystallization behavior (Jahurul *et al*., 2013). Virgin coconut oil is generally produced using fresh coconut fruits at room temperature without the use of solvents or chemical products (Rohman et al., 2021); this oil is colorless and has a fresh coconut aroma (Mohammed *et al.,* 2021). The biological properties and health benefits of lauric acid, short-chain triglycerides, as well as phytochemicals, have been extensively studied for their potential in disease prevention. Active compounds in coconut oil include tocopherols, tocotrienols, phytosterols, flavonoids, polyphenols, phospholipids, and medium-chain triglycerides (MCT) (Hitlamani et al., 2023). Likewise, the awareness of consumers about the health benefits of coconut oil has created value within the processing of food products.

The intermediate cities in Colombia strengthen their business fabric based on microenterprises. Market diversification is a post-pandemic business strategy that has proven to be effective in keeping microenterprises operationally functional. Microenterprises are defined as productive economic units in agro-industrial, commercial or service activities in rural or urban areas. Among the post-pandemic strategies for the reactivation and economic growth of microenterprises that have proven beneficial is support for innovation, development of technologies and products that allow companies to grow and position themselves nationally (Agudelo, 2021). Currently, its implementation is favored through the articulation Company-Academy-State under Law 2069, entrepreneurship law (Congreso de Colombia, 2020). In this research, a physicochemical characterization of two commercial fruit fillings, three fruit fillings elaborated in the production plant of a Caucana company from guava grown in their properties, as well as the coconut oil used for their manufacture was carried out in order to evaluate the compliance with the desired requirements for the elaboration of bakeable products such as filled cookies and to promote the generation of a new line of products.

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MATERIALS Y METHODS

The analysis of the physicochemical parameters of 5 samples of bakable fruit fillings was carried out: 3 samples of Guava filling (FRG1, FRG2 y FRG3) prepared in the company's production plant, following the general scheme shown in Figure 1, and two commercial samples of filling, one of Blackberry (RCZ), and one of Chocolate (RCCH). A sample of coconut oil used as an ingredient in the FRG1 filler formulation was also characterized.

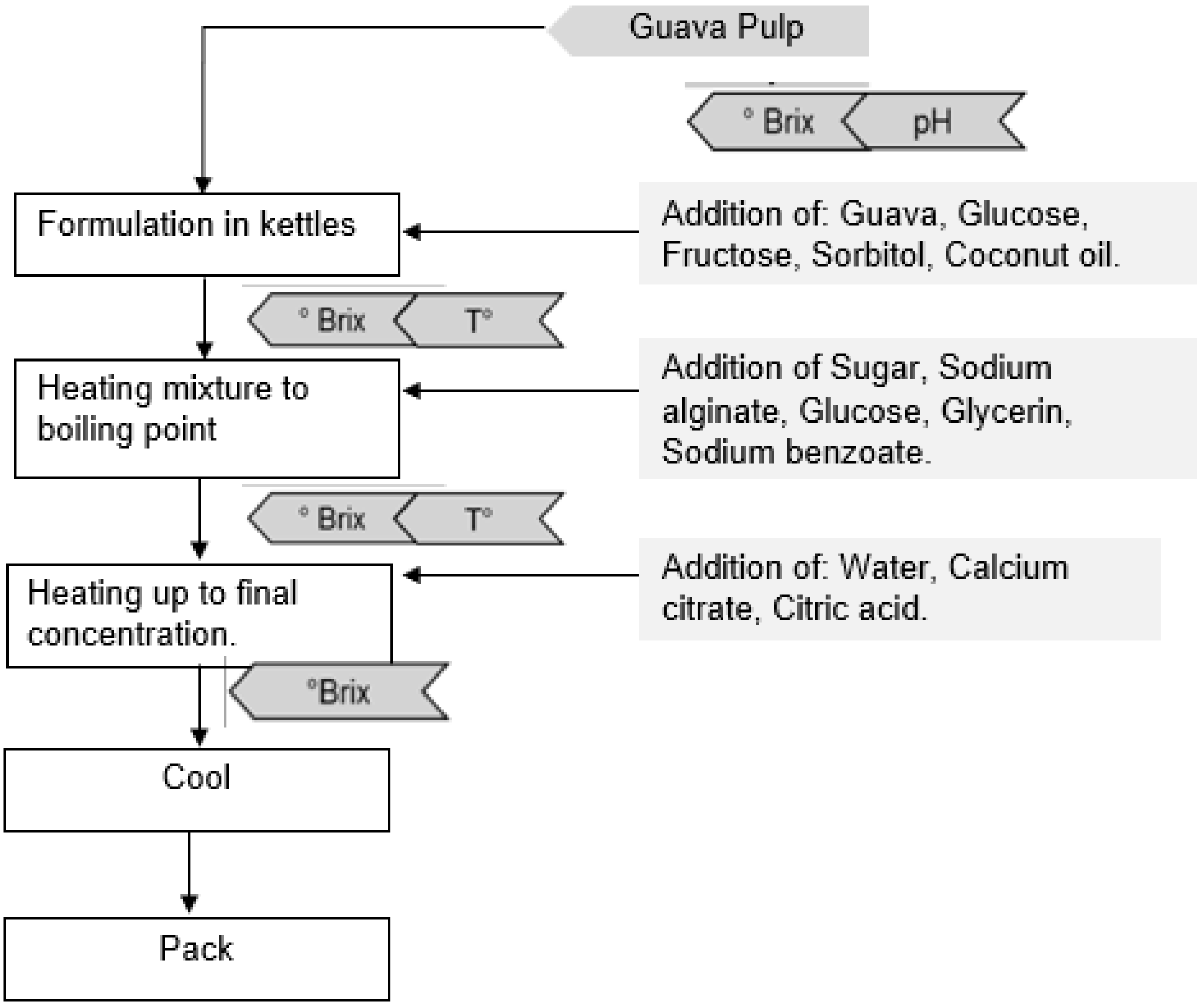

Figure 1. *General scheme of the formulation of guava fillings (Own source).*

The analyses of the fruit fillings were performed following the protocols reported in Table 1. Given the consistency of the samples and in order to estimate a routine control procedure for pH and percentage of soluble solids, an indirect methodology was also implemented based on a dilution of the sample or an alternative method by direct heating, respectively. All analyses were carried out in triplicate.

Table 1. Methodologies used for the analysis of physicochemical parameters of fruit fillings.

Parameter	Direct method	Indirect or alternative method
pH	AOAC 981,12. Sample mass ±5,0000 g	10,0000 g of sample diluted with hot distilled water. Shaking for 20 s.
Soluble solids (%)*	AOAC 932,12	2,5000 g of sample diluted with hot distilled water. Shaking for 20 s.
Humidity (%)	AOAC 934,01 Sample mass ±0,5000 a 3,0000 g	N. A
Total solids (%)	Hoyos et al. (2015) Sample mass ± 0,5000 g *t*=3h	Direct heating Sample mass ± 0,500 g *t*=3h
Ethereal extract (%)	AOAC 960,39 Solvent: ethyl ether *t*= 4h.	N. A
Ashes (%)	AOAC 942,05 Heating ramp. t_{FRG}=5h; t_{RCM}=4h; t_{RCCH}=10h;	N. A
Artificial colors	Hoyos et al. (2015) Sample mass ±10,0000 g l_{lana}=30 cm	N. A

*This parameter in the chocolate sample was only analyzed by the indirect method.

In coconut oil, the density, percentage of moisture, ash, crude protein, acidity index, percentage of free fatty acids, refractive index and saponification index were analyzed. These determinations, were carried out as described by Hoyos et al. (2015) in the Fats and Oils section. ANOVA analysis to compare some parameters of interest was performed with Statgraphics Centurion XVI software.

RESULTS AND DISCUSSION

Fruit fillings

In Table 2 the average values of the physicochemical parameters analyzed in the fruit filler samples by direct and indirect methodologies for pH and soluble solids, and sand and non-sand methods for total solids are shown.

Table 2. Physicochemical parameters of fruit fillers.

PARAMETER	METHOD	SAMPLES				
		FRG1	FRG2	FRG3	RCZ	RCCH
pH ± *s*	Direct	4,150±0,001 %CV=0,024	4,220±0,005 %CV=0,137	4,160±0,021 %CV=0,502	3,840±0,001 %CV=0,600	5,500±0,050 %CV=0,932
	Indirect	4,070±0,001 %CV=0,024	4,140±0,008 %CV=0,193	4,090±0,009 %CV=0,220	3,060±0,010 %CV=0,188	4,89±0,010 %CV=0,204
S.S* ± *s* (%)	Direct	78,80±0,05 %CV=0,01	77,000±0,17 %CV=0,23	75,90±0,001 %CV=0,024	79,600±0,000 %CV=0,000	N. A
	Indirect	65,300±0,01 %CV=0,01	60,600±2,36 %CV=3,901	65,330±0,67 %CV=1,068	65,900±0,000 %CV=0,000	70,600±0,10 %CV=0,142
Humidity ± s (%)		22,650±0,23 %CV=1,015	17,050±0,27 %CV=1,580	18,830±0,30 %CV=1,588	19,840±0,170 %CV=0,843	0,680±0,014 %CV=2,170
S.T ± s (%)	With Sand	86,680±0,31 %CV=0,352	87,190±0,69 %CV=0,779	89,260±1,03 %CV=1,155	89,110±1,060 %CV=1,119	99,440±0,50 %CV=0,502
	No Sand	89,360±0,47 %CV=0,528	87,190±0,54 %CV=0,618	88,910±0,51 %CV=0,569	82,390±0,30 %CV=0,361	99,300±0,36 %CV=0,365
Ethereal Extract ± *s* (%)		0,860±0,011 %CV=1,330	0,850±0,017 %CV=2,002	0,450±0,015 %CV=3,283	0,220±0,014 %CV=0,634	29,150±1,74 %CV=5,980
Ash ± *s* (%)		1,350±0,064 %CV=4,740	1,450±0,020 %CV=1,18	1,390±0,010 %CV=0,435	0,470±0,010 %CV=2,890	2,290±0,050 %CV=2,350
Dye		Absence	Absence	Absence	Absence	Presence

The soluble solids present in the samples were determined using a direct and an indirect method, finding significant differences between them ($p<0,05$) (see Table 3).

Table 3. ANOVA of the factors evaluated with a 95% confidence level.

Comparison factor	Parameter on which it is evaluated	Reason f	p value
pH			
Direct method - Indirect method	RCZ	685,05	0,0000
	RCCH	60,52	0,0000
FRG2-FRG3	Indirect Method	23,14	0,0086
Soluble solids			
Direct method - Indirect method	RCZ	13667,17	0,0000
FRG2-FRG3	Direct method	121,00	0,0004
Direct method - Indirect method	FRG2	143,57	0,0003
	FRG3	1245,48	0,0000
Humidity			
FRG2-FRG3	Humidity	58,87	0,0016

It should be noted that the chocolate filling could not be measured by the direct method, since it consists of an optical measurement and this filling is very opaque, so this parameter was only determined by the indirect method, which proved to be accurate (since its %CV was 0,142).

The parameters found in the samples can be contrasted with those reported by the technical data sheets of this type of products; however, the comparison is not made with those obtained by the indirect methods, since they are not reported under such conditions. The pH values obtained by the direct method for FRG1, FRG2 and FRG3, 4,15, 4,22 and 4,16 respectively, do not conform to the desired values for this type of products, a range of 3,2-4,0 (FAMESA 404, *s.f.*). In terms of soluble solids, only FRG2 yielded measurements in the desired range (77-83%), however, it would be advisable to increase the sugar content in this formulation, since it is just at the limit of what is acceptable.

The formulation of a thermostable filling requires consideration of the effect of variables such as the nature of the fruit (and the form in which it is added, for example, dehydrated, diced, etc.), the pH, the total soluble solids (°Brix) or the artificial colorant available; for FRG1, FRG2 and FRG3, which have a pH above the allowed one, the amounts of citric acid/calcium citrate pair should be regulated in the formulation, so as to improve the acidity parameter without significantly affecting the stability of the filling or the baking capacity which, as reported, can be influenced by hydrocolloidal stabilizers such as pectin or sodium alginate (Young, 2003).

The RCZ filler meets the parameters of pH (3,0-4,0) and soluble solids (rango de 77-83%) (FAMESA 425, *s.f.*) and differs greatly from those reported for sour cherry (43,38-44,86 °Brix) (Tesli, 2023). RCCH does not comply with the acidity range, which in this case is the same as RCZ, since both are commercial.

The content of total solids is important and a determinant of properties such as hardness; At values higher than 65 °BRIX microbial growth is inhibited, increasing the half-life of the product and generating an intermediate aqueous activity (Miquelim, 2011), in addition, it reduces the risk of syneresis, since it allows reaching an aqueous equilibrium between the filling phases and the baked dough, thus reducing the tendency for uncontrolled moisture migration, which can damage the quality of the product and increase its microbiological susceptibility (Tesli, 2023). At concentrations of 80°brix, a browning effect can be seen in the filling, due to the amount of sugar available for caramelization (Mahapatra, 2014).

The moisture percentages determined ranged between 17,050 and 19,840 for the guava and blackberry fillings, while that of the chocolate filling was less than 1% (see Table 2), consistent with the total solids content. Some physicochemical analyses carried out on bakable fruit fillings prepared from commercial recipes report moisture values of 20.50%, which is directly related to the microbiological, chemical and rheological stability of the filling, which tends to increase with the growth of simple sugars, since it has greater availability of hydroxyl groups that bind with water, and can fluctuate with storage time due to aqueous migration in the filling structure. However, acidic pH favor microbiological safety when the filler is subjected to a thermal process (Carcelli, 2022), so the durability of FRG1, FRG2, RCZ and RCCH fillers is favored by their acidity. However, sour cherry fillings report moisture contents from 48,36-52,62%; the wide ranges are due to the use of raw materials with different water contents, as well as the specific procedures used in the preparation of each fruit filling. This is a factor to take into account, since it has been shown that high baking temperatures can accelerate the rate of moisture loss in water vapor, so that the filling dries and shrinks, which can destroy the structure of the baking dough, and then having a favorable moisture content in the filling becomes a way to improve the quality of the product (Cauvain, 2010). According to the above, the chocolate-RCCH filler with a moisture content of 0,68% would present the lowest risk of forming cracks for this reason, when compared to the other guava fillers FRG2, FRG3 and blackberry-RCZ, which presented much higher moisture contents (17,05, 18,83 and 19,84% respectively). The results of total solids collected after determining the moisture of the 3 samples were recorded in Table 2. For these determinations, two methods were used, one of which required the use of calcined sand and the other did not.

Total solids are considered as the dry matter that remains after the removal of water in the structure, and it has been found that in biological matrices temperatures of up to 365°C can be reached for its elimination (Mauer, 2017), n Table 2 are expressed the values moisture (%) and total solids (%), being the sum of these parameters for FRG2 of 104,24%, for FRG3 of 108,09% and for RCZ of 108,95%; in these cases the value exceeds 100%, and this could be explained by the presence of water bound to the hydrophilic structures of the hydrocolloids such as sodium alginate that were added in the preparation of the filler.

In bread doughs have been found increases in water retention after adding sodium alginate after baking at 210°C during 30 minutes (Tabara, 2016), such effect could explain the overestimation in the dry matter value of the fillings.

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Facultad de Ingenierías y Arquitectura
Universidad de Pamplona

The contents of ethereal extract and ashes determined were higher than those reported for sour cherry fillings, ashes of ±0,30%, fats of 0,03% (Tesli, 2023), however, because there are differences in the type and quantity of raw material, preparation methodologies and different uses that are intended for the final product, these values only constitute a bibliographic reference but not a quality standard or a regulation on the nutritional composition of these products: in this case, a contribution is made to the literature on the physicochemical analysis of guava, blackberry and chocolate fillings.

The dye test is purely qualitative, and therefore, a coloration present or absent is determined. Therefore, Figure 2, in which the final results were observed, is presented.

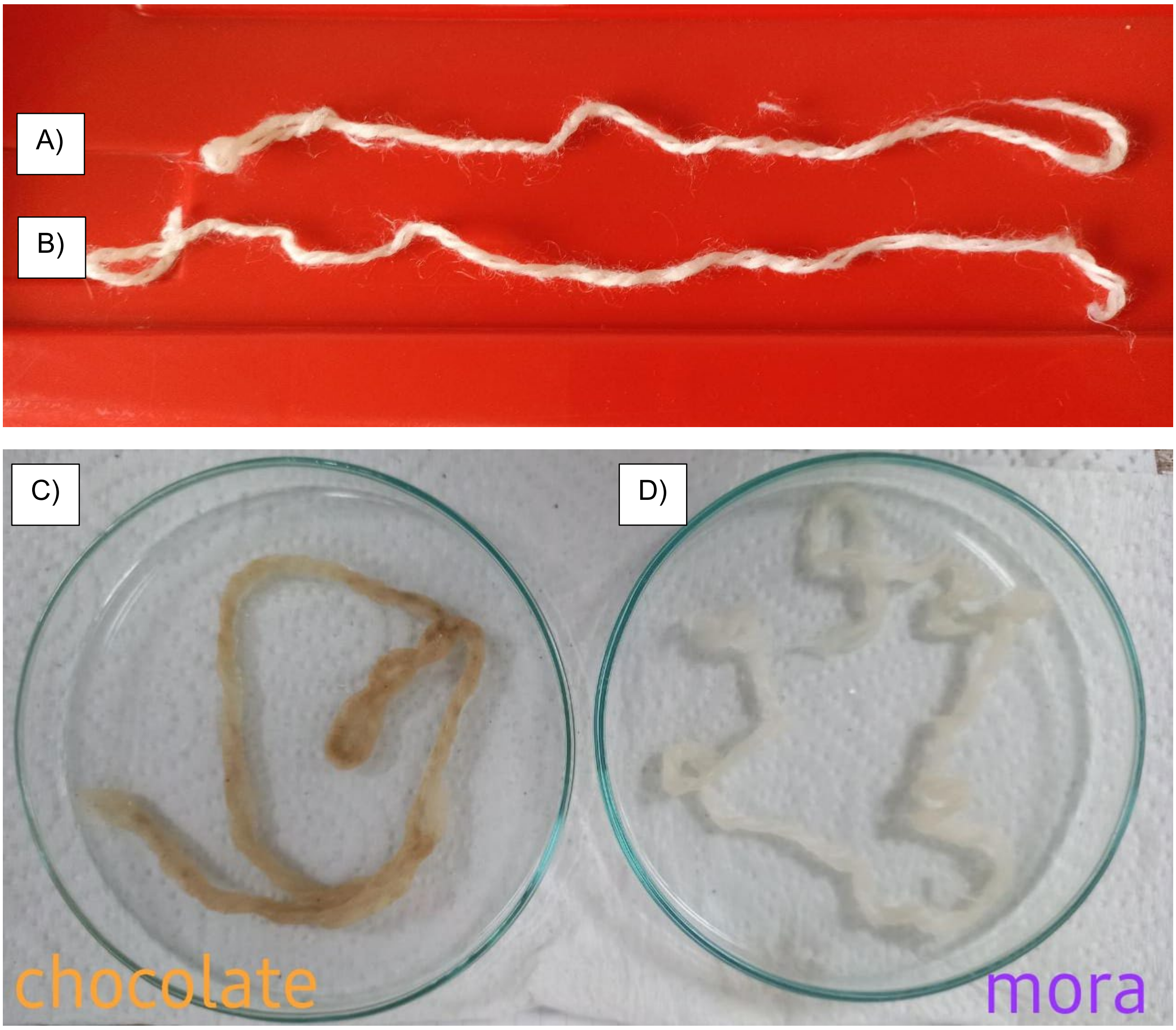

Figure 2. *Dye Testing. A) FRG2, B) FRG3, C) RCCH, D) RCZ (Own Source).*

In Figure 2 it can be observed that only the chocolate filling presented artificial colorants, however, according to the FAMESA technical data sheet, both the blackberry and guava fillings FRG2 and FRG3 present carmine colorant; in this case, it is possible that the sensitivity of the method is not sufficient to detect the colorant, so it is recommended to use a more sensitive and adequate analysis technique such as HPLC or Uv-Vis. In the FRG1, FRG2 and FRG3 fillings, the presence of colorants was not expected according to their processing (see Figure 1).

Coconut oil

Different physicochemical analyses were performed on the coconut oil with which the formulation was made to prepare FRG1, the results were reported in Table 4.

Table 4. Physicochemical results of coconut oil.

Parameter	Value found
Humidity	0,0960±0,0004% CV=4,579
Density	0,9020±0,023g/cm^3 CV=2,537
Crude protein	0,011±0,000% CV=0,0
Ash	0,0097±0,0003% CV=2,790
% Free fatty acids	0,0470±0,0001% CV=2,183
Saponification index	259,38±2,028 meq KOH/g CV=0,782

Codex Alimentarius Stan 210 (1999), establishes that the maximum tolerable moisture and volatile matter in refined coconut oil is 0,2 %, and, considering that after 3 h the weight remained constant and the mean was 0,0960%, the sample meets the technical standard in this case.

The density of coconut oil according to NTC 252 (ICONTEC, 2015) is in the range of 0,917-0,919 g/mL, therefore the value of 0,902 g/mL is below the accepted values, however, considering that density is a parameter that can vary with factors such as age, rancidity or treatments to which the oil is subjected, according to Bernal de Ramírez (1993), this change is not very significant nor necessarily indicative of any problem with the quality of the oil.

The crude protein content reported for crude oil varies from 0,53-9,26% depending on the extraction methodology (David-Jacob *et al.,* 2020), and considering that the oil is refined, a much lower value is expected so 0,011% is an acceptable value (James *et al*., 2020).

Now then, it was proceeded to determine the acidity index and the percentage of free fatty acids present in the coconut oil sample. The results obtained were recorded in Table 4. The maximum acceptable acidity expressed as lauric acid is 0,2%; the value of 0,0470% is an indicator of the low acidity present in the sample and also complies with NTC 252 (ICONTEC, 2015).
Finally, it was determined the Saponification Index of the coconut sample, and the results

of the determination were recorded in Table 4.

According to NTC 252, the saponification index of coconut oil should be between 250-264 mgKOH/g, so that 259,38 mgKOH/g complies with the Colombian Technical Standard.

Considering some previous studies carried out by the research group (unpublished data) of refined coconut oils, the values reported are close, so the coconut oil used is of good quality.

CONCLUSIONS

The guava fillings formulated as FRG1, FRG2, FRG3 and chocolate filling (RCCH) did not comply with the pH ranges established in the FAMESA technical data sheet, so it is necessary to increase the acidity of these fillings, and one option is to add a greater amount of citric acid/calcium citrate. In terms of soluble solids only the guava filling with FRG2 formulation was in the desired range, in the rest of the formulations the sugar content should be increased, but without affecting baking quality parameters such as syneresis or baking index. The blackberry filling (RZC) complied with the pH and soluble solids parameters of its data sheet.

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