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Isolation and physico-chemical and rheological characterisation of the Brazilian jalap starch (Operculina tuberosa Meisn.)

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ABSTRACT

The properties of the jalap starch (Operculina tuberosa Meisn.) were investigated and compared with other already known starches (potato and wheat starch). The jalap starch presented peak viscosity lower than the one from potato but higher than wheat starch, while the stability during the cooling down was higher than potato and wheat starch. The jalap starch presented X-ray pattern of type-A, which is typical of those from wheat starch. The rheological and physico-chemical characteristics presented by this source of starch were intermediate between those from wheat and potato, which makes it a promising commercial source to be explored, mainly in areas with food scantiness as in the Brazilian Northeast.

1. Introduction

The jalap (Operculina tuberosa Meisn.) is a "climber" which belongs to the *Convolvulaceae* family and is common in the secondary vegetation of coastal areas (Matos, 1998). They are found in tropical regions comprised between Antilles and Brazil and temperate regions of the Mexicans Andes, in muddy areas and areas of deep soil (Planchon & Bretin, 1937). This family of 55 genuses include 1650 species of herbs, shrubs, many of which are aerial (Enriquez et al., 1992; Noda, Miyahara, Kawasaki, & Okabe, 1987; Shellard, 1961). One of the most interesting characteristics of this family is the presence of arrays of secretory cells of glycosidic resins in foliate tissue and, especially, in its roots. These substances constitute one of the chemotaxonomics characteristic of this family, and the employment in the traditional medicine of some species (*Convolvulus, Exogonium, Ipomoea, Merremia* and *Operculina*) is associated to the purgative properties of its resins (Garcia-Argáez & Pérez-Amador, 1997; Pereda-Miranda & Bah, 2003; Pérez-Amador, García-Argáez, Contreras, Herrera, & Ríos, 1998). The jalap resin is composed of two distinct types: jalapin and *jalapurgin* or *convolvulin*. The *jalapurgin*, the main active component, is an odorless, white glycoside which is more irritable than the *jalapin* and in large doses can act as poison (Culbreth, 1927).

The Brazilian jalap has as its main species *Operculina macrocarpa* Urb. (syns *C. macrocarpus* L., *O. macrocarpa* (L.) Farwel), *O. alata* (Ham) Urban whose synonym is *I. operculata* (Matos, 1997, 1998; Ono, Kubo, Miyahara, & Kawasaki, 1989), and I. tuberosa L. or *O. tuberosa* Meissner (Matos, 1997, 1998). All those species are known popularly as "*batata de purga*" (purge potato). It is used due to its laxative and purgative properties, against skin diseases and in the treatment of the leukorrhea (Martins, Castro, Castelani, & Dias, 2000; Matos, 1982; Michelin & Salgado, 2004). It is also used as menstruation regulator (Michelin & Salgado, 2004).

The roots of O. *tuberosa* can reach up to 40 cm in length, as was the case in this study. They are hard and difficult to break or crunch, and are found mainly in the Brazilian Northeast, where the roots are big, starchy and rich in resins (Matos, 1998).

The jalap powder sold commercially is rich in starch. However, its starch is considered of less interest by virtue of the importance of its resins. Phytochemical investigations on the resin glycosides were initiated during the second half of the XIX century. However, the structures of their active components had remained poorly known and still are for some members of these purgative root species (Pereda-Miranda & Bah, 2003). Scientific researches about the physico-chemical properties of jalap starch are unknown in the literature. This study has the objective to investigate the physico-

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chemical and rheological properties of the jalap starch, comparing it with other known sources of starch.

2. Materials and methods

2.1. Raw material

The jalap roots (O. tuberosa Meisn.) were collected in the rural zones of São Luís/MA (Brazilian Northeast) during the months of May and June. For the purpose of comparison, commercial wheat (*Triticum aestivum*) and potato (*Solanum tuberosum* L.) starches were used.

2.2. Isolation and purification of the starches

The roots were washed, peeled and grated manually. Enough water was added to allow the sieving of the mass rich in starch in sieves of 200 "mesh". After this stage the suspension was left to decant for 24 h and the red coloured supernatant was discarded and the sediment washed several times over until the white powder was obtained. The sample was dried in a stove with forced air circulation at 40 °C until humidity of approximately 10% was reached.

2.3. Chemical composition of starches

The jalap starch was submitted to moisture and ash analysis according to the methods 930.15 and 942.05 of AOAC (1990), respectively. The total nitrogen content was determined in micro Kjeldahl equipment, and the protein content was calculated by the conversion factor 5.75, in agreement with method 984.13 of AOAC (1990). The total lipids content was determined gravimetrically in agreement with method 954.02 of AOAC (1990) with small modifications after extraction with methanol in Soxhlet apparatus. The amylose content was determined according to the method 6647 of ISO (1987).

The phosphorus content (P) was determined as described in Zakharov, Motyguilin, and Girmutdinov (2000), with some modifications. Experiments were performed on an atomic absorption spectrometer 1100B (Perkin-Elmer,) with a deuterium background corrector, an HGA-700 atomizer, and an AS-70 autosampler. The light source was a phosphorus hollow-cathode lamp. Atomic absorption was measured at the wavelength of 213.5 nm, and the spectral slit width was 0.7 nm. Graphite furnaces with a pyrolytic cover and pyrographite platforms from Perkin-Elmer were used throughout.

2.4. Swelling power and solubility

Swelling power and solubility of starch sample were determined by the procedure described by Schoch (1964). About 1 g of starch and 40 mL of water were submitted to heating for 30 min at 55, 65, 75, 85 and 95 °C. After cooling, the slurry of starch was centrifuged 5000g for 30 min. The supernatant was carefully separated from the precipitate, and 10 mL was removed to evaluate the percent of soluble, while the sediment was weighed for determination of the percentage of swollen granules.

2.5. Pasting characteristics

A Rapid Visco Analyser (RVA) RVA-4 (Newport Scientific Pty. Ltd., Australia) was used for pasting determinations. About 4.5 g of starch were mixed with 25 mL of distilled water to obtain a total weight of 29.5 g in the RVA cup. The samples were maintained for 1 min at 50 °C and the temperature increased progressively up to 95 °C (13 °C/min.), held at 95 °C for 3 min. then cooled to 50 °C (13 °C/min.) and held for 3 min. The test was concluded after 13 min. The analyses were run in triplicate.

2.6. Gelatinization

The T_o (onset temperature), T_p (peak temperature), T_c (final temperature) and ΔH (gelatinization enthalpy) parameters, were obtained by analysis in a differential scanning calorimeter DSC-50 (Shimadzu, Japan). Approximately 3 mg of starch were weighed in aluminium capsules and suspended in 15 μL of distilled water to obtain the starch/water proportion of 1:5 (w/w). The samples were then placed to rest for 1 h and the sealed capsules heated from 25 to 100 °C (10 °C/min.).

2.7. Granule morphology

2.7.1. Optic microscopy

The granule morphology was studied by a polarized and bright-field optics microscope, (Jenalab Pol, Carl Zeiss JENA, Germany), from the Microscopy Laboratory of the Biology Department of Federal University of Ceará. The starch powder was suspended in aqueous solution of glycerol and a drop was spread in a microscope slide and re-covered with a cover slip. The samples were observed at a magnification of 50× in polarized and bright-field optics and photographed with a digital camera.

2.7.2. Scanning Electron Microscopy (SEM)

The morphology and the size of the granules were determined with the use of a scanning electron microscope Philips XL-30 (Philips, Eindhoven, Netherlands), from the Microscopy Laboratory of the Mechanical Engineering Department of UFC. The samples were mounted on aluminium stubs and coated with a 50 μm thick gold film in an Emitech K550 Sputter Coater, operating at 5 mA and 50 kV. Starch granule diameter range was estimated by measuring 10–20 randomly selected granules from triplicates microphotographs.

2.8. X-ray diffraction

The analysis was carried out in an X-ray diffractometer Dmax-B (Rigaku, Japan), with a copper radiation in line Kα ($\lambda = 0.1542$ nm), operating at 40 KV and 25 mA. The region of scanning of the diffraction angle (2θ) was 3–40° (1/2°/min.).

3. Results and discussion

3.1. Isolation and chemical composition of the starches

The jalap is a hard and fibrous root. This characteristic and harvest time affects enormously the starch extraction yield, which was low, around 12% (g starch/g fresh roots). Another parameter which probably also influenced the starch extraction yield was the high amount of resins impregnated in starch powder, which made the purification process, difficult.

The results of chemical composition of the starches are shown in Table 1. The amylose content was found to be inside the normally verified range for the starch of roots, which was near to those of the cassava, but lower than that of the wheat.

The phosphorus content in the jalap starch was low in relation to wheat and potato starches. When compared to cereals, legume and tubers starches and potato starch, were the only ones to have relatively high phosphorus content. The majority of those are in phosphate ester form substituted in anhydroglucose molecules of amylopectin. The presence of phosphate groups in amylopectin