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PHYSICO-CHEMICAL PARAMETERS EVALUATION OF MANIHOT CASSAVA FLOURS MARKETED IN SANTARÉM – PA

AVALIAÇÃO DE PARÂMETROS FÍSICO-QUÍMICOS DAS FARINHAS DEMANDIOCA COMERCIALIZADAS EM SANTARÉM – PA

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ABSTRACT: The state of Pará, especially Santarém, is a large producer of cassava root, most of this production is destined for the manufacture of flour. The present work aims to evaluate the physicochemical quality parameters of the flour sold in Santarém. 149 samples were collected in 5 municipal fairs and later the granulometry, pH, alcoholic acidity, moisture content and ash in these samples were evaluated in comparison with Brazilian legislation. It was observed that no sample was classified as fine flour, and only 3 samples had moisture above the permitted level. However, in relation to ash and alcoholic acidity contents, 37.6% and 100% of the samples presented values above the allowed. In addition, the samples showed great variations in the parameters evaluated, which indicates a lack of standardization in the production stages and in the storage of the product.
Keywords: Manihot esculenta Crantz, pH, alcoholic acidity, ash content, granulometry.

RESUMO: O estado Pará, com destaque para Santarém, é grande produtor de raiz de mandioca, a maior parte dessa produção é destinada à fabricação de farinhas. O presente trabalho visa avaliar parâmetros físico-químicos de qualidade da farinha comercializados em Santarém. Foram coletadas 149 amostras em 5 feiras municipais e posteriormente foram avaliados a granulometria, pH, acidez alcoólica, teor de umidade e cinzas nessas amostras em comparação a legislação brasileira. Foi observado que nenhuma amostra se enquadrou como farinha fina e, apenas 3 amostras apresentaram umidade acima do permitido. No entanto, em relação aos teores de cinzas e acidez alcoólica, 37,6% e 100% das amostras apresentaram valores acima do permitido. Além disso, as amostras apresentaram grandes variações nos parâmetros avaliados o que indica uma falta de padronização nas etapas de produção e no armazenamento do produto.
Palavras-chave: Manihotesculenta Crantz, pH, acidez alcoólica, teor de cinzas, granulometria.

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INTRODUCTION

Cassava (*Manihot esculenta Crantz*) is a species of easy propagation that adapts even in soils with low fertility, which allows its cultivation in several poor regions of Africa and America. In Brazil, cassava cultivation stands out, with the North and Northeast regions being major producers (BAYOUMI *et al.,* 2010; ALBUQUERQUE *et al.*, 2009; EMBRAPA, 2018).

In 2020, the national production of root was 18,205,120 tons per planted area and harvested area, contributing with an income to the GDP (gross domestic product) of R$ 10,887,678.00 and in that same year only the state of Pará contributed with 20 % of the Gross Value of Production (GVP). Although production has shown an increasing drop since 2017, Pará has been the largest producer of cassava in Brazil since 2001, occupying the first place in the ranking in terms of production. More recently, in 2020, production was calculated at 3,813,369 tons, which is equivalent to 20.95% of national production (IBGE, 2021).

Until 2015, the municipality of Santarém showed a growing increase in cassava root production, becoming the second municipality with the highest national production until 2017. However, this production decreased until 2020 (IBGE, 2021). The main by-product of cassava in a production chain is flour. It is estimated that 22.1% of the national production of cassava is destined to the production of flour, 10.0% to the production of starch and 2.0% to consumption in natura. (MATTOS *et al.*, 2006). While in Pará more than 90% of the production of cassava root is destined to the manufacture of flour (MODESTO JÚNIOR and ALVES, 2021). This is because, in the North region, the average annual per capita consumption of flour is around 10.8 kg, while the national average is 2.3 kg and in other regions the average is even lower (IBGE, 2021).

It is important to highlight that according to Brazilian legislation (BRASIL, 2011), flour is the product obtained by grinding the edible vegetable part, which can undergo further technological processes. For classification and standardization purposes, it recognizes three types of flour that correspond to the Group: (1) Dry (does not go through the fermentation process); (2) Water (flour that goes through the fermentation stage), (3) Bijusada (irregular flakes appearance).

As for the determining characteristics of quality, "The granulometry is the distribution of particles and granules that make up the product, according to their sizes [...] it is an important quality aspect in the standardization of cassava flour manufactured by the artisanal process." (SILVA *et al.,* 2015). Through granulometry it is possible to determine the respective Class of each flour group, which can be Coarse, Medium or Fine.

Tapioca is considered a starchy product derived from cassava root, not being properly recognized as flour (SILVA *et al.*, 2012; COÊLHO, 2019). Normative Instruction No. 52 of November 7, 2011 establishes the maximum moisture content of up to 13% for dry and water flours to be commercialized (BRASIL, 2011). And the Normative Instruction nº 23 of December 14, 2005 determines the maximum limit of 15% of humidity for Tapioca (BRASIL, 2005). Brazilian legislation also establishes that the ash value must be equal to or less than 1.4% for water and dry group of flours and less than 0.50% for Tapioca.

These parameters are directly associated with the processing and proper storage of the flour until it reaches the consumer (CHISTÉ and *et al.*, 2007; ANDRADE, 2014). As flour in the municipality of Santarém is sold mainly at municipal fairs, the present work aims to evaluate physicochemical parameters of flour quality in order to compare with the values established in Brazilian legislation. In addition, as there is no standardized classification in relation to the class or group to which the flour belongs, we seek to classify them in terms of origin and granulometry.

MATERIALS AND METHODS

The flour samples were acquired in the five main municipal fairs of Santarém. They correspond to the Mercadão 2000 (2º42'01" S and 54º72'95"W), Aeroporto Velho (2º43'82"S and 54º71'31"W), Cohab (2º44'45"S and 54º70'33"W), Prainha (2º43'65"S and 54º70'50" W) and Candilha (2º42'03"S and 54º71'65"W) fairs, during the months of January and February 2020. The samples were grouped into four categories according to the classification of the group and class in which they belong: A (Tapioca/Sagú) with 30 samples, B (Dry/Coarse) with 66 samples, C (Dry/Medium) with 39 samples and D (Water/Coarse) with 14 samples, totaling 149 samples.

The mass of all samples was standardized at approximately 400 g for further analysis. All analyses, with the exception of granulometry, were performed in triplicate.

The granulometric analysis and classification was carried out following Normative Instruction No. 52 of November 7, 2011 and Normative Instruction No. 23 of December 15, 2005 of the Ministry of Agriculture, Livestock and Supply (BRASIL, 2011).

With the exception of Tapioca, 100 g of each sample were separated into 250 mL beakers for later manual sieving using

1 mm and 2 mm mesh sieves, respectively. The granulometry was measured accordingto the following formula:

$$Granulometry = \frac{m_r}{m_t} x$$

where mr is the mass retained in the mesh in g and mt is the total mass of the sample in g.

The dry flour is classified into three groups: fine (100% of the granules pass through the 2 mm mesh and up to 10% are retained in the 1 mm mesh), coarse (more than 10% of the granules are retained in the 2 mm mesh) and medium (when it does not fit the other classifications).

The flour from the water group was also divided into three classes, namely: fine (up to 10% of the granules are retained in the 2 mm mesh sieve), medium (10 to 15% of the granules are retained in the 2 mm) and coarse (more than 15% of the granules are retained in a 2 mm mesh sieve).

The flour related to the Tapioca group is not graded according to the granulometric classification, due to its different preparation method. And it has two classes: (1) Tapioca granulated, the granules have an irregular polyhedral shape of different sizes; (2) Pearl Tapioca or artificial sago, the granules have irregular spherical shapes, also of different sizes.

As for the determination of moisture, the method proposed by the Adolf Lutz Institute (2008) was followed with some modifications. Approximately 7.0 g of each sample were separated into beakers, after which the samples were taken to the oven (model 400 – 7D Series 29401/12, Ethik Technology, São Carlos, Brazil) with forced air circulation at 105 °C during 24 h. After this period, the samples were cooled and the dry mass was measured as follows:

$$Moisture(\%) = \frac{M_u - M_s}{M_u} x\ 100$$

Where Mu is the mass of the wet sample, Ms is the dry mass of the sample.

The analysis of ash contents was performed according to the method proposed by the Adolf Lutz (2008). To determine the ash contents, approximately 2.0 g of each sample previously dried in an oven at 105 ºC for 24 h were measured in crucibles, which were subsequently taken to a muffle furnace (Jung model LF0612, Blumenau, Brazil) at 600 °C for 6 h. After this time, the sample was cooled and the ash percentage was measured:

$$Ashes\ (\%) = \frac{M_{ac} - M_c}{M_s} \times 100$$

Where Mc is the mass of the empty crucible, Mac is the mass of the crucible with ash and Ms is the sample of the dry sample.

The potentiometric process was used to evaluate the pH, using a digital pH meter (model HI2020 HANNA, Barueri, Brazil) previously calibrated with buffer solutions at pH 4.0, 7.0 and 10.0 (Adolfo Lutz, 2008). In 100 mL beakers, 5 g of each sample and 50 mL of deionized water were added. The samples were left to rest for 1 h and after that, the pH measurement was performed.

The determination of alcohol-soluble acidity was carried out following the Analytical Standards of the Instituto Adolfo Lutz (2008) for flours and similar products. Approximately 1,25 g of each sample was measured along with 25 mL of 96 ºGL ethyl alcohol were added to 125 mL beakers and allowed to stand for 24 h. Afterwards, the samples were titrated with 0,0096 mol L-1 NaOH solution standardized with potassium bifthalate, using 0,1 g of phenolphthalein dilued in 10 ml ethyl alcohol as an indicator. The blank was prepared using 20 mL of 96 ºGL ethyl alcohol. The titratable acidity was calculated using the formula:

$$Ac = \frac{(V - V') \times f}{P \times c} \times 100$$

where V is the volume of NaOH solution spent, V' is the volume of NaOH solution spent on the blank titration, f is the factor of the 0.0096 mol L-1 NaOH solution, P is the mass of the sample used and c the factor of correlation which is equal to 100 for 0,01 mol L-1 NaOH solution.

Statistical analysis was performed using the GENES Software version 1990.2020.28, where the analysis of variance of triplicates, standard deviation and Tukey test for comparing means at a 5% probability level were performed.

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RESULTS

The results of the particle size analyses, pH, total acidity and moisture and ash contents are expressed in Table 1.

Table 1: Result of the analysis of granulometry (classification), humidity, pH, ash and titratable acidity.

Sample	Popular Name	Group/Class	Humidity %	pH	Ash %	Aciditymeq/100g
A	Tapioca	Tapioca/Sagú	9,86±0,94[a]	4,56±0,26[b]	1,21±1,14[b]	8,94±1,77[b]
B	White, manioc, coarse,fine,butteryflour, medium	Dry/Coarse	9,23±2,29[a]	1,46±0,65[c]	5,54±0,53[a]	16,33±7,81[a]
C	Fine white, buttery, farofa, common flour	Dry/Medium	8,72±1,54[a]	5,41±0,55[a]	1,47±0,48[b]	18,75±8,9[a]
D	Puba, common flour, coarse, sifted, puba medium	Water/Coarse	8,96±1,13[a]	5,2±0,42[a]	1,65±1,39[b]	15,96±6,05[a]

Results of ash percentages expressed on a dry basis. Means followed by the same letter in the column do not differ statistically by Tukey's test at the 5% probability level.

Based on the granulometric analysis, all samples of flour D'água were classified as Coarse. While the flours of the Dry group were classified as Medium (39 samples) and Coarse (67 samples). All samples of Tapioca were classified as Tapioca or Sagú.

Moisture contents ranged from 5.2% to 25.7% for flours from the Dry group and from 6.9% to 10.0% for flours from the D'água group. In Tapioca, the moisture content was in the range of 8.5% to 12.5%. Of the total of 149 samples analyzed, 13% for dry and water flour and 15% for Tapioca presented contents higher than the maximum allowed by Brazilian legislation.

The acidity values in the range of 7,1 to 14,1 meq. NaOH100g-1 of Tapioca sample were observed, in D'água flour samples the acidity ranged between 7.7 and 30.1 meq. NaOH 100g-1 of sample and between 7,1 and 54,4 meq. NaOH100g-1 of flour sample from the dry group. With the exception of Tapioca, all samples showed high acidity, a value above the limit established by legislation, up to 3,0 meq.NaOH100g-1 for dry flour and up to 5,0 meq. NaOH100g-1 for flour of water group.

The pH ranged between 4.04 and 5.11 in samples of Tapioca, between 4,59 and 5,75 in flours from the water group and between 4,11 and 6,30 in flours from the Dry group.

Brazilian legislation (BRASIL, 2011) establishes that the ash value is equal to or less than 1,4% for water and dry flours and less than 0,50% for Tapioca. In this aspect, it was observed that the ash percentage varied between 0,08% and 3,85% in Tapioca and 18 of the 30 samples of this flour presented values above 0,50%.The ash content varied between 0.84% and 6.13% in samples from the Dry group and between 0,76% and 6,36% in samples from the D'água group. Of the total of 119 samples of these two classes, 56 presented ash contents higher than allowed by legislation.

The standard deviation allows us to observe how the samples of each group differed from each other. The greater the standard deviation, the greater the variability of the samples. This is indicative of the lack of standardization during the processing and obtaining of the flour.

The Tukey teste (Table 1) indicates that not much variability between groups A, B, C and D.

DISCUSSION

In Table 1, it contains the 'popular' of each sample collected. This 'popular name' refers to how familiar the seller or producer is with the particular type of flour in the region where it was produced. There is enormous variation in the identification of flour between fairs and between producers. It is possible to find flours with similar aspects and different names.

Although many producers and dealers determine several samples as Fine, in the classification by sieving, no sample belonging to this class was found.This highlights the lack of standardization among producers during the production process, screening and selection of the processed product.

The flour of the Tapioca group, due to its method of preparation and its unique characteristics of irregular granules, is classified according to the shape of its granules and not by sieving. It was found that all samples belong to the class of pearly Tapioca or sagú (BRASIL, 2005). Specifically in the North region, Tapiocaconsists of an expanded sagú, which is one of the main consumed products obtained from cassava starch (SILVA, 2011). The purpose of the granulometry for Tapioca is just to determine if it is granulated or pearly, this is not a parameter that has a great weight when it comes to quality, but it is important for the visual standardization of the product.

For the evaluation of the moisture content in the samples, Normative Instruction Nº 52 of November 7, 2011, was taken into account, which establishes the maximum moisture content of up to 13% for dry and water flours to be marketed (BRASIL, 2011). And Normative Instruction Nº 23 of December 14, 2005, which determines the maximum limit of 15% moisture for Tapioca (BRASIL, 2005). Humidity values above these limits favor the growth of microorganisms and consequently deterioration of the product in a short period of time (CHISTÉ et al., 2007). While lower humidity values are favorable to maintain the stability and durability of the product, hindering the development of microorganisms and preventing product deterioration (SILVA et al., 2015; DIAS and LEONEL, 2006).

The step of the production process responsible for reducing the moisture of the dough for the preparation of flour is pressing and drying. Pressing "drains excess water, facilitates operation and drying (ÁLVARES et al., 2009). Thus, the low moisture levels achieved depend directly on good drying and adequate storage (ÁLVARES et al, 2013).

The samples that presented low humidity, according to Dias and Leonel (2006), can also be explained by the complementary roasting process to which it is submitted, in which much of the moisture is removed. These authors found moisture contents in flours of the dry and water groups in the range of 3.10% to 11.57%, values similar to those found in this work. Other authors such as Álvareset al. (2013), Souza et al. (2008) and Silva (2011) also found values close to those found in this work.

Acidity is an important parameter as it confers sensory characteristics. This can manifest itself in higher or lower values (high acidity or low acidity), according to its production process, always paying attention to the typical consumption patterns and habits of the respective region (BRASIL, 2011).

The Legislation makes the consumption unfeasible for values above the established, since it is recommended to commercialize flours with low acidity. The MAPA (Ministry of Agriculture, Livestock and Supply) regulates that the acceptable amount of acidity for flours of the dry group is up to 3.0 meq.NaOH100g-1. While for flours from the D'water group, the acceptable limit is up to 5.0 meq.NaOH100g-1. Values below the established limit, the acidity is considered low and values above the acidity is considered high.

It was found that, in terms of acidity, with the exception of Tapioca in which acidity is not a limiting factor for quality, all samples did not meet the standards of the legislation. Chisté and Cohen (2010) in their research with flours from the Belém region evaluated the acidity of ten samples of flour of water group, of which nine were within the standards determined by Brazilian legislation. These values ranged from 4.76 to 10.21 meq.NaOH for 100 g of sample.

In another work, Chisté*et al.* (2007) also found in their analysis higher acidity values in cassava flour, which ranged from 6.50 to 10.10 meq.NaOH for 100 g of sample.The increase in acidity occurs for two main reasons: one is the fermentation of cassava roots that are left submerged in water for a certain period, and the other due to the concentration of acids due to the roasting process (Chisté*et al.*, 2007).

In flours of the water group, "the beginning of the fermentation process is accompanied by a rapid drop in the concentration of dissolved oxygen, caused by aerobic amylolytic bacteria, capable of consuming oxygen and producing organic acids such as lactic, buritic, acetic, among others (BEZERRA, 2000).

Acidity is also indicative of the time and intensity of root fermentation. The greater the acidity, the greater the intensity of fermentation or time of the pubic process of the roots, in the case of flours (DIAS and LEONEL, 2006).

In flours of the dry group, the process to obtain the flour should be continuous, without interruptions. However, it is observed that in some Flour Houses, this process suffers interruptions. The crushed cassava mass is exposed overnight at room temperature, which can result in fermentation and consequent increase in acidity (Chisté*et. al.*, 2006).

The pH is a parameter that is related to the acidity found in the samples, as it is an indication of potential acidity. Based on pH, flour samples can be classified into three groups: low acid (pH > 4.5), acidic (pH 4.5 to 4.0) and very acidic (pH < 4.0) (SOUZA *et al.*, 2008).

Dias and Leonel (2006) found values close to pH in the range of 4.16 to 4.64 in flours from the D'água and Seca groups and an average value of 4.31 in Tapioca sold in Pará. Silva (2011) verified that in samples of Tapioca commercialized in Santarém, the average pH was 5.75. It is worth noting that the pH and acidity of the flour also depend on the variety of cassava, on adequate storage, among other parameters (SILVA, 2011).

For Brazilian legislation, pH is not a determining parameter that makes the consumption and commercialization of cassava flour unfeasible. However, it is a very important factor in food conservation, as it limits the development of microorganisms, most of which grow at a pH greater than 4.5 (SUAREZ*et al*., 2013).

As a result of acidity, pH can also influence the oxidative browning of plant tissues, interfering with visual characteristics, flavor and aroma (LEME *et al*., 2007), as a consequence it interferes with the sensory quality of the flour.

During the production of cassava flour from the water group, the cassava roots are submerged in water for a period of 4 days, resulting in their fermentation (CHISTÉ*et al*., 2007). This characteristic in the production step results in an increase in the amount of acids and a decrease in the pH value. In flours of the Dry group, which do not undergo the fermentation process, the acidity and pH can increase according to the pressing time. Álvares*et al*. (2009) when analyzing the behavior of the dough during the pressing phase for flour production, found that there was a gradual reduction in pH and an increase in acidity according to the pressing time.

This is because after crushing the cassava mass, the mass usually remains stored at room temperature around 25 to 35 ºC in the North of the country, and pressed the next day, adding to the mass moisture, results in favoring the occurrence of fermentation, increasing the acidity concentration and reducing the pH (CHISTÉ*et al*., 2007). Pressing is done to remove excess moisture from the dough, both in the production of flour from the D'água group and from the Dry group.

The ash corresponds to the fixed mineral residue, that is, mineral material that is present in the product (BRASIL, 2011). The ash content is directly related to the content of non-volatile metals and minerals contained in the samples (ANDRADE, 2014).

The ash content in cassava flour may be related both to the intrinsic characteristics of the roots and to the manufacturing process (CHISTÉ and COHEN, 2010). According Souza*et al*. (2008) ash values higher than allowed may be indicative of significant levels of Ca, P, Fe and Mg or product contamination by foreign materials in failures during the processing steps.

According to Ros*et al*. (2013), cassava roots showed a significant response to fertilization, regarding the addition of P, Ca, K and Mg, with an increase in root productivity. These minerals belong to groups that may not be fully volatilized and remain present in the sample even after incineration. It can increase the percentage of fixed mineral residue.

If the amount of minerals in the soil increases, it is expected that their availability to the plant will also increase. This factor is reflected in the mineral composition in the roots and consequently in the composition of its by-products, such as flour.

The variation observed (Table 1) in the analyses is due to process of obtaining the flour, which can vary a lot because the production of flour in the region is still artisanal and differs among producers, as it originates from different places within the Santarém region; the way in which the flours are stored, conditions are not always ideal; the storage time, it is possible to lose some chemical properties such as starch and accumulate acids when associated with high temperature and humidity; and among other conditions related to production and storage.

Attention needs to be made to improve plant health care in the production and marketing phase. Handling in cleaner environments, free from the presence of animals and insects, storage in dry and airy places, marketing of the properly package product are improvements that can be made in order to get better quality of the final product.

Producers need to be heeded for the physical and chemical parameters that enable the commercialization of the product. And follow the instructions proposed by the Legislation.

CONCLUSIONS

The identification of flours in the Santarém region is not similar between producers and resellers. Although many samples were identified as Fine flour, none fit into this class that indicates the lack of existing standardization when obtaining the product.

Only 3 samples showed moisture levels above the allowed, which may be associated with the correct preparation of the same or even the samples have been obtained in the Amazon dry period.

Regarding acidity, with the exception of Tapioca, all samples showed levels above those allowed by Brazilian legislation, which may be associated with fermentation during the manufacturing and storage process or acid concentration during roasting.

As for the fixed mineral residue, approximately 56 samples showed levels above the permitted levels, which is directly associated with the nutrients present in cassava or possible contamination due to failures in handling during the processing of the product.

The results in general show that there is a lack of standardization during the production of flour and contamination of the samples during the processing steps. And they indicate the need to pay attention to the inspection of products and the need for proper training of producers by competent bodies to improve the manufacture and handling of flour.

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