Rapid analysis for predicting the expansion of the *polvilho azedo*

Análises rápidas para prever a expansão do polvilho azedo

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ABSTRACT
Sour cassava starch (*polvilho azedo*) is a chemically and enzymatically modified starch. It is used to prepare the traditional baked goods. Because of its satisfactory expansion property, a light and alveolar structure is produced in baked food prepared with sour cassava starch, without adding any chemical or biological ferment. This study reports some easy and simple procedures for pre-estimating sour cassava starch expansion property. The correlation between the native cassava starches and the laboratory-produced sour cassava starch was analyzed. An investigation among the local bakers was carried out for inquiring into the *polvilho azedo* choice, and it disclosed some product brands. The *polvilho azedo* samples from these brands were also evaluated. Acid factor, pH, specific volume, swelling power and intrinsic viscosity characteristics were directly correlated with expansion property. The procedures for determining acid factor, pH and weight loss are easy and accessible methodologies which might be carried out at small industries and bakeries.
Keywords. cassava, expansion, fermentation, rheology, starch.

RESUMO
O polvilho azedo é um amido de mandioca em que são feitas modificações químicas e enzimáticas, sendo usado na fabricação de produtos tradicionais, como pão de queijo, em função de sua propriedade de expansão, que induz a formação de estrutura leve e alveolar, mesmo sem adição de qualquer produto químico ou fermento biológico. O presente trabalho foi realizado com o objetivo de estabelecer alguns procedimentos técnicos simples para efetuar a avaliação prévia da propriedade de expansão do polvilho azedo a ser utilizado. Foi estudada a relação entre os amidos de mandioca nativos e os do polvilho azedo produzido em laboratório. Um questionário foi aplicado entre os padeiros locais com o objetivo de averiguar o motivo para efetuar a escolha do polvilho azedo, o que conduziu a algumas marcas; essas marcas selecionadas foram também analisadas neste estudo. As características referentes a fator ácido, pH, volume específico, poder de inchamento e viscosidade intrínseca apresentaram boa correlação com a propriedade de expansão. As determinações de fator ácido, pH e perda de peso são procedimentos fáceis e acessíveis para serem efetuados em pequenas indústrias e padarias.
Palavras-chave. mandioca, expansão, fermentação, reologia, amido.

INTRODUCTION

Food and beverages from tropical roots are produced throughout the world involving numerous and diverse processes, resulting in functional compounds, additives and special properties of these novel or traditional products, when compared to the original raw material[1].This is the case of the sour cassava starch (*polvilho azedo*), for example, a naturally modified starch with special properties.

Numerous works intend to chemically show the expansion property of *polvilho azedo*, a fermented and sun dried cassava starch, considered a chemically and enzymatically modified product, with acidic characteristics, perforated granules and high expansion capacity[2-8]. Expansion is a natural characteristic of *polvilho azedo*, defined as the growth rate of the dough during oven cooking, also referred to as expansion rate[9]. It is directly related to specific volume, expressed in $cm^3.g^{-1}$. *Polvilho azedo* with good performance in bakery results in products with soft, alveolar structures, differently from non-fermented cassava starches, referred to as native cassava starch[10].

In comparison to native cassava starch *polvilho azedo* is a product of low pH, high acid factor, low swelling power and low intrinsic viscosity, which implies a loss in the polymerization degree, due to structural changes during fermentation and sun drying[11,12].

Several works state the importance of hydrolysis and sun drying to define the properties of *polvilho azedo*, which contribute to dough expansion[2-8]. Meanwhile, there still is the uncertainty about physicochemical parameters to ensure commercialization of *polvilho azedo* of good-performance.

Nowadays sour cassava starch industries use a practical assay, making a sample of specimen (or *biscuit*) and if it expands, the *polvilho azedo* is suitable for commercialization. However, industries and bakeries either need to know the physicochemical variables for an easy determination or depend on a simplified analysis to determine the best *polvilho azedo*. A study[10] suggested a chemical model of the expansion of *polvilho azedo*, aiming to find any variables that could be directly or indirectly related with it. The variables which could be considered in a *polvilho azedo* quality control system are: acid factor, pH, specific volume, swelling power, and intrinsic viscosity. Meanwhile, those which are more accessible for small industries and bakeries would be the pH and the acid factor. In spite of specific volume representing an easy analysis, it takes longer to be performed and also is very similar to the one which is being currently used to determine the best *polvilho azedo* in the industry. The determination of swelling power requires the use of a centrifuge and of intrinsic viscosity requires an elaborate method, which is inaccessible for small industries and bakeries[10].

The sour cassava starch (*polvilho azedo*) industry is traditionally a familial business. The processing units are classified, at the most, as medium sized enterprises[13,14]. The major effect of this reality is the lack of access to advanced technologies for quality control of raw material, products, and processes. At the same time this situation persists, the preference for *polvilho azedo* derivatives is increasing. It is used in the production of cheese bread, biscuits and cookies. The expansion of the dough occurs without any addition of yeast and is gluten free. The classification of *polvilho azedo* as a gluten free product contributes to its increasing demand and justifies concern for the standardization of the *polvilho azedo* quality, which is still considered as a rustically made product[15].

Based on results by Marcon et al.[10] and on several other published works, including advanced techniques for analysis such as ATR-FTIR (Attenuated total reflectance-Fourier-transform infrared spectroscopy), DSC, X-Ray diffraction, and others, this present work intends to show variables of easy determination that could be suggested as an easy analysis to predict the performance of *polvilho azedo*. The present work was performed with the objective of suggest some easy assay procedures to predict sour cassava starch expansion property accessible to bakers and cassava industries.

MATERIALS AND METHODS

A comparison was made on the physicochemical and expansion characteristics of native cassava starch (five different trademarks and one mix of them, resulting in six samples) and sour cassava starch (*polvilho azedo*) samples that, according to the opinion of bakers, were of good performance (three different trademarks), more fourteen good trademarks, totalizing seventeen (17) samples of commercial sour cassava starch; as well as of the sour cassava starches samples produced in laboratory (twenty samples) with simulation of its industrial production and with modifications[25] and also with addition of glucose syrup were also included in the investigation.

Interview with bakers

All bakers, of every bakery, in the centre of Florianópolis downtown, Santa Catarina State, Brazil, were interviewed. The interview included open questions about the criteria they use when choosing a brand of *polvilho azedo*.

Physicochemical analysis

All samples were characterized for moisture content (method AOAC 921.10)[16] to express results in dry basis. Acid factor, pH, expansion rate, swelling power, and specific volume were determined for all samples, and intrinsic viscosity was determined for commercial native cassava starch (five samples) and commercial *polvilho azedo* (three samples).

The pH values were measured by using a Quimis pHmeter (model Q 400A). Acid factor was determined with samples of 25 g, with addition of up to 50 mL of distilled water. The dispersion was titrated with a standard 0.1 N HCl to pH 3.0 under agitation. The acid factor corresponds to the volume of acid consumed (mL).[17]

Swelling power was evaluated at 90 °C, as described by[18], with modifications. The analysis was carried out in 50 mL leak proof centrifuge tubes by taking a suspension of starch in deionized water (0.4 g starch and 36 g deionized water) in water bath under agitation at the selected temperature. After 30 minutes for cooling, the volume was completed until obtaining 40 g and centrifuged (3,000 rpm/20 minutes), and all supernatant was collected. The pellets containing the swollen starch granules were weighed and the ratio between the final mass and the initial dry matter was considered as the swelling power. The solubility was expressed in weight percentage and the swelling power in weight gain.

The determination of baking expansion capacity followed the procedures proposed by the CERAT – Tropical Root Centre.[19] The formulation was made with 50 g of sample with 40 mL boiling water for the production of the dough. The dough was divided into five balls of approximately 10 g each and baked at 200 °C for 25 minutes in an electric oven. The diameter of each dough ball was measured with a Vernier calliper (Metrica, Italy) before and after baking. Baking expansion capacity was calculated by using the relation between the initial average diameter of the dough balls before baking and their final average diameter after baking (their diameters are irregular)[20].

Specific volume of the dough balls after cooking was determined through the rape seed displacement method and calculated as displacement/weight ($cm^3.g^{-1}$)[21].

The Intrinsic viscosity[22] of the cassava starch samples was measured with a thermostatically viscometer Schott, AVS 350, CT 52, at constant temperature 30 °C, capillary diameter 0.63 mm; capillary constant (K) of 0.01511 $mm^2.s^{-2}$. Solutions of the cassava starches samples at 0.002, 0.003, 0.004, 0.005, 0.006 and 0.007 g/mL were prepared in 1.0 N KOH. To determine the flow time of the solvent (0.1 N KOH) and of the starch solutions, exactly 10 mL of solutions were transferred to the 100 mL tube of the viscometer. Specific viscosity was calculated by Equation 1.

$$\eta_{sp} = (\eta/\eta_o - 1) = (t/t_o - 1) \qquad \textbf{(Equation 1)}$$

Where: η_{sp} = specific viscosity; η = starch solution viscosity; η_o = solvent (KOH 0.1 N) viscosity; t = flow time of starch solution; t_o = flow time of solvent.

The results of the reduced viscosity (specific viscosity/concentration) for each concentration of starch solution were plotted and intrinsic viscosity was determined by extrapolation to concentration zero of the starch solution. The point of intersection between the straight and the y axis of the concentration zero of the starch solution was considered as intrinsic viscosity.

Data analysis

The individual co-relation between each one of the analyzed variables and expansion rate was examined using the Statistical 6.0 software. Variables that showed the greatest value for the co-relation coefficient were adapted to preview the expansion capacity of *polvilho azedo*. All analyses were performed in triplicate.

DISCUSSION

The general opinion of bakers reveals that *polvilho azedo* is a product without any standard of quality, which leads those professionals to establish a relation of fidelity with determined trademarks only, which in their opinion were the best. Based on their opinion about which *polvilho azedo* is the best, it was possible select the three best trademarks to be used in this study as a model of expansion property.

All results were organized for construction of co-relations to establish the best variables to preview the performance of expansion. Table 01 shows the value of co-relation coefficients for several parameters of the starch quality.

Table 1. Co-relation coefficient between expansion rate, and acid factor, pH, swelling power, specific volume and specific viscosity for several samples of polvilho azedo with high and low expansion performance and native cassava starch

Sour cassava starch quality parameter	Expansion rate(*) co-relation coefficient R
Swelling power	0.52
Acid factor	0.81
pH	0.92
Intrinsic viscosity (**)	0.93
Specific volume	0.97

** Data from samples of native cassava starch (n = 6) and sour cassava starch produced in laboratory (n = 20) and commercial trademarks (n = 17). ** Intrinsic viscosity determined to: Native cassava starch (n = 5); commercial sour cassava starch (n = 3)*

The swelling power is a variable with important relation with the expansion rate, in accordance with preliminary study[23]. Due to starch granule de-polymerization sour cassava starch shows low swelling power, which increases expansion. When analyzing all the data with high density of results of all the samples, commercial *polvilho azedo*, *polvilho azedo* produced in laboratory, and native cassava starch, the co-relation coefficient between expansion rate and swelling power is ~ -0.34. Meanwhile, when analyzing more specifically only the commercial *polvilho azedo* and native cassava starches, it becomes evident that the co-relation is higher. The decrease in swelling power is inversely related with expansion rate, with a co-relation value of ~ -0.51. Swelling power could be a variable indicated to predict the expansion performance of *polvilho azedo*. However, studies with the variables: pH, acid factor, specific volume and intrinsic viscosity would offer better answers to predict the expansion performance of *polvilho azedo*. There are several published works about polymeric networks in several kinds of dough and natural and synthetic polymers, but none about the relation between swelling power and expansion rate of sour cassava starch, *polvilho azedo*.

The reduction in pH value, as consequence of fermentation, is a known event in the *polvilho azedo* production process. This fermented product shows a low characteristic pH zone when compared with native cassava starches. The relation between pH and expansion rate for all the samples analyzed shows co-relation coefficient of ~ -0.68, which proves that expansion increases with the reduction of pH.

The analysis of the relation of the expansion rate and pH, restrictive to native cassava starches and commercial *polvilho azedo* samples, co-relation coefficient (~0.92), reveals that pH is an important variable to predict the expansion performance of *polvilho azedo*.

This strong inverse relation between pH and expansion comprises a fact largely published about *polvilho azedo*, as a product with low pH[7,24-26].

Although the acid factor has been a variable which is little studied in the academic scenario it represents a volume of 0.1 N hydrochloric acid that is necessary for the starch dispersion pH to achieve the value of 3.0. Indirectly, it indicates the chemical nature of the starch granule in relation to its sensitivity to changes in pH. When comparing *polvilho azedo* to native cassava starch, which is a non-fermented product that has low acid factor, the acid factor can show the kind of chemical change that occurs after fermentation. Therefore, the acid factor is a variable that increases with the increase of the expansion rate.

Co-relation coefficient between expansion rate and acid factor has a lower value when all the samples in this work are compared. Because of the faster production of the *polvilho azedo* produced in laboratory, this co-relation can be dramatically changed when comparing only native cassava starches samples with commercial *polvilho azedo* of the bakers' preference.

This prospect improves when analyzing the relation between acid factor and expansion rate only with native cassava starches and commercial *polvilho azedo* samples, thereby achieving the co-relation coefficient of ~ 0.81. Several works have been published attempting to explain the expansion property of *polvilho azedo*, but none have been published about the relation between the expansion rate and the acid factor.

Attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy and chemometric analysis applied to the samples in study (unpublished data) indicated that absorption wavebands at 1650-1500 cm^{-1}, typical of nitrogen-containing compounds, revealed some important differences compared to native cassava starches. Since the acid factor analysis considers the pH reduction on titration with 0.1N HCl and because *polvilho azedo* has greater acid factor in comparison to native cassava starch, these results can show an indication of buffering nitrogen material, which prevents pH reduction and is predominantly present in the *polvilho azedo* samples.

Another variable to be considered is the specific volume that means the volume/weight relation of samples of specimen constituted by the starches in study. This variable is closely related to the expansion rate. Through an analysis of all the sample spectra, it is possible to achieve a high co-relation coefficient (r=0.89) between the specific volume and expansion rate.

This important co-relation increases (r=0.97) when analysis is performed only between native cassava starches and commercial *polvilho azedo* samples.

According to several authors[8,10-12] cassava starch fermentation in the *polvilho azedo* production process results in starch granule depolymerization. Intrinsic viscosity determination for the samples studied showed this change, with partial reduction of the polymerization degree of amylose and amylopectin. There is a high inverse co-relation (r = -0.93) between intrinsic viscosity and expansion rate, when analyzing native cassava starches and commercial *polvilho azedo* samples.

As consequence of this depolymerization, some authors have already published that there are some differences in amylose content in fermented and non-fermented starch granules. Therefore, according to our work and to unpublished data, there is no important relation between amylose content of native cassava and fermented starch granules. This fact leads to the conclusion that the bounds and the proximity amylose-amylose/amylose-amylopectin/amylopectin-amylopectin, and the relation of amylose in starch granule are sufficiently diverse to consider amylose as a parameter of reference to predict expansion performance of *polvilho azedo*.

Considering all the co-relations shown in this work, in crescent order, swelling power, acid factor, pH, intrinsic viscosity and specific volume could be useful as indicator to predict the expansion performance of *polvilho azedo*. Therefore, considering some of these analyses, producers of *polvilho azedo* and bakers could become dependent on laboratories to perform more elaborated analysis to predict the performance of *polvilho azedo* such as on intrinsic viscosity and swelling power. Today bakers and small industries of *polvilho azedo* use to make a sample of specimen, which can be quite similar to the specific volume evaluation. Therefore, this present work suggests that pH and acid factor should be used to simplify the prediction of the performance of *polvilho azedo*.

The results of the samples studied in this work show that the commercial *polvilho azedo* with more

Table 2. Expansion rate and percent of weight loss of several samples of *polvilho azedo* with high and low expansion performance, compared to native cassava starch

Samples	Expansion rate	Loss of Weight (%)
Native Cassava Starch		
	1.3	34
	1.4	28
	1.3	30
	1.4	31
	1.5	30
	1.5	32
n=6		
Average±SD	**1.4±0.09**	**30.83±2.04**
Sour cassava starch produced in laboratory		
Low performance		
	1.4	29
	1.5	32
	1.4	31
	1.3	32
	1.4	32
	1.5	36
	1.5	39
	1.5	30
	1.4	34
n=9		
Average±SD	**1.43±0.07**	**32.72±3.11**
High performance		
	2.2	41
	2.1	40
	2.2	42
	2.1	40
	2.1	39
	2.1	40
	2.2	42
	2.2	41
	2.2	42
	2.2	41
	2.3	40
n=11		
Average±SD	**2.7±0.06**	**40.73±1.01**
Commercial sour cassava starch		
	2.0	39
	2.0	41
	1.9	41
	1.9	38
	1.9	39
	1.9	38
	1.9	39
	2.1	37
	2.0	41
	1.9	40
	1.9	37
	2.1	39
	2.2	45
	2.2	40
	2.1	42
	2.1	40
n=17		
Average±SD	**2.01±0.11**	**39.82±1.98**

desirable expansion property has pH values between 3.47 and 4.18, and acid factor values between 3.17 and 4.36. These minimum and maximum values can be useful as limits to predict the performance of *polvilho azedo*.

All *polvilho azedo* samples produced in laboratory showed a good performance in expansion property, however those samples showed acid factor values lower than the minimum limit suggested in this work. This method[25] is different from the one traditionally used for production of *polvilho azedo* because it is a rapid method, requiring only half the time of the traditional method. To predict the performance of *polvilho azedo* produced by this rapid method, pH and specific volume could be used as variables.

Additionally, the weight of the samples of specimen can also be related with their expansion because the weight of the cooked product made with a good *polvilho azedo* is lower than a *polvilho azedo* with low expansion performance. Considering the method used in this work for determination of specific volume[21], we can find a high amount of data, enough to establish a relation between the lost of weight sample of specimen and the expansion rate. According to this consideration, expansion can be estimated by converting the difference in weight, before and after cooking, into percentage of weight loss during cooking. Table 2 shows data obtained for weight loss in products with better performance, compared to low performance and native cassava starch. Therefore, it is possible to predict that a good *polvilho azedo* must show weight loss around 40 to 45% after cooking. This evaluation is independent of pH or acid factor determination, but it is advantageous in comparison to the traditional method used in bakeries because it uses only a minimal quantity of sample, oven, and scale and also because it reduces time of assay.

The determination of pH, acid factor, and weight loss can be used to choose a good *polvilho azedo*. The intension is that this fermented product is capable of being developed to establish lower variations between several fermentations by the same producer and also to facilitate the availability of a greater number of trademarks preferred by bakers. At the same time, familial industries of *polvilho azedo* need the simplest methodology to predict the quality of their products.

Studies on *polvilho azedo* quality control or with the aim to improve the process and the product should be considered in view of the increasing opportunities to preserve the small and familial industries dedicated to production of *polvilho azedo*. Just as much as several wine and cheese producers worldwide, the industry of cassava starch fermentation must be developed and valued.

CONCLUSION

According to the opinion of bakers who use *polvilho azedo*, there is great difficulty to find a good trademark of this product. Consequently, they establish a relation of customer fidelity when they eventually find a good product.

Good performance of *polvilho azedo* means a final product with good expansion capacity. Among several quality variables such as intrinsic viscosity, swelling power, specific volume, pH, and acid factor, only the last two could contribute to predict performance of *polvilho azedo* for products obtained through the traditional process, which takes long time for fermentation.

Polvilho azedo produced by rapid fermentation from fermented native cassava starch does not respond similarly to traditional *polvilho azedo*, due to the low acid factor compared to higher acid factor obtained in traditional products. Therefore, the inclusion of the percentage of weight loss during cooking can be a good variable, along with pH and acid factor, to establish a prediction of the expansion performance of *polvilho azedo*.

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