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Preparation and Characterization of Red Dragon Fruit (*Hylocereus polyrhizus*) Starch as an Excipient in Solid Dosage Form

Melisa Nia Alfiona, Intan Martha Cahyani*, Wulan Kartika Sari

College of Pharmaceutical Sciences, Yayasan Pharmasi Semarang, Jl. Letjend Sarwo Edie Wibowo Plamongansari-Pucanggading, Semarang 50193, Jawa Tengah, Indonesia

*Corresponding author: Intan Martha Cahyani | Email: intanmartha20@gmail.com

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Abstract: Starch is often used as a filler, crusher, and binder in solid preparations. One source of starch can be found in red dragon fruit stems. When making starch, browning often occurs which causes the flour to become brownish, which can reduce public acceptance. Efforts are made to prevent browning in the starch making process by using sodium bisulfite solution. This study aims to determine the effect of different sodium bisulfite soaking times on the characteristics of red dragon fruit stem starch *(Hylocereus polyrhizus)* to be used as a solid preparation additive and to determine the length of sodium bisulfite soaking time that can produce red dragon fruit stem starch *(Hylocereus polyrhizus)* characteristics that meet the standards of solid preparation additives. The results of the analysis showed significant differences in yield, moisture content, flow velocity, angle of repose, pH, solubility, expandability, compressibility index, bulk density, tap density, true density, and Hausner index. FTIR analysis showed that red dragon fruit stem starch contains starch functional groups. Based on the results of the red dragon fruit stem characterization test, the best treatment was obtained, namely 1 hour soaking, producing starch with physical characteristics suitable for solid preparation additives.

Keywords: red dragon fruit stem; starch; sodium bisulfite; characterization

1. INTRODUCTION

Red dragon fruit plants after harvest will be pruned to quickly stimulate the growth of new flowers. The remaining red dragon fruit stem will only be discarded because it is considered as waste and its utilization is still very minimal, so it is very necessary to handle it so that it does not become a problem if it is not handled properly, but the high water content of the red dragon fruit stem makes its shelf life very short, by making dragon fruit powder is expected to extend its shelf life. Powder from red dragon fruit stems that have been peeled off the skin contains starch, making it suitable for use as an additive to solid preparations [1].

Starch has many benefits and has long been used as a food ingredient or additive or excipient in solid preparations. The use of starch in the pharmaceutical field, especially in tablet preparation formulas, is used as a filler, crusher, or as a binder [2].

Processing of red dragon fruit stems into starch flour is done through several processes such as washing, removal of unwanted parts, size reduction, drying, crushing and sieving. In the flouring process, browning often occurs which causes the flour to become brownish so that it can reduce public acceptability [3]. Many efforts are made to prevent browning in these foodstuffs by using sodium bisulfite and bleaching solutions.

A study needs to be conducted to determine the effect of sodium bisulfite soaking time on the characterization of starch from red dragon fruit stems *(Hylocereus polyrhizus)* as an additive to solid preparations.

2. MATERIALS AND METHODS

2.1. Materials

The materials used in making starch and characterization testing of red dragon fruit stem starch are red dragon fruit stem *(Hylocereus polyrhizus)* from Gembong-Pati, distilled water, iodine pro analisis (Merch), $Na_2S_2O_3$ Pro Analisis (Smartlab), potato starch, cassava starch, corn starch, kaffein (Sigma Aldrich) , diclofenac sodium, ascorbic acid (Merch), paraffin liquid (ROFA).

2.2. Preparation of Red Dragon Fruit Stem Starch

The red dragon fruit stems obtained were then washed and peeled off the outer skin, then sliced thinly with a thickness of about ± 0.2 mm. The cleaned red dragon fruit stems were then isolated by soaking in sodium bisulfite ($NaHSO_3$) solution with a temperature of 40°C only at the beginning of soaking (temperature is not maintained) with a concentration of 500 ppm, the weight ratio of red dragon fruit stems: solution is 1:2 with variations in soaking time of 1 hour, 2 hours, and 3 hours. Red dragon fruit stems are blended until they become a slurry, then stirred and kneaded with the aim of accelerating the release of starch from the protein or gum that covers it, then filtered with a filter cloth gradually. Settled for 24 hours until the starch separated from the soaking water. The precipitated starch was then washed with water 2-3 times until white (brownish white) starch was produced. The red dragon fruit stem starch sediment obtained was then dried at 60° C until a certain moisture content (⩽ 15%). The dried red dragon fruit stem starch was pulverized with a grinder, and sieved with a 100 mesh sieve

2.3. Physical Characteristics Testing

2.3.1. Organoleptical

The plants used in this study have been confirmed by plant determination conducted at the Pharmaceutical Biology Laboratory of the College of Pharmacy, Yayasan Pharmasi Semarang. The results of plant determination showed that the plant was a red dragon fruit plant (*Hylocereus polyrhizus*). Then the red dragon fruit stem starch was tested to observe the shape, odor, color and taste [4].

2.3.2. Yield

Red dragon fruit stem starch with sodium bisulfite soaking at soaking times of 1 hour, 2 hours, and 3 hours was weighed and the yield was calculated.

2.3.3. Moisture Content

Red dragon fruit stem starch was weighed as much as 1 gram and put into the moisture analyzers (Ohaus), the moisture analyzers was measured at 110° C.

2.3.4. Identification

Red dragon fruit stem starch solution as much as 5 mL is put into a test tube and dripped with iodine solution as much as 5 drops, observe the color changes that occur [5][6].

2.3.5. Microscopy

Red dragon fruit stem starch is placed on a glass object, then covered with a cover glass, and observed the shape of the hilum and lamella of dragon fruit stem starch under a microscope at 1000x and 400x magnification.

2.3.6. pH Examination

Red dragon fruit stem starch was weighed as much as 1 gram and suspended with distilled water as much as 10 ml, pH was measured using a pH meter (WalkLAB) [7].

2.3.7. Flow Speed and Angle of Repose

Starch powder was weighed as much as 25 grams, put into a funnel whose bottom was closed, then the bottom of the funnel was opened so that the granules could flow and then the time was recorded and the height and radius were measured [8][9].

2.3.8. Water Content

The crucible was heated in an oven at 105°C for 30 minutes, and then tared to constant weight. Red dragon fruit stem starch was weighed as much as 1 gram, put into the krus, dried in an oven at 105°C for 30 minutes with the lid open, then put in a desiccator for 15 minutes. Dried until the weight of the crucible was constant [10].

2.3.9. Ash Content

The red dragon fruit stem starch was weighed as much as 1 gram, put into a crucible, then incinerated with a muffle furnace at 600°C for 3 hours. Then the crucible was cooled in a desiccator for 10 minutes and weighed [11].

2.3.10. Starch Content Analysis

Red dragon fruit stem starch was weighed as much as 50 grams and dissolved in distilled water as much as 50 ml of distilled water. The sample was pipetted 6 ml, then put in a 10 ml volumetric flask and added 1% iodine as much as 0.5 ml and then ad 10 ml. The absorbance was measured with a UV-Vis spectrophotometer (Shimadzu 1240) double beam at a maximal wavelength of 400-800 nm is (461.20 nm) obtained from the standard amylum maydis, and measured the standard series with concentrations of 300, 400, 500, 600, 700 and 800 ppm, then obtained a linear equation that will be used to determine the starch content of red dragon fruit stems [12].

2.3.11. Swelling Power and Solubility

Red dragon fruit stem starch was weighed as much as 2.5 grams, made a suspension of 50 ml of distilled water (2.5 g/50 ml), taken 10 ml and put into a test tube, heated in a waterbath at 60°C for 30 minutes. After the waterbath, it was centrifuged at 3000 rpm for 15 minutes. The precipitate was separated and weighed, then dried in an oven at 130°C for 2 hours, the dried precipitate was weighed and the swelling power and solubility were calculated.

Swelling power and solubility are calculated based on equations 1 & 2

$$\% S = \frac{A}{W} \times 100\% \quad \ldots\ldots (1)$$

$$\% SP = \frac{D}{W(1-S)} \times 100\% \quad \ldots\ldots (2)$$

Keterangan :

%S = Solubility

%SP = Swelling power

A = Weight of substance after oven (substance after oven)

W = Weight of dry matter

D = Sediment weight (starch wet) [13].

2.3.12. Compressibility Index, Tap Density, Bulk Density, and Hausner Ratio

Red dragon fruit stem starch was placed in a 100 ml measuring cup and the initial volume (Vo) was recorded and tested for impermeability. Determination of 10, 500, 1250 times was carried out and the compressible volume was obtained [14].

2.3.13. Analysis of Fourier Transform Infrared (FTIR) Spectrum

Red dragon fruit stem starch was weighed as much as 2 mg using an analytical balance, measured the absorption with an FTIR spectrophotometer (Agilent Technologies Cary 630 FT-IR) at a wavelength of 4000 - 370 cm^{-1}

2.3.14. Particle Size Distribution

Particle size testing using particle size analyzer (PSA) Laser Scattering Particle Size Analyzer LA-960.

2.3.15. Microbial Contamination Test

The media used for Total Plate Count (ALT) testing is Plate Count Agar (PCA) while the Yeast Mold Number (AKK) is Potato Dextrose Agar (PDA) [14].

3. RESULTS AND DISCUSSION

The characterization test of red dragon fruit stem starch *(Hylocereus polyrhizus)* which includes organoleptic test, yield test, moisture content test, amylum qualitative test, flow rate, angle of repose, pH test, water content, ash content, solubility, swelling power, compressibility index, Hausner index, tap density, bulk density, true density, microscopic test, starch content analysis, microbial contamination test, FTIR, SEM test, and particle size analyzer (PSA) test. The results of the evaluation of red dragon fruit stem starch characteristics can be seen in Table 1.

Table 1. Characterization Test Results of Red Dragon Fruit Stem Starch

No	**Evaluation**		**Results**			**literature data *Corn Strach***
			1 h	**2 h**	**3 h**	[14][15]
1	Organoleptics	Shape	Powder	Powder	Powder	Powder
		Color	Broken- White	Broken- White	Broken- White	White
		Odor	Odorless	Odorless	Odorless	Odorless
		Flavor	Flavorless	Flavorless	Flavorless	Flavorless
2	Yield (%)		1.43 ± 0.03	1.39 ± 0.02	1.34 ± 0.02	-

continued table 1…

3	Moisture content (%)	2.40 ± 0.23	3.00 ± 0.25	3.48 ± 0.25	10 - 15
4	Identification	Dark blue	Dark blue	Dark blue	Dark blue
5	Flow rate (g/second)	1.05 ± 0.11	0.80 ± 0.05	0.59 ± 0.03	7.99
6	Angle of repose (°)	25.90 ± 1.11	29.89 ± 1.69	32.67 ± 1.25	25 - 30
7	pH	6.13 ± 0.10	5.67 ± 0.20	5.40 ± 0.05	4.0 – 7.0
8	Water content (%)	8.44 ± 0.96	10.94 ± 0.88	12.82 ± 0.73	-
9	Ash content (%)	0.40 ± 0.04	0.53 ± 0.08	0.52 ± 0.06	0.20 – 0.38
10	Swelling power (%)	30.72±10.90	58.22 ± 15.04	84.01 ± 8.75	-
11	Solubility (%)	5.37 ± 2.66	12.44 ± 4.93	18.48 ± 8.75	6 - 8
12	Compressibility index (%)	27.5 ± 5.75	30.5 ± 4.51	36.25 ± 3.30	24 - 30
13	Hausner ratio	1.39 ± 0.12	1.43 ± 0.06	1.58 ± 0.08	-
14	Tap density (g/mL)	0.50 ± 0.03	0.53 ± 0.03	0.61 ± 0.03	0.64 – 0.83
15	Bluk denity (g/mL)	0.36 ± 0.01	0.37 ± 0.01	0.39 ± 0.01	0.47 – 0.59
16	True density (g/mL)	2.38 ± 0.22	2.83 ± 0.16	3.05 ± 0.25	1.478
17	Particle size (μm)	216	235	229	2 - 32
18	Starch content (%)	58.66 ± 3.6	58.89 ± 4.97	56.32 ± 5.51	-

Description: average result of testing 4 replicates along with ±SD

Organoleptical tests of red dragon fruit stem starch with soaking times of 1 h, 2 h, and 3 h showed almost the same results is powder, broken-white, tasteless and odorless, but the 3 h soaking showed slightly whiter starch results. The results of the yield test obtained by red dragon fruit stem starch the longer the soaking time the resulting yield will be more. The difference in the yield of red dragon fruit stem starch is due to the material that is too long soaked, the water content in the red dragon fruit stem and other components contained in the red dragon fruit stem will dissolve in the soaking water. The yield test results can be seen in Table 1.

The results of the moisture content obtained from red dragon fruit stem starch with a soaking time of 3 h are greater than the red dragon fruit stem starch with a soaking time of 1 h and 2 h, this is because the longer the soaking time, the higher the absorbed water content will be. In the results of testing the flow rate that can be seen in Table 1 shows that the longer the immersion of the flow rate produced will be smaller, while in testing the angle of respose, the longer the immersion time will be the greater the angle of repose produced. Stationary angle testing is related to water content, the lower the water content in the sample, the less water content in the sample so that the flow properties are faster, where the faster flow rate indicates that the starch flows freely so that a small stationary angle is formed.

Red dragon fruit stem starch with a soaking time of 3 h is lower and tends to be more acidic than the pH of starch with soaking times of 1 and 2 h. The longer the soaking time and the higher the concentration of sodium bisulfite used causes the pH of the starch produced to be more acidic, because in water sodium bisulfite will break down into sulfuric acid (H_2SO_3) which can reduce pH.

In testing the water content, it was concluded that the longer the soaking time, the higher the water content. The difference in the amount of water content of red dragon fruit stem starch is due to the length of soaking so that the absorption of water by the tissue increases. The results of the ash content test can be seen in Table 1, indicating that the length of soaking time of red dragon fruit stem starch has no effect on the ash content value.

From the test results of solubility and expandability, it was found that red dragon fruit stem starch with a soaking time of 3 h had the highest solubility and expandability values compared to red dragon fruit stem starch with a soaking time of 1 h and 2 h. Solubility is related to expandability, if the higher the expandability of a starch, the solubility of the starch will increase. The higher the expandability value, the more water is absorbed. The results of the expandability and solubility tests can be seen in Table 1.

Microscopic examination using a binocular microscope connected to an optilab application with a microscope magnification of 400x and 1000x showed that dragon fruit stem starch was round and flat, hilus and lamella were not clearly visible. It can be seen that red dragon fruit stem starch with a soaking time of 1 h is not much different from red dragon fruit stem starch with a soaking time of 2 h and 3 h. Microscopic test of red dragon fruit stem starch can be seen in Figure 1. Microscopic test results of red dragon fruit stem starch samples have round and flat particle shapes, hilus and lamella are not clearly visible. Scanning Electrone Microscopy (SEM) test results of red dragon fruit stem starch can be seen in Figure 2.

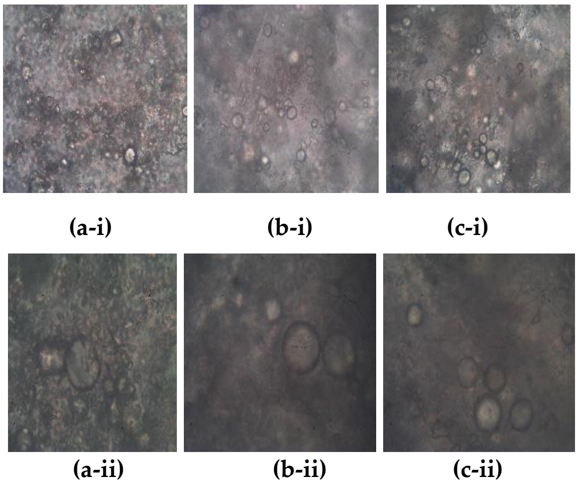


Figure 1. Microscopic of Red Dragon Fruit Stem Starch (a) Soaking Time 1 h, (b) Soaking Time 2 h, (c) Soaking Time 3 h with (i) 400x and (ii) 1000x Magnification

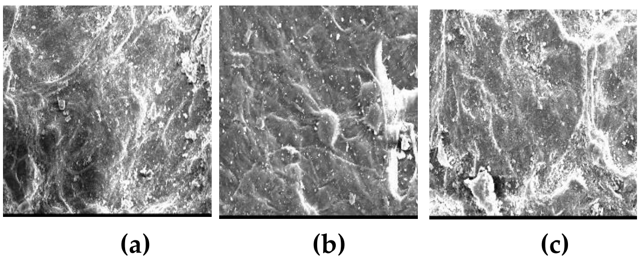


Figure 2. Scanning Electrone Microscopy (SEM) of Red Dragon Fruit Stem Starch (a) Soaking Time 1 h, (b) Soaking Time 2 h, (c) Soaking Time 3 h with 1000x Magnification

SEM test was conducted to determine the size of the red dragon fruit stem starch that has been made. Scanning Electrone Microscopy (SEM) test results can be seen that red dragon fruit stem starch with 1000x magnification shows the surface of the sample is not smooth and uneven and has an average particle size of 10 μm. Red dragon fruit stem starch still meets the general microgranule size requirements of 1-1000 μm [16].

The results obtained from the analysis of starch content can be seen in Table 1, the highest starch content is in red dragon fruit stem starch with a soaking time of 2 h. The differences found in the three samples can be influenced by the level of purity during the process, because the more mixtures such as fiber, sand or impurities that participate in starch, it can affect the resulting starch content. The results of FTIR absorption of red dragon fruit stem starch can be seen in Figure 3.

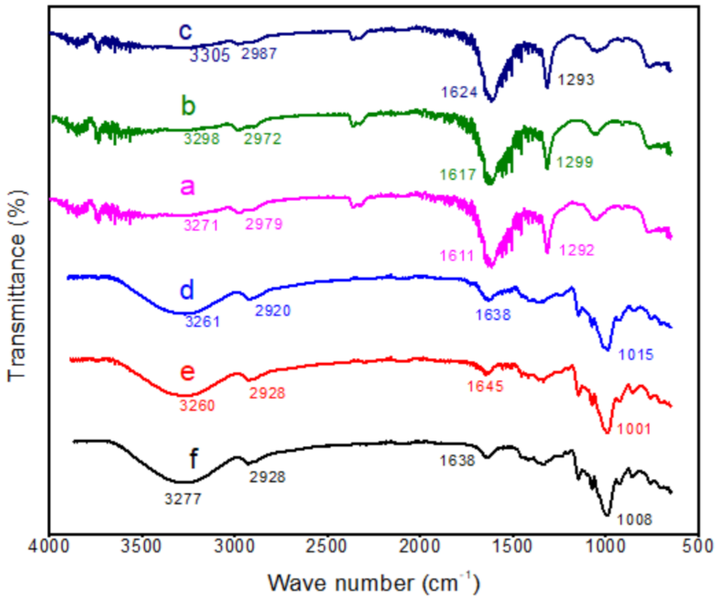
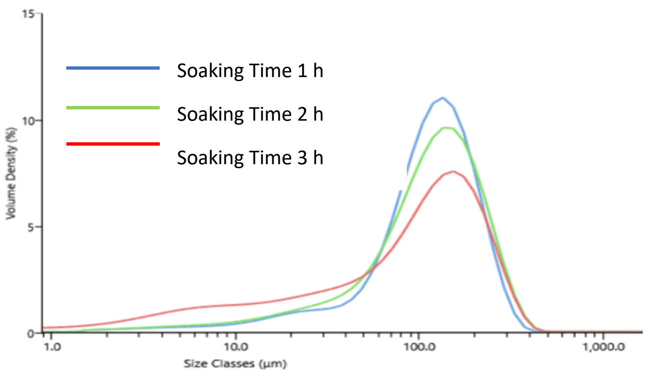


Figure 3. FTIR images of Red Dragon Fruit Stem Starch (a) Soaking Time 1 h, (b) Soaking Time 2 h, (c) Soaking Time 3 h, (d) Potato Strach, (e) Corn Starch and (f) Cassava Starch

Red dragon fruit stem starch with soaking time of 1 h 3271 cm^{-1}, 2 h 3298 cm^{-1}, and 3 h 3305 cm-1 showed the peak intensity of hydroxyl group (-OH) strain. The absorption bands 2979 cm^{-1}, 2972 cm^{-1}, and 2987 cm^{-1} show the absorption of (-CH3) aliphatic strain. The 1611 cm^{-1}, 1617 cm^{-1} and 1624 cm^{-1} absorption bands showed the presence of (-C=O). Carbonyl groups (C=O) are formed due to the presence of alcohol groups in starch that undergo oxidation. The 1292 cm^{-1}, 1299 cm^{-1} and 1293 cm^{-1} absorption bands show (-C-O) absorption, so it can be concluded that red dragon fruit stem starch contains starch functional groups.

The average results of compressibility index and Hausner ratio on red dragon fruit stem starch with 1 h soaking time are lower, when compared to 2 h and 3 h soaking time. A lower compressibility index or lower hausner ratio indicates better flow properties than higher ones. The percent compressibility result is influenced by particle size and its distribution [17].



Soaking Time (h)	Dx (10) (µm)	Dx (50) (µm)	Dx (90) (µm)
1	38.5	118	216
2	31.7	120	235
3	7.04	97.6	229
Mean	25.7	112	226
SD	16.5	12.2	9.90
RSD (%)	64.3	11.0	4.37

Figure 4. Particle Size Distribution Test Results

Particle size distribution testing using PSA (Particle Size Analyzer) Malven® Mastersize 3000 (Malvern Instruments, UK) the average results of particle size distribution testing can be seen in Figure 4. The higher the number of Dx used, the greater the distribution of samples in the test. Based on the results obtained, it is concluded that the particle size distribution of red dragon fruit stems does not meet the requirements, because it does not enter the range of 2 - 32 μm [15] and the length of dragon fruit soaking time has no effect on particle size distribution.

ALT and AKK microbial contamination testing can be seen in Table 2. it can be seen that the colony results obtained are still within the range of predetermined limits, so that red dragon fruit stem starch is still safe to use as an additive to solid pharmaceutical preparation.

Table 2. Total ALT and AKK Contamination of Red Dragon Fruit Stem Starch

Red Dragon Fruit Stem Starch Samples with Variation of Soaking Time Number (CFU/mL)	**Total (CFU/mL)**		
	ALT	**AKK Day-5**	**AKK Day-7**
1 h	$6.1 X 10^2$	$1.7 X 10^2$	$2.3 X 10^2$
2 h	$5.2 X 10^2$	$5 X 10^1$	$4.8 X 10^2$
3 h	$6.7 X 10^2$	$2.3 X 10^2$	$5.2 X 10^2$

4. CONCLUSION

Based on the characteristic tests carried out, it can be concluded that differences in soaking time can affect the characterization of each sample of red dragon fruit stem starch, There are significant differences (p-value<0,05) in the test results of yield, moisture content, flow rate, angle of repose, pH, solubility, swelling power, compressibility index, density and hausner index.

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