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Food Hydrocolloids from Butternut Squash (*Cucurbita moschata*) Peel: Rheological Properties and Their Use in *Carica papaya* Jam

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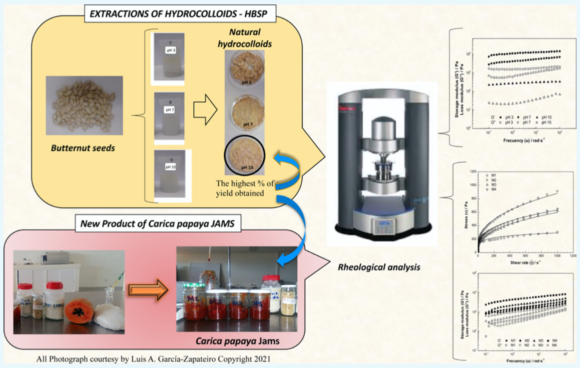
ABSTRACT: Hydrocolloids are a class of functional ingredients that are widely used in the development of food structures. The hydrocolloids are mainly polysaccharides and some proteins that are applied in various food products. For this reason, natural sources that are friendly to the environment must be sought for their extraction. Therefore, this study aimed to extract hydrocolloids from butternut squash (*Cucurbita moschata*) peels—HBSP—and determine the proximal composition and rheological properties as well as their use effect in a microstructure product like fruit jam from *Carica papaya*. Hydrocolloids were obtained from butternut squash at pH 3, 7, and 10 and at different temperatures, presenting higher yield values at 80 °C with higher carbohydrate and protein contents and non-Newtonian flow behavior type shear-thinning. In order to analyze the influence of HBSP on the rheological properties of the microstructured product, the samples were employed as a partial substitute of pectin in *C. papaya* jam (CPJ), showing a positive effect on the jam matrix due to the addition of hydrocolloids. The physicochemical properties of jams did not present significant differences. CPJ presents non-Newtonian behavior type shear-thinning adjusting to the Herschel–Bulkley model. The dynamic viscoelastic rheological test characterized the jam as a gel-like state when the storage modulus values were higher than the loss modulus values in the frequency ranges studied. Regarding the addition of HBSP, this modified the color parameter, presenting a reddish color with an increase in tonality, and the sensory evaluation showed that the M3 sample was better than the other products, with a higher level of satisfaction. The obtained results show that butternut squash peel is suitable for the obtention of hydrocolloids, and they can be used as a raw material in the development and formulation of food products, as well as their byproducts can be used to solve problems with organic waste from the agroindustry in an environmentally friendly way.



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■ INTRODUCTION

Hydrocolloids are macromolecules with high molecular weight and can be dispersed or dissolved in water under appropriate conditions and employed as emulsifiers, thickeners, texture modifiers, and stabilizers in order to improve the physicochemical, rheological, and microstructural properties of food products.[1,2] Large, linear, and flexible polysaccharides increase viscosity even at low concentrations. This property allows hydrocolloids to be the major ingredient in liquid and semisolid-type foods.

Today, there is much interest in the use of natural ingredients in the food industry. The demand for hydrocolloids,[3] for example, pectin, starch, carrageenan, galactomannan, alginate, cellulose and its derivatives, and extraction hydrocolloids from plants and animals, has increased. However, plant-based additives are of most interest because of their friendly image in the eyes of consumers and because they are low cost, safe, and biodegradable. Therefore, they are ideal as suspending agents, gelling agents, encapsulating agents, viscosity modifiers, and sugar crystal growth inhibitors for reducing syneresis and for flavor release control when added to products.[4]

Butternut squash (*Cucurbita moschata*) is considered a healthy and functional vegetable because it is rich in flavonoids, phenols, carbohydrates, α-tocopherol, β-carotene, minerals, and amino acids.[5−7] Some studies have demonstrated that butternut squash peel (BSP) presents a high percentage of carotenoids, provitamin A, and pectin;[8] nevertheless, they are usually discarded as an agricultural byproduct for feed, becoming an important raw material from the industrial foods and scientific points of view. Accordingly, the beneficial

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effects of these carotenoids and polysaccharide contents in the substantial pumpkin byproducts have attracted great interest from researchers and manufacturers, making them of interest for the obtention of hydrocolloids.

Microstructured products like jams and jellies make fruits available for consumption out of season.[9] Jam is a food prepared by boiling fruit pulp with sugar and other ingredients such as pectin, acid, preservative, flavoring agent, and coloring agent;[10] the amount and type of sugar added, acidity, proportion, and the kind of pectin used affect its rheological and microstructural properties. Jam has a three-dimensional network of pectin in the pulp−sucrose−acid system with certain degrees of elasticity, involving a combination of noncovalent contributions such as hydrophobic and hydrogen-bonding interactions.[11−13] The use of natural additives and ingredients is a current trend in the food industry, with consumers showing an increasing interest in natural food and ingredients.[14] This study aims to utilize the waste BSP for the obtention of hydrocolloids and evaluate their proximal composition and rheological properties and their use in the design, standardization, and characterization of the new jam-type product from *Carica papaya*, taking into account that it is a fruit with a high nutritional value, is a source of vitamins A, C, and E, minerals, carotenoids, phenolic compounds, fiber, and folate, and is used traditionally by many countries due to its nutritional value and health benefits.[15]

■ RESULTS AND DISCUSSION

Extraction Properties of HBSP. The extraction yield (g hydrocolloid/g dried BSP × 100) of the hydrocolloids from butternut squash peel (HBSP) (Table 1) has showed different values depending on the pH and extraction temperature, from 0.87 to 4.01%.

Table 1. Extraction Conditions and Yield of Hydrocolloids from Squash Peel[a]

no.	temp.	pH	yield (%)
1	30	3	1.74 ± 0.36^{a}
2	30	7	0.87 ± 0.12^{a}
3	30	10	1.95 ± 0.23^{a}
4	55	3	1.97 ± 0.23^{ab}
5	55	7	1.36 ± 0.25^{ab}
6	55	10	3.56 ± 0.32^{ab}
7	80	3	2.33 ± 0.23^{b}
8	80	7	1.93 ± 0.65^{b}
9	80	10	4.01 ± 0.32^{b}

[a] Values with different letters (a, b) in the column differ significantly ($p < 0.05$).

Therefore, the extraction was performed at 1:10 peel/water ratio, regarding the proportion of peel in water, resulting in good relation with the transfer of mass and energy to the product and favoring greater extraction of polysaccharides.[16]

An increase in the temperature during extraction presents a significant growth of hydrocolloid extraction ($p < 0.05$), showing a linear correlation ($R^2 > 0.93$) for all pH (Figure 1). At 30 °C, the extraction presents the lowest values, while 80 °C presents the highest yield extraction value.

Then, in all cases, a significant variation in the yield values ($p < 0.05$) was observed, and the lowest values were at pH 3 and the highest at pH 10. The slope values increase with pH. However, the intercept was higher at pH 3 and the lowest at pH 7; but the yield values at pH 10 were higher than others in the studied temperatures. These results were in line with Lin and Lai[17] when alkaline conditions were employed to obtain higher hydrocolloid extraction yield.

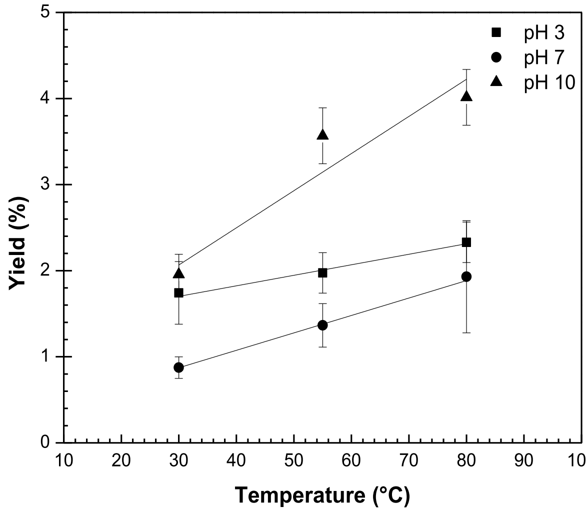

Figure 1. Extraction yield of hydrocolloids from the squash (*Cucurbita moschata*) peel.

Proximal Composition and Physicochemical Properties of HBSP. Considering the higher extraction yields, the analysis of proximal composition and physicochemical properties of hydrocolloids obtained at 80 °C was performed, and this result is shown in Table 2. The pH of extraction presented a high relation with their proximal composition when an increase in moisture was observed with the increase in pH, presenting values between 4.32 and 12.07% similar to the report which used other gums such as xanthan (10.20%) and guar (8.60%)[18] and lower than that of Arabic gum (15%).[19] The total ash content of hydrocolloids was between 5.41 and 9.02%, where the samples extracted at pH 3 and 10 presented the highest values and that at pH 7 presented the lowest; this parameter is indicative of physiological minerals,[20] which indicates that the HBSP obtained in alkaline and acid mediums could present the highest amount of sodium, potassium, iron, calcium, or magnesium, the principal minerals identified in the peel of squash.[21] The amount of protein was similar in all case studies, presenting values of 16.32 for pH 3, 18.09 for pH 7, and 18.89 for pH 10. These results obtained are higher than those for guar gum (8.20%),[18] fenugreek seed (7.24%),[22] and xanthan gum (5.40%) and the lowest− for Arabic (2.31%) gum.[19]

The amount of carbohydrate from the peel was the highest at alkaline conditions with values of 64.18 ± 0.32% and the lowest at neutral conditions, which indicated that the purity of HBSP was relatively high, mainly consisting of saccharides. These values are similar to the total carbohydrate contents reported in the literature for sage seed gum (69.96−71.05%)[23] and basil seed gum (70.63%).[24] In the same way, the relation with the total sugar was similar when the percentage at pH 10 was 41.39 ± 0.12%, and at pH 7, it was 27.47 ± 0.53%. The result of the proteins obtained for all samples was similar, being 16.32 ± 0.04%, 18.09 ± 0.10%, and 18.90 ± 0.09%. The highest values of these percentages can be attributed to the pectin type found in the cell walls of vegetables.[25]

The pH 10 was the treatment with the better condition for isolating hydrocolloids due to the higher content of carbohydrate (64.17 ± 0.31%) and the good relationship

Table 2. Proximal Composition of Hydrocolloids from the Squash Peel at 80 °C[a]

pH	moisture	ash[b]	protein[b]	fat[b]	carbohydrate[c]	total sugar[c]
pH 3	4.32 ± 0.01^{bc}	9.02 ± 0.02^{c}	16.32 ± 0.04^{b}	5.62 ± 0.10^{c}	38.22 ± 0.39^{a}	34.95 ± 0.11^{a}
pH 7	9.08 ± 0.20^{bc}	5.41 ± 0.88^{c}	18.09 ± 0.10^{b}	2.18 ± 0.26^{c}	30.52 ± 0.52^{a}	27.47 ± 0.52^{a}
pH 10	12.07 ± 0.10^{bc}	8.22 ± 0.80^{c}	18.89 ± 0.09^{b}	6.45 ± 0.01^{c}	64.17 ± 0.31^{a}	41.39 ± 0.11^{a}

[a] Values with different letters (a–c) in the column differ significantly ($p < 0.05$). [b] Expressed in (% db) dry basis. [c] Expressed in (% wb) weight basis.

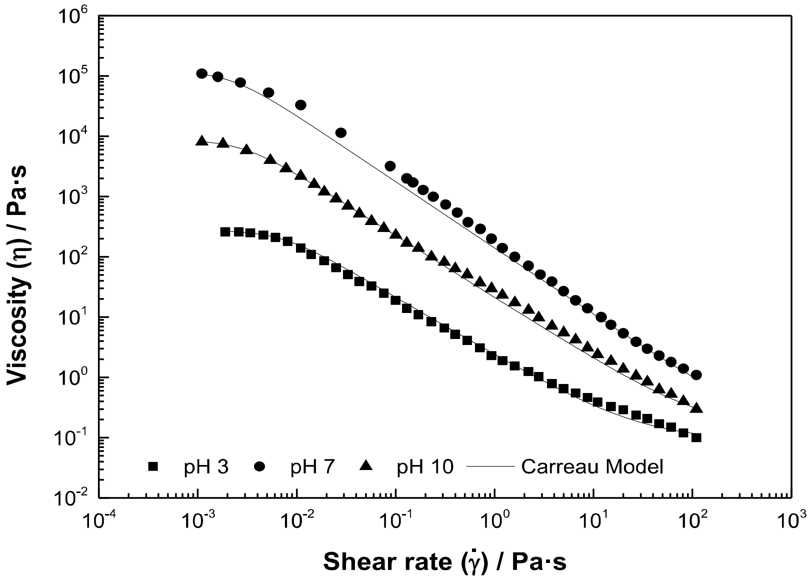

Figure 2. Viscous curve of hydrocolloids at 80 °C and pH 3, 7, and 10.

Table 3. Rheological Properties of Hydrocolloids from the Peel of Squash at 80 °C[a]

pH	η_o	η_∞	λ	s	R^2
pH 3	276.855 ± 4.70^{a}	0.09 ± 0.01^{b}	143.51 ± 1.13^{a}	0.48 ± 0.03^{a}	0.99
pH 7	122349.41 ± 3.86^{a}	0.15 ± 0.04^{b}	470.63 ± 1.13^{a}	$0.55 \pm 0,14^{a}$	0.99
pH 10	8763.88 ± 41.27^{a}	$0.13 \pm 0,06^{b}$	350.28 ± 6.77^{a}	0.52 ± 0.01^{a}	0.99

[a] Values with different letters (a, b) in the column differ significantly ($p < 0.05$).

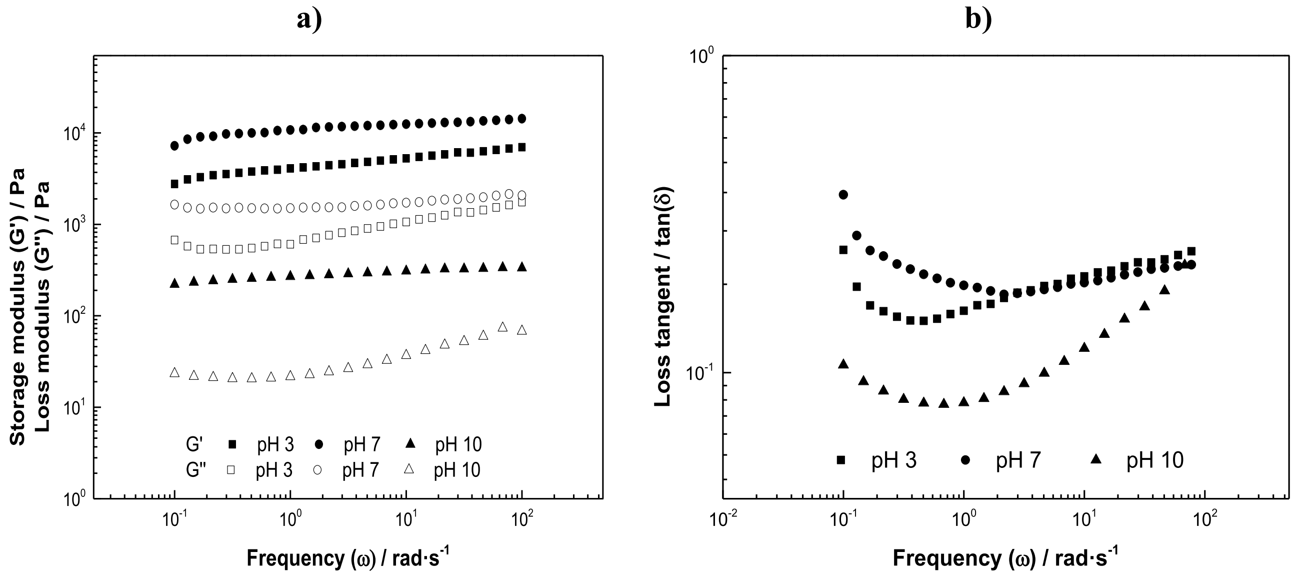

Figure 3. Dynamic rheology properties of hydrocolloids at 25 °C in the linear viscoelastic region. (a) Storage and loss modulii; (b) loss tangent.

with protein (18.89 ± 0.09%). The higher amounts of protein and carbohydrates are related to the technological properties of hydrocolloids. HBSP is considered a good alternative as an ingredient for their use and improves the rheological properties of food products due to the fact that the mixture of protein and polysaccharide molecules can associate either by covalent

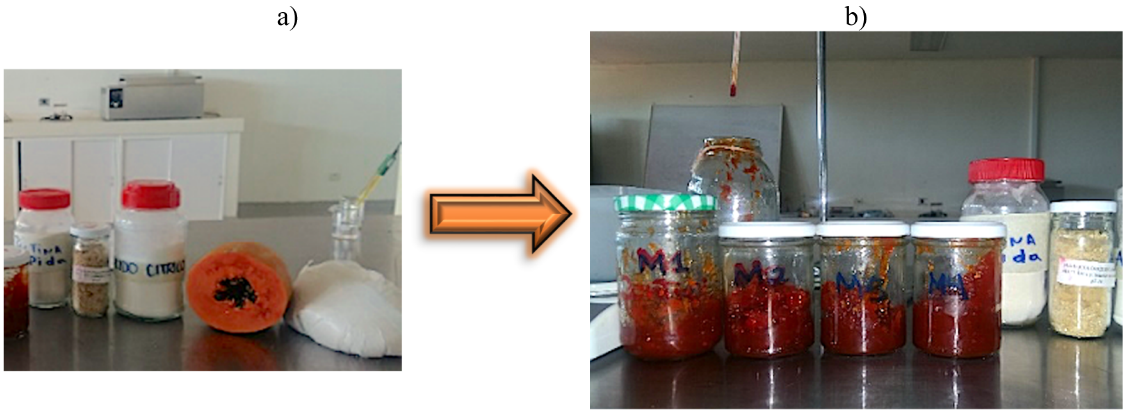

Figure 4. (a) Papaya Carica and HBSP and (b) four samples of CPJ. Photograph courtesy Luis A. García-Zapatiero Copyright 2021.

interactions, giving rise to strong and specific biopolymers,[26] or by noncovalent bonds through electrostatic, hydrophobic interactions, steric exclusion, and hydrogen bonding.[27]

Rheological Characterization of HBSP. Figure 2 shows the experimental steady-shear viscous curves of HBSP obtained at 80 °C and different pH at 3, 7, and 10. Samples revealed strong shear thinning behavior with a variation in viscosity (η) with 10 orders of magnitude. The Carreau model was used to describe the viscosity and the shear rate relationship (eq 1)

$$\eta = \eta_\infty + \frac{\eta_o - \eta_\infty}{[1 + (\lambda\dot{\gamma})^2]^s} \tag{1}$$

where η_o is the zero shear rate viscosity, η_∞ is the infinite shear rate viscosity, λ is the time constant of Carreau, and s is the behavior index of power law, respectively.

Table 3 shows the adjusting parameters. The rheological behavior was similar for the three samples extracted: sample pH 7 presented the highest values of η_o, η_∞, λ, and s, followed by the samples at pH 10 and pH 3. The shear-thinning behavior makes the hydrocolloids from squash peel a potential ingredient in developing food products because their characteristics include desirable attributes in providing satisfaction to the consumer.

The frequency sweep of hydrocolloids obtained at 80 °C under different pH is shown in Figure 3a. The data showed that the storage modulus (G') stays higher than the loss modulus (G''), so the elastic response system was higher than the viscous response over the whole range of frequencies studied, which corresponds closely to that of a gel increase with the increased frequency.

In addition, the hydrocolloids showed mainly elastic behavior. This was observed for basil seed gum and hydrocolloids obtained from *Ocimum basilicum* L. seeds,[28] described by a strong association of cross-linked molecules within the diluted gum solution network.[29] Figure 3b is shown as the tangent of the phase ($\tan(\delta) = G''/G'$); similar $\tan(\delta)$ was obtained with values between 0.222 and 0.253, which suggests a concentrated amorphous polymer rather than a gel.[30] The dynamic analysis can give information about the time or frequency dependence and the structural behavior of components. The obtained results are essential to predict the material behavior through processing manipulation.[31]

Design and Proximal Composition of *C. papaya* Jam. HBSP extracted at 80 °C and pH 10 was chosen for the design and standardization of microstructured products of *C. papaya* jam (CPJ) due to its higher content of carbohydrate and protein, so that they presented a defined gel structure. The raw materials and four samples of CPJ are shown in Figure 4a,b, respectively.

Four jams were obtained (Table 4) using 0.5% of pectin (M1) and replacing 25, 50, and 75% of the pectin percentage with hydrocolloids (sample M2, M3, and M4) in order to obtain a jam with similar consistency, employing an alternative to pectin and considering that hydrocolloids can modify the rheological and sensorial properties of food products[32] and their nutritional quality and physical aspects.

Table 4. Formulation of CPJs

jams	gelling agent
M1	pectin 0.5%
M2	pectin 0.375% and HBSP 0.125%
M3	pectin 0.250% and HBSP 0.250%
M4	pectin 0.125% and HBSP 0.375%

All CPJ products obtained were stable—the syneresis phenomenon did not occur in any of the samples. The proximal composition of jams is presented in Table 5. All samples were prepared with the total soluble solid content as commercial products.[33] In this case, the samples show a slight increase in pH, with values ranging between 2.99 and 3.77, but there was a decrease in acidity with the addition of HBSP. This parameter is one of the most important factors in the jam process, which should be monitored and controlled. Indeed, acidity is an imperative factor influencing pectin gelation, texture, and the overall quality of fruit jams.[34]

All samples present similar values, but not a significant difference (Table 5), in the content of moisture (±45%), carbohydrate (±57%), ash (±2%), and protein (±0.46%). In the case of fat, a significant decrease was observed with the increase in the percentage of hydrocolloids, suggesting hydrocolloids as a possible additive employed as a fat substitute in food products.

Rheological Properties of CPJ. The rheological properties of the steady-state shear rate of CPJ establish a quality control method for product development and processing calculations.[35] Figure 5a shows a decrease in the apparent viscosity with the increase of shear rate. The effect of HBSP on the apparent viscosity of jams on steady-state rheological behavior was observed, where a small shear rate and η exhibited a Newtonian plateau followed by a shear-thinning zone. In this region, a Newtonian plateau region was increased,

Table 5. Proximal Composition and Physicochemical Properties of CPJ[a]

jams	pH	°Brix	acidity[c]	moisture (%)	ash[b] (%)	fat[b] (%)	carbohydrate (%)	protein (%)
M1	2.99 ± 0.01^{a}	64.00 ± 0.01^{b}	0.42 ± 0.01^{c}	45.67 ± 0.02^{d}	2.40 ± 0.09^{a}	6.45 ± 0.02^{a}	56.54 ± 3.00^{e}	0.46 ± 0.01^{c}
M2	3.32 ± 0.01^{a}	63.87 ± 0.01^{b}	0.54 ± 0.03^{c}	44.44 ± 0.97^{d}	2.15 ± 0.02^{a}	3.71 ± 1.84^{a}	57.89 ± 0.40^{e}	0.41 ± 0.01^{c}
M3	3.77 ± 0.01^{a}	67.00 ± 0.01^{b}	0.41 ± 0.04^{c}	44.62 ± 0.64^{d}	2.21 ± 0.02^{a}	2.81 ± 0.96^{a}	54.20 ± 0.31^{e}	0.43 ± 0.01^{c}
M4	3.27 ± 0.01^{a}	64.66 ± 0.01^{b}	0.31 ± 0.02^{c}	45.28 ± 0.21^{d}	2.75 ± 0.06^{a}	1.43 ± 0.33^{a}	57.50 ± 0.20^{e}	0.43 ± 0.01^{c}

[a] Values with different letters differ significantly ($p < 0.05$). [b] Expressed in (%db) dry basis. [c] Expressed in percentage of citric acid.

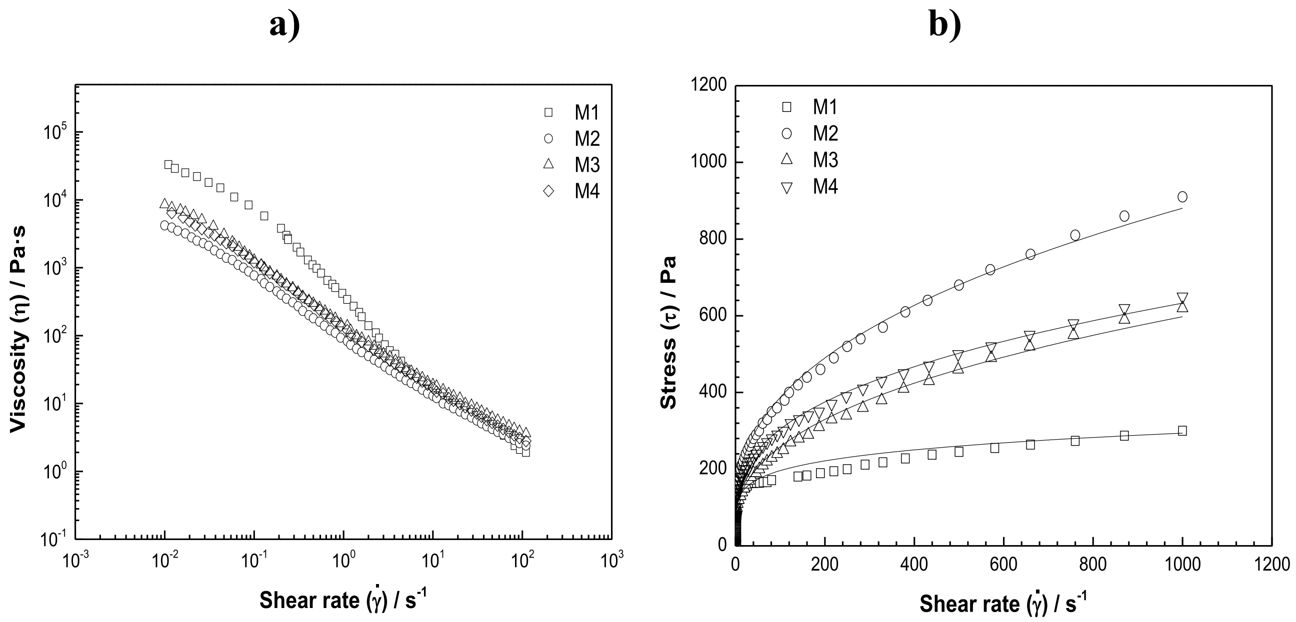

Figure 5. Influence of shear rate on the (a) flow curve and (b) viscosity curve of CPJ.

Table 6. Rheological Properties of CPJ at 25 °C[a]

	Herschel–Bulkley parameters					viscoelastic properties at 1 rad·s^{-1}				
sample code	τ_o	k (Pa·s^n)	n	R^2	G'_o (Pa·s^n)	n	G'' (Pa)	η^* (Pa·s)	tan δ	G^* (Pa)
M1	1.39 ± 0.06^{a}	86.53 ± 4.37^{a}	0.17 ± 0.03^{b}	0.90	4189.45^{c}	0.15^{a}	970.3^{c}	4439^{b}	0.222^{a}	4439^{b}
M2	73.53 ± 5.60^{a}	47.26 ± 4.64^{a}	0.41 ± 0.01^{b}	0.97	2082.04^{b}	0.18^{a}	556.1^{ab}	2284^{a}	0.251^{a}	2284^{a}
M3	42.92 ± 3.02^{a}	$32.76 \pm 2\text{-}49^{a}$	0.41 ± 0.01^{b}	0.98	1193.66^{a}	0.19^{b}	266.4^{a}	1222^{a}	0.223^{a}	1222^{a}
M4	68.19 ± 5.86^{a}	41.19 ± 5.92^{a}	0.37 ± 0.01^{b}	0.95	1453.08^{a}	0.19^{b}	378.2^{ab}	1541^{a}	0.253^{a}	1541^{a}

[a] Values with different letters (a–c) in the column differ significantly ($p < 0.05$).

considering the pectin percentage quantified by the constant viscosity at a low shear rate. The sample without the addition of HBSP presents a high initial value of viscosity at a lower strain rate than the others. At 1 s^{-1}, the apparent viscosity decreases from 340 to 83 Pa·s between M1 and M2, respectively. At a constant low shear rate, viscosity values were obtained showing a Newtonian plateau region, followed by a potential drop, identified as the shear-thinning region and infinite viscosity at a high shear rate. This behavior is explained by the increase in the alignment of the molecules that constitute it as HBSP, sugar, and pectin and the hydrodynamic force generated.[36]

The flow behavior of CPJ is shown in Figure 5b; the shear stress increases with the shear rate. The flow of jams presents yield stress, indicating an interactive structure that must be broken down before flow. The experimental data were fitted employing the Herschel–Bulkley model with an R^2 range value between 0.90 and 0.98 (eq 2)

$$\tau = \tau_o + k\dot{\gamma}^n \tag{2}$$

where τ_o is the yield stress, k is the consistency coefficient, and n is the flow behavior index; the results are presented in Table 6. Adjusting the experimental data to eq 2 could determine the non-Newtonian behavior. The k value obtained was 47.26, 32.76, and 41.19 Pa·s^n for M2, M3, and M4, respectively. A higher value of k was shown as a more consistent jam,[37] and these values were bigger than reported by Sagdic et al.[38] for a rosehip jam and within the range shown for peach jam.[39] The n values were less than 1 in all cases, indicating that all samples present shear thinning behavior. The addition of HBSP increases the n values (Table 6) which means an increase in pseudoplasticity.

The yield stress (τ_o) is the amount of energy necessary to initiate flow, while the energy needed to maintain flow is expressed by the plastic viscosity; this parameter is important for industrial handling, storage, processing, and transport of concentrated suspension.[37] Afterward, the addition of HBSP led to the obtention of a product with a higher yield stress and consistency index, which showed a passing from solid to liquid state behavior, so it has been shown that a structural network

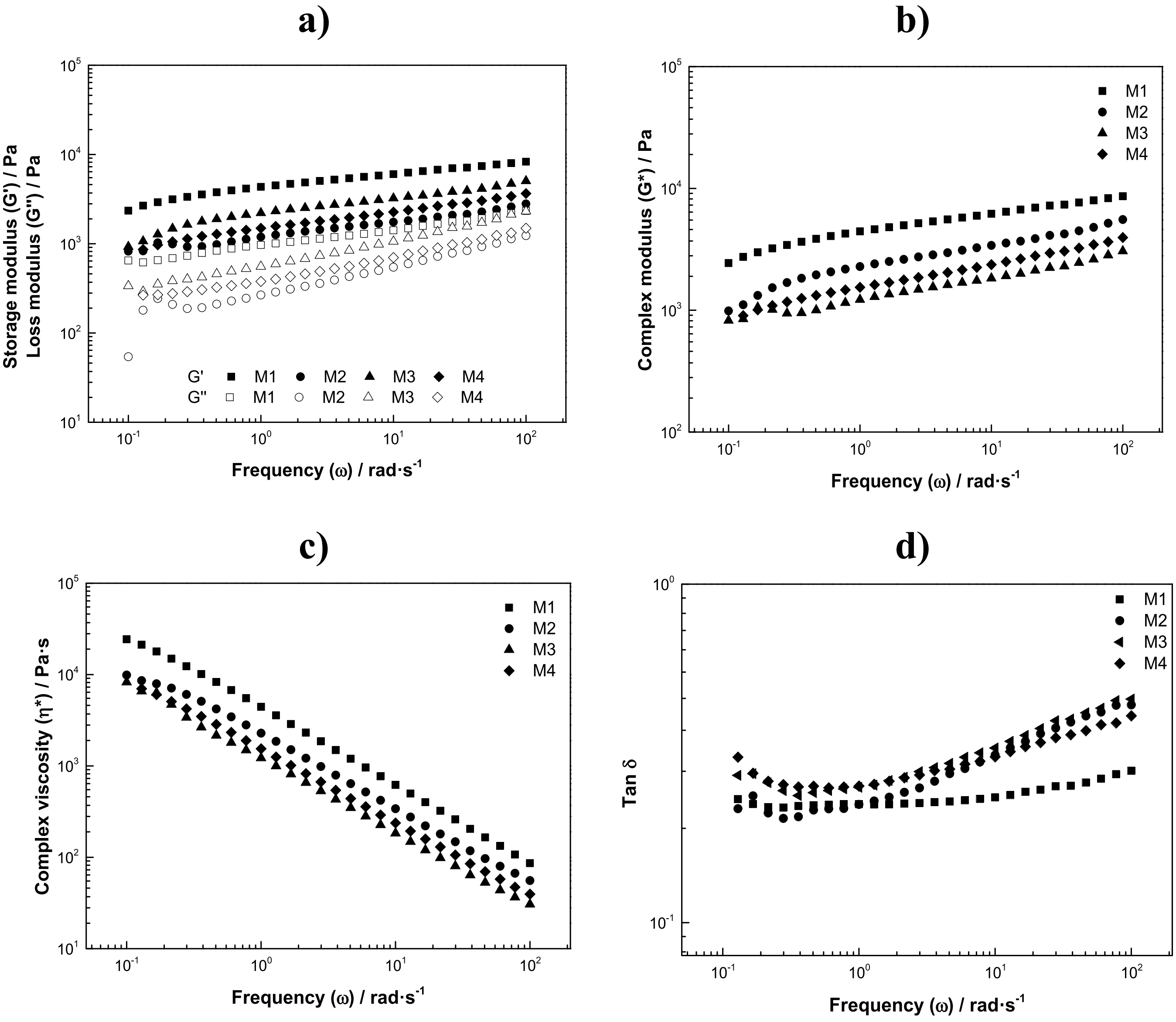

Figure 6. Mechanical spectra obtained in the dynamic oscillatory test of CPJ. (a) Storage and loss modulus, (b) complex modulus, (c) complex viscosity, and (d) loss tangent.

exists in liquid jams that needs to be disrupted before flow can take place due to the interactions between the solid particles. As the content of hydrocolloids dispersed in a fluid increased, so too did the rheological properties. This behavior was reinforced by the higher content of carbohydrates and proteins in HBSP employed, which favored the formation of junction zones between pulps and pectin chains.[40] The addition of HBSP led to obtaining jams with higher yield stress and pseudoplasticity parameters and a more moderate consistency than the samples prepared with pectin.

Dynamic Rheology Properties of CPJ. CPJs were analyzed in order to determinate their viscoelastic properties as functions of G', G'', complex viscosity (η^*), complex modulus (G^*), and loss tangent (tan δ) in the linear viscoelastic regime (LVR). G' and G'' moduli present a frequency dependence, showing $G' > G''$ (Figure 6a) describing jam as a product that has predominantly solid behavior.[41] This behavior can be attributed to the polysaccharides and the chemical structure present in the pulp.[42] The gel behavior has been reported for gabibora jam[40] and apple jelly.[43] Viscoelastic properties of CPJ are reported in Table 6. The experimental data of G' (Figure 6b) as the function of ω was fitted with the potential function (eq 3)

$$G' = G'_o \cdot \omega^n \qquad (3)$$

where G'_o is the predicted value of G' at $\omega = 1$ rad·s^{-1} and n is the slope of log G' versus log ω, showing R^2 values between 0.94 and 0.99. G'_o increases with the percentage of hydrocolloid added, while n decreases. M2, M3, and M4 present values of G'_o less than control samples but with the greatest value of n. G^* of CPJ with different percentages of hydrocolloids as a substitute of pectin, when the jam sample structure declines with the amount of pectin.

η^* (Figure 6c) decreases as a function of frequency; the sample's mechanical spectra correspond to the entangled macromolecules of solutions,[30] and then this parameter is related to the global viscoelastic response. η^* covers the elastic and viscous properties of CPJ under no-flow conditions and was found to be very high at the low angular frequency, if

Table 7. Effect of HBSP on Redness (a^*), Yellowness (b^*), Lightness (L^*), Chroma (C^*), Hue Angle (Hue), and Color Change (ΔE)[a]

sample code	L^*	a^*	b^*	C^*	Hue	ΔE
M1	42.73 ± 1.24^d	48.40 ± 1.43^b	37.27 ± 1.39^d	61.09 ± 1.85^b	1.03 ± 0.04^a	
M2	34.19 ± 0.23^c	49.45 ± 0.76^b	33.05 ± 1.02^c	59.48 ± 1.17^b	1.27 ± 0.03^b	46.47 ± 2.36^a
M3	28.09 ± 3.26^b	41.39 ± 4.23^{ab}	27.75 ± 0.76^b	49.59 ± 3.57^a	1.29 ± 0.19^b	191.79 ± 79.35^b
M4	23.72 ± 2.20^a	38.02 ± 1.09^a	21.38 ± 0.99^a	43.62 ± 1.22^a	1.59 ± 0.09^c	363.25 ± 49.84^c

[a] Values with different letters (a−c) in the column differ significantly ($p < 0.05$).

Table 8. Sensorial Analysis of CPJ Products[a]

CPJ	color	odor	flavor	consistency	general acceptability
M1	3.71 ± 0.21^a	3.33 ± 0.15^a	3.53 ± 0.16^a	3.71 ± 0.17^a	3.58 ± 0.16^a
M2	3.60 ± 0.10^a	3.37 ± 0.15^a	3.50 ± 0.05^a	3.84 ± 0.06^b	3.58 ± 0.05^a
M3	4.01 ± 0.10^b	3.50 ± 0.26^a	3.75 ± 0.07^a	4.05 ± 0.09^{bc}	3.83 ± 0.04^b
M4	3.90 ± 0.10^b	3.27 ± 0.06^a	3.67 ± 0.12^a	3.86 ± 0.08^b	3.70 ± 0.18^b

[a] Values with different letters (a−c) in the column and row differ significantly ($p < 0.05$).

compared with the viscosity of water, and decreased with increasing angular frequency, corroborating that all samples present pseudoplastic behavior, as reported for fruit jams.[41,44]

In order to analyze the values of G^* and η^*, a frequency values of 1 rad·s^{-1} was chosen as representative of the viscous modulus and complex viscosity of each sample (Table 6). An increase in hydrocolloid did not influence the values of tan δ and decreased the G^* values, confirming the sample's viscosolid behavior. Tan δ (Figure 6d) values were low, indicating that CPJ has more solidlike behavior.[45] The values were closer, although M1 presents the lowest values when the values of M2, M3, and M4 are similar. This shows that the samples with hydrocolloids have similar viscoelastic properties, and there is no significant change in the bonding involved in the formation of the gel structure.

Instrumental Color Analysis of CPJ. Appearance is a crucial consideration for food products; color is vital for receptive analysis. The measurements of chroma (C^*), hue angle (Hue), and color change (ΔE) were made employing eqs 4−6

$$C^* = \sqrt{a^2 + b^2} \tag{4}$$

$$\text{Hue} = \tan^{-1}(b^*/a^*) \tag{5}$$

$$\Delta E = \sqrt{(\Delta L^{*2}) + (\Delta a^{*2}) + (\Delta b^{*2})} \tag{6}$$

The CPJ presented an intense red color, as indicated by CIElab parameters obtained and shown in Figure 4. With regard to the benefit of the papaya red color present, a variation of this parameter with the percentage of HBSP resulted in decreased redness (a^* values), yellowness (b^* value), lightness (L^*), and chroma (C^*) and an increase of Hue angle (Hue), as shown in Table 7. The positive and high values of a^* corroborate the redness color of samples. M1 and M2 present the highest a^* values when the increase of HBSP shows a slight decrease of this parameter. The changes in redness and yellowness of CPJ can be evaluated by C^*, calculated according to eq 2 previously shown. A greater C^* value represents a more pure and intense color;[46] thus, when the samples present a decrease of C^*, there is a less intense color in samples. Thus, the Hue angle presents significant differences ($p < 0.05$), and showing a slight increase of these parameters indicated conservation and intensification of a reddish color in all samples.

The total color difference is a way to estimate the differences in color between the two samples.[47] Thus, the samples prepared with pectin (M1) were compared with samples with HBSP as a partial substitute (M2, M3, and M4). There were significant differences ($p < 0.05$) in terms of the ΔE values between the CPJ with HBSP (Table 7) and M1. The sample with the higher difference was the M4 sample, with 0.375% of HBSP. Overall, the addition of hydrocolloids provided a significant difference in the measured color parameters, preserving the red color parameter and increasing their tonality.

Sensorial Evaluation of CPJ. *CPJs* were considered acceptable (Table 8). The hedonic categories of color, odor flavor, consistency, and general acceptability depends on the percentage of hydrocolloids added; nevertheless, a similar result for the four samples in the different parameters "neither like, nor dislike" excepting the color and consistency of sample M3 (pectin 0.250% and HBSP 0.250%) showing "i like it" results, in which significant difference is observed in the scores between all the formulations and the sample control.

The attribute of the color of CPJ (M1, M2, M3, and M4) was 3.71 ± 0.2, 3.60 ± 0.10, 4.01 ± 0.10, and 3.90 ± 0.10, respectively, where M3 score for color was more than the control sample (M1) (Table 8). Odor scores were 3.33 ± 0.15, 3.37 ± 0.15, 3.50 ± 0.26, and 3.27 ± 0.06 for M1, M2, M3, and M4, in which the addition of HBSP increased the odor attribute.

The perception texture is also related to consistency, where the sample M3 presents the highest valorization, with M4 very close. In addition to these products, they contain high HBSP in their formulation, which indicates that the hydrocolloid content has a strong effect on the sensory perception due to the microstructural, rheological, and physicochemical changes in microstructured products. It is known that the high viscosity of the solutions can prevent the translational motion of the molecules, retarding their collision and even inhibiting rotational movement and hampering the formation and growth of crystals.[48] The overall acceptability of the samples was 3.58 ± 0.16, 3.58 ± 0.05, 3.83 ± 0.04, and 3.70 ± 0.18 for M1, M2, M3, and M4, respectively, which reveal the better acceptability and positive valorization of jams.

The rheological parameters are related strongly to sensorial parameters, such as spreadability, creaminess, thickness, smoothness, and taste perception. An increase in viscosity in the system decreased the perception of sweetness,[37] and creaminess is affected inversely by changes in the critical stress or strain.[49] The addition of hydrocolloids modifies the rheology and improves the physical stability and overall mouth-feel properties, so they may contribute to an increase in the desirable overall characteristics;[50] therefore, the samples with intermediate values of yield stress, consistency, and flux index present the highest acceptability valorization.

■ CONCLUSIONS

Hydrocolloids with a good carbohydrate−protein ratio from butternut squash (*C. moschata*) peels were obtained, improving the interactions of the molecules and giving rise to a strong and specific biopolymer. The HBSP obtained at 80 °C and pH 3, 7, and 10 presents a non-Newtonian flow behavior type shear thinning and with elastic properties. The obtained results have shown that BSP is a good source for the obtention of hydrocolloids for application in food products, improving the functionality. These ingredients contributed to stability, evidenced by an increase in the elastic modulus predominating the solidlike behavior. CPJ made with different percentages of HBSP results in different behavior compared to the control, having function as thickeners, gelling agents, and stabilizers, leading to a product without syneresis and presenting a higher elastic modulus. The tan δ values were similar, demonstrating the significant contribution of hydrocolloids in the elastic and viscous components in the formulation of microstructural products. The CPJ presented an intense red color, supporting the benefit of the papaya red color. The sample with higher differences was the M4 sample, with 0.375% of HBSP. Overall, the addition of hydrocolloids provided a significant difference in the measured color parameters, preserving the red color parameter and increasing their tonality. All CPJ samples standardized with different proportions of HBSP presented good acceptability. According to the sensory analysis, the sample M3 (0.250%) presented the highest valorization in acceptance by the panelists. Thus, the addition of HBSP improves the acceptability parameters of jams, increasing their overall desirable characteristics. The interaction of HBSP with other polysaccharides modifies the rheological and sensorial properties of food matrices, playing an important role in the acceptability of food products by increasing the physical stability of foods and the overall mouth-feel properties.

■ MATERIALS AND METHODS

Butternut squash (*C. moschata*) and *C. papaya*, with similar weight and commercial maturity, were purchased at the local market (Cartagena, Colombia). Ethanol (99.5% purity), sodium hydroxide (NaOH, pellets for analysis), petroleum ether (grade for analysis), and buffer solutions (boric acid/potassium chloride/sodium hydroxide) were purchased from Merck Millipore (Munich, Germany). Citric acid, sodium bicarbonate, and sucrose were purchased from Tecnas S.A. (Itagui, Colombia). Phenolphthalein and pectin from the citrus peel (galacturonic acid, ≥74%, dried basis) were purchased from Sigma-Aldrich Co. (St. Louis, MO, USA).

Isolation of Hydrocolloids. Hydrocolloid extraction was done employing a 3^2 experimental design to find the effect of pH and temperature extraction (Table 1). The extraction of hydrocolloids was performed using the methods described by Ibañez and Ferrero[51] and Orgulloso-Bautista et al.[52] with some modifications. Initially, the peels were dried at 25 °C for 72 h and ground. Solid−liquid extraction was carried out with distillate water (1:10 peel: water ratio) for 4 h at a specific temperature (30, 55, and 80 °C). The pH was adjusted using acetic acid and NaOH. The mixture was centrifuged for 15 min at 6500 rpm, and the supernatant was recollected. After that, the viscous solution was mixed with ethanol in a 1:1 ratio in order to precipitate the hydrocolloid-based extract. The mixture was centrifuged, and the precipitate was recollected and lyophilized for 48 h.

Design and Formulation of the Microstructure Product Type Jam. CPJ was prepared following the procedure described by Quintana et al.[53] Briefly, the pulp puree was mixed with half the amount of sugar and stirred at 25 °C until the dissolution of sugar. The pH was adjusted to 3.0 ± 0.05 with citric acid while waiting to reach 70 ± 1.0 °C. The rest of the sugar, previously mixed with pectin and hydrocolloids, was added and stirred. The jam was heated until it reached 65 °Brix. Hot jams were poured into glass jars with screw caps and stored at 4 °C until use. Four CPJ formulations were prepared by substituting the commercial pectin with HBSP.

Proximal Composition and Physicochemical Properties. The proximal composition of hydrocolloids and CPJs was done following the methods described by the Official Methods for Analysis (AOAC).[54] The moisture was determined by dehydration in an oven at 105 °C for 4 h; the fat content was determined by the Soxhlet method employing hexane as a solvent; the total protein content was determined by Kjeldahl methods and the total carbohydrate was obtained by subtracting the amounts of water, protein, total fat and ash from 100. The physicochemical properties such as pH, soluble solids, and acidity were determined according to the method described by AOAC.[54]

Rheological Characterization of Hydrocolloids and CPJs. The rheological properties of HBSP on a wet basis and CPJs were evaluated following the methodology described by Quintana et al.[55] and were done using a Haake Mars 60 (Thermo-Scientific, Germany) rheometer equipped with a coaxial cylinder (inner radius 12.54 mm, outer radius 11.60 mm, and cylinder length 37.6 mm). Initially, the temperature was fixed using a Peltier system, and each sample was equilibrated at 600 s before the rheological test to have the same recent past thermal and mechanical history.

Steady-Shear Test. The continuous shear rate test was performed from 10^{-3} to 10^3 s^{-1} at 25 °C.

Oscillatory Test. The stress amplitude sweep test was carried out within the range of 10^{-3} to 10^3 Pa, with an angular frequency of 1 Hz on all samples, in order to determine the LVR. The frequency sweep test was done from 10^{-2} to 10^2 rad·s^{-1} in the linear viscoelastic range at 25 °C.

Color Analysis of Jams—CPJ. Color parameters of CPJ were evaluated using a colorimeter. The measurements included L^* (lightness), a^* ($+a^*$ = redness, $-a^*$ = greenness), and b^* ($+b^*$ = yellowness, $-b^*$ = blueness) per triplicate.

Sensorial Evaluation of Jams—CPJ. Sensorial evaluation by sensory descriptive analysis of CPJ was performed. A panel of 30 tasters, 15 men and 15 women, were recruited among staff and students at the University of Cartagena. The panelists were instructed to evaluate each sample individually. Sensory analysis of the samples consisted of the evaluation of color, flavor, and consistency attributes employing the hedonic scale following the descriptors: i dislike it a lot = 1; i dislike it a little = 2; i neither like nor dislike it = 3; i like it = 4; and i like it a lot = 5. The samples were served at room temperature in transparent plastic glass identified with three-digit random numbers. Each panelist tasted approximately the same amount of each sample, and mineral water was provided to the assessors to rinse their mouth. Panelists were given room-temperature water to cleanse the palate before the presentation of the samples, as described by the technical guide GTC 165.[56] The results were expressed as a percentage of acceptability.

Statistical Analysis. All analyses were done in triplicate. The significative treatments were determined using one-way analysis of variance with Tukey HSD (honestly significant difference); grouping at 95% confidence level was performed using Statgraphics Centurion XVI.

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Notes

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