

## Modification of (*Dioscorea alata* L) starch with propylene oxide to make edible film

Miksusanti<sup>1</sup>, Herlina<sup>1</sup>, Wiwin<sup>1</sup>

<sup>1</sup>Chemistry Study Program, Faculty of Mathematics and Natural Science, University of Sriwijaya, Palembang 300062, Indonesia,

\*Corresponding author e-mail: [miksusalbi2000@yahoo.com](mailto:miksusalbi2000@yahoo.com)

### ABSTRACT

The research about modification uwi starch (*Dioscorea alata* L) by using propylene oxide has been done. Concentration of propylene oxide were 6%(v/w), 8%(v/w), and 10%(v/w). The amilograf parameter after modification were characteristic breakdown viscosity 43 BU and setback viscosity 975 BU. The modification starch has edible properties according to FDA (food and drug administration) which have degree of modification < 7%, degree of substitution < 0,1 and propylene oxide concentration < 10%(v/w). The best propylene oxide in making of edible film was 8% (v/w). The starch control can be made into edible film with thickness 0,136 mm, tensile strength 20,4605 MPa and elongation 22%. Modification starch of uwi can be made into edible film with thickness 0,146 mm, tensile strength 25,3521 Mpa, elongation 30% and water vapor transmission 7,2651 g/m<sup>2</sup>/24 hours. FTIR characterization of uwi starch showed the occurrence of hydroxypropylation. The peak spectrum at 2900 cm<sup>-1</sup> showed bonding of C-H from methyl group, which is characteristic for modification starch with hydroxypropyl. Characterization with scanning electron microscopy showed that modification of uwi starch has turned the granule of starch to be fully swallon.

*Keywords:* Modification starch, uwi starch (*Dioscorea alata* L), propylene oxide, edible film

### 1. INTRODUCTION

Polysaccharides from tuber starch can be used as raw material for edible film making. Starch is often used in the food industry as a biodegradable plastic polymer film to replace undegradable plastic, because it is cheap, easily available, renewable and can provide good physical characteristics. Edible films made of polysaccharides have several advantages, namely either to protect the product against oxygen, carbon dioxide and lipids but very low water vapor resistance (Bourtoom, 2008). Uwi starch has the potential to be modified because the functional properties of this starch are acceptable as the film. Edible film from uwi starch has low resistance to water vapor. The chemical properties of uwi starch needs to be improved by modification.

Modified starch is mostly done in various ways to obtain starch with certain characteristics. Modifications can be carried out by crosslinking (Munarso *et al.*, 2004), hydroxypropylation (Febby *et al.* 2012), etherification (Dzulkefly *et al.*, 2007), and etherification (Aziz *et al.*, 2004; Choi and Kerr, 2003; Lawal, 2009). In this research modified uwi starch was conducted using propylene oxide to produce a lower gelatinization temperature, the optimum swallon of starch granule and resistant to retrogradation (Lawal, 2009). Uwi tuber is one food starch that has not been utilized in maximum way (Richana and Sunarti, 2004). This tuber contains

starch that include of amylose and amylopectin which can be used to create a thin layer film, to serve as an alternative material of edible filmmaking.

### 2. EXPERIMENTAL SECTION

#### 2.1. Modification of Uwi Starch

Concentration of propylene oxide (C<sub>3</sub>H<sub>6</sub>O) that was used were 0%, 6%, 8% and 10%. Modification procedures of uwi tuber starch were made in accordance with Lingfeng and Ya-Jane procedures (2000). 15 g of sodium sulfate (15% of the dry weight of uwi starch) was dissolved in 186 mL of deionized water in a 300 mL at room temperature, forming a solution of sodium sulfate. Uwi starch was weighed 100 g (dry weight) and then put in a solution of sodium sulfate with a magnetic stirrer to form a suspension. pH was set at 11.5 by the addition of 1% NaOH solution and stirred for 10 minutes.

Propylene oxide was added with variation 0%, 6%, 8% and 10% (v/w) of the dry weight of starch into the suspension. Flask was closed and the solution was stirred at room temperature for 1 hour before the temperature rose to 45 °C. Stirring was continued constantly using a dry incubator at a speed of 150 rpm for 24 hours and the pH was lowered to 5.5 using HCl 1 N. Suspension temperature was lowered to 30 °C. The suspension was filtered using whatman paper no. 4, while washed with distilled water for 5 times. Drying is carried out at temperature of 40 °C in an oven for 18 hours. Parameters observed for uwi starch modification are pattern of amilograf, modification levels and degree of substitution.

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**2.2. Modification levels JECFA 2009**

Levels of modified starch with propylene oxide performed using ninhydrin reagent. Ninhydrin reagent prepared from 3% solution of 1,2,3-triketohidrin crystals in a solution of 5% sodium bisulfite. Modified starch samples were weighed as much as 50-100 mg and put in a 100 mL volumetric flask, and were added 1 N sulfuric acid at 25 mL. Then unmodified starch samples were treated in the same way as the above treatment. The flask was placed into a water bath and then heated until the sample is solved, after that it was cooled and diluted with water to a volume of 100 mL samples as well as unmodified starch.

Solution of 1 mL were taken and put into a 25 mL test tube lids. The test tube immersed in cold water and then was dropped with 8 mL of concentrated sulfuric acid. The solution was mixed until homogeneous and the reaction tube is placed into the water bath for 3 minutes. After that it was transferred to an ice bath until the solution became cool. Ninhydrin was added by 0.6 mL through the tube and stirred. After that the tube was placed into a water bath at temperature of 25 °C for 100 min. Each test tube concentrated sulfuric acid is added until the volume reaches 25 mL and mix for several times.

3 ml of solution measured with spectrophotometer. Unmodified starch used as a reference and the absorbance was measured at 591 nm. Curve of calibration was made with standard solutions containing 10; 20; 30; 40 and 50 µg propylene glycol each mL. The calculation of hydroxypropyl as follows.

$$\%HP = \frac{K \times 0.7763 \times F}{w} \times 100$$

Where HP is hydroxypropyl, W is weight of sample (mg), F is force, and K is concentration of propylene glycol

**2.3. Determination of the substitution degree (Wurzburg 1989)**

Determination of the substitution degree of the modified starch can be calculated by using the calculation as shown below.

$$DS = \frac{162 \times \%hydroxypropyl / 58}{100 - (\frac{57}{58} \%hydroxypropyl)}$$

Where 162 is the molecular weight of glucose, 58 is the molecular weight of propylene oxide, and 57 is the molecular weight of propylene oxide-1 atom H

**2.4. Pattern Amilograf of Ravid Visco Analyzer**

Analysis of the rheological properties of samples performed by using RVA Newport Scientific Pty Master Tec. Ltd., Warriewood, Australia. RVA tool set using a modified standard 2 with basis

Table 1. Effect of Modification with Propylene Oxide on Amilograf Pattern of Uwi Starch

Concentration of propylene oxide (%)	Gelatinization Temperature		Viscosity		
	Initial (°C)	Maximum (°C)	Maximum (BU)	Breakdown (BU)	Set back (BU)
0	88 ± 0.36	90.7 ± 0.16	1946.5 ± 0.38	547 ± 0.41	977.5 ± 0.42
6	82.75 ± 0.27	90 ± 0.22	4197 ± 0.11	1407.5 ± 0.36	1608 ± 0.16
8	77.5 ± 0.15	82 ± 0.46	2407.5 ± 0.20	43 ± 0.28	975 ± 0.25
10	76.5 ± 0.21	85.5 ± 0.12	4379.5 ± 0.43	2377.5 ± 0.12	834 ± 0.43

weight samples 3,4 g and water content of 25,26 g. Distilled water was put into aluminum RVA canister and weighed with certain weight. Then the samples incorporated into the canister which had contained the distilled water and stirred using a stirrer until evenly mixed.

Canister contains samples that have been mounted on the RVA, was heated and cooled with constant stirring. Sample was heated to a temperature of 50 °C and was maintained for 1 minute. Samples were heated from 50 °C to 95 °C with a speed of 6 °C/minute, and a temperature of 95 °C and were maintained for 6 minutes. Sample was cooled to a temperature of 50 °C with 6 °C/minute, then maintained at 50 °C for 3 minutes. Measurement results with this tool include viscosity peak or peak viscosity, hot paste viscosity or trough viscosity, viscosity changes during heating or breakdown, cold paste viscosity, final viscosity and viscosity changes during cooling or setback.

**2.5. Edible Film of Modified Starch**

14 grams of uwi starch which has been modified with propylene oxide 8% (v / w) was dissolved in 230 mL of distilled water, stirred and filtered to obtain the starch solution, then heated at temperature of 73 °C. Glycerol and CMC 10% (w/w) was added slowly.

**2.6. Characteristic of Edible Film**

**2.6.1. Test of Film Thickness**

Film thickness was measured by using a micrometer to approximately 0,001 mm in five different places, one in the middle of the film and four others around the surface of the film. Measurements of the result in mm.

**2.6.2. Tensile strength and percent elongation**

Tensile strength and percent elongation (elongation to break) were measured with Tensile Testing Equipment. Prior to measurement, the film was conditioned indoor temperature 28°C, humidity (RH) of 50% for 48 hours. Instron grip separation Initial set at 50 mm, crosshead speed of 8.0 i. Sample mounted vertically on a clamp that will move in the opposite direction, so that the film will experience withdrawal until the end. The maximum tensile force (F) which is capable of being held by the film will be read in digital numbers. Percent elongation calculated from the change in the distance of film:

$$\text{Tensile Strength} = F/A$$

Where F is force (N) and A is large (mm<sup>2</sup>)

Table 2. Modification levels and degree of substitution

Concentration of Propylene Oxide (%)	Modification Levels (%)	Degree of Substitution
0	0	0
6	1.118±0.30	0.031±0.27
8	1.800 ±0.21	0.051±0.30
10	3.100 ±0.31	0.089±0.28

$$\text{Elongation (\%)} = \{ \Delta J (\text{cm}) / J_0 (\text{cm}) \} \times 100\%$$

### 2.6.3. Water Vapor Transmission Test ( Cup Method )

Water vapor transmission rate was measured by using a water vapor transmission rate tester. The film was measured previously conditioned at room temperature 28°C and humidity (RH) of 50% for 24 hours. The cup was weighed to the nearest 0.0001 g, then placed in a humidity chamber, closed the fan run. The cup was weighed every day at almost the same and determined weight gains (mg) and time (hours). Water vapor transmission rate value was calculated with the following equation: Moisture absorbing material was placed in the cup so that the surface is 3 mm of the film to be tested. The cup was placed part facing up. Film were put into the cup until cover all of the cup.

$$\text{LTUA} = 4,8 \times m / t \text{ (g/m}^2 \text{ / 24 hours)}$$

Where m is weight gain (mg per hour), t is time last weighing between 2 (hour)

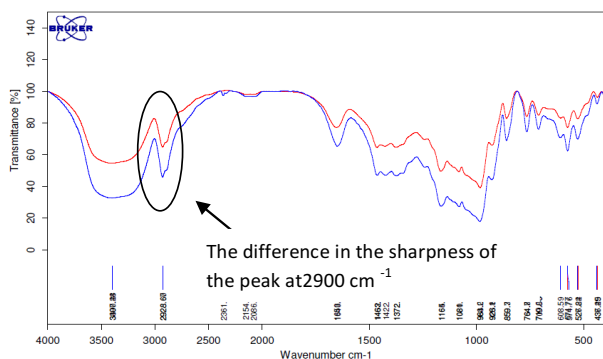
$\Delta J$  is the distance difference before and after drawn (cm),  $J_0$  is the distance between the two end clamps of the film before the drawn (cm)

## 3. RESULT AND DISCUSSION

### 3.1. Pattern of Amilograf

Amilograf starch pattern is determined from the ability of starch granules to swell and rupture, which causes swelling of the starch granules and increasing the viscosity of the suspension. Modified of uwi starch amilograf have a different pattern with uwi natural starch (starch controls).

Modified uwi starch have a decreased in initial gelatinization



- IR spectrum of uwi starch before modification
- IR spectrum of uwi starch after modification

Fig 1. IR spectrum of natural uwi starch and modification of uwi starch with the addition of Propylene Oxide 8%

temperature (SAG), decrease in peak gelatinization temperature (SPG), increase in the maximum viscosity and breakdown value (except for the addition of propylene oxide concentration 8%) and decrease in the value of setback (except for the addition of propylene oxide concentration 6%).

This amilograf pattern changes due to changes in the chemistry of starch granules due to the substitution reaction of hydroxyl groups of starch. According to Flence (1985), the modification of starch will change gelatinization characteristics, the viscosity, solid relationship, tendency of gel formation and dispersion of the hydrophilic properties of natural starch.

The effect of chemical substitution reaction on hydroxyl groups of starch and the physical properties of the resulting film of modified starches is better. Where a decline in water vapors and increase tensile strength and percent elongation of the edible film. Lawal (2009) also report that finger millet starch modification with propylene oxide decreased the initial temperature and the peak temperature of gelatinization. Finger millet starch modified using propylene oxide 10, 20, 30 and 40 mL produces initial gelatinization temperature of 57.2 °C; 56.1 °C; 55.0 °C and 54.9 °C lower than finger millet starch control of 63.6 °C. Gelatinization peak temperature also decreased from 66.9 °C; 68.8 °C; 64.8 °C; 64.5 °C lower than finger millet starch control (74, 1) °C. But instead of finger millet starch modification increases the viscosity of each peak 381 BU, 398 BU, 420 BU and 436 BU higher. The uwi starch that have been modification experienced the decline in the temperature of early gelatinizes and the temperature of the peak gelatinization. This was caused by the cluster hydroxyl that was change along the chain of the polymer especially amylose has been substituted with the cluster hydrokxipropil of the ether and caused the weakness of the internal tension of the structure of the granule of the starch, so as to reduce the strength of the association that caused the fast starch to experience the swelling.

The process of the modified uwi starch's swelling will cause the temperature gelatinization faster compared by the control starch. The decline in the temperature gelatinization of this starch will also influence the physical characteristics of the breakdown of the film and percent of elongation. Films from starch that has a low gelatinization temperature will produce the film with tensile strength and elongation percent better than the films of starch that has a high gelatinization temperature (Table 4). This happens because of the low gelatinization temperature will result in a more stable starch granules so that when made into a film will produce the film edible with compact and robust properties (Bourtoom, 2008). The increase in the viscosity after modification of the natural starch (the Table 1) was caused by the modification of the starch. This process facilitates penetration of water in the granule of the starch and weakened the structure of the granule of the starch. (Han et al, 2005) reported that the modification of the rice starch with propylene oxide showed the increase in the viscosity. Breakdown values obtained from starch amilograf results granule stability in the heating process. The stability of paste viscosity values calculated from the difference between the maximum value of viscosity at 95 °C.

The higher the level difference paste viscosity during the process shows that the starch is increasingly unstable. Table 1 shows that the higher of the maximum viscosity the value breakdown (stability pasta) greater. Choi and Kerr (2003) said that the modification of the starch with propylene the oxide also could reduce retrogradation the starch. Retrogradation the starch could be seen from the value setback. The table 1 showed that thought setback the modification starch with propylene the oxide was smaller compared by the natural starch.

Increasing in the concentration propylene the oxide 6% did

Table 3. Effect of Hydroxypropyl to Thickness of Edible Film

Edible Film of starch	Thickness (mm)
Natural Uwi starch	0.136±0.24
Modification Uwi starch	0.146±0.35

Table 4. Effect of Modified Uwi Starch on Tensile Strength and Percent Elongation Edible Film

Edible Film of starch	Tensile Strength (N/mm <sup>2</sup> )	Elongation (%)
Natural of Uwi Starch	20.460±0.23	22 ±0.15
Modification of Uwi Starch	25.352±0.31	30 ±0.26

Table 5. Effect of Starch Modification against Water Vapor Transmission Edible Films

Edible Film from Starch	Water Vapor Transmission (g/m <sup>2</sup> /24 hours)
Natural of Uwi Starch	100 ± 0.31
Modification of Uwi Starch	7.260 ± 0.42

not decline the setback compared with the natural starch. The increase of propylene oxide with the concentration 6% had the level substitution 0.03, so as the starch that had been hydroxypropylated still could not reduce retrogrades although reduced the temperature of early and the temperature of starch gelatinization.

### 3.2. Modification Levels and Degree of Substitution

The level of modification was the number of hydroxypropyl ether that reacted to the chain of the polymer amylose. Whereas the level of substitution (DS) was defined as the number in general of hydroxyl that was changed the polymer amylose. The functional compound that replaced from the hydroxyl in this case was hydroxypropyl of ether. Table 2 showed that the use propylene oxide according to FDA condition.

According to U.S. and Drug Administration (FDA) in modification of the starch the use propylene oxide not more than 10% with the level of modification < 7% and with level of substitution not more than 0.1. According to Aziz et al. (2004) the level of substitution (DS) had the high solubility and the increase of DS could improve the characteristics of hydrophobic of thermal starch. From the three treatments, the concentration 8% gave the smallest breakdown value. The modification of uwi starch with propylene oxide 10% had the value of substitution approached 0.1 so it cannot be used to produce edible film.

### 3.3. Identification of the IR Spectrum

Spectroscopy FTIR was used to confirm the occurrence of the modification of the starch. Yuliasih et al. (2007) said that Spectra FTIR the starch produced by the modification showed the relative pattern that had not been changed in the basic structure.

The change only happened in the intensity of the certain peak. According to Duanmu et al. (2007), the existence of the peak that emerged to the area 2900 cm<sup>-1</sup> indicate the existence of the association of C-H from the cluster methyl that was characteristic for the modification group hydroxypropyl. Fig 1 showed the FTIR spectra of natural uwi starch and modified uwi starch.

### 3.4. Characterization Of Edible Film

Characterization edible film needs to be done in order to know the quality of edible film to apply as a food wrap. Characterization had been done to edible film of natural and modified starch with the addition of propylene oxide 8% (v / w). Parameters that can be used to see the quality of the food packaging is among others thickness, tensile strength and percent elongation of the film edible also water vapor transmission.

The parameter is formed from the natural starch uwi broke because amylose content of the starch is not too high is only ± 22%. While the film of starch modification produces films that are not broken and has better characteristics by increasing thickness, tensile strength, percent elongation and reduction of water vapor transmission. Amylose is one of the compiler molecules of the starch where it can be used in the making of the film and a strong gel. High amylose will make the film becomes more compact because amylose is responsible for the formation of the film matrix. According to Bourtoom (2008) that making an edible film of the high amylose corn starch (75%) would be better than making edible film of normal corn starch (25%).

### 3.5. Thickness of Edible Film

Thickness of edible film is a physical trait which is affected by the concentration of dissolved solids in the solution of the film and the size of the glass plate.

Table 3 shows that the modification edible film of the starch has higher thickness than hydroxypropyl edible film where 0.136 mm becomes 0.146 mm. Emmanuel (2005) reported that the increasing of modification sago starch by crosslinking from 0.126 mm becomes 0.145 mm; 0.172 mm and 0.139 mm with variation of POCl<sub>3</sub>. Modified starch help starch granules swallow rapidly. This process causes the change in viscosity of the starch and made the film thickener.

### 3.6. Tensile Strength and Percent Elongation of Edible Film

Tensile strength is the maximum tensile force which can be held on the film until disconnected. Great tensile strength value means quality packaging films as well as good. Film edible with small tensile strength values indicate that the film is less well used as packaging for its physical characteristics are less robust and easily broken.

Elongation is the maximum length change before the film edible disconnected. Percent elongation showed the film's ability to stretch to the maximum. Edible films with low elongation values indicate that the film is rigid and brittle.

Table 4 shows that an increase in the tensile strength of the film edible natural starch and modified uwi starch are 25.3521; 20.4605 MPa and elongation of the film also increase from 22% to 31%. This occurs because propyl ether groups in the starch molecules through modification reaction can react as an internal plasticizer that can increase the flexibility of the film (Wuzburg, 1989).

### 3.7. Water Vapor Transmission of Films Edible

Permeability is one important factor in food packaging, because it is closely related to the shelf life of food products. Permeability values serve to estimate the shelf life of products packaged and to determine the appropriate foodstuffs packaged.

Table 5 shows a decline in water vapor transmission films made from natural uwi starch than films made from modified uwi



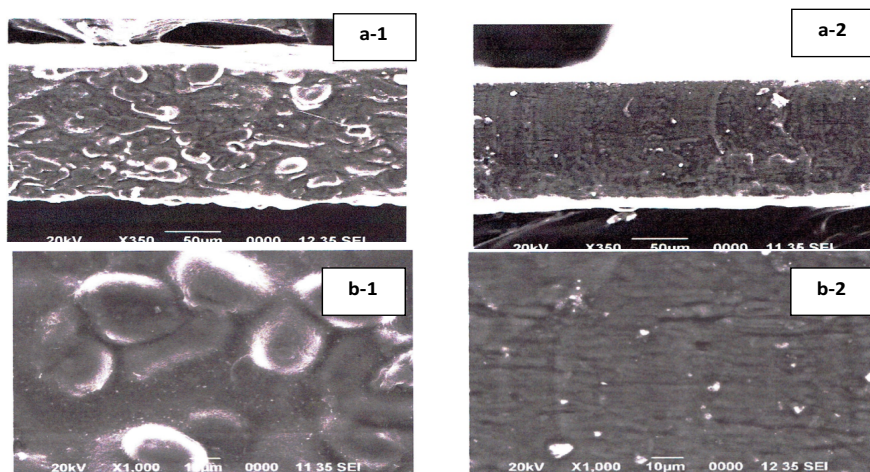


Fig 2. Scanning electron microscope images of transversal and surface section of films made from uwi starch (a-1;a-2 for uwi starch control, and b-1; b-2 for modified uwi starch with propylene oxide)

starch of  $100 \text{ g} / \text{m}^2 / 24 \text{ hours}$  to  $7.2651 \text{ g} / \text{m}^2 / 24 \text{ hours}$ . Edible films made from natural uwi starch have water vapor transmission of  $100 \text{ g} / \text{m}^2 / 24 \text{ hours}$ . Water vapor transmission of this film was large, indicates the film is very easy to absorb moisture.

Water vapor transmission of edible film from modification starch was smaller than the natural starch films due to the replacement of OH groups on the glucose units with a group hydroxypropyl ether leads to reduced the ability to bind water. DetdangchanN et al (2014) stated that due to the hydrophilic nature of the chemical structure of starch is widely available with hydroxyl group (OH) which has the ability to bind water. In replacing, the hydroxyl group with Hydroxypropyl ether made polymer matrix become denser due the increase in molecular weight of Hydroxypropyl ether. Yuliasih et al (2007) suggest that modification of starch can make the arrangement polymer matrix denser so that the water vapor transmission rate is lowered.

### 3.8. Morphology of Edible Film From Natural and Modified Uwi Starch by SEM

Film edible from natural and modified uwi starch was carried out through process of gelatinization. Gelatinization temperature recorded  $77^\circ\text{C}$ . Based on the results of the scanning electron microscopy (SEM) the film control shows still have starch granules. At this temperature natural starch have not swallon completely. Edible film from modified starch look smooth and no longer contain starch granules. This indicates that at temperature of  $77^\circ\text{C}$  all modified starch granule completely swollen. This phenomenon is supported by the data of tensile strength and percent elongation of the film. Control starch films have tensile strength values  $20,40 \text{ N} / \text{mm}^2$  while the modified starch film has a tensile strength of  $25.30 \text{ N} / \text{mm}^2$ . In natural starch film, gelatinization process is incomplete and caused polysaccharide chain still wrapped tightly in starch granules so the tensile strength becomes weaker than modified starch films. It is also strengthened by the data of percent elongation from modified starch films. It has greater elongation than natural starch film. Based on morphology form SEM, it can be explained that water vapor transmission rate of the control starch film ( $100 \text{ g} / \text{m}^2 / 24 \text{ h}$ ) is greater than the modified starch films ( $7.26 \text{ g} / \text{m}^2 / 24 \text{ h}$ ). The natural starch granules were pack in film randomly causing a gap between starch granules and it is easy to absorb water molecule.

## 4. CONCLUSION

Natural Uwi starch has the characteristics with initial gelatinization temperature  $88^\circ\text{C}$ , maximum Gelatinization temperature  $90^\circ\text{C}$ , maximum viscosity 1946.5, viscosity of breakdown 547, and viscosity of setback 977.5. Uwi starch which has been modified by the addition of propylene oxide with various concentration of 6%, 8% and 10% showed decrease in gelatinization temperature and increase of maximum viscosity. Modified starch by the addition of propylene oxide 8% (v / w) has the effect that can improve characteristics of edible film where an increase in mechanical properties such as the thickness from 0.136 mm to 0.146 mm, tensile strength from 25.3521 to 20.4605 MPa, percent elongation from 22% to 31% and decreased permeability of water vapor transmission from  $100 \text{ g} / \text{m}^2 / 24 \text{ hours}$  to  $7.2651 \text{ g} / \text{m}^2 / 24 \text{ h}$  compared to unmodified starch.

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