International Journal of Biological Macromolecules 76 (2015) 320–325

Contents lists available at ScienceDirect

International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac

Physicochemical and tablet properties of *Cyperus alulatus* rhizomes starch granules

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ARTICLE INFO

Article history:
Received 3 December 2014
Received in revised form 2 March 2015
Accepted 3 March 2015
Available online 10 March 2015

Keywords:
Starch
Cyperus alulatus
Rhizome
Tablet properties

ABSTRACT

The starch extracted from rhizomes of *Cyperus alulatus* (CA) was characterized for its physicochemical, morphological and tableting properties. Rhizomes of CA yield a significant quantity of starch granules (CASG) i.e., 11.93%. CASG was characterized in terms of moisture, ash and amylose contents, solubility and swelling power, paste clarity and water retention capacity. The swelling power was found to be significantly improved with the increase in temperature. Scanning electron micrographs revealed that the granule's surface was smooth, the granules were spherical, mostly round, disc like, and the size range was 6.65–12.13 μm. Finger print region in FTIR spectra confirmed its carbohydrate nature. The evaluated micromeritic properties of extracted granule's bulk density, tapped density, Carr's index, Hausner ratio, true density and porosity render unique practicability of CASG being used as an adjuvant in pharmaceutical solid dosage forms. Tablets prepared by using CASG showed higher mechanical strength and more disintegration time, which depicted the characteristic binding nature of the starch granules. As CASG is imparting better binding properties in less concentration and also it can be used in combination with the established starches to get the synergistic effect; this starch can be used commercially in the tablet preparation.

1. Introduction

Starch is a very attractive source for the development of biodegradable polymers. It is a resourceful biopolymer with great potential for pharmaceutical industrial applications. It can be found in the form of discrete semi crystalline particles, whose size, shape, morphology and composition depend on the botanical origin and are called as granules [1]. The properties of starch depend largely on the content of amylose and amylopectin as well as the shape and size of the granules.

Starches are broadly signed as multipurpose excipients in a range of solid dosage forms, especially as binding agents, diluents and disintegrants in tablet formulations due to their suitable physicochemical properties as well as their relative cheapness and inertness [2,3]. The adaptability of starches implies a need to continue to develop new starch excipients with appropriate properties to meet the special needs of drug formulators and the demands of novel formulations like delayed release drug designing.

Plants of the *Cyperaceae* family grow widely in tropical, subtropical, and temperate regions throughout the world. This is one of the earliest known and most important edible herbs, and several chemical investigations have been reported for members of this genus for Ethnomedical considerations. From one of them, *Cyperus alulatus* (CA), a crude drug prepared from the rhizomes (CAR) is used in indigenous medicine for abortive purposes [4]. It has been reported that Cyperus genus sedges contain almost equal or higher quantity of starch as potato or sweet potato tubers. As these are the underutilized plants, these can be used as an alternative source of starch to the existing commercial starches like potato, rice and maize as these sources are also used as a staple food [5]. The characterization and utilization of the *C. alulatus* starch have not been reported till the date. Hence, the present study is aimed to investigate the physicochemical and tableting properties of starch granules extracted from the rhizomes of *C. alulatus* (CASG).

2. Materials and methods

2.1. Raw materials

The plant species *C. alulatus* (CA) and its rhizomes were collected from Tamil Nadu, India and were authenticated (Sheet No. CNH/56/2013/Tech.II/22, specimen No. C-01) by Dr. V. P. Prasad, Botanical Survey of India, Kolkata 711103, West Bengal, India.

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http://dx.doi.org/10.1016/j.ijbiomac.2015.03.004
0141-8130/

2.2. Starch extraction

The collected rhizomes of *C. alulatus* were thoroughly washed with distilled water to take away the soil and other contaminants. Then, the rhizomes were cut into small pieces of approximately 2–3 cm length, and were steeped in aqueous sodium hydroxide (0.05%, w/v) solution at room temperature for 24 h [6]. After steeping, the liquid was drained off and the rhizomes were washed repeatedly with the sufficient amount of distilled water until the pH is neutral. The rhizome pieces were wet milled to slurry with the sufficient amount of distilled water. The starchy suspension was collected by passing the slurry through a sieve and was kept for settling of the starch. Then, the upper liquid layer was decanted, and the starch sediment was air dried. The dried powder was weighed and the yield (%, w/w) was calculated. Finally, the starch granules of *C. alulatus* rhizomes (CASG) were stored in air tight bags for further uses.

2.3. Elemental analysis

Using an Elemental Analyzer (Make – M/s Elementar, Germany; Model-Vario EL III), the carbon, hydrogen, sulphur and nitrogen elements of CASG were analyzed.

2.4. Physicochemical characteristics of the extracted CASG

2.4.1. Determination of moisture, ash, pH, amylose and mineral contents

Moisture content was evaluated by heating 1 g of CASG in an oven at 105 °C to constant weight. Ash residue was assessed by incinerating a pre-weighed sample at 500 °C in a muffle furnace for 12 h [7]. A 1% (w/w) suspension (sample in distilled water) was used to determine the pH. The amylose content was determined by the method described by Varma et al. [8]. An ICP-OES (inductively coupled plasma optical emission spectroscopy) spectrometer (optical 2100DV, Perkin Elmer, USA) was utilized to analyze the mineral content of the sample. For the process dried CASG (0. 2 g) was digested in microwave digestion unit with H_2O_2/HNo_3 mixture.

2.4.2. Swelling and solubility power of CASG

The process described by Deepika et al. was used to determine the swelling and solubility power [9]. A 1% (w/v) suspension of CASG was weighed into a graduated centrifuge tube with distilled water. The tubes containing slurries were immersed and heated in water bath at a temperature range from 30 to 90 °C at 10 °C interval for 30 min, with continuous shaking for every 5 min during heating. The tubes were cooled to room temperature and then centrifuged for 30 min at 3000 rpm. The supernatant was carefully sucked into pre-weighed Petri dishes and evaporated in the hot air oven at 110 °C for 4 h. After drying the supernatant, the variation in the weight of the petri dish was taken as the weight of soluble starch percentage. Solubility was determined as gram of soluble starch per gram starch on the dry weight basis. The weight of the swollen starch was measured and used to estimate the swelling power as gram of sediment paste per gram starch. All the measurements were done in triplicate. Solubility and swelling power were calculated as:

$$\text{Swelling power}(\%) = \frac{M_{WS}}{M_{Ds} \times (100 - \%\text{solubility})} \times 100 \quad (1)$$

$$\text{Solubility power}(\%) = \frac{M_{ss}}{M_{Ds}} \times 100 \quad (2)$$

where M_{DS} is the mass of dried starch (g); M_{SS} is the mass of soluble starch (g) and M_{WS} is the mass of wetted starch (g).

2.4.3. Water retention capacity (WRC) of CASG

Estimation of WRC of CASG was done by following the procedure depicted by Kulkarni et al. [10]. A suspension (1 g dry starch in 15 mL distilled water) was agitated for 1 h and then was centrifuged for 10 min at the rate of 3000 rpm. The supernatant was depleted from the wetted starch and was drained for 10 min. The mass of wet starch was taken, and the result was specified as percent (w/w) on the dry weight basis.

2.4.4. Paste clarity of CASG

A 2% CASG dispersion was prepared in distilled water according to the method of Deepika et al. [9]. It was heated in a boiling-water bath for 30 min with continuous stirring. After cooling, the dispersion was stored at 4 °C for 5 days. Using distilled water as blank, the % transmittance (paste clarity) was measured at 640 nm using UV–VIS Spectrophotometer for five days at a gap of 24 h.

2.5. Morphological characteristics of the CASG

Scanning electron microscope (JEOL – Japan, JSM 6390 LV) at an accelerating voltage of 5–10 kV was used to study the morphology of CASG. For this study, the dried sample of CASG was sprinkled on the double sided adhesive tape, which was mounted on the aluminium stub, and then it was coated with platinum to make the sample conductive.

2.6. Infrared spectroscopy of CASG

To determine the chemical nature of CASG, Fourier transforms infrared spectrophotometer (FTIR-8400 S, Shimadzu, Japan) was used. The FTIR spectrum was obtained by using KBr pellets of CASG, and by operating the instrument at the frequency range 4000–400 cm^{-1}.

2.7. Powder characteristics

2.7.1. Bulk, tap and true densities of CASG

As per European Pharmacopoeia, bulk density was calculated as the ratio of weight (W_{ds}) of CASG sample to initial volume (V_i) and tap density was calculated as the ratio of weight (W_{ds}) of same sample to final volume (V_f) after tapping. The initial volume (V_i) occupied by the powdered sample (1 g) after placed in 10 mL of measuring cylinder was noted and the final volume (V_f) was noted down after tapping gently 100 times on the table. The measurements were done in triplicate [7].

True density (T_d) was determined using acetone as solvent by liquid displacement method and calculated as:

$$T_d = \frac{W_{ds}}{[(A + W_{ds}) - B] \times S} \quad (3)$$

where W_{ds} is the weight of dried sample, S is the specific gravity of solvent, A is the weight of the bottle with solvent, B is the weight of the bottle with solvent and dried sample.

2.7.2. Powder flow ability of CASG

The powder flow ability of CASG was ascertained by using Carr's Index (CI) and Hausner ratio (HR). HR was calculated as the ratio of tapped density (D_{tapped}) to bulk density (D_{bulk}) of the dried sample [11]. Determination of CI was done using the following equation.

$$CI = \frac{D_{tapped} - D_{bulk}}{D_{tapped}} \times 100 \quad (4)$$

2.7.3. Angle of repose

The angle of repose (θ) was estimated by the fixed funnel and free standing cone method. Which was explained as, a funnel was