Isolation and Evaluation of Physicochemical of Pregelatinized Starch as Pharmaceutical Excipient: Albizia Saman

Elasari Dwi Pratiwi(✉), Fransisca Dita Mayangsari, Diah Indah Kumala Sari, Devi Ristian Octavia, and Irma Susanti

Faculty of Health Sciences, Universitas Muhammadiyah Lamongan, Lamongan, East Java, Indonesia
edpratiwi8@gmail.com

Abstract. Albizia saman seeds contain 24.2% starch which can be used as an excipient in pharmaceutical preparations. However, natural starch has unfavorable characteristics, so it needs to be modified. Pre-gelatinization is one of the physical modification methods which can improve the characteristics of natural starch. This study aimed to evaluate the physical characteristics of pregelatinized starch of Albizia saman seeds as an excipient in pharmaceutical preparations. The current research consisted of two stages. The first stage included carrying out the phytochemical screening process for Albizia saman seeds, isolating starch from Albizia saman seeds, and determining the starch content of Albizia saman seeds using the Luff Schoorl method. In the second stage, the starch from Albizia saman was physically modified (pregelatinized) using a 1:1 ratio and then evaluated. The results of the phytochemical screening confirmed that the Albizia saman seeds were positive for containing secondary metabolites of alkaloids, flavonoids, and saponins. The determination of the starch content of the Albizia saman using the Luff Schoorl method was 95%. Furthermore, pregelatinized starch of Albizia saman seeds had good characteristics as evidenced by the results of the particle size distribution (251–425 μm), moisture content (0.064%), flow rate (8.45 g/s), angle of repose (0.6°), ash content (1.89%), and compressibility index (12.4%). To sum up, the pregelatinized starch of Albizia saman seeds have good characteristics in accordance with the requirements of the Indonesian Pharmacopoeia, so it can be developed as an excipient raw material in pharmaceutical preparations.

Keywords: Evaluation · isolation · pre-gelatinization · starch · Albizia saman

1 Introduction

Albizia saman has plenty of benefits and is easily found in Lamongan Regency. However, people are not familiar with its usage because they lack knowledge about the contents. Albizia saman produces abundant seeds which are round in shape and reddish-brown [1–3]. In her research, Anil mentioned that Albizia saman seeds contain 64.6 mg carbohydrates and 1.26 mg protein, which makes it good and has the potential to be an excipient in pharmaceutical preparations [4].

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Pharmaceutical excipients such as fillers, disintegrants, and binders are excipients derived from starch [5]. Starch is a natural polymer composed of a branched structure called amylopectin and a straight structure called amylose [6, 7]. Utilization of natural starch is still very limited because it has poor flow and compressibility properties, which affect the uniformity of the tablet content and result in the therapeutic effect. Therefore, a modification process is needed for starch, such as pre-gelatinization [8, 9].

Pre-gelatinization is a physical modification of starch. It is obtained by pregelatinizing and drying the starch suspension. Among the starch modification methods, pregelatinization is more commonly used in the pharmaceutical industry as an excipient in solid dosage forms[10]. Another advantage of starch modification is easily soluble in water, has good viscosity, flowability, and compressibility, and can expand well. With various advantages resulting from modification of pre-gelatinization, it can provide or guarantee a uniform dose and has very good disintegration or dissolution properties [11].

As an initial step in the development of starch as an excipient for pharmaceutical preparations, a process of isolation and evaluation of the results of pregelatinized starch from Albizia saman seeds was carried out. This study aimed to evaluate the physicochemical characteristics of pregelatinized starch of Albizia saman seeds. It is hoped that the contribution of the research results will be able to improve and answer new challenges in the use of Albizia saman seeds starch as an excipient for pharmaceutical preparations.

2 Materials and Methods

2.1 Materials

The tools used in this study were an oven (Memmert UN30), sieve shaker (MBT Sieve Shaker AG-515), furnace (B-ONE Muffle Furnance 1200 °C Cap 7.2 L), analytical scale (Durascale DAB-E223), water bath (Faithful DK-2000-IIIL), desiccator, granule tester, and glassware. Meanwhile, the ingredients were Albizia saman seeds, 2N hydrochloric acid (CV Jayarindo Pratama Laboratory), Mayer reagent (Nitra Kimia), Bouchardat reagent, methanol, ether (EMSURE®), ethyl acetate (CV Jayarindo Pratama Laboratory), ethanol 95% (CV Jayarindo Pratama Laboratory), concentrated hydrochloric acid (CV Jayarindo Pratama Laboratory), 1% FeCl3 (Medical and Laboratory Supplier), Aquades (Bratachem), sodium metabisulfite (Bratachem), Luff Schoorl Solution.

2.2 Methods

2.2.1 Identification of Secondary Metabolic Compounds

The procedures for the identification of secondary metabolites referred to the 1989 MMI guidelines and various works of literature. Identification of secondary metabolites of Albizia saman seeds included the identification of alkaloids, flavonoids, saponins, and phenols [12–14].

2.2.2 Isolation of Albizia Saman Seeds

Isolation of starch was carried out by soaking the materials. Albizia saman seeds were soaked in distilled water (Aquades) at a ratio of 1:4 for 24 h. Then, it was strained to produce a starch precipitate. It was soaked again using sodium metabisulfite (Na2SO4) for 12 h. After that, it was dried in an oven at 40 °C to produce good-quality starch [15, 16].

2.2.3 Determination of Albizia Saman Seeds Starch

100 mg of Albizia saman seeds starch was added with 50 mL of distilled water and 5 mL of 25% HCl. The suspension was heated at 100 °C for 3 h and neutralized using 25% NaOH to pH 7. The suspension was added with distilled water up to 100 ml. Then, 25 mL of the sample solution was pipetted, and 25 mL of Luff Schoorl's solution was added to an Erlenmeyer. The solution was distilled and brought to a boil. Moreover, the solution was cooled, and added by 15 mL of 20% Cl and 10 mL of 25% H2SO4. The sample solution was dripped with 2 mL of starch indicator and titrated using 0.1 N Na2S2O3 solution [17].

2.2.4 Albizia Saman Seeds Pre-gelatinization

Pregelatinized starch was made with a ratio of 1:1 between starch and water, and it was heated in a water bath at 55 °C for 10 min while continuously stirred. The suspension formed was dried in an oven at 60 °C for 24 h. After the starch dried, it was sieved with a mesh sieve No. 20 to produce pregelatinized starch [18, 19].

2.2.5 Evaluation of Pregelatinized Starch of Albizia Saman Seeds

The evaluation of pregelatinized starch of Albizia saman seeds included organoleptic, particle size distribution, moisture content, flow rate, angle of repose, ash content, and compressibility index [12].

a. Organoleptic
   Starch was observed visually by looking at its shape, color, and smell.
b. Solubility
   100 mg of starch was moistened with 96% ethanol, added with 25 ml of distilled water, stirred for 30 min, and then filtered. The filtrate obtained was then oven-dried at 105 °C, and the residue obtained was weighed to determine the amount dissolved.
c. pH
   Starch was dissolved in 100 ml of distilled water. Then, the pH was measured using a pH meter.
d. Moisture Content
   The weighing bottle was dried at 105 °C for 30 min and cooled in a desiccator for 15 min, and then weighed (W2). One gram of pregelatinized starch powder was put into a weighing bottle and weighed (W). Then, the powder was dried at 105 °C for 60 min, cooled in a desiccator for 15 min, and weighed again (W1). The moisture content should not be more than 15%.

e. Ash Content

A total of 1 g of starch was placed in a crucible cup. Next, it was put in a furnace at 600 °C for 3 h, cooled in a desiccator, and then weighed.

f. Compressibility Index

A total of 25 g of granules were put into a 100 ml measuring cup, and then the volume was measured (bulk volume). The measuring cup containing the sample was then tapped 300 times to obtain a compressed volume. The final granule volume was measured after setting, and then it was weighed to determine the weight (b/v). The compressibility index of less than 18% provided good flow properties.

g. Flow Rate

A total of 100 g of starch were weighed. Starch was poured through the rim of the funnel slowly into a funnel with a closed bottom. The bottom of the funnel was slowly opened, and the starch was allowed to drain out. The time required (seconds) for all the starch to pass through the funnel was recorded using a stopwatch. The ideal flow rate criteria for powders were more than 10 g/s.

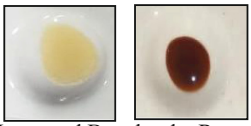
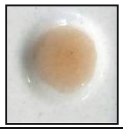
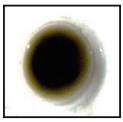
3 Result and Discussion

Albizia saman seeds were selected by considering the age and color to produce good quality starch. Albizia saman seeds powder was made by grinding (blender) and sifting it (Fig. 1.). Furthermore, the powder was subjected to a phytochemical screening process, including tests for alkaloids, flavonoids, saponins, and phenols. The results of the phytochemical screening confirmed that the Albizia saman seeds powder contained alkaloids, flavonoids, and saponins (Table 1).

The process of Albizia saman seeds powder isolation used an alkaline steeping method. Alka-line steeping was performed by soaking the powder in distilled water and sodium metabisulfite with a ratio of 4:1 to obtain starch of good quality (Fig. 2). One of the quality criteria used as an excipient for pharmaceutical preparations was high starch content. In this study, the determination of starch content used the Luff Schoorl method [13]. The Luff Schoorl method applies the iodometric principle, where free iodine is used as the basis for determining the concentration [7]. If a strong oxidizing agent such as H2SO4 is present in a neutral or slightly acidic solution, the addition of excess iodide ion will make the oxidizing agentless and will liberate I2 equivalent to the amount of

Fig. 1. Albizia saman Seeds

Table 1. Screening Phytochemical of Albizia saman Seeds

Screening Phytochemical	Results	
Alkaloids	 Mayer and Bouchardat Reagent	+
Flavonoids		+
Saponins		+
Phenols		-

oxidizing agent. The results of determining the starch content of Albizia saman seeds using the Luff Schoorl method were 95%.

The next stage was the pre-gelatinization of Albizia saman seeds starch using a ratio of Albizia saman seed starch and distilled water (1:1). The physical characteristics results of pregelatinized Albizia saman seeds starch were organoleptic, solubility, pH, water content, ash content, compressibility index, and flow properties. The results of the organoleptic test and solubility test (Table 2) showed that the characteristics of

Fig. 2. Pregelatinized Starch from Albizia saman Seeds

Table 2. Evaluation of Physicochemical Properties of Pregelatinized Starch of Albizia saman Seeds

No.	Evaluation	Albizia saman Seeds Powder	Pregelatinized Starch of Albizia saman Seeds	Compendial
1.	Organoleptic			
	Color	White-brown	White-brown	White-brown
	Texture	Powder	Powder	Powder
	Smell	Odorless	Odorless	Odorless
2.	Solubility	Practically insoluble in water	Practically insoluble in water	Practically insoluble in water

pregelatinized starch were in accordance with the starch standard, including a white to brownish color, powder form, odorless, and practically insoluble in water (Fig. 3).

The results of the pH test indicated that the pH of Albizia saman seeds starch and the pregelatinized starch of Albizia saman seeds met the requirements in the range of 5.0 to 8.0. Likewise, in testing the ash content and moisture content, the results were within the range required by the Indonesian Pharmacopoeia.

Flow rate testing is an important aspect in the manufacture of pharmaceutical preparations, especially in the manufacture of directly compressed tablets. Based on Fig. 4,

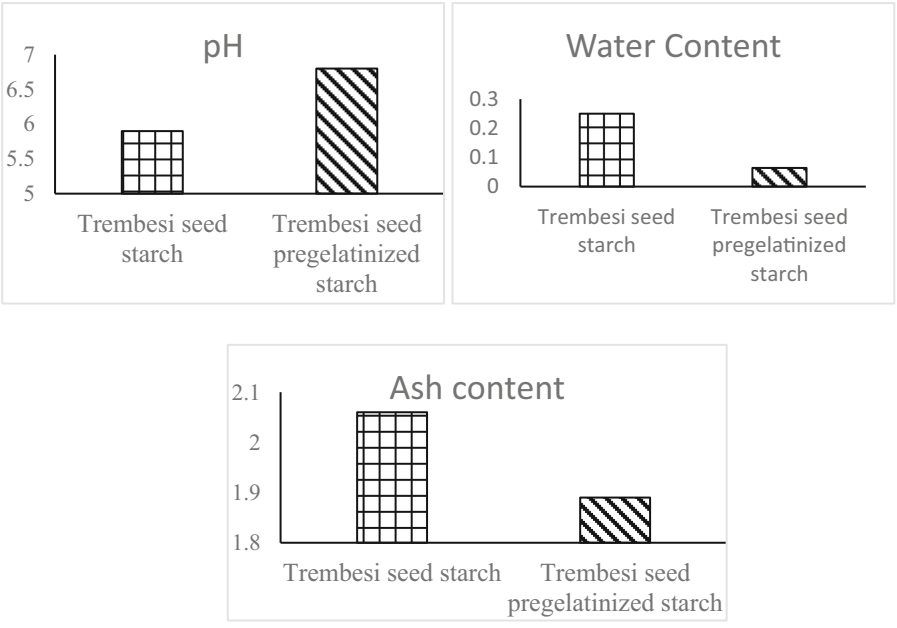


Fig. 3. The Characteristics of pH, Water Content, and Ash Content of Albizia saman Seeds Pregelatinized Starch

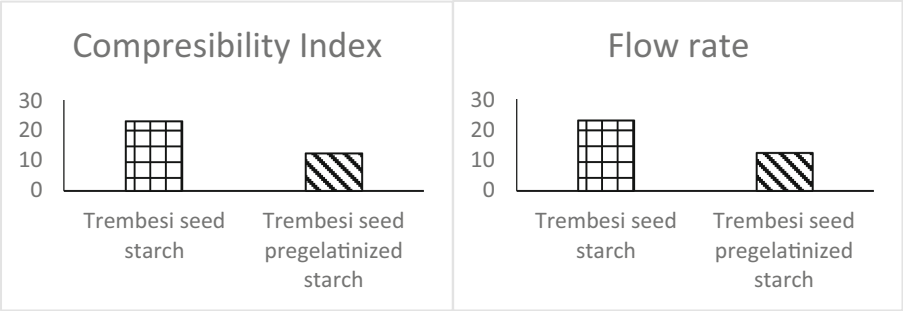


Fig. 4. The Characteristics of Compressibility Index Test and Flow Rate of Pregelatinized Starch of Albizia saman Seeds

pregelatinized starch had a good flow rate in accordance with the flow rate requirements of 10 g/s compared to Albizia saman seeds starch which had a poor flow rate. The size of the powder particles was a factor that affected the change in flow rate. The fine particle size limited the ability of the powder to flow freely due to the adhesion between the particles by gravity. Meanwhile, the compressibility test is a parameter that shows the ability of a material to make tablet preparations using the direct compression method. The compressibility index is closely related to the particle size and particle size distribution. The results indicated that both pregelatinized starch of Albizia saman seeds and Albizia saman seeds starch had a good compressibility index in accordance with the requirements of the Indonesian Pharmacopoeia, namely <18%. Thus, modification of pregelatinized starch from Albizia saman seeds can improve the physicochemical properties of starch, so it can be used and developed as an excipient in pharmaceutical preparations [14].

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