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## QUALITY PARAMETERS AND PHYSICAL-CHEMICAL CHARACTERIZATION OF COPIOBA CASSAVA FLOUR: ELEMENTS TO ITS GEOGRAPHICAL INDICATION

### ATRIBUTOS FÍSICO E FÍSICO-QUÍMICOS DA FARINHA DE MANDIOCA PRODUZIDA NO VALE DO COPIOBA/BRASIL: UM ESTUDO COMPLEMENTAR À INDICAÇÃO GEOGRÁFICA

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**Abstract:** The aim of this study was to characterize the Copioba and Common cassava flours produced in Copioba Valley (located in Recôncavo Baiano, Brazil) and outside of it, verifying compliance to Identity and Quality Parameters (IQP), in addition to physical-chemical and morphological compositions, contributing to a request for Geographical Indication (GI) product. 100% of the A and C met the requirements for moisture and ash. The exception was the fiber content, in which 20% samples of B did not attend, and of starch content, in which 20% (A) and 30% (B and C) presented values lower than the parameters of the legislation ( $\geq 80\%$ ). The Scanning Electron Micrographs also showed morphological features differentiate to A. The A is mainly Type 1, with lower classification as Out of Type, while those of C are mostly Type 3 and Out of Type. The B is distributed in the 4 Types. These parameters together are considered differentiating factors of Copioba flours regarding flour produced in other regions, which, along with the climatic characteristics and particular historical notoriety recognized can support a request for GI, bringing social and economic benefits for the region and appeal for the safe use of the product.

**KEYWORDS:** *Manihot sculenta* Crantz; Cassava flours; Copioba Valley; Recôncavo Baiano.

**Resumo:** O objetivo deste estudo foi caracterizar as farinhas de copioba e mandioca produzidas no Vale de Copioba (localizado no Recôncavo Baiano, Brasil) e fora dele, verificando a

conformidade com os parâmetros de identidade e qualidade (PIQ), além das composições físico-químicas e morfológicas, contribuindo para um pedido de indicação geográfica (IG) do produto. 100% das amostras A e C atendiam aos requisitos de umidade e cinzas. A exceção foi o teor de fibras, em que 20% das amostras de B não corresponderam, e o teor de amido, em que 20% (A) e 30% (B e C) apresentaram valores inferiores aos parâmetros da legislação ( $\geq 80\%$ ). As Micrografias Eletrônicas de Varredura também mostraram características morfológicas diferenciadas de A. A amostra A é principalmente do Tipo 1, com classificação mais baixa como fora do tipo, enquanto as amostras C são principalmente do Tipo 3 e fora do tipo. As amostras B estão distribuídas nos 4 tipos. Esses parâmetros juntos são considerados fatores diferenciadores das farinhas de Copioba em relação às farinhas produzidas em outras regiões, as quais, juntamente com as características climáticas e a notoriedade histórica reconhecida, podem apoiar uma solicitação de IG, trazendo benefícios sociais e econômicos para a região e apelando ao uso seguro do produto.

**PALAVRAS-CHAVE:** *Manihot sculenta* Crantz; Farinha de Mandioca; Vale do Copioba; Recôncavo Baiano.

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## INTRODUCTION

Brazil is the main center for the diversification of cassava (CARVALHO, 2002), being one of the major world producers, with annual production of 27.7 million tons, 7.6% of world production (FAO, 2018).

The basic production process of flour is summarized in the following steps: harvesting, transportation, peeling, washing, grating, pressing, crushing, roasting, sorting and packaging (FIALHO and VIEIRA, 2011). Currently, most cassava roots in Northeast Brazil is processed as flour, and the production chain for this flour is characterized by the use of family farming work, in hundreds of small units called cassava flour houses (“casas de farinha”) (SILVA et al., 2017). In Bahia State, the region of the Recôncavo Baiano has favorable conditions for the cassava cultivation, whereas to geographic location, rainfall, climate and suitable soils (RIBEIRO et al., 1990).

As a historic region for flour production, the Copioba Valley, is known for the production of cassava flour with distinguished quality. The Copioba Valley is located south central region, watching the Copioba river and cutting the municipalities of Nazaré, Maragojipe and São Felipe. The prestige of the name Copioba led to its exploration, associating the name as a form to obtain more appreciation and relevance (MATOS et al., 2012). Due to the notoriety flour produced in this region, the procedure used for its production was spread from out Copioba Valley, being produced today in all Recôncavo Baiano (DRUZIAN and

NUNES, 2012; DRUZIAN, MACHADO and SOUZA, 2012; MATOS et al., 2012).

The valorization of handmade farms by consumers takes into account the socio-cultural aspects in the environments in which cassava flour is manufactured and favors the development of local communities (OLIVEIRA et al., 2018). “Copioba flour” from Copioba Valley is produced by artisanal techniques traditionally used in the region, the “flour houses” are semi-mechanized and historically recognized as top quality flour with peculiar characteristics that can be easily identified by the final consumer.

Products that are eligible for geographical indication (GI) must meet the identity and quality standards laid down by legislation. Products that are eligible for GI must comply with legislation aimed at the producers’ organization, the processing methods and the quality criteria for production standardization. (BRANCO et al., 2013; SILVA et al., 2017). Regions for which the processes of GI are recognized there is an improvement in the local economy, including an increase in value-added products as well as further activities such as tourism (DRUZIAN and NUNES, 2012; DRUZIAN, MACHADO and SOUZA, 2012).

The produced by small farmers represent 70-80% of total cassava flour in the country, aimed primarily at the local trade. It is because the gap of standardization in production, and little control of the parameters of identity and quality, has hindered the access of the product to other markets (SILVA et al., 2017).

The consumers are becoming more and more conscious and sensitive to the environmental and ecological impact of food production and distribution. Protection of collective intellectual property and food innovation can lead the path of ethics and sustainability. The objective of this study was to characterize the Copioba and Common flours produced in Copioba Valley and out of it, verifying compliance to Identity and Quality Parameters (IQP), in addition to physical, physical-chemical and composition morphological, contributing to a request for GI product.

## MATERIAL AND METHODS

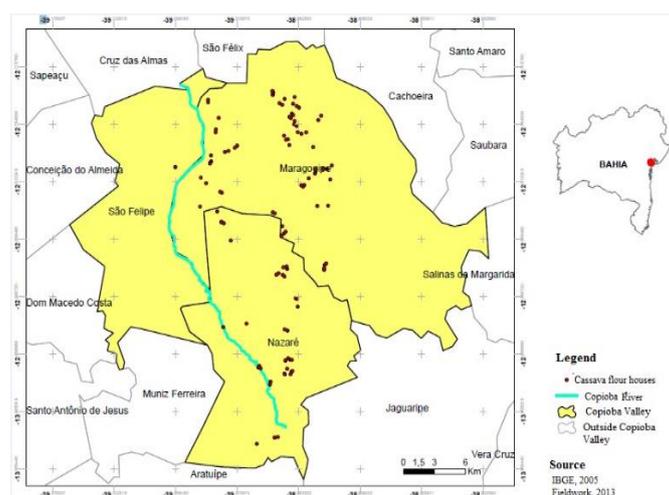
### Material

### Sampling and Location

A total of 30 samples, of which 10 samples of Copioba type flour (A) and 10 samples of Common type flour (B), were collected in the flour houses of Copioba Valley/BA. Another 10 samples of Copioba flours were collected in cities outside the Copioba Valley/BA (C) (Table 1). All samples were produced by the traditional method with mechanized steps. An exploratory study was recorded and identified geographically flour houses in the Copioba Valley (Figure 1). Each treatment was represented by ten samples randomly collected by lot.

**Table 1.** Classification and origin of cassava flour collected in the Copioba Valley and in cities outside the Copioba Valley/BA.

Sample	Classification	Origination
A1, A2, A3, A4, A5, A6, A7, A8, A9, A10	Copioba	Copioba Valley
B1, B2, B3, B4, B5, B6, B7, B8, B9, B10	Common	
C1, C2, C3, C4, C5, C6, C7, C8, C9, C10	Copioba	Outside Copioba Valley (Couties Muniz Ferreira and Aratuípe)



**Figure 1.** Location of the cities of Copioba Valley and outside Copioba Valley, in State of Bahia, Northeast of Brazil. (Source: Adapted from information from the Instituto Brasileiro de Geografia e Estatística, 2005).

### Methods

The parameters of the legislation in

Normative Instruction N°. 52/2011 (BRAZIL, 2011), analyzed in this study

were: moisture content, ash content, crude fiber, acidity, starch content, grain size and dirt research. Others complementary parameters not provided for in legislation such as crude protein content, total lipid content, water activity and composition morphological. These parameters aid in the characterization of the flour samples, and consequently in their identity and quality.

#### **Identity and Quality Parameters (IQP)**

The moisture content of the flour was determined by the oven drying method (method 925.09) in which pre-weighed samples were placed over silica gel overnight and then oven-dried at 105 °C for 24 h. The moisture content was considered the percentage of weight loss in relation to the original sample (AOAC, 2005).

The ash content was calculated by high temperature incineration in an electric muffle furnace (550 °C) and expressed as a percentage of the initial sample weight (method 923.03) (AOAC, 2005). The overall content of crude fiber obtained by digesting the material in a solution of H<sub>2</sub>SO<sub>4</sub> 1.25% w/v for 30 minutes, then NaOH 1.25% w/v for 30 minutes (AOAC, 1995). The acidity was determined according to AOAC (1995) (method 942.15).

The starch content was determined according to the Analytical Standards of the Instituto Adolfo Lutz (IAL, 2008), which after hydrolysis of the sample, determining the concentration of starch is performed by titrimetric method using the Fehling reagent for determination of reducing sugar.

To determine the particle size of the flour (particle size), an electromagnetic stirrer (BERTEL<sup>®</sup>) was used. The agitator consists of nine mesh with openings of different diameters: 0.075 mm, 0.125 mm

0.250 mm, 0.425 mm, 0.710 mm, 0.850 mm, 1.00 mm, 1.40 mm and 2.00 mm. In all the determinations, 100 g of flour was placed and subjected to a uniform shaking for 15 min. The amount of sample retained on each sieve as weighed, and the retention was calculated in percentage.

The dirt research was reviewed by the official AOAC (1995) method 965.39B, consisting of acid hydrolysis and float. Flour samples were digested in hot HCl, filtered, washed with water, suspended in an oil phase oil-aqueous mixture, filtered. The material was examined under a stereoscopic microscope (OPTON, Mod – TIM-2B / TIM-2T), under magnification 40x, isolated dirt being identified, counted and classified into different categories (whole insects, insect fragments, by human textiles, fragments of plastic, glass, silica).

#### **Additional analyzes for characterization of samples**

The content of crude protein (method 926.86) was determined based on hydrolysis and subsequent distillation of the sample, by assessing the percentage of nitrogen in the sample Micro-Kjeldahl method. Factor was 5.46 x % N for conversion to protein (AOAC, 2005).

Total lipids were extracted using chloroform/methanol/ water as solvent, according to the methodology proposed by Bligh and Dyer (1959), with values expressed also as a percentage. Measurements of activity water (Aw) of the flour samples were determined in meter, decagon brand AQUALAB LITE<sup>®</sup>. Sample preparation and use of the apparatus was performed according to manufacture instructions.

The surface of the starch samples of cassava flour Copioba type and Common

type was observed using images from scanning electron microscopy (SEM), generated equipment (Model JEOL JSM-6390LU) Service for Electron Microscopy Research Center Oswaldo Cruz/FIOCRUZ Salvador- BA. The samples of flour were fixed on a double-sided tape, on a support of aluminum and metallized with a layer of gold. After metallization, the samples were observed with 1,600 x magnification and bar 10 µm, 20 µm, 50 µm and 500 µm.

### **Statistical analysis**

The results obtained from cassava flours samples were analyzed according to a completely randomized design with 30 treatments. In order to evaluate if there is a difference between the types and origin of cassava flour, for the three treatments, the randomization test was performed using the Kruskal-Wallis test statistic at 1% and 5% significance levels. The null hypothesis (H0) is that the samples come from the same population, that is, that there is no difference between the treatments. The results were analyzed using ASSISTAT software version 7.7 beta (INPI 0004051-2.2011). The moisture, ash, starch, crude fiber, acidity, crude protein, total lipids, activity water were done in triplicate.

### **RESULTS AND DISCUSSION**

Identity and Quality Parameters of cassava flour of type Common and Copioba from inside and outside the Copioba Valley and other complementary parameters of quality are described in Table 2 e 3. The IQP were compared to those established by legislation (BRAZIL, 2011).

Moisture is an essential parameter in the storage of cassava flour, with greater than 13% can provide levels of microbial growth and deterioration in short time (CHISTÉ et al., 2006), while lower

percentages are favorable to greater stability and shelf life. The moisture content ranged from 1.67 to 7.58% for samples from within Copioba Valley and for samples of Common flour into the Valley, the change was from 3.74 to 8.21%. For from 3.53 to 7.31% for those from Copioba outside the Valley (Table 2, Figure 2), with all samples in accordance with standards established by the Brazilian legislation (BRAZIL, 2011) (until 13%). There were no statistical differences among the analyzed groups.

Ash is defined as the total mineral material present in the product, with the stipulated maximum value of 1.4% (BRAZIL, 2011). The ash content ranged from 0.65 to 1.16% for samples Copioba flour from Valley, between 0.62 to 1.11% for samples of Common flour in the Valley, while the variation was from 0.46 to 1.15 % for those from outside the Valley (Table 2, Figure 2A). All samples met the standards required (BRAZIL, 2011). There were no statistical differences among the analyzed groups. Higher ash values can indicate excess sand, improper processing, such as incomplete washing and peeling.

As regards the crude fiber content (BRAZIL, 2011) sets the value of 2.3% on a dry basis, the maximum amount of raw fiber. The food fibers have the characteristic of complexing with components (toxic or not) and dragging it with the feces (RAUPP et al., 1999). The crude fiber content ranged from 1.7 to 2.19% for samples Copioba from Valley (100% conformity) and Common flours from Valley the variation was between 1.73 to 2.76%, and 20% of the samples with values higher the established values. For samples those from outside the Valley ranged from 1.79 to 2.23%, all the samples (100%) attended the established

values. (Table 2, Figure 2A). There were no statistical differences among the analyzed samples.

The current legislation (BRAZIL, 2011) determines the parameter of acidity due to the manufacturing process, where cassava flour for dry classification groups will be considered low acid values up to 3.0 meq NaOH.100 g<sup>-1</sup> or for high values above 3.0 meq NaOH.100 g<sup>-1</sup>. In this study found that the acidity ranged from 3.83 to 6.65 meq NaOH.100 g<sup>-1</sup> for Copioba samples from Valley, and for samples of Common flour into the Valley, the interval also was higher (1.73 to 6.30 meq NaOH.100 g<sup>-1</sup>). For Copioba outside the Valley higher interval (1.50 to 6.72 meq NaOH.100 g<sup>-1</sup>) (Table 2, Figure 2A), similar to Comom flour samples of into the Valley. There were no statistical differences among the analyzed groups.

These results showed that all 10 samples of flour Copioba from Valley, 100% are classified as "high acidity" (Table 2, Figure 2A). According to Dias and Leonel (2006), the acidity of cassava flour is a result of the mass fermentation process, the higher the acidity, the higher the exposure to fermentation. Studies with flours of high acidity presented higher scores in sensorial preference ordering tests (CHISTÉ and COHEN, 2010).

The samples analyzed, 8 samples (26.7%) do not comply with the legislation. Of these, 02 (20%) Copioba samples from Valley, 03 (30%) Common samples from Copioba Valley and 03 (30%) Copioba samples from outside the Valley. Mattos et. al (2012) found similar results (78.99% to 90.55%) for flours type Copioba marketed at fairs in Salvador, Bahia, Brazil.

The results obtained ranged from 0.64 to 1.07% for the Copioba samples from Valley, and for the Common flour samples from Valley, the variation was 0.81 to 1.09%. For from Copioba outside the Valley the variation was 0.89 to 1.20% (Table 3, Figure 2A), being lower than those obtained by Santos et al., (2012), which report levels of 1.60 to 3.30% for cassava flour marketed in the municipal market of Cruz das Almas, Bahia, Brazil. The protein crude content from Copioba samples from outside the Valley was higher than the Copioba samples from Valley ( $p < 0.05$ ). This difference between the groups may also be associated with the quality of the soil and the seasonal conditions of the production of cassava roots inside and outside the Valley

Cassava flour is a product that has low lipid content (CHISTÉ et al., 2007). The lipid content ranged from 0.35 to 0.73% for the Copioba samples from Valley and the Common flour samples from Valley the variation was 0.39 to 0.72%. For the samples from Copioba outside the Valley the variation was 0.37 to 0.60% (Table 3, Figure 2A). There were no statistical differences among the analyzed groups. Chisté et al. (2006) also reports that this characteristic may consider intrinsic characteristics of cassava roots.

In this study the water activity ranged from 0.18 to 0.50 for the Copioba samples from Valley, and the Common flour samples from Valley the variation was (0.27 to 0.66). The interval higher 0.22 to 0.47, for those Copioba outside the Valley (Table 3, Figure 2A). There were no statistical differences among the analyzed groups.

**Table 2.** Identity and Quality Parameters of cassava flour specified by legislation (Brazil, 2011).

Samples	Identity and Quality Parameters					
	Moisture (%)	Ash (%)*	Fiber crude (%)*	Acidity (meq NaOH.100g <sup>-1</sup> )	Starch (%)*	
Copioba Valley	A1	6.14±0.14	1.12±0.03	2.09±0.04	6.22±0.14	82.68±0.62
	A2	4.45±0.15	0.71±0.05	1.84±0.02	6.65±0.23	86.19±1.00
	A3	7.52±0.31	1.03±0.01	1.54±0.03	5.19±0.51	85.49±0.54
	A4	1.67±0.12	0.98±0.00	1.47±0.03	5.89±0.25	80.76±1.32
	A5	3.97±0.09	1.16±0.04	2.11±0.03	4.71±0.27	82.54±0.99
	A6	4.03±0.63	0.65±0.01	1.85±0.01	5.58±0.08	86.06±0.46
	A7	6.03±0.11	0.96±0.01	2.19±0.02	4.95±0.25	78.16±1.23
	A8	3.39±0.12	0.67±0.01	1.81±0.02	4.12±0.15	80.54±1.37
	A9	7.58±0.15	1.10±0.01	2.12±0.01	3.83±0.02	77.98±1.01
	A10	5.06±0.11	0.82±0.02	1.90±0.02	5.93±0.23	82.10±0.36
	Mean±SD	4.98±1.87 <sup>a</sup>	1.89±0.02 <sup>a</sup>	1.89±0.02 <sup>a</sup>	5.31±0.91 <sup>a</sup>	82.25±3.00 <sup>a</sup>
Range	1.67 - 7.58	0.65 - 1.16	1.47- 2.19	3.83 - 6.65	77.98 - 86.19	
Conformity <sup>1</sup>	100%	100%	100%	100% (high)	80.0%	
Common Valley	B1	6.10±0.16	0.70±0.04	1.91±0.01	5.33±0.10	80.81±1.39
	B2	4.30±0.28	1.11±0.07	2.10±0.02	4.78±0.13	82.14±0.53
	B3	4.40±0.78	0.76±0.01	2.24±0.03	6.04±0.17	83.55±1.19
	B4	4.96±1.34	0.84±0.03	2.27±0.04	6.30±0.15	81.12±1.22
	B5	6.83±0.15	0.78±0.01	2.19±0.03	3.70±0.21	79.45±0.77
	B6	3.74±0.17	0.98±0.04	2.16±0.04	4.47±0.22	82.51±0.76
	B7	7.49±0.25	0.88±0.01	1.73±0.08	4.93±0.12	76.78±1.32
	B8	7.53±0.08	0.71±0.09	2.21±0.02	6.09±0.33	77.19±1.13
	B9	8.21±0.09	0.74±0.01	2.76±0.03	1.73±0.14	80.03±0.37
	B10	8.10±0.07	0.62±0.03	2.36±0.01	1.86±0.06	81.34±1.17
	Mean ±SD	6.17±1.70 <sup>a</sup>	0.81±0.15 <sup>a</sup>	2.19±0.27 <sup>a</sup>	4.52±1.65 <sup>a</sup>	80.49±2.19 <sup>a</sup>
Range	3.74-8.21	0.62-1.11	1.73-2.76	1.73-6.30	76.78-83.55	
Conformity <sup>1</sup>	100%	100%	80%	20.0% (low), 80.0% (high)	70.0%	
Copioba outside Valley	C1	3.53±0.08	0.68±0.02	2.09±0.01	5.41±0.55	73.47±1.20
	C2	6.93±0.19	0.91±0.05	2.11±0.08	1.70±0.12	81.08±1.61
	C3	5.82±0.32	0.85±0.01	2.10±0.09	1.50±0.22	85.22±1.14
	C4	4.02±0.28	1.15±0.02	2.23±0.01	5.19±0.32	80.92±1.17
	C5	4.39±0.50	0.46±0.02	1.83±0.02	6.65±0.09	71.18±1.07
	C6	5.26±0.49	0.78±0.06	2.01±0.04	6.05±0.11	80.31±0.91
	C7	7.31±0.10	0.94±0.05	2.12±0.03	2.02±0.10	79.86±1.52
	C8	5.29±0.45	1.00±0.03	2.13±0.02	6.72±0.08	83.89±0.31

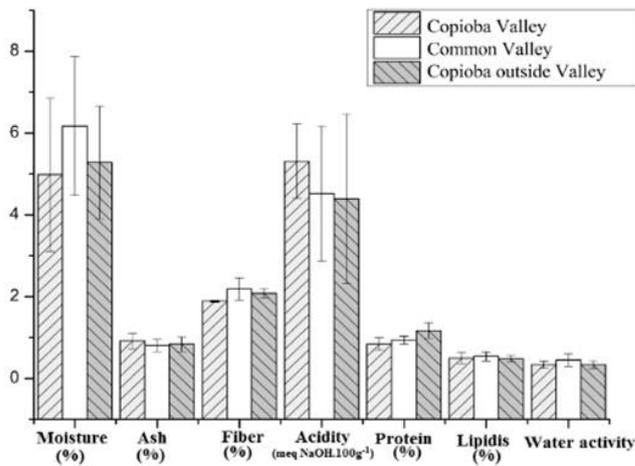
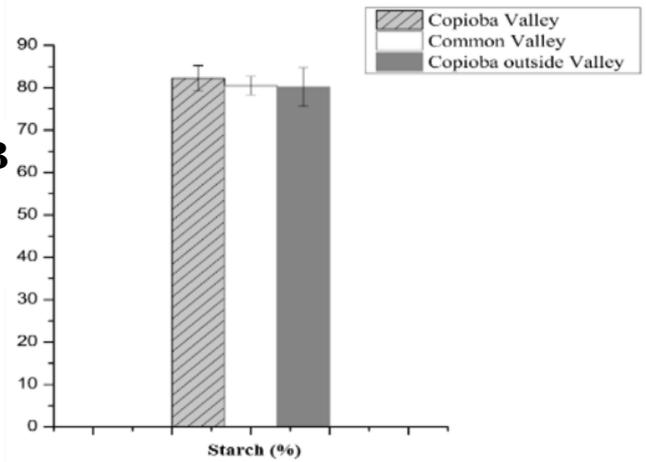
C9	6.62±0.25	0.92±0.01	2.00±0.02	3.18±0.23	81.83±1.03
C10	3.61±0.31	0.71±0.00	2.15±0.05	5.44±0.43	84.15±1.17
Mean ±SD	5.28±1.38 <sup>a</sup>	0.84±0.18 <sup>a</sup>	2.08±0.11 <sup>a</sup>	4.39±2.07 <sup>a</sup>	80.19±4.54 <sup>a</sup>
Range	3.53 - 7.31	0.46 - 1.15	1.79-2.23	1.50 - 6.76	71.18 – 85.20
Conformity <sup>1</sup>	100%	100%	100%	30.0% (low), 70.0% (high)	70.0%
Legislation <sup>1</sup>	< 13%	≤ 1.40%	≤ 2.3	≤ 3 (low), > 3 (high)	> 80%

<sup>1</sup>Brazil (2011). Equal letters in the same column indicate that there is no significant difference between the results for the Kruskal-Wallis test ( $p < 0.05$ ). (\*) Values expressed on a dry basis.

**Table 3.** Complementary parameters of quality of cassava flour.

Samples	Complementary Parameters			
	Protein crude (%)	Total Lipid (%)	Water activity	
Copioba Valley	A1	0.81±0.04	0.68±0.03	0.42±0.00
	A2	0.79±0.03	0.52±0.00	0.33±0.00
	A3	0.74±0.02	0.35±0.00	0.50±0.00
	A4	0.64±0.03	0.46±0.01	0.18±0.00
	A5	0.82±0.04	0.59±0.02	0.27±0.00
	A6	1.07±0.04	0.42±0.01	0.24±0.00
	A7	0.93±0.03	0.42±0.02	0.43±0.01
	A8	0.65±0.02	0.73±0.03	0.34±0.00
	A9	1.03±0.03	0.42±0.02	0.32±0.00
	A10	0.95±0.03	0.37±0.01	0.34±0.00
	Mean±SD	0.84±0.15 <sup>b</sup>	0.50±0.13 <sup>a</sup>	0.34±0.09 <sup>a</sup>
	Range	0.64 - 1.07	0.35 - 0.73	0.18 - 0.50
Common Valley	B1	1.09±0.03	0.53±0.01	0.35±0.00
	B2	0.94±0.02	0.67±0.02	0.31±0.01
	B3	0.92±0.04	0.57±0.01	0.27±0.01
	B4	1.04±0.04	0.72±0.03	0.43±0.00
	B5	1.00±0.03	0.40±0.01	0.43±0.00
	B6	0.81±0.03	0.48±0.02	0.31±0.00
	B7	0.81±0.04	0.60±0.02	0.62±0.00
	B8	0.86±0.04	0.59±0.03	0.52±0.01
	B9	1.06±0.02	0.41±0.01	0.66±0.00
	B10	0.88±0.01	0.39±0.01	0.64±0.00
	Mean ±SD	0.94±0.10 <sup>ab</sup>	0.54±0.11 <sup>a</sup>	0.45±0.15 <sup>a</sup>
	Range	0.81-1.09	0.39-0.72	0.27-0.66
Copioba outside Valley	C1	1.37±0.05	0.48±0.01	0.20±0,00
	C2	1.09±0.05	0.38±0.01	0.42±0,00
	C3	1.02±0.03	0.45±0.00	0.36±0,00
	C4	1.49±0.05	0.47±0,01	0.24±0,00
	C5	1.13±0.05	0.51±0.20	0.30±0,00
	C6	1.35±0.03	0.60±0.20	0.33±0,00
	C7	1.25±0.03	0.48±0.01	0.47±0,00
	C8	0.99±0.03	0.37±0.01	0.47±0,00
	C9	1.08±0.02	0.49±0.01	0.43±0,00
	C10	0.89±0.02	0.60±0.00	0.22±0,00
	Mean ±SD	1.17±0.19 <sup>a</sup>	0.48±0.08 <sup>a</sup>	0.34±0.10 <sup>a</sup>
	Range	0.89 – 1.49	0.89 - 1.20	0.20 - 0.47

Equal letters in the same column indicate that there is no significant difference between the results for the Kruskal-Wallis test ( $p < 0.05$ ). (\*) Values expressed on a dry basic.

**A****B**

**Figure 2.** Identity and Quality Parameters (IQP) of cassava flour, other complementary parameters of quality established by legislation (Brazil, 2011). A) Moisture, ash, fiber, acidity, protein, lipids and water activity. B) Starch.

The particle size distribution is the granules which constitute the product is very important in the standardization of quality cassava flour produced by artisanal process. The legislation (Brazil, 2011) set that the cassava flour dry group, according to its particle size, will be classified into three (3) class: a) fine: when 100% of the product passing through the sieve with an aperture of 2 mm and is retained up to 10%, including, in the sieve with mesh size 1 mm, b) coarse: when the product is held in more than 10% in the sieve with an aperture of 2 mm, and c) medium: when the cassava flour does not fit none of the above classes. The results of particle size according to the parameters of existing legislation can be seen in Table 4 and Figure 3.

The samples granulometry from Common Valley was higher than the Copioba samples from Valley ( $p < 0.05$ ). Getting so evident that all samples of flour Copioba (inside and outside the Valley) are 90% classified as "fine" class which may be a characteristic of this type of flour; regarding the Common flour from outside Valley only 60% are classified as "fine" and 40 % as "medium" (Table 4, Figure 3).

It can be seen, therefore, that flours are classified in different percentages in the 4 types stipulated to legislation (BRAZIL, 2011; Table 4). The Copioba Valley flour samples are mostly classified in Types 1 and 2 (60% of samples). To Common flour from Valley, half is of Type 1 and 2 and the other half as Type 3 and Outside of Type, where it is believed that this behavior is due to non-complete removal of the bark and the central fiber of roots during root processing, which resulted in fiber contents higher than 2.3% in two samples and starch lower than 80% in three samples (Table 1). Copioba flour outside Valley are mostly classified as Type 3 and outside of type (70%), because three samples had starch contents lower than 80% (Table 1). This is because the starch content is a characteristic that varies mainly with the production process, and the removal of the starch during the production of the flour the product quality loses, as already reported by Aryee et al., (2006). In this case, 20% of Copioba flour from Valley, and 30% of Common flour from Valley, and Copioba flour outside Valley have been classified as "Out of Type", and according legislation this information must

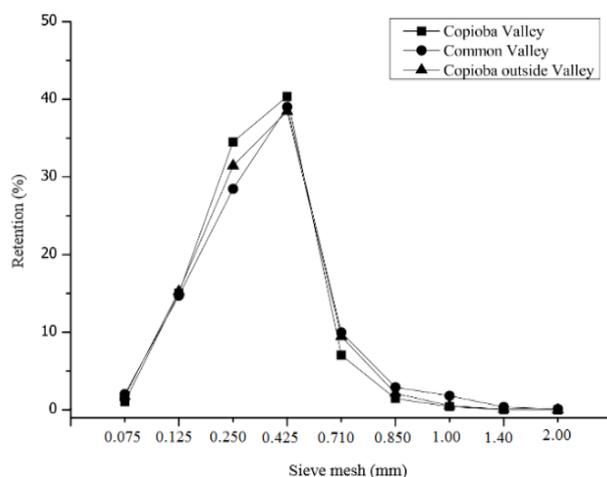
be added to the label or flour to be re-marketed.

**Table 4.** Granulometry (1.00 or 2.00 mm) and classification of three cassava flours in Fine, Medium or Coarse by sieving.

Denomination	Samples	Retained flour by mass (%)		Classification <sup>1</sup>
		2.00 mm	1.00 mm	
Copioba Valley	A1	0.00	0.09	Fine
	A2	0.00	0.01	Fine
	A3	0.00	1.15	Fine
	A4	0.00	0.02	Fine
	A5	0.00	0.10	Fine
	A6	0.02	0.12	Medium
	A7	0.00	0.00	Fine
	A8	0.00	1.74	Fine
	A9	0.00	1.14	Fine
	A10	0.00	0.00	Fine
		Mean±SD	0.00±0.01 <sup>b</sup>	0.44±0.65 <sup>b</sup>
Common Valley	B1	0.00	0.72	Fine
	B2	0.00	1.11	Fine
	B3	0.01	6.40	Medium
	B4	0.03	2.93	Medium
	B5	0.00	3.05	Fine
	B6	0.00	0.67	Fine
	B7	0.00	0.62	Fine
	B8	0.00	0.19	Fine
	B9	0.66	1.41	Medium
	B10	0.57	1.09	Medium
		Mean±SD	0.13±0.26 <sup>a</sup>	1.82±1.87 <sup>a</sup>
Copioba outside Valley	C1	0.00	0.39	Fine
	C2	0.00	1.11	Fine
	C3	0.00	1.07	Fine
	C4	0.00	0.03	Fine
	C5	0.00	0.09	Fine
	C6	0.04	0.28	Medium
	C7	0.00	0.12	Fine
	C8	0.00	0.64	Fine
	C9	0.00	0.61	Fine
	C10	0.00	1.57	Fine
		Mean±SD	0.00±0.01 <sup>b</sup>	0.59±0.51 <sup>ab</sup>

<sup>1</sup>Brazil (2011). Equal letters in the same column indicate that there is no significant difference between the results for the Kruskal-Wallis test ( $p < 0.05$ ).

The classification of Copioba Valley, Common Valley, and Copioba outside Valley flours in "dry group / process", "fine or medium / granulometry", in Type 1, 2, and 3, is shown in the Table 5.



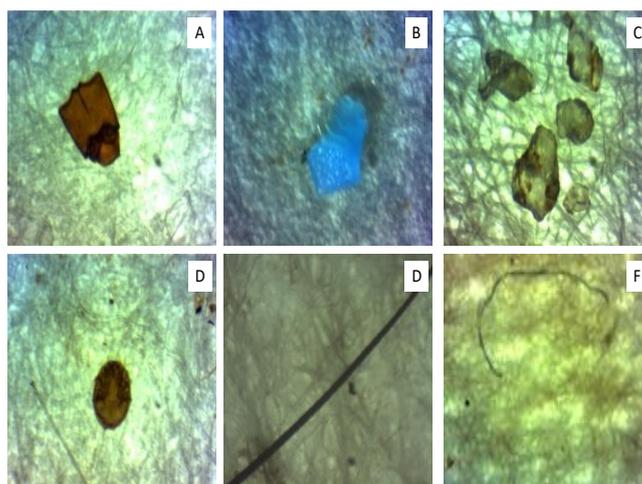
**Figure 3.** Particle size distribution of Copioba and Common cassava flours from the Valley and Copioba flour from outside the Valley by sieving.

**Table 5.** Classification of Copioba Valley, Common Valley and Copioba outside Valley flours of the "Dry group / process", "Fine or Mean / granulometry", in four Types (Brazil, 2011).

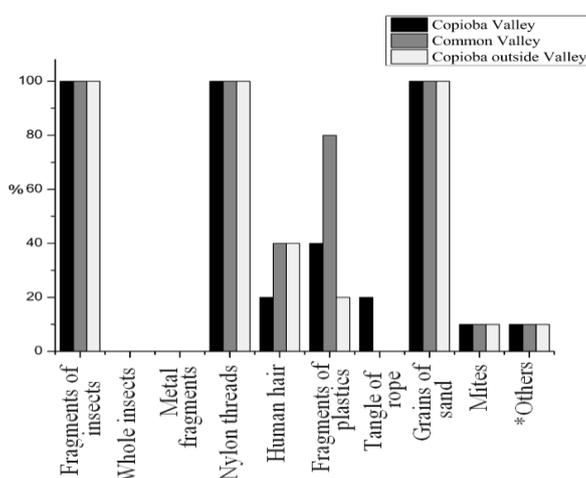
(Brazil, 2011)	Cassava flours		
	Copioba Valley	Common Valley	Copioba outside Valley
Starch content (%)	77.98 - 86.19	76.78 - 83.55	71.18 - 85.20
Ash content (%)	0.65 - 1.16	0.62 - 1.11	0.46 - 1.15
Crude fiber (g.100 <sup>-1</sup> )	1.47- 2.19	1.73 - 2.76	1.79 - 2.23
Classification			
Type 1 (%)	20	20	0
Type 2 (%)	40	30	30
Type 3 (%)	20	20	40
Out of Type (%)	20	30	30

In 100% of samples of cassava flour (Copioba and Common) the presence of insect fragments, sand grains and nylon wires were detected (Figure 5). Another relevant occurrence is the presence of plastic fragments that appear in 40% of the Copioba flour from Valley, in 80% of the Common flour samples from Valley, and in

20% of Copioba flour from outside the Valley. The presence of human hair appears in 20% of the Copioba flour from Valley, 40% in the Common from Valley, and 40% of Copioba from outside Valley. The presence of mites and other foreign matter appear in 10% of all types of flour.



**Figure 4.** Major occurrences of foreign matter obtained by stereoscopic microscope (40x): Fragment of insect (A); fragment of plastic (B); grains of sand (silica) (C); mite (D), human hair (E) and wire nylon (F).



**Figure 5.** Percentage of un Conformities the of the cassava flour from Copioba Valley; Common Valley; and Copioba outside Valley, with the parameters of foreign matter established by Brazilian legislation (BRAZIL, 2011). \* Foreign material not identified.

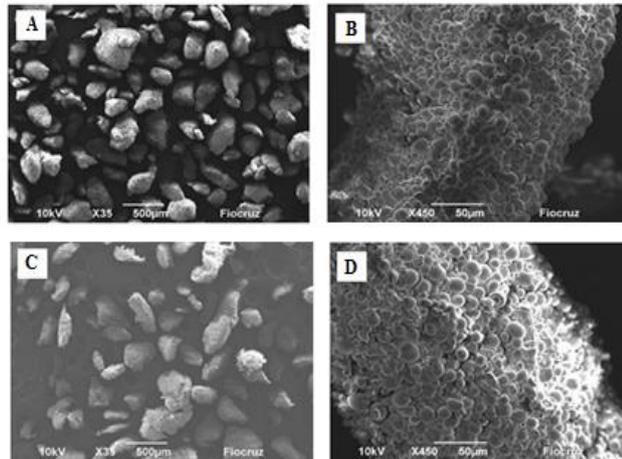
The high un Conformities found regarding foreign matter can be justified by a quantitative and exploratory study with 72 “flour houses” of Copioba Valley produced by Silva et al., (2017), whom related that none of units met more than 60% of the requirements, (building conditions; equipment and utensils; workers in the production area, food handling, and sales; raw material and products displayed for sale; and production flow, food handling, sale and quality control), which is below the recommended cutoff and indicates poor hygienic sanitary conditions. Equipment

and utensils made up the group with the lowest performance (4.54%), whereas the highest performance was observed in raw material and products displayed for sale (45.42%). However, the authors related that simple changes are possible, and would not only have positive effects on the hygienic sanitary profiles off “flour houses” but would also have an important social impact.

Morphology of cassava flour plays an important role in its functionality. The general appearance of cassava flours (Copioba and Common from Valley) was evaluated by micrographs obtained in a

scanning electron microscope (Figures 6 and 7), being possible to observe the

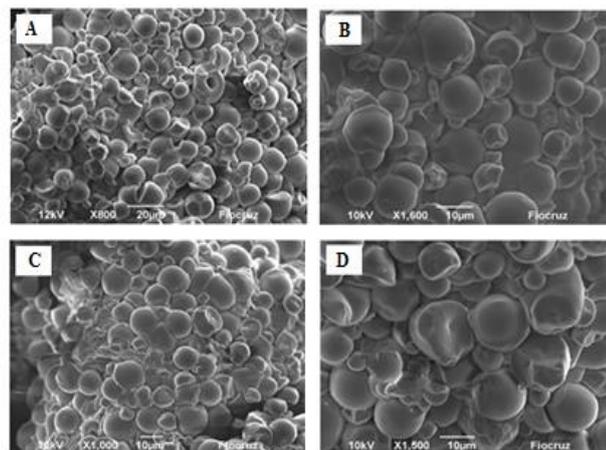
morphology and morphological changes of starch granules.



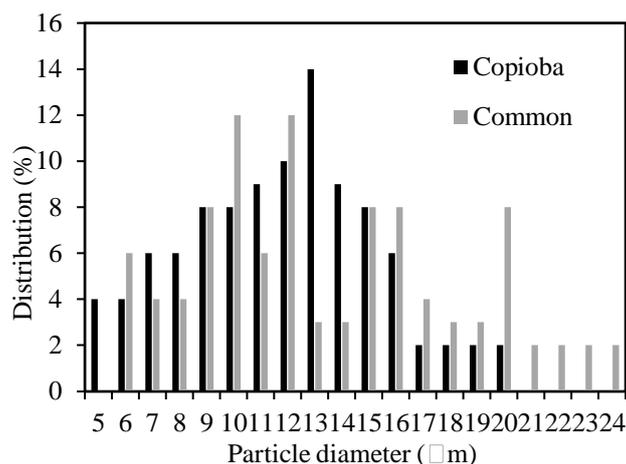
**Figure 6.** Scanning electron micrographs of cassava flour Copioba Valley (A, B in 35x and 450x, respectively) and Common cassava flour from Valley (C, D in 35x and 450x, respectively). (bar: A and C = 500 μm; B and D = 50 μm).

Starch granules are presented together with other (cluster) to form a larger particle, such as round, oval, and truncated shapes as well as a wide range of dimension (5–24 μm, Figure 8). In general, the average starch granule size of Copioba flour from Valley (5-20 μm, higher % in 9-16 μm) was similar to that of Common flour from Valley (6-24

μm, higher % in 9-20 μm), but with different distribution. Hence, small and medium-sized granules form the bulk class of granule types found in cassava starches, being in agreement with the results of the granulometry and classification of the cassava flours in Fine, Medium by sieving (Table 5).



**Figure 7.** Scanning electron micrographs of Copioba flour (A, B in 800x and 1,600x, respectively) and Common flour from Valley (C, D in 1,000x and 1,500x, respectively). (bar: A = 20 μm; B, C and D = 10 μm).



**Figure 8.** Particle size distribution of Copioba and Common flours from Valley obtained by Scanning electron micrographs.

## CONCLUSIONS

The flours were mostly considered of the high acidity, especially the Copioba from Valley flour. The three flours are extremely dry, with low moisture and water activity, and 90% of Copioba flours, regardless of origin, are classified as Fine, while only 60% of Common flours falls within this classification.

The foreign matter present in 100% of samples was composed of insect fragments, plastic fragments, grains of sand (silica), in addition, human hair and nylon in smaller quantities, indicating that basic hygiene care in flours processing is necessary. These parameters are extremely important for the establishment of a regulation of use in implementation of a geographic indication seal of the Copioba cassava flour.

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