

# Physical and rheological properties of xanthan gum agglomerated in fluidized bed: Effect of HPMC as a binder

G.Y. Jeong, J.H. Bak, B. Yoo\*

Department of Food Science and Biotechnology, Dongguk University-Seoul, Goyang, Gyeonggi 410-820, Republic of Korea

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## ABSTRACT

Physical and rheological properties of agglomerated xanthan gum (XG), commonly used as a food thickener for the management of the patients with dysphagia (swallowing difficulty), were investigated at different concentrations (0, 2, 4, and 6% w/w) of hydroxypropyl methylcellulose (HPMC) as a binder in the fluidized bed agglomeration process. Flow characteristics of agglomerated XG powder were evaluated using Carr index (CI) and Hausner ratio (HR). The agglomerated XG powders obtained by HPMC binder exhibited a better flowability and higher porosity than the agglomerated powder without binder due to the size enlargement of XG powder. Dynamic moduli ( $G'$  and  $G''$ ) of agglomerated XG powders at 2% and 4% HPMC were significantly higher than those of other powders. The  $\tan \delta$  values of agglomerated powders with HPMC binder were much lower than that of an agglomerated powder without HPMC, indicating that their elastic properties were enhanced because of the addition of HPMC binder. Results suggest that the use of HPMC in agglomeration process could considerably enhance the flow characteristics and rheological properties of XG powder.

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## 1. Introduction

Fluidized bed agglomeration can be defined as the size enlargement process in which fine particles join or bind with one another, resulting in an aggregate porous structure much larger in size than the original material, such that the original particles can still be identified [1,2]. The process includes fluidizing particles with hot air, to allow their individualization and circulation, and spraying a liquid (i.e. binder solution) onto or into the fluidized bed of particles [3]. In the food industry, a major application of fluidized bed agglomeration is in the production of a wide variety of instant products, such as dairy powders (milk powders and cocoa powders), beverage powders (coffee, tea, and fruit juice powder), starch-based products (soup mix and cereal flours), and food thickeners, in which primary particles are agglomerated to give a granule with improved dispersion and dissolution compared to the original primary particles. Food thickeners, which consist mainly of gums, have been widely used to improve and control the rheological properties of food products. In particular, thickened liquids prepared with food thickeners are often used in the management of dysphagia (swallowing disorder) to improve bolus control and to help prevent aspiration for the management of the patients with dysphagia because thickened fluids move slowly through oropharynx [4–6]. Therefore, the rheological

properties of food thickeners are important in the management of the patients with dysphagia. However, it takes long times to completely disperse and hydrate the concentrated gum ( $\geq 1\%$ ) as materials of food thickeners. Commercial instant food thickeners are commonly agglomerated for quick dispersion and thickening in various liquid (water, fruit juices, milk, tea, etc.) without the formation of lumps [7]. Among food thickeners, xanthan gum (XG)-based thickeners are commonly used for the management of dysphagia due to its great pseudoplasticity and elasticity which are related to the easy and safe swallowing [5]. Studies on the rheological and physical characterization of agglomerated XG-based thickeners are important because the use of instant food thickener for the patients with dysphagia increases due to aging population [8].

The use of binders in fluidized bed agglomeration is one of the most frequent methods to modify the structure of agglomerates [9]. It is well known that the binders affect the rate of particle size enlargement, densities, and morphology of the agglomerates and that binder type and concentration play important roles in agglomeration [10]. However, different binders have different bonding efficiencies due to varied chemical compositions, mechanical properties, concentration, viscosity, and inter-particle interactions between the particles and the binders [9]. In food systems, a water-based binder solution (food gums, starches, and sugars) is applied on the particle surface to increase the adhesion forces such that the wetted particle collide and stick together [11]. It is known that the agglomeration of particles depended on the properties of binder, particularly the type of binder and the concentration of the binder solution [10]. However, no attempts have been made to

\* Corresponding author at: Department of Food Science and Biotechnology, Dongguk University-Seoul, 32 Dongguk-ro, Ilsandong-gu, Goyang, Gyeonggi 10326, Republic of Korea.

E-mail address: [bsyoo@dongguk.edu](mailto:bsyoo@dongguk.edu) (B. Yoo).

investigate the effect of gum solution as binder on the physical and rheological properties of agglomerated XG prepared by fluidized bed agglomeration. In particular, no studies have been conducted to determine the effect of hydroxypropyl methylcellulose (HPMC), which is commonly used as a binder in pharmaceutical and food applications, on physical and rheological properties of agglomerated XG. The aim of this study was to investigate the influence of HPMC binder concentration on the flow and dynamic rheological properties, and physical properties (flowability, cohesiveness, porosity, and particle size distribution), scanning electron microscopy (SEM) analysis, and morphological properties of agglomerated XG powder prepared by fluidized bed agglomeration.

## 2. Materials and methods

### 2.1. Materials and fluidized bed agglomeration process

Commercial xanthan gum (XG) powder (12.3% moisture) (CP Kelco, Atlanta, GA, USA) was used to produce agglomerated XG powder and hydroxypropyl methylcellulose (HPMC) (LOTTE Fine Chemical Co., Ltd., Incheon, Korea) was also used as a binding agent (binder) in agglomeration process. A top-spray fluidized bed granulator (Fluid Bed Lab system, Dae Ho Tech. Co., Ltd., Hwaseong, Korea) was used to carry out the agglomeration of XG powders. The experimental binder solution was prepared by dissolving HPMC at three different concentrations (2, 4, and 6% w/w) in deionized water with continuous stirring for 1 h at room temperature. A binder solution (only deionized water) with no added HPMC was also used for the preparation of agglomerated XG powder. The HPMC binder solutions were kept overnight (16 h) at 4 °C to completely hydrate before feeding to a spray nozzle. XG powder weighing 1500 g added to the chamber and fluidized by upward flowing air stream. The temperature of powder was maintained  $45 \pm 1.0$  °C by controlling inlet temperature. The flow rate of binder solution (900 ml) was set at 15 ml/min by peristaltic pump. The spray pressure was 1.2 bar. When the binding solution had been used up, the product was dried with fluidizing for 10 min.

### 2.2. Flowability and cohesiveness measurement

The flowability and cohesiveness of agglomerated powders were quantified using the Carr index (CI) [12] and Hausner Ratio (HR) [13] which represent a measure of the ability of a particulate solid to flow. The CI and HR values were calculated using the tapped ( $\rho_{\text{tapped}}$ ) and poured bulk densities ( $\rho_{\text{bulk}}$ ). The  $\rho_{\text{tapped}}$  and  $\rho_{\text{bulk}}$  were calculated as the ratio of mass/volume of powder poured into a 100 ml glass graduated cylinder respectively before and after tapping. For the  $\rho_{\text{tapped}}$  determination, the cylinder was tapped 1250 times at a rate of 200 taps/min using a tap density volumeter (BT-301, K-ONE Ltd., Seoul, Korea). Both CI and HR values were calculated from  $\rho_{\text{tapped}}$  and  $\rho_{\text{bulk}}$  of the powder as shown Eqs. (1) and (2).

$$\text{CI} (\%) = \frac{(\rho_{\text{tapped}} - \rho_{\text{bulk}})}{\rho_{\text{tapped}}} \times 100 \quad (1)$$

$$\text{HR} = \frac{\rho_{\text{tapped}}}{\rho_{\text{bulk}}} \quad (2)$$

Flowability (%) of powders based on CI was classified as follows: <15: very good; 15–20: good; 20–35: fair; 35–45: bad; >45: very bad [12]. Powders with HR < 1.2 are classified as free-flowing, while those with a HR of 1.2–1.4 were considered as intermediate flowing, and those with a HR > 1.4 as highly cohesive and non-flowing [14].

### 2.3. Porosity measurements

Agglomerated XG powder (1.0 g) was poured into a 10 ml graduated cylinder. Then, 5 ml of petroleum ether was added to suspend the powder at room temperature for 1 min, and particles on the wall of the cylinder were rinsed down with additional 1 ml of petroleum ether. The total volume of petroleum ether with suspended powder particles was determined. The particle density ( $\rho_{\text{particle}}$ ) was calculated using Eq. (3):

$$\rho_{\text{particle}} = W_p / (V_t - 6) \quad (3)$$

where,  $W_p$  is the weight of powder (g), and  $V_t$  is the total volume of petroleum ether and suspended powder (ml).

Porosity ( $\epsilon$ ) of powder was calculated by the relationship between the tapped ( $\rho_{\text{tapped}}$ ) and particle ( $\rho_{\text{particle}}$ ) densities of the powder as shown Eq. (4):

$$\epsilon = (\rho_{\text{particle}} - \rho_{\text{tapped}}) / \rho_{\text{particle}} \quad (4)$$

where, tapped density was described previously.

### 2.4. Particle size distribution

The particle size of the agglomerated XG was measured by the dry method in a Laser diffraction particle size analyzer (Mastersizer 3000E, Malvern Instruments Ltd., Worcestershire, UK). On the other hand, span was used to determine the particle size distribution as follows:

$$\text{Span} = (d_{90} - d_{10}) / d_{50} \quad (5)$$

where  $d_{10}$ ,  $d_{50}$ , and  $d_{90}$  are the particle diameters at 10%, 50%, and 90% in the cumulative size distribution, respectively.

### 2.5. Scanning electron microscope (SEM) analysis

The agglomerate were attached to aluminum stubs using two-sided adhesive carbon tape and coated under vacuum with platinum-palladium before examination by scanning electron microscope (SEM) (Hitachi S-3000N, Hitachi Ltd., Tokyo, Japan). SEM was performed at 20 kV and magnification of 120 $\times$ .

### 2.6. Preparation of thickened samples for rheological measurements

In general, the thickened fluids for the management of dysphagia were prepared by mixing the food thickener at 1–3% concentrations with the fluid foods. In this study, the gum solution was prepared by dispersing the agglomerated XG (1.0 g) with 100 ml deionized water under continuous stirring at room temperature for 2 h. After dispersion, the solution was kept overnight (16 h) at room temperature to completely hydrate the agglomerated XG powder. Each solution was prepared in triplicate.

### 2.7. Rheological properties

Flow and dynamic rheological measurements were carried out using a controlled stress rheometer (Haake RheoStress 1, Haake GmbH, Karlsruhe, Germany). The plate-plate geometry with a diameter of 35 mm was used. Each sample was loaded between the parallel plates at 25 °C and compressed up to obtain a gap of 500  $\mu\text{m}$ , and then equilibrated at 25 °C for 5 min before rheological measurements. All rheological measurements were performed in triplicate.

Flow behavior was evaluated in the controlled strain mode to obtain flow rheological data (shear stress and shear rate) over a shear rate range of 0.1–300  $\text{s}^{-1}$  at 25 °C. To describe the flow properties of the samples, the shear stress-shear rate data were fitted to the power law

model (Eq. (6)):

$$\sigma = K \cdot \dot{\gamma}^n \quad (6)$$

where  $\sigma$  is the shear stress (Pa),  $\dot{\gamma}$  is the shear rate ( $s^{-1}$ ),  $K$  is the consistency index ( $Pa \cdot s^n$ ), and  $n$  is the flow behavior index (dimensionless).

Dynamic viscoelastic properties were evaluated using small amplitude oscillatory rheological measurements. All samples were analyzed by frequency sweeps which were performed between 0.63 and 62.8  $rad\ s^{-1}$  of angular frequency ( $\omega$ ) at 2% strain value in order to determine the storage (or elastic) modulus ( $G'$ ), loss (or viscous) modulus ( $G''$ ), and loss tangent ( $\tan \delta$ ;  $G''/G'$ ). All samples also remained between the plates for 5 min (rest period) before measurements. All tests were carried out in triplicates.

### 2.8. Statistical analysis

Statistical significance was assessed by Analysis of variance (ANOVA) and Duncan's multiple range test for significant differences using Statistical Analysis System (SAS) software version 9.2 (SAS Institute, Cary, USA). The level of significance was set at  $p < 0.05$ .

## 3. Results and discussion

### 3.1. Particle diameter and size distribution

The particle size values of agglomerated XG powders are summarized in Table 1. The particle diameter significantly increased ( $p < 0.05$ ) with an increase in the HPMC binder concentration. This may be due to the increased viscosity and particle coating by the HPMC binder. The greater feed viscosity can contribute to the formation of larger droplets during atomization, consequently increasing the particle size [15]. The span values of agglomerated XG powders with different binder concentrations were in the range of 1.22–1.47, and the lower span value (1.22) was observed at a 2% binder concentration, indicating a narrow distribution. The lower span value also indicates a lower breakage rate of particles. In contrast, higher span values, which may be caused by the mixing of fine particles with large particles and the breakage of the particles during the agitation, indicate a wide size distribution and a high polydispersity [16]. This result showed that the higher binder concentration led to the formation of larger particles. From these observations, it was found that HPMC binder concentrations affected both the particle sizes and size distributions of the resultant agglomerated XG powders.

### 3.2. Flow characteristics

The porosity, Carr index (CI), and Hausner ratio (HR) of agglomerated XG powders with different binder concentrations are shown in Table 2. The CI and HR values of agglomerated powders decreased with an increase in the binder concentration from 0 to 6%, indicating that flowability and cohesiveness were significantly affected by the

**Table 1**

Particle sizes and distributions for agglomerated XG powders with different HPMC binder concentrations.

Concentration (%)	$D_{10}$ ( $\mu m$ ) <sup>a</sup>	$D_{50}$ ( $\mu m$ ) <sup>a</sup>	$D_{90}$ ( $\mu m$ ) <sup>a</sup>	Span
0	$63.4 \pm 1.27^d$	$126 \pm 2.08^d$	$225 \pm 2.89^d$	$1.28 \pm 0.01^b$
2	$74.8 \pm 1.63^c$	$144 \pm 0.58^c$	$251 \pm 3.79^c$	$1.22 \pm 0.04^c$
4	$118 \pm 3.21^b$	$250 \pm 2.31^b$	$485 \pm 7.77^b$	$1.47 \pm 0.01^a$
6	$171 \pm 2.31^a$	$382 \pm 1.00^a$	$731 \pm 3.06^a$	$1.46 \pm 0.01^a$

Values are means of three measurements  $\pm$  SD.

Means with different lowercase letters (a–d) within each column are significantly different ( $p < 0.05$ ).

<sup>a</sup>  $d_{50}$ ,  $d_{10}$ ,  $d_{90}$  are values of the particle diameter at 50%, 10%, and 90% in the cumulative size distribution, respectively.

HPMC binder addition and concentration. Agglomerated XG powders with HPMC (2, 4, and 6% HPMC) exhibited a better flowability and lower cohesiveness compared to the agglomerated powder without HPMC. In particular, all powders exhibited a fair flowability (20–35 CI) as classified by the CI values, indicating that the fluidized-bed agglomeration in the presence of HPMC binder improved the flowability of agglomerated XG powder due to its large size of agglomerated powder. The cohesiveness of agglomerated powders decreased with an increase in particle size, demonstrating better flowability with the intermediate cohesiveness (1.2–1.4 HR). These results showed that binder addition and higher binder concentration improved the flowability and cohesiveness of XG powder, indicating that the size enlargement of XG by fluidized bed agglomeration prepared with HPMC binder had a major influence on powder flow characteristics.

Porosity ( $\epsilon$ ) is defined as the void fraction in the powder sample. Porosity increased with increasing binder concentration. Agglomerated powders containing smaller particles, i.e. those prepared with lower binder concentrations, showed significantly higher  $\rho_{bulk}$  and  $\rho_{tapped}$  (Table 2) but lower porosities than those with larger particles, i.e. those prepared with higher binder concentrations. The porosity of agglomerates with higher binder concentrations was significantly higher than that of the agglomerates with lower binder concentrations. This may be attributed to the large droplet size of XG with a HPMC binder at higher concentrations, which could lead to the formation of large globular clusters of XG powder. There were no noticeable changes in  $\rho_{particle}$  values among the agglomerated XG powders prepared with different binder concentrations (Table 2), indicating that they had similar density of agglomerates [1].

### 3.3. Morphology

Fig. 1 shows the SEM images of the non-agglomerated (a) and agglomerated XG powder samples (b–e) with different binder concentrations. The particle size was larger after agglomeration; thus, the non-agglomerated powder had a small and compact particle, whereas the agglomerated powder had large, porous, and irregularly shaped particles. Agglomerated food powders can vary in size, shape, and structure [17]. The particle sizes of agglomerated XG powders (c–e) with HPMC were much larger than that of agglomerated powders without HPMC (b), indicating that individual agglomerates may be held together by the HPMC binder and XG powder. Their size also increased with an increase in the binder concentration from 2 to 6%. Overall, binder addition and concentration had noticeable effects on the shape and size of the agglomerates, demonstrating that the agglomerated XG powders prepared with a HPMC binder had a large, porous, and irregularly shaped particles.

### 3.4. Rheological properties

The experimental results of shear stress ( $\sigma$ ) versus shear rate ( $\dot{\gamma}$ ) well fitted the power law model (Eq. (6)) with high  $R^2$  (0.98–0.99) (Table 3). All samples exhibited shear-thinning behavior with flow behavior index values ( $n = 0.15$ – $0.17$ ). The high shear-thinning behavior may be attributed to the unique rigid, rod-like conformation and high molecular weight of XG [10]. The  $n$  values (0.15) of agglomerated XG powders with HPMC binder were lower than that (0.17) of the agglomerated powder without HPMC, indicating that the agglomerated XG powder with HPMC binder was a less sticky and had better mouthfeel than the agglomerated powder without HPMC. It is known that a high  $n$  value results in a sticky mouthfeel, whereas a low  $n$  value results in a smooth mouthfeel [18]. However, there were no noticeable changes in consistency index ( $K$ ) values among the agglomerated XG powders with different binder concentrations, indicating that the concentration of HPMC did not affect the consistency of the agglomerated XG powders with HPMC.

**Table 2**  
Porosity and flow characteristics of agglomerated XG powders with different HPMC binder concentrations.

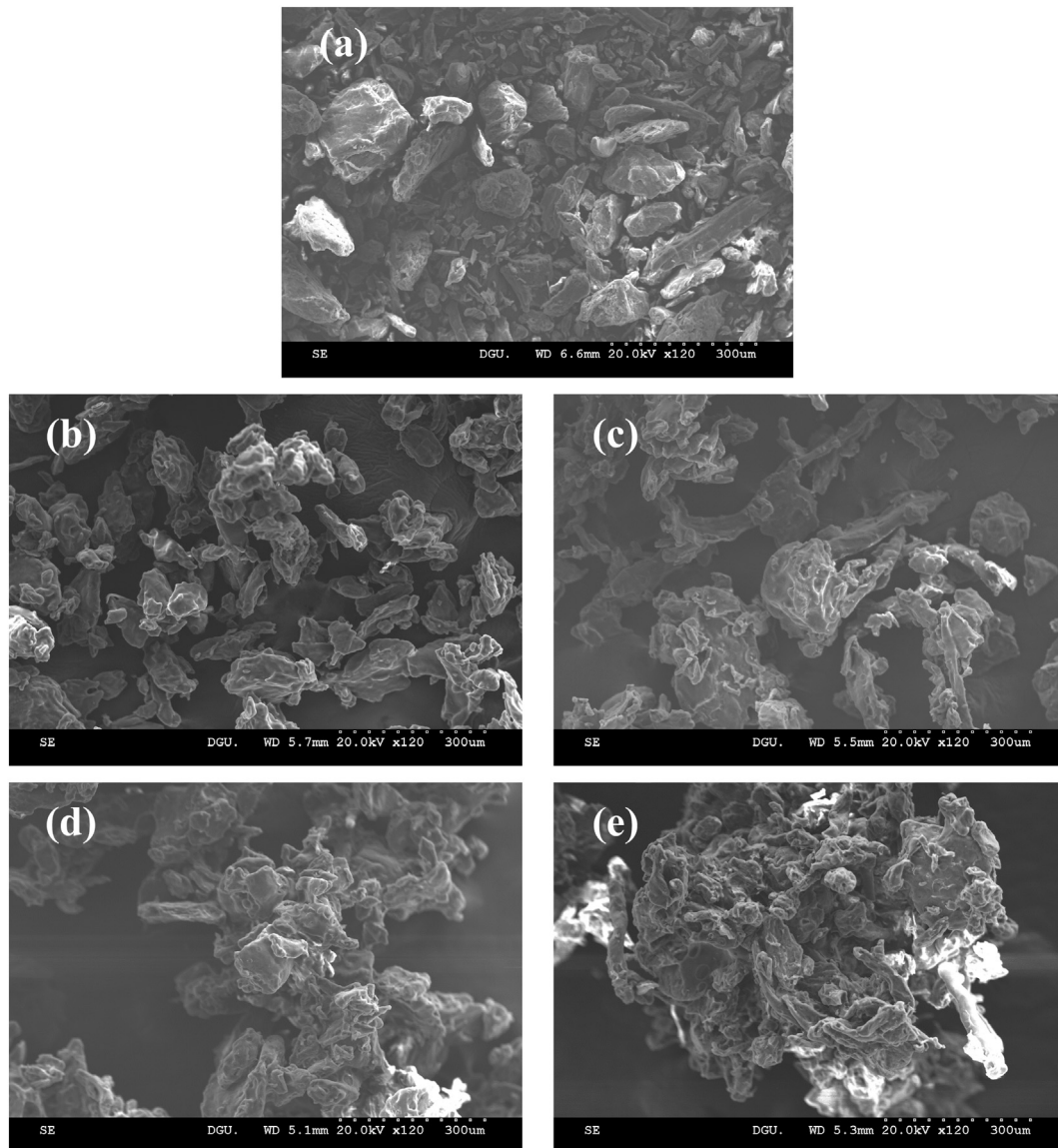
Concentration (%)	$\rho_{\text{bulk}}$ (g/cm <sup>3</sup> )	$\rho_{\text{tapped}}$ (g/cm <sup>3</sup> )	$\rho_{\text{particle}}$ (g/cm <sup>3</sup> )	Porosity (%)	CI (%)	HR
0	0.44 ± 0.00 <sup>a</sup>	0.58 ± 0.00 <sup>a</sup>	1.67 ± 0.00 <sup>a</sup>	65.0 ± 0.00 <sup>d</sup>	25.1 ± 0.16 <sup>a</sup>	1.33 ± 0.00 <sup>a</sup>
2	0.40 ± 0.01 <sup>b</sup>	0.52 ± 0.01 <sup>b</sup>	1.67 ± 0.00 <sup>a</sup>	69.0 ± 0.00 <sup>c</sup>	22.4 ± 0.39 <sup>b</sup>	1.29 ± 0.01 <sup>b</sup>
4	0.27 ± 0.00 <sup>c</sup>	0.35 ± 0.00 <sup>c</sup>	1.67 ± 0.00 <sup>a</sup>	79.2 ± 0.00 <sup>b</sup>	22.0 ± 0.26 <sup>b</sup>	1.28 ± 0.00 <sup>b</sup>
6	0.21 ± 0.00 <sup>d</sup>	0.27 ± 0.00 <sup>d</sup>	1.62 ± 0.10 <sup>a</sup>	83.3 ± 1.21 <sup>a</sup>	21.2 ± 0.24 <sup>c</sup>	1.27 ± 0.00 <sup>c</sup>

Values are means of three measurements ± SD.

Means with different lowercase letters (a–d) within each column are significantly different ( $p < 0.05$ ).

Viscoelastic properties are an important factor for evaluating XG-based food thickeners used for people with swallowing difficulty. The storage modulus ( $G'$ ) and loss modulus ( $G''$ ) of agglomerated XG powders with different HPMC concentrations are also shown in Table 3. The dynamic moduli ( $G'$  and  $G''$ ) of agglomerated XG powders with HPMC were much higher than those of the agglomerated XG powders without HPMC, indicating that the addition of HPMC in the agglomeration process had a synergistic effect on the viscoelastic properties of the agglomerated powder, which may be attributed to better intermolecular interaction between HPMC and XG. In particular, the dynamic moduli ( $G'$ : 18.2 Pa;  $G''$ : 5.37 and 5.44 Pa) of agglomerated powders at 2% and

4% binder concentrations were relatively higher than those of other agglomerated powders, indicating the marked effect of HPMC on viscoelastic properties. Indeed, the dynamic behavior of thickened agglomerated powder with HPMC may be affected by binder material properties, as reported by Li et al. (2012) [19]. The  $\tan \delta$  ( $G''/G'$ ) value (0.30) of the agglomerated powders with 2%, 4%, and 6% HPMC was lower than that (0.34) of the agglomerated powder with 0% HPMC, demonstrating that  $\tan \delta$  decreased with the addition of HPMC. However, no significant change in  $\tan \delta$  (0.30) was observed among the agglomerated powders (2–6% HPMC) that had a synergistic HPMC effect on the viscoelastic properties of XG powder (Table 3). The greater



**Fig. 1.** SEM micrograph for the non-agglomerated XG powder (a) and agglomerated XG powders (b–e) prepared with different HPMC binder concentrations. (b) 0% HPMC, (c) 2.0% HPMC, (d) 4.0% HPMC, (e) 6.0% HPMC. Magnification 120 $\times$ .

**Table 3**

Flow and dynamic rheological properties of agglomerated XG powders with different HPMC binder concentrations.

Conc. (%)	K (Pa·s <sup>n</sup> )	n (–)	G' (Pa)	G'' (Pa)	tan δ
0	7.58 ± 0.18 <sup>a</sup>	0.17 ± 0.01 <sup>a</sup>	12.2 ± 0.26 <sup>c</sup>	4.10 ± 0.03 <sup>c</sup>	0.34 ± 0.01 <sup>a</sup>
2	7.65 ± 0.12 <sup>a</sup>	0.15 ± 0.00 <sup>b</sup>	18.2 ± 0.52 <sup>a</sup>	5.44 ± 0.04 <sup>a</sup>	0.30 ± 0.01 <sup>b</sup>
4	7.69 ± 0.19 <sup>a</sup>	0.15 ± 0.01 <sup>b</sup>	18.2 ± 0.23 <sup>a</sup>	5.37 ± 0.03 <sup>a</sup>	0.30 ± 0.00 <sup>b</sup>
6	7.55 ± 0.15 <sup>a</sup>	0.15 ± 0.01 <sup>b</sup>	16.8 ± 0.47 <sup>b</sup>	5.01 ± 0.07 <sup>b</sup>	0.30 ± 0.01 <sup>b</sup>

Values are means of three measurements ± SD.

Means with different lowercase letters (a–d) within each column are significantly different ( $p < 0.05$ ).

change in  $G'$  than in  $G''$  indicated that the addition of HPMC increased the solid character of agglomerated XG powder and effectively contributed to the elastic properties of agglomerated powder, improving rheological quality for the agglomerated XG-based food thickener. These observations suggest that the viscoelastic properties of agglomerated XG powder were strongly affected by HPMC binder addition.

#### 4. Conclusions

The physical and rheological properties of agglomerated XG powder prepared using HPMC as a binder in the fluidized bed agglomeration process were evaluated. These properties were compared between agglomerated XG powders at different binder concentrations. The agglomeration process with HPMC binder produced a larger, porous, and irregularly shaped particles with improved flowability and cohesiveness. The results of physical and rheological analysis suggest that careful agglomeration is required to prepare instant XG-based food thickeners with desirable mouthfeel for the patients with dysphagia when HPMC is used as a binder. In particular, the larger size of agglomerate prepared with HPMC at higher concentrations could result in viscoelastic properties with higher dynamic moduli ( $G'$  and  $G''$ ) values. This effect may be attributed to better intermolecular interaction between HPMC and XG during the agglomeration process. The knowledge of the specific rheological properties of agglomerated XG powders prepared with HPMC binder would be useful in developing formulation of XG-based food thickeners for patients with dysphagia. Further studies on various types of gum-based binders are needed to expand on the results of this study.

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