

Effect of Variations in Acetic Acid Concentration on the Physicochemical Characteristics of Sweet Potato Starch Isolation Results

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ABSTRACT

This study aims to analyze the effect of variations in acetic acid concentration (0; 0.5; 1; 1.5; and 2%) on the physicochemical characteristics of starch. The parameters analyzed included moisture content, water absorption, swelling power, and solubility, as well as structural characterization using Fourier Transform Infrared (FTIR). Data analysis was carried out with one-way ANOVA at a significance level of 5%. The results showed that the variation in acetic acid concentration had a significant effect on water content and swelling power ($p < 0.05$). The value of swelling power increases at certain concentrations and decreases at higher concentrations, indicating a change in the granules. Conversely, water absorption and solubility were not significantly different ($p > 0.05$). FTIR analysis showed that the addition of acetic acid did not cause covalent chemical modifications, but rather only affected the physical interactions and hydrogen bonds of starch granules.

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INTRODUCTION

Starch is a natural biopolymer that is widely used in various fields, such as food, pharmaceuticals, and bio-based materials, due to its renewable properties, biodegradable, and relatively inexpensive [1,2]. Sweet potatoes (*Ipomoea potatoes* L.) are known as one of the foods that is rich in carbohydrates, both in Indonesia and globally, and contains various bioactive compounds such as anthocyanins that give sweet potatoes certain chemical characteristics [3,4]. Thanks to its high availability and easily modifiable properties, sweet potato starch has a great opportunity to be used as a raw material in various sectors of the food and non-food industry [5,6]. However, to maximize this utilization, understanding its characteristics is very important. The structure of starch granules composed of amylose and amylopectin can be improved in functionality through physical, chemical, and enzymatic modifications by manipulating the hydrogen bonds in them [7].

The modification is carried out using acetic acid through the interaction between hydroxyl groups ($-OH$) on starch polymer chains with acetic acid as a modification agent [8]. In addition, acetate-derived compounds are associated with molecular interactions at starch hydroxyl groups that can affect the structure and conformation of polymers [9]. These

interactions occur at the molecular level, influencing how functional groups in starch behave, but do not necessarily change the chemical structure of starch entirely. Under certain conditions, the presence of acetic acid can play a role in affecting the regularity of starch granule structure as well as interactions between polymer chains. Therefore, this process is associated with changes in the molecular structure of starch, both partial and limited, which are further related to the physicochemical characteristics of starch, such as water absorption, solubility, and other functional properties [10].

Several studies have shown that acid treatment can alter the structure of starch granules, thereby affecting their gelatinization ability and functional properties for food and non-food industry applications [11,12]. This is reinforced by research (Bolande and Oni, 2015), which showed that modification using acetic acid in a mixture of sweet potato and cassava starch simultaneously increased the water absorption, solubility index, and resistance of the gel to the freezing cycle-liquid, despite a reduction in viscosity and oil binding ability [13]. Therefore, variations in acetic acid concentrations in the starch isolation process are an important aspect to be studied to understand the changes in physicochemical properties that play a role in determining the final characteristics of starch [14].

This study focuses on the study of the effect of acetic acid variation at low concentrations on the physicochemical characteristics of isolated sweet potato starch in order to obtain a comprehensive understanding of starch characteristics at each level of treatment. This study presents the analysis of several parameters of physicochemical characteristics of starch, including swelling power, solubility, water absorption, and moisture content, as well as the identification of functional group changes using FTIR analysis due to treatment of variations in acetic acid concentration. The results of this study are expected to provide scientific information regarding the relationship between acid treatment and changes in the structure and physicochemical properties of isolated sweet potato starch.

RESEARCH METHODS

Tools and Materials

The equipment used includes *Pyrex* brand glass beaker, knives and cutting boards for material preparation, *Advan* brand blender, 100 mesh sieve, mixing rods, *Memmert brand digital oven*, *Gyrozen brand centrifuges*, and *Kern ABS 220-4N brand analytical balance*. In addition, supporting equipment is also used in the form of *an Iwaki* brand measuring gourd, a *Pyrex brand measuring cup*, a *magnetic stirrer* to homogenize the mixture, universal pH indicator paper to regulate the pH of the solution during the neutralization process, and a centrifuge tube to support the starch isolation and processing process. The analysis of functional groups in starch was carried out using the *Shimadzu* brand Fourier Transform Infrared (FTIR) instrument.

The material used in this study is purple sweet potato obtained from Munduk Village, Buleleng Regency, Bali. The chemicals used include glacial acetic acid from *Smartlab* as a concentration variation treatment, pH control, Sodium Hydroxide, and Aquadest used as solvents and detergents during the starch isolation process.

Research Stages

This stage of research includes the process of isolating sweet potato starch with modification treatment using acetic acid, followed by testing the physicochemical properties of the starch produced and characterizing the functional groups using FTIR instruments. The isolation process begins with the preparation of ingredients, followed by soaking treatment using acetic acid solutions at various concentrations, then drying and refining of starch until a homogeneous starch powder is obtained. A detailed description of each stage of the process is provided in the following paragraphs.

Starch Insulation

Following the procedure (Vithu et al., 2020), the isolation of purple sweet potato starch begins with the extraction of 100 g of fresh samples that have been mashed with aqueous (1:3 w/v). The suspension of the filtered results is left for 4–6 hours until sedimentation is formed. The resulting sediment is then cleaned through two flushes with aqueducts, then dried at a temperature of 45–50 °C for 12–24 hours until dry starch is obtained. To ensure size homogeneity, the dried starch is ground and filtered using a 100 mesh sieve [15].

Acetic Acid Treatment in Starch

The isolated sweet potato starch was treated with variations in acetic acid concentrations of 0% (control), 0.5%, 1%, 1.5%, and 2% (w/v) with a starch-to-acetic acid solution ratio of 1 : 5 (w/v). The starch is dispersed in aqueous solution until it forms a homogeneous suspension, then an acetic acid solution is added according to the treatment, and the mixture is then homogenized using a magnetic stirrer at room temperature for 30 minutes. After treatment, the starch suspension is neutralized using a 1 M NaOH solution until it reaches a pH close to neutral, then washed with water and centrifuged to separate the starch deposits. The starch deposits are then dried in the oven at 50 °C to a constant weight, ground, and stored in a sealed container before physicochemical analysis and FTIR characterization.

Physicochemical Analysis

Yield analysis was carried out by following the methods that have been reported in the study (Hartesi et al., 2022) [16]. The analysis of swelling power and solubility was conducted with reference to the method (Martínez-rubio and Londoño-restrepo, 2025) [10]. Water absorption was analyzed based on the approach reported by (Tatiana et al., 2021) [17]. The moisture content was analyzed using the gravimetric method using an oven at a temperature of 105 °C until a constant weight is obtained [18]. Each physicochemical parameter was analyzed using a different starch sample to avoid the influence of previous treatments on the test results.

FTIR Analysis

Spectrum Fourier Transform Infrared (FTIR) natural sweet potato starch and modified sweet potato starch with acetic acid were analyzed using the brand's FTIR instrument, *Shimadzu*, with the KBr pellet method, referring to the procedure reported by (Cecilia et al., 2026) [19]. The spectrum was recorded in the range of 4000–400 cm^{-1} . The identification of acetyl groups is based on the appearance of carbonyl absorption bands (C=O) in the range of 1730–1750 cm^{-1} . This analysis aims to evaluate changes in the chemical structure of starch as a result of the modification process, which is indicated by the difference in the infrared spectrum between starch before and after treatment.

Data Analysis

The physicochemical characteristics of sweet potato starch, including moisture content, swelling power, solubility, and water absorption, were analyzed quantitatively by calculating the mean and standard deviation from three repetitions. The effect of variations in acetic acid concentration on each parameter was tested using *One-Way Analysis of Variance* (ANOVA) at a significance level of 5% ($\alpha = 0.05$). If the ANOVA results showed a significant difference ($p < 0.05$), the Tukey test was used to identify differences among treatment groups. FTIR spectrum analysis was performed qualitatively by identifying absorption peaks in the wavenumber range of 4000–400 cm^{-1} . Changes in peak position and intensity were compared between the control sample and the treatment sample to evaluate possible changes in functional groups due to acetic acid treatment. The analyzed data is presented in the form of tables and graphs to facilitate the interpretation of the results.

RESULTS AND DISCUSSION

Yield

Yield is an important parameter in the evaluation of the efficiency of the starch isolation process because it reflects the proportion of starch that has been successfully separated from the raw material matrix. In general, the starch content in tubers ranges from 16–24% of the dry weight, but the yield value of the insulation is usually lower due to loss during the extraction, filtering, and drying processes [20]. In this study, starch isolation from 100 grams of sweet potatoes resulted in a yield value of 12.09%. This value is still within the range reported in several previous studies, where the yield of fresh sweet potato starch generally ranges from 10–20%, depending on the variety, extraction method, and process conditions [19,20].

The value of starch yield after treatment with acetic acid is presented in Table 1. The increase in starch yield at a concentration of 2% acetic acid is thought to be related to the increased efficiency of the separation of non-starch impurities and the deposition process of starch granules. At this concentration, acetic acid has not caused degradation in starch granules, so the mass loss is relatively small. The lower starch yield at the concentration of 0.5% acetic acid is thought to be due to the low separation efficiency, so that some of the starch granules are still carried in the supernatant phase during the washing and centrifugation process. In contrast, at a concentration of 2%, acetic acid is more effective in releasing non-starch components without causing granule degradation, so that mechanical losses are reduced, and starch yields are increased [23]. This shows that at low to moderate acid concentrations, the morphology of starch granules is still maintained, and the hydrolysis process that occurs does not cause significant structural damage. This condition allows most starches to remain stable during the treatment process so that the loss of starch mass becomes very small. This is in line with the data in the table, which shows that the yield value of starch increased from 89.89% at the concentration of acetic acid 0.5% to 95.98% at the concentration of 2%. The increase in yield indicates that the acid treatment does not cause significant starch degradation, so most of the starch can still be retained during the isolation process.

Table 1. Starch Yield Value After Treatment of Acetic Acid Concentration Variation

Acetic Acid Concentration (%)	Rendemen (%)
0.5	89.89
1	90.43
1.5	93.20
2	95.98

Several previous studies have stated that acid hydrolysis in starch tends to invade the amorphous region first, while the structure of starch granules is generally maintained under low–medium concentration treatment conditions, resulting in severe granule degradation and relatively small starch mass loss [24].

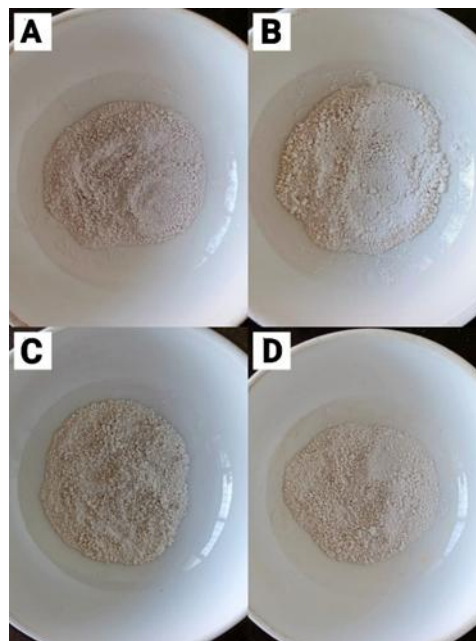


Figure 1. Starch Isolated After Treatment with Acetic Acid Variations with Concentration (A) 0.5% (B) 1% (C) 1.5 (D) 2.

Table 2. Physicochemical Characteristics of Sweet Potato Starch Acetic Acid Addition

Concentration (%)	Swelling Power (g/g)	Solubility (%)	Water Absorption (g/g)	Moisture Content (%)
0	15.84 ± 0.99 ^a	10.97 ± 1.72 ^a	24.83 ± 2.26 ^a	12.52 ± 0.42 ^a
0.5	16.62 ± 0.58 ^a	9.74 ± 1.48 ^a	28.07 ± 2.03 ^a	12.37 ± 0.30 ^a
1	16.94 ± 0.12 ^a	13.18 ± 1.67 ^a	28.72 ± 1.12 ^a	11.92 ± 0.12 ^b
1.5	11.94 ± 0.90 ^b	12.03 ± 1.11 ^a	29.09 ± 4.10 ^a	11.87 ± 0.11 ^b
2	11.86 ± 0.99 ^b	10.94 ± 0.35 ^a	26.64 ± 0.33 ^a	10.70 ± 0.23 ^c

Remarks: The data are presented as mean ± standard deviation (n = 3). Different superscript letters in the same column showed a significant difference at the 5% level (p < 0.05).

Swelling power

The results of the one-way ANOVA in Table 2 show that variations in acetic acid concentration have a significant effect on the value of swelling power (p < 0.05). This suggests that increased acetic acid concentrations cause noticeable changes in the starch granules' ability to absorb water and expand. Tukey's follow-up tests confirmed that the 0–1% concentration treatment was not significantly different from each other, but was significantly different from the 1.5% and 2% treatments. Swelling power describes the ability of starch granules to absorb water and expand when exposed to water under heating or stirring conditions, reflecting the interaction between water and the molecular structure of starch [25]. The bubble power is affected by the structural composition of starch, specifically the interaction between amylose and amylopectin when given water and heat. The hydrogen bonds between starch molecules are broken so that water enters the granules and causes swelling [24,25].

In the results obtained, swelling power at the concentration variation of 0–1%, there is a relatively high value and no real difference, while at a higher concentration of 1.5% and 2%, there is a significant decrease in swelling power. Increase in numbers swelling power indicates that up to this concentration, the starch structure is still able to absorb water with a relatively stable capacity. The relatively stable value of swelling power at low concentrations indicates that the starch granule structure is still able to absorb water and expand well. Under these conditions,

acid treatment has not provided a significant disruption to the structural integrity of starch granules. However, at higher concentrations of acids, the decrease in swelling power is thought to be related to the occurrence of partial hydrolysis in the amorphous regions of starch that are more accessible to acid molecules [28]. These results are in line with several studies that report that acid treatment in starches can modify functional properties such as swelling power through changes in amorphous structure and reorganization of hydrogen bonds without producing new functional groups on the FTIR spectrum [26,27]. The decrease in bubble power at concentrations of 1.5% and 2% is thought to be related to the increasing influence of acid hydrolysis on the internal structure of starch. At higher concentrations of acids, the hydrolysis process tends to attack amorphous regions of starch that are more accessible to acid molecules, leading to partial breakage of polysaccharide chains as well as changes in the hydrogen bonds between starch molecules. Such changes can disrupt the structural integrity of starch granules and reduce their ability to retain water during heating. As a result, the ability of granules to expand optimally decreases, resulting in lower swelling power values at higher acid concentrations.

Solubility

The research data in Table 2 shows that the solubility value of starch is in the range of 9.74–13.18%. The results of the variance analysis (ANOVA) showed that the treatment did not have a significant effect ($p > 0.05$) on the observed parameters. These results indicate that the use of acetic acid up to a concentration of 2% has not had a significant impact on the resulting solubility properties, as shown by the similarity of the superscript notation in the follow-up test results. At the concentration of acetic acid 1%, there is a tendency to increase solubility, which is related to the increase in amylose release due to some disturbances in the amorphous region [24]. These changes occur because acid treatment can weaken the interaction of hydrogen bonds between starch molecules, making it easier for water to enter the granules and facilitating the release of some of the amylose, which contributes to increased starch solubility [31]. However, at a concentration of 1.5–2% solubility value decreased again, but did not make any real difference.

Decreased solubility at higher concentrations can be caused by the reorganization of starch structure, where the stronger influence of acid hydrolysis can cause changes in the molecular makeup of starch as well as increase interactions between polysaccharide chains. This condition has the potential to cause steric resistance and reduce the flexibility of the starch chain, so that the ability of granules to absorb water becomes more limited, and the dissolution process does not take place optimally [8]. The results showed that the addition of acetic acid up to a concentration of 2% only provided a small change or slight modification in the solubility of sweet potato starch.

Water Absorption

Based on the results in Table 2, the one-way ANOVA test at a significance level of 5% ($\alpha = 0.05$), the treatment of variations in acetic acid concentration did not show a significant difference in water absorption ($p > 0.05$), so no further tests were carried out in Turkey. This shows that statistically, changes in acetic acid concentrations have not had a significant effect on the starch's ability to bind water. Thus, although numerically there is a variation in water absorption values, the difference is not large enough to be statistically significant. In Table 2, water absorption shows a tendency to increase from a concentration of 0.5% to 1.5%, then decrease at a concentration of 2%. Theoretically, increased water absorption at low to moderate concentrations can be attributed to the occurrence of partial hydrolysis in amorphous areas of starch granules. Amorphous regions are known to be more susceptible to acid attack than crystalline regions, so mild acid treatment can open up internal structures without damaging the overall shape of the granules [20]. Increased water absorption at a concentration of 0.5-1.5% indicates an increase in the accessibility of hydroxyl groups to water molecules due to partial disruption of internal hydrogen bonds.

Research by (Singh et al., 2003) [32], stating that changes in amorphous structure can increase the ability of granules to interact with water due to increased intermolecular space. However, at a concentration of 2%, there is a decrease in water absorption. This suggests that at higher concentrations of acids, there is a more intensive molecular degradation occurring. Further hydrolysis can lead to the breaking of the chains of amylose and amylopectin, making the granule structure less stable in retaining water [24]. Increased acid concentrations can improve hydration properties in the early stages, but at higher concentrations, there is a decrease in water absorption due to further depolymerization.

Moisture Content

Based on the results of the moisture content analysis in Table 2, an average value was obtained that showed a decreasing tendency as the concentration of acetic acid increased, especially in the 2% treatment. The results of the one-way ANOVA analysis with a significance level of 5% showed that the variation in acetic acid concentration had a real effect on starch. Statistically, the value ($p < 0.05$) indicates a real difference between treatments. Turkey's follow-up test results showed that the concentrations of 0% and 0.5% were in the same group and did not differ markedly, as did the 1% and 1.5% treatments. Meanwhile, at the concentration of 2%, it is significantly different and produces the lowest moisture content compared to other treatments. These results were similarly reported by Polnaya and Lopulalan (2020) [28], which states that increased concentrations of acetic acid can affect the moisture content of starch through changes in the internal structure of starch that impact starch's ability to retain water.

Molecularly, acid treatment causes partial hydrolysis in starch granules, especially in amorphous regions that are more susceptible to acid attack than crystalline regions [20]. At low to moderate concentrations, the hydrolysis that occurs is still limited so that the granule structure remains relatively stable. However, at higher concentrations, it makes molecular degradation more intensive and can lead to a reduction in the number of free hydroxyl groups capable of interacting with water molecules [24]. In addition, the shortening of the amylose and amylopectin chains due to further acid hydrolysis resulted in a decrease in the degree of polymerization and the size of starch molecules, thereby decreasing the capacity of three-dimensional tissue formation that plays a role in water retention [33].

The shortening of the amylose and amylopectin chains due to acid hydrolysis leads to a reduction in the length of the polysaccharide chains, which play a role in the formation of water-binding structures [34]. Although in the early stages of treatment, there is an opening of granular structures that can increase interaction with water, the increase in acid concentrations actually leads to more intensive degradation of the structure. This condition reduces the starch's ability to form stable internal tissues in holding water molecules. As a result, the water absorption capacity decreases, or water is more easily released during the drying process.

FTIR Analysis

Fourier Transform Infrared (FTIR) analysis aims to identify changes in the molecular structure of starch after acetic acid addition treatment. The spectrum of treated and untreated starch FTIR is presented in Figure 2. The spectrum showed that both the control sample and the treatment sample had a typical absorption pattern of polysaccharides without the emergence of significant new bands. This indicates that the acetic acid treatment does not cause the formation of new functional groups such as carbonyl esters, so the modifications that occur are physical or in the form of internal structural disturbances, not permanent chemicals. These findings are in line with (Yadav et al., 2025) [35]. Acid hydrolysis at low concentrations generally only triggers structural reorganization in amorphous regions, as evidenced by the absence of the typical uptake of carbonyl ester groups (C=O) that usually appear at wave number $1700\text{--}1750\text{ cm}^{-1}$, which indicates that no chemical substitution reaction occurs during the modification process.

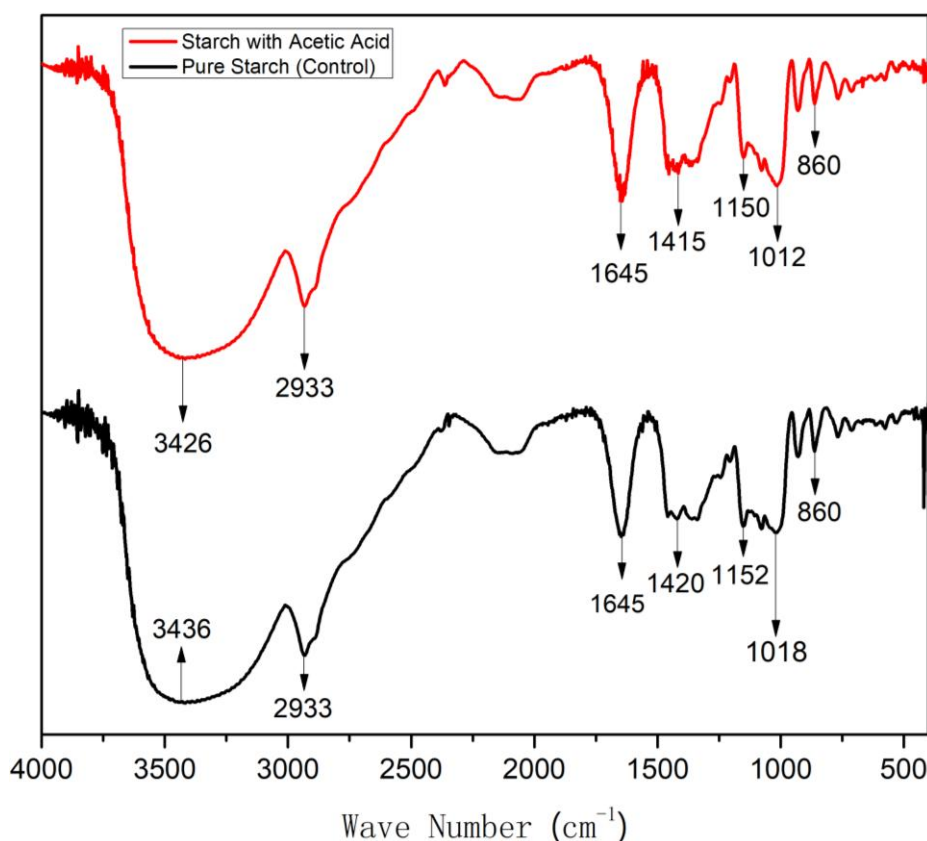


Figure 2. FTIR spectrum of sweet potato starch control and starch treated with acetic acid.

Absorption tape at 3200 range–3600 cm^{-1} shows hydroxyl group ($-\text{OH}$) strain vibrations reflecting the presence of hydrogen bonds in the starch matrix [36]. Absorption at wave number 2933 cm^{-1} associated with the strain vibration ($\text{C}-\text{H}$) of the glucopyranose ring, indicating that there was no significant shift between control and treatment [37]. This stability confirms that the hydrolysis that occurs is mild and does not damage the main structure of the polysaccharides. At 1645 cm^{-1} the wave number shows the vibration of water molecules adsorbed in the non-crystalline domain of starch [37]. Absorption tape at wave number 1150–1000 cm^{-1} related to vibration ($\text{C}-\text{O}-\text{C}$) and ($\text{C}-\text{O}$) of glycosidic bonds, showing changes especially around 1022 cm^{-1} . This region is sensitive to the degree of kriticalin starch. Absorption wavelengths 1047 cm^{-1} and 1022 cm^{-1} are often used as an indicator of the regularity of the crystal structure [38].

Spectrum showing wavelength 1420 cm^{-1} . About the vibration of bending CH_2 deformation ($\text{C}-\text{H}$), which is often associated with the degree of regularity of starch crystal structure. Changes in the intensity of these bands after acid treatment indicate a reorganization of the internal structure due to the disruption of hydrogen bonds [35,36]. Strong band at 1146–1150 cm^{-1} is a strain vibration ($\text{C}-\text{O}-\text{C}$) of glycosidic bonds a-1.4 that makes up the polysaccharide backbone [37]. At wavelength 860–930 cm^{-1} , relating to skeletal vibration bonds a-1.4 glycosides and anomeric structures of glucose, which are sensitive to changes in the degree of starch crystallinity [39].

CONCLUSION

Starch treatment with variations in acetic acid concentration affects the physicochemical characteristics of starch. The results of the ANOVA analysis prove that variations in acetic acid concentration have a significant effect on swelling power values and moisture content, but do not have a significant effect on water solubility and absorption. The results of the swelling power test increased to a concentration of 1%, then decreased to a concentration of 1.5%, which showed a change in starch granules due to acid treatment. The results of the moisture content test decreased as the concentration of acetic acid increased because acetic acid treatment can affect the availability of free hydroxyl groups in binding and retaining water. Meanwhile, water absorption and solubility values at variations in acetic acid concentration of up to 2% showed no significant difference. The results of the FTIR analysis showed that the acetic acid treatment did not produce a new functional group, because the spectrum pattern obtained was relatively similar to that of the control starch. However, there are changes in the value of swelling power and moisture content. This suggests that these changes in properties are not internal interactions, especially the weakening of hydrogen bonds between starch molecules. The weakening of this interaction increases the accessibility of water into the granule structure, thereby affecting the starch's ability to expand and retain water.

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Hopefully, the results of this research can make a useful scientific contribution to the development of chemistry, especially in the field of starch characterization and modification.

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