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Gel Textural Characteristics of Corn, Cassava and Yam Starch Blends: A Mixture Surface Response Methodology Approach

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Blends of native starches can be used to obtain special sensory properties avoiding the use of chemically modified starches. The mixture design approach was used to analyze the textural properties (hardness, adhesiveness, cohesiveness and gumminess) of gels obtained with different proportions of yam, corn and cassava starches (6% total solids) and related to microstructural characteristics. Maximum limits of 60% yam starch and 70% corn starch and minimum level of 30% cassava starch were fixed to minimize syneresis under storage. Hardness, adhesiveness and gumminess increased with the proportion of corn starch in the blends. The lowest values of hardness corresponded to the blends containing higher proportions of cassava starch, that has the lowest amylose content. Corn starch was the component that less contributed to cohesiveness. The characteristic high cohesiveness of cassava starch pastes (related to its higher amylopectin content) was reduced when it was mixed in adequate proportions with yam and/or corn starches. Gels containing only yam starch presented syneresis values close to 40% after 24 h storage at 4°C; the decrease of the maximum level of yam starch to 60% as well as the inclusion of cassava starch in the blends reduced weight losses. Disadvantages found in gels containing individual starches, such as exudate in yam and corn starch gels, and excessive cohesiveness in cassava starch gels, are minimized improving their possible applications, when blends are used.

Keywords: Corn; Cassava; Yam; Starch blends; Texture

1 Introduction

Starch is the most important source of carbohydrates in human diet. Though corn starch is the most widely used as food ingredient, starches from other botanical sources show different functional properties related to their capacity as adhesives, binders, stabilizers, thickeners, gelling and film forming agents, emulsions and foams stabilizers and water retention agents. These different characteristics allow their application with different industrial purposes: nutritional, technological and sensory ones [1].

Some characteristics of the starches can determine and even limit their industrial applications: stability under stirring, different pH conditions and heating treatment, paste clarity and texture, tendency to retrogradation and syneresis. Functional properties are not only affected by starch: water ratio but also by temperature, heating and stirring rates during gel preparation [2].

Among microstructural factors affecting functional properties of starch, the following can be mentioned: granule morphology and size, amylose:amylopectin ratio, presence of other components of the granule [3, 4] and length of amylopectin ramified chains [5, 6].

Aqueous suspensions of corn (*Zea mays*) starch, when submitted to gelatinization temperatures form viscous pastes and opaque gels with a strong tendency to retrograde. On account of its tendency to retrograde the use of this starch in foods requiring storage at low temperatures is not recommended except when hydrocolloids are added as stabilizers [7].

Cassava (*Manihot esculenta*) starch, with a relative high content of amylopectin, is utilized by the food industry due to its high paste clarity, cohesive texture of pastes, low tendency to retrogradation and good gel stability [4]. Other advantageous characteristics are its low range of gelatinization temperature (between 65 and 70°C) and fast increase of the viscosity during gelatinization [8].

Yam (*Dioscorea alata*) starch granules are trigonal-rounded shaped, with a high amylose content (28 to 30%). Gelatinization temperature ranges between 71 and 83°C.

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This starch shows a high tendency to retrogradation and syneresis, low paste clarity and high gel strength leading to a firmer consistency at room temperature after thermal treatment [9–12].

Different studies were performed on the behavior of starch blends from different botanic sources in the food industry, mainly in systems formulated with natural products, avoiding the use of chemically modified starches to obtain the desired sensory properties [13, 14]. Blends of native starches may be formulated to reach at least some of the desired characteristics of the modified ones.

The mixture design approach [15] has been widely used to obtain product formulations. In this type of experiments, two or more ingredients are blended in different proportions and the resulting mixture characteristics are analyzed. Responses depend only on component proportions. It is possible to study how mixture properties are affected by changes in component ratios and synergic or antagonist effects of a combination of two or more components can be easily identified. Nowadays mixture design experiments are considered as a useful technological tool to optimize product formulations and processes, to accelerate product development reducing costs, to improve the transfer of research results to product manufacture and to solve production problems [16].

The objectives of this work were:

- to analyze the effect of starch (yam, corn and cassava) blends on textural properties and exudate production of gels under refrigerated storage,
- to analyze these results according to microstructural changes in the system.

2 Materials and Methods

2.1 Materials

Yam starch was extracted from fresh yam (*Dioscorea alata*) tubers using the method describe by Alves et al. [10]. Corn starch was provided by Corn Products (Curitiba, Brazil) and cassava starch by Hikari Ind. Com. (São Paulo, Brazil). Yam, corn and cassava starches contained (d.b.), respectively, 0.50, 0.29, 0.28% lipids; 0.16, 0.27, 0.11% protein; 0.13, 0.06, 0.22% ashes and 99.21, 99.38, 99.39% carbohydrates.

Amylose contents for yam, corn and cassava starches were 28.5, 25.1, 19.2%, respectively.

2.2 Experimental design

Starch blends were prepared with the proportions defined by the simplex-centroid experimental design for mixtures of three components expanded with internal points, with constraints, in accordance with the software Statistica 5.1 (Stat Soft, Oklahoma, USA). Two experimental designs were applied. The first one includes a maximum limit of 60% yam starch in the blend. This upper bound restriction was necessary because preliminary tests showed that higher concentrations of this starch (> 60%) led to very strong gels with important exudate production (syneresis) after a 3–4 h setting. Thus, only experimental data in a subtrapezium (Fig. 1b) of the full triangle area [15] of the mixture design could be studied (Tab. 1, first design). The second experimental design (Tab. 1) was used for texture measurements during storage. The defined region was more restricted in order to evaluate only those gels that were stable for at least one day without syneresis. To attain this objective, maximum levels of yam and corn starches were fixed at 60 and 70% respectively, and the minimum level of cassava was 30%.

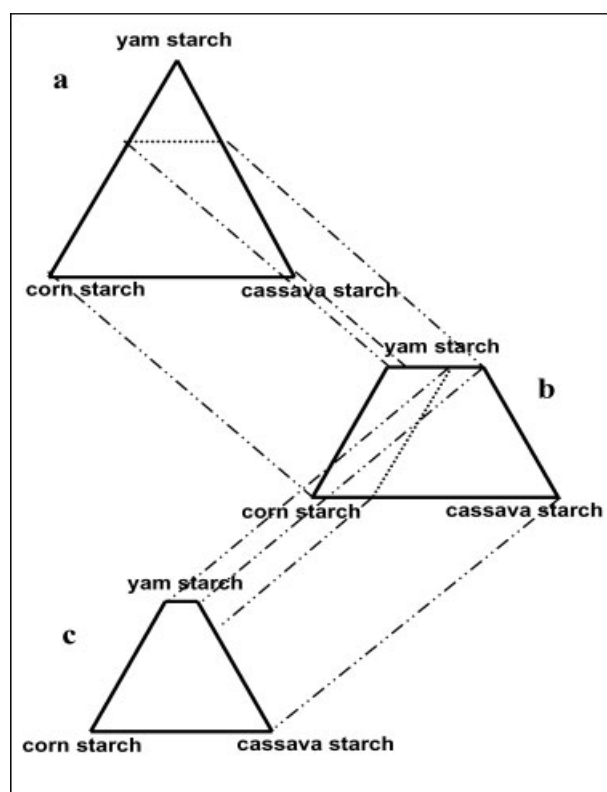


Fig. 1. (a) Experimental region for centroid design. (b) Experimental region defined for the first experimental design (maximum proportion of yam starch in the mixture: 60%). (c) Experimental region defined for the second experimental design (maximum proportion of yam starch in the mixture: 60%, maximum proportion of corn starch: 70% and minimum proportion of cassava starch: 30%).

Tab. 1. Simplex-centroid mixture designs with constraints.

Run	Starch proportions ¹					
	First design			Second design		
	X_1	X_2	X_3	X_1	X_2	X_3
1	1.00	0.00	0.00	0.00	1.00	0.00
2	0.00	1.00	0.00	0.70	0.30	0.00
3	0.33	0.34	0.33	0.00	0.40	0.60
4	0.50	0.50	0.00	0.10	0.30	0.60
5	0.20	0.20	0.60	0.00	0.70	0.30
6	0.67	0.00	0.33	0.35	0.65	0.00
7	0.00	0.67	0.33	0.40	0.30	0.30
8	0.00	0.40	0.60	0.05	0.35	0.60
9	0.40	0.00	0.60	0.20	0.50	0.30

¹ $X_1 + X_2 + X_3 = 1$ or 100% $X_1 =$ corn starch $X_2 =$ cassava starch $X_3 =$ yam starch

2.3 Gel samples preparation

Starch gels were prepared by heating aqueous suspensions (450 mL, 6% starch d.b.) of the pure or blended starches in a Brabender ViscoGraph (Brabender Instruments, Inc., Duisburg, Germany) to 95°C (3°C/min heating rate) and holding this temperature for 10 min. The hot pastes were poured into cylindrical plastic tubes (30 mm diameter and 45 mm height) that were covered, cooled at room temperature and stored for 1, 3, 5 or 7 days at 4°C. Gels were placed at room temperature at least 1 h before texture and exudate analyses were done.

2.4 Microscopy analysis

Starch gels for microscopy observations were prepared according to Karam [8]. Small gel samples were fixed under vacuum (24 h) with a solution of formaldehyde, acetic acid and ethanol. Samples were then immersed in ethanol solutions of increasing concentration: 50%, 70%, 80%, 90%, 100% and in absolute ethanol-xylene solution; clarification was performed in pure xylene. Samples were embedded in paraffin by low rate infiltration and sections of 10 µm thickness were mounted and stained with iodine solution. Observations and image digitalization were performed with a Leica DMLB microscope and Leica DC100 software (Wetzlar, Germany).

2.5 Exudate measurements

Exudate measurements in starch gels were performed following the method proposed by Billaderis [17]. Samples were prepared and stored as it was previously

described. The water losses were determined by differences in the weight of the samples, after 1, 3, 5 and 7 days storage and expressed as percentage of exudate in the gels. Analyses were performed in triplicates.

2.6 Texture analysis

Gel texture parameters were evaluated using a TA.XT2i Texture Analyser (Texture Technologies Corp., NY, USA) equipped with the software XTRAD. The gels were penetrated (15 mm) with a cylinder probe 15 mm in diameter and 30 mm long (SMS P/0.5). Force-time curves were obtained at a crosshead speed of 2 mm/s, during two penetration cycles. Gel strength, adhesiveness, cohesiveness and gumminess of gels were determined in triplicates. Gel strength or hardness is defined as the maximum force registered during the first compression cycle. Adhesiveness is the negative area obtained during the first cycle. Cohesiveness is determined as the ratio between the positive area of the second cycle and the positive area of the first cycle. Gumminess is calculated as the product of cohesiveness and hardness.

2.7 Amylopectin retrogradation by differential scanning calorimetry (DSC)

Amylopectin retrogradation was determined by DSC using a polymer equipment (Rheometric Laboratories, Surrey, UK). Approximately 10 mg of the refrigerated starch gels were weighed in coated aluminum pans, that were hermetically sealed. An empty double pan was used as reference. Scans were performed from 20 to 100°C at a controlled constant rate of 10°C/min. After each test, pans were punctured and heated at 105°C; the exact dry weight was measured to determine the water content of the samples. Runs were performed in duplicates.

2.8 Statistical analysis

The Scheffé canonical equation (special cubic) was used to model the experimental data for three components (Eq. 1)

$$Y = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3 \quad (1)$$

where Y is the studied response, $\beta_1, \beta_2, \beta_3, \beta_{12}, \beta_{13}, \beta_{23}$ and β_{123} are the regression coefficients and X_1, X_2 and X_3 are the levels of corn, cassava and yam starches in the blends, respectively. As the analyzed mixture designs included constraints, the mass fraction values (X) are rescaled so that the low and high factor settings for each factor are transformed to 0 and +1 respectively; these

Tab. 2. Mean values of starch gels¹ textural parameters corresponding to the first day of storage at 4°C.

Starch proportions			Hardness	Adhesiveness	Gumminess	Cohesiveness
Corn	Cassava	Yam	[N]	[N·s]	[N]	(dimensionless)
1.00	0.00	0.00	0.934(±0.085)	1.438(±0.045)	0.427(±0.018)	0.458(±0.021)
0.00	1.00	0.00	0.193(±0.003)	0.021(±0.020)	0.113(±0.002)	0.581(±0.012)
0.33	0.34	0.33	0.284(±0.030)	0.560(±0.091)	0.158(±0.004)	0.559(±0.046)
0.50	0.50	0.00	0.363(±0.008)	0.557(±0.022)	0.217(±0.010)	0.597(±0.016)
0.20	0.20	0.60	0.291(±0.016)	0.364(±0.005)	0.147(±0.005)	0.507(±0.038)
0.67	0.00	0.33	0.481(±0.013)	0.729(±0.027)	0.246(±0.020)	0.512(±0.028)
0.00	0.67	0.33	0.184(±0.001)	0.227(±0.032)	0.107(±0.006)	0.578(±0.033)
0.00	0.40	0.60	0.172(±0.011)	0.200(±0.014)	0.100(±0.004)	0.584(±0.018)
0.40	0.00	0.60	0.571(±0.018)	0.588(±0.032)	0.289(±0.017)	0.505(±0.029)

¹ 6% starch (dry basis).

Values between parentheses correspond to standard deviations.

recoded component settings are called “pseudo-components”. This transformation makes the coefficients comparable in size.

Responses were submitted to analysis of variance (ANOVA) to determine: the significance of the regression model at 5% level (*F* test), coefficient of variation (CV) and the adjusted coefficient of determination (R^2) [15]. Positive values for β indicate synergistic effects while negative values indicate antagonism. Full models were simplified by eliminating non-significant terms ($p > 0.05$).

Triaxial diagrams were obtained from the polynomial equations for each property using the software Statistica for Windows 5.1 (StatSoft, Oklahoma, USA).

3 Results and Discussion

3.1 Texture analysis

Tab. 2 shows mean values of the texture properties of starch gels analyzed 24 h after preparation.

Statistical analysis indicated that all the responses followed normal distributions and ANOVA (Tab. 3) showed that all the models were significant ($p < 0.05$). However, high coefficients of variation ($> 20\%$) for hardness, adhesiveness and gumminess indicate that, in these cases, models can only be used to show behavior tendencies in the analyzed region, but they are not useful for predictive purposes. With reference to cohesiveness, the adjusted coefficient of determination was 76.3% but the responses predicted by the model were close to the experimental values (coefficient of variation = 4.31%) so it could be useful for predictive purposes.

Tab. 3. Mathematical modeling¹ of the textural properties: regression coefficients and analysis of variance (ANOVA).

Regression coefficients	Texture properties			
	Y_1	Y_2	Y_3	Y_4
β_1	0.908	1.396	0.399	0.468
β_2	0.213	0.023	0.105	0.590
β_3	0.673	0.221	0.313	0.537
β_{12}	-0.762	-0.450	-	0.249
β_{13}	-1.176	-0.729	-0.439	-
β_{23}	-1.097	0.504	-0.480	-
β_{123}	-	-	-	-
Model	0.038	0.014	0.021	0.016
significance (p)				
CV [%]	23.69	20.97	22.18	4.31
R^2 (adjusted)	0.86	0.93	0.829	0.763

¹ $Y = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3$, where Y_1 = hardness; Y_2 = adhesiveness; Y_3 = gumminess; Y_4 = cohesiveness; X_1 = corn starch, X_2 = cassava starch, X_3 = yam starch
CV = coefficient of variation
 R^2 = coefficient of determination

As shown in the contour graphs in Fig. 2a, b, c corresponding to models Y_1 , Y_2 , and Y_3 respectively; hardness, adhesiveness and gumminess increased with the proportion of corn starch in the blends. The curved traces toward corn starch vertex in Figs. 2a and c showed the interaction effect (β_{12} and β_{13} coefficients) of this component with the other ingredients on the responses.

According to Morris [18] and Fannon and BeMiller [19], main factors affecting functional properties of gelatinized starches are: firmness of the continuous amylose matrix,

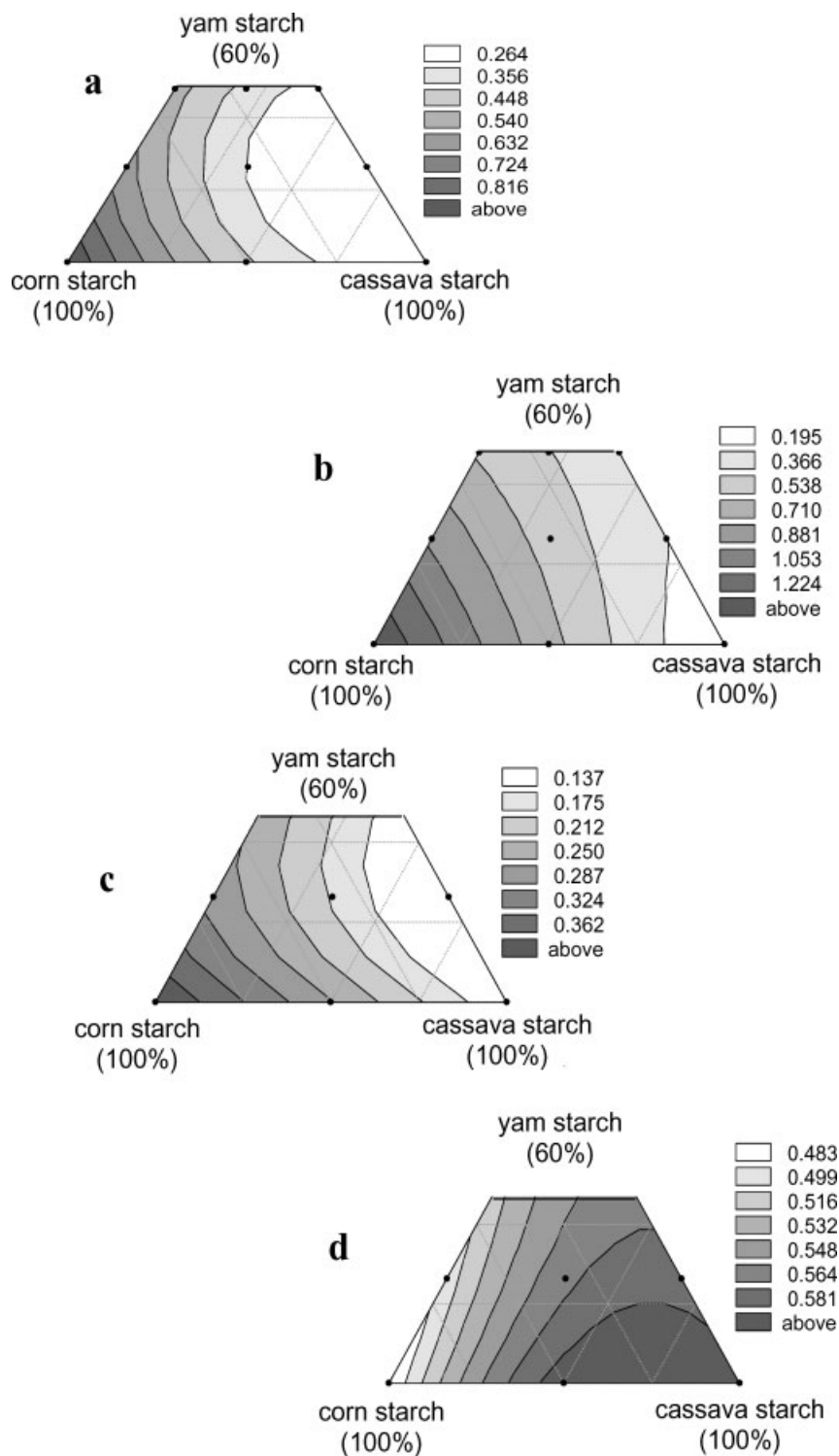


Fig. 2. Effect of corn:cassava: yam starch proportions on different textural properties of gels: (a) hardness, (b) adhesiveness, (c) gumminess and (d) cohesiveness.

stiffness and volume of the remaining starch granules and their interactions. Thus, gel firmness must be related to amylose matrix as well as to the filling effect of swollen granules [18].

The lower values of hardness corresponded to the blends containing higher proportions of cassava starch. Cassava starch, having the lowest amylose content among the components of the blends contributed to decrease the hardness of the blends.

It was reported in literature that the length of amylopectin branches is related to gel strength and other functional properties [20, 21]. For the tested starches the ratio between short chain/long chain amylopectin ramifications were corn = 5.32; cassava = 2.7 and yam = 2.3 [8]. Therefore, the decrease in hardness found in blends with high proportions of cassava starch can be attributed not only to the low amylose content but also to the low ratio between short chain/long chain amylopectin branches.

In addition micrographs not shown of the gels containing 100% cassava starch showed that molecular dispersion was complete and granules have lost their integrity leading to softer gels.

Gels of cassava/yam starches have comparable hardness to those of cassava starch alone, although it could be expected that yam starch incorporation to blends led to an increased hardness. A possible explanation to this result is an incomplete leaching of yam starch amylose (main contribution to gel hardness) during heating time (10 min).

Figs. 3b and a show micrographs corresponding to a yam starch gel and a blend of yam, corn and cassava starches, respectively. In Fig. 3b a greater loss of granule identity can be observed. On the other hand, when yam starch is blended with cassava and corn starches, its granules were only partially swollen and seemed like rods. This inhibition of granule swelling in yam starch can be attributed to a higher resistance of yam starch to shear and heating treatment [10] and a lower availability of water by yam granules when other starches with lower gelatinization temperatures are incorporated to the blend. Differential scanning calorimetry (DSC) studies [8] evidenced shoulders or only one peak in the thermograms of blends containing at least 30% yam starch; thus, granules of corn and cassava starch, that gelatinize at lower temperatures (65.6 and 62.7°C, respectively), decreased the

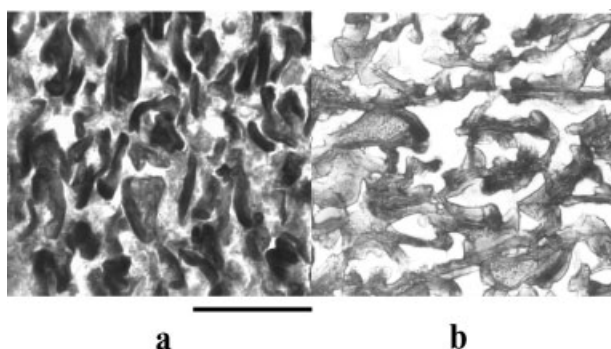


Fig. 3. Micrographs of (a) 0.1:0.3:0.6 corn:cassava:yam starch blend, (b) yam starch paste. Content of solids: 6%. Bar corresponds to 100 μm .

swelling capacity of yam starch granules. However, DSC results as well as loss of birefringence showed that in all cases starches were completely gelatinized.

The other contribution to gel elasticity is the interaction between the granule and the continuous phase formed by amylose as can be verified through studies on gel microstructure. As cassava and corn starches have lower gelatinization temperatures, their granules capture the water molecules before yam starch; thus yam starch granules remain partially swollen. These yam granules embedded in the amylose matrix also contribute to gel firmness. However, decrease of yam starch swelling and amylose leaching in starch blends led to softer gels with respect to systems containing only yam starch.

The lower adhesiveness observed in pastes containing a major proportion of cassava starch (Fig. 2b) can be related to the amylose molar masses of the different starches. Cassava starch exhibits the higher amylose molar mass among the three studied starches [8]. *Mua* and *Jackson* [21] reported an inverse linear relationship between adhesiveness and amylose molar mass in gels obtained with amylose fractions and a linear relationship between amylopectin ramifications and adhesive force in gels formed with amylopectin fractions. In the obtained blends, amylose is the main component present outside the granule and it strongly influences gel characteristics.

Fig. 2c shows the response surface corresponding to gumminess (= hardness \times cohesiveness) The similarity with Fig. 2a indicates that hardness is the most important contribution.

In Fig. 2d, it can be observed that corn starch is the component that less contributes to cohesiveness. The region with the highest values of this property was delimited by a approximately triangular zone whose vertices were: 56% corn starch/44% cassava starch; 20% corn starch/20% yam starch/60% cassava starch and 100% cassava starch. Thus, the characteristic high cohesiveness of cassava starch pastes (related to its higher amylopectin content) can be reduced when it is mixed in adequate proportions with yam and/or corn starches. A decrease in the cohesiveness of the gels can be interesting for technological applications of cassava starch, because this attribute is not desired in many food products such as puddings, creams, fillings and sauces [22].

3.2 Texture parameters changes along storage time

Texture parameters of gels prepared according to the second experimental design and stored during 1, 3, 5 and 7 days at 4°C are shown in Figs. 4a, b and c (hardness,

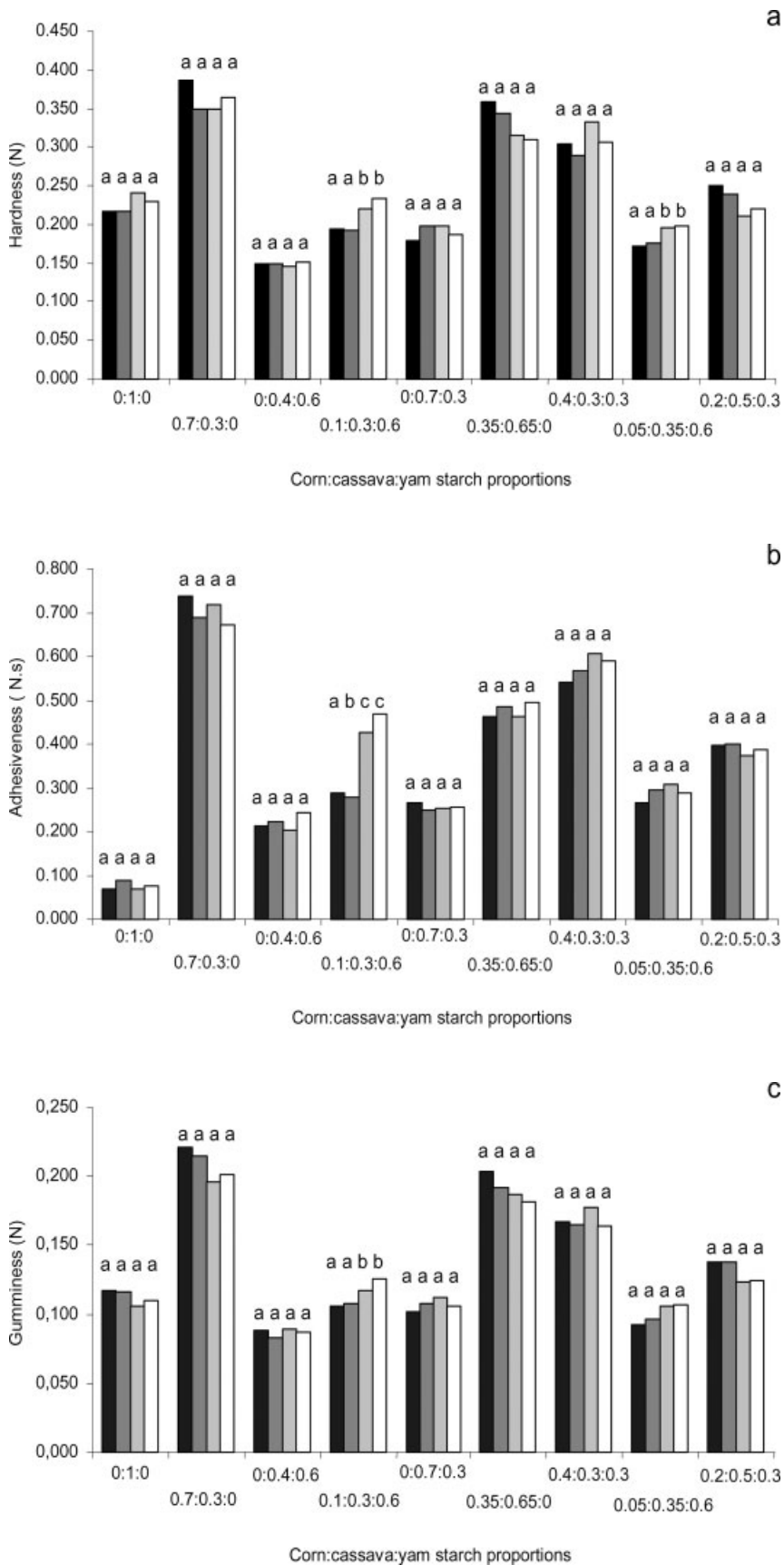


Fig. 4. Effect of storage time at 4°C on different textural properties of corn:cassava:yam starch mixtures. (a) hardness, (b) adhesiveness, (c) gumminess after (■) 1, (▣) 2, (▨) 5 and (□) 7 days of storage. For each gel composition, bars with the same letter do not differ significantly ($p < 0.05$).

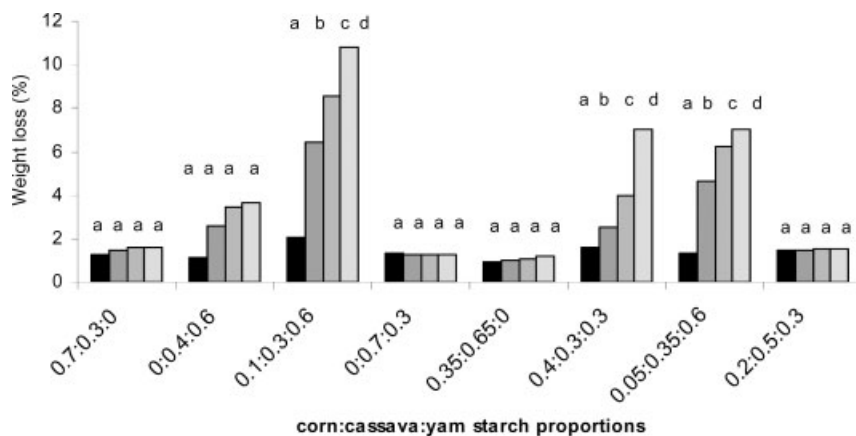


Fig. 5. Weight loss (% exudate) of corn: cassava: yam starch mixtures after (■) 1, (▒) 2, (▓) 5 and (□) 7 days of storage at 4°C. For each gel composition, bars with the same letter do not differ significantly ($p < 0.05$).

adhesiveness and gumminess, respectively). No significant changes were detected within the storage period studied in the present work, except for some specific combinations. DSC studies of gels showed that no significant amylopectin recrystallization occurred during the 7-days storage period at 4°C. These results indicate that amylose retrogradation, occurring within the first 24 h, was the main factor contributing to gel characteristics.

With respect to hardness, gels obtained from a mixture of corn/cassava/yam starches in the proportions 0.1:0.3:0.6 and 0.05:0.35:0.6 showed a significant increase in hardness ($p > 0.05$) between the third and fifth day of storage and then remained without significant changes (Fig. 4a). These gels have a higher content of yam starch and also lower contents of corn starch. When corn starch was removed, as in sample 0.0:0.4:0.6, the hardness of gel was stable.

Adhesiveness and gumminess did not show significant changes during the analyzed period except for the blend 0.1:0.3:0.6 (Figs. 4b and c). Cohesiveness did not show significant differences ($p < 0.05$) between the first and seventh day of storage in any case. Stability of the textural parameters during the storage period, exhibited by some mixtures, is a useful characteristic comparable to that obtained with modified starches.

3.3 Exudate production

Fig. 5 shows the loss of weight (related to starches syneresis) for gels prepared with corn/cassava/yam starch mixtures at different storage times. It was verified that the systems containing only cassava starch (mixture 0: 1: 0) or the blends with high cassava starch levels (mixtures 0: 0.7:0.3; 0.35:0.65: 0; 0.2:0.5: 0.3) had non-significant exudate production.

Pure yam starch gels showed syneresis values close to 40% after 24 h storage at 4°C [8]. On the other hand, the blend with 40% cassava starch and 60% yam starch showed an exudate level of only 3.64% after seven days of storage, thus reduction of the maximum level of yam starch to 60% as well as cassava starch incorporation allowed to considerably decrease in exudate production. The presence of cassava starch could contribute to a lower mobility of amylose leached from yam starch, that has a strong tendency to retrograde. Probably due to this fact, in gels with predominance of yam starch the syneresis was controlled up to the first day of storage but increased significantly ($p > 0.05$) along the storage period.

The increase of hardness during storage, in samples with high proportion of yam starch (Fig. 4a) can be related to water losses. However, other blends did not show this correspondence probably due to compensation provided by the presence of cassava starch at a minimum level of 30% that could hinder amylose chains realignment.

4 Conclusions

The results of this work indicate that gels with different textural properties can be obtained from the combination of the three studied starches. On the other hand gels with similar properties can be obtained varying the formulation between the limits of each level within the response surface. This fact allows a certain flexibility when formulating starch based products according to availability and costs.

Disadvantages found in gels obtained from the use of the individual starches (exudate in yam and corn starch gels, excessive cohesiveness in cassava starch gels) could be minimized when the starches are used as part of blends, thus improving further applications.

Mixture design approach was fundamental for detection of synergistic or antagonistic effects between the starches and also to furnished a complete vision of textural properties variations within the experimental region.

Acknowledgements

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