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Physicochemical and release kinetics of natural and retrograded starch of Indian palmyrah shoots

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ABSTRACT

Starch was isolated from the shoots of Indian palmyrah (*Borassus flabellifer* L.) and it was subjected to the process of retrogradation. The influence of retrogradation on morphological, physicochemical and drug release properties was studied. Retrogradation of native starch changed its morphology from oval, elliptical to crystalline rods. Due to retrogradation there is an increase in amylose content and better hydration capacity, swelling and solubility power. The micromeritic properties of native and retrograded starch uncover its usage as excipients in tablet manufacturing. The retrograded starch showed better powder characteristics to that of native starch. The characteristic peaks for D-glucopyranosyl ring confirms the carbohydrate nature of starch. The TGA data reveals that the retrograded starch shows less bound water to that of native starch during the first decomposition step. In-vitro release study reveals that the retrograded starch attained a better release retardant property and was best explained by Hixson–Crowell model. The result showed that retrograded starches can be used for the preparation of sustained release tablets.

1. Introduction

The palmyrah or toddy palm (*Borassus flabellifer* L.) is an ancient plant belonging to the family *Arecaceae*. It is widely distributed and cultivated in tropical Asian countries such as India, Thailand, Bangladesh, Myanmar, Sri Lanka, Malaysia, etc. [1]. The palm is very multifaceted and it can produce several beneficial products. The shoot of *Borassus flabellifer* L. (fleshy food storage scales) grow by the germination of seed to a height of 12–15 cm and the surplus of carbohydrate content is reserved in the means of starch [2]. The shoots are boiled, dried, grounded and sieved to give *Borassus flabellifer* L. flour, having a high nutritive and energy value which is comparable to rice and wheat flour. These shoots have considerable amount of fibre and are also responsible for low glycemic index. These shoots are regarded as common food stuffs and are eaten after boiling and could be a substitute for many existing starches [3].

Starch is a biocompatible, biodegradable, renewable and inexpensive polymer, abundantly occurring in nature as a major polysaccharide reserved in higher plants [4,5]. Starch is having a very constructive role in tablet production due to its inertness, cheapness and can be used as filler, binder, disintegrant, glidant, emulsifying and suspending agent [6]. There is an extensive work done in the starches isolated from the tubers like potato, cassava, sweet potato, etc., due to their wide range of availability [7]. However, Indian palmyrah shoot starch as binder in tablet formulation needs to be explored.

Retrogradation, a recrystallization process which occurs during storage after starch pasting and there is a change in crystalline structure which leads to the development of ordered double-helical structure where restructuring of amylose and amylopectin takes place [8]. During retrogradation, the rapid and irreversible gelation of amylose and slower reversible recrystallization of amylopectin chains takes place. In the first stage of retrogradation, association of linear amylose molecules occurs and later on the amylopectin molecules crystallizes to form rigid starch gel [9].

During the process of retrogradation, there is an increase in resistant starch content which increases the stability of starch while storing. The increase in resistant starch content would effect the adjuvant properties of the starch. The current work aims to explore the effect of retrogradation on the physicochemical characteristics and drug release kinetics of tablets prepared using *Borassus flabellifer* L. shoot starch.

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2. Materials and methods

2.1. Materials

Drug diclofenac sodium was kindly received as gift sample from Metrix Healthcare Private Limited, Ahmedabad, India. All chemicals used were of analytical grade and were procured from Sigma–Aldrich (India). Analytical reagent grade chemicals and ultrapure water from Millipore water purification system (Millipore, United States) was used to prepare all solutions.

2.2. Starch isolation

The *Borassus flabellifer* L. (Indian palmyrah) shoots was procured from the market of Andhra Pradesh, southern region of India, where the species of *Borassus flabellifer* L. is abundantly found. The shoots were identified with the help of standard local flora and its identification was confirmed with the herbarium in Andhra university. The young shoots of *Borassus flabellifer* L. were collected and the shoots were washed with distilled water to remove the traces of contaminants. Then, the shoots were sliced into pieces and subjected for wet grinding. The obtained slurry was steeped in 0.05% w/v NaOH solution for 24 h at 25 °C [10]. After 48 h of steeping, the resultant slurry was washed with distilled water until the supernatant liquid becomes clear. Then the slurry was passed through a sieve to remove the fibrous content and a filtrate of white suspension was obtained. The starch suspension was allowed for settling and the upper supernatant liquid was decanted. Thus, the sedimented starch obtained was allowed for air drying till 48 h. Finally the dried starch (BS) was packed in tightly closed in polyethylene bags and the percentage yield was calculated.

2.3. Retrogradation of starch

Retrogradation of starch was done according to the method described by Zięba et al. [11]. A starch suspension of 10% w/v concentration was prepared from native starch and was subjected for heating to a temperature of 70 °C for 30 min, with continuous stirring to form a paste so as to prevent the sedimentation of starch. The resulted suspension was kept in a water bath for 6 h at a temperature of 94 °C to form a thick paste. Thus the suspension was left for 12 h at a temperature of 20 °C and then it was frozen in ultralow temperature cabinet at −20 °C for 3 days and unthawed for 2 days at 20 °C. This repeated freeze thaw cycles results in the formation of a spongy structure which is further rinsed with distilled water, subjected to drying at a temperature of 35 °C for 24 h in an air flow dryer. Finally it was grounded and passed through a sieve of mesh diameter 400 μm and the finely powdered material (RBS) was stored in polyethylene bags for further use.

The percent of retrogradation rate was computed according to the following equation [12]:

$$\text{Retrogradation rate}\,(\%) = \frac{m}{m_0} \times 100 \tag{1}$$

where, m_0 and m are the weights of native (g) and retrograded starch (g) respectively.

2.4. Physicochemical characteristics of native and retrograded starch

2.4.1. Proximate analysis of starches

The proximate analysis of starches such as moisture content, pH, mineral content and water holding capacity (WHC) of native and retrograded starch were determined according to the method described by Rodrigues and Emeje [13].

2.4.2. Amylose content

The amylose content of native and retrograded starch is determined by the method described by Srichuwong et al. [14]. To 100 mg of starch, 1 ml of 95% ethanol was added and boiled for 10 min. Then it was cooled and made up to 100 ml with distilled water. To the 5 ml of the above solution, 1 ml of 1 N acetic acid, 2 ml of iodine solution were added and the volume was made up to 100 ml with distilled water. After 20 min, the absorbance of the starch solution was determined spectrophotometrically at 620 nm against the blank.

$$\%\text{Amylose} = 3.06 \times \text{Absobance} \times 20 \tag{2}$$

Here, 3.06 is conversion factor.

2.4.3. Elemental analysis

The elements like carbon and hydrogen were determined by using an Elemental Analyzer (Make – M/s Elementar, Germany; Model-Vario EL III).

2.4.4. Water holding capacity (WHC)

Water holding capacity of native and retrograded starch samples of *Borassus flabellifer* L. was determined according to the method described by Deepika et al. [15]. A suspension of 1 g starch was prepared in centrifuge tube with 15 ml of distilled water and the tube was subjected to agitation for 1 h. Then it was centrifuged at 3000 rpm for 10 min. The supernatant liquid thus obtained was decanted and the wet starch was weighed.

$$\text{WHC}\ (\%) = \frac{W_{WS}}{W_W} \times 100 \tag{3}$$

Here, W_{WS} is the weight of wet starch (g) and W_S is the initial weight of starch (g) on dry basis.

2.4.5. Swelling and solubility power

The suspensions of starch (1%, w/v) was prepared by using distilled water and are subjected to a different temperatures of 30 °C, 40 °C, 50 °C, 60 °C, 70 °C, 80 °C, 90 °C for 30 min in temperature controlled water bath. The samples were left for cooling at room temperature and are centrifuged at 3000 rpm for 15 min. In a pre-weighed petridish, the supernatant liquid thus obtained was carefully decanted and the swollen sedimented starch was weighed [16,17]. Then the supernatant liquid was evaporated overnight at 110 °C and residue was obtained. The weight of this residue represents the solubilized portions of starch in water [18,19]. Swelling and solubility power were calculated according to the equations [20]:

$$\text{Swelling power}\,(\%) = \frac{W_{WS}}{W_{WS} \times (100 - \%\ \text{Solubility})} \times 100 \tag{4}$$

$$\text{Solubility}\ (\%) = \frac{\text{weight of soluble starch} \times 100}{W_S} \tag{5}$$

where, W_{WS} is weight of wet starch (g) and W_S weight of starch on dry weight basis (g).

2.5. Micromeritic properties

2.5.1. Bulk and tapped densities

A weighed amount (W_t) of each of the powder samples was placed in a 100 ml cylinder of a volumeter and the volume occupied without tapping was noted as bulk volume (V_o). Then it is subjected for 100 tappings and the volume was noted as tapped volume (V_f). The bulk density (ρ_b) and tapped density (ρ_f) were computed as the ratio of weight to volume. These parameters were performed statistically with triplicate analysis [21].