

# Morphological structure and crystallinity of ‘Rainha’ sweet potato starch by heat–moisture treatment

Mônica Tejo Cavalcanti<sup>1\*</sup> , Natália Silva de Farias<sup>2</sup>, Albanete da Nóbrega Cavalcante<sup>1</sup>,  
Mônica Correia Gonçalves<sup>1</sup>, Adriano Sant’Ana Silva<sup>1</sup> and Roberlúcia Araújo Candeia<sup>1</sup>

<sup>1</sup>Unidade Acadêmica de Tecnologia de Alimentos, Centro de Ciências e Tecnologia Agroalimentar, Universidade Federal de Campina Grande – UFCG, Pombal, PB, Brasil

<sup>2</sup>Programa de Pós-graduação em Ciência dos Alimentos, Universidade Federal de Santa Catarina – UFSC, Florianópolis, SC, Brasil

\*[monicatejoc@yahoo.com.br](mailto:monicatejoc@yahoo.com.br)

## Abstract

Heat-moisture treatment is type of physical modification that which cause changes in the technological characteristics of the starch. One of the sources of starch that presents potential of use of this treatment is sweet potato of the variety ‘Rainha’ (*Ipomoea batatas*). Therefore, the objective of this work was extract the sweet potato starch, apply the heat-moisture treatment in different relative humidity conditions (15, 20, and 25%), and characterize the starches as to water absorption capacity, morphology and crystallinity. Starch extracted from sweet potato resulted in a product of high purity. All modified starches showed a higher water absorption capacity when compared to native starch. The morphology of the starch granules remained unchanged after the modification and the same was observed with respect to the crystallinity. However, modified 15% moisture starch showed significant changes in the amylose content, water absorption, and crystallinity, these characteristics extend the use of this starch, for use in foods.

**Keywords:** *physical modification, tubercle, quality.*

**How to cite:** Cavalcanti, M. T., Farias, N. S., Cavalcante, A. N., Gonçalves, M. C., Silva, A. S., & Candeia, R. A. (2019). Morphological structure and crystallinity of ‘Rainha’ sweet potato starch by heat-moisture treatment. *Polímeros: Ciência e Tecnologia*, 29(2), e2019016. <https://doi.org/10.1590/0104-1428.03917>

## 1. Introduction

The sweet potato (*Ipomoea batatas*) belongs to the Convolvulacea family and is an easily-adaptable rustic tuberous vegetable tolerant to dry seasons. It is a relatively low cost production crop of great economic and social importance in developing countries<sup>[1]</sup>.

Starch is the main component of this root and corresponds to about 50-80% of dry matter, consisting of amylose and amylopectin in different proportions. The process of extracting this carbohydrate is of great interest to the food, pharmaceutical and chemical industry, because it presents physicochemical characteristics as, solubility, swelling power, pasting property, thermal, among others<sup>[2]</sup>.

In its native form, starch often does not possess appropriate physicochemical properties for some types of processing. In this sense, the modifications of starches from alternative sources provide the amplification of use and favor the commercialization of starch.

Among the types of modifications, the physical modification by heat-moisture treatment has been highlighted with the advantage that the obtained starch is considered a natural and highly safe material<sup>[3]</sup>. Huang et al.<sup>[4]</sup> verified that this method applied to sweet potatoes promoted significant changes in increasing paste temperature and starch content, and decrease in viscosity and relative crystallinity.

Based on this approach, the objective of the study was to evaluate morphological and structural characteristics, water absorption capacity, as well as the physicochemical characteristics of ‘Rainha Branca’ sweet potato starch subjected to heat-moisture treatments.

## 2. Materials and Methods

### 2.1 Starch extraction

Medium-sized ‘Rainha Branca’ sweet potatoes were purchased in the city of Campina Grande, Paraíba, Brazil. Starch was obtained by the method described by Adebawale et al.<sup>[5]</sup> with adaptations. The potatoes were ground with distilled water in a ratio of 1:2 (w/v). The mixture was filtered (70 mesh) and the filtrate was allowed to stand at 5°C for 8h. The supernatant was discarded and the starch was dried in air circulating oven at 40°C until final moisture of 13%.

### 2.2 Physicochemical characterization

Sweet potato starch was evaluated for water content using an Infrared Moisture Analyser until constant weight (brand Marte, ID 200), ash, protein content and lipid content were determined using the AACC<sup>[6]</sup> method, the results were expressed as dry basis (% db). Total

carbohydrate content including fiber was calculated by difference. Apparent amylose content from starch was determined according to the methodology described by Martinez & Cuervas<sup>[7]</sup>.

### 2.3 Modification of starch

Starch modification by heat–moisture treatment (HMT) was carried out according to the method described by Horndok & Noomhorm<sup>[8]</sup>, where, the obtained starch had its water content adjusted to 15 (HMT15), 20 (HMT20) and 25% (HMT25) for posterior heat treatment.

### 2.4 Water and oil absorption capacity

Water and oil absorption capacity were determined according to the method of Beuchat<sup>[9]</sup> at 25 ° C.

### 2.5 Morphological characterization

The morphological evaluation was performed by a JEOL JSM 5800LV Scanning Electron Microscope (SEM) with 3kV. The determination of particle size was performed based on micrographs with a magnification of 2000x using ImageJ software.

Structural characterization was performed by X-ray diffraction in a SHIMADZU X-ray diffractometer (XRD 6100) with an angular sweep of  $10^\circ < 2\theta < 50^\circ$  in the Bragg-Brentano assembly  $\theta$ - $2\theta$  system using Cu ( $\text{K}\alpha 1$ ) radiation, a sweep scan of 0.02 ( $2\theta$ ), and 0.6 second interval for each sample. The crystallographic phases of the samples and the standard JCPDS plots representing them were determined by the X-ray diffraction curves.

### 2.6 Statistical analysis

The data starch water absorption capacity were submitted to analysis of variance ANOVA and the Tukey test ( $p \leq 0.05$ ), using Software Assisat 7.7

## 3. Results and Discussion

### 3.1 Physicochemical characterization

The starch extracted from 'Rainha' sweet potatoes has its own characteristics, with low levels of minor constituents such as ash (0.36%, db), lipids (0.37%, db) and proteins (0.07%, db). The starch presented good quality and efficacy in the implemented extraction process, with carbohydrate content (sugars plus fibers) of about 85.57%.

As described in the literature, sweet potato starch is considered to have high amylose content and to have influence on starch properties<sup>[10]</sup>. When comparing the apparent amylose content in the native starch (30.39%, db) and those physically modified, we observed that the modified starch at 15% moisture presented lower values (28.85%, db) than other samples (HMT20 of 33.97% db, HMT25 of 31.19% db), which can be the result of additional interactions between the amylose-amylose and amylose-amylopectine chains which modify the starch matrix making the amylose more insoluble and unavailable for quantification<sup>[11]</sup>.

### 3.2 Water and oil absorption capacity

Sweet potato starch presented low water absorption capacity (WAC), however, the modified starch presented higher values of starch compared to the native, where: 0.76 ( $\pm 0.04$ ) g/100g in the native starch; 0.95 ( $\pm 0.02$ ) g/100g in the HMT15; 0.96 ( $\pm 0.02$ ) g/100g in HMT20; and 0.85 ( $\pm 0.01$ ) g/100g in HMT25. Similar behavior was observed regarding oil absorption capacity (OAC), where the modified starches presented higher values of starch, being 1.01 ( $\pm 0.02$ ) g/100g in the native starch; 1.22 ( $\pm 0.07$ ) g/100g in the HMT15; 1.23 ( $\pm 0.02$ ) g/100g in the HMT20; and 1.34 ( $\pm 0.05$ ) g/100g in the HMT25. Starch with low water absorption capacity is indicated as an ingredient in products that require both low retention of water and fat, improving characteristics such as product crunchiness.

### 3.3 Morphological characterization

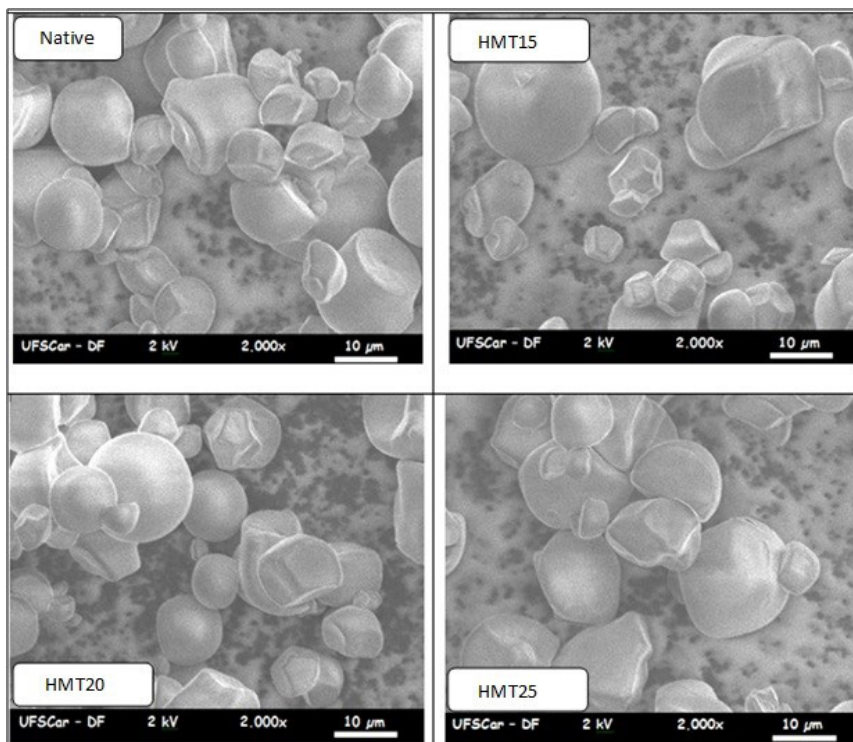
The size of the starch granules is an important parameter that affects their physicochemical properties and applications. Micrographs of the starch (Figure 1) show surface roughness and few cracks/fissures in modified starches in comparison to original starch. However, all starch types presented ellipsoidal and rounded granules, with noticeable agglutination tendency in starches modified by heat-moisture treatment.

The 'Rainha' sweet potato native starch sample presented ellipsoidal granules and a smooth surface, with the granules size corresponding to about 5.51  $\mu\text{m}$ . A small increase in particle size by 6.84 and 5.51  $\mu\text{m}$  was observed for the morphologies of the granules corresponding to modified starches at 15% RM (HMT15) and 25% RM (HMT25), except for the sample of 20% RM (HMT20) that was smaller with 5.00  $\mu\text{m}$ . Average particle size may be related to the amylose content, considering that the HMT 20 treatment had the highest amylose content.

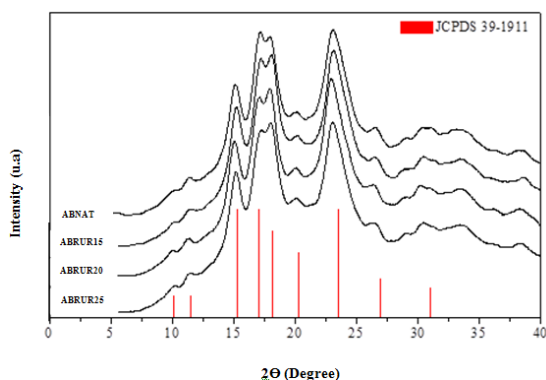
Because starch granules are partially crystalline, they are defined based on the interplanar spaces and the relative intensity of the X-ray diffraction lines. Peak regions observed for sweet potato starch are characteristic of standard type A chains, which assume short to medium starch chain lengths (Figure 2).

The ratio of the starch crystallinity state (amorphous/crystalline) is very important to understand its stability and application. The diffraction curve allows us to identify these characteristics, in addition to establishing the starch classification, which according to Yu et al.<sup>[12]</sup> and Zeng et al.<sup>[13]</sup> can be classified as type A, B, C and V starch. According to these authors, type A starch is a typical cereal starch, Type B is associated with tuberoses or amylose rich materials, and Type C starch resembles pea starch and various bean starches. Moreover, Type C can be considered as a mixture of the XRD patterns of starches A and B. Type V starch is a type of crystalline starch typical of complexes formed between amylose and lipid.

The starches were characterized as Type A due to the presence of XRD curves at reflection points  $2\theta = 10, 11, 15, 18$  and  $23^\circ$ . The amylose content found in the starches corroborates with the crystallinity results, once, ranged from 28.85 to 33.97% indicating low content.



**Figure 1.** Micrographs of native starch granules and modified starch at 15% RH (HMT15), 20% RH (HMT20) and 25% RH (HMT25) of 'Rainha Branca' sweet potato, 2000x magnitude.



**Figure 2.** X-ray diffraction curves of native starch and modified starch at 15% RH (HMT15), 20% RH (HMT20) and 25% RH (HMT25) of 'Rainha Branca' sweet potato.

Waramboi et al.<sup>[14]</sup> conducted characterization studies of sweet potatoes from New Guinea and Australia, and found that sweet potatoes obtained a type A crystallinity pattern and peak values of 15; 17; 17.9 and 22.8° for 2 $\theta$ ; values similar to those found in this study.

Based on the crystallinity values of the modified starches, the increase of the crystallite in relation to the original starch (7.37%) can be verified. However, the HMT 15 stood out from the others, obtaining 8.43% crystallinity.

This parameter corroborates with the results of the micrographs (Figure 1), which illustrates the difference in the morphology of the granule, considering that the 15% RM

sample presented larger particle size with less agglutination. This result can be explained because the modified sample at 15% relative moisture has lower amylose content, increasing its crystallinity.

#### 4. Conclusions

The extraction of sweet potato starch results in a product of high purity and good quality. The heat–moisture treatment caused morphological changes and water absorption capacity. Modified 15% moisture starch presented considerable characteristics for use in fried food products as texture enhancers.

#### 5. References

- Nunes, M. U. C., Jesus, A. F., Lima, I. S., Santos, L. S., & Cruz, D. P. (2012). Produtividade de genótipos de batata-doce com diferentes colorações de raízes em cultivo orgânico. *Horticultura Brasileira*, 30(2), S5542-S5548. Retrieved in 2017, June 17, from <https://www.alice.cnptia.embrapa.br/alice/bitstream/doc/949283/1/Produtividade.pdf>
- Zhu, F., Yang, X., Cai, Y., Bertoft, E., & Corke, H. (2011). Physicochemical properties of sweetpotato starch. *Starch*, 63(5), 249-259. <http://dx.doi.org/10.1002/star.201000134>.
- Bemiller, J. N. (1997). Starch modification: challenges and prospects. *Starch*, 49(4), 127-131. <http://dx.doi.org/10.1002/star.19970490402>.
- Huang, T.-T., Zhou, D.-N., Jin, Z.-Y., Xu, X.-M., & Chen, H.-Q. (2016). Effect of repeated heat-moisture treatments on digestibility, physicochemical and structural properties of

- sweet potato starch. *Food Hydrocolloids*, 54(Part A), 202-210. <http://dx.doi.org/10.1016/j.foodhyd.2015.10.002>.
5. Adebowale, K. O., Olu-Owolabi, B. I., Olayinka, O. O., & Lawal, O. S. (2005). Effect of heat-moisture treatment and annealing on physicochemical properties of red sorghum starch. *African Journal of Biotechnology*, 4(9), 928-933. Retrieved in 2017, June 17, from <https://www.ajol.info/index.php/ajb/article/view/71104>
  6. American Association of Cereal Chemists – AACC. (1995). *Approved methods* (8th ed.). Saint Paul: AACC.
  7. Martinez, C., & Cuervas, F. (1989). *Evaluación de la calidad culinaria y molinería del arroz. Guía de estudio para ser usada como complemento de la unidad auditorial sobre el mismo tema* (3. ed., 73 p.). Cali: CIAT. Retrieved in 2017, June 17, from <http://hdl.handle.net/10568/54016>
  8. Hormdok, R., & Noomhorm, A. (2007). Hydrothermal treatments of rice starch for improvement of rice noodle quality. *Lebensmittel Wissenschaft und Technologie*, 40(10), 1723-1731. <http://dx.doi.org/10.1016/j.lwt.2006.12.017>.
  9. Beuchat, L. R. (1977). Functional and electrophoretic characteristic of succinylated peanut flour proteins. *Journal Agriculture Chemistry*, 25(2), 258-260. <http://dx.doi.org/10.1021/jf60210a044>.
  10. Feng, W., Zhang, W., Wang, H., Ma, L., Miao, D., Liu, Z., Xue, Y., Deng, H., & Yu, L. (2015). Analysis of phosphorylation sites on autophagy proteins. *Protein & Cell*, 6(9), 698-701. <http://dx.doi.org/10.1007/s13238-015-0166-0>. PMID:26081468.
  11. Kaur, M., & Singh, S. (2019). Influence of heat-moisture treatment (HMT) on physicochemical and functional properties of starches from different Indian oat (*Avena sativa* L.) cultivars. *International Journal of Biological Macromolecules*, 122, 312-319. <http://dx.doi.org/10.1016/j.ijbiomac.2018.10.197>. PMID:30385334.
  12. Yu, L., Zhai, H., Chen, W., He, S., & Liu, Q. (2013). Cloning and functional analysis of lycopene  $\epsilon$ -Cyclase (IbLCYe) gene from sweetpotato, *Ipomoea batatas* (L.) Lam. *Journal of Integrative Agriculture*, 12(5), 773-780. [http://dx.doi.org/10.1016/S2095-3119\(13\)60299-3](http://dx.doi.org/10.1016/S2095-3119(13)60299-3).
  13. Zeng, J., Li, G., Gao, H., & Ru, Z. (2011). Comparison of A and B starch granules from three wheat varieties. *Molecules (Basel, Switzerland)*, 16(12), 10570-10591. <http://dx.doi.org/10.3390/molecules161210570>. PMID:22183883.
  14. Waramboi, J. G., Dennien, S., Gidley, M. J., & Sopade, P. A. (2011). Characterisation of sweet potato from Papua New Guinea and Australia: physicochemical, pasting and gelatinisation properties. *Food Chemistry*, 126(4), 1759-1770. <http://dx.doi.org/10.1016/j.foodchem.2010.12.077>. PMID:25213955.

Received: June 17, 2017

Revised: Jan. 28, 2019

Accepted: Apr. 09, 2019