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Physicochemical properties and release characteristics of starches from seeds of Indian Shahi Litchi

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ABSTRACT

Many conventional sources of starches are from staple foods. Non-conventional and cheap sources of starch are being explored. Starch was isolated from Shahi Litchi seeds using two extraction media; acidic (citric acid 0.3%, w/w; LC) and alkaline (NaOH 0.5%, w/w; LN). Each starch was investigated for various properties such as structural, morphological and functional. The percentage yield of LN and LC was 11% and 12.6%, respectively. Morphological properties of both starches show same structural makeup, but compound granules were in LN starch. Moisture content, amylose content was found to be higher LC starch than in LN starch, which indicates that extraction media affects the properties of starch. FTIR confirmed the carbohydrate nature of the both isolated starches. TGA data of both starches reveal slight difference in stability with temperature. In vitro release of both starches shows the release up to $58.95 \pm 0.04\%$ and $67.184 \pm 0.07\%$ in 5 h for LN and LC, respectively, that indicates that these starches can be used in delayed drug delivery and targeting drugs to the colon.

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1. Introduction 21

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Starch, pre-eminent carbohydrate constituent in most of the 22 plants, is widely used in industries for processing food and non-23 food articles owing to its diversified applications. A comprehensive 24 research has already been done on the properties of main starches 25 of commerce obtained from seeds (corn, wheat and rice), tubers and 26 roots (potato, cassava, and yams) because of their prompt avail-27 ability [1–3]. Starches from different sources are varied in their 28 morphological, structural and physicochemical properties physi-29 cochemical properties. The diverse applications of starch demands 30 specific functional characteristics [4,5]. In pharmaceutical indus-31 32 tries, starch is used as an excipient for drug delivery, as binder, disintegrants, suspending agent and emulsifying agent [6]. The lit-33 erature contains sparing information on isolation and properties of 34 starches from non-conventional sources, such as seeds of fruits. 35

Shahi Litchi is available in Muzzafarpur district of Bihar, India. The seed of this plant is considered to be a waste, but it contains 37 a large amount of starch. However, these starches have not been 38 explored for its utility in drug delivery. Therefore, the present study 39 includes some physicochemical, thermal, functional and morpho-40 logical study of starch isolated from Indian Shahi Litchi seeds by 41

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using different extraction media and its application in drug delivery for its utility in pharmaceutical industries as an excipient.

2. Materials and methods

2.1. Materials

Authenticated Shahi Litchi seeds were procured from the National Research Centre for Litchi, Muzzafarpur, Bihar, India. All chemicals used were of analytical grade reagents and procured from Sigma-Aldrich (India). Analytical grade chemicals and pure water from Millipore water purification system (Milli-pore, United States) was used for the preparation of all the solutions.

2.2. Isolation of starch from Litchi seeds

Isolation of starch from Shahi Litchi seeds was done by the method as described by Gorosquera et al. [7] and Kulkarni et al. [8] with little modification. Approximately weighed seeds (500 g) were peeled and steeped in citric acid solution (0.3%, w/w) and sodium hydroxide (0.5%, w/w) separately, and seeds were then ground using low speed blender with water (500g seeds:500g distilled water) for 2 min. The resulting homogenate was passed through the sieve 80 # and the liquor was kept aside for settling. The residue retained on the sieve was rinsed with distilled water to get maximum starch recovery. The sediment starch was washed

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thoroughly until the washing water was clean. The starch cake
obtained was allowed to dry in air and packed in an airtight container. The starch isolated using sodium hydroxide, and citric acid
was termed as LN and LC respectively. The percentage yield of each
starch was calculated on a dry basis of the material taken.

A 2.3. Moisture, ash, amylose, pH, element and mineral content determination

Ash content of isolated starches was determined by incinerating 70 the sample at 500 °C [9]. Moisture content of Litchi seed starches 71 was estimated by heating the samples at 105 °C to a constant weight 72 in hot air oven [9], pH of isolated starch was determined by using 73 digital pH meter. Estimation of amylose content was done by the 74 method of [10]. The amount of carbon, hydrogen, nitrogen and 75 sodium were determined by using Elemental Analyzer (M/s Ele-76 mentar, Germany; Model-Vario EL III). For determination of mineral 77 contents in starch, ICP-OES method was used. Shortly, starch sam-78 79 ples were first treated with a digestion mixture in a microwave digester. Resulting clear solution was then taken into 100 ml vol-80 81 umetric flask and fill up to the mark with Millipore water. This solution was used to determine mineral content. 82

83 2.4. Water-holding capacity

Water holding capacity (WHC) of isolated starches was evaluated as per the method described by Yamazaki [11] and Medcalf and Gills [12] with slight modification. Starch suspension was prepared by using 1 g of starch in 15 ml of distilled water, and the resulting suspension was stirred for 1 h. It was followed by centrifugation at 3000 rpm for 10 min. Supernatant was then poured off, and wet starch was weighed. Water holding capacity was calculated in percent (w/w) on the dry basis.

92 2.5. Micromeritic properties

Micromeritic properties of each starch were assessed in for bulk
 density, tapped density, angle of repose, Carr's index and Hausner's
 ratio. All these properties were evaluated according to method as
 described in European Pharmacopoeia, 2008 [13].

2.5.1. True density

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Liquid displacement method was used to calculate true density [14].

$$TD = \frac{W_1}{(A+W_1)-B} \times SG$$
(1)

 W_1 = weight of sample; SG = specific gravity of solvent; *A* = weight of the bottle + solvent; *B* = weight of the bottle + solvent + sample.

103 2.5.2. Porosity

¹⁰⁴ Porosity [15] of isolated Litchi seed starches was determined as

$$P = 1 - \left(\frac{BD}{TD}\right) \times 100$$
 (2)

Here P = porosity; BD = bulk density; TD = true density.

107 2.6. Swelling and solubility power

To analyze swelling and solubility power method of Chen et al. [16] was used. 2% (w/v) suspension of each starch was heated at different temperature of 30, 40, 50, 60, 70, 80 and 90 °C for 30 min, being stirred every 20 min. Suspensions were then centrifuged at 3000 rpm and supernatant containing water soluble material

Table 1

Composition of formulation of tablet using different percentage of isolated starches (LN and LC).

Ingredients	Quantity per each tablet (% w/w)		
	1	2	3
Drug	71.4	71.4	71.4
Lactose	19.1	18.1	17.1
Starch	2.5	3.5	4.5
Sodium CMC	5	5	5
Talc	1	1	1
Magnesium stearate	1	1	1

1 for LN1 and LC1, 2 for LN2 and LC2, and 3 for LN3 and LC3.

was dried and weighed. The residue left was swollen starch. The swelling and solubility power was computed as

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Swelling power (g/g) =
$$W_{WSt} \times \frac{100}{W_{St}} \times (100 - \% \text{ solubility})$$
 (3)

Solubility (%w/w) = Weight of soluble starch $\times \frac{100}{W_{St}}$ (4)

Here W_{WSt} = weight of wet starch (g); W_{St} = weight of starch on the dry weight basis (g).

2.7. Morphology of Shahi Litchi seed starches

The morphology of each sample was observed under Scanning electron microscope (JEOL-Japan, JSM 6390 LV). The dried samples of starches were affixed to a metal stub with double sided adhesive tape. The affixed samples were then coated with platinum to make them conductive and morphology of starches was observed under an accelerating voltage of 10 kV.

2.8. Infrared spectroscopy of starches

The structure of both starch samples was evaluated by Fourier transform infrared (FTIR-8400 S, Shimadzu, Japan) spectroscopy. The IR spectra was determined by mixing the samples with KBr in the ratio of 1:1 with range of 4000–400 cm⁻¹ and pressing into tablets before passing through radiation.

2.9. Thermal properties

Thermo gravimetric analysis apparatus (DTG-60, Shimadzu, Japan) was used to study thermal properties of each starch sample. Samples were loaded in platinum pan, and it was heated at a rate of 10 °C from 25 °C to 400 °C. Thermal analysis was done in the presence of nitrogen (flow rate 50 ml/min).

2.10. Tablet preparation

Tablets were prepared using various concentrations of isolated native starches (2.5%, 3.5%, 4.5%, w/w). These different concentrations are represented as LN1 (2.5%), LN2 (3.5%), LN3 (4.5%) for starches isolated using sodium hydroxide and LC1 (2.5%), LC2 (3.5%), LC3 (4.5%) for starches isolated using citric acid. Paracetamol was used as a model drug and lactose, magnesium stearate, sodium CMC and talc were taken as excipients. All the ingredients were subjected to grind to obtain a required degree of fineness and passed through sieve no. 80 individually. Required quantities of ingredients (Table 1) were taken, and granules were prepared using wet granulation method. The properties of granules such as bulk density, tapped density, true density, angle of repose, Carr's index, Hausner's ratio, angle of repose and porosity were studied. 16 station tablet punching machine (Cadmach, Ahmadabad, India) was used for granule compression.

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