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Effects of pulsed electric field treatments on some properties of tapioca starch

Zhong Han[a], Xin An Zeng[a,*], Nan Fu[a,b], Shu Juan Yu[a], Xiao Dong Chen[b], John F. Kennedy[c]

[a] College of Light Industry and Food Sciences, South China University of Technology, China
[b] Department of Chemical Engineering, Monash University, Australia
[c] Chembiotech Laboratories, Institute of Advanced Science and Technology, 5 The Croft, Buntsford Drive, Stoke Heath, Bromsgrove, Worcestershire B60 4JE, UK

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ABSTRACT

Tapioca starch-water dispersions (8.0%, w/w) were subject to pulsed electric fields (PEF) at 30, 40 and 50 kV cm^{-1}, respectively. The effect of the intense energy input (Q) of PEF treatment (over 28.85 J g^{-1}) on the functionality of tapioca starch was investigated. Scanning electron microscopy analysis revealed the dissociation and damage of PEF treated tapioca starch granules. X-ray diffraction analysis demonstrated that tapioca starch tended to transform from crystalline to amorphous structure after PEF treatments. It was found that higher energy input (>49.36 J g^{-1}) could destroy the crystal region of tapioca starch. Meanwhile, with increasing electric field strengths, the gelatinization temperature, the gelatinization enthalpy, the peak viscosity and the breakdown viscosity of PEF-treated samples showed a remarkable decrease. These results suggest that PEF treatments would be a promising physical method to modify the properties of starch in order to obtain desired products in food industry.

1. Introduction

The starch granule structure is complicated and strongly depends on the botanical origin. Despite several decades of investigation on the crystalline ultrastructure of starch, many questions still remain unresolved, including the distribution of ordered and unordered areas in the granule, the size distribution of crystalline areas and the organization of mixed A- and B-type granules (Buleon, Colonna, Planchot, & Ball, 1998; Maaruf, Che Man, Asbi, Junainah, & Kennedy, 2001).

The technology of pulsed electric fields (PEF) treatment is a non-thermal food processing method, which treats pumpable liquid materials in a processing chamber with high intensity electric pulses (over 10 kV cm^{-1}) at short duration (less than 40 μs). PEF treatment has been widely used, not only for nonthermal pasteurization, but also for enhancing chemical reactions and modifying large molecules (Ade-Omowaye, Angersbach, Taiwo, & Knorr, 2001; Han, Zeng, Yu, & Chen, 2009; Han, Zeng, Zhang, & Yu, 2009; Jeyamkondan, Jayas, & Holley, 1999). The technique has been successfully applied to process various liquid foods with low viscosity and suitable electrical conductivity, such as milk, soymilk, pea soup, wine, liquid egg and the juice of oranges, apples, grapes, and carrots (Knorr & Angersbach, 1998; Knorr, Geulen, Grahl, & Sitzmann, 1994; Li, Chen, Liu, & Chen, 2008; Marselles-Fontanet & Martin-Belloso, 2007; Sampedro, Rivas, Rodrigo, Martinez, & Rodrigo, 2007; Torregrosa, Esteve, Frigola, & Cortes, 2006). Numerous studies have demonstrated that the prominent advantages of PEF treatment include the low processing temperature, continuous processing nature, short treatment time, and uniform treatment intensity. Concerning the application of PEF on starch modification, as evidenced in our previous paper (Han, Zeng, Yu, et al., 2009; Han, Zeng, Zhang, et al., 2009), we found that PEF treatments exhibit positive effect on degradation of large molecular materials. However, up to now, few researches have focused on the effect of energy dissipation of PEF on the properties of starch.

Among all types of starches, tapioca starch is one of the most widely used starches in food, paper and chemical engineering industries. The objective of this work is to investigate the energy dissipation of PEF treatment on the physicochemical properties of native tapioca starch, in order to develop a useful method to produce modified starch for industrial application.

2. Materials and methods

2.1. Materials

Commercial tapioca starch (13.20% moisture) was purchased from Dacheng Company (Chang Chun, China). KCl and ethanol were obtained from Hui Shi Biochemical Regant Co. Ltd. (Shanghai, China). All reagents used in this experiment were analytical grade.

* Corresponding author. Fax: +86 20 8711 3668.
E-mail address: xazeng@scut.edu.cn (X.A. Zeng).

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2.2. PEF system

Native tapioca starch was treated in a bench-scale, continuous PEF system (SCUT-PEF Team, South China University of Technology, China). The treatment chamber is composed of two parallel copper plate electrodes and a tubular insulator body made of Teflon. The native tapioca starch samples were pumped (Watson Marlow 323E/D Pump, USA) through the treatment chamber to receive the PEF treatment. The flow rate of the suspension was controlled by a rotameter (Model FM-01, Ningbo Jiutian Meter Company, China). The input voltage and pulse waveform were monitored with a two-channel digital oscilloscope (Tektronix TDS220, Beaverton, OR). Two type K thermocouples were inserted in the inlet and outlet of the chamber to measure the sample temperature. The temperature of the sample before PEF treatment was room temperature and did not exceed 50 °C after each treatment. A water-bath, adjusted to room temperature, was used to cool the samples immediately after PEF treatment. Before and after PEF treatments, the PEF system was cleaned and disinfected with 75% (v/v) ethanol solution and rinsed with sterile distilled water.

The equipment and processing parameters were as follows: square-wave form, bipolar; pulse frequency, 1 kHz; pulse duration time, 10 μs; field strength, 0, 30, 40 and 50 kV cm^{-1}; electrode diameter, 0.30 cm; electrode gap, 0.30 cm; and sample flow rate, 60 mL min^{-1}. The number of pulses (n = 21.37) and the total treatment time (t = 214 μs) were obtained from the following expressions:

$$n = \frac{Vf}{u} \quad (1)$$

$$t = n\tau \quad (2)$$

where V represents the volume of the treatment chamber (mL), f is the pulse frequency (Hz), $\boldsymbol{u}$ is the flow rate of starch suspension (mL s^{-1}) and τ is the pulse duration (μs) (Calderon-Miranda, Barbosa-Canovas, & Swanson, 1999; Unal, Yousef, & Dunne, 2002).

The energy input (Q) was calculated by Eq. (3) as below (Abram, Smelt, Bos, & Wouters, 2003; Min, Min, & Zhang, 2003; Perez & Pilosof, 2004; Zhao et al., 2008):

$$Q = E^2 t\sigma \quad (3)$$

where Q is the energy input (J m^{-3}), E is the electric field strength (V m^{-1}), σ is the electrical conductivity of the product (S m^{-1}) calculated for each process temperature, and t is the total treatment time (s).

The energy on temperature rise of solution (Q_s) was calculated by the following equations:

$$Q_s = TC_s \quad (4)$$

$$C_s = C_w - \frac{C_w X}{100} \quad (5)$$

where T is the temperature rise of solution (°C), C_s is the specific heat of the solution (J kg^{-1} °C^{-1}), C_w is the specific heat of the water (4200 J kg^{-1} °C^{-1}), and X is the water content of tapioca starch.

2.3. PEF treatment

Native tapioca starch suspension (8%, w/w) was prepared by adding tapioca starch to de-ionized water at 25 °C. KCl was added to the suspension in order to adjust its electric conductivity to 150 μS cm^{-1}. The suspension was mixed by magnetic stirring and pumped through treatment chamber to receive PEF treatment at 30, 40, and 50 kV cm^{-1}, respectively. The sample temperature was controlled below 50 °C using a water-bath. After PEF treatments, the samples were immediately cooled to room temperature, which was then vacuum filtered and dried in an oven at 40 °C for 24 h. Dry starch samples were collected for following analysis.

2.4. Scanning electron microscopy (SEM)

The SEM photos of all tapioca starch samples were taken with an E-SEM XL30 (FEI Company, Eidhoven, the Netherlands) microscope equipped with a SE detector of secondary electrons. Samples were fixed on a specimen holder using double-sided scotch tape and sputter-coated with gold (5 min, 2 mbar). After coating, samples were transferred to the microscope and observed with an acceleration voltage of 20 kV and a vacuum of 9.75×10^{-5} torr.

2.5. X-ray diffraction

The X-ray diffraction (XRD) analysis was conducted with a D/max 2200X-ray diffractometer (Tokyo, Japan), equipped with a conventional copper target X-ray tube, with operating conditions at 30 kV and 30 mA. The X-ray source was Cu Kα radiator. Data were collected from 2θ of 4.00° to 60.00° (θ being the angle of diffraction) with scanning speed of 12° min^{-1}. Native tapioca starch and PEF-treated tapioca starch samples were dried at 40 °C to constant moisture in a vacuum oven; then 50.00 mg of samples were added into the slide for packing prior to X-ray scanning.

2.6. Differential scanning calorimetry

The gelatinization properties of native tapioca starch and PEF-treated tapioca starch were analysed by a Perkin-Elmer DSC-7 (Perkin-Elmer Corp. Norwalk, CT, USA). Samples were sealed in an aluminium pan, which was mixed with water at a ratio of 3:7 (w/w). The sampled were heated from 20 to 90 °C at rate of 10 °C min^{-1}. A capsule filled with water was used as a reference. The onset, peak, and conclusion temperatures (T_o, T_p, and T_c) together with gelatinization enthalpy (ΔH_{gel}) were quantified.

The temperature range for gelatinization (R) was calculated as (Sandhu & Singh, 2007).

$$R = 2 \times (T_p - T_o) \quad (6)$$

2.7. Pasting behaviour

Pasting properties were studied on 6% (w/v) starch suspension (28.00 g dry starch dispersed in 432.00 mL distilled water) on a Brabender Viscograph-E (Germany). The mixture was gradually heated (while being stirred at 75 rpm) from 30 to 95 °C at 1.5 °C min^{-1}, and then held at 95 °C for 30 min. After that, the mixture was cooled from 95 to 50 °C at the same rate and then held at 50 °C for 30 min (Barminas et al., 2008). The viscosity was expressed by Brabender units (BU). The peak viscosity (maximum viscosity during pasting), breakdown (BD) viscosity (difference between the peak viscosity and minimum viscosity during pasting), setback viscosity (difference between the maximum viscosity during cooling and the peak viscosity during pasting), final viscosity, and pasting temperature were determined.

2.8. Statistical analysis

All of the sample analyses were carried out in triplicate and statistical analysis was performed using SAS v. 6.12 (SAS Institute Inc. Cary, NC, USA). The Student's t-test was used to estimate significant differences among means at a probability level of 5% ($p < 0.05$).

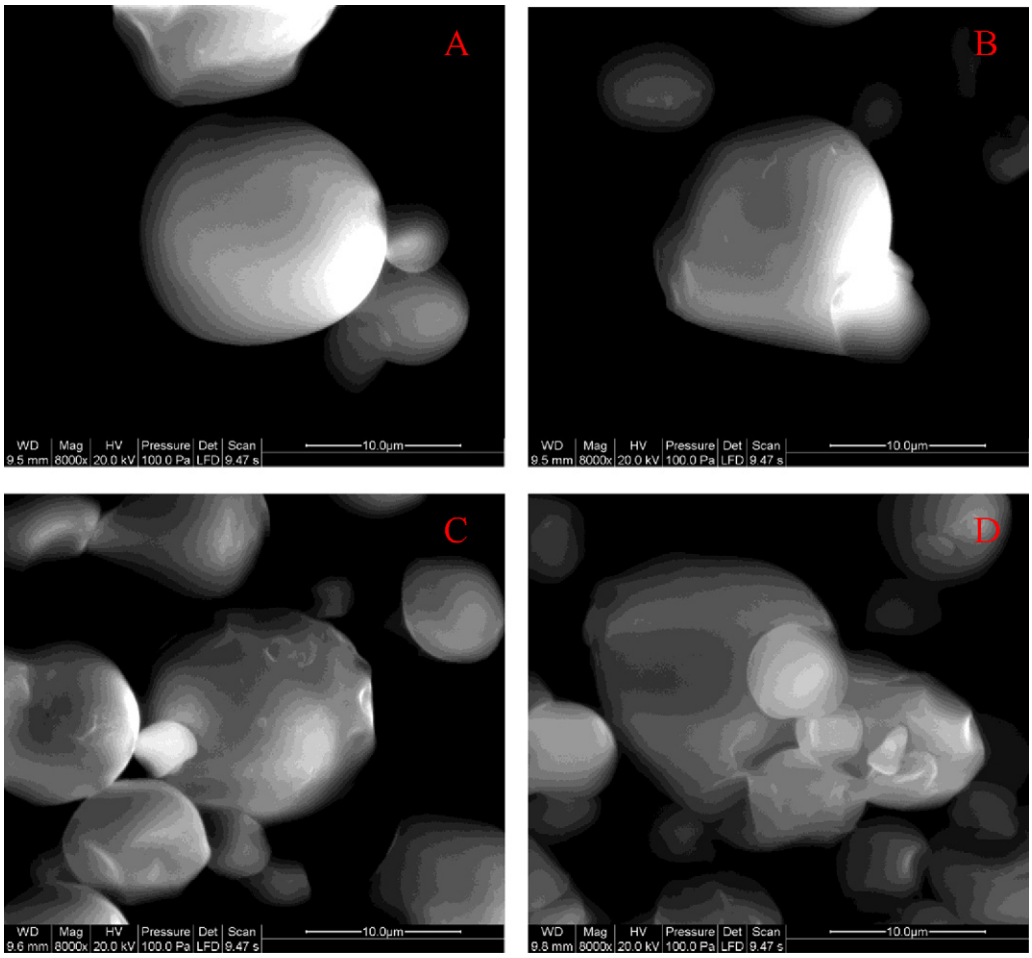

Fig. 1. SEM micrographs of tapioca starches treated at different electric field strengths: (A) native; (B) 30 kV cm^{-1}; (C) 40 kV cm^{-1}; (D) 50 kV cm^{-1}.

3. Results and discussion

3.1. Energy analysis

Table 1 summarizes the energy input (Q) of PEF treatments calculated by Eq. (3), the temperature rise (T), and the energy required for the temperature rise of solution (Q_s) calculated by Eq. (4). The energy input (Q) of PEF treatment at 30, 40, and 50 kV cm^{-1} were 28.85, 51.29, and 80.14 J g^{-1}, respectively. Meanwhile, the energy dissipated on the solution leading to temperature rise (Q_s) were 19.59, 26.58, and 30.78 J g^{-1} at 30, 40, and 50 kV cm^{-1}, respectively. It can be seen that the differences ($Q - Q_s$) between Q and Q_s at the same electric strength increased along with the increase of electric strength, which were 9.26, 24.71, and 49.36 J g^{-1} at 30, 40, and 50 kV cm^{-1}, respectively. It was thus inferred that with increased energy input (Q) of the PEF treatment, more energy ($Q - Q_s$) was consumed by tapioca starch, which should affect the physicochemical properties of tapioca starch.

Table 1
The energy input (Q) of PEF, the temperature rise and the energy on temperature rise of solution (Q_s) at different electric field strengths.[a]

Sample name	T (°C)	Q (J g^{-1})	Q_s (J g^{-1})	$Q - Q_s$ (J g^{-1})
30 kV cm^{-1} treated	4.7 ± 0.1	28.85 ± 0.03	19.59 ± 0.05	9.26 ± 0.04
40 kV cm^{-1} treated	6.3 ± 0.2	51.29 ± 0.07	26.58 ± 0.09	24.71 ± 0.08
50 kV cm^{-1} treated	7.3 ± 0.1	80.14 ± 0.09	30.78 ± 0.11	49.36 ± 0.10

[a] Values represent the means ± standard deviation for triplicate measurements.

3.2. Scanning electron microscopy

The SEM micrographs of all the tapioca starch granules are shown in Fig. 1. The surface morphology of native tapioca starch granules was smooth, oval, and irregularly shaped. After subjected to the PEF treatment at 30 kV cm^{-1}, some roughness or damages emerged on the surface of tapioca starch granules. With the treatment at 40 kV cm^{-1}, some pits emerged and some small starch particles aggregated together forming bigger ones, indicating that the granule structure of native starch had been altered by PEF. With the PEF treatment at 50 kV cm^{-1}, the granular shape of tapioca starch seem to disappear, replaced by some congregated fragments with gel-like structures. It is interesting to observe that the newly generated particles were smaller than the native starch granules, which may suggest that they might be the lost envelopes of granules. Similar phenomena have been reported in our previous papers (Han, Zeng, Yu, et al., 2009; Han, Zeng, Zhang, et al., 2009).

Blaszczak, Valverde, and Fornal (2005) suggested that the outer part of starch granule had a very dense layer which enhanced its resistance to external physical stresses. In the current study, after PEF treatment with increasing electric strengths, the increased energy could act on the fragments as well as the inner part of starch granule. As a result, starch granule might lose the protection of envelope; the resultant granule could absorb water more

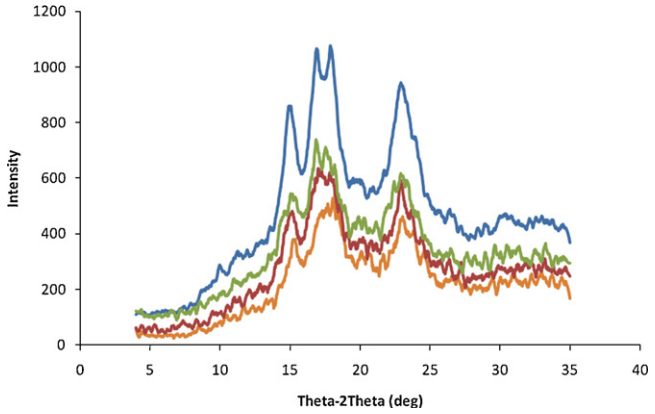

Fig. 2. X-ray powder diffraction patterns of tapioca starches treated at different electric field strengths: (A) native; (B) 30 kV cm^{-1}; (C) 40 kV cm^{-1}; (D) 50 kV cm^{-1}.

effectively and swell more easily, resulting in the increase of aggregation among particles.

3.3. X-ray diffraction pattern

The X-ray diffraction patterns of tapioca starch under different electric field strength are shown in Fig. 2. The main peaks of native tapioca starch were at approximately 15°, 17°, 18° and 23° (2θ), indicating that its structure is C-pattern, with relative crystallinity of 24.2%. After PEF treatments, the relative crystallinity of the treated tapioca starch was 18.1%, 11.4%, and 7.2% for the 30, 40, 50 kV cm^{-1}, respectively. Furthermore, it was interesting to observe that the double peaks around 17° and 18° turned into a single one after being treated at 50 kV cm^{-1}. From the energy analysis, the $(Q - Q_s)$ values at 30, 40, and 50 kV cm^{-1} were 9.26, 24.71, and 49.36 J g^{-1} respectively. The results thus suggested that higher energy (>49.36 J g^{-1}) input from the PEF treatment could destroy the crystal region of tapioca starch.

According to our previous papers (Han, Zeng, Yu, et al., 2009; Han, Zeng, Zhang, et al., 2009), PEF treatment is capable of destroying the crystalline region of starches, by offering higher energy for the reaction between tapioca starch granules and water molecules. This results in a distinct trend for tapioca starch granules to transform from crystal to non-crystal. Further investigation should be done to confirm this point in the future.

3.4. Thermal analysis

The DSC results of all starch samples are presented in Table 2. The graphical representations of DSC are shown in Fig. 3.

Compared to the T_o/T_p values of native tapioca starch (62.78/68.78 °C), the T_o/T_p values of PEF treated tapioca starch samples decreased along with the increase in electric strengths, which were 62.73/68.12 °C, 62.21/66.63 °C, and 61.76/65.94 °C at 30, 40 and 50 kV cm^{-1}, respectively. It can be seen that for PEF-treated tapioca starch, the decrease of its T_p was smaller than that of the T_o value, leading to the contraction of gelatinization temperature range (Table 2).

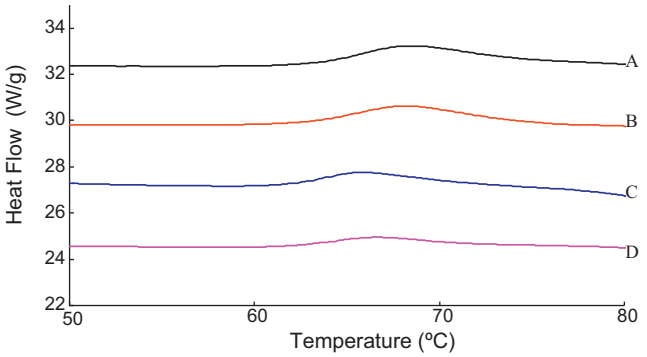

Fig. 3. DSC thermograms of gelatinization properties for PEF treated tapioca starches and native potato starch: (A) native; (B) 30 kV cm^{-1}; (C) 40 kV cm^{-1}; (D) 50 kV cm^{-1}.

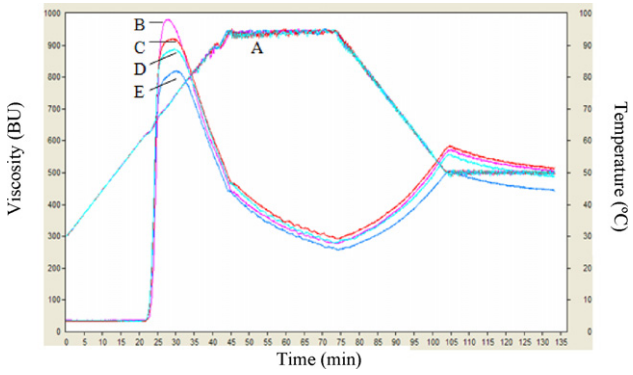

Fig. 4. Brabender viscosity of native and PEF treated tapioca starches as response to the applied temperature program (A): (B) native; (C) 30 kV cm^{-1}; (D) 40 kV cm^{-1}; (E) 50 kV cm^{-1}.

The gelatinization enthalpy (ΔH_{gel}) values of PEF-treated tapioca starch samples also showed a decrease as the electric field strength was increased. When the electric field strength was increased from 0 to 30, 40, and 50 kV cm^{-1}, the ΔH_{gel} values showed a remarkable decrease from 16.06 to 8.87, 4.82 and 3.80 J g^{-1}, respectively. From the energy analysis, the $(Q - Q_s)$ values were 9.26, 24.71, and 49.36 J g^{-1} at 30, 40, and 50 kV cm^{-1}, respectively, which indicated that the energy offered by PEF could affect the structure of tapioca starch.

Eliasson and Gudmunsson (1996) reported that gelatinization temperatures could be related to the degree of perfection of crystallites in the starch granules, and whereas gelatinization enthalpies could be related to the degree of crystallinity. Wang et al. (2008) also demonstrated the correlation between ΔH_{gel} and the crystalline region of starch. During the PEF treatment, some of the double helices present in crystalline and non-crystalline regions of the granule could be disrupted. XRD analysis showed that the crystalline region of the PEF-treated tapioca starch was destroyed. Therefore, during heating process in DSC, few double helices would unravel and melt during gelatinization; water molecules could

Table 2
DSC measurements for gelatinization properties of PEF treated tapioca starches and the native tapioca starch.[a]

Starch	T_o (°C)	T_p (°C)	T_c (°C)	ΔH_{gel} (J g^{-1})	R (°C)
Native sample	62.78 ± 0.02	68.78 ± 0.01	78.14 ± 0.03	16.06 ± 0.12	12.00 ± 0.10
30 kV cm^{-1} treated	62.73 ± 0.03	68.12 ± 0.02	75.15 ± 0.05	8.87 ± 0.07	10.78 ± 0.02
40 kV cm^{-1} treated	62.21 ± 0.07	66.63 ± 0.09	72.31 ± 0.05	4.82 ± 0.06	8.84 ± 0.12
50 kV cm^{-1} treated	61.76 ± 0.11	65.94 ± 0.10	72.61 ± 0.09	3.80 ± 0.05	8.36 ± 0.10

[a] Values represent the means ± standard deviation for triplicate measurements.

Table 3
The whole viscosity behaviours of PEF treated tapioca starches and native tapioca starch: (1) native; (2) 30 kV cm^{-1}; (3) 40 kV cm^{-1}; (4) 50 kV cm^{-1}.

Evaluation point		Beginning of gelatinization	Maxium viscosity	Start of holding	Start of cooling	End of cooling	End of final holding	Breakdown
(1)	Time (min)	22.83	27.92	43.33	73.33	103.33	133.33	703
	Viscosity (BU)	51	982	496	279	557	505	
	Temperature (°C)	62.9	71.3	94.1	94.6	50.6	50.3	
(2)	Time (min)	22.42	29.00	43.33	73.33	103.33	133.33	626
	Viscosity (BU)	47	921	517	295	572	512	
	Temperature (°C)	62.4	72.8	91.6	94.6	50.6	50.0	
(3)	Time (min)	22.25	29.58	43.33	73.33	103.33	133.33	608
	Viscosity (BU)	46	889	505	281	546	489	
	Temperature (°C)	62.2	73.8	92.5	94.2	50.4	50.0	
(4)	Time (min)	22.42	30.17	43.33	73.33	103.33	133.33	560
	Viscosity (BU)	49	820	469	260	493	444	
	Temperature (°C)	62.4	74.7	93.1	94.3	50.8	50.1	

react with molecules in crystalline region more easily, causing the decrease of ΔH_{gel} of gelatinization (Gunaratne & Hoover, 2002).

3.5. Pasting characteristics

The viscosity parameters of all tapioca starch samples are summarized in Fig. 4 and Table 3.

It is demonstrated in Table 3 that the peak viscosity also showed a decrease with increasing electric field strength, with the lowest value observed at 50 kV cm^{-1}. When the electric field strength was increased from 30 to 40 and 50 kV cm^{-1}, the difference of peak viscosity between native and treated sample showed a corresponding increase from 61 to 93 and 162 BU, respectively. Peak viscosity reflects the ability of the starch granules to swell freely before their physical breakdown (Karaoglu, 2006). Tester and Morrison (1990) reported that amylopectin contributes to swelling, whereas the amylose and lipids inhibit swelling for native starch. After the PEF treatment, the surface structure of the tapioca starch granules was destroyed (as can be seen from SEM pictures) and the crystalline structure, consisting of amylose and amylopectin, was also destroyed (as shown in XRD patterns), resulting in the decrease of peak viscosity.

The breakdown viscosity also decreased with increasing electric field strength. When the electric field strength was increased from 0 to 30, 40, and 50 kV cm^{-1}, the breakdown viscosity was correspondingly decreased from 703 to 626, 608, and 560 BU, respectively. After the PEF treatments, the decreased breakdown viscosity of modified starch indicated that the stableness of hot starch paste became better. Hence we could use the PEF-treated tapioca starch instead of native starch in many industrial applications such as sterilization of canned food.

The last portion of Brabender pasting curve demonstrated the changes of viscosity associated with gelation and retrogradation during cooling. Compared with the setback viscosity of native tapioca starch (425 BU), the setback viscosity decreased with increasing electric field strength, which was 349, 343, and 327 BU for the electric strength of 30, 40, and 50 kV cm^{-1}, respectively. Meanwhile, the final viscosity was also changed with increasing electric field strength, which was 512, 489, and 444 BU at 30, 40, and 50 kV cm^{-1}, respectively.

4. Conclusions

In this study, the energy change under various intensity PEF treatments (up to 50 kV cm^{-1}) and the corresponding physicochemical properties of tapioca starch were investigated. The results from SEM images showed that dissociation, denaturation and damage of tapioca starch granules had been induced by PEF treatments. The X-ray diffraction pattern showed an obvious loss of crystalline structure after the PEF treatment at 50 kV cm^{-1}, and from energy analysis, higher energy (>49.36 J g^{-1}) offered by PEF could completely destroy the crystal region of tapioca starch. DSC analysis showed a decrease in gelatinization temperatures (T_o and T_p) and gelatinization enthalpy (ΔH_{gel}) with increasing electric field strengths. On the other hand, it was explored from Brabender rheological analysis that the peak viscosity, the breakdown viscosity as well as the final viscosity were all decreased in PEF-treated samples. Therefore, the energy change under PEF treatment should deserve further more detailed research for studying the intragranular molecular arrangement of tapioca starch granules.

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