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Comparing the effect of four different thermal modifications on physicochemical and pasting properties of breadfruit (*Artocarpus altilis*) starch

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Abstract

The present work compared the effect of different thermal treatments namely heat-moisture treatment (HMT), microwave heating treatment (MHT), heat pressure treatment (HPT) and osmotic pressure treatment (OPT) on physicochemical and pasting properties of breadfruit starch. The results showed that all modified starches exhibited a change in granule morphology, crystalline type and relative crystallinity, pasting and functionality. HMT and HPT-treated starches displayed a loss in physical integrity of the granules. Following MHT treatment, granular structures disappeared to some extent accompanied by the appearance of small holes on the granule surface. HMT, MHT and HPT altered the X-ray diffraction pattern of the starch from B to A+B-type, whereas OPT changed the type to A-type. All modified starches exhibited a decrease in relative crystallinity as compared to the native starch. HMT, HPT and OPT induced an increase in pasting temperature and a reduction in setback. All thermal treatments reduced the peak viscosity and breakdown viscosity of native starch. OPT treatment resulted in the pronounced change of peak, breakdown and setback viscosity. All modification methods did not alter the gel strength but increased water absorption capacity of the starch.

Introduction

Breadfruit (*Artocarpus altilis*) contains approximately 76.7% carbohydrates (Adebowale *et al.*, 2005b). The short shelf life of fresh breadfruit is a problem commonly encountered for perishable food. In order to extend its shelf life, processing breadfruit into flour or starch is seen as an alternative. Studies on the application of breadfruit starch or flour in a range of food products have been carried out such as noodles (Akanbi *et al.*, 2011) and infant food (Amagloh *et al.*, 2007). Breadfruit-based products also have the potentials to mitigate type II diabetes and obesity (Turi *et al.*, 2015).

Starch can be used as a main raw material or food additive in the food industry. Native breadfruit starch however, exhibits limited application in the food industry due to its poor paste clarity and readily retrograded characteristic. In order to improve the properties, starch modification has been commonly applied (Nwokocha and Williams, 2011). Study on breadfruit starch modification has been previously conducted using heat-moisture treatment (HMT; Tan *et al.*, 2017). Other studies have also reported on the functional and pasting properties of native breadfruit starch (Akanbi *et al.*, 2009), and heat moisture treated, annealed, oxidized and acetylated breadfruit starch (Adebowale *et al.*, 2005b). However, the information on the effect of other physical modifications using osmotic pressure (OPT), microwave heating (MHT) and heat pressure (HPT) on the physicochemical and pasting properties of breadfruit starch is still limited. Physical modifications using heat, moisture, pressure, shear, or radiation have been widely used owing to the absence of any chemical residues (Adebowale *et al.*, 2005b; Huang *et al.*, 2016). HMT, MHT, HPT and OPT are some physical methods which have been widely developed recently. Considering that different treatments might result in different responses, it is thus interesting to compare the effect of the above treatments on starch. It is expected that the starch modification could improve some properties of the native breadfruit starch which in turn would be useful in industrial applications. The present work was therefore aimed to compare the effect of different physical modifications (HMT, MHT, HPT, and OPT) on the physicochemical and pasting properties of breadfruit starch.

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Materials and methods

Starch extraction

Mature unripe breadfruit was bought from Indramayu Regency, Indonesia. Starch from breadfruit was extracted based on the method described by Adebowale *et al.* (2005b) with slight modifications. Briefly, the breadfruit was peeled, sliced and washed thoroughly with water. Next, the breadfruit was blended with water at 1:4 ratio (w/v), and sieved using a filter cloth. The filtrate was precipitated for 12 h. Precipitated starch was washed with water, and centrifuged (Becman, model TJ-6) at 3,500 rpm for 30 min The resulting precipitate was oven-dried (Shel Lab FX-14-2) at 50°C for 24 h, milled (FCT-Z300) and sieved (80 mesh).

Preparation of modified breadfruit starches

For HMT, the method described by Sui *et al.* (2015) was followed. Firstly, the moisture content of starch was adjusted to 30%, equilibrated at 4°C for 24 h, and heated at 100°C for 16 h in an oven (Memmert UNB 300). The HMT-starch was further oven-dried (Shel Lab FX-14-2) at 50°C for another 12 h.

For MHT, the method described by Luo *et al.* (2006) was followed. Firstly, the moisture content of starch was adjusted to 30%. The starch was then placed in a microwave-safe container, equilibrated at 4°C for 24 h, and placed in a microwave oven (Sharp R-222Y(W)) at 1 W/g for 20 min. The MHT-starch was further oven-dried (Shel Lab FX-14-2) at 50°C for another 12 h.

For HPT, the HMT procedures described earlier was followed except that the moisture content of starch was adjusted to 20%. Next, the starch was further heated at 120°C for 1 h in an autoclave (Raypa AE-28 Dry) following which it was cooled to room temperature, removed from the bottle, and further oven-dried (Shel Lab FX-14-2) at 50°C for another 12 h.

For OPT, the method described by Pukkahuta *et al.* (2008) was followed. Firstly, 100 g (d.b.) starch was placed into a glass bottle and added with saturated sodium sulphate solution (200 mL). The mixture was then heated at 120°C for 1 h in an autoclave (Raypa AE-28 Dry) following which it was cooled to room temperature. washed with distilled water (500 mL × 8) and placed in a centrifuge tube. The starch was precipitated by centrifugation at 3,500 rpm before the pellet was oven-dried (Shel Lab FX-14-2) at 50°C for 12 h.

Granular morphology

The scanning electron microscopy (SEM) (JEOL JSM-6360 LA at 15 kV) was used to determine the granule morphology of dried starch samples by spreading them on an aluminium plate and coating with gold/palladium at 8-10 mA for 10-15 min. Representative digital images of starch granules were obtained at 1,000, 5,000 and 10,000× magnifications.

Starch crystallinity

PANalytical X'Pert PRO series PW3040/x0 was used to measure the X-ray diffraction pattern of breadfruit starch samples with Cu-K α-radiation with a wavelength of 1.54060 nm as X-ray source at 30 mA and 40 kV. The diffraction angle (2θ) of scanning was from 5–50° with a scanning step time of 2.9050 s.

Pasting properties

The pasting properties of breadfruit starch samples were analysed using Rapid Visco Analyser (RVA StarchMaster 2, Warriewood, Australia). Briefly, the starch samples were separately weighed (3.5 g) and distilled water (25 mL) was added. The starch suspension was poured into RVA canister, equilibrated (50°C, 1 min), heated to 95°C (3.7 min), held (95°C, 2.5 min), cooled (50°C, 3.8 min) and kept at 50°C for another 2 min. At first 10 s, the RVA paddle was rotated at 960 rpm to disperse the starch. After that, the paddle was rotated at 160 rpm.

Swelling volume and solubility

The breadfruit starch samples (0.35 g d.b. for each treatment) were placed into a centrifuge tube, and distilled water (12.5 mL) was added. Starch samples were mixed using vortex mixer for 20 s, heated in water bath at 92.5°C and regularly stirred for 30 min. Next, the starch samples were cooled for 1 min in ice water, and then centrifuged (3,500 rpm, 30 min). The supernatant was then separated, measured and oven-dried to measure the percentage of solubility (Collado and Corke, 1999). The following equations were used, respectively.

$$\text{Swelling Volume (mL/g)} = \frac{\textit{total volume - supernatant Volume}}{\textit{Weight of sample (d.b)}}$$

$$\text{Solubility (\%)} = \frac{\textit{Weight of dried supernatant}}{\textit{Weight of sample (d.b)}} \text{ x } 100\%$$

Water Absorption Capacity (WAC)

Distilled water (10 mL) was added to breadfruit starch samples (1 g) in a centrifuge tube, and vortexed. The solutions were then conditioned at room temperature (26 ± 2°C, 1 h) and centrifuged

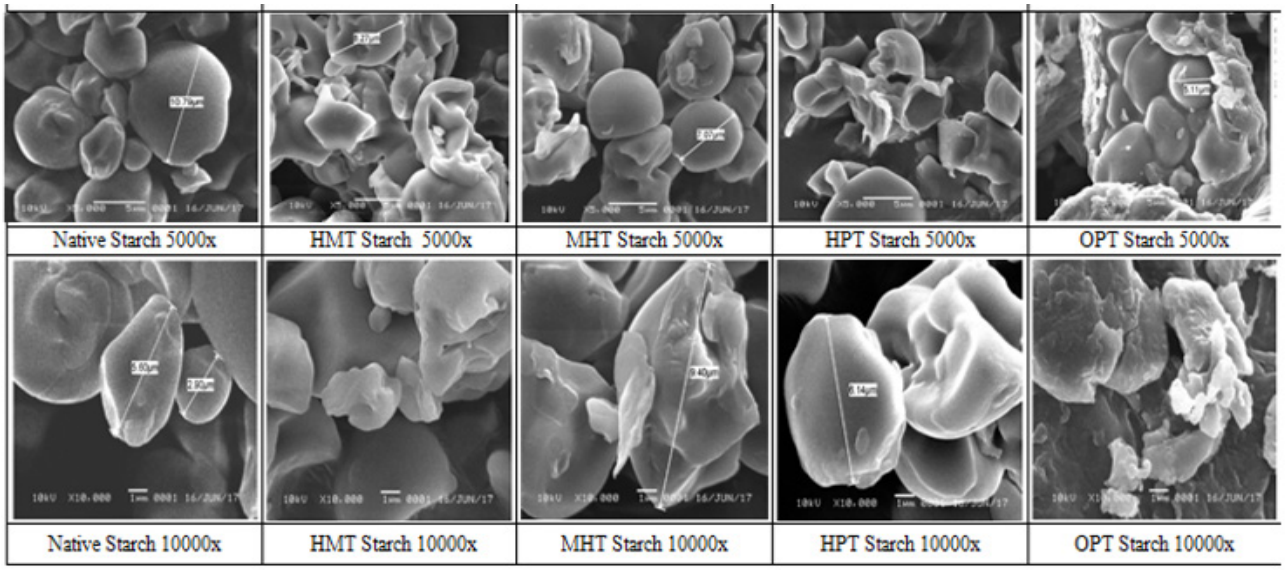

Figure 1. Granules morphology of native and modified breadfruit starches.

(3,500 rpm, 30 min). The volume of supernatant was measured (Beuchat, 1977). The WAC was calculated as the ratio between the volume of water absorbed and the weight of sample (d.b.).

Freeze-Thaw stability (% syneresis)

Distilled water was added to starch samples in centrifugal tubes (8% w/v), and adjusted to pH 6.5. The starch solutions were then placed in shaking water bath, heated to 95°C, held for 30 min, and cooled to 50°C. After that, the starch pastes were placed at 4°C for 24 h, and then frozen at -20°C for 48 h. The frozen starch samples were thawed (25°C, 4 h) and centrifuged (3,500 rpm, 15 minutes). Finally, the supernatant was calculated (Wattanachant *et al.*, 2003) .

$$\text{Syneresis (\%)} = \frac{\textit{Weight of supernatant}}{\textit{Weight of starch paste}} \times 100\%$$

Gel strength

Starch samples were mixed with distilled water (11% w/v d.b.) (95°C, 30 min) and stirred continually. The resulting pastes were separately poured into pipe containers (2.5 cm diameter, 3 cm high) at room temperature to form a gel, left for 1 h, covered with aluminium foil, then refrigerated (4°C, 24 h). The gel strength was analysed by a Texture Analyser (TA-XT2) (Zhu *et al.*, 2009).

Statistical analysis

Analysis of Variance (ANOVA) was used to compare the sample means at 95% confidence level ($p < 0.05$); then Duncan's Multiple Range Test was used to determine the significant difference between groups using SPSS Version 17.

Results and discussion

Starch granule morphology

The native starch granules of breadfruit exhibited irregular shapes (spherical, elliptical, polyhedral), and ranged from 3.0 to 7.9 µm (Figure 1), which is in line with another study (Tan *et al.*, 2017). Following thermal modification, a deformation of the starch granules was clearly visible. The granules broke and lost the round shape. This damage might have occurred in the granules with weak tissue structure. The loss of physical integrity in HMT and HPT-treated starch granules might be caused by partial gelatinisation as indicated by swelling, separation, and granular aggregation/fusion (Deka and Sit, 2016). The starch experienced pressure and heating outside the granules, resulting in a hollow shape in the granules surface. According to Tan *et al.* (2017), disruption might have occurred within the granule where the tissue structure was weak. The pressure and heating outside the starch during HMT might have led to a formation of compact granule and even resulted in concavities on the surface. The result on OPT-treated starch was different from the others. OPT-treated starch did not show its original form because of the merging of many granules. Pukkahuta *et al.* (2008) reported that OPT on corn starch induced a deformation on its granule surface. Similarly, MHT also altered the granule morphology in which the granular structures disappeared in conjunction with the appearance of small holes on the surface. Luo *et al.* (2006) have studied the impact of microwave treatment on corn starch and found that the granule surface has been changed probably due to the rearrangement or transfer of the molecular structure; hence, the formation of porous surface and a cavity at the centre granules.

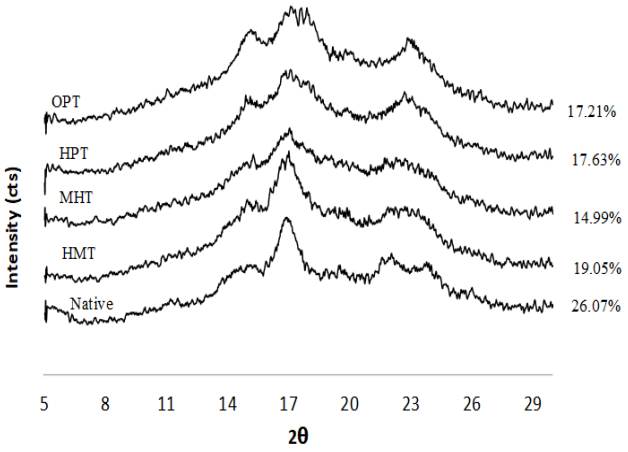

Figure 2. X-Ray diffraction patterns of native and modified breadfruit starches.

Starch crystallinity

The X-ray diffraction (XRD) patterns of native and modified breadfruit starch samples are presented in Figure 2. Native breadfruit starch sample exhibited diffraction peak at 5.4°, 15.3°, 17.1°, 22.3°, 24.7° (2θ) indicating the typical B-type pattern (Pukkahuta *et al.*, 2007). All heat treatments altered the X-ray diffractograms of breadfruit starch samples. The peak at 5.4° (2θ) was absent in HMT-treated starch. Furthermore, it showed higher peaks at 15.4°, 17.03° and the peaks at 22.3° and 24.7° (2θ) merged into a single peak at 22° (2θ). Therefore, HMT altered the XRD pattern of breadfruit starch samples from B to A+B-types pattern, which is in agreement with previous works on waxy potato starch (Lee *et al.*, 2012) as well as true yam and potato starches (Gunaratne and Hoover, 2002). Gunaratne and Hoover (2002) reported that the change in crystalline type was caused by the vaporisation of the 36 water molecules in the central channel of the B-unit cell and movement of a pair of double helices into the central channel. Both MHT and HPT-treated starches displayed similar type to HMT-treated starch. The starches exhibited diffraction peak at 15.2°, 17.1°, 22.7°, and 15°, 17.2°, 22.9° (2θ) respectively. Luo *et al.* (2006) reported that microwave treatment on amylomaize V starch changed the XRD of native starch from B to A+B type pattern. It produced double-helix chains with the starch crystallites shifting and leading to a crystalline array that was more ordered than that in native starch. This result is also in agreement with previous study on hydrothermally treated potato starch (Lee *et al.*, 2011). The XRD of OPT-treated starch was altered from B to A-type pattern. The starch exhibited diffraction peak at 15.3°, 17.1°, 18.0° and 23.0° (2θ), which is in agreement with OPT potato starch (Pukkahuta *et al.*, 2007). Furthermore, Lehmann and Robin (2007) explained that A and B-type pattern differed in their water content and packing of double helices. The relative crystallinity (RC) of the all modified starches was lower than that of the native starch, which was similar to previous studies on potato and true yafm (Gunaratne and Hoover, 2002), and also corn starches (Pukkahuta *et al.*, 2008). The RC reduction in HMT-treated starch resulted from a disruption of amylopectin crystallites (Chung *et al.*, 2009) while in MHT-treated one was induced by the change in double-helix chains (Luo *et al.*, 2006).

Pasting properties

The amylographs of native and modified starches are presented in Figure 3 and Table 1. The pasting temperature (PT) of HMT-, HPT-, and OPT-treated starches increased significantly as compared to that of the native starch. Our finding is in line with previously reported studies (Adebowale and Lawal, 2003; Adebowale *et al.*, 2005b). Thermal modification at high temperatures allows the formation of more complex bonds between amylose in the crystalline region and amylopectin in the amorphous region, resulting in a new, more strongly and closely bonded crystalline formation. The formation of the crystalline structure causes the starch to require a higher temperature to absorb water (Takahashi *et al.*, 2005).

All thermal treatments tested in the present work significantly reduced the peak viscosity (PV). This is in agreement with previous work on HMT of corn starch (Chung *et al.*, 2009), HMT of normal and waxy corn starches (Sui *et al.*, 2015), MHT of normal and waxy corn starches (Lee *et al.*, 2007), HMT and OPT of sago starches (Pukkahuta and Varavinit, 2007), as well as MHT of corn and rice starch (Uthumporn *et al.*, 2016). The decrease in PV might be due to the increase in the extent of amylose-amylose and amylose-amylopectin chain interactions occurring during the modification process (Gunaratne and

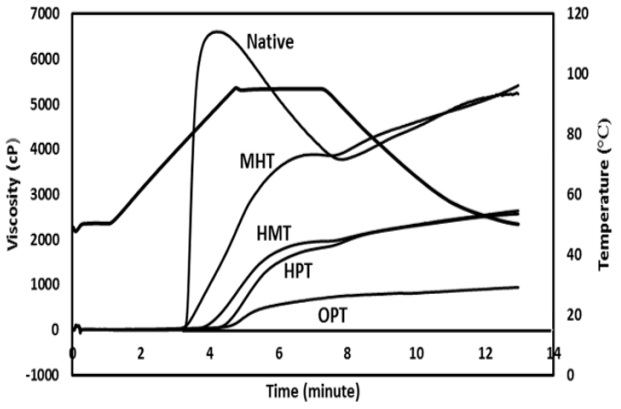

Figure 3. Rapid Visco Amilograms of native and modified breadfruit starches.

Table 1. Pasting properties of native and modified breadfruit starches.

Treatment	Pasting Temperature (°C)	Peak Viscosity (cP)	Hot Paste Viscosity (cP)	Breakdown Viscosity (cP)	Final Viscosity (cP)	Setback Viscosity (cP)
A (Native)	76.65 ± 0.82^{a}	6607.67 ± 318.70^{d}	3769.0 ± 141.49^{c}	2838.67 ± 231.05^{b}	5222.8 ± 160.86^{c}	1453.83 ± 49.72^{c}
B (HMT)	80.33 ± 0.90^{b}	1963.50 ± 199.29^{b}	1934.7 ± 174.71^{b}	28.83 ± 9.39^{a}	2636.5 ± 286.69^{b}	701.83 ± 122.93^{b}
C (MHT)	74.97 ± 0.35^{a}	3881.00 ± 101.53^{c}	3852.8 ± 103.88^{c}	21.00 ± 9.84^{a}	5413.2 ± 134.81^{c}	1646.50 ± 226.32^{c}
D (HPT)	87.98 ± 0.55^{c}	1809.83 ± 93.56^{b}	1783.2 ± 86.04^{b}	26.67 ± 8.69^{a}	2566.7 ± 109.22^{b}	783.50 ± 57.15^{b}
E (OPT)	88.86 ± 2.75^{c}	694.0 ± 161.77^{a}	673.8 ± 163.42^{a}	20.17 ± 3.40^{a}	940.0 ± 178.25^{a}	266.17 ± 20.93^{a}

Means with different letter in each column are significantly different ($p < .05$)

Corke, 2007). Furthermore, Pukkahuta *et al.* (2008) explained that the decrease in PV could also be due to the formation of the amylose-lipid complex during the heating process indicated by the presence of a peak at around 20° (2θ) in a diffractrogram when characterised by XRD. The presence of such a peak in the present work however was not obvious from the X-ray diffractogram suggesting the absence of amylose-lipid complex. It is therefore suggested that the decrease in PV was not due to the amylose-lipid complex.

All modified starches exhibited significantly lower breakdown viscosity (BDV) than native starch, suggesting that the starch granules became more stable to the continuous heating and agitating. This reduction will reduce the destabilisation effect on the amorphous region on crystallite melting (Gunaratne and Hoover, 2002). Significant decrease in BDV has been found for HMT of corn starch (Chung *et al.*, 2009), HMT and OPT of potato starches (Pukkahuta *et al.*, 2007), HMT and OPT of sago starches (Pukkahuta and Varavinit, 2007), MHT of normal and waxy corn starches (Lee *et al.*, 2007), white sorghum starch (Olayinka *et al.*, 2008), and finger millet starch (Adebowale *et al.*, 2005a).

HMT, HPT, and OPT modifications caused a decrease in SBV of native breadfruit starch. This result is in line with other studies on HMT of breadfruit starch Adebowale *et al.* (2005b), OPT of potato starch (Pukkahuta *et al.*, 2007), HMT of finger millet starch (Adebowale *et al.*, 2005a), and HMT of white sorghum starch (Olayinka *et al.*, 2008). According to Sun *et al.* (2014) the decrease in SBV after HMT treatment was affected by the presence of rigid non-fragmented swollen granules, amount of leached amylose, amylose chain length and granular size.

Functional properties

The functional properties of native and modified breadfruit starch samples are presented in Table 1. Swelling volume of modified starches were not significantly different from native breadfruit starch except for OPT which decreased in swelling volume. The decrease in swelling volume of OPT-treated starch was linked to the presence of SO_4^{2-} ions during the modification process which inhibited the absorption of water by starch granules. The addition of structure-forming ions (F-, K +, and SO_4^{2-}) has been known to decrease swelling volume of corn starch (Wang *et al.*, 2017). This result is in agreement with a previous work on OPT of corn starch (Pukkahuta *et al.*, 2008).

All modification treatments significantly increased the solubility of the breadfruit starch samples except for MHT-treated starch which was not significant. The trend of increased solubility in the present work is in line with previous studies on different starches modified with the same treatments employed in the present work (Kurakake *et al.*, 1997; Adebowale *et al.*, 2005a; Pukkahuta *et al.*, 2008; Dundar and Gocmen, 2013). The increase in solubility might be attributed to the increase of amorphous region of treated starch as indicated by the decrease in the crystallinity index of starch following modification. The increase in solubility with the decrease in crystallinity was also observed in the ozonated starch (Cahyana *et al.*, 2018) and HMT of corn starch (Pukkahuta *et al.*, 2008). The amorphous region was reported to be more susceptible to dissolve in hot distilled water than those of crystalline regions and thus high amorphous region exhibited high solubility (Pukkahuta *et al.*, 2008).

In terms of water absorption capacity, the native and modified starches displayed a lower water absorption capacity (WAC; Table 2). The WAC of all modified starches slightly increased compared to that of the native starch. The increase in WAC was caused by the starch tendency to have rising hydrophilic properties (Adebowale *et al.*, 2005b). Increased WAC was also found in HPT-modified sweet potato starch (Babu and Parimalavalli, 2013).

Table 2. Functional properties of native and modified breadfruit starch.

Treatment	Swelling Volume (mL/g d.b)	Solubility (%)	Water Absorption Capacity (g/g d.b)	Gel Strength (gf)	Syneresis (%)
A (Native)	7.72 ± 0.36^{bc}	2.67 ± 0.18^{a}	1.75 ± 0.05^{a}	2.72 ± 0.15^{a}	1.51 ± 0.37^{a}
B (HMT)	7.02 ± 0.32^{b}	6.10 ± 0.61^{c}	2.03 ± 0.02^{ab}	3.00 ± 0.25^{a}	25.84 ± 2.62^{c}
C (MHT)	7.87 ± 0.76^{c}	2.80 ± 0.75^{a}	2.63 ± 0.46^{c}	3.23 ± 0.37^{a}	25.99 ± 1.65^{c}
D (HPT)	8.10 ± 0.61^{c}	4.33 ± 0.56^{b}	2.23 ± 0.14^{b}	3.09 ± 0.24^{a}	23.70 ± 2.80^{c}
E (OPT)	6.07 ± 0.13^{a}	10.00 ± 1.06^{d}	2.82 ± 0.22^{c}	2.89 ± 0.11^{a}	14.57 ± 2.27^{b}

Means with different letter in each column are significantly different ($p < .05$)

All modified starches exhibited no significant effect on gel strength (Table 2). Hormdok and Noomhorm (2007) reported that the gel hardness of HMT-treated rice starch depended on its moisture content. Rice starch treated with 15 and 20% moisture exhibited a decrease in gel strength, but the treatment with 25% moisture did not affect the gel strength of starch. According to Choi and Kerr (2003), the textural properties of gels depend on the ratio of amylopectin and amylose, the interaction between the dispersed and continuous phases and the volume and deformation of the granules.

All of the modified starches underwent a significant increase in syneresis value as compared to the native starch (Table 2). The increase in the value showed that thermally modified starch had a low storage stability. The increase in syneresis might be due to the randomly generated interaction which reduced the water holding capacity of starch gel (Yadav *et al.*, 2013). The weak bonds in amorphous region might increase the distance between amylose chains when granule swells, facilitating the expulsion of the amount of water during thawing. Based on the results obtained in the present work, it was apparent that thermal modification treatment increased syneresis of native starch which was the drawback of the modification from the point of view of starch stability. Therefore, the modified starch in the present work might not be appropriate for frozen food applications.

Conclusions

All thermal modifications applied in the present work altered the granule morphology, crystalline characteristics, pasting and functional properties of native starch. Native breadfruit starch granules were small in size ranging from 3.0 to 7.9 μm, and the granules exhibited spherical, elliptical and polyhedral shapes. All thermal modifications caused deformation of the starch granules. HMT and HPT-treated starch granules lost their physical integrity. Granular structures disappeared along with the appearance of small holes on the granule surface when MHT was applied. X-ray diffractogram of native breadfruit starch exhibited a typical B-type pattern whereas all modified starches displayed significantly altered X-ray diffractogram. HMT, MHT, and HPT changed the XRD pattern of breadfruit starch samples from B to A+B-types, while OPT altered the pattern to A-type. The relative crystallinity (RC) of all modified starches was lower than the native starch (MHT<OPT<HPT<HMT). The pasting temperature of HMT, HPT, and OPT starches increased significantly compared to the native starch (OPT>HPT>HMT). Moreover, all modified starches exhibited a decrease in peak and breakdown viscosity. Meanwhile, HMT, HPT, and OPT caused a decrease in setback viscosity of the starch. All modifications did not alter the gel strength but increased syneresis and water absorption capacity of the breadfruit starch samples.

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