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Research article

PHYSICAL PROPERTIES IMPROVEMENT OF GADUNG (*Dioscorea hispida* Dennst.) STARCH OXIDIZED BY HYDROGEN PEROXIDE

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Abstract

Oxidation is chemical modification which alters and improves the properties of starch. This study investigated the effect of oxidation using hydrogen peroxide on Gadung starch towards its physical characteristics. The different concentrations of oxidant (0; 2; 4; 6 and 8%) were studied. The effect of oxidation were evaluated based on swelling power, solubility, freeze thaw stability, viscosity, and whiteness of the oxidized starch. A significant difference was observed on the value of swelling power, solubility, freeze-thaw stability, viscosity and whiteness of Gadung starch. H_2O_2 at 4% concentration showed the best swelling power with 263% increase than that of native starch. The highest solubility was shown by 8% H_2O_2 concentration which was 55.66 g/100g. The syneresis of 8% H_2O_2 was also the lowest, 11.67% which showed the highest freeze thaw stability. The viscosity has decreased to 94.3% compared to native starch at 8% H_2O_2. The whiteness of oxidized starch is increased along with increasing concentration of hydrogen peroxide that given.

1. Introduction

Gadung (*Dioscorea hispida* Dennst) tuber is an easy-growing vine in tropical climates such as in Indonesia, which also wildly grows in the forest. Gadung has very good nutrition with a starch content of 38.80%, with amylose and amylopectin portions of 12.42% and 87.58%, respectively (Santoso et al. 2015). Therefore, Gadung has been considered one of the staple foods in Indonesia. Despite the potential of Gadung being very high, the tuber is underutilized and have not been commercially important in the food industry (Kumoro et al. 2014). Part of the reason is due to its cyanide content, which is toxic; thus, it needs a special step of preparation to reduce it to an acceptable/safe level (Kumoro et al. 2016). The other reason is its derived product's poor characteristics, which limits the potential application. For instance, Gadung starch, as the main derivative, has a very low solubility of about 5.6 g /100 g (Subroto et al. 2024).

Native starch has many disadvantages, such as lack of heat resistance, too high viscosity, and a high tendency of retrogradation and syneresis (Hazarika and Sit 2016). Hence many researchers are motivated to modify native starch's properties in order to enhance its utilization. Modification of starch can be done either by physically, chemically and enzymatically. Among starch chemical modifications, oxidation is a popular technique. The technique includes reaction that occurs between starch and oxidizing agent under controlled temperature, pH, and time. Several benefits have been reported from starch oxidation, such as lower viscosity, high thermal stability, brighter color, and good binding ability (Moreno et al. 2017). Hydroxyl groups in the starch hydrocarbon chain, primarily at C-2, C-3, and C-6 positions, are converted to carbonyl and/or carboxyl groups by the oxidation process, thus inducing physical and chemical characteristics change (Ojogbo, Ogunsona, and Mekonnen 2020).

Conventional oxidation reactions usually use inorganic substances such as hypochlorite as oxidizing agents. However, with environmental concerns as a priority and also potential residue that may cause food safety concerns, hydrogen peroxide has gained much interest from researchers. Hydrogen peroxide is characterized by its low cost and easily decomposed; hence, it is safe for the water environment and leaves no harmful residues in food products (Kumoro et al., 2015). Oxidized starch prepared by hydrogen peroxide has already been investigated in many starchy material such as in wheat starch (Sun et al. 2017a), maize starch and sweet potato starch (Zhang et al. 2012). However, there has been no research on the effect of oxidation using hydrogen peroxide on the characteristic of Gadung starch. Therefore, this study aimed to improve Gadung starch's properties by the use of oxidation method and to evaluate the effect of oxidation using hydrogen peroxide on its swelling power, solubility, viscosity, freeze-thaw stability and whiteness.

2. Materials and methods

2.1. Starch Isolation

Gadung starch was isolated according to (Shofiyah et al. 2020) method with some modifications. Gadung tubers (obtained from farmer groups in Podorejo Mijen, Semarang, Indonesia) were cleaned, peeled, cut and soaked in 10% salt solution for 48 hours in order to remove the cyanides. Gadung was crushed with a blender and added with water at a ratio of 1:3. Gadung slurry was then filtered to produce a starch suspension. Starch suspension was precipitated for 24 hours. The precipitate was then separated and dried by oven at 45 ºC for 24 hours. The dried starch was passed through 100 mesh sieve.

2.2. Oxidation

The oxidation of Gadung starch was carried out based on (Sumardiono et al., 2021). Appropriate amount of $CuSO_4.5H_2O$ (MERCK, Germany) was added to 42% (w/w) starch suspension to achieve concentration of 0.04 g of Cu_2+/100 g starch. The mixture was stirred at 250 rpm (IKA RW 20 overhead stirrer, Germany) and heated for 15 min at 40 °C. Subsequently, 35% (w/w) solution of H_2O_2 (MERCK, Germany) was added dropwise in order to achieve the final H_2O_2 concentration of 0; 2; 4; 6 and 8% of dry starch (v/w). The reaction was continued for 45 min. The slurry was then washed with aquadest and filtered. After drying at 45 °C for 24 hour, the resulted starch was passed through 100 mesh sieve. The treatments were carried out in four repetitions.

2.3. Swelling Power and Solubility

Swelling power and solubility were evaluated according to (Kusumayanti, Handayani, and Santosa 2015) method with some modifications. Starch was dispersed in aquadest at the concentration of 0.5 g/100 g. The starch dispersion was heated to 95 °C and kept for 30 min under constant stirring. The resulted starch paste was immediately cooled in an iced water bath until it reached ambient temperature. A 10 g of suspension was prepared in a centrifuge tube and then centrifuged at 2300 rpm for 30 min (Hettich Zentrifugen, Germany). An aliquot of decanted supernatant was then dried in an oven at 110 °C for 4 h. Swelling power and solubility were calculated using below equations where W is the total weight of the suspension in the tube, W1 is the weight of dry tube, W2 is the weight of the tube after the supernatant was decanted and W3 is weight of the dried supernatant.

$$\text{Swelling power (g/g)} = \frac{W_2 - W_1}{W \times 0.5/100} \quad (1)$$

$$\text{Solubility (g/100g)} = \frac{W_3 \times 100}{W \times 0.5/100} \quad (2)$$

2.4. Swelling Power and Solubility

The measurement of freeze-thaw stability was based on the method described by (Siswo Sumardiono et al. 2022). Starch suspension at the concentration of 5 g/100 g was heated at 95 °C while constantly stirred for 30 min and then brought to room temperature in an iced water bath. A 10 g of resulting starch paste was put in a centrifuge tube and frozen at -14 °C for 24 hours. Frozen starch was then thawed at 30 °C for 1.5 hours followed by centrifugation at 2300 rpm for 30 min. The supernatant was decanted and weighed. Freeze-thaw stability is shown as % syneresis which calculated as follow:

$$\text{\% Syneresis} = \frac{\text{Supernatant (g) x 100\%}}{\text{Total weight of suspension in the tube (g)}} \quad (3)$$

2.5. Viscosity

The viscosity was determined as the Final Viscosity according to the method described by (Siswo Sumardiono et al. 2021) with some modifications. Starch in aquadest suspension (15 g/100 g) was prepared and heated at a temperature of 95 °C for 15 min. The viscosity was measured by viscometer (Rion VT-06, Japan) after the suspension was cooled to 50°C.

2.6. Whiteness

Whiteness was determined by digital colorimeter using CIE L*a*b colour scale. Whiteness was expressed as L (lightness) value which value ranges from 0 (black) to 100 (white).

2.7. Statistical Analysis

Data were statistically analyzed using Analysis of Variance (ANOVA) with 5% level of significance. Duncan Multiple Range Test (DMRT) was applied to any significant difference.

3. Results and discussions

3.1. Swelling Power

As can be seen in Fig 1, the swelling power of native and oxidized Gadung starch was significantly different ($P<0.05$). The swelling power of native starch (0%) showed increasing value by adding H_2O_2 at concentrations of 2% and 4%, followed by a decrease of 6% and 8%. Native Gadung starch had the lowest swelling power with 4.09 g/g, while the highest swelling power was found at an H_2O_2 concentration of 4%, which was 14.85 g/g, representing an increase of about 263%.

A similar scenario was reported by (Wang et al., 2003), where the swelling power of corn starch increased by 37.3% as the oxidant concentration was increased from 0.5% to 1% but further decreased at concentrations of 1.5% and 2% with a reduction of thepercentage of 25%. On the contrary, (Halal et al., 2015) found a decreasing swelling power in barley starch with increasing oxidant concentration. Research (Sun et al. 2017b) has also reported that the swelling power of wheat starch, which is oxidized by 12% hydrogen peroxide, was decreased from the native one with a reduction rate of about 6.8%. Comparatively, the current study showed a much higher increase in swelling power at the optimum concentration than other similar studies.

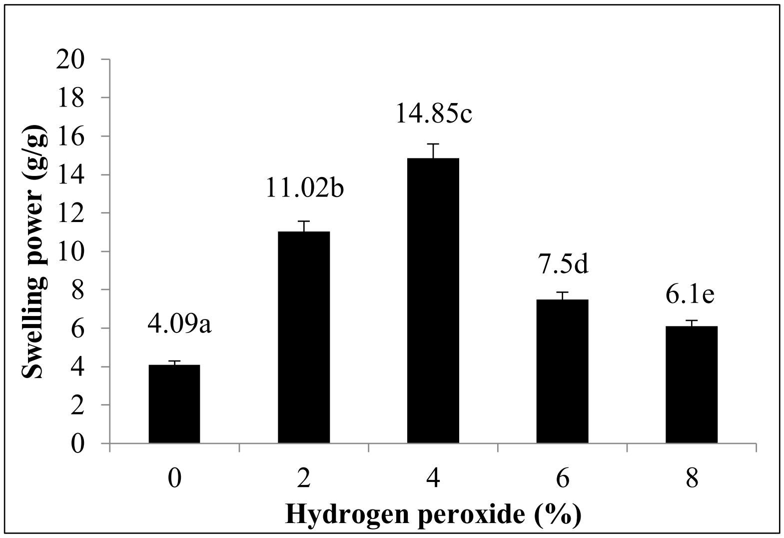

Figure 1. Swelling power of native and oxidized gadung starches.

Swelling power has been used to demonstrate differences among various types of starches because it indicates the starch's water-holding capacity (Pramana et al. 2024). In the current study, swelling power has reached the peak at the H_2O_2 concentration of 4% and then decreased. The increase can be explained by the fact that at the lower oxidant concentration, the oxidation reaction mainly affects the amylose fraction and results in the amylopectin: amylose ratio being higher. It is in agreement with (Vanier et al., 2017), who argued that amylose readily reacts with an oxidizing agent, possibly due to its linear structure, which makes it more susceptible to oxidative degradation. Therefore, less oxidizing agent was available for the oxidation of the amylopectin. Amylose depolymerization causes a decrease in the degree of crystallinity, and water molecules present in the system can be readily accessed by amylopectin molecules, thus causing an increase in starch swelling power (Cahyono et al., 2023).

The following trend of swelling power decrease at concentrations 6% and 8% was speculated to be caused by the amount of hydroxyl radicals in the system being too high. It resulted in the oxidation of hydroxyl groups from both amylose and amylopectin into carboxyl groups and cleaving the polymer into a shorter chain (Sumardiono et al. 2024) . The depolymerization of amylopectin molecules causes the starch to lose its ability to absorb water, hence decreasing its swelling power (Kumoro et al. 2020).

3.2. Solubility

Fig 2 shows that the oxidation treatments gave a significant ($P<0.05$) effect on the solubility of Gadung starch. The solubility of the Gadung starch increased with the increasing oxidant concentration, where the highest solubility was at an 8% concentration of H_2O_2 at 55.66 g/100g. The solubility enhancement from the native starch reached 351%. This value is higher when compared with an even higher oxidation level (12% H_2O_2) in wheat starch (Sun et al., 2017), which had a solubility increase of about 158%. A comparable result was reported by (Sun et al. 2017a), where a 391% solubility increase was found in corn starch oxidized by 12% H_2O_2 concentration.

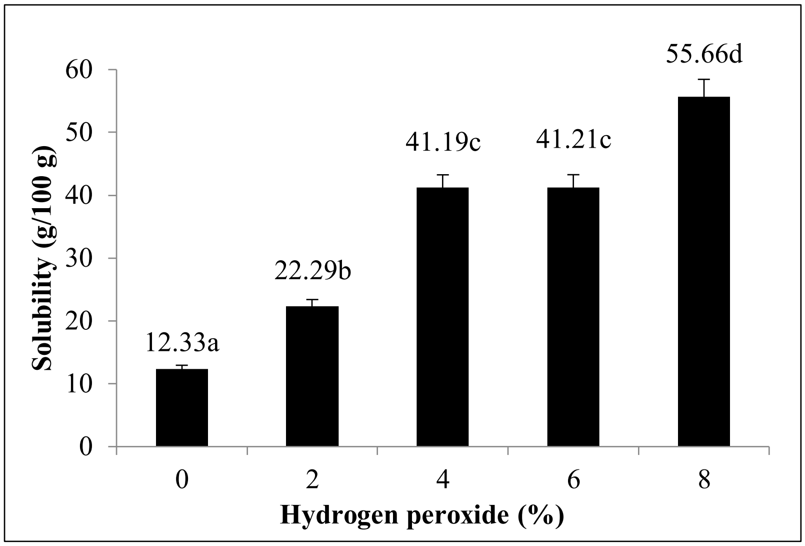

Figure 2. Solubility of native and oxidized Gadung starch.

Solubility indicates the percentage of starch molecules released after the swelling of starch granules (Rahma et al. 2017). The positive correlation between solubility and oxidant concentration has also been reported by (Halal et al. 2015) in barley starch, whose solubility increased substantially as the amount of oxidant (NaOCl) increased. The increased solubility is caused by the depolymerization of starch granules and the weakening of the starch structure by the oxidation process. According to (Fonseca et al. 2015), the formation of carboxyl groups lead to porous structures on starch granules and hence it could absorb water but could not hold it. The degradation of starch molecules increases the mobility of the starch molecules and makes the molecules interact easier with water, most probably due to the increased polarity (Sumardiono et al., 2017). High-soluble starch can be applied in the encapsulation of food products and also as a food additive to products that require high solubility (Sunarti, Pasaribu, and Winarti 2020).

3.3. Freeze-thaw Stability

Starch's freeze-thaw stability can be reflected in its syneresis, which is defined as the portion of water that separates out after freezing and thawing (Kumoro et al. 2014). This stability is particularly relevant to the potential application of modified starch in frozen foods, e.g., sausages, and meatballs. Therefore, in the current study, freeze-thaw stability is expressed as the magnitude (in percent) of syneresis. Lower % syneresis represents higher freeze-thaw stability.

As shown in Fig 3, the H_2O_2 oxidation had a significant effect ($P<0.05$) on the starch syneresis value. The percentage syneresis of oxidized starch decreased with increasing numbers of oxidants that were given. The highest syneresis was shown by native starch in the amount of 47.83%, while the lowest syneresis was shown in oxidant concentration of 8% with 11.67%, showing an approximate 75% reduction. This reduction percentage is higher than cross-linked oxidized corn starch at 12% oxidant concentration, which decreased syneresis from its native starch by about 55.2% (Arunyanart and Charoenrein 2008). Similarly, it is also higher than syneresis reduction in wheat starch oxidation (12% oxidant level), about 26.3% of its native starch (Sun et al. 2017a). The higher syneresis reduction at a lower oxidant level shows the effectiveness of H_2O_2 oxidation treatment in improving the Gadung starch's freeze-thaw stabili

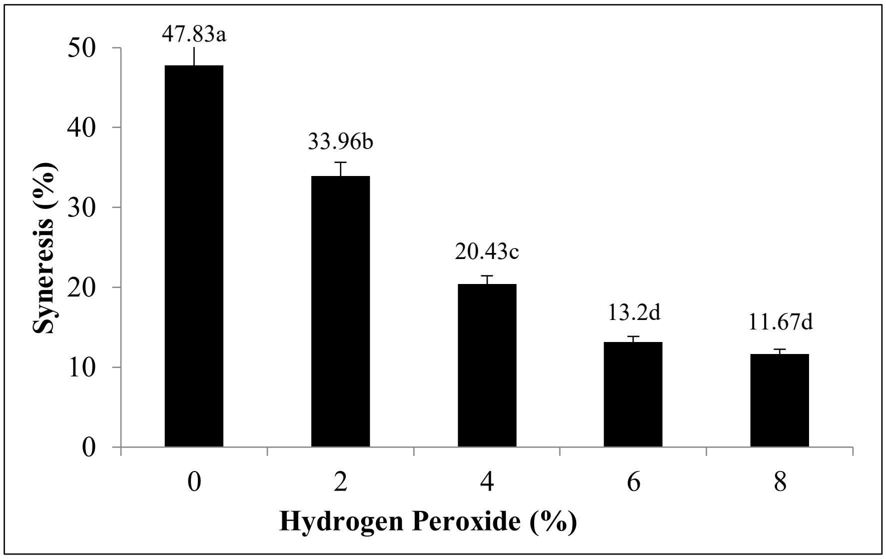

Figure 3. Freeze-thaw stability of native and oxidized Gadung starch.

The increasing freeze thaw stability can be attributed to the formation of carboxyl groups in oxidation reaction. Higher oxidant concentration results in more carboxyl groups and thus produces higher freeze thaw stability (Sumardiono et al., 2021). Carboxyl and carbonyl functional groups reduce the syneresis of starch because the carboxyl group has a strong hydrophilicity that prevents the release of water and improves water-holding capacity (Wardhani et al. 2023).

3.4. Viscosity

The viscosity, expressed as final viscosity, was measured at the starch paste's temperature of 50°C after its complete gelatinization at 95 °C heating condition. The simulated setup can illustrate the possible application of the oxidized starch, for instance, as an added substance in instant soup or beverage powder. Typical preparation of such products may include dilution with hot water, to induce gelatinization, prior to consumption which normally done at milder temperature.

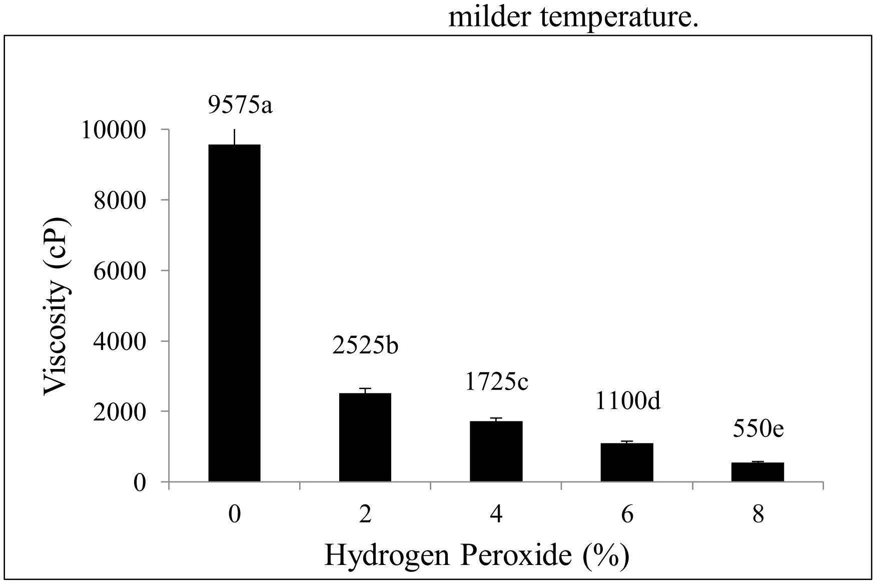

Figure 4. Viscosity of native and oxidized Gadung starch.

As can be seen in Fig 4, H_2O_2 oxidation treatment gave significant effect ($p<0.05$) to Gadung starch final viscosity. Starch paste viscosity decreased with increasing oxidant level. The highest starch paste viscosity was shown by native starch that was 9575 cP, while the lowest viscosity was resulted by oxidized starch with the highest oxidant concentration which was 550 cP, hence showing a reduction percentage of 94.3%. This reduction value is greater than that of oxidized wheat starch, which has decreased by about 89.2% from its native starch (Sun et al. 2017a). It is also higher than that reported by (Sandhu et al., 2008) where the final viscosity of oxidized corn starch has lowered about 32% from its native starch.

Viscosity is influenced by the ability of starch to be hydrated and its water-binding capacity (Pratama et al. 2018). Oxidation may cause disintegration of starch glycosidic bond resulting in loss of water-binding capacity, so that the viscosity of starch decreases (Sugiharto 2023). Furthermore, (Vanier et al. 2017) argued that the presence of carboxyl groups when compared with the hydroxyl groups would weaken the structure of the starch granules and contribute to lower the viscosity of starch.

3.6. Whiteness

The L value could be used as a parameter for characterizing starch color, and it is a direct measurement of its whiteness (Pramono et al. 2021). A higher L value (closer to 100) indicates a whiter material. Fig 5 shows that the whiteness of oxidized Gadung starch increased significantly ($p<0.05$) as the oxidant concentration increased. The highest L value was 83.89, shown by the highest oxidant level (8%). It represents an increase of about 4.71% from the native Gadung starch. The result is in accordance with (Vanier et al. 2017) study which reported an increase of 1.2% whiteness in oxidized potato starch if compared with its native starch.

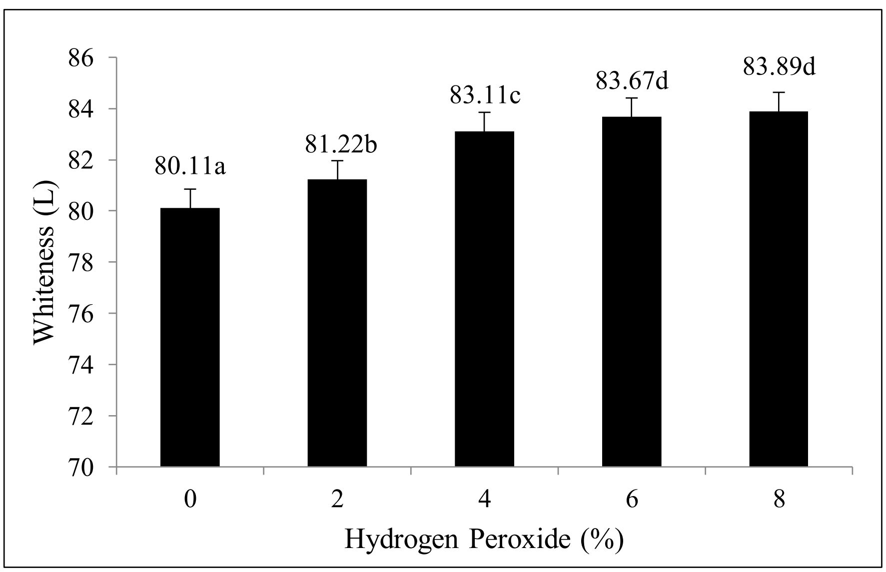

Figure 5. Whiteness of native and oxidized Gadung starch.

Oxidizing agents, which are generally used in starch modification, such as hydrogen peroxide or sodium hypochlorite, could function as bleaching agents. A bleaching agent enhances the white colour by oxidizing the pigments except white. Research by (Argüello-García et al., 2014) stated that sodium hypochlorite increases the whiteness value of starch by oxidizing impurities such as carotene, xanthophyll, and other related pigments. Therefore, oxidizing/bleaching agent is useful to improve the starch color, which normally undergoes a browning reaction during the extraction and drying process (Al-Baarri, Legowo, and Widayat 2018).

4. Conclusions

It can be concluded that the difference oxidant concentration that given has a significant effect on the value of swelling power, solubility, freeze-thaw stability, viscosity and whiteness of Gadung starch. Giving an oxidant concentrations above 4% reduce the value of Gadung starch swelling power. Solubility, freeze-thaw stability and whiteness increased when the H_2O_2 concentration was increase. The higher concentration of oxidant that given resulted lower Gadung oxidized starch paste viscosity.

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