

Rosa M. Alves^a, María Victoria Grossmann^a, Cristina Ferrero^b, Noemi E. Zaritzky^c, Miriam N. Martino^c, Maria Rita Sierakoski^d

^a Departamento de Tecnologia de Alimentos, Universidade Estadual de Londrina, Londrina, PR, Brazil

^b CIDCA-Facultad de Ciencias Exactas-Universidad de La Plata, La Plata, Pcia de Buenos Aires, Argentina

^c CIDCA-Facultad de Ciencias Exactas and Depto Ingeniería Química, Facultad de Ingeniería, La Plata, Pcia de Buenos Aires, Argentina

^d Departamento de Bioquímica, Universidade Federal de Parana, PR, Brazil

Chemical and Functional Characterization of Products Obtained from Yam Tubers

Aiming to find alternative uses of yam (*Dioscorea alata*) in the food industry, three products (flour, starch and mucilage) were obtained from yam tubers and characterized. Flours were obtained from peeled fresh tubers, dried at two different temperatures (30 and 55 °C) and then milled. Starch and mucilage were extracted from mashed yam before drying. Products were characterized by their chemical, structural, morphological, rheological and thermal properties. Flours showed differences in pasting, thermal and water absorption properties as a function of drying temperature. Starch contained 30% amylose. The analysis of monosaccharide composition of starch showed traces of mannose, xylose and rhamnose. The onset gelatinization temperature was 74.4 °C for starch, whereas those for flours varied between 71.5 and 74.4 °C. The relatively high gelatinization temperature and the absence of breakdown in the amylograph test evidenced the stable structure of starch when submitted to heat and shear. The mucilaginous material contained 55.36% protein and 43.05% starch, and also an acidic polysaccharide fraction linked to a neutral fraction. Flow curves of 1% mucilage dispersions showed a pseudoplastic behavior with high pH sensitivity. Results demonstrated that yam products have promising applications in the food industry.

Keywords: *Dioscorea alata*; Mucilage; Gelatinization

1 Introduction

The main sources of starch supply are corn, cassava, potato, wheat and rice. Yam tubers are another potential starch source [1–3]. Yam (*Dioscorea alata*) is a food crop of economic value in southern Brazil [4] and other countries from Asia and Africa. Yam processing and yam flour applications in the food industry have been studied by several researchers [5–12]. The main application claimed for yam flour has been in bread products and snacks. Increasing interest in other ingredients derived from yam tubers is arising, as well.

Starch extraction from yam tubers is difficult because during the milling process mucilaginous compounds occur, that increase the viscosity of the system [1]. However, this mucilaginous material could be useful as a thickener in food products.

The knowledge of chemical composition and structure of a potential food ingredient could help to predict its functional properties under different processing and storage conditions.

The objectives of the present work were to perform a physico-chemical characterization of the flour, starch and

mucilaginous material obtained from yam tubers in order to evaluate their potential use as food ingredients.

2 Materials and Methods

2.1 Raw Material

Tubers of *Dioscorea alata* were obtained from farmers of Londrina (Brazil) region.

2.2 Separation of the different fractions

2.2.1 Flour preparation

Tubers with different shapes and sizes were washed, hand peeled and trimmed to remove defective parts. Then the tubers were sliced and dried in an air convection oven (30 °C for 40 h) up to 13% moisture. The dried slices were first hammer milled (MR 340 mill, Metalúrgica Roma, São Paulo, Brazil) and then reground in an Alpine mill (Augsburg, Germany) equipped with a 0.297 mm screen. The flour was packed and stored for further analysis. A second flour sample was obtained by following the same procedure and drying at 55 °C. This higher temperature was selected because it was observed that the mucilaginous fraction, rich in proteins, partially lost the thickening properties after being heated at 55 °C. Since these proteins are also present in the flour, the effect of the drying temperature on flour properties was analyzed.

Correspondence: Maria Victoria Grossmann, Departamento de Tecnologia de Alimentos, Universidade Estadual de Londrina, Caixa Postal 6001, 86051-970-Londrina, PR, Brazil. e-mail: victoria@uel.br.

2.2.2 Mucilaginous material extraction

Yam mucilage was obtained using a modification of the method described by Fedeniuk and Biliaderis [13]. Tubers were peeled, sliced and homogenized in large quantities of distilled water containing 0.075 % (w/v) of sodium metabisulfite using a laboratory blender. The suspension was transferred to a large vessel and another volume (10 L/kg tuber) of sodium metabisulfite solution was added. The material was allowed to settle and ethanol was added until no more mucilage was flocculated from the supernatant. Flocks were removed and freeze dried. Deliberately, this fraction was not purified in order to analyze the properties of this low cost ingredient. Although it was not purified, for simplicity it will be named "mucilage" in the present work.

2.2.3 Starch extraction

Starch was extracted as reported by Alves et al. [3]. The remaining material after mucilage separation was sieved (0.250 mm) and slurried in sodium metabisulfite solution (0.075 %, w/v). The suspension was allowed to settle again and the resuspension and sedimentation operations were repeated ten times. Finally the sedimented material was treated with sodium hydroxide (0.15 or 0.10 %, w/v) and exhaustively washed with distilled water until pH 7.0. The recovered starch was sieved (0.105 mm), treated with 70 % ethanol and dried in a forced air oven until 12 % moisture.

2.3 Physico-chemical characterization

2.3.1 Starch granule morphology

Starch samples obtained by the described method were observed with an optical Jenamed-Zeiss microscope under polarized light (Jena, Germany).

2.3.2 Chemical composition

Moisture, protein ($N \times 6.25$), lipids and ash were determined according to AOAC methods [14]. Starch content was estimated using the ICC Standard n° 123/1, being $d = 184.8$ [15]. Amylose content of starch was determined by the method of Jarvis and Walker [16]. Non-starch carbohydrates were obtained by difference. Each analytical determination was performed in triplicate.

2.3.3 Monosaccharide determination

Neutral monosaccharide contents in the samples were determined by acid hydrolysis with M trifluoroacetic acid (TFA) at 100 °C for 5 h [17]. The solutions were evaporated to a residue, which was repeatedly dissolved in H₂O and evaporated. The products from the hydrolysis procedure were reduced with NaBH₄ and acetylated with pyridine-Ac₂O (1:1 v/v) for 12 h at 25 °C. To identify the uronic

acid in the structure of polysaccharides, the products from acid hydrolysis were submitted to carboxyl reduction as described by Blake and Richards [18] modified. This process was repeated two times. Then, the neutral oligosaccharides produced were submitted to total hydrolysis with 2 M TFA at 100 °C for 5 h. The liberated monosaccharides were converted into alditol acetates by successive NaBH₄ reduction and acetylation with Ac₂O-pyridine. The resulting alditol acetates were analyzed by GLC using an HP model 5890-2, with a DB-225 capillary column at 220 °C, a flame ionization detector at 250 °C, and nitrogen as carrier gas (J. & W. Scientific, CA, USA).

Gas chromatography-mass spectrometry (GC-MS) analyses were performed using a Finnigan Mat ion trap (model 410) mass spectrometer, incorporating a DB-225 capillary column (30 m × 0.25 mm i.d.) (J. & W. Scientific, CA, USA), with He as carrier gas. Injections were carried out at 50 °C, the column temperature was then programmed to rise to 230 °C at a rate of 4 °C/min, and held at that temperature until the end of the run. Scans were carried out at m/z 40-420 every 2 s at 70 eV.

To determine the presence of glucuronic and galacturonic acids, a paper chromatography was performed. Fifty milligrams of each sample were dispersed in 3 mL of distilled water. After a rest period of 3 h, 2 M TFA was added and samples were hydrolyzed during 2.5 h at 100 °C. After hydrolysis, TFA was evaporated and samples were washed several times with distilled water. The obtained materials were solubilized in 0.5 mL distilled water and analyzed using the ascending technique and Whatman N°1 chromatographic paper as support. A mixture of benzene, butanol, pyridine and water (1:5:3:3) was used as solvent and silver nitrate in alkaline medium as staining agent. As standards, glucuronic and galacturonic acids were used.

2.3.4 Differential scanning calorimetry

Thermograms were obtained using a DSC Polymer equipment (Rheometric Laboratories, Surrey, UK). Aqueous suspensions of starch or flour (10%, w/w) and mucilage (15%, w/w) were prepared in batch. Under constant stirring, aliquots of the suspension (10-15 mg) were weighed in coated aluminum pans and hermetically sealed. Scans were performed from 20 to 120 °C at a controlled constant rate of 10 °C/min. After each test, pans were punctured and the exact dry weight of each sample was determined. Enthalpies were calculated on a starch dry-weight basis subtracting protein content. Runs were performed in triplicates.

2.3.5 Pasting properties of yam starch

Brabender amylographic patterns were obtained for flours and starch (Brabender Amylograph Pt 100, 700 cm-g,

Brabender OHG, Duisburg, Germany). Samples were heated at 1.5 °C/min from 30 to 95 °C and kept for 20 min at 95 °C. Cooling was performed at 1.5 °C/min to attain 50 °C. Dry matter concentration was 6 % (w/v) in all cases.

2.3.6 Paste clarity

Paste clarity was determined according to the method of Craig et al. [19]. Transmittance was determined for 1% (w/v) yam starch dispersions, employing a Femto spectrophotometer (São Paulo, Brazil).

2.3.7 Rheology characterization of the mucilage

Rheological measurements on mucilage (1%, w/v) at different pH (4, 6 and 8) were done using a Haake Rotovisco RV2 (Karlsruhe, Germany) with a coaxial cylinders (NV) sensor system. Temperature was maintained constant at 20 °C with a thermostatic system. Flow properties were studied from 0 to 1385 s⁻¹ shear rate with the following program: 3 min to attain the maximum shear rate, 1 min at the maximum shear rate and 3 min to attain to 0 s⁻¹. Two runs were performed for each sample.

2.3.8 Statistical analysis

Statistical analysis involved ANOVA, mean comparison and regression analysis. Specific softwares Statistical Analysis System for Windows (1996, USA) and Statistica by Statsoft (1995, USA) were used.

3 Results and Discussion

3.1 Chemical composition of tubers and derived ingredients

The chemical composition of peeled yam tubers, expressed as % of fresh weight was: 71.0% moisture; 2.7% protein, 0.1% lipids, 23.8% starch and 0.7% ash. Solid content (29%) agrees with the results (25–32%) reported by Agbor-Egbe and Rickard [20] and Valetudie [1]. The starch and protein contents (dry basis) were 82.0 and 5.9%, respectively. Other authors describe tubers with contents varying from 65 to 80% starch and 2.1 to 7.3% protein [1, 12].

The results of the proximate analysis and extraction yield of the products obtained from tubers are shown in Tab. 1. Starch was the most important component of flour (88%), that presented also a significant protein content (6.9%). Similar results were reported by Valetudie [1] and Muzac-Tucker et al. [21]. The levels of proteins (0.18%), lipids (0.16%) and ash (0.15%) of the starch fraction indicated that it was fairly pure. Starch amylose content was 30.0%, greater than those reported by Emiola and Delarosa [22]

Tab. 1. Chemical composition (% dry basis) and yield (% dry weight of tubers) of ingredients obtained from yam.

	Flour	Starch	Mucilage
Starch	88.71 (0.07)	98.30 (0.05)	43.05 (0.03)
Proteins ^a	6.90 (0.02)	0.18 (0.02)	55.36 (0.05)
Lipids	0.25 (0.01)	0.16 (0.01)	0.11 (0.01)
Ash	1.60 (0.04)	0.15 (0.01)	0.10 (0.01)
NS carbohydrates ^b	2.54 (0.01)	1.21 (0.01)	1.38 (0.02)
Yield	97.03	88.70	0.14

^a N × 6.25. ^b Non-starch carbohydrates.

Standard deviations are shown between parentheses.

and Valetudie [1] (21.0 to 24.4%) but lower than 34%, obtained by Cruz-Gay and Gonzalez [23].

Chemical composition of mucilaginous material showed 55.36 % protein, that can be part of the mucilage structure, as reported by Shi-Shun-Tsai and Fen-Ji-Tai [24], and also from contaminant protein. Similarly, the detected glucose mainly related to starch (43.05%, Tab. 1) could originate from other polysaccharides.

Yam starch granules showed a characteristic almond shape, with noncentered hilum located at the more acute pole (Fig. 1). Their sizes ranged between 8–27 μm width and 12–33 μm length. Similar values were obtained by Rosenthal et al. [25].

3.2 Monosaccharide composition

The monosaccharide composition of all the products extracted from yam tubers was analyzed by GC-MS after acid hydrolysis and conversion to alditol acetate derivatives (Tab. 2). According to these results, it was verified that yam flour was composed by 93.4% of glucose units that can be

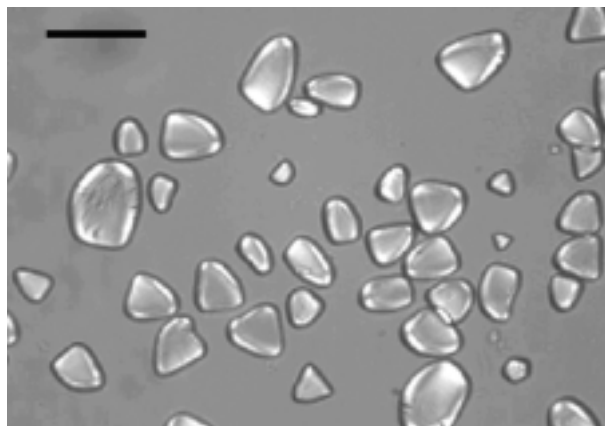


Fig. 1. Micrograph of yam starch native granules under polarized light (bar = 50 μm).

Tab. 2. Monosaccharide composition of ingredients obtained from yam tubers.

Ingredient	Monosaccharides [mol%] ^a						
	Arabinose	Galactose	Glucose	Mannose	Rhamnose or fucose	Xylose	Inositol
Flour ^b	0.5	1.0	93.40	0.6	1.0	0.4	3.1
Starch ^b	1.0	-	89.50	5.10	1.0	0.5	2.9
Mucilage ^b	9.8	-	55.40	30.3	-	-	4.5
Mucilage ^c	56.0	-	2.5	-	21.5	20.0	-
Mucilage ^d	47.0	3.8	16.5	11.0	7.0	12.7	2.0

^a As alditol acetates.^b Hydrolysis with 1 M trifluoroacetic acid (TFA) -neutral fraction.^c Hydrolysis with 2 M TFA.^d Hydrolysis with 2 M TFA, carboxyl-reduced with borate buffer two times and hydrolyzed with 2 M TFA- acidic fraction.

assigned to the starch present. Detection of xylose, galactose, mannose, arabinose, inositol and rhamnose or fucose units show also the presence of other polysaccharides. By paper chromatography, glucuronic acid was detected, indicating the existence of acid constituents.

As it was expected, in the starch sample the principal detected monosaccharide was glucose but others units like

mannose, arabinose, fucose and xylose were also found, in levels under 8%.

In the mucilage, a significant level of glucose was detected and this can be interpreted as a possible starch contamination. Among the presence of other polysaccharides, an acidic fraction was found that is more resistant to acid hydrolysis and that is covalently linked to a neutral fraction. Inositol was identified in the neutral fraction (obtained by acidic hydrolysis with 1 M TFA) and in less quantity in the acidic one (obtained by acidic hydrolysis with 2 M TFA, carboxyl-reduced with borate buffer two times and hydrolyzed with 2 M TFA). The presence of glucuronic acid unit was verified by paper chromatographic analysis. In the alditol acetates quantification, applying carboxyl-reduction method before acetylation, the glucuronic acid units were converted to neutral units. This explains the increased content of glucose in the acidic fraction, whose units can be bound to rhamnose units. The presence of others neutral monosaccharides such as arabinose, xylose, mannose and galactose units suggest that the mucilage is composed by one or more acidic heteropolymers of highly complex structure.

3.3 Thermal properties of yam products

In Fig. 2 four DSC typical thermograms obtained for flour, starch and mucilage are compared. For yam flour and yam starch, endotherms ranging from 71 to 85 °C can be attributed to starch gelatinization.

Mean values of the onset, peak and final gelatinization temperatures (Tab. 3) are higher than those reported for corn starch (63.2, 69.0 and 75.2 °C, respectively) or potato starch (58.7, 62.6 and 68.1 °C, respectively) indicating that yam starch has a more ordered structure [26]. The high T_g

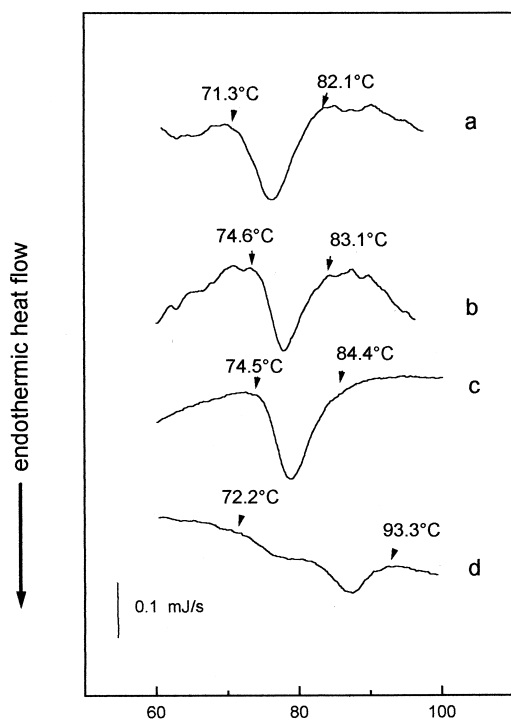


Fig. 2. Thermograms for a) yam flour dried at 30 °C (10% w/w, dry basis), b) yam flour dried at 55 °C (10% w/w, dry basis), c) yam starch (10% w/w, dry basis) and d) mucilage (17% dry basis). Onset and final temperatures are shown.

Tab. 3. Thermal properties of ingredients obtained from yam.

	Thermal transition temperatures ^a [°C]			Gelatinization enthalpy [J/g starch dry basis]
	T_o	T_p	T_f	
Starch	74.4 ^a (0.1)	78.8 ^a (0.1)	84.9 ^a (0.1)	17.3 ^a (0.8)
Flour dried at 30°C	71.5 ^b (0.3)	76.3 ^b (0.1)	81.8 ^b (0.4)	11.9 ^b (1.7)
Flour dried at 55°C	74.4 ^a (0.4)	77.6 ^c (0.2)	82.7 ^b (0.8)	10.5 ^b (0.4)
Mucilage	72.0 ^b (0.7)	88.1 (0.8) (second peak)	94.3 (1.3)	nd

T_o = onset temperature; T_p = peak temperature; T_f = final temperature.

Standard deviations are shown between parentheses.

Within each column, values with the same letter are not significantly different.

nd= not determined.

is related to the resistance to swelling of the starch granule, as was observed before by *Emiola* and *Delarosa* [22].

Onset gelatinization temperature of yam starch was not significantly different from that of flour dried at 55 °C, however it showed a higher enthalpy value than both flour samples. Yam flours showed significant differences in onset and peak values, depending on the temperature used for drying process. Yam flour dried at 55 °C showed a significantly higher value than yam flour dried at 30 °C. These differences could be attributed to protein-starch interaction or protein denaturation.

Since the mucilage contains starch and proteins, two superimposed peaks can be observed in this case (Fig. 2, curve d). The first one, according to the temperature range, can be attributed to starch gelatinization. The second one could be due to the denaturation of proteins.

3.4 Pasting properties

Brabender patterns of both flour samples (dried at 30°C and 55°C) and starch are shown in Fig. 3.

Curves patterns corresponding to starch samples showed a marked higher viscosity than for flour samples. Flour samples showed a lower viscosity probably due to the presence of other components that make starch pasting more difficult and also to its lower starch content. Yam flour dried at 55 °C showed a higher viscosity during heating and cooling and a slightly lower pasting temperature (69 °C) than the flour dried at 30 °C (pasting temperature = 70.5 °C). This behavior could be attributed to protein modifications during the more drastic heat treatment at 55 °C. These modifications could increase protein water absorption affecting protein-starch interactions. Patterns of both flour and starch pastes indicated also a great stability to heat treatment and mechanical stress as can be inferred from the absence of breakdown. Viscosity increased during the entire heating period; this behavior continued along cooling, indicating a pronounced tendency

to retrogradation related to the relatively high amylose content of this starch.

The higher initial viscosity of flour samples as compared with starch can be attributed to the presence of other constituents (proteins, other polysaccharides), that are partially eliminated during starch extraction.

Paste clarity of yam starch, expressed as percent of transmittance (% T) was 4.0 ± 0.2 while cassava pastes showed a % $T = 24.0 \pm 0.2$ in the same conditions (unpublished data from our laboratory). This can be attributed to higher amylose content of yam starch (30 %) compared to cassava starch (16-20 %). According to *Wang* et al. [27] starches containing higher contents of amylose showed relative lower transmittance (%) values.

3.5 Viscosity properties of the mucilage

Dispersions of the mucilage material at a concentration of 1 % (w/v) showed a non-Newtonian flow behavior and high sensitivity to medium pH. At pH=4 protein coagulation was observed. At pH=6, apparent viscosities calculated at 692.5 s^{-1} and 1385 s^{-1} were 46.9 (SD=0.9) and 32.5 (SD=0.5) respectively. Flow curves showed a pseudo-plastic and slightly thixotropic behavior. At pH 8 the mu-

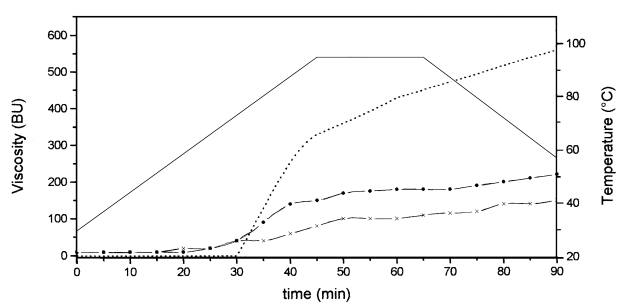


Fig. 3. Brabender Amylograph patterns of: — x — yam flour dried at 30 °C, — ● — yam flour dried at 55 °C and ----- yam starch. — Temperature profile (on the right Y axis).

mulilage suspension was more clear and no coagulation was observed. Apparent viscosity values were 27.0 and 19.9 (at 692.5 s^{-1} and 1385 s^{-1} , respectively). The flow behavior was pseudoplastic but not thixotropic. The strong influence of the medium pH on the solubility, thickening capacity and type of rheological behavior of mulilage dispersions can be related to the high concentration of proteins in this complex material.

4 Conclusions

Ingredients obtained from yam tubers contained starch as the main constituent. However, mucilaginous material contained both starch and proteins as major components. Flours dried at different temperatures showed different thermal and pasting properties. Yam starch supports unfavorable processing conditions: pastes of yam starch can be heated without showing a structure breakdown. This characteristic would allow this ingredient to be included in starch based foods requiring good stability under thermal treatment and an opaque appearance. Its relatively high content of amylose could limit the use of this starch in foods requiring storage at low temperatures since retrogradation is promoted. Further studies concerning yam amylose characteristics are necessary for a complete explanation of the particular properties of this starch. The mulilage showed a marked sensibility to pH and thermal conditions because of the high content of proteins and starch. Because of this instability, it seems that this mucilaginous material could not be applied without a previous purification in food formulations.

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