Pharmacy Education (2023) 23(2) 47 - 52
https://doi.org/10.46542/pe.2023.232.4752

IAI SPECIAL EDITION

RESEARCH ARTICLE

Morphology and physicochemical properties of starch extracted from Indonesian ginger

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Keywords
Morphology
Physicochemical property
Starch
Zingiber officinale

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Abstract

Background: Starch of ginger (*Zingiber officinale*) is potentially developed as a pharmaceutical excipient of dosage forms. In Indonesia, there are three varieties of ginger, namely *Z. officinale* var. Roscoe, *Z. officinale* var. Amarum, and *Z. officinale* var. Rubrum. **Objective:** To characterise the morphology and physicochemical properties of starches from the three ginger varieties. **Method:** The morphology of ginger starch was characterised using a light microscope and Scanning Electron Microscope (SEM). Physicochemical properties were determined using several methods from previous studies. **Result:** Starch granules of three ginger varieties were elliptical and showed an eccentric hilum with lamella. Starch of *Z. officinale* var. Amarum (SZA) had enormous size and led the highest swelling power, solubility, density, emulsion capacity, and moisture content. Starch of *Z. officinale* var. Roscoe (SZRo) had a more acidic pH (6.44 ± 0.02) without foaming capacity. SZA and SZRo had higher browning temperatures, lower gelatinised temperatures, and water absorption. Starch of *Z. officinale* var. Rubrum (SZRu) exhibited the highest charring temperature (279.00 ± 1.00 °C). Particle size distribution of all starches showed that more than 98% of particles had less than 179 µm in size. **Conclusion:** Ginger starch can be potentially developed as an excipient of pharmaceutical dosage forms in an optimum formula.

Introduction

Starch is the main carbohydrate reserve and a source of abundant polysaccharides in plants. It is stored as granules in chloroplasts of green leaves, amyloplasts of seeds, tubers, and rhizomes. Starch consists of about 10-38% of amylose and amylopectin. It is easily isolated with high purity, and the process requires low cost. Many industries have used starch in their production processes, including pharmaceutical industries as fillers, glidants, binders, disintegrants, gelling agents, bulking agents, encapsulating agents, and water retention agents in various pharmaceutical dosage forms (Adebowale *et al.*, 2014). The amount of starch used as excipients or additives for pharmaceutical dosage forms is still limited. Therefore, further exploration is needed to obtain sources of starch from various plants in Indonesia.

One source of starch potentially developed as an excipient for pharmaceutical dosage forms is ginger (*Zingiber officinale*) rhizome because of its abundance and low price. In addition, ginger starch is an unutilised secondary product of various production processes, such as essential oils isolation, the production of syrup, candy, and other food or beverage products containing ginger.

In Indonesia, there are three common ginger varieties, namely elephant ginger (*Zingiber officinale* var. Roscoe), yellow ginger (*Zingiber officinale* var. Amarum), and red ginger (*Zingiber officinale* var. Rubrum) (Ningsih *et al.*, 2020). In previous studies, several parameters from ginger starch were characterised, but no study was conducted to characterise the starch of three ginger varieties (Adebowale *et al.*, 2014; Afolayan *et al.*, 2014; Kolawole *et al.*, 2013). The difference in ginger varieties may cause differences in their contents, including starch

characteristics. Therefore, it is necessary to characterise rhizome starch from three varieties of ginger. The study evaluated the morphology and physicochemical properties of starch from three ginger varieties.

Method

Material

Rhizomes of three ginger varieties were collected in Kencong Subdistrict, Jember, East Java, Indonesia. The plant was authenticated at Plant Laboratory, State Polytechnic of Jember, with voucher 16/PL17.3.1.02/LL/2019. The other materials were distilled water and iodine solution obtained from Brataco (Jakarta, Indonesia).

Isolation of ginger starch

The fresh rhizomes were washed, peeled, sliced, added distilled water and milled to obtain a slurry. It was soaked for five hours, washed with distilled water, and filtered. The filtrate was kept for two hours for separation. The supernatant was removed by decantation. The starch was re-dispersed, decanted to eliminate impurities, and dried using the oven (Afolayan *et al.*, 2014).

Morphology analysis

Starch powder was placed on an object glass, dripped iodine solution, and covered by a cover glass. It was analysed using a light microscope (Olympus BX53F). In addition, the starch powder was put on SEM (Hitachi Tabletop Microscope TM 3000) specimen stub using adhesive tape and coated in an ion sputter coater (Hitachi E-1045) with platinum. The analysis used five kV of accelerating voltage.

Physicochemical properties

Swelling power

0.1 g of starch powder was added to ten mL of distilled water, heated for 30 min at 50-90 °C, and stirred. The supernatant and starch paste was separated by centrifugation at 1500 rpm for 20 minutes and decantation. The supernatant was removed, and the paste was weighed. The swelling power was determined using the following formula:

$$swelling\ power = \frac{weight\ of\ starch\ paste}{weight\ of\ starch\ powder}$$

Solubility

Half gram of starch powder was added to ten mL of distilled water, heated for 30 minutes at 50-90 °C, and shaken for 30 min. It was centrifuged at 1500 rpm for 30 min and decanted to obtain five mL of the supernatant. Then, it was dried at 105 °C for 24 hours and weighed. The solubility was calculated using the following formula:

$$\%solubility = \frac{weight\ of\ dissolved\ starch}{weight\ of\ starch\ powder}\ x\ 100$$

pH

Two grams of starch powder was dispersed in ten mL of distilled water and vortexed for five minutes. Determination of pH was carried out using a pH meter.

Browning and charring temperature

Starch powder in several capillary tubes was placed in melting point apparatus to obtain browning and charring temperature.

Foam capacity

One gram of starch powder was vortexed in 50 ml of distilled water for five minutes.

Emulsion capacity

One gram of starch powder was mixed with five mL of distilled water for 30 sec, added to five mL of coconut oil and mixed for 30 sec. The mixture was separated by centrifugation at 1600 rpm for five minutes. The volume of oil emulsified per gram of starch powder was calculated as emulsion capacity.

Gelatinisation temperature

One gram of starch powder was dispersed with ten mL of distilled water, heated until a slurry was formed, and the temperature was measured as gelatinisation temperature.

Water absorption

0.5 g of starch powder was mixed with ten mL of distilled water for two minutes, separated by centrifugation at 1500 rpm for 20 minutes and decantation. The supernatant was removed, and the hydrated starch was weighed. The weight of water absorbed per 100 grams of starch powder was calculated as water absorption.

Moisture content

Six grams of starch powder was placed in the moisture analyser apparatus. The temperature was set to 105 °C.

Tapped density

The starch powder was placed in a measuring cylinder (100 mL) set in a tap density tester, and tapped 500 times to obtain compressed volume. Tapped density was calculated by dividing the weight of starch powder by its volume.

Bulk-density

The starch powder was placed in a measuring cylinder (100 ml) for its volume. Bulk density was calculated by dividing the weight of starch powder by its volume.

True density

The empty pycnometer was weighed (a), followed by weighing the pycnometer containing distilled water as solvent (b) and weighing the solvent (c). One gram of starch powder was placed in the pycnometer, and weighed (d). The solvent was poured until the pycnometer volume and the total weight was measured (e). True density was calculated using the following formula:

$$true\ density = \frac{d-b}{(d-b)+(c-e)}\ x\ \frac{c-b}{a}$$

Compressibility

Compressibility of the starch powder was determined using Carr's index and the Hausner ratio was calculated using the following formula:

$$Carr's\ index = \frac{tapped\ density - bulk\ density}{tapped\ density}\ x\ 100\%$$

$$Hausner\ ratio = \frac{tapped\ density}{bulk\ density}$$

Distribution of particle size

Determination of particle size distribution was carried out using a sieving test. 100 grams of starch powder was sieved at 30 rpm for 20 minutes. The starch powder left on each sieve was weighed.

Statistical analysis

The data were expressed as mean values ± standard deviation (n=3) and analysed using one-way analysis of variance (ANOVA) with the Least Significance Different (LSD) method to test significant differences. Values of $p<0.05$ showed significant differences among samples ($\alpha=0.05$).

Result

Rhizomes of *Zingiber officinale* var. Roscoe, *Zingiber officinale* var. Amarum, and *Zingiber officinale* var. Rubrum yielded 4.33%, 4.41%, and 4.46% of starch powder, respectively. The yield difference could be due to a variety of species affecting its chemical composition. The starch was a white tasteless, non-hygroscopic, and odourless powder.

Figures 1 and 2 showed the light microscope and SEM microscope images of starch powder for three ginger varieties as morphological characteristics of isolated starch.

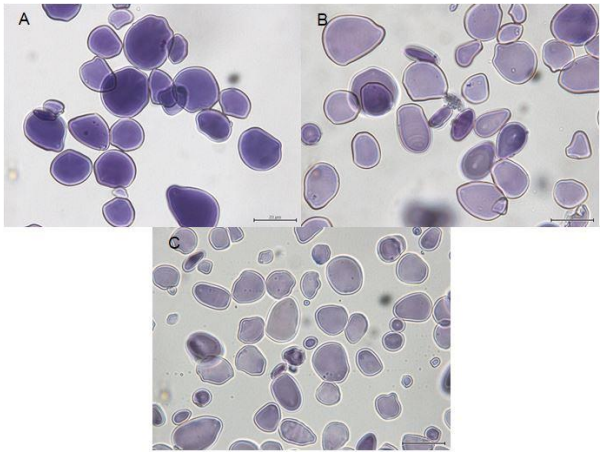

Figure 1: Micrograph of SZRo (A), SZA (B), and SZRu using light microscope in 1000x magnification

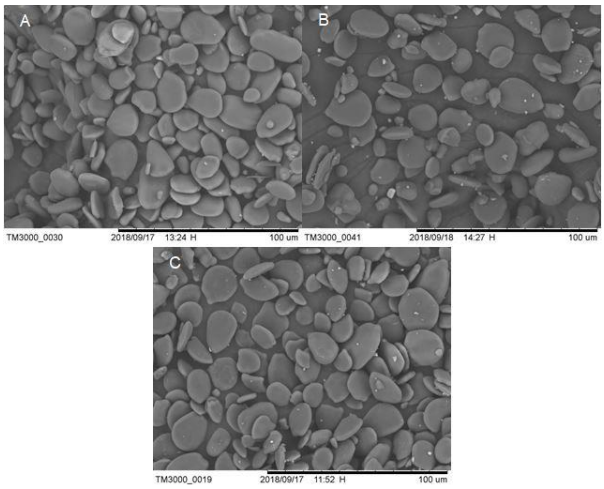

Figure 2: Micrograph of SZRo (A), SZA (B), and SZRu using SEM in 1000x magnification

The swelling power and solubility of SZRo, SZA, and SZRu for a temperature range of 50-90 °C are shown in Figure 3. SZA has higher swelling power, solubility, and moisture content than other starch (Table I). However, all starches meet the maximum permitted moisture content for herbal medicines (12.0% w/w) (Badan Standarisasi Nasional, 2019). In addition, all starches show a similar capacity for water absorption.

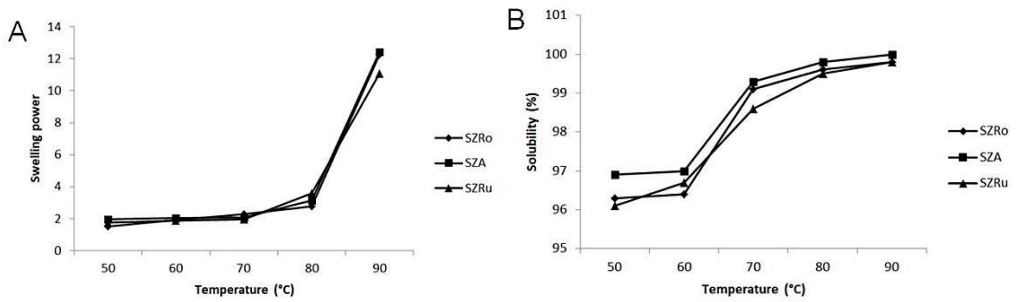

Figure 3: Swelling power and solubility of starch powder from three ginger varieties

SZA indicates a more alkaline pH (Table I). The browning and charring temperatures are high for all starches i.e. more than 244 °C and 271 °C. All starches have similar browning and charring temperatures. The results show that SZRo has no foam capacity. Meanwhile, SZA and SZRu have low foam capacity. SZA exhibits the highest emulsion capacity. Its gelatinisation temperature is not significantly different from SZRu. Tapped and bulk density measurements show similar values for all starches. Carr's index and the Hausner ratio of SZRu exhibited the highest value. It is also known that most of the particle sizes are less than 179 µm.

Table I: Several physicochemical properties of starch powder of three ginger varieties

Parameters	SZRo	SZA	SZRu
Granule size			
Length (µm)	12-32	11-34	5-28
Width (µm)	8-25	10-26	5-18
Water absorption (%)	62.62±5.03^{ab}	59.23±8.05^{a}	70.00±3.46^{b}
Moisture content (%)	4.08±0.31^{a}	5.07±0.12^{b}	3.47±0.42^{a}
pH	6.44±0.02^{a}	6.65±0.03^{b}	6.55±0.01^{c}
Browning temperature (°C)	248.33±0.58^{a}	249.33±0.58^{a}	244.00±1.00^{b}
Charring temperature (°C)	271.33±0.58^{a}	275.00±1.00^{b}	279.00±1.00^{c}
Foam capacity (%)	-	50.00±0.00^{a}	66.67±28.87^{a}
Emulsion capacity (%)	46.96±0.23^{a}	48.69±0.67^{b}	43.75±1.58^{c}
Gelatinisation temperature (°C)	64.67±1.15^{a}	65.83±3.01^{ab}	67.67±1.15^{b}
Density			
Tapped density (g/mL)	0.80±0.08^{a}	0.81±0,01^{a}	0.79±0.00^{a}
Bulk density (g/mL)	0.60±0.06^{a}	0.61±0.00^{a}	0.58±0.00^{a}
True density (g/mL)	5.10±0.00^{a}	5.07±0.00^{b}	5.06±0.00^{c}
Compressibility			
Carr's index	25.00±0.00^{a}	25.33±0.58^{a}	26.00±0.00^{b}
Hausner ratio	1.33±0.00^{a}	1.34±0.01^{ab}	1.35±0.00^{b}
Particle size:			
<179 (µm)	98.37±0.32^{a}	98.14±0.19^{a}	99.19±0.08^{b}
180-249 (µm)	0.49±0.05^{a}	0.57±0.07^{a}	0.23±0.01^{b}
250-424 (µm)	0.35±0.08^{a}	0.39±0.03^{a}	0.21±0.06^{b}
425-849 (µm)	0.33±0.03^{a}	0.42±0.04^{b}	0.20±0.15^{c}
≥850 (µm)	0.19±0.07^{a}	0.31±0.21^{b}	0.08±0.07^{a}

†Data are average of samples (mean) ± SD (n=3). The different superscript letters (in the same row) showed significant differences according to the LSD test ($p < 0.05$).

Discussion

The morphology of SZRo, SZA, and SZRu is elliptical and has an eccentric hilum with lamella. SZRo and SZA show similar granule sizes. Meanwhile, SZRu has a smaller length and width. The size variation may be caused by its variety difference. The morphology of all starches is

similar to those reported by previous studies i.e. oval, cordate, polyhedral and angular with a diameter of 4-40 µm (Braga *et al.*, 2006; Adebowale *et al.*, 2014; Gavrilova *et al.*, 2022).

SZA shows higher swelling power than other starch indicating its stronger bonding force in starch granules (Adebowale *et al.*, 2014). The swelling profile exhibited a trend of increase with the increase in temperature for all starches. The swelling power shows its digestibility, ease of use in solution formulation, and ability to be used as a disintegrant (Nuwamanya *et al.*, 2010; Chowdary & Enturi, 2011;). The starch of all varieties exhibited an increase in swelling power at 80 °C and there was a rapid increase at 90 °C. Thereby, it is unsuitable for application in pharmaceutical formulations that desires high swelling at low temperatures, such as most tablets. The result also correlated with the water absorption characteristic of the starches at high temperatures. Water absorption differences may be caused by different polymeric chains' interaction with the amorphous and crystalline granules (Zhang *et al.*, 2005). Material with a smaller amorphous proportion has a lower water-holding capacity and absorbs less water (Boyer *et al.*, 1976). In the solubility profile, SZA exhibited the highest value, indicating its use in a solution dosage form. However, all starches showed an increase in solubility significantly at 70 °C. The result showed that an increase in temperature caused an increase in solubility. During the heating process of starch-water suspension, the crystalline structure is interrupted because the hydrogen bonds are damaged. The water molecules are linked by hydrogen bonding to hydroxyl groups of amylose or amylopectin and thereby causing an increase in swelling power and solubility (Hoover, 2001). The swelling and solubility profile of the study was similar to that of ginger from Abuja, Nigeria (Kolawole *et al.*, 2013).

The pH of SZRo, SZA, and SZRu was 6.44±0.02, 6.65±0.03, and 6.55±0.01, respectively. Starch with a pH range of 3.00-9.00 was usually used as excipients in the pharmaceutical, cosmetics, and food industries (Omojola *et al.*, 2010). A previous study showed that the pH of ginger was 6.54±0.03 (Kolawole *et al.*, 2013). The study showed that all starches resulted in similar browning and charring temperatures. This allowed the starches to be heated at high temperatures during the manufacturing process without changing colour or charring.

Foam capacity measurement indicated that SZA and SZRu had similar fat content, but SZRo had no fat content. Meanwhile, the high emulsion capacity of SZA was correlated with its higher ability as an emulsifier (Afolayan *et al.*, 2014). Another study showed that ginger starch has a foam capacity of 5±0.10% and an emulsion capacity of 5.7±0.1 (Kolawole *et al.*, 2013). The different results may be caused by different plant origins and growing locations. Gelatinisation occurs when starch is heated in excess water. Thereby, water diffuses into starch granules to swell because of amorphous phase hydration (Jiménez *et al.*, 2012). This process is characterised by transition temperatures and gelatinisation enthalpies in the paste for each species. The high temperature corresponds to increased stability, the resistance of the granule structure to gelatinisation, and a high degree of crystallinity (Tester *et al.*, 2004). The previous study showed a gelatinisation temperature of 78 ± 0.0 °C for ginger starch (Afolayan *et al.*, 2014).

The density determination of the study showed that starch from all varieties had similar values. It implied that all starches had similar maximum weight reduction during the packing process and compatibility to form a tablet (Hasan *et al.*, 2015). Carr's index and the Hausner ratio represent the flow ability and compressibility of starch. All starches showed that the value of Carr's index was more than 15%, and the Hausner ratio was more than 1.25. It suggested that all starches had fair flow properties and compressibility (Hasan *et al.*, 2015; Patel *et al.*, 2012). Most starch particles from all varieties had a size of less than 179 µm. The smaller the particle size, the larger the surface area for water absorption. It caused the starch tent to quickly absorb the surrounding moisture and increase its moisture content. The moisture sorption capacity represents material moisture sensitivity and relative physical stability of the tablets stored under humid conditions (Ohwoavworhua & Adelakun, 2010).

Conclusion

Ginger starch of three varieties has similar morphology and physicochemical properties. The study shows that the starches are potentially developed as an excipient in an optimum formula to imply the requirements of pharmaceutical dosage forms.

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