

## Effect of temperature on the textural, thermal and microstructural properties of wheat flour/high amylose corn starch gels

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### Abstract

Textural, thermal and microstructural properties of single component gels and binary composite gels (BCG) of high amylose corn starch (Hylon VII) mixed with wheat flour at different wheat flour/Hylon VII (WF/H) ratios (95:5, 90:10 and 85:15) and temperatures (100, 121 and 135°C) were investigated. The visual appearance showed that as Hylon VII was increased in BCG, the stronger gel was achieved. Textural results confirmed by increasing Hylon VII, the firmness was increased, but the springiness, cohesiveness and adhesiveness were reduced. Moreover, the BCG at high temperatures showed the higher level of Hylon VII, the higher water solubility index would be achieved. The gelatinization enthalpy ( $\Delta H$ ) and peak gelatinization temperature ( $T_p$ ) increased by improving the content of amylose in BCG. Hylon VII showed the lowest peak viscosity and the BCG gel containing high amount of Hylon VII indicated a reduction in the paste viscosity. The differences in the microstructure of WF and Hylon VII gels were also reflected the pasting properties of the gels. Consequently, BCG of WF/H develops the stronger gel which can withstand at high thermal processing such as retort to improve the shelf-life of the final product.

**Keywords:** Composite gel; pasting properties; texture; microstructure; gelatinization.

### Introduction

The most abundant polysaccharide and most important dietary source of carbohydrates is plant starch. Among different kinds of starch, corn starch is an interesting ingredient with a precious position in the food, nutraceutical, textile and biomedical industries due to its thickening, gelling and bulking characteristics (Sandhu & Singh, 2007). The granules of starch are comprised of two types of  $\alpha$ -glucans including amylose and amylopectin, which are approximately 98 to 99% of the dry weight of starch (Carvalho *et al.*, 2007). Amylose is the chief material in gel forming which can also be linked with intact starch granules or fragments (Ott & Hester, 1995), whereas the swelling and gelation are associated with amylopectin (Maningat & Seib, 2010; Atwell, 2001; Krogars *et al.*,

2002). Corn starch can be classified into three groups based on the ratios of amylose to amylopectin. Normal corn starch has about 27% amylose, whereas “waxy starch” and “high amylose corn starch” possess less than 15% and more than 40% amylose, respectively. “High amylose corn starch” is a derivative form of the hybrid corn, which can be improved the strength and crunchiness of the product (Huang, 1995). A superior instance of high amylose corn starch is Hylon VII which contains about 70% amylose (Carvalho *et al.*, 2007; Kibar *et al.*, 2010).

Starch gels are an interesting issue from a colloidal chemical standpoint as well as their relation to other carbohydrate gels such as agar or cellulose. They are generally known as composite systems, consisting of swollen particles embedded in a three-dimensional (3-D) network of aggregated amylose chains. When starch granules are heated in excess water at a particular temperature, the swelling gets to be irrevocable and the structure of the granule is significantly modified. The structure of the gel is depends on the starch concentration, amount of leached-out

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components, configuration of swollen granules and the ratio of amylose/amylopectin as well as their interactions (Luo *et al.*, 2012; Carvalho *et al.*, 2007).

Functionality of starch solutions during thermal processing is critical in food operations. The inexpensive techniques such as micro visco-amylograph (MVA) and rapid visco-analyser (RVA) can be utilized to track the important quality changes during thermal processing (Suh & Jane, 2003). However, RVA has been previously applied to develop gels to determine fundamental rheology of starch systems and survey the extent of interaction among a mixture of food components (Zhang & Hamaker, 2003; Shim & Mulvaney, 2001).

Although, the functionality changes of starch upon processing and monitoring of its physicochemical modification were previously studied (Zhang & Hamaker, 2003; Sriburi & Hill, 2000), but the effect of thermal processing on the paste viscosity of starch systems particularly in presence of Hylon-VII is still unknown. Furthermore, numerous studies have been reported on the individual physicochemical properties of high amylose corn starch (Cieřla & Eliasson, 2003) and Hylon VII (Kibar *et al.*, 2010; Shi *et al.*, 1998), but the mixture of wheat flour with Hylon VII starch at different temperatures and concentrations did not investigate yet.

Therefore, the aim of current work was to evaluate the textural, gelling, pasting, thermal and microstructural properties of the single component gel (SCG) and binary composite gel (BCG) of wheat flour and Hylon VII starch at different ratios and temperatures. Certainly, the understanding of the functional properties of the gel mixture will be useful in selecting the appropriate ratio for high heat treatments such as cooking and improving the eating quality of final products like as noodle.

### Material and methods

High amylose corn starch as Hylon VII (~70%) was donated by National Starch & Chemical Company (10 Finderne Avenue Bridgewater, New Jersey, USA). Wheat flour

type 000 without any additives was purchased from Zarin Company (Mashhad, Iran). The approximate protein, fat, moisture and mineral content were measured following the methods of Association of Official Analytical Chemist Society (AOAC, 2000). The carbohydrate was also calculated by subtracting the amount of other ingredients from 100.

### Gel preparation

Single component gels (SCG) and binary composite gels (BCG) were prepared according to the procedure of Foo *et al.* (2013) and Tan *et al.* (2015) with slight modifications. The stock solution of individual biopolymer of Hylon VII and wheat flour (10% w/w) was dispersed in distilled water. The BCG of Hylon VII and wheat flour at different ratios of WF/H including 95:5, 90:10 and 85:15 were prepared at the total biopolymer concentration of 10%. All the solutions were stirred for 5 min and left overnight to ensure complete hydration. Excessive stirring was avoided to ensure that no fragmentation was imparted to the samples. Then, the samples were put in retort for 30min at temperatures 100, 121, 135 °C for SCG of Hylon VII starch gel and 100, 121 °C for BCG of WF/H at different ratios of 95:5, 90:10 and 85:15.

When the solutions were still warm, they transferred into the cut off plastic syringes (20 ml; inner diameter: 19 mm). A thin layer of paraffin oil was initially used to avoid adhesiveness of gels and reduce the friction between the gel and inner surface of the syringes during unmoulding. All the gels were cooled down to room temperature (25 °C) and refrigerated at 4 °C for at least 18 h. The gels were gradually removed from the syringe by pushing the plunger. Then, they were cut into 20 mm in length by using a sharp razor and were left to equilibrate at room temperature for at least 1 h prior to the textural analysis.

### Texture profile analysis (TPA)

Textural properties of gels were evaluated by using a TA-TX2 Texture Analyser (Stable Micro Systems Ltd., Surrey, UK), attached

with a 5 kg load cell. The cylindrical sample was compressed by using a 75 mm diameter compression platen at constant speed of 1.0 mm/s to a distance of 4 mm. The deformation level was set at 80% strain of the original sample height (Koliandris *et al.*, 2008). In order to avoid barrel effect during compression, the bottom plate and the top of the gel were covered with a thin layer of paraffin oil. The gel strength was evaluated according to the method provided by the Gelatin Manufacturers Institute of America (GMIA) testing standard. At least five measurements were recorded for each type of gel.

Textural characteristics of gels including modulus or modulus of deformability as a measure of firmness (Pons & Fiszman, 1996), hardness or the strength of gel structure as a measure of maximum force at any time during the first compression cycle (Rosenthal, 1999), and cohesiveness as the ratio of the area of work during the second compression to the area of work during the first compression were determined. The latter determines the degree of difficulty in breaking down the internal gel structure (Li *et al.*, 2004). In this study, cohesiveness was expressed as the percentage ratio of the peak area during the second compression to the peak area during the first compression.

#### Pasting properties

The pasting properties of the samples were determined using a Rapid Visco-Analyzer (RVA) (TCW, Newport Scientific, NSW, Australia). Briefly, 3 g of samples (wheat flour, mixed with different levels of Hylon VII starch) were transferred into an RVA aluminum canister and was mixed with 25 mL of distilled water. The sample was dispersed at a shear rate of 960 rpm for 10 s followed by a constant shear rate at 160 rpm. The samples were pasted according to the programmed heating and cooling cycle and the approved method ICC Standard No. 162 (Sriburi & Hill, 2000). The temperature profile was hold at 50°C for 1 min, ramped to 95°C at rate of ~12°C/min, hold at 95°C for 2.70 min, cool

back to 50°C at the same rate, hold at 50 °C for 2min. Each analysis took 13 min and was performed in triplicates.

The paste viscosity responses of RVA curves were: onset paste temperature (initial increase in viscosity) (PT), peak viscosity (maximum viscosity during heating) (PV), breakdown viscosity (the difference between the maximum and minimum peak viscosity after heating ramp), setback viscosity (difference between the maximum viscosity during cooling and the lowest viscosity after the heating ramp), trough (the lowest viscosity during holding time), final viscosity (FV) and peak time (PTM).

#### Water solubility and absorption index

The water solubility (WSI) and water absorption indices (WAI) were determined following the procedure described by Bujang (2006), with slight modifications. Gel was prepared according to the procedure explained by Teck (2012). The solution samples were put in the retort for 30 min at the temperatures from 100 to 121 °C and transferred into a centrifuge tube. Then, the mixture was centrifuged at 3000 g for 10 min. The supernatant was dried in an aluminum plate in the oven at 70 °C for 24 h. Then, it was cooled in desiccator and weighed. WSI is calculated by using the Eq. 1:

$$WSI (\%) = \frac{\text{weight of dissolved solid in supernatant}}{\text{weight of dry solids in original sample}} \times 100$$

(1)

The sediment formed after centrifuged in WSI was used to calculate WAI as follows:

$$WAI = \frac{\text{weight of sediment formed}}{\text{weight of dry solids in original sample}}$$

(2)

#### Differential scanning calorimetry (DSC)

Thermal properties of the gels were determined using a differential scanning calorimeter (Q100, TA Instruments, Inc., New Castle, USA). Starch samples (3±0.01 mg) were weighed into a steel DSC pan and mixed with 9 µL of distilled water (starch/water ratio 1:3). Then, the samples (~13 mg) were carefully stirred, hermetically sealed and left

for 1 h at room temperature to equilibrate. Heating was applied out from 30 to 200 °C at a rate of 5 °C/min. A sealed empty pan was also used as a reference. The onset temperature ( $T_o$ ), peak gelatinization temperature ( $T_p$ ), and conclusion temperature ( $T_c$ ) were measured as well as the gelatinization enthalpy change ( $\Delta H$ ) from the area of the endotherm peak using the Universal Analysis 2000 Software (TA Instruments, Inc., New Castle, USA).

#### Scanning Electron Microscopy (SEM)

The gel samples were initially frozen to -50°C and subsequently were freeze-dried. The freeze-dried gels were cut to the size of 0.5×0.5×0.5 cm using a razor blade, to expose the cross-sectional (CS) surface. A conductive double-sided tape was used to fix the gel samples to the round aluminum stub prior to spotting them with a thin layer of gold-palladium by using a sputter coater Quorum SEM coating system for about 90 s. The morphology of the gel was observed at 100× magnifications under a SEM (EVO, MA 10; ZEISS, German) with an accelerating voltage of 5 kV.

#### Statistical Analysis

All the experimental data were analyzed in triplicates and presented in mean  $\pm$  standard deviation (SD). The statistical evaluation between the treatments were subjected to One-way analysis of variance (ANOVA), using SPSS version 19.0 for windows software. A significant level of  $P < 0.05$  was maintained throughout the study.

#### Results and discussion

The visual appearance of the wheat flour, Hylon VII and BCG of WF/H at various ratios and temperatures are presented in Fig.1. It can be seen that Hylon VII develops self-standing or “hard gel” at concentration 10% at temperatures 121 and 135°C. In addition, Hylon VII gels were opaque and white in color with sticky and elastic texture (Fig. 1 b,c). The similar findings were also reported for modified tapioca starch gels (13%) (Foo *et al.*, 2013). In contrary, wheat flour produces

yellowish color gel with the attributes of “soft gel” or yield free standing gels without sagging (Fig. 1 d, e). For BCG two regimes of temperatures including 100 and 121°C were studied. As the amount of Hylon VII was increased in the mixed gel at 100 °C, the more elastic and firm gel was obtained (Fig.1. f-h). Similarly, by increasing the level of Hylon VII in BCG at 121°C, the stronger gel was achieved (Fig. 1. i-k). From technical point of view, it is more critical to have strong and elastic gels to endure the harsh conditions in retort and sterilization processing. Therefore, textural properties of SCG and BCG of WF/H at different ratios and temperatures were investigated to achieve the suitable composite gel for industrial applications.

#### Textural properties of gels

Gels can be defined as a substantially dilute cross-linked system, which may be weak or strong depending on their flow behaviour in steady-state (Gulrez *et al.*, 1980). Textural properties of SCG of Hylon VII, wheat flour and BCS gel at different ratios and temperatures are shown in Table1. It was found that the gel development did not occur at 100 °C (Fig. 1 a), which may be attributed to the limited swelling of the granules would leave unabsorbed water and as a result Hylon VII did not gelatinize during cooking at boiling water (100 °C) (Rendleman 2000; Ott & Hester, 1965). In contrast, high-amylose corn starch has shown strong gel and less stickiness at higher temperatures (Kasemsuwan *et al.*, 1998; Carvalho *et al.*, 2007).

The maximum firmness was obtained for Hylon VII at 135 °C (224 N) and followed by SCG of Hylon VII at 121 °C (136 N). This phenomenon could be related to the required level of solubility of amylose in gel forming and structure (Ott & Hester, 1965). The gelation is the formation of a three-dimensional network and contains only amylose and water. The molecular connection that happens after cooling of the gelatinized starch, generally known as retrogradation, leads to the elimination of water from gel and

dehydration of the material (Sobolewska-Zielińska *et al.*, 2010; Ott & Hester, 1965). The main reason for the rupture of starch gels is the retrogradation which is associated with

the syneresis of water. Therefore, the starch gel showed severe syneresis and the lowest rupture at 121 °C.

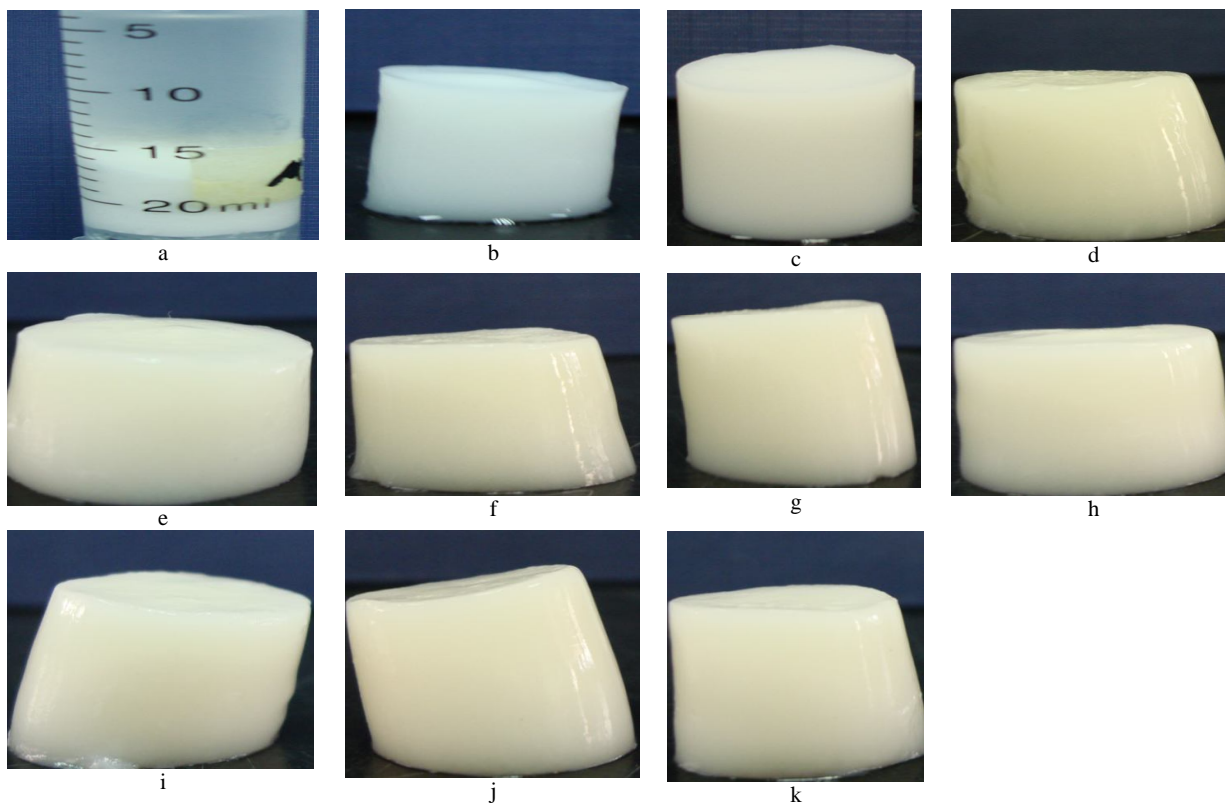


Fig. 1. Appearance of SCG of Hylon VII gels at :(a) 100 °C, (b) 121 °C, (c) 135 °C; SCG of wheat flour gels at: (d) 100 °C, (e) at 121 °C and BCG at different ratios and temperatures including: (f): WF/H 95:5 at 100 °C, (g) WF/H 90:10 at 100 °C, (h) WF/H 85:15 at 100 °C, (i) WF/H 95:5 at 121 °C, (j) WF/H 90:10 at 121 °C and (k) WF/H 85:15 at 121 °C.

Table 1. Textural properties of Hylon VII starch, wheat flour and BCG of WF/H at different temperatures.

| WF/Hylon VII | Temperature (°C) | Firmness (N)                | Springiness (%)        | Cohesiveness (%)       | Adhesiveness (g)          |
|--------------|------------------|-----------------------------|------------------------|------------------------|---------------------------|
| 0:100        | 121              | 136.77 ± 10.13 <sup>b</sup> | 27.11±03 <sup>a</sup>  | 2.2±0.01 <sup>c</sup>  | 8.5 ± 0.72 <sup>a</sup>   |
| 0:100        | 135              | 222.40 ± 45.15 <sup>a</sup> | 30.13±04 <sup>a</sup>  | 0.91±0.3 <sup>f</sup>  | 4.5 ± 0.74 <sup>b</sup>   |
| 100:0        | 100              | 32.5 ± 1.92 <sup>a</sup>    | 16.7±0.6 <sup>a</sup>  | 24.02±0.3 <sup>a</sup> | 3.92 ± 0.59 <sup>a</sup>  |
| 95:5         | 100              | 32.92 ± 0.81 <sup>a</sup>   | 16.01±0.1 <sup>b</sup> | 21.04±0.2 <sup>b</sup> | 3.6 ± 0.45 <sup>a</sup>   |
| 90:10        | 100              | 33± 2.32 <sup>a</sup>       | 15.0± 0.4 <sup>c</sup> | 22.34±0.1 <sup>c</sup> | 3.4 ± 0.24 <sup>a</sup>   |
| 85:15        | 100              | 35.6 ± 1.90 <sup>a</sup>    | 13.22±0.1 <sup>d</sup> | 22.11±0.4 <sup>d</sup> | 4.1±0.64 <sup>a</sup>     |
| 100:0        | 121              | 18.85 ± 1.97 <sup>c</sup>   | 11.87±0.1 <sup>e</sup> | 8.8±0.4 <sup>a</sup>   | 13.13 ± 1.80 <sup>a</sup> |
| 95:5         | 121              | 23.05 ± 1.84 <sup>b</sup>   | 14.1±0.0 <sup>d</sup>  | 7.8±0.1 <sup>b</sup>   | 11.27 ± 1.59 <sup>a</sup> |
| 90:10        | 121              | 30.2 ± 2.32 <sup>a</sup>    | 15.02±0.2 <sup>c</sup> | 6.1±0.4 <sup>c</sup>   | 8.83 ± 1.04 <sup>b</sup>  |
| 85:15        | 121              | 36.65 ± 3.52 <sup>a</sup>   | 17.06±01 <sup>b</sup>  | 4.2±0.0 <sup>d</sup>   | 4.06±0.56 <sup>c</sup>    |

\*. Values with similar letters in the same column do not differ significantly ( $P < 0.05$ ).

Furthermore, results showed that adding Hylon VII at different ratios had no significant effect on the gel firmness at 100 °C (Table 1), which may be related to the

limited swelling of the high amylose corn starch. The starch granules of the wheat are gelatinized and swollen in excess water below 100°C. Depending on the amount of the wheat

flour protein, the gluten network protein holds up the soluble wheat flour starch into the boiling water (Maningat & Seib, 2010). The granules of Hylon VII are diffused in water during boiling, but they are not swollen (Błaszczak *et al.*, 2007). Coagulation of gluten in wheat flour plays an important role in making gel, whereas Hylon VII starch may be considered as filler. On the other hand, the gel samples illustrated various results with different significances at 121 °C. By increasing the content of Hylon VII to BCG, firmness increased and adhesiveness decreased. The lowest firmness was found for the sample did not have any Hylon VII (WF). This shows that all wheat starches have been gelatinized and the gel structure is “soft gel” and due to the high leaching of amylose, the highest adhesiveness was obtained for SCG of wheat flour. During heating, the thermal energy breaks hydrogen bounds among the molecules of the starches. Hylon VII starch granules were hydrated by diffusion of water. The granules swell and lose their structure; the amylose leaches out and leads to the gelation process. During cooling, retrogradation happened and the distances among the starch molecules decreased, and finally the water was taken out of the gel and dehydration occurred. So, the high gel strength values reported for Hylon VII starch are perhaps related to the retrogradation of amylose (Sobolewska-Zielińska *et al.*, 2010; Sandhu & Singh, 2007; Kibar *et al.*, 2010). The results showed that by increasing the level of Hylon VII starch, the firmness increased and the adhesiveness decreased (Table 1).

#### Pasting properties

Pasting behaviours of wheat flour mixed with different levels of Hylon VII are reported in Table 2. The paste viscosity profiles of all samples were smooth curves without sharp peaks. The highest PV as a measure of gelatinization (239.5 RVU) was observed for wheat flour (Fig. 2). Starch gelatinization as a

complex phenomenon occurs when the internal crystalline structure of the starch granules is lost by heating in the presence of excess water (Carvalho *et al.*, 2007). The higher PV indicates high amylopectin content and higher resistance to retrogradation (Van Hung *et al.*, 2006). As the Hylon VII was increased in the BCG of WF/H, the pasting properties were reduced (Fig. 3). Peak viscosity was decreased from 148, 127 and 107 RVU when wheat flour was substituted with Hylon VII at 95:5, 90:10 and 85:15, respectively.

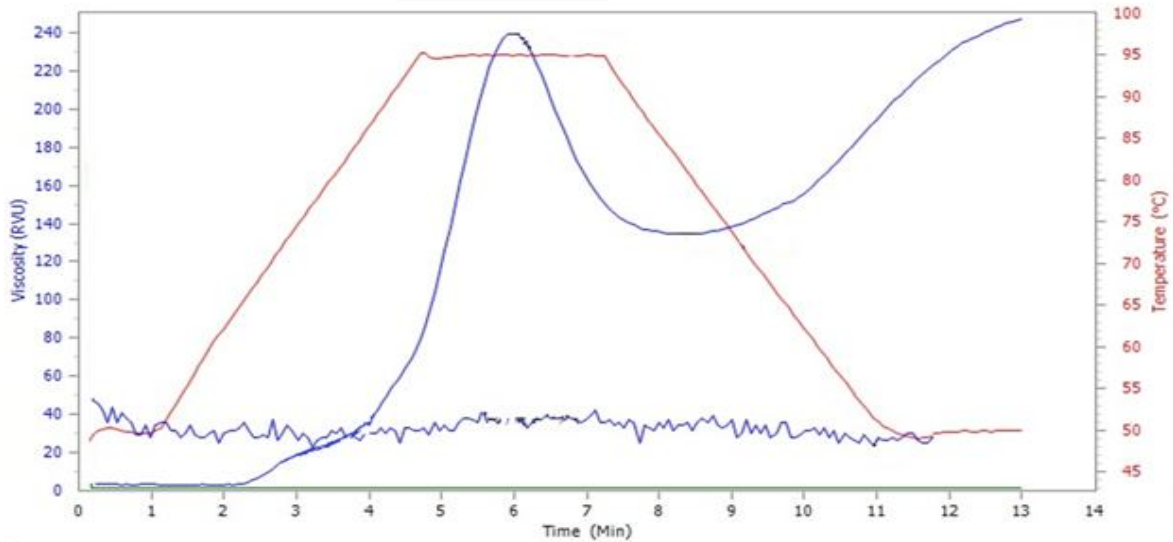
Holding samples at 95 °C under constant shear rate caused the disruption of granules, leaching and alignment of starch molecules, which consequently led to viscous breakdown known as trough. The FV is the paste viscosity at the end of the cooling cycle and is largely determined by the retrogradation of soluble amylose by cooling (Delcour & Hosney, 2010). Although, the setback viscosity is the recovery of viscosity during cooling of cooked starch and is known as FV minus the holding viscosity (Islas-Rubio *et al.*, 2014). As expected, setback viscosity which is associated with the degree of retrogradation of starch paste was found higher for wheat flour (Fig. 2). Breakdown, FV and setback viscosities were decreased by substituting of wheat flour with Hylon VII, due to reduction of amylopectin from wheat flour caused by starch dilution effect, promoted by Hylon VII addition (Fig. 3). Similar findings were also found for BCG of rice flour/defatted soy flour and whey protein/Hylon VII (Sereewat *et al.*, 2015; Carvalho *et al.*, 2007), which may be attributed to the competition of rice starch or whey protein to hydration as well as the restraint action of amylose to swelling, which results to lower viscosity. The low gelatinization peak and the high onset temperature of Hylon VII could be explained by the high amylose content and low level of amylopectin (Carvalho *et al.*, 2007).

**Table 2. Paste behaviours of Hylon VII, WF and WF/H mixtures in RVA at 10% concentration.\***

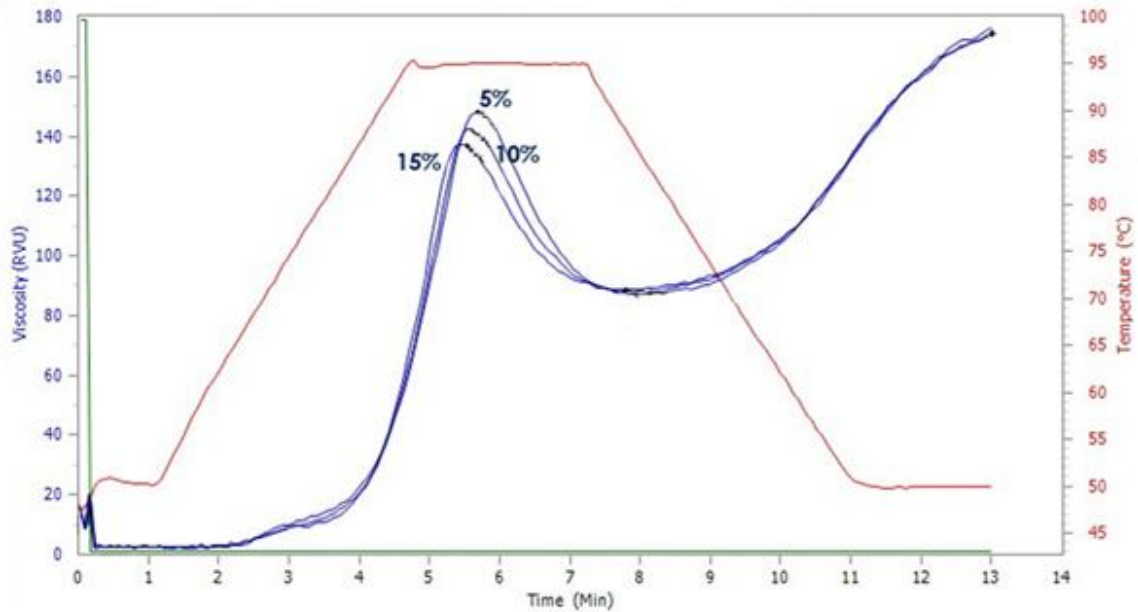
| WF:H  | Peak viscosity (RVU)     | Trough (RVU)             | Breakdown (RVU)          | Final viscosity (RVU)  | Setback (RVU)            | Peak time(min)           | Pasting temperature (°C) |
|-------|--------------------------|--------------------------|--------------------------|------------------------|--------------------------|--------------------------|--------------------------|
| 100:0 | 239.5 ± 0.1 <sup>a</sup> | 134.5 ± 0.1 <sup>a</sup> | 105 ± 1.4 <sup>a</sup>   | 250 ± 0.1 <sup>a</sup> | 115.5 ± 0.2 <sup>a</sup> | 2.2 ± 0.1 <sup>d</sup>   | 43 ± 0.0 <sup>c</sup>    |
| 95:5  | 148.1 ± 1.0 <sup>b</sup> | 87.2 ± 2.3 <sup>b</sup>  | 61 ± 0.8 <sup>b</sup>    | 180 ± 0.5 <sup>b</sup> | 92.83 ± 0.1 <sup>b</sup> | 2.5 ± 0.4 <sup>c,d</sup> | 43 ± 0.8 <sup>c</sup>    |
| 90:10 | 127.6 ± 0.4 <sup>c</sup> | 78.9 ± 0.9 <sup>c</sup>  | 48.66 ± 1.2 <sup>c</sup> | 160 ± 0.4 <sup>c</sup> | 81.08 ± 0.3 <sup>c</sup> | 3 ± 0.0 <sup>b</sup>     | 44 ± 0.2 <sup>b</sup>    |
| 85:15 | 107.7 ± 1.2 <sup>d</sup> | 69.7 ± 0.7 <sup>d</sup>  | 38 ± 0.0 <sup>d</sup>    | 140 ± 0.7 <sup>d</sup> | 70.33 ± 1.1 <sup>d</sup> | 3.4 ± 0.1 <sup>a</sup>   | 45 ± 0.1 <sup>a</sup>    |
| 0:100 | n.a.                     | n.a.                     | n.a.                     | n.a.                   | n.a.                     | n.a.                     | n.a.                     |

\*. Mean values in the same column with different letters are significantly different at p < 0.05.

\*. All the viscosities are presented in Rapid Visco Unit (RVU), which equals to 12 cp. n.a. is not applicable



**Fig.2. RVA pasting profiles of Hylon VII starch (the bottom graph) and wheat flour (WF) at 10%.**



**Fig. 3. RVA pasting profiles of BCG of wheat flour mixed with Hylon VII. 5%: WF/H (95:5), 10%: WF/H(90:10), 15%: WF/H**

(85/15).

**Water solubility and absorption index**

For gel structure, the amount of amylose in the soluble is necessary (Ott & Hester, 1965). Water solubility index (WSI) was used as a scale of the degradation of starch components, and often determines the level of free molecules leached out from the starch granules. Water absorption index (WAI) measures the rate of water absorbed by starch. Water solubility index (WSI) shows mobility and amount of soluble polysaccharide released from the starch components. WAI and WSI can be used as index of starch gelatinization (Bujang, 2006). The reverse relationship between the water absorption and amylose content led to the result that as the amylose content increased, the water absorption increased and water solubility decreased (Kibar *et al.*, 2010; Rodriguez-Sandoval *et al.*, 2012). The gel forming and ability of heated composite flour and Hylon VII starch to absorb water is determined using water

absorption index (WAI) and the fragment granules with residual amylose is responsible for water absorption (Hongsprabhas, 2007). The water solubility and water absorption indices for Hylon VII starch at various temperatures are provided in Fig. 4. The results revealed that both the water absorption and water solubility indices depend on the temperature. As temperature increases, WAI increases and WSI decreases. The low gelatinization temperature of Hylon VII may be due to the more water absorption by more amylose content. It can be found from Fig. 5, by increasing level of amylose, WSI decreased at 100 °C. It may be due to the development of Hylon VII percentage, reduced amount of wheat flour and the reduction of starch gelatinization. While at a high temperature (121 °C), the results indicated that by increasing the level of Hylon VII more water absorption and more gelatinization are observed (Fig. 5).

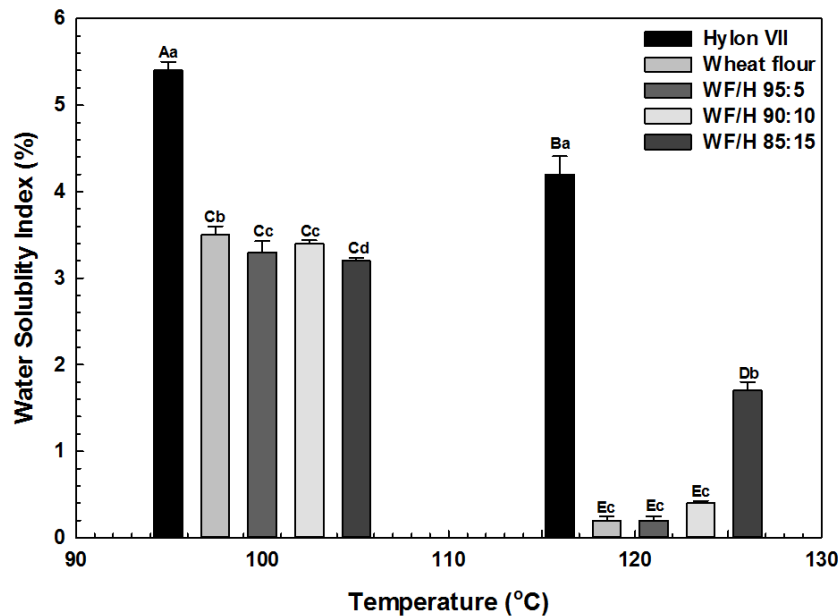


Fig. 4. The WSI of SCG and BCG of H/WF at different ratios and temperatures. The upper and lower case letter are corresponding to the statistical significant difference between treatments and within treatments, respectively ( $p < 0.05$ ).



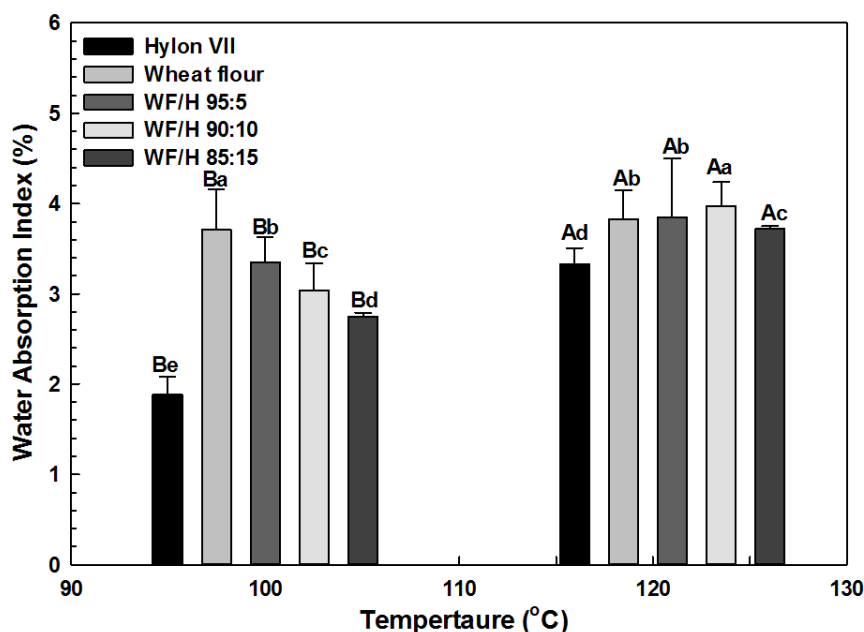


Fig. 5. The WAI of SCG and BCG of H/WF at different ratios and temperatures. The upper and lower case letter are corresponding to the statistical significant difference between treatments and within treatments, respectively ( $p < 0.05$ ).

#### Differential scanning calorimetry (DSC)

Thermal properties of the gels are summarized in Table 3 and their corresponding DSC thermograms are shown in Fig. 6. All the samples showed an endothermic peak, which shifted to higher temperatures as the wheat flour was substituted by Hylon VII. As a result, the WF/H of 85:15 was shown the higher  $T_o$ ,  $T_p$  and  $T_c$  than that of the other samples (Table 3). Gelatinization temperature of the gels directly depend on the amylose content and increases with enhancing the amylose content (Table 3) (Matveev *et al.*, 2001; Zaidul *et al.*, 2008). The gelatinization temperature ( $T_p$ ) of wheat flour and Hylon VII were observed at 61.91 and 107.50 °C, respectively (Fig. 6). Whereas, the  $T_p$  of BCG of WF/H at ratio 95:5 (~107.83 °C) was obviously closer to Hylon VII. However, the addition of Hylon VII to wheat flour caused enhancement of  $T_p$ , which issolely related to the gelatinization transition of the wheat flour starch granules (Zaidul *et al.*, 2008). Moreover,  $T_o$ ,  $T_p$  and  $T_c$  of Hylon VII were greater than the others gels except BCG of WF/H at ratio 85:15, which may be attributed to  $\beta$ -Type crystalline form of amylopectin as

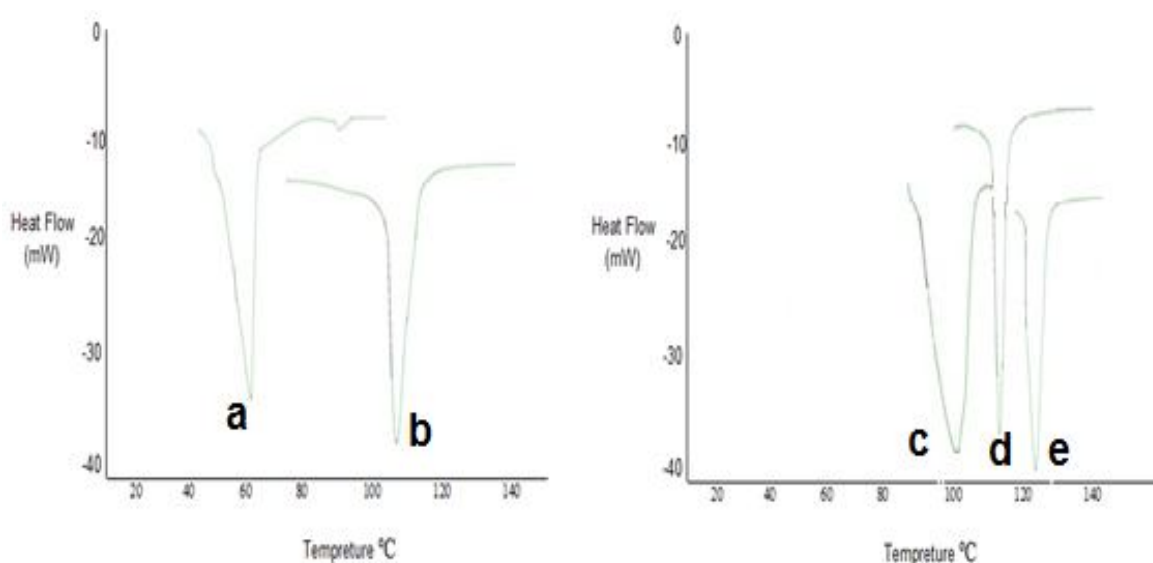
well as the hydrogen bonding between the chains in high amylose structures, resulting in higher gelatinization temperatures (Kibar *et al.*, 2010). Therefore, water uptake of Hylon VII was limited due to this chemical hindrance and needs higher temperatures for complete gelatinization. In contrast, the lowest  $T_o$  (39.77 °C) and  $T_c$  (92.09 °C) were found for wheat flour, whereas the highest values of  $T_o$  and  $T_c$  were seen for BCG of WF/H at ratio 85:15 (Table 3).

Since, the BCG of WF/H at ratio of 85:15 had the highest amylose content, it would require more energy to initialize the starch gelatinization. Therefore, the more gelatinization enthalpy ( $\Delta H$ ) was observed for the sample. The enthalpy reduction of wheat flour can be attributed to its more fiber, which is in agreement with the findings of other workers (Duta & Culetu, 2015; Sabanis *et al.*, 2009; Katina *et al.*, 2006). As the amount of amylose was increased,  $T_p$ ,  $T_o$ ,  $T_c$  and the gelatinization enthalpy ( $\Delta H$ ) were raised. In fact, increasing the level of Hylon VII would reduce the gelatinized wheat flour starch (Fig. 6).

**Table 3. DSC results of the gelatinization peak.**

| WF:Hylon VII | T <sub>o</sub> ( °C)     | T <sub>p</sub> ( °C)      | T <sub>c</sub> ( °C)    | ΔH(J/g)                 |
|--------------|--------------------------|---------------------------|-------------------------|-------------------------|
| 100:0        | 39.77±0.15 <sup>e</sup>  | 61.91±0.7 <sup>e</sup>    | 92.09±0.10 <sup>d</sup> | 245.4±0.8 <sup>d</sup>  |
| 95:5         | 100.75±0.13 <sup>d</sup> | 107.83±0.3 <sup>c</sup>   | 130.00±0.2 <sup>b</sup> | 483.70±0.2 <sup>b</sup> |
| 90:10        | 102.5±0.2 <sup>c</sup>   | 109.12±0.2 <sup>b</sup>   | 132±0.1 <sup>c</sup>    | 512.3±0.6 <sup>c</sup>  |
| 85:15        | 108.75±0.3 <sup>a</sup>  | 118.11±0.0 <sup>a</sup>   | 145.00±0.0 <sup>a</sup> | 845.6±0.2 <sup>a</sup>  |
| 0:100        | 101.74±0.2 <sup>b</sup>  | 107.50±0.4 <sup>c,d</sup> | 130.01±0.6 <sup>b</sup> | 238.8±0.1 <sup>e</sup>  |

\*. Mean values in the same column with different letters are significantly different at  $p < 0.05$ .



**Fig. 6.** DSC thermogram of gel samples, a: WF, b: Hylon VII, c: BCG of WF/H at ratio 95:5, d: BCG of WF/H at ratio 90:10, e: BCG of WF/H at ratio 85:15.

#### Microstructure of gels

The SEM images were obtained from cross-section of the gels, which illustrated the interior morphological structure of the gel network. The microstructural SEM images of SCG and BCG of wheat starch and Hylon VII at different temperatures are shown in Fig. 7. It can easily be seen very distinct structures in which SCG of wheat flour and Hylon VII has a smooth structure, well defined by the holes left where air and water entrapped in the gel (Fig 7 a, b and c, d). The wheat flour gel showed large fibrous strands with large void spaces and no remnant of granular structure which can entrapped water and produced a cream-yellowish gel. On the other side, the SCG of wheat flour was constructed of porous and cellular structure with interconnected thin walls (Fig.7 a). In comparison, SCG of Hylon VII illustrated a homogenous “honeycomb”

network of interconnected pores with an average size of about 100  $\mu\text{m}$ . The extensive amylose network was formed with granular structure indicating that it has more thermo-mechanical resistance structure (Fig. 7b). Due to the larger pore size, it lets the light pass easily through the structure, SCG of Hylon VII showed opaque gel with lighter color than that of wheat flour. As a result, it can be concluded that wheat flour gelatinized at lower temperature ( $\sim 62$  °C) than HylonVII gels (107.5 °C), which is in agreement with previous work on tapioca starch(66.2 °C), corn starch and maize starch ( $\sim 69.9$  °C) (Carvalho *et al.*, 2007; Carvalho & Mitchell, 2000; Tan *et al.*, 2015; Li *et al.*, 2007). Furthermore, it can be concluded that the fibrous thin network strands of wheat flour gels led to weak and soft gels as indicated by the low hardness in TPA. In contrary, the globular structure of

Hylon VII cause to strong and hard gel as confirmed by the textural studies. The differences in the microstructure of wheat flour and Hylon VII gels were also reflected the pasting properties of the gels.

#### **Effect of Hylon VII on the BCG microstructure**

As wheat flour substituted by Hylon VII, the gel structure was reinforced (Fig.1 c, d). Therefore, Hylon VII acted like as a filler between retrograded amylose entanglements and a more compact structure was formed (Fig. 7c, d). In contrast, the presence of wheat flour in BCG work a plasticizer by preventing molecular rearrangement of amylose leading to reduced rigidity. It was also similarly found for BCG of high amylose starch gels mixed with whey protein (Carvalho *et al.*, 2007). However, the independent Hylon and wheat starch networks were not distributed evenly throughout the gel. As a result, the textural properties of BCG were relatively weaker than the respective SCG, particularly for Hylon VII-SCG. Similar findings were observed for maize starch-egg white and amylase-egg white gels (Tan *et al.*, 2015; Li *et al.*, 2007). The more densely packed structure of SCG was also in agreement with the work of Rodriguez-Hernandez *et al.*, 2006 in which maize starch promoted segregation of gel led to a higher local polymer concentration and more compact networks for the mixed gels of waxy maize starch and gelatin.

#### **Effect of temperature on the BCG microstructure**

During cooking, the starch granules of the wheat flour become swollen and the granules gelatinized have lower resistance to enzyme which can be digested easier than high amylose starch (e.g. Hylon VII). High amylose starch is not gelatinized when cooked in water at 100°C (Rendleman, 2000). The effect of different temperatures on the microstructure of SCG and BCG gels are illustrated in Fig.7 d, e and f. It indicates the potential interactions between the coagulated proteins and the gelatinized wheat starch components at different temperature (Fig.7). As shown, the surface structure becomes softer and sticky as

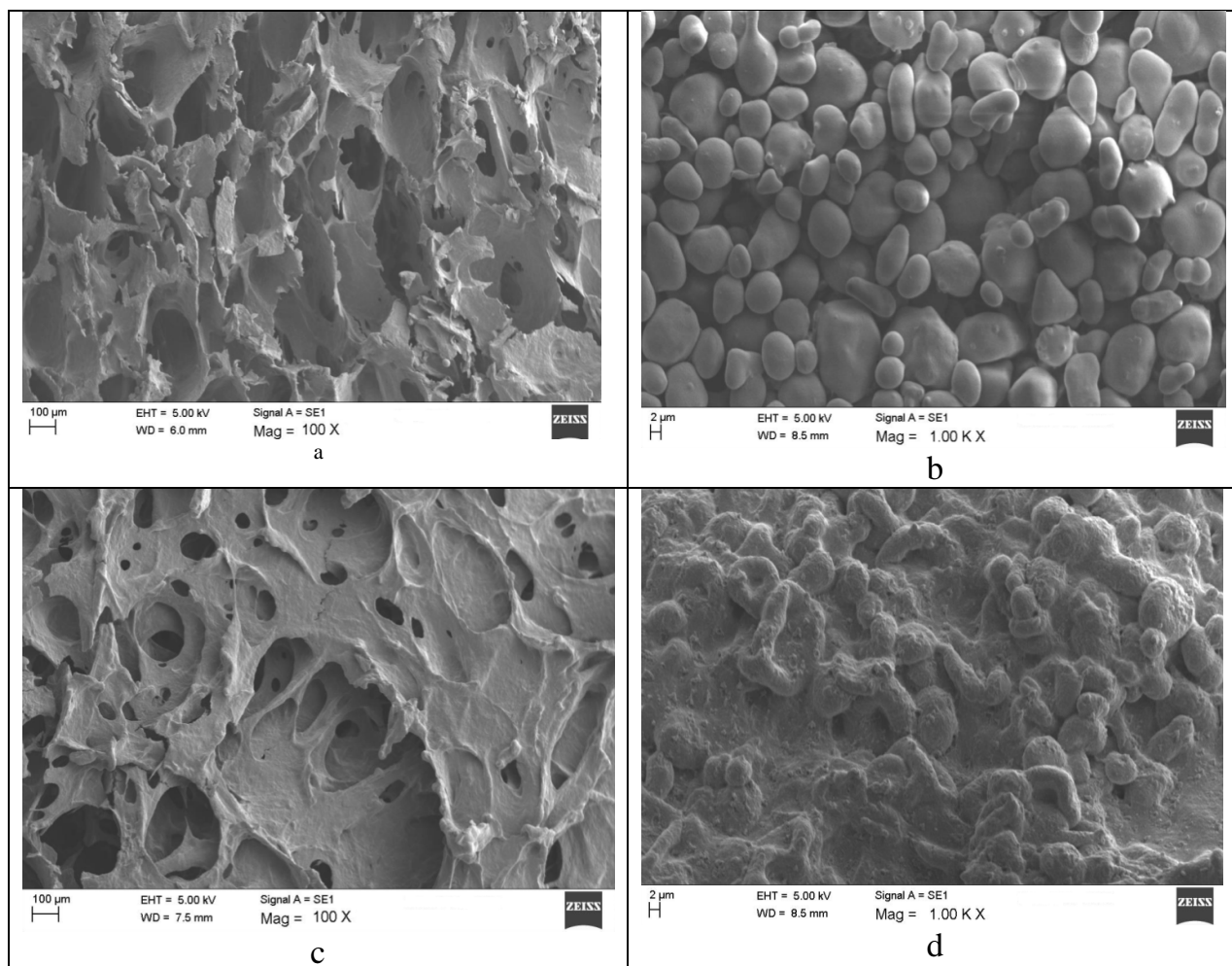
the temperature increased. The surface areas interconnected by the fibrils that contain gluten protein and some material leached from the wheat starch granules. Starchy filamentous network and strongly swollen starch granules are visible. As it is clear, the granules are not gelatinized at 100 °C. Dissolution in water is very slight at that temperature and the starch granules are not completely gelatinized at 121 °C. Raw starch granules can still be observed at the core. This may be, due to the fact that Hylon VII does not swell and gelatinize during cooking in boiling water (100 °C) and the swelling of Hylon VII starch granules is limited at higher temperatures. In this state, the Hylon VII starch gelatinization is not yet complete, and amylose enrichment occurs without more leaching, so the amylose is quite dense and still have crystalline. Therefore, by adding Hylon VII starch to wheat flour at 100°C, the surface of gel structure becomes rougher than higher temperatures. Since, Hylon VII was showed high gel strength, particularly when mixed with wheat flour, it can withstand at high thermal processing such as retort processing.

#### **Conclusion**

The effect of high temperatures and high amylose corn starch on the pasting properties as well as textural, thermal and microstructural of wheat flour gels were studied. By increasing the Hylon VII level in the BCG of WF/H, due to reduction of amylopectin from wheat flour caused by starch dilution effect, the pasting properties were reduced. It can be concluded that the competition of wheat flour for hydration and the restraint action of amylose to swelling, results to lower viscosity. The low gelatinization peak and the high onset temperature of Hylon VII could be explained by the high amylose content and low level of amylopectin. The highest WAI was found for Hylon VII at 121 °C, which may be related to higher diffusion of amylose to the granule surface. The combination that exhibit harder gels, tend to have more and higher Hylon VII level and the gel firmness is depend on retrogradation of starch gels. Hylon VII starch

has a low gelatinization level at 100 °C, and dissolution in water at that temperature was slight. Increasing the amylose level in gels cause firmness, strength and tightness in the gel network structure at retort temperature (121°C). Gel formation depends on the degree of hydration, the concentration of amylose in the soluble and rate of temperature. Thermal studies showed that the BCG of WF/H at ratio 85:15 had the greatest  $T_o$ ,  $T_p$  and  $T_c$  than that of the other samples. Due to the more amylose content, it had more gelatinization enthalpy ( $\Delta H$ ). In fact, increasing the level of Hylon VII would reduce the gelatinized wheat flour starch. SEM results confirmed that the fibrous thin network strands of wheat flour gels led to weak and soft gels as indicated by the low hardness in TPA. In contrary, the globular

structure of Hylon VII cause to strong and hard gel as confirmed by the textural studies. The differences in the microstructure of wheat flour and HylonVII gels were also reflected the pasting properties of the gels. As wheat flour substituted by Hylon VII, the gel structure was reinforced. As a result, the textural properties of BCG were relatively weaker than the respective SCG, particularly for Hylon VII-SCG. Cooked starch gels by increasing amylose, showed higher viscosities at higher shear rates. Consequently, BCG of WF/H develops the stronger gel which can withstand at high thermal processing such as retort to improve the shelf-life of the final product



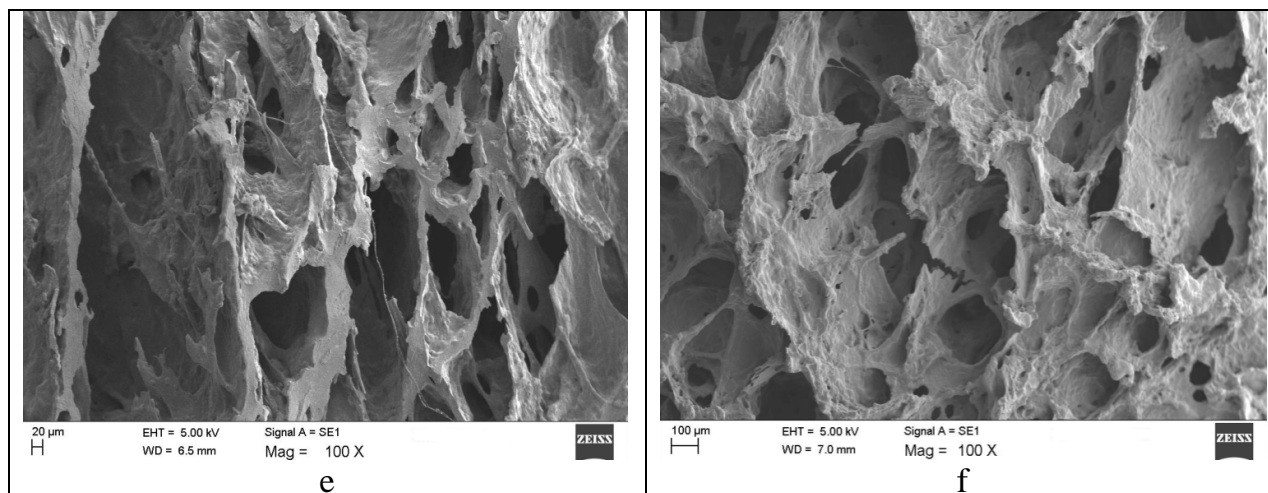


Fig. 7. SEM micrographs of the SCG at 100 °C: (a) WF , (b) H; at 121 °C: (c) WF , (d) H; BCG of (e) WF/H at ratio 85:15 at 100 °C and (f) BCG of WF/H at ratio 85:15 at 121 °C.

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## اثر دماهای بالا بر ویژگی‌های بافتی، حرارتی و ریزساختمانی ژل‌های ترکیبی آرد گندم / نشاسته ذرت آمیلوز بالا

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### چکیده

ویژگی‌های بافتی، حرارتی و ریزساختمانی ژل‌های تک جزئی نشاسته ذرت آمیلوز بالا و آرد گندم و ژل‌های دوجزئی (BCG) در نسبت‌های مختلف و دماهای 100، 121 و 135 درجه سانتی‌گراد مورد بررسی قرار گرفته است. نتایج نشان داد که ژل‌های دوجزئی با مقادیر بالای نشاسته قوی‌تر بودند. افزایش نشاسته منجر به سفتی بیشتر، کاهش فنریت، چسبندگی و پیوستگی ژل شد. افزون بر این، ژل‌های دوجزئی در دماهای بالا شاخص‌های جذب آب بالاتری را در مقادیر بالای نشاسته نشان دادند. آنتالپی ژلاتیناسیون و دمای پیک ژلاتیناسیون ژل دو جزئی با افزایش مقدار آمیلوز افزایش یافت. ژل نشاسته و پس از آن ژل دو جزئی با مقدار بالای آمیلوز، کمترین پیک ویسکوزیته را نشان دادند. اختلاف در ریزساختمان ژل‌های آرد گندم و نشاسته آمیلوز بالا خواص خمیری آنها را به خوبی منعکس می‌نماید. در نتیجه ژل دوتایی آرد گندم و نشاسته آمیلوز بالا ژل‌های قوی‌تری بودند و می‌توانند شرایط فراوری حرارتی بسیار شدید مانند استریلیزاسیون و اتوکلاو را تحمل نمایند که در نهایت به افزایش مدت ماندگاری محصول نهایی منتهی می‌شود.

واژه‌های کلیدی: ژل ترکیبی، خواص خمیری، ریزساختمان، ژلاتیناسیون، بافت