



Effect of heat-moisture treatment on the physicochemical properties of native canistel starch

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Abstract

The modification process in starch can improve starch characteristics and expand its application into food products. The objective of this research was to study the effect of heat moisture treatment (HMT) modification on the properties of new canistel starch and study their potential application to food products. The research methods were isolating the native starch and modifying it using HMT modification. Pasting profile, moisture content, starch and amylose content, yield, color, granule morphology, and crystallinity properties of native and HMT canistel starches were analyzed. Statistical analysis showed that HMT modification increased the initial and peak temperatures of gelatinization, peak time, trough viscosity, final viscosity, and setback value. However, it decreased peak viscosity and breakdown levels. In addition, this modification significantly reduced the yield, brightness, and amylose content of canistel starch. HMT modification made a more tenuous structure than native starch, however it did not change spherical shape and small size of the granules. Also, HMT-modified canistel starch had higher crystallinity degree than native starch. Based on these obtained properties, native canistel starch was suitable for frozen food, whereas HMT starch can be applied in noodle processing and as a thickener.

Keywords: *Pouteria campechiana*; gelatinization; modified starch; morphology structure; crystalline property.

Practical Application: This study provides a new canistel starch using heat-moisture treatment modification on the physicochemical properties of canistel starch. Its use can be as non-gluten raw material in the starch-based foods processing. In addition, this study provides a method for utilizing canistel fruit and extending its shelf life significantly by turning it into starch.

1 Introduction

Canistel (*Pouteria campechiana*) is a fruit originating from Mexico and Central America which is included in sapodilla (Costa et al., 2010). Canistel fruit is usually consumed directly or used as a mixture in making cookies, biscuits, jams, lunkhead, and fruit juice. Also, canistel fruit has also been converted into flour (Pertiwi et al., 2020a, b) and has been applied to several products including wet noodles (Aminullah et al., 2020) and steamed brownies (Pertiwi et al., 2018). The application of non-gluten flour, such as in the manufacture of noodles, is very dependent on the ability of the starch to produce a compact and robust network (Muhandri et al., 2011).

Starch is a polysaccharide which is widely used in the food industry for various purposes. There are different sources of starch in plants, including tubers (potatoes, cassava, sweet potatoes), seeds (wheat, corn, rice), stems (sago), and fruit (banana, breadfruit). Several studies of native starch from fruits have been carried out, namely banana starch (Waliszewski et al., 2003; Rodriguez-Marín et al., 2010; Zhang & Hamaker, 2012), lindur starch (Jacoeb et al., 2014), breadfruit starch (Marta et al., 2019), sweetsop and soursop starch (Nwokocha & Williams, 2009), jackfruit starch (Kittipongpatana & Kittipongpatana, 2011), pumpkin starch (Stevenson, 2003), kiwi starch (Li & Zhu, 2017; Stevenson et al., 2006), and avocado starch (Builders et al., 2010). According to Fortuna et al. (2001), native starch has limited use

for application in various products in the food industry because of its characteristics which tend to be insoluble in cold water, require a long time to cook, and has low stability. Therefore, starch modification is needed to improve its utilization and characteristics for the desired products. Neelam et al. (2012) reported that heat-moisture treatment (HMT) could reduce granule swelling and peak viscosity as well as increase thermal stability. Heat-moisture treatment (HMT) was defined as a physical method that involves heat treatment at temperatures above the gelatinization temperature (80-120 °C) with limited moisture content or less than 35% (Collado et al., 2001). This treatment can change the physicochemical properties of starch granules such as the crystal structure, swelling power capacity, gelatinization, paste properties, and retrogradation (Hoover, 2010; Hormdok & Noomhorm, 2007; Jyothi et al., 2010). This HMT starch was suitable for product applications that are resistant to heat, mechanical treatment, and acidic condition such as in the manufacture of noodles, thickeners and textures in soups, sauces, dairy products, and bread (Taggart, 2004). Also, He et al. (2021) reported the drying treatment on canistel starch for its structural and physicochemical properties. Research on native and heat moisture treated canistel starches has only been slightly documented and it would be a valuable science and knowledge about fruit starch.

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This study aims to study the effect of HMT modification on native canistel starch on the pasting profile, physicochemical, and crystallinity properties and to determine its potential to be applied to food products based on the gelatinization properties obtained.

2 Materials and methods

2.1 Isolation of native canistel starch (Gbadamosi & Oladeji, 2013)

10 kg of fully ripe canistel fruit (Padalarang of Bandung, West Java, Indonesia) was peeled and split, the seeds separated from the fruit flesh, and washed thoroughly. Then the resulting 6 kg of canistel flesh was grated and mixed with water in a ratio of 1:5 (one part fruit, five parts water). The mixture was stirred and then filtered using a filter cloth, the filtrate was then held in a container for 3 hours. After the starch settled, the supernatant water was removed. This washing was repeated until the supernatant water was clear, after which the sediment was taken. The sediment of starch was dried in an electric food dehydrator type MKS-DR10 (manufacturer of PT Toko Mesin Maksindo, Indonesia) at 50 °C for 17 hours and then cooled. Dry starch was mashed using a disc mill type FFC-15 (Shandong-Jimo Agricultural Machinery, China) and sieved using a 100 mesh sieve to obtain a fine starch.

2.2 Heat moisture treatment modification of canistel starch (Collado et al., 2001)

The native canistel starch sample was adjusted to a moisture content of 28% by spraying distilled water (Sumber Kimia, Indonesia) and balanced in the URG-168SE type UCHIDA refrigerator (PT Maspion, Indonesia) at 5 °C for 12 hours to uniform the moisture content. The moisture-adjusted samples were placed in a foil-covered baking sheet and heated in an oven (Memmert, Germany) for 3 hours at 110 °C. The modified starch sample was then cooled to room temperature and dried at 50 °C in an electric food dehydrator type MKS-DR10, and dried for 8 hours. Starch was milled using a disc mill type FFC-15 and sifted for 100 mesh.

2.3 Pasting analysis (Collado et al., 2001)

A suspension of 3 g of starch in 25 g of distilled water underwent controlled heating and cooling cycle with constant stirring in the Rapid Visco Analyzer-StarchMaster2 (Pertent, Sweden). Samples were held at 50 °C for 1 minute, heated to 95 °C at 6 °C/min, then held for 5 minutes. After that, cooled to 50 °C at 6 °C/min, and held for 5 minutes. The following data were recorded as the parameter of pasting time from start to peak viscosity (Ptime); the temperature at the peak viscosity (Ptemp); peak viscosity (PV); hot paste viscosity/trough viscosity (HPV); breakdown (PV - HPV); cold paste viscosity/final viscosity (CPV); and setback. All tests were repeated twice.

2.4 Starch yield (Association of Official Analytical Chemists, 2005)

The starch yield was determined by calculating the weight of the starch produced by the weight of whole fruit, the weight of fruit flesh, and the weight of native starch based on Equation 1.

$$\%Yield = \frac{\text{weight of the starch (g)}}{\text{weight of whole fruit (g)}} \times 100\% \quad (1)$$

2.5 Color analysis (Hutchings, 1999)

Five grams of canistel starch sample was placed in a transparent container. CR-300 type chromameter (Konica Minolta Holdings Inc, Japan) was prepared and calibrated. After the standard was printed on the screen, the sample test can be carried out. The chromameter's light eye was attached as close as possible to the sample and illuminated using a tool, and then the value would be printed on the screen. The measurement parameters were the values of L, a, and b.

2.6 Moisture content (Association of Official Analytical Chemists, 2005)

Moisture content analysis was performed using the oven method. The porcelain cup was preheated at 100-105 °C for 30 minutes then cooled in a desiccator and weighed. The sample was weighed as much as 2 grams in a weighed dish, then heated in an oven for 6 hours at 100-105 °C. After that, the sample was cooled in a desiccator for 30 minutes and weighed. The determination of moisture content followed Equation 2.

$$\text{Moisture content (\%)} = \frac{A - B}{C} \times 100\% \quad (2)$$

where A was the mass of the container and sample before drying process, B was the mass of the container and sample after drying process, and C was the mass of wet sample.

2.7 Starch content (Badan Standardisasi Nasional, 2011)

Five grams of sample was weighed and put into an Erlenmeyer flask, then 200 mL of 3% HCl solution was added and boiled for 3 hours with an upright cooler. Then cooled and neutralized with 30% NaOH solution then 3% CH₃COOH was added for the acidic condition. The mixture was transferred to a 500 mL flask, and distilled water was added to the mark, then filtered. A 10 mL of filtrate was pipetted into a 500 mL Erlenmeyer, then 25 mL of Luff-Schoorl solution, some boiled stones, and 15 mL of distilled water were added. The mixture was boiled for 13 minutes, then cooled in an ice-filled tub. Then 15 mL of 20% KI solution and 25 mL of 25% H₂SO₄ were added slowly into the cold mixture, and then it was titrated immediately with 2 to 3 mL of 0.1 mol L⁻¹ Na₂S₂O₃ solution (V1) and did the work for blank (V2). A glucose weight table was used for calculating the glucose weight, which equivalent to the reduced CuSO₄·5H₂O. The amount of Na₂S₂O₃ needed to find the glucose weight in the table was the reduction of the blank titer volume (V2) with the sample titer volume (V1). Glucose level and starch content followed Equations 3 and 4, respectively.

$$\text{Glucose level (\%)} = \frac{W \times fp}{W_1} \quad (3)$$

$$\text{Starch content (\%)} = 0.90 \times \text{glucose level} \quad (4)$$

where W = sample weight (mg); W₁ = weight of glucose based on the table (mg); and fp = dilution factor.

2.8 Amylose content (Apriyantono et al., 1989)

A standard curve was made to determine the amylose content using spectrophotometry method. A 40 mg of pure amylose was put into a test tube, and 1 mL of ethanol 95% and 9 mL of NaOH 1 mol L⁻¹ were added into it. Then heated in boiling water for 10 minutes and cooled. The solution was pipetted as much as 1, 2, 3, 4 and 5 mL each into a 100 mL measuring flask. To each measuring flask, 0.2, 0.4, 0.6, 0.8, and 1 mL of 1 mol L⁻¹ acetic acid was added into each flask, and then 2 mL of iodine solution (1 gram of iodine and 10 grams of KI added with distilled water to reach a volume of 500 mL) was added to each. The mixture was fixed evenly and allowed to stand for 20 minutes. The intensity of the formed blue color was measured by a UV-Vis spectrophotometer at a wavelength of 625 nm. A standard curve was made by plotting the amylose content on the X-axis and the absorbance on the Y-axis. Then a linear equation, which was a relationship between them, was calculated.

The procedure for testing amylose levels in the sample is the same as for making a standard curve. 5 mL of the solution, which contained 100 mg of a sample with 1 mL of 95% ethanol and 9 mL of 1 mol L⁻¹ NaOH, was pipetted into a 100 mL flask, then 1 mL of 1 mol L⁻¹ acetic acid and 2 mL of iodine solution were added into it until to the mark. The amylose content was calculated using the obtained linear equation from the standard curve.

2.9 Morphology structure of starch (Pukkahuta et al., 2008)

The starch flour was placed on the sample holder using double-side insulation. The sample was coated with gold, then inserted into the SNE-4500M scanning electron microscope (SEM) instrument (SEC, Korea). The starch structure was observed on the monitor using a magnification scale of 1500 times with an acceleration voltage of 10 kV.

2.10 XRD (X-ray diffraction) analysis

X-ray diffraction measurements of starch were carried out using a XRD Shimadzu type XD61 diffractometer with Cu K α radiation (40 kV and 30 mA). The scanning regions of the diffraction angle (2 θ) were 5.0131-79.9711° at steps of 0.0260° and scan step time of 22.4400 s. Relative crystallinity was calculated by comparing the area of the crystalline peak with the total area (crystalline + amorphous).

2.11 FTIR (Fourier Transform Infrared) spectra analysis

The FTIR spectra of native and heat moisture treated canistel starches were measured using a FTIR Nicolet i5 spectrometer Thermo Fischer (Thermo Fischer Inc.). The KBr pellet method was used for the sample preparation. The spectra, recorded against an empty cell as the background, were acquired at wavelength between 4000-600 cm⁻¹ with a 36 times scan.

2.12 Statistical analysis

The SPSS® ver 25 programs via One Way ANOVA and Duncan's post hoc test with α of 5% was used for analyzing the

data in this research. ImageJ software and Microsoft Excel® were used to analyze starch granule shape, size, and distribution. OMNIC® software was used to analyze the infrared spectra. While, HighScore Plus of PANalytical® combine with MAUD open free software and ICDD database were used in analyzing crystallinity properties of canistel starch.

3 Results and discussion

3.1 Physical and chemical properties of canistel starch

Canistel starch is analyzed for physical and chemical qualities, including yield, color, moisture content, starch content, and amylose content. The results of the physicochemical analysis of canistel starch are shown in Table 1.

Statistical analysis in Table 1 shows that HMT-modified starch has a lower yield value than native canistel starch. This lower yield due to additional processes from native starch, such as heating and sieving. Desrosier (1988) stated the drying temperature affected the water evaporation from the material so that it could reduce the yield. This low yield is similar to banana starch that was 5.54-5.93% per fruit weight (Palijama et al., 2020), although it is lower than avocado starch of 20.5% per weight of fresh fruit (Builders et al., 2010). HMT modification also reduces the brightness of canistel starch. Deka & Sit (2016) and Winarno (2004) reported that thermal modification or processing caused the Maillard reaction between reducing sugars from starch and amino groups in proteins, which could change color and aroma. Based on the resulted a and b values and substitute them to Equation 5,

$$^{\circ}\text{Hue} = \tan^{-1}(b/a) \quad (5)$$

the native and HMT-modified canistel starches have $^{\circ}\text{Hue}$ of 150.90° and 58.36° which refer to a yellow green and a yellow red color (Hutchings, 1999), respectively.

Native and HMT-modified canistel starch are categorized into high amylose starch, which have more than 25% (Suwannaporn et al., 2007). Table 1 shows that the HMT modification does not significantly affect starch content, but it can reduce amylose content compared to native starch. This reduction is in line

Table 1. Physical and chemical properties of canistel starch.

Parameter	Native	HMT modification
	Yield (%)	
Per whole fresh fruit	6.23 ^a	4.28 ^b
Per fruit flesh	8.47 ^a	6.41 ^b
Per native starch	100.00 ^a	88.83 ^b
	Color	
L	93.70 ^a	81.69 ^b
a	3.99	5.17
b	-2.22	8.39
Moisture content (%)	9.44 ^a	11.87 ^a
Starch content (%)	66.10 ^a	69.33 ^a
Amylose content (%)	36.14 ^a	29.52 ^b

Note: Different superscript letter on the same line indicates significantly different at $\alpha = 0.05$.

with Chung et al. (2009), Herawati et al. (2010), and Hoover & Vasanthan (1994). Hoover & Vasanthan (1994) and Hoover & Manuel (1996), explained that HMT modification could form an extra amylose-lipid complex which caused lower amylose content than native starch.

3.2 Granule morphology of canistel starch

Scanning using SEM instruments plays an important role in understanding of the starch granule structure and the changes that occur in modified starch. The starch granule can be seen more clearly in Figure 1.

Figure 1 shows that HMT-modified canistel starch tends to have larger cavities between the starch granules. Syamsir et al. (2012) reported the presence of cavities in the centre of the HMT-modified arrowroot starch granules. Pukkahuta et al. (2008) and Watcharatewinkul et al. (2009) also reported that HMT-modified corn and canna starches, respectively, showed a more tenuous appearance than native starch. This morphological structure strengthens the pasting profile. For example, Kartikasari et al. (2016) and Singh et al. (2004) explained that a denser arrangement of starch granules had higher peak viscosity and breakdown values. Pukkahuta et al. (2008) also reported that the HMT-modified maize starch had more tenuous morphology, lower peak viscosity, breakdown, and setback than native maize starch. Analysis of the size and shape of the granules can be seen in Figure 2 and Table 2.

Figure 2 and Table 2 show the size of the native and HMT-modified canistel starch granules ranging from 5-9.99 μm with a distribution percentage of 83% and 87% and average sizes of 7.53 μm and 7.60 μm , respectively. This data indicates that canistel starch is categorized into a small starch according to Lindeboom et al. (2004) and similar to passion fruit starch of 6.4-7.8 μm (Kwok et al., 1974). Small granules have a bigger superficial area that allows fast hydration that would enhance

the water uptake and increase the viscosity, swelling, and gelatinization capacity properties (Cornejo-Ramírez et al., 2018). Table 2 shows canistel starch granules roundness of 0.80-0.82 indicate a spherical shape, that is similar to pejobaye starch and pineapple starch (Jane et al., 1994). This roundness is higher than that of sweetsop and soursop starches reported by Nwokocha & Williams (2009). Table 2 also shows that HMT modification does not change the granule's size and shape. Many studies such as Kaur et al. (2006), Stute (1992), Hoover & Vasanthan (1994), Hoover & Manuel (1996), Gunaratne & Hoover (2002), Adebowale et al. (2005), Tattiyakul et al. (2006), Khunae et al. (2007), Pukkahuta et al. (2008), and Vermeylen et al. (2006) reported that HMT modification on starch did not lead to detectable morphological changes.

3.3 Pasting properties of canistel starches

The measurement of the pasting profile aims to determine the properties of canistel starch gelatinization during the cooking process. The data of canistel starch pasting profile can be seen in Table 3.

The initial pasting temperature is the temperature when the starch granules begin to absorb water which can be seen by starting to increase in viscosity (Lestari et al., 2015). The initial and peak temperatures for gelatinization of native canistel starch were 66.55 $^{\circ}\text{C}$ and 79.85 $^{\circ}\text{C}$, respectively. These results

Table 2. Size analysis of native and HMT-modified canistel starch granules.

Particle characteristics	Native starch	HMT Starch
Particle number	37	37
Maximum diameter (μm)	10.84	14.72
Minimum diameter (μm)	5.00	4.70
Average granule size (μm)	7.53	7.60
Roundness	0.82	0.80

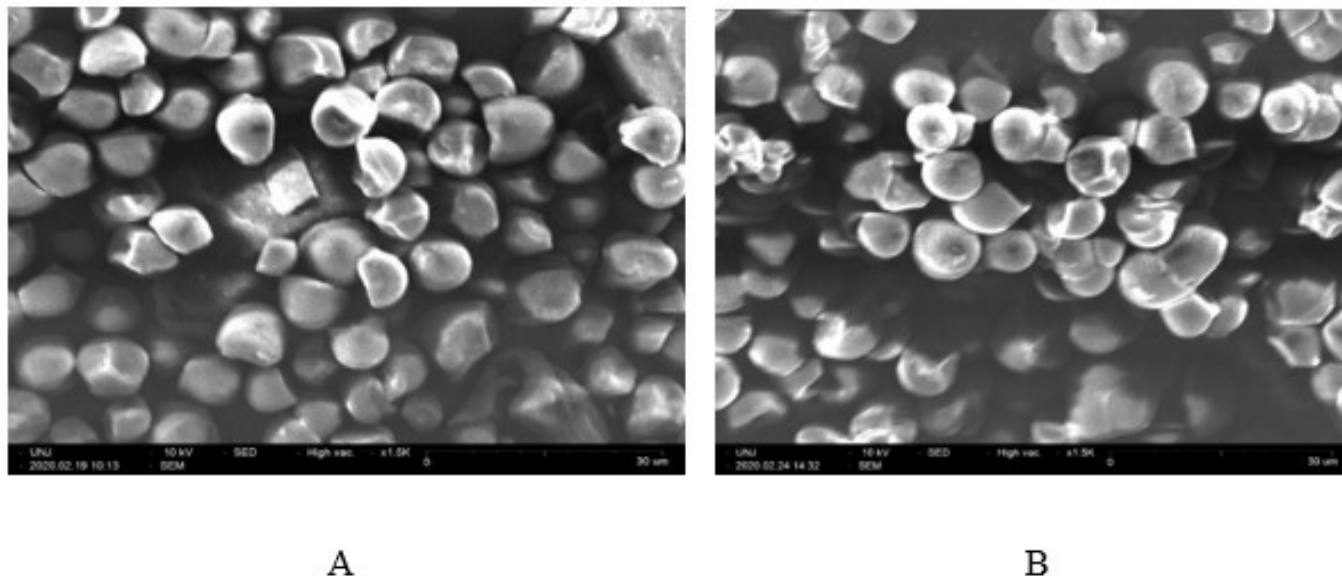


Figure 1. Morphology of native canistel (A) and HMT (B) starch under a Scanning Electron Microscope (SEM) at a magnification of 1500x.

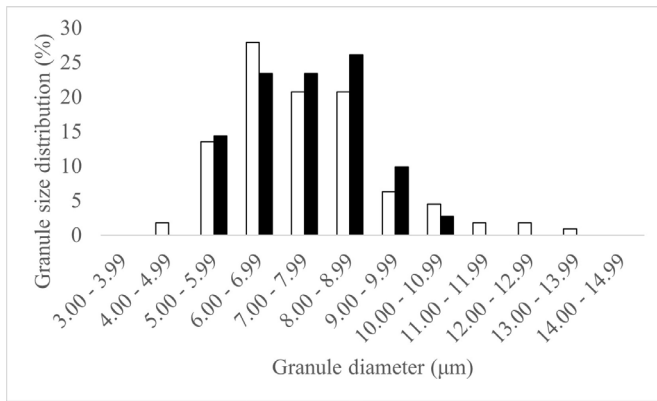


Figure 2. Diameter distribution of native (solid fill) and HMT-modified (no fill) canistel starch.

Table 3. Gelatinization profiles of native and HMT modified canistel starches.

Parameter	Native	HMT modification
Initial gelatinization temperature (°C)	66.6 ^a	68.2 ^b
Peak gelatinization temperature (°C)	79.9 ^a	94.8 ^b
Peak viscosity (cP)	3162 ^a	2052 ^b
Peak time (minute)	3.46 ^a	5.30 ^b
Trough viscosity (cP)	1223 ^a	1571 ^b
Final viscosity (cP)	2350 ^a	3318 ^b
Breakdown (cP)	1940 ^a	481 ^b
Setback (cP)	1127 ^a	1747 ^b

Note: Different superscript letter on the same line indicates significantly different at $\alpha = 0.05$. Initial and peak temperatures of gelatinization.

are similar to Stevenson (2003) and Zhang & Hamaker (2012) on pumpkin and banana starch, respectively. Table 3 shows that HMT modification can increase the initial and peak gelatinization temperatures of canistel starch. The higher initial temperature of gelatinization in HMT-modified starch is in line with several studies such as breadfruit starch (Marta et al., 2019), sago starch (Purwani et al., 2006), rice starch (Puncha-arnon & Uttapap, 2013), water chestnut starch (Yadav et al., 2013), and corn starch (Pukkahuta et al., 2008). Shafie et al. (2016) reported that the higher the pasting temperature indicates the potential for resistance to swelling in the material. Based on the data, it shows that HMT-modified starch is more resistant to heat and requires a higher temperature to gelatinize.

3.4 Peak viscosity

Table 3 shows that native canistel starch has a peak viscosity of 3162 cP. Several previous studies reported the peak viscosity of other native fruit starches, namely kiwi starch of 3000 cP (Stevenson et al., 2006), pumpkin starch of 2208-2688 cP (Stevenson, 2003), sweetsop starch of 6939 cP and soursop starch of 5147 cP (Nwokocha & Williams, 2009), breadfruit starch of 6607.67 cP (Marta et al., 2019), and banana starch of 2016 cP (Zhang & Hamaker, 2012). The statistical analysis shows that HMT-modified starch has a lower peak viscosity than native

starch that is about 2052 cP. This situation also occurred in breadfruit starch (Marta et al., 2019), sago starch (Purwani et al., 2006), sweet potato starch (Collado et al., 2001), sorghum starch (Olayinka et al., 2008), pearl millet starch (Balasubramanian et al., 2014), rice starch (Horndok & Noomhorm, 2007), corn starch (Pukkahuta et al., 2008), and water chestnut starch (Yadav et al., 2013). According to Yadav et al. (2013), low peak viscosity affected the limited swelling capacity, and this was due to reorganization in the granule, and Collado et al. (2001) stated that starch with limited swelling capacity was the ideal type of starch for making noodles.

3.5 Peak time

The peak time is the time when the Rapid Visco Analyzer (RVA) reads the maximum value of viscosity/peak gelatinization during the heating process (Kusnandar, 2011). The native canistel starch has a peak time of 3.5 minutes which is close to the peak time of sweetsop starch of 4.3 minutes and soursop starch of 4.69 minutes (Nwokocha & Williams, 2009) and breadfruit starch for 3.7 minutes (Marta et al., 2019). Table 3 shows that the HMT modification causes the starch to thicken more slowly to reach its peak viscosity compared to the peak time of native canistel starch. Collado et al. (2001) reported that HMT-modified sweet potato starch could increase the peak time of native starch from 7.6 minutes to 11.8 minutes. The HMT-modified sago starch (Pukkahuta & Varavinit, 2007) and water chestnut starch (Yadav et al., 2013) also have an increase in peak time from their native form. The shorter the peak time leads to the shorter the cooking time for the starch paste. These HMT-starch characteristics are suitable for increasing the viscosity of in soups and sauces that can provide sufficient viscosity at the beginning of the cooking process (Sitanggang et al., 2018).

3.6 Trough viscosity and breakdown

The trough viscosity value of the native canistel starch of 1223 cP is similar to that of pumpkin starch, which was 1242-1926 cP (Stevenson, 2003). Table 3 shows an increase in the trough viscosity value of HMT-modified starches. This was also reported in HMT-modified sago starch (Purwani et al., 2006) and sweet potato starch (Collado et al., 2001).

Breakdown is defined as the difference between peak viscosity and trough viscosity, which indicates the stability of the paste during the heating (Shafie et al., 2016). The native and HMT-modified canistel starches have breakdown values of 1940 cP and 481 cP, respectively. Pukkahuta et al. (2008) explained that the reduction in the breakdown was caused by the formation of the bonds between amylose and fat so that it can reduce granule swelling and can improve the stability of the paste during the heating. In addition, low breakdown indicates the ability to withstand a long period of heating and stirring (Lorlowhakarn & Naivikul, 2006). This breakdown reduction is also reported in sweet potato starch of 51 cP (Collado et al., 2001), modified rice starch of 158.04 cP (Horndok & Noomhorm, 2007), and corn starch of 161 cP (Chung et al., 2009). According to Beta et al. (2001), the lower the breakdown value led to the higher the hardness of the starch gel so that it can reduce the cooking

loss value. In making noodles, the low breakdown viscosity is expected so that the noodles have a low cooking loss value.

3.7 Setback

The statistical analysis shows that the HMT modification on canistel starch can increase the setback viscosity value from 1127 cP to 1747 cP. Yousif et al. (2012) explained that starch modification, including HMT, can increase the setback viscosity value of native starch. Changes in viscosity during cooling occur due to reassociation of amylose molecules, and low setback viscosity indicated a low rate of starch retrogradation (Shafie et al., 2016). This higher setback viscosity contributes to the higher cohesiveness and hardness of the paste after cooling (Sitanggang et al., 2018). Therefore, starch with a high setback allows it to be used as an ingredient in making noodles. By having higher cohesiveness and hardness, the noodles will have a lower cooking loss during production.

3.8 Final viscosity

Final viscosity refers to the viscosity of the fully gelatinized dispersion of starch after cooling of the resulting paste to a specific temperature. The statistical analysis shows that the HMT-modified starch has a higher final viscosity than the native canistel starch. This result is consistent with Purwani et al. (2006) and Collado et al. (2001), respectively, who reported an increase in the final viscosity of the native starch in sago starch and sweet potato starch, respectively.

Schoch & Maywald (1968) classified the viscosity patterns of the Brabender amylograph into four types, namely types A, B, C, and D. These different types will produce starches with different characteristics so that they will affect the application into food products. Based on the pasting profile, native canistel starch has high swelling properties due to its high peak viscosity, thickens quickly, less stable to heating, and short cooking time. This type of starch is suitable for use as a filler for soups and sauces, cakes, and it can also be used in the manufacture of frozen food because of its low setback value. Based on this profile, native canistel starch is included in B-type pasting starch, which was described by (Singh et al., 2005) stated that arrowroot starch with B-type pasting profile was characterized by low peak viscosity, breakdown and final viscosity. Stute (1992) reported that HMT modification resulted in decreased peak viscosity and breakdown, and increased final viscosity. HMT modification can increase the resistance of starch to heat, mechanical treatment, and acid by increasing the gelatinization temperature (Taggart, 2004). Based on pasting profile, HMT-modified canistel starch is a C-type pasting profile due to its lower peak viscosity so that it affects the limited swelling capacity. The applications of this starch include being used as raw material for noodles, thickening and texturing in soups, sauces, dairy products, and baked goods (bread and processed cakes).

3.9 Crystallinity of native and HMT canistel starches

XRD analysis of canistel starches

Native and HMT-modified canistel starches show a typical and similar X-ray pattern (Figure 3), with major peaks at $2\theta = 15.0^\circ$,

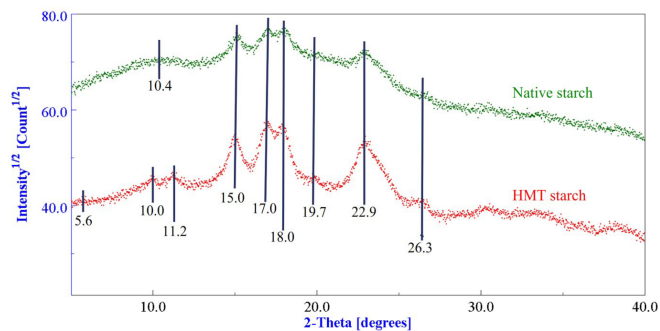


Figure 3. XRD pattern for native and HMT canistel starches.

17.0° , 18.0° and 22.9° with weak diffraction peaks at $2\theta = 10.4^\circ$, 19.7° , and 26.3° for native; and $2\theta = 5.6^\circ$, 10.0° , 11.2° , 19.7° , and 26.3° for HMT starch.

Jyothi et al. (2010) reported no significant difference between the XRD patterns for native and HMT modified starches. Also, these results are similar to several types of starch such as corn starch (Chung et al., 2009) and cassava starch (Jyothi et al., 2010), with the XRD pattern of HMT canistel starch similar to potato starch, which has a peak at $2\theta = 5.5^\circ$ (Manek et al., 2012). Zobel (1988) and Lim et al. (2001) explained that starch, which has a peak at $2\theta = 5.5^\circ$ with weak intensity, was classified as a B-type crystal. This result indicates that the HMT modification changes the starch's crystalline structure from A- to B- type.

In addition, there are peak intensities differences between the native and HMT modified canistel starches. Klein et al. (2013) and Deka & Sit (2016) observed a reduction in HMT-modified starch's peak intensities on rice and taro starches, respectively. This condition was also reported by Lim et al. (2001) for potato starch and Hoover & Vasanthan (1994) for yam starch. The intensities reduction on HMT of B-type starches has been explained by Hoover & Vasanthan (1994) due to the hydrate water bridges' rupture, which causes the adjacent double helices to move apart and assume orientations that are not in the perfect parallel crystalline array. Adjacent double helices in crystallites of B-type starches are mainly linked by hydrate water bridges and, to a limited extent, by direct hydrogen bonding (Leach et al., 1959).

The quantitative analysis on the XRD shows that the degree of crystallization in HMT canistel starch is higher (52.70%) than the native starch (47.80%). This was also reported by Vermeylen et al. (2006) for potato starch subjected to HMT at 130°C , which attributed to double helices' decoupling from the amylopectin backbone. This decoupling renders the double helices sufficiently mobile to become organized in perfect/larger crystallites (Vermeylen et al., 2006). Zheng et al. (2020), Cai et al. (2015), and Yoo & Jane (2002) reported that the starch crystallinity was inversely proportional to the amylose content. According to the previous analysis, native starch has higher amylose levels than HMT canistel starch (36.14% vs. 29.52%). Ao & Jane (2007) reported B-type starch granules to have low amylose content with high crystallinity degree than A-type crystal. This result also strengthens the formed peak at $2\theta = 5.6^\circ$ in HMT modified starch, which indicates the B-type crystal starch. According to Wu & Sarko (1978), the A- and

B-type crystalline starch granules were composed of parallel double helices in a hexagonal arrangement.

3.10 Infrared profiles of canistel starches

FTIR spectrometry is a sensitive method for studying starch structure changes in short-range orders (van Soest et al., 1995). Figure 4 shows 19 absorption peaks formed at wavenumbers of 4000 - 600 cm^{-1} with two major absorption peak regions.

The area at 3258 and 2929 cm^{-1} in Figure 4 are O-H and C-H bond stretching, respectively (Irudayaraj & Yang, 2002); and the region of fingerprint region at 1200-800 cm^{-1} . van Soest et al. (1995) and Cael et al. (1975) explained that the glucan ring vibration band was overlapped by COH stretching and bending vibration and the C-O-C glycoside bond vibration in the region of 1200-800 cm^{-1} . Absorption peaks of 1151, 1124, and 1104 cm^{-1} are assigned to CO and CC stretching with some COH contributions. Meanwhile, the peaks at 1078, 1042, 1014, 1000, and 929 cm^{-1} are subjected to COH bending and CH₂-related modes. Also, Karwasra et al. (2017) explained that the peak around 1015 cm^{-1} was due to the C-O of C-O-C in polysaccharides, and the peaks at 1081 and 1160 cm^{-1} were associated with anhydrous-glucose ring C-O stretch. In addition, Wang et al. (2010) and Miao et al. (2014) reported that peaks close to 930 cm^{-1} assigned to the skeletal mode vibration of α -(1-4) glycosidic linkage, as well as Cael et al. (1975) stated that a region of 862 cm^{-1} indicated the presence of COC stretching bands and CH deformation.

Apart from these two main areas, Lee et al. (2004) reported that the C-O-C stretching occurs at 1634 cm^{-1} , and C-H bending is exhibited at 1455 cm^{-1} . A single peak at about 1634 cm^{-1} is assigned to the tightly bound water present in starch due to its hygroscopic nature (Fang et al., 2002; Nzenguet et al., 2018). The peak at 1417 cm^{-1} is related to C-H bending of CH₂, and the peaks at 1204, 1243, and 1337 cm^{-1} are associated with O-H bending of primary or secondary alcohols. Meanwhile, the wavenumbers at 764 dan 709 cm^{-1} were attributed to the pyranoid ring (Karwasra et al., 2017). Overall, no significant differences in the native and HMT canistel starches' spectra pattern, and

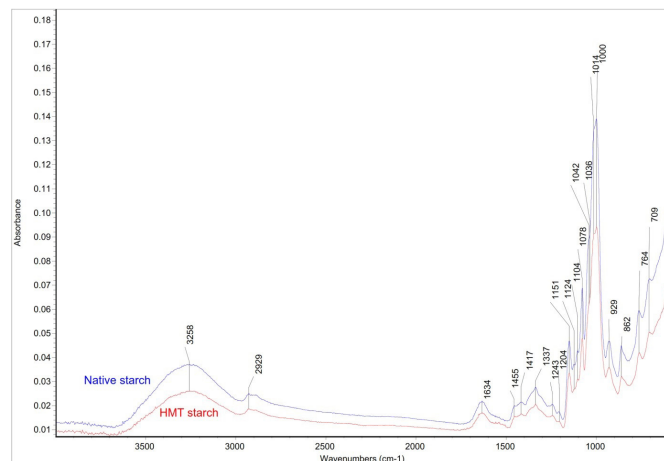


Figure 4. FTIR spectra of native and HMT-modified canistel starches.

there is no change in the functional group (Figure 4). However, a change in the absorption intensity occurs, which is related to the change in the crystalline phase and amorphous starch. Similar observations have been reported for maize, potato, and corn starches by Sui et al. (2015), Varatharajan et al. (2011), and Chung et al. (2009), respectively.

van Soest et al. (1995) also explained that FTIR spectroscopy had been applied to determine starch crystallinity. The IR absorbance band at 1047 cm^{-1} is sensitive to the crystalline structure; the band at 1022 cm^{-1} or 1016 cm^{-1} in another study (Sui et al., 2015) is related to the amorphous structure; the band at 994 cm^{-1} is associated to the intramolecular hydrogen bonding of the hydroxyl groups at C-6; and the valley at 1035 cm^{-1} is short-range order characteristic. The wavenumbers similar to these in the canistel starch spectra are 1042, 1014, 1000, and 1036 cm^{-1} . A lower ratio of 1042/1014 cm^{-1} in native canistel starch (0.666), than HMT starch (0.674), indicates native starch has a lower crystallinity. This ratio comparison strengthens the XRD analysis in which the native starch has lower degree of crystallinity than HMT-modified starch. In addition, native starch has a higher ratio of 1042/1036 cm^{-1} (0.989) indicates that it has a higher amount of short-range order than HMT canistel starch (0.969). This result is reinforced by higher absorbance intensity at 1000 cm^{-1} in native starch which contributed to a higher degree of double-helical order (short-range order) (Zhang et al., 2013).

4 Conclusion

HMT modification could increase the initial and peak temperatures of gelatinization, peak time, trough viscosity, setback, and final viscosity and decrease peak and breakdown viscosities. In addition, HMT-modified canistel starch had a lower yield, brightness, amylose content than the native starch. These starches were polygonal spherical with an average size of 7.53 - 7.60 μm with a roundness of 0.80. However, the HMT-modified starch had a more stretchable position between the granules than the native starch. Furthermore, HMT-modified canistel starch had a higher crystallinity degree and B-type crystalline structure than the native starch (A-type crystalline). Based on these properties, native canistel starch was included in the B-type paste, and A-type crystalline with a high swelling capacity thickens quickly, was less stable to heating, and had a short cooking time. On the other hand, HMT-modified starch was a C-type paste and B-type crystalline with a limited swelling capacity and resistance to heat and stirring.

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