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Effects of Heat/moisture Treatments on Physicochemical and Morphological Characteristics of Cassava Starch

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Abstract

This study investigates the influences of different heat/moisture treatments on amylose leaching (AML), resistant starch (RS) content, crystalline and morphological characteristics of cassava starch (CS): microwave heat/moisture treatment (MWT) and hot air oven heat/moisture treatment (HWT). CS with 35% of moisture content was subjected to MWT and HWT. Both treatments were performed at 60°C for 30 min. MWT- and HWT- treated CS were subsequently incubated at 4°C and 60°C for 24 h. They were then dried at 50°C for 24 h for further analyses. Native CS was used as a control. The results showed that MWT and HWT noticeably reduced AML in the temperature range 70-80ºC of treated CS, attributing to an increase interaction between amylose-amylose and/or amylose-amylopectin. In addition, RS content of MWT- and HWT-treated CS (all conditions) was significantly lower than that of native CS (control) (p<0.05). X-ray diffraction (XRD) revealed that total crystallinities (TC) of MWT- and HWT-treated CS followed by 4ºC and 60ºC incubation, respectively, were slightly increased compared with that of native CS (control), while A-type crystalline structure of MWT- and HWT-treated CS (all conditions) remained unchanged. Scanning electron microscopy (SEM) image showed significant changes in structure of starch granules after MWT. The result in this study indicates that MWT and HWT reduced the AML and RS values of CS. There was higher aggregation of starch granules in MWT-treated CS than occurred in HWT-treated CS.

Keywords: Cassava starch, Microwave heat/moisture treatment, Hot air oven heat/moisture treatment, Physicochemical and morphological characteristics

1 Introduction

Cassava starch produced from a cassava tuber is the most available starch in Thailand. Starch consists of two different structural polysaccharides; amylose (17-24%) and amylopectin (76-83%). Physiochemical properties of starch (i.e., digestibility, crystallinity, viscosity, swelling, and amylose leaching) are affected by heat processing (Osunsami et al., 1989; Charles et al. 2005). Heat/moisture treatment is a physical modification method that can alter properties of starch (Dupuis et al., 2014). Previous studies focused on the effects of heat/moisture treatments on the physicochemical and structural characteristics of starches. Specifically, microwave heat/moisture treatment (MWT) was used to treat Canna edulis Ker starch (Zhang et al., 2009), and waxy and non-waxy rice starches (Anderson and Guraya, 2006). Hot air oven heat/moisture treatment (HWT) was used to treat sweet potato starch (Xia et al., 2016).

Despite numerous studies to determine the physicochemical properties of MWT-treated and HWT-treated starches, there exists no study on comparison of the effects of MWT and HWT on the physicochemical and morphological properties of CS. Therefore, this study investigates the effects of heat/moisture treatments on the amylose leaching (AML), resistant starch (RS) content, crystalline and morphological characteristics of cassava starch (CS). The study focused on two heating methods: microwave heat/moisture treatment (MWT) and hot air oven heat/moisture treatment (HWT).

2 Materials and Methods

2.1 Materials

Cassava starch (CS) was provided by Thaistarch Co., Ltd., Nakhon Ratchasima, Thailand. All chemicals used in this study were of analytical grade.

2.2 Chemical composition analysis

Amylose, ash, and moisture content were analyzed according to the procedures of Varatharajan et al. (2011). The analyses were performed in triplicate.

2.3 Preparation of heat/moisture treatment of cassava starch (CS)

Moisture content of CS was adjusted to 35% (w/w) by adding of distilled water and mixing with a food mixer (Otto HM-273, Thailand). The mixtures were then kept at 4ºC for 24 h. The moisture-adjusted CS was then divided for microwave heat/moisture treatment (MWT) and hot air oven heat/moisture treatment (HWT) using the protocols as follows:

MWT: the moisture-adjusted CS was transferred to a microwave digestion vessel and placed in a microwave digestion/extraction system (Multiwave 3000SOLV, Anton Paar, Austria) following the procedure in Vatanasuchart et al. (2011) with certain modifications. To avoid starch gelatinization, the maximum temperature of microwave irradiation was kept at 60ºC. The moisture-adjusted CS was heated for 30 min.

HWT: the moisture-adjusted CS was transferred to a test tube covering with cap and put in a hot air oven. The moisture-adjusted CS was then heated at 60ºC for 30 min.

After treatment, both MWT- and HWT-treated CS were incubated at 4ºC and 60ºC for 24 h by controlling the moisture content. The MWT- and HWT-treated CS were subsequently dried at 50ºC for 24 h using hot air oven, sieved through 100-mesh and stored in desiccator until further analyses.

2.4 Amylose leaching (AML) analysis

AML analysis followed the procedure in Vatanasuchart et al. (2010) with some modifications. In the analysis, MWT- and HWT-CS (20 mg) in water (10 mL) were heated at 60–100ºC in sealed tubes for 30 min. The tubes were shaken by hand every 5 min to suspend the starch slurry. The tubes were then cooled to room temperature and centrifuged at 10,000 rpm for 10 min. The supernatant (1 ml) was withdrawn and amylose content was determined. AML was expressed as percentage of amylose leached per 100 g of dry starch. The analyses were performed in triplicate.

2.5 Resistant starch (RS) content analysis

RS content of native, MWT- and HWT-treated CS was measured using Megazyme Resistant Starch Kit (Megazyme Inc., Wicklow, Ireland) based on the Method 2002.02 of the Asscoication of Official Analytical Chemists AOAC according to the method described by Xia et al. (2016). The analyses were performed in duplicate.

2.6 X-ray diffraction (XRD) measurement

X-ray diffractograms were obtained using Power X-ray Diffraction (XRD-D2 Phaser, Bruker, USA). Samples were scanned with Cu-Ka radiation (λ = 0.154 nm). The X-ray generator operating conditions were 30 kV and 10 mA. Samples were tightly packed in the sample holder and scanned in the angular range of 2θ = 4–35º with a scanning rate of 1.0 s. The total crystallinity (TS) was calculated following the formula in Mutungi et al. (2009). The measurements were performed in duplicate.

2.7 Scanning Electron Microscopy (SEM) analysis

Scanning electron microscopy (SEM, JSM-6010LV, JEOL, Japan) was used to reveal the effect of heat/moisture treatments on morphology of CS. The native, MWT- and HWT-treated CS were mounted on a metal stub and sputter-coated with thin gold film. Microstructure and surface characteristics of the samples were observed.

2.8 Statistical analysis

The statistical analysis was carried out using Minitab® 17 statistical software. One-way analysis of variance (ANOVA) was used to analyze the data. When significant (p<0.05) differences were found, Tukey's test was used to determine the differences among means.

3 Results and Discussion

3.1 Composition of native cassava starch (CS)

Table 1 Composition of native CS

Sample	Composition (%, dry basis)	
	Amylose	Ash
Native CS	29.84 ± 0.96	0.07 ± 0.02

The chemical composition of native CS was analyzed and calculated as percent dry-weight (Table 1). Amylose and ash content were 29.84 ± 0.96% and 0.07 ± 0.02%, respectively. The amylose content was higher than 15.9-22.4% in Charles et al. (2005). The variations of amylose content are probably due to the differences in species and growth conditions of native CS.

3.2 Effect of MWT and HWT on the amylose leaching (AML)

In figure 1, AML of native, MWT- and HWT-treated CS (under all conditions) increased with increasing temperature. These results were consistent with Zhang et al. (2009) and Vatanasuchart et al. (2011). In this study, the native CS showed higher

AML than MWT- and HWT-treated CS in the temperature range 70-100ºC. These results indicated that the MWT and HWT affected starch structure. However, no significant differences in the AML between MWT- and HWT-treated CS were observed. The observations are agreeable with Zhang et al. (2009), who reported that microwave irradiation altered the starch structure. Moreover, Vatanasuchart et al. (2011) documented that the heat/moisture treatment reduced the AML of starch attributing to (1) additional interaction between amylose-amylose and/or amylose-amylopectin and (2) decrease in granular swelling.

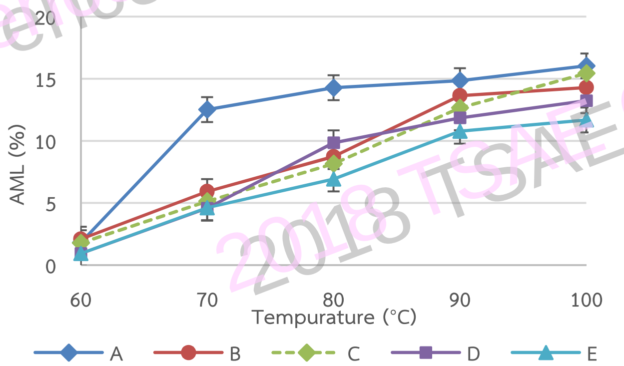

Figure 1 Amylose leaching (AML) of native, MWT- and HWT-treated CS in the range 60-100ºC, where A denotes the native CS, B and C denote MWT-treated CS followed by incubation at 4ºC and 60ºC, respectively, and D and E denote HWT-treated CS followed by incubation at 4ºC and 60ºC, respectively.

3.3 Effect of MWT and HWT on the resistant starch (RS) content

Table 2 RS content of native, MWT- and HWT-treated CS

Treatment[1]	RS (%, dry basis)[2]
A	5.27 ± 0.68^a
B	2.46 ± 0.22^b
C	2.14 ± 0.05^b
D	1.48 ± 0.02^b
E	3.22 ± 0.17^b

[1] A denotes the native CS, B and C denote MWT-treated CS followed by incubation at 4ºC and 60ºC, respectively, and D and E denote HWT-treated CS followed by incubation at 4ºC and 60ºC, respectively.
[2]The value represent the means of duplicate ± standard deviations. The different letters in the column represent the difference among treatment at $p<0.05$.

RS content of native, MWT- and HWT-treated CS is shown in Table 2. Native CS had RS value of 5.27±0.68%, slightly lower than 6.8-14.0% in Charles et al. (2005). This value was consistent with Vatanasuchart et al. (2009), who reported that RS content of cassava pearl was 4.5±1.0%. The variations of RS values are possibly due to the differences in species and growth conditions of native CS. The RS values of MWT- and HWT-treated CS (all conditions) were significantly lower than that of the native CS (control) ($p<0.05$). This study indicates that the RS content was affected by the heat/moisture treatments. The decreasing trend of RS values was consistent with that of the AML, revealing that the structure change after the heat/moisture processing of CS. In addition, Vatanasuchart et al. (2009) reported that the reduction of RS value (type III) was due to gelatinization and retrogradation after processing when compared to the RS value (type II) of native CS.

3.4 Crystalline structure of native, MWT- and HWT-treated CS by XRD

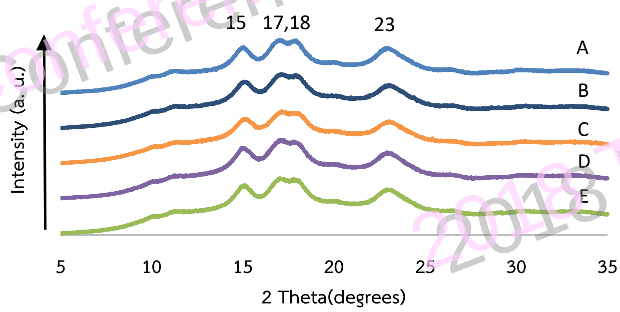

Figure 2 X-ray diffractograms of native, MWT- and HWT-treated CS in the range 60-100ºC, where A denotes the native CS, B and C denote MWT-treated CS followed by incubation at 4ºC and 60ºC, respectively, and D and E denote HWT-treated CS followed by incubation at 4ºC and 60ºC, respectively.

XRD patterns of native, MWT- and HWT-treated CS are shown in Figure 2. All diffraction patterns have four planes at 2θ about 15, 17, 18 and 23° indicating A-type pattern. This result is consistent with Mutungi et al. (2009), who reported that both A- and mixtures of A- type and B-type patterns were found in native cassava starch depending on genotypic and field growth conditions. In this study, the intensity of the reflection at 2θ = 15° and 18° of MWT-treated CS followed by incubation at 60ºC (C) and HWT-treated CS followed by incubation at 4ºC

(D) slightly decreased compared to the native CS (A), indicating imperfect crystallites with a lower ability to diffract X-rays (Mutungi et al., 2009).

Total crystallinities (TC) are summarized in Table 3. The TC of MWT-treated CS followed by incubation at 4ºC (B) and HWT-treated CS followed by incubation at 60ºC (E) slightly increased compared to that of NCS. Meanwhile, the MWT-treated CS followed by incubation at 60ºC (C) had the lowest TC (p<0.05). According to Mutungi et al. (2009), the increase in total crystallinity corresponded to higher resistant starch content.

Table 3 Total crystallinity (TC) of native, MWT- and HWT-treated CS

Treatment[1]	TC (%)[2]
A	29.90 ± 0.20[ab]
B	30.30 ± 0.20[ab]
C	26.80 ± 0.50[c]
D	28.90 ± 0.30[b]
E	31.35 ± 0.05[a]

[1]A denotes the native CS, B and C denote MWT-treated CS followed by incubation at 4ºC and 60ºC, respectively, and D and E denote HWT-treated CS followed by incubation at 4ºC and 60ºC, respectively.
[2]The value represent the means of duplicate ± standard deviations. The different letters in the column represent the difference among treatment at p<0.05.

3.5 Morphology of native, MWT- and HWT-treated CS

SEM images was used to investigate the microstructures of native, MWT- and HWT-treated CS. In Figure 3A, the native CS had a somewhat smooth surface, oval and concavity shape and loose granules. In Figure 3B and C, the MWT-treated CS appeared to be rough surface with irregular shape, compact and tight, and aggregation of granules. This result was probably due to the destruction of starch structures by heating and recrystallization during incubation. Some granules of HWT-treated CS were become compact, irregular shape, and aggregation of granules but less than the MWT-treated CS (Figure 3D and E). These results revealed that the morphologies of both MWT- and HWT-treated CS were affected by the heat/moisture treatment. Similar results were revealed heat/moisture treated potato (Xia et al., 2016). The starch granules were changed from loose granules to be compact structures after heat/moisture treatment.

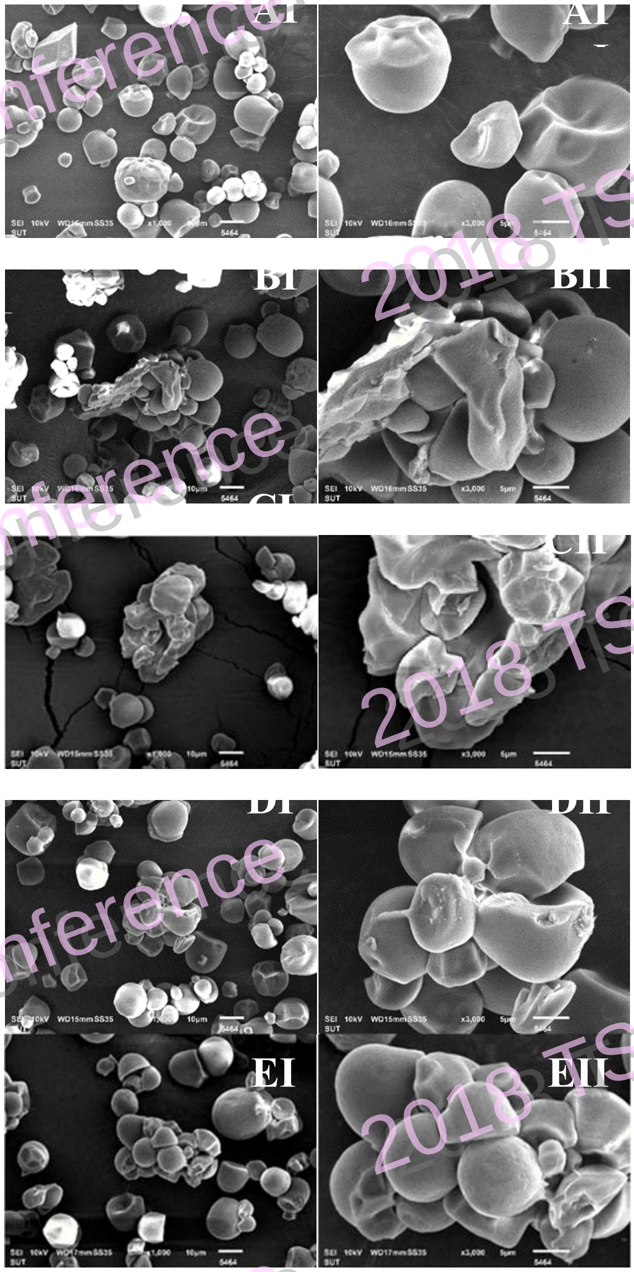

Figure 3 The SEM images of: (A) native NS, (B and C) MWT-treated CS followed by incubation at 4ºC and 60ºC, respectively, (D and E) HWT-treated CS followed by incubation at 4ºC and 60ºC, respectively. The magnifications are (I) 1000x and (II) 3000x.

4 Conclusions

MWT and HWT noticeably lowered the AML in the temperature range 70-100ºC of treated CS. In addition, RS type III value was reduced by heat/moisture treatments of CS. A-type crystalline structures of MWT- and HWT-treated CS (all conditions) remained unchanged. Moreover, there was minimally variation in the TC between MWT- and HWT-treated CS. Microwave heating significant affected the structure of starch granules of treated CS by induced destruction and aggregation of starch granules. Future works are needed to investigate for increasing the RS value in treated CS

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