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Characterization of Pregelatinized, Pentanol-Modified, and Acetylated Pratama Taro Starch

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Tablets are pharmaceutical dosage forms consisting of active ingredients and excipients. One commonly used excipient is starch. Pratama taro is a plant containing a high source of starch. The use of native starch in tablets has limitations, such as poor flow properties and compressibility. This study aimed to modify native starch through pregelatinization, pentanol, and acetylation. The research was conducted using an experimental laboratory method, starting with the sorting of pratama taro tubers, starch isolation, modification, and characterization of the resulting starch. Characterization results showed that the flow rates of native, pregelatinized, pentanol-modified, and acetylated starches were 1.9 ± 0.19, 2.15 ± 0.37, 1.35 ± 0.15, and 1.95 ± 0.14 grams/second, respectively, with angle of repose values of 20.49 ± 1.99, 17.12 ± 1.99, 22.92 ± 1.18, and 20.06 ± 1.97°. The compressibility values were 20.23 ± 5.31, 15.47 ± 3.03, 21.09 ± 2.8, and 19.09 ± 2.05%. Based on the obtained results, pregelatinized native starch exhibited better flow properties compared to pentanol and acetylation modifications.

Keywords: starch modification, characterization, pratama taro

1. INTRODUCTION

Indonesia has a tropical climate and an abundance of natural resources with the potential to heal various diseases and serve as raw pharmaceutical materials (Yassir and Asnah, 2019). However, utilization of medicinal raw materials in Indonesia is currently still limited (Octavia et al., 2019). Data collected by the Ministry of Industry in 2021 shows that 95% of pharmaceutical raw materials in Indonesia are still imported (Ministry of Industry, 2021). About 70% of drug raw materials are imported from China, 20% from India, and the rest from the United States and the European Union (Ministry of Industry, 2021). One raw pharmaceutical material (excipient) found in Indonesia is starch (Hartesi et al., 2020).

Starch can be used as an excipient in tablet manufacturing when physically and chemically modified (Octavia et al., 2019). Research on pregelatinization modification using potato starch at a starch-to-water ratio of 1:1.25 yielded a flow rate of 9.95 grams/second and optimal compressibility percentage of 9.44% in formula 9 (Hartesi et al., 2020). Research utilizing starch from taro corms (Colocasia esculenta Schott) as filler across four paracetamol tablet formulas at concentrations of 8.35% (FI), 12% (FII), 15.3% (FIII) and 18.4% (FIV) demonstrated that all four paracetamol tablet preparations met granule

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and finished tablet test requirements (Apriyanto et al., 2017).

Pratama variety taro (Colocasia esculenta (L.) Schott var. Pratama) is a newly developed taro strain first mass propagated in mid-2016 (Nurliani et al., 2019). Pratama taro starch can serve as a matrix in potassium diclofenac tablet preparations (Kurniadi et al., 2023). Starch content in taro tubers ranges from 13-29% with moisture levels of 63-85% (Karmakar et al., 2014). Poor flow properties, compressibility, and matrix formation of natural starch limits pharmaceutical applications (Hartesi et al., 2020). Modifications like pregelatinization (Hartesi et al., 2020), pentanol addition (disrupts amylose-amylopectin bonds), and acetylation using acetic acid or starch acetate can improve flow and compressibility (Octavia et al., 2019; Astuti et al., 2023). The urgency of this research is to improve the flow properties and compressibility of Pratama taro starch for pharmaceutical suitability.

2. RESEARCH METHODOLOGY

This study is a laboratory experiment aimed at determining the printed mass and physical properties of diclofenac potassium sustained release tablets using pregelatinized, pentanol-modified and acetylated Pratama taro starch (Colocasia esculenta (L.) Schott var. Pratama).

2.1 Determination of Pratama taro plant

Determination of Pratama taro was carried out at the Biology Laboratory, Faculty of Pharmacy, Muhammadiyah University of Purwokerto to verify the accuracy of the plant used in the study.

2.2 Isolation of starch from Pratama taro

The 9-month-old Pratama taro used was cleaned of attached dirt under running water. The tubers were then peeled and washed repeatedly until clean. A total of 6 kg of taro tubers were weighed, then soaked in water for 1 hour to soften the tuber tissue for easier crushing. The soaked taro was then blended into Pratama taro paste. The obtained Pratama taro paste was added with water at a 1:2 ratio, while stirring to release more starch from the tuber cells.

The Pratama taro tuber paste was filtered through cloth so the starch passed through as a starch suspension while the pomace remained in the cloth. The Pratama taro starch suspension was allowed to settle for 8 hours in a container. The obtained sediment was then drained to separate the starch from the water. The Pratama taro starch sediment without water was dried at 60°C for 12 hours then cooled. It was then sieved using an 80 mesh to produce Pratama taro starch with a uniform size (Irhami et al., 2019).

2.3 Physicochemical modification of Pratama taro starch

a. Pregelatinization modification

The modification process was carried out by preheating water to 45°C on a hot plate, then mixing it with Pratama taro starch at a 1:1 ratio at 45°C for 10 minutes using a 300 rpm homogenizer until a viscous mass formed. The viscous mass was then oven dried at 45°C for 48 hours. After the starch was dry, it was sieved through a 60 mesh (Hartesi et al., 2020).

b. Pentanol modification (disruption of amylose-amylopectin bonds)

Starch/amylum modification with pentanol results from the disruption of bonds between amylose and amylopectin. The procedure involves mixing Pratama taro starch and pentanol at a 1:1 ratio using a homogenizer for 30 minutes at room temperature. The starch was then dried at 45°C and sieved through an 80 mesh (Octavia et al., 2019).

c. Acetylation modification with acetic acid (starch acetate)

This modification was done by mixing 250 g of Pratama taro starch and 750 mL of water (1:3 ratio), homogenizing the mixture at around 27°C. Then, 16 mL of glacial acetic acid was slowly added into the mixture with occasional stirring for 30 minutes. This was followed by repeated 3-time filtration, separating the liquid from the starch, then oven drying the starch. After drying, it was sieved through an 80 mesh (Octavia et al., 2019).

2.4 Characterization of Pratama taro starch

Starch quality must first be evaluated according to the requirements, including organoleptic testing, flow rate, angle of repose and compressibility percentage.

a. Organoleptic testing

The general appearance of starch was observed including shape, color, taste, smell and particle size using a Particle Size Analyzer (PSA) to characterize the particle size of pregelatinized, pentanol-modified and acetylated starch.

b. Flow rate testing

A granule flow tester was used to determine flow rate. 25 grams of sample was weighed and placed in the tool funnel and the time taken for complete sample flow was recorded. Good flow rate is 4-10 grams/second (Edy and Mansauda, 2020).

c. Angle of repose measurement

First, 25 grams of granules was weighed and placed in a granule flow tester. The surface was flattened and the sample was allowed to flow freely while measuring the slope angle. This evaluates the powder flow properties of starch. It involves using a funnel to observe the flowing powder's radius and height, which depends on factors like particle size, shape and moisture (Aulton and Taylor, 2018).

d. Evaluation of compressibility percentage

Compressibility highly relates to density, particle size and shape. A tapped density tester was used by weighing the powder sample (Aulton and Taylor, 2018). To determine compressibility percentage, the sample was placed in a 100 mL volumetric flask and tapped 200 times using a tapped density tester. The final powder volume was observed and the percentage compressibility calculated.

3. RESULTS AND DISCUSSION

3.1 Plant Determination Results

The results of the determination proved that the plant used by the researchers matched the studied plant species, namely pratama taro with the scientific name Colocasia esculenta (L). Schott var. Pratama. The pratama taro plant can be seen in Figure 1.

Figure 1. Pratama taro plant

Pratama taro can reach a height of 1.5-2 meters, with tuber sizes ranging from 1.5-2 kg at 8 months of planting age. The pratama taro tubers used in this study were then subjected to starch isolation and modification treatments.

3.2 Pratama Taro Starch Isolation Results

The isolation of pratama taro starch with an initial tuber weight of 6 kg yielded 833.132 grams of pratama taro starch. The yield value was then calculated by dividing the used taro tubers by the resulting pratama taro starch, obtaining a yield value of 13.89%. The obtained yield was not significantly different from a previous study on sweet potato starch isolation using oven drying at 60°C for 6 hours and sieving with an 80 mesh sieve, which resulted in an average starch yield of 11.92% (Irhami et al., 2019).

3.3 Physicochemical Modification Results of Pratama Taro Starch

a. Pregelatinization Modification

Pregelatinization modification was carried out by heating at 60°C to obtain starch with larger particle size and increased particle density, thus improving flow rate and compressibility (Hartesi et al., 2022). The result of pregelatinization modification with a ratio of 250 grams of pratama taro starch and 250 mL of water (1:1) yielded 234.89 grams. During the pregelatinization process, there were procedures involving mixing with distilled water at a certain ratio, oven drying, and sieving using a mesh, which could lead to some starch remaining in the container during the process. Previous research on pregelatinization modification of white glutinous rice starch resulted in the best flow properties of 9.99 grams/second compared to native starch that did not flow through the funnel, which also affected the good compressibility percentage value (Hartesi et al., 2023).

b. Pentanol Modification (Amylose-Amylopectin Bond Cleavage)

A total of 250 grams of native starch modified with pentanol produced 233.98 grams of pentanol-modified starch. The principle of pentanol modification is that pentanol compounds bind to the -O- group on the alpha C atom, which then breaks the alpha-1,4 glycosidic bond connecting one glucose to another, resulting in starch with a smaller molecular size than native pratama taro starch. In a previous study, pentanol-1 modification exhibited good hardness with scales of 5, 6, and 7, influenced by the small and fine powder size that filled the tablet mold well (Mahalia et al., 2020).

c. Acetylation Modification with Acetic Acid (Acetylated Starch)

Dilakukan pada suhu kamar 27ºC dengan Acetylation was carried out at room temperature (27°C) by mixing 250 grams of starch with 750 mL of distilled water (1:3), then slowly adding glacial acetic acid, filtering with cloth, and oven-drying at 60°C (Octavia et al., 2019). The acetylation modification yielded 194.97 grams. Acetic acid modification involves inserting an acetyl group from acetic acid compounds to replace the -OH group present in starch through an acetylation reaction, resulting in acetylated starch. This reduces the strength of hydrogen bonds between starch molecules and causes starch granules to dissolve easily in water. The yield of acetylation modification was the lowest compared to pregelatinization and pentanol modifications, possibly due to some starch remaining during filtration. Acetylated starch showed better solubility and swelling percentages compared to native starch, which is advantageous for industrial-scale use, especially in the pharmaceutical field (Octavia et al., 2019).

3.4 Characterization of Pratama Taro Starch

a. Organoleptic Test

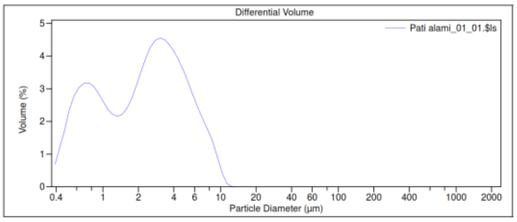
Organoleptic testing was conducted to determine the properties of the material through visual observation. The established appearance can include texture, color, taste, and odor of the starch (Dadang and Pertiwi, 2020). The results showed that pentanol-1 modified starch had a powder form with a stickier texture and darker color compared to all other modifications and native starch.

This can occur because pentanol-modified starch has a smaller size, and the presence of adhesive and cohesive forces causes the resulting starch to have a sticky and smoother texture (Octavia et al., 2019). The results of the organoleptic test are presented in Table 1.

Table 1. Organoleptic test results

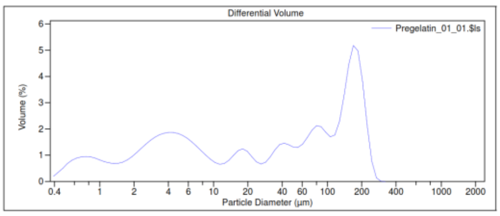
Modification	Texture	Color	Taste	Smell
Native starch	Fine powder	White	Bland	No discernible odor
Pregelatinized	Fine powder	White	Bland	No discernible odor
Pentanol-1	Fine and somewhat sticky powder	Slightly darker white	Bland	No discernible odor
Acetylated	Fine powder	White	Bland	No discernible odor

Further starch characterization was performed using a Particle Size Analyzer (PSA) instrument to determine the particle size of the samples. The particle size test results can be seen in Figure 2.



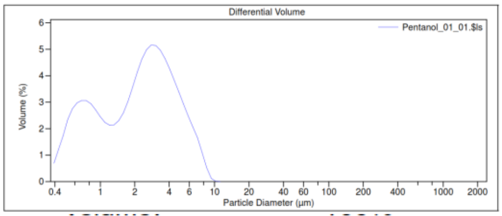
Mean: 2.835 µm

(a) Native Pratama Taro Starch



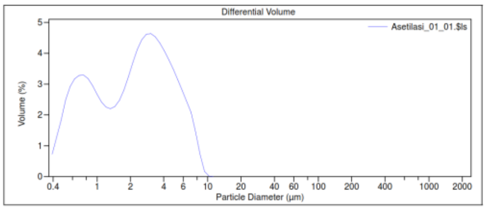
Mean: 66.83 µm

(b) Paregelatinized Starch



Mean: 2.619 µm

(c) Pentanol-modified Starch



Mean: 2.662 µm

(d) Acetylated Starch

Figure 2. Particle size test results using PSA instrument

Pregelatinization modification can increase the particle size of native pratama taro starch. This result is consistent with a previous study on pregelatinized canna starch, which had a larger particle size of 25-30 µm (Azhary et al., 2019). Based on Figure 2, native taro starch, pregelatinized, pentanol-modified, and acetylated starches had particle sizes of 2.835, 66.84, 2.619, and 2.662 µm, respectively. It is known that the average particle size of pentanol and acetylation-modified starches was smaller than native starch. Particle size is one of the factors influencing flow rate. From the conducted tests, pregelatinized starch showed the fastest powder flow ability. Changes in the structure of pregelatinized starch granules and the swelling characteristics of starch in water cause the starch size to tend to be larger, thus improving flow rate (Hartesi et al., 2020).

b. Flow rate Test

The flow rate test aims to achieve uniform flow to ensure that each tablet has the same or nearly the same powder mass (Aulton and Taylor, 2018). The flow rate test was conducted to determine the flow properties of the samples. The obtained flow rates fell within the good range of 4-10 grams/second (Edy and Mansauda, 2020). With a good flow rate, the powder can flow freely and fill the mold space.

Table 2. Starch flow rate test results

Type of modification	Flow rate (g/second)
Native starch	1.9 ± 0.19
Pregelatinized	2.15 ± 0.37
Pentanol-1	1.35 ± 0.15
Acetylated	1.95 ± 0.14

Based on Table 2, pratama taro starch had the slowest flow criteria compared to modified starches, both pregelatinized and acetylated. The flow rate of a sample can be influenced by the size and shape of the sample (Rohmani and Rosyanti, 2019). According to Figure 2, the particle size of pregelatinized starch was the largest (66.83 µm), resulting in faster flow ability compared to other starches. Flow time directly affects the magnitude of the angle of repose formed; the lower the angle of repose, the faster the flow rate (Siregar and Wikarsa, 2010).

This is consistent with the changes in the structure of pregelatinized starch granules and the swelling characteristics of starch in water, having larger particle sizes, which can improve flow rate (Hartesi et al., 2020). Additionally, acetylation-modified starch produces more stable starch, allowing the powder to flow well from the hopper (Nurhayati, 2019). This is in line with a previous study that resulted in an average flow rate of 9.95 g/second for pregelatinized potato starch (Hartesi et al., 2020).

In the flow rate test results of pentanol-1 modified starch, the slowest flow rate of 1.35 ± 0.15 g/second was obtained. This occurred because pentanol-1 modification has stickier characteristics compared to other modifications, resulting in poor flow properties (Octavia et al., 2019). Small and fine particle sizes can increase cohesive forces and slow down powder flow (Mahalia et al., 2020).

c. Angle of Repose Test

The angle of repose is a characteristic of the internal friction and cohesion of particles, thus describing the flow rate. The test results showed that powders with a good angle of repose fall within the range of 25-35° (Aulton and Taylor, 2018). The angle of repose test is part of the flow rate test; the larger the angle of repose formed, the slower the flow properties (Hartesi et al., 2023). The angle of repose can be a determinant of the flowability of a powder (Nawangsari, 2019).

Table 3. Angle of repose test results

Sample	Angle of repose (°)
Native starch	20.49 ± 1.99
Pregelatinized	17.12 ± 1.99
Pentanol-1	22.92 ± 1.18
Acetylated	20.06 ± 1.97

Based on Table 3, only pregelatinized starch had the lowest angle of repose, consistent with its flow rate ability in the previous test. The smaller the angle formed in the granule flow tester, the faster the sample's flow rate. Larger particle sizes will result in a good angle of repose (Hartesi et al., 2023).

The angle of repose is proportional to the flow rate. The smaller or faster the flow time, the smaller the angle of repose formed, indicating low cohesion between particles and better fluidity (Puspadina et al., 2021). The angle of repose increases with increasing water content (relative humidity) and decreases with increasing particle size (Badwan et al., 2015). In the PSA test results, pregelatinized starch had the largest particle size, while pentanol modification resulted in a particle size of 2.619 µm. The small particle size of pentanol-modified starch can be a factor in the small static angle obtained in this study.

This is in line with a previous study on pregelatinized red sweet potato starch, which showed that the obtained angle of repose met the requirements with the best starch to water ratio (1:1.25) of 25.49° and ratio (1:1) of 26.92° (Hartesi et al., 2022). Furthermore, pregelatinized white glutinous rice starch resulted in a good angle of repose with an average of 25.77° (Hartesi et al., 2023).

d. Compressibility Percentage Test

Powder compressibility is defined as the tendency of a powder to reduce its volume when placed in a confined space and subjected to pressure or load (Aulton and Taylor, 2018). Compressibility is higher if the difference between the true density and tapped density is greater (Hartesi et al., 2023). The test results showed that powders with good compressibility fall within the range of 11-20% (Aulton and Taylor, 2018).

Table 4. Compressibility percentage test results

Sample	Compressibility (%)
Native starch	20.23 ± 5.31
Pregelatinized	15.47 ± 3.03
Pentanol-1	21.09 ± 2.8
Acetylated	19.09 ± 2.05

Based on Table 4, the compressibility percentage test results showed that the starches could be compressed well, as they met the required range. Particle size, shape, and density affect compressibility percentage; the lower the powder density, the better the flowability or flow rate (Sirisha et al., 2012).

This is related to the angle of repose and flow rate test results that fall within the required range. The magnitude of the compressibility percentage is determined by the powder's properties when filling the interparticle space and compacting, so a small compressibility percentage indicates that the powder has a good ability to arrange particles and a lower compressibility percentage indicates a faster flow rate (Mahalia, 2018). Compressibility is related to powder characteristics during compression; good results will produce tablets with a long disintegration time (Hartesi et al., 2022).

This is consistent with a study showing that pregelatinized white glutinous rice starch had a compressibility percentage that met the requirements with the best average result of 10.32% (Hartesi et al., 2023). Additionally, the best compressibility results for pregelatinized red sweet potato starch were 9.60% for the starch to water ratio of (1:1) and 9.50% for the ratio of (1:1.25), indicating that the results met the requirements (Hartesi et al., 2022).

The limitation of this study is that the utilization of modified starch in tablet dosage forms as an excipient has not been carried out to prove the benefits obtained from the modifications that have been performed.

4. CONCLUSION

The results of this study showed that among the modifications made to pratama taro starch, pregelatinization modification exhibited the best characterization in terms of flow rate, angle of repose, and compressibility. The particle size of pregelatinized starch increased by 30 times compared to native taro starch.

5. RECOMMENDATIONS

Pratama taro starch can be used as an excipient in tablets by applying pregelatinization modification.

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