

Factors Affecting the Starch Extraction from Jackfruit Seed (*Artocarpus heterophyllus* L.)

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Abstracts: The study was conducted to investigate some factors affecting the starch extraction from jackfruit seeds (JS), including: Jackfruit seed drying temperature, extraction time and ratio of water to jackfruit seed flour (JSF). The JS were dried at various temperatures (50, 60 and 70°C) before grinding into flour. Starch was extracted in 4 levels of time (5, 6, 7 and 8 hours) interacted with 4 ratios of water to JSF (10, 15, 20 and 25 times, v/w). The efficiency of the extraction process was evaluated through the yield and the physicochemical properties of JSS, such as starch content, the color (L value and browning index - BI), water absorption index (WAI), water solubility index (WSI) and swelling power (SP); the scanning electron microscopy (SEM) and the average particle diameter (APD). The drying temperature of 60°C is considered suitable for obtaining good flour for performing starch extraction. Starch extraction from the JSF was carried out with the ratio of water to JSF of 15 times (v/w) for 6 hours at ambient temperature. The yield was 44.96±0.29%; the starch content was 92.06±0.48%. The JSS exhibited the values of WAI, WSI and SP in range of from other starches. The APD of JSS is 6.90–8.50 µm and is classified as small granules. All physicochemical properties as well as the APD of JSS were interrelated. These results showed that the extraction is high yield. The extracted JSS has high starch content and suitable physicochemical properties. When purified effectively, it has the potential to become food or effective complementary ingredients in the development of functional food products.

Keywords: Drying, Extraction, Jackfruit Seeds, Starch, Yield.

1. INTRODUCTION

Jackfruit (*Artocarpus heterophyllus* Lam) is a popular fruit tree widely grown in South and Southeast Asia such as Bangladesh, India, Myanmar, Nepal, Thailand, Vietnam, China, Philippines, Indonesia, Malaysia and Sri Lanka [1]. In Vietnam, jackfruit is distributed mainly in the Southeast region (Binh Phuoc and Binh Duong) and in the Mekong Delta (Tien Giang, Vinh Long, Long An, Hau Giang, Ben Tre). The growing area of mainly Thai jackfruit variety in recent years has increased to several tens of thousands of hectares. In particular, Hau Giang is one of the two provinces of the Mekong Delta that has the best quality source of Thai jackfruit and up to now Hau Giang has an area of 7,972 hectares of jackfruit with the yield of 79,830 tons/year.

Despite being a delicious fruit, jackfruit has not been exploited and used effectively due to the low percentage of fruit pulp that is only about 29% of the fruit weight [2]. Waste products are mainly seeds, skins, core and latex that generate many environmental pollution wastes from jackfruit processing factories. Among them, JS account for about 10-15% of the fruit weight [3], which shows their high carbohydrate content, especially starch, accounting for about 77.8% per dry weight [4]. In order to know whether starch can be used as a stabilizer, gelling agent or thickener in the food industry or in the cosmetic and pharmaceutical fields, it is necessary to consider the physicochemical properties of the starch, that are related to the composition and size of the starch granules [5]. The fresh JS cannot be stored for long time and they usually are discarded or steamed for immediate use [6]. To effectively exploit this source of waste, it is necessary to create suitable starch extraction procedure for using the starch source from them. Some studies from other countries have applied JSF as a supplement to other food products. However, there has not been any research on extracting starch from Thai jackfruit seeds in Hau Giang area to effectively use this local by-product source.

2. MATERIAL AND METHODS

2.1 Jackfruit Seeds and Flour Preparation

Jackfruit seeds (*Artocarpus heterophyllus* L.) were collected from mature Jackfruit fruits that were grown in Hau Giang province, Vietnam. The seeds were washed and peeled off manually. The clean seeds were dried (50, 60 and 70°C) to reduce the moisture content to about 55% to remove the seed coats easily. For the next step, the seeds were soaked in the solution of 5% NaOH for 4 minutes at room temperature to remove brown layers [7]. The washed seeds were sliced into 2-2.5 mm thickness slides and dried in an oven until their moisture content is about 10%. The seed slides were ground into flour by using a dry-grinder.

2.2 Starch Extraction

The extraction of JSS was carried out according to the method of Noor et al. [7] with a little modification. A sample of JSF (50 g) was mixed with distilled water (The ratio of water to flour were 10; 15; 20 and 25 v/w) and soaked (soaking time were 5; 6; 7 and 8 hours) at room temperature. The mixture was stirred carefully and was filtered after soaking. The remaining sediment was washed with distilled water for three times. The filtrates were combined and precipitated overnight at 4°C in refrigerator. The supernatant was discarded to obtain the crude starch that was cleaned with distilled water. This step was repeated three times and the starch cake was dried at 50°C in an oven until their moisture content is about 10-12%. The starch was fine ground by using a dry-grinder and sieved through 0.125 mm stainless sieve. The fine starches were packed in a PA bag and kept at 4°C until analysis.

2.3 Determination of the Yield

The yield was calculated according to equation (1) [8]:

$$\text{The yield (\%)} = \frac{\text{Extracted starch weight (db)} \times 100}{\text{Flour weight (db)}} \quad (1)$$

2.4 Determination of the physicochemical properties of JSS

The starch content

The starch content was determined by Lane and Eynon method.

The protein content

The protein content was determined by Kjeldahl method

The ash content

The ash content is determined according to TCVN 8124:2009

The crude fiber content

The crude fiber content is determined according to TCVN 4998:1989

Color measurement

The color attributes (Hunter L*, a*, and b* values) were measured by colorimeter (CR-20, Konica Minolta, Japan). Browning index (BI) was determined using the equation (2) [9]:

$$\text{BI} = [100(x-0.31)]/0.17 \quad (2); \text{ Where: } x = (a^* + 1.75L^*)/(5.645L^* + a^* - 3.012b^*)$$

Determination of WAI, WSI and SP of starch

Starch sample (2 g) was suspended in 20 ml of distilled water. The dispersion was stirred for 30 min and centrifuged at 4000 rpm for 30 min. The supernatants were poured into pre weighed petri dish and the residue was weighed after oven drying at 70°C until the moisture content is about 10%. WAI and WSI were calculated using following equations [7]:

$$\text{WAI (g/g)} = \text{weight of sediment} / \text{weight of dry solids}$$

$$\text{WSI (\%)} = \text{weight of dissolved solids in supernatant} \times 100 / \text{weight of dry solids}$$

The calculation of SP using following equation [10]:

$$\text{SP (g/g)} = \text{weight of sediment} / [\text{dry weight of sample} \times (1 - \text{WSI} / 100)].$$

Starch morphology evaluation and APD of starch determination

The starch granules morphology was quantified using a scanning electron microscopy (SEM) and the diameter of 50 granules were measured, the APD of starch was calculated.

2.5 Data analysis

Data were analysed for Analysis of Variance (ANOVA) to determine the significant differences in the means. Means were separated using LSD (Least Significant Difference) test ($p < 0.05$). Results were presented as mean values and standard deviations in Tables and graphs.

Multiple regressions were performed to determine the multi-variable regression equation between the response variable and the dependent variables. Multiple-Variable Analysis was performed to determine whether there is a correlation between each pair of variables. The Statgraphics Centurion 19 software was used for all methods of data analysis.

3. Results and Discussion

3.1 Effects of Seed Drying Temperature on JSS Properties

3.1.1. The Yield and the Starch Content

The influence of seeds drying temperature on the yield and starch content are shown in Table 1. The yield and starch content of JSS were in the range 30.14-44.89% and 86.54-91.94%, respectively. The result of yield was in agreement with Thanh et al. [11] (40.77%); While this value from other researches varied from between 18.9-32.11% [12; 5]. The starch content was slightly lower than the result of Wong et al. [12] (92.6%), but higher than that from the result of Noor et al. [7] (83.83-86.71%). The difference in the yield and starch content from previous studies was due to the application of different starch extraction methods.

Table 1. Effects of seed drying temperature on the yield and the starch content of extracted JSS

| Drying temperature (°C) | Yield (%) | Starch content (%) | Drying time (Hours) |
|-------------------------|-------------------------|-------------------------|---------------------|
| 50 | 44.41±0.55 ^a | 91.03±0.96 ^a | 25 |
| 60 | 44.89±0.28 ^a | 91.94±0.38 ^a | 20 |
| 70 | 30.14±0.27 ^b | 86.54±0.45 ^b | 10 |

(Mean±SD, n=3, Different letters in each column indicate a significant difference at 5% level of significance)

There was no change in the yield and starch content of JSS when the seeds were dried at 50 and 60°C, but all these values were significantly reduced when jackfruit seeds were dried at 70°C. The cassava starch yield was not significantly affected by the drying temperature in range of 40-60°C [13]. Ren et al. [14] reported that corn starch yield decreased with increasing drying temperature from 60 to 100°C. The decreases in starch yield as the drying

temperature increases resulted from the partial gelatinization of starch and the protein denaturation during the hot air drying [14].

3.1.2. The Color

The drying temperature had no significant effect on the L value of starch (Table 2), there was a tendency to increase b* and to change in a* value, the BI of starch also varied. There was no different in BI of JSS when drying temperature in range of 50-60°C, but BI increased significantly when drying seed at 70°C. The trend of changing the starch color was similar to the result of Ziegler et al. [15] on corn starch when the materials were dried at 40-70°C.

Table 2. Effects of seed drying temperature on the color of extracted JSS

| Drying temperature (°C) | L* | a* | b* | BI |
|-------------------------|-------------------------|-------------------------|------------------------|-------------------------|
| 50 | 95.17±0.29 ^a | 0.42±0.23 ^a | 7.44±0.52 ^c | 8.31±0.52 ^b |
| 60 | 95.47±0.21 ^a | -0.52±0.05 ^b | 8.31±0.26 ^b | 8.48±0.29 ^b |
| 70 | 95.27±0.28 ^a | 0.22±0.19 ^a | 9.62±0.21 ^a | 10.55±0.30 ^a |

(Mean±SD, n=3, Different letters in each column indicate a significant difference at 5% level of significance)

3.1.3. Functional Properties

Water absorption index (WAI), water solubility index (WSI), and swelling power (SP) of JSS are shown in Table 3. WAI of JSS was in range of 2.26 to 2.82 (g/g) and increased with the increase of drying temperature. The tendency of WAI change is also confirmed by Ahmed et al. [16]. The WAI change may be due to the difference in the degree of participation of hydroxyl groups to form hydrogen and covalent bonds between starch chains [16].

The values of WSI were from 11.67 to 12.38% and was not significantly affected by drying temperature in range of 50-70°C. The WSI values were similar to the results of Noor et al. [7]. Ahmed et al. [16] found a tendency to increase WSI with the increasing in drying temperature.

The SP of JSS was in the range of 5.35-6.09 (g/g) and increased with the increase of drying temperature. The SP values in this study were lower than that of Noor et al. [7] (8.79-9.87 g/g) but higher than that of Thanh et al. [11] (2-3 g/g). The increase of SP with grain drying temperature was also confirmed from some previous research results [16; 17]. According to Ahmed et al. [16], the increase in SP at high drying temperature can be explained by starch gelatinization at a certain temperature leading to disrupt the molecular organization inside the grain and the interaction between starch and water increases, resulting in significantly increased swelling power.

Table 3. Effects of seed drying temperature on the functional properties of JSS

| Drying temperature (°C) | WAI (g/g) | WSI (%) | SP (g/g) |
|-------------------------|------------------------|-------------------------|------------------------|
| 50 | 2.26±0.12 ^b | 11.88±0.75 ^a | 5.35±0.27 ^b |
| 60 | 2.75±0.11 ^a | 11.67±0.57 ^a | 5.44±0.25 ^b |
| 70 | 2.82±0.09 ^a | 12.38±0.57 ^a | 6.09±0.15 ^a |

(Mean±SD, n=3, Different letters in each column indicate a significant difference at 5% level of significance)

3.2 Effects of Extraction Time and The Ratio of Water and Flour on Jss Properties

3.2.1. The Yield and Starch Content

The extraction time and water-to-flour ratio have effected significantly on the yield, and starch content of extracted starch (Fig 1.a; b). The yield and starch content tended to be higher when extracting for 6-7 hours and there was a significant difference from that of starch extracted for 5 or 8 hours. Noor et al. [7] announced that the most suitable time for extracting JSS in water is 6 hours. The prolonged extracting time allowed more starch components to escape from the cells [18]. However, too long extracting time could lead to lower starch yield, mainly due to the increase in starch hydration and swelling which reduces the filtrate amount making it difficult to facilitate

sedimentation [19]. They were the reasons why the effective starch extraction time was 6-7 hours.

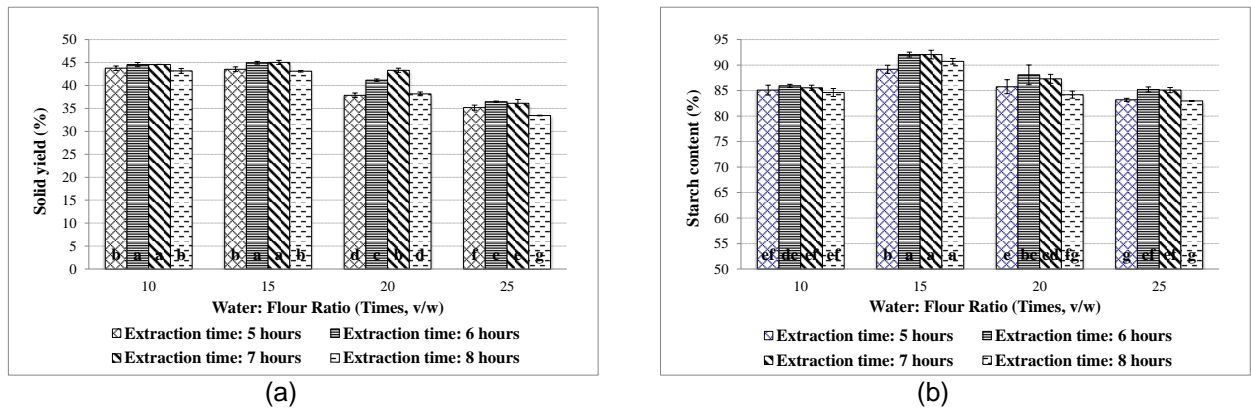
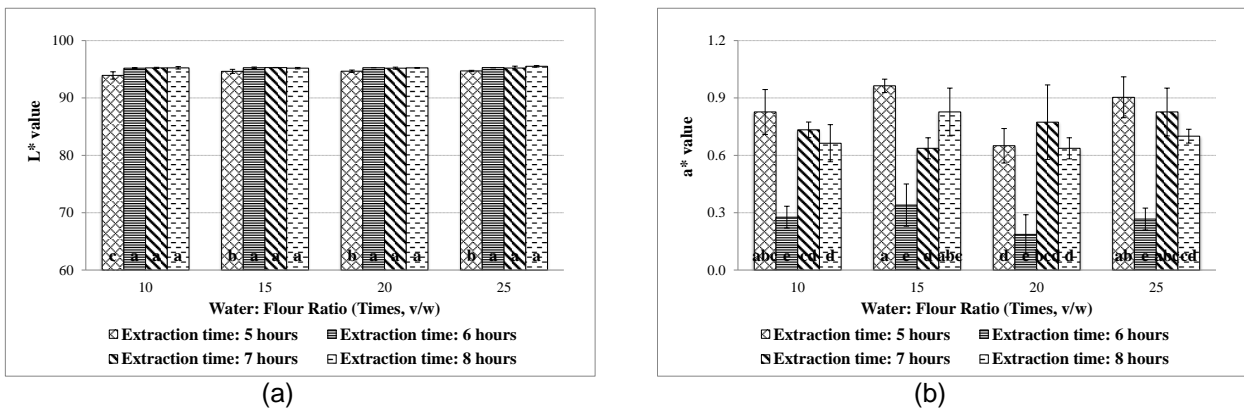


Fig 1. Effect of extraction time and the water and flour ratio on: (a) the yield; (b) starch content

The highest yield obtained when the water to flour ratio was in range of 10-15 times that was significantly different from the others. The highest starch content corresponded to water to flour ratio of 15 times (Fig 1). Lower water to flour ratio (10 times) or higher ratio (20-25 times) resulted in a significant reduction in starch content. Thank et al. [11] showed that starch production increased with the ratio of water to flour used, but Liu et al. [20] gave the opposite result. Noor et al. [7] extracted JSS with a water to flour ratio of 20 times.

3.2.2. The Color

As the ratio of water has increased, the whiteness (L^* value) of starch has tended to increase. However, there was no significant difference in L^* as the water to flour ratio from 15 to 25 times.



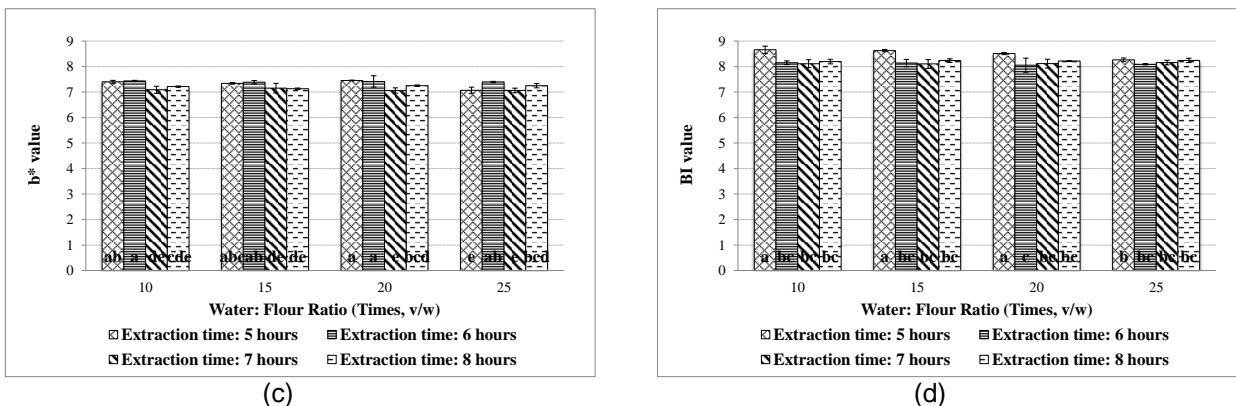


Fig 2. Effect of extraction time and the ratio of water and flour on the starch color: (a) L* value; (b) a* value; (c) b* value; (d) BI value

Extraction time also had a similar effect on starch whiteness. Extraction time from 6 to 8 hours starch whiteness was not statistically significant (Fig. 2) but was significantly higher than from starch extracted for 5 hours. The L* values of starch were high and in the range of 93.92-95.52. Thanh et al. [11] announced the L* value of JSS as 89.76. The prolonged time and lots of water in contact with the flour makes it easier to dissolve and remove color compounds. The values of a* and b* were influenced by the time and water to flour ratio during starch extraction (Fig. 2). However, the change in BI is completely consistent with the L* value. The whiter the sample (higher L* value), the lower BI value and vice versa.

3.2.3. Functional Properties

The variation of WAI, WSI and SP according to extraction time and water to powder ratio have followed the quadratic multivariable regression equation (Fig. 3), specifically as follows:

$$WAI = - 0.0675X^2 - 0.0022Y^2 + 0.9191X + 0.07438Y \quad (R^2 = 0.948)$$

$$WSI = 0.016125Y^2 \quad (R^2 = 0.940)$$

$$SP = 1.8966 - 0.06875X^2 + 1.15835X + 0.06213Y - 0.00872XY \quad (R^2 = 0.932)$$

Where X: Extraction time (Hours) and Y: Water to flour ratio (Times, v/w)

The WAI of JSS was in range of 2.64-3.19 g/g. This value was lower than that found by Noor et al. [7] (around 8.3 g/g), but higher than found by Karadbhajne and Yatin [21] (1.15 g/g). The WAI of starch from 3 varieties of Thai rice has shown similar values [22]. The WAI of JSS has tended to increase with the increasing in water to flour ratio and prolonged extraction time (Fig. 3). According to Nuwamanya et al. [23], protein and carbohydrate content in starch would influence water absorption capacity because of the presence of hydrophilic regions in both components. In addition, the variation in WAI of starch could be due to the difference in the degree of the engagement of hydroxyl groups to form hydrogen and covalent bonds between starch chains [24].

WSI refers to the amount of dissolved solids in a sample and it reflects the amount of polysaccharides released from starch granules through a measurement of the conversion rate of starch during processing [25]. In this study, the WSI of the extracted starch ranged from 9.09% to 13.51%. Similar observation has been reported by Noor et al. [7]. The WSI of starch from a variety of average amylose content Japanese rice was 9.5% [26]. WSI tended to decrease with increasing extraction time and water to flour ratio. WSI as being dependent on the morphological structure of starch granules such as size, shape and texture; these determine the extent which free polysaccharides are released in the presence of water [27; 28].

JSS exhibited SP in the range of 6.12-6.70 (%). It was slightly lower compared to finding from Noor et al. [7] (8.79-9.87 g/g), while another study conducted by Karadbhajne and Yatin [21] showed a SP reading as 0.37 (g/g). SP of JSS was slightly lower than that of rice starch (7.0-16.2 g/g) from the result of Kong et al. [26]. SP tended to increase with the increasing in extraction time and water to flour ratio. SP is a measure of hydration capacity through the measurement of the weight of the swollen starch granules and their occluded water [29]. It is involved in non-covalent bonds between starch molecules and depends on many factors such as amylose-amylopectin ratio, chain length and molecular weight distribution, degree of branching of starch chains [30].

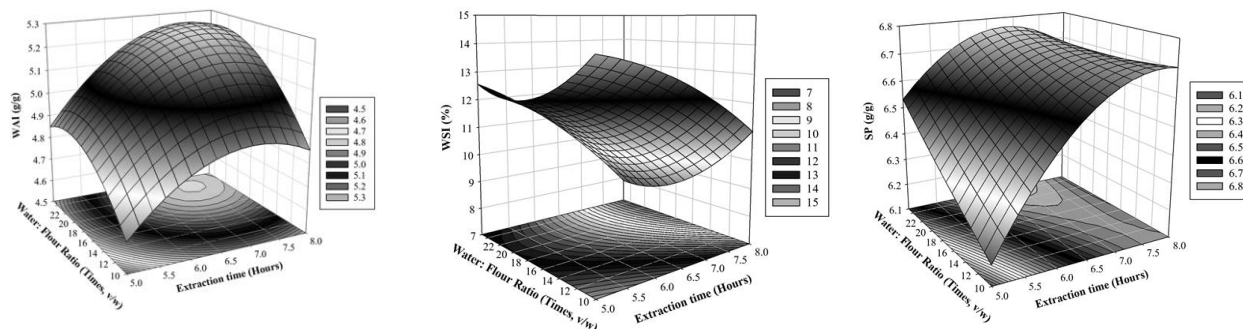


Fig 3. Effect of extraction time and the ratio of water and flour on: (a) WAI; (b) WSI; (c) SP

3.2.4. SEM and APD

The scanning electron microscopy analysis using 1000x magnification showed granules with round and semi-oval or some bell shapes (Fig. 4). The size distribution of granules was in range of 5.5-11 μm and it showed the similar results with observations of Bobbio et al. [31] who reported that JSS has round or bell shapes, ranging in size from 7 to 11 μm as well as the result of Madruga et al. [32] that APD were 6–13 μm .

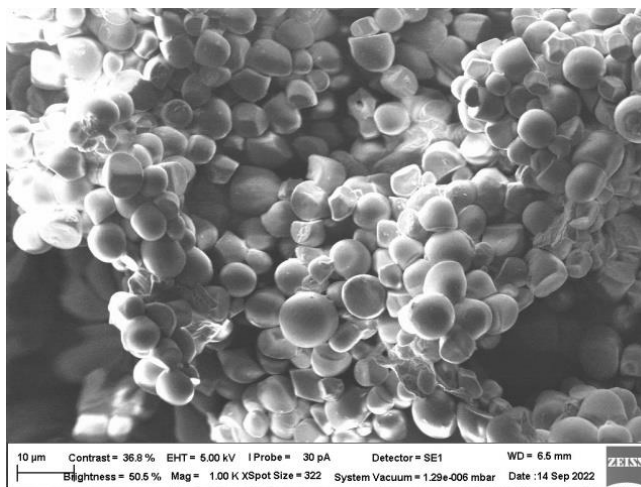


Fig 4. Scanning electron micrographs of JSS extracted with the water to flour ratio of 15 times (v/w) and the extraction time of 6 hours

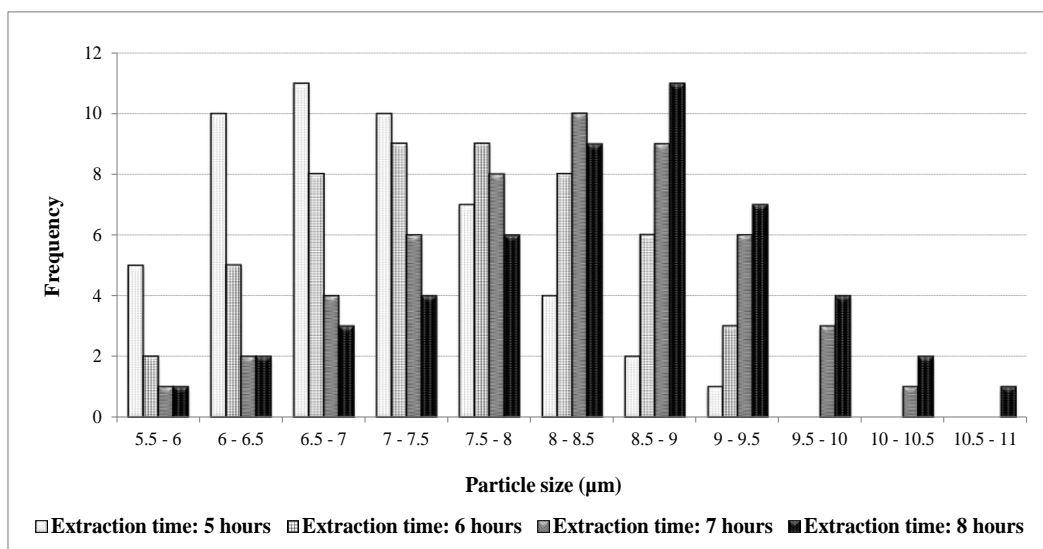


Fig 5. Effect of extraction time on size distribution of starch

The prolonged extraction time has increased in APD (Figure 5). The APD of JSS was in range of 6.9-8.5 µm and obeyed the quadratic regression equation with extraction time and water-to-powder ratio with a R² of 0.994 (Fig. 6). This range of APD was very consistent with the results of Lubis et al. [33] and of Tongdang [34]. Based on the standard of size classification, JSS is classified as small granules that has a APD of 5-10 µm [35]. The APD and shape observed for starch in this study are typical of jackfruit seeds which have been growing around the world.

The quadratic regression equation of ADP according to extraction time and water to flour ratio is as following:

$$APD = 1.85855 - 0.05875X^2 + 1.23855X \quad (R^2 = 0.994); \text{ Where X: Extraction time (Hours)}$$

The above equation showed that ADP did not depend on the water to flour ratio

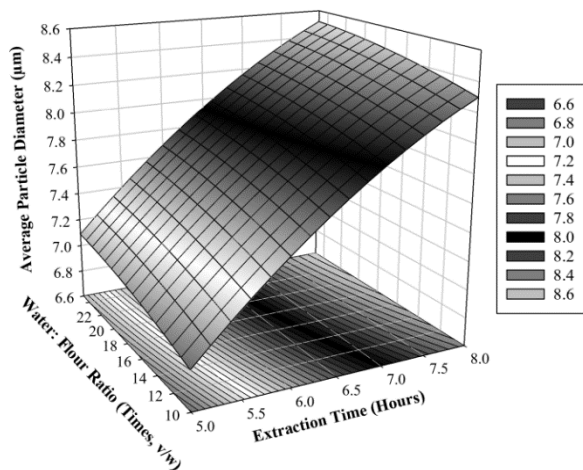


Fig 6. Relationship between APD with extraction time and the water to flour ratio

3.2.5. Multiple-Variable Analysis

Most of physicochemical properties of JSS were correlated with extraction time, there were the yield and starch content was correlated with water to flour ratio (Table 4). All physicochemical properties of JSS were correlated APD, where the relationship between APD to WSI and BI were negative. The WAI, WSI and SP were all correlated,

L* value and BI were all correlated. Wickramasinghe and Noda [36] analysed and confirmed the correlation between the physicochemical properties of rice starch.

Table 4. Pearson correlation coefficient and p-value of the correlation between two variables

| | Yield | Starch content | WAI | SP | WSI | L* | BI | APD |
|----------------------|--------------|----------------|-----------|-------------|--------------|-------------|--------------|--------------|
| Extraction time | -0.039 | -0.032 | 0.615 (*) | 0.734 (**) | -0.795 (***) | 0.712 (**) | -0.530 (*) | 0.980 (***) |
| Water to flour ratio | -0.873 (***) | -0.324 | 0.589 (*) | 0.348 | -0.474 | 0.242 | -0.199 | 0.145 |
| Yield | | 0.664 (**) | -0.327 | -0.2101 | 0.398 | -0.114 | 0.012 | -0.122 |
| WAI | | | | 0.756 (***) | -0.845 (***) | 0.738 (**) | -0.653 (**) | 0.728 (**) |
| SP | | | | | -0.656 (**) | 0.911 (***) | -0.909 (***) | 0.804 (***) |
| WSI | | | | | | -0.614 (*) | | -0.828 (***) |
| L* | | | | | | | -0.859 (***) | 0.766 (***) |
| BI | | | | | | | | -0.607 (*) |

(*): *p*-value < 0.05; (**): *p*-value < 0.01 and (***): *p*-value < 0.001

3.3 Comparison the Chemical Composition of JSF and JSS

The main chemical compositions of JSF and JSS were presented in Table 5.

Table 5. The compositions of JSF and JSS

| | Water content (%) | Starch (% DW) | Protein (% DW) | Crude fiber (% DW) | Ash (% DW) |
|-----|-------------------|---------------|----------------|--------------------|------------|
| JSF | 6.89±0.65 | 75.87±1.81 | 12.11±0.29 | 4.36±0.47 | 3.13±0.05 |
| JSS | 6.43±0.10 | 92.06±0.48 | 5.12±0.34 | 1.62±0.21 | 0.14±0.07 |

Table 5 showed that the starch extraction process maintained small amounts of protein, fiber and ash, so a purification method (using protease and cellulase enzymes) is needed to obtain high purity jackfruit starch.

CONCLUSIONS

The starch extraction of JSF showed the high yield and starch content. Starch granules were round, some semi-oval or bell-shaped and the particle size was classified as small, which was a common feature of starch granule structure from jackfruit seeds in Asia. WAI, WSI and SP of JSS are not much different from rice starch, so if further purified, JSS is used in the formulation of food products as well as a supplementary ingredient in food processing, cosmetics and medicine.

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