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Physicochemical properties of extrusion-modified konjac glucomannan

Orawan Tatirat[a], Sanguansri Charoenrein[a], William L. Kerr[b,*]

[a] Department of Food Science and Technology, Faculty of Agro-Industry, Kasetsart University, Bangkok, Thailand
[b] Department of Food Science and Technology, College of Agricultural and Environmental Science, University of Georgia, Athens, GA 30602 USA

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ABSTRACT

Konjac glucomannan was extruded and subsequently ground under four conditions denoted: KGM1 (33% solids, 90 °C), KGM2 (24% solids, 90 °C), KGM3 (24% solids, 90 °C, die restriction), and KGM4 (24% solids, 110 °C). SEM and particle size analysis showed that extruded KGM had slightly larger and rougher particles. The water absorption index was decreased to 47.92–128.80, as compared to 153.64 for the control (KGMC). The crystallinity index increased to 2.97 and 3.42 for KGM3 and KGM4 samples, as compared to 1.72 for the control. Zero-shear viscosity of 0.5% solutions decreased to 0.36–3.01 Pa s. All samples were shear-thinning and data were best fitted by the Cross model. Based on capillary viscometry, molecular weights were decreased to 2.7×10^5–9.9×10^5, as compared to 1.2×10^6 for the control. In most cases, properties were most altered by higher temperature and shear, and to a lesser extent by lower solids in the feed.

1. Introduction

Konjac glucomannan (KGM) is a major component of the konjac tuber (*Amorphophallus konja*). KGM is a neutral heteropolysaccharide composed of β-1,4 linked D-mannose and D-glucose in the ratio of 1.6:1. There are short branches at some of the C-3 positions and 5–10% of the glucomannan backbone has *O*-acetyl substitutions at the C-6 position (Li, Xie, & Kennedy, 2006; Takigami, 2000). KGM has a relatively high molecular weight (M_w) of $\sim 10^5$–10^6, while the degree of crystallinity is low (Li & Xie, 2003; Xiaoyan et al., 2010). Dispersions of KGM (0.5%) had the highest viscosity amongst 12 polysaccharides tested, and exhibit shear-thinning behavior (Yaseen, Herald, Aramouni, & Alavi, 2005). Moreover, KGM has very high water absorbency, absorbing as much as 100 g of water per g of sample (Koroskenyi & McCarthy, 2001). Strong elastic gels are formed with concentrations of KGM greater than 2% in the presence of alkali (Huang, Takahashi, Kobayashi, Kawase, & Nishinari, 2002).

KGM is generally recognized as safe (Takigami, 2000) and has been shown to provide several health benefits. It is not hydrolyzed by digestive enzymes in humans, and as it promotes healthy micoflora may be considered as a prebiotic (Chen, Fan, Chen, & Chan, 2005). It has also been demonstrated to reduce cholesterol levels (Martino, Martino, Carnevali, Forcone, & Niglio, 2005) and associated risk factors for heart disease in type 2 diabetes (Vuksan, Jenkins, Spadafora, Sievenpiper, & Owen, 1999). KGM is used in various applications including drug delivery, as part of coating and encapsulation materials, as an emulsifier and in cosmetic ingredients (Zhang, Xie, & Gan, 2005). It has also been used as a gelling and bulking agent in foods, and as a stool softener. While KGM has substantial promise for food, health and nutraceutical products, there is some concern with choking risks related to high water absorption of the powder. In addition, it would be useful to have broader range of KGM properties available. Thus, it would be helpful to have methods for modifying KGM to alter water absorption, viscosity or other properties. Chemical modifications of KGM have been done, including acetylation, methylation, and oxidation (Gao & Nishinari, 2004; Kishida, Okumasu, & Kamata, 1978). However, these procedures are somewhat involved and have a limited throughput. Complex physical methods of modification have also been attempted including sonication, irradiation, and pressure–temperature treatments (Samil, Abdelhameed, Ang, Morris, & Harding, 2009).

Extrusion is commonly used to produce regular shaped food, polymer, pharmaceutical and even metal products in a system that provides excellent control over temperature, pressure and shear conditions. It is also known to modify material structure and can be used as a reactor for polymerization and other chemical reactions in a process known as reactive extrusion. It is a very practical and efficient way to modify food polysaccharides (Wolf, 2009), as by breaking polymer chains or increasing resistance to starch digestion. There are three broad areas of research on the extrusion-modification of food ingredients. Extrusion of starch, cereal grains and other starchy materials is widespread, and substantial research has been conducted on the modification of starch during extrusion

* Corresponding author. Tel.: +1 706 224 0461; fax: +1 706 542 1050.
E-mail address: wlkerr@uga.edu (W.L. Kerr).

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(Davidson, Paton, Diosady, & Larocque, 1984; Gomez & Aguilera, 1983; Sriburi, Hill, & Barcly, 1999). A second area of study involves co-extrusion of starch with non-starch polysaccharides. For example, Adamu (2001) extruded cornstarch with guar gum in the presence of specific additives, and was able to increase the amount of resistant starch. Finally, only a few studies have been done on the use of extrusion cooking to modify non-starch polysaccharides. For example, Cote and Willet (1999) studied ultrasonication, high pressure jet-steam cooking and twin-screw extrusion to reduce the molecular weight and viscosity of dextran. Sereno, Sandra, and Mitchell (2007) used extrusion and drying to produce a more particulate xanthan biopolymer with modified rheological properties.

The objective of this research was to investigate the use of extrusion cooking for modifying KGM. A key interest was to reduce water absorption and swelling that may contribute to choking risks when powdered samples are ingested. This work also examined the effects of modifying KGM on rheological behavior and molecular structure. Based on previous studies, four different extrusion conditions were examined and the effects on several physical properties were examined including particle structure, water absorption, soluble solids, crystallinity, flow rheology, molecular weight, and ability to form gels.

2. Materials and methods

2.1. Materials

KGM with 92.6% glucomannan content was purchased from DKSH (Thailand) Limited. Commercial grade ethanol (95% purity) was mixed with distilled water as needed for extrusion.

2.2. Extrusion conditions

Extrusion was performed in a co-rotating twin-screw extruder (Hermann Berstorff Laboratory, ZE25×33D), with a length to diameter ratio of 33:1. Extrusion was done both with and without an exit die. The diameter of the exit hole was 30 mm. When used, the die had 4 square holes, each 3 by 3 mm. The barrel contained seven sections. Temperature profiles in the first (feed end) to fifth zones were kept at 30, 40, 50, 70 and 90 °C. The temperature of the sixth zone was set at 90 or 110 °C, and the temperature of seventh zone was kept at 10 °C below that of the sixth zone. Konjac glucomannan was fed into the feed hopper and mixed with 20% ethanol to give the desired solids content. Preliminary studies showed that KGM could not be successfully extruded with water only. At relatively low solids content (20%, w/w), a multitude of lumps was observed in the mixture and it was so thick that it was difficult to operate the screw in a continuous manner. At higher solids content (~80–90%), the extruded KGM burned when using only water as solvent. In 20% ethanol, however, the mixture yielded a coarse texture that was suitable for extrusion without slipping. Four extrusion conditions (in addition to the control) were considered based on preliminary studies, in which response surface methodology was used to analyze KGM water absorption properties. Conditions were chosen so as to give a broad range of WAI. These included (Table 1): KGMC – non-extruded control; KGM1 – 33% solids, 90 °C, no die; KGM2 – 24% solids, 90 °C, no die; KGM3 – 24% solids, 90 °C, with die; and KGM4 – 24% solids, 110 °C, no die. Extruded KGM samples were dried at 45 °C for 15 h, then ground with a mortar and pestle and sieved to ~150 μm through a 100 mesh screen. KGM powder was sealed with silica gel pouches in polyethylene-aluminum laminate bags.

2.3. Scanning electron microscope

Scanning electron microscopy (SEM) was conducted on a JEOL (JSM-5410LV) microscope (JEOL, Ltd., Tokyo, Japan) at an acceleration voltage of 15 kV. Dried KGM samples were sprayed on stubs previously covered with double-sided adhesive tape and then coated with gold. Various regions of each sample were examined, and morphological features were photographed at a magnification of 200× or 500×.

Table 1
Extrusion conditions at different solids content and barrel temperature[a]. Screw speed was fixed at 300 rpm.

Condition names	Extrusion conditions		
	Solids content (%)	Barrel temperature (°C)	Die insert
KGMC	–	–	–
KGM1	33	90	Without die
KGM2	24	90	Without die
KGM3	24	90	With die
KGM4	24	110	Without die

[a] Temperature of barrel in sixth heating zone.

2.4. Determination of particle size

Particle sizes were determined using a Mastersizer 2000 equipped with a Scirocco 2000 dry powder feeder (Malvern Instrument Ltd., UK). Dried sample (3–5 g) was blown into the sealed flow cell into the path of the light scattering laser. The sizes were reported as both average particle size and particle size distribution.

2.5. Determination of WAI and WSI

Water absorbency and solubility properties were measured by the water absorption index (WAI) and water solubility index (WSI). The method was based on that of Anderson, Conway, Pfeifer, and Griffin (1969) with some modification. A sample (0.150 ± 0.001 g dry basis) was weighed and dispersed with 30 ml distilled water into a 50 ml centrifuge tube containing a magnetic stir bar. The mixture was vortexed and inverted to break any clumps. The suspension was stirred on a magnetic stir plate for 30 min. After removing the stir bar, the mixture was centrifuged at 1500 × *g* for 15 min. Finally, the supernatant fraction was poured into a tared metal canister and dried at 80 °C for 24 h. The weight of the dried material represented the soluble solids. The tube with the remaining sediment was weighed after the supernatant was removed. The WAI and WSI were calculated by:

$$\mathrm{WAI} = \frac{\text{wt of sediment} - \text{wt of solids in supernatant}}{\text{wt of total solids} - \text{wt of solids in supernatant}} \quad (1)$$

$$\mathrm{WSI} = \frac{\text{wt of solids in supernatant}}{\text{wt of total solids}} \quad (2)$$

2.6. Determination of crystallinity

Crystallinity of the dried samples was determined using an X'Pert Pro X-ray diffractometer (XRD) (PANalytical, Singapore). The tube voltage and current were 40 kV and 30 mA, respectively. Powder samples were scanned continuously at a rate of 5.00° min^{-1} in the range of 5–60°. The crystallinity index (the proportion of crystalline to non-crystalline material) was calculated from the peak height (I_p) divided by the full width at half height ($w_{1/2}$) (Herdianita, Browne, Rodgers, & Campbell, 2000):

$$X_c = \frac{I_p}{w_{1/2}} \quad (3)$$

The counts per second at peak height were also reported.

2.7. Determination of flow behavior

Flow behavior of KGM samples was determined using a SR5000 dynamic rheometer (Rheometrics Scientific, Piscataway, NJ) equipped with a temperature-controlled Couette cell. The sample (0.5%, w/w) was dissolved in deionized water at room temperature (~25 °C) for 15 h. Viscosity data was collected at shear rates from 0.1 to 1000 s^{-1} at a constant temperature of 30 °C. The apparent viscosity (η) versus shear rate ($\dot{\gamma}$) data were best fit by the Cross model:

$$\eta = \frac{\eta_o}{[1 + (K\dot{\gamma})^{1-n}]} \quad (4)$$

where η_o is the zero viscosity extrapolated at zero shear; K is a constant associated with breaking of structural linkages; n is a dimensionless constant.

2.8. Determination of intrinsic viscosity and molecular weight (M_w)

The intrinsic viscosity and molecular weight of the KGM samples was determined using a Model 0C Ubbelohde viscometer (Cannon Instrument Co., State College, PA). Samples were prepared by dissolving KGM into deionized water to a concentration of 0.05 g/dL and mixing at room temperature of 25 °C for 15 h. The dispersion was centrifuged at 1500 × g and the supernatant fraction used to make dilutions of 0.04, 0.03, 0.02 and 0.01 g/dL. The kinematic viscosity (η/ρ) was calculated by multiplying the flow time by the capillary tube constant. The relative viscosity (η/η_o) was determined by comparison with the measured viscosity for pure water (η_o). The reduced viscosity was calculated as $[((\eta/\eta_o) - 1)c]$. Finally, the reduced viscosity was plotted against the concentration (c), and the intrinsic viscosity $[\eta]$ determined by extrapolating to zero concentration. Molecular weights (M_w) for the KGM samples were determined using the Mark–Houwink equation, with constants as determined by Kishida et al. (1978):

$$[\eta] = 6.37 \times 10^{-4} M^{0.74} \quad (5)$$

2.9. Statistical analysis

A completely randomized block design was used in this study. Determinations of particle size, WAI, WSI, flow behavior, intrinsic viscosity, M_w and XRD were performed in duplicate. Multifactorial analysis of variance (ANOVA) was performed using SPSS 12.0 for Windows (Somers, NY).

3. Results and discussion

3.1. Effect of extrusion conditions on morphology and particle size

Scanning electron micrographs of non-extruded and extruded KGM samples are presented in Fig. 1. Non-extruded samples did not show a regular granular structure. While konjac flour normally consists of white oval-shaped sacs, the granule is broken during the process of KGM extraction (Ohashi, Shelso, Moirano, & Drinkwater, 2000; Takigami, 2000). After extrusion, the surface roughness of all extruded samples increased, and those undergoing higher temperature and shear had the most convoluted surfaces. Particles produced at lower temperature (and with no die) had sharper edges and less surface indentations. Studies on modified starch have shown that resultant particles also exhibit increased roughness after extrusion (Lopez-Rubio, Htoon, & Gilbert, 2007). No studies have been reported on KGM or similar hydrocolloids.

The particle size distribution of the control and extruded samples is shown in Fig. 2. The average peak particle size of

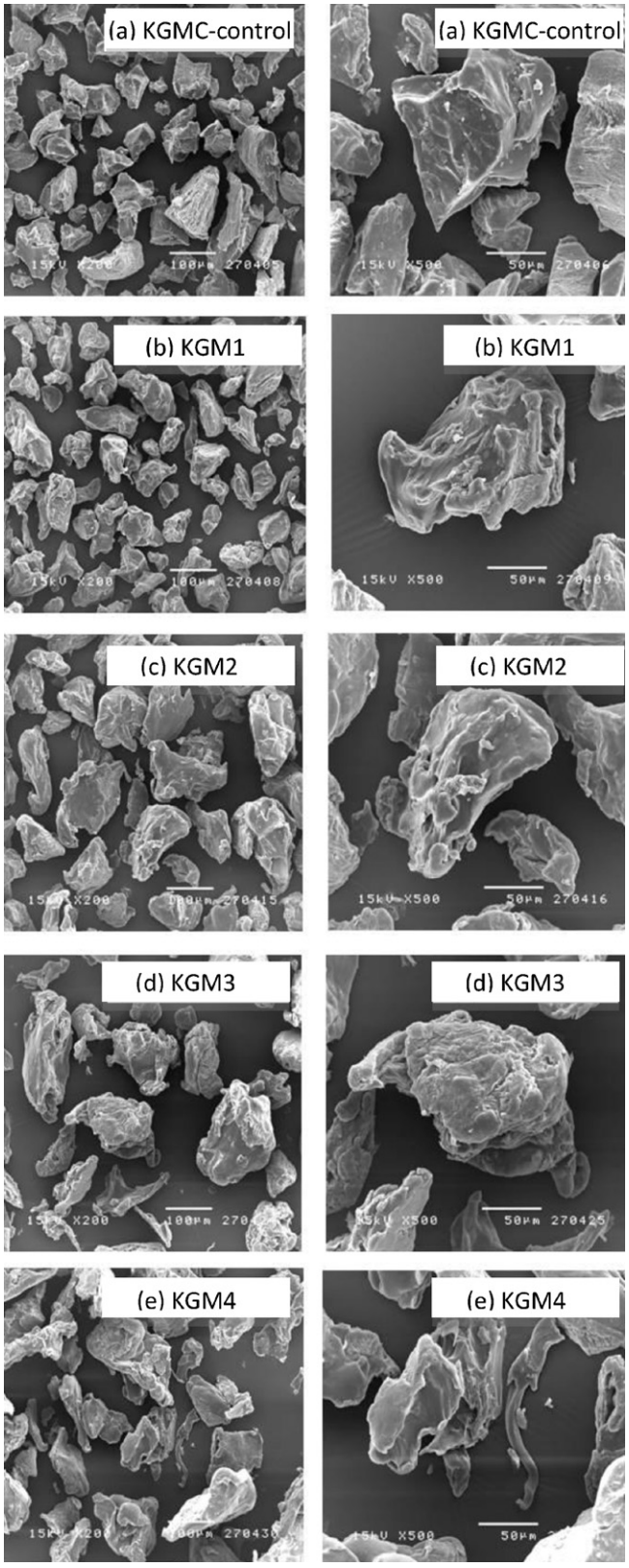

Fig. 1. SEM photographs of extrusion-modified KGM powder at 200× (left column) and 500× (right column): (a) non-extruded (KGMC), (b) KGM1, (c) KGM2, (d) KGM3, and (e) KGM4.

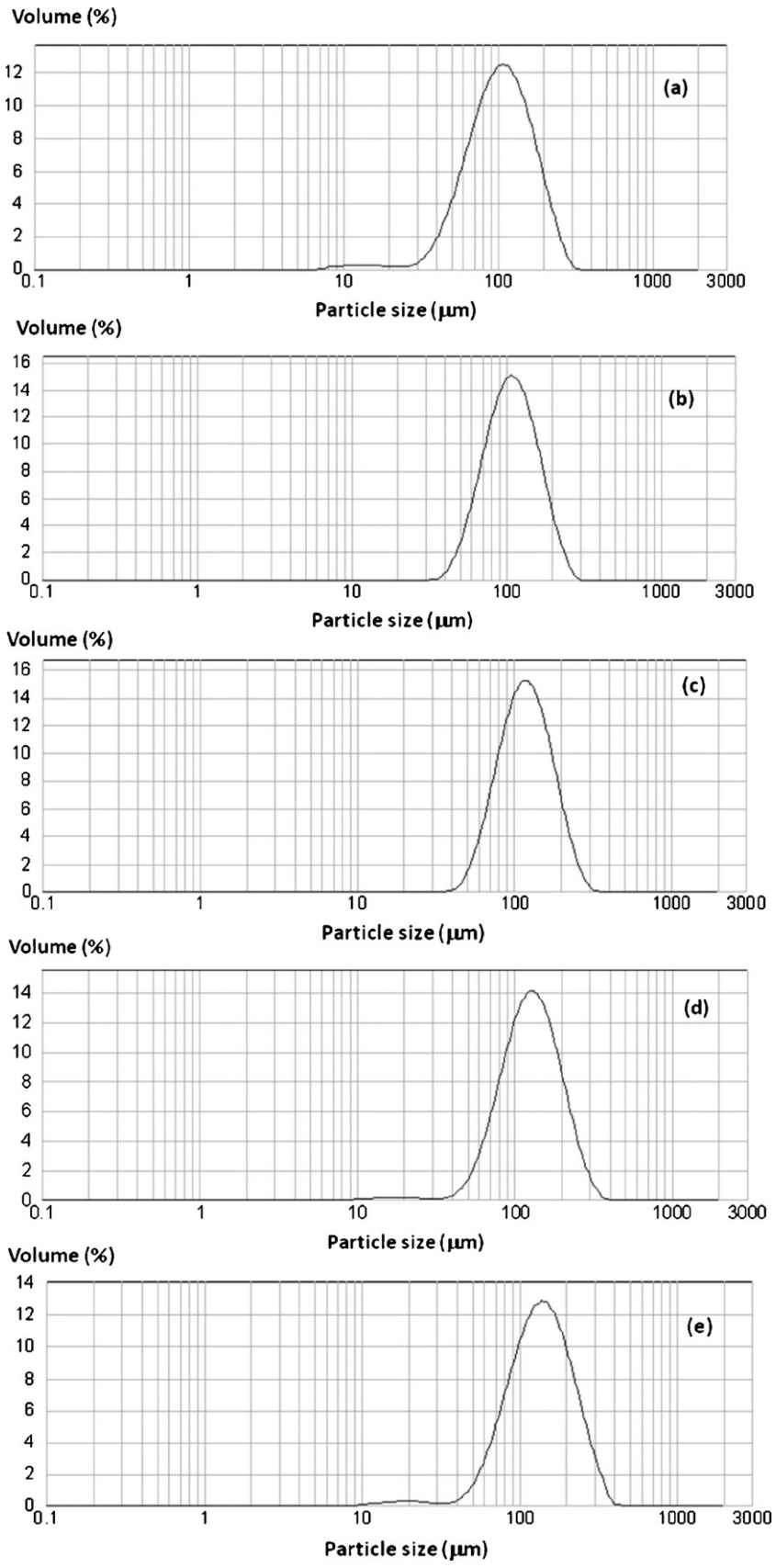

Fig. 2. Particle size distribution of extrusion-modified KGM powder: (a) non-extruded (KGMC), (b) KGM1, (c) KGM2, (d) KGM3, and (e) KGM4.

Table 2
WAI and WSI values of extrusion-modified KGM.

Samples	WAI	WSI
KGMC	153.64 ± 4.70e	0.71 ± 0.02e
KGM1	128.80 ± 2.31d	0.63 ± 0.02d
KGM2	85.71 ± 1.32c	0.47 ± 0.01c
KGM3	47.92 ± 1.32a	0.33 ± 0.01a
KGM4	55.49 ± 1.27b	0.37 ± 0.01b

Values in a column followed by different letters are significantly different ($p < 0.05$).

Table 3
Crystallinity index and peak intensity for extrusion-modified KGM.

Samples	Crystallinity index	Peak intensity (counts/s)
KGMC	1.72 ± 0.04a	527 ± 5a
KGM1	1.69 ± 0.03a	500 ± 15a
KGM2	1.77 ± 0.05a	534 ± 3a
KGM3	2.97 ± 0.12b	589 ± 6b
KGM4	3.42 ± 0.87b	613 ± 40b

Values in a column followed by different letters are significantly different ($p < 0.05$).

non-extruded samples was 112.4 μm. Those for KGM1, KGM2, KGM3 and KGM4 were 116.1 μm, 126.4 μm, 136.4 μm and 144.3 μm, respectively. Particles in the latter three groups were slightly, though significantly, larger than those of the control. The shape and roughness of KGM1 and KGM2 particles were not notably different. As these groups were processed at 33% and 24% solids and at the same temperature, the decrease in solids did not affect particle morphology. However, the average size of KGM2 particles was bigger than KGM1 particles. The non-extruded group also had a small fraction of particles at ~10 μm. This fraction was not present for samples extruded at 90 °C and without a die, but smaller particles were evident in samples produced with the die and at 110 °C. Evidence of relatively small particle fragments was also seen in the SEM micrographs.

3.2. Effect of extrusion conditions on WAI, WSI

The effects of extrusion conditions on water absorption index (WAI) and water solubility index (WSI) are shown in Table 2. The WAI ranged from 153.64 for non-extruded KGM to 47.92 for KGM3 samples. In general water absorption decreased for samples extruded at higher shear and temperature conditions. The WSI ranged from 0.71 for non-extruded KGMC to 0.33 for KGM3. Thus, the amount of water-soluble solids decreased with higher temperature and shear conditions. WAI and WSI values were substantially less for samples from the KGM2 group (85.71 and 0.47) as compared to those from the KGM1 group (128.8 and 0.63). This indicates that decreased solids content of the feed had a profound effect on the water absorption and solubility properties of the product. It should be noted that ethanol was used with water in the feed stream as a plasticizer. Thus, decreased solids content would coincide with an increase in the ethanol/water fraction, and presumably a more mobile and less viscous paste. At a given screw rpm, this allows for greater mixing and shearing of the mix. Indeed, results in a subsequent section show that the average molecular weight of KGM2 extruded at 24% solids and 90 °C was lower than that of KGM1 extruded at 33% solids and 90 °C. One theory is that WAI for KGM2 samples is lower, as shorter polymer chains are less able to become entangled and entrap water. Another possibility is that both WAI and WSI are decreased due to deacetylation produced by lower solids and higher barrel temperatures during extrusion.

Increased extrusion temperature produced an even greater change in WAI and WSI. Changing the extrusion temperature from 90 °C (KGM2) to 110 °C (KGM4), and without the die, decreased WAI from 85.71 to 55.49, and WSI from 0.47 to 0.37. Inclusion of the die had a greater influence than even temperature. At 90 °C, inclusion of the die lowered the WAI to 47.92 and the WSI to 0.33. Presumably, incorporation of the die created even greater shear conditions, and indeed the extruder pressure was much greater when the die was incorporated.

As mentioned previously, there are several health benefits and possible applications of KGM, but high water absorption (as much as 100 g water/g dry KGM) has been a concern (Chen et al., 2005; Koroskenyi & McCarthy, 2001). Thus extrusion is a good alternative for producing KGM with lower WAI in a controlled manner.

3.3. Effect of extrusion conditions on crystallinity

To gain further understanding of the effects of extrusion on the properties of KGM, X-ray diffraction patterns were collected for the modified KGM powders. All samples had a peak at approximately $2\theta = 20°$. KGM3 and KGM4 samples had somewhat narrower peaks, indicative of increased crystallinity. This is shown by the crystallinity indices (Table 3). For KGMC, KGM1 and KGM2 samples, the crystallinity index (X_c) varied from 1.69 to 1.77, though there were no significant differences in X_c or peak intensity (500–534 counts/s) amongst the treatment groups. For KGM3 and KGM4 samples, the crystallinity was greater (2.97 and 3.42) and peak intensity was greater (589 and 613 counts/s).

Glucomannans exist in nature as part of the plant cell wall, particularly in softwoods and bulbs. It is a part of the hemicellulose polysaccharides that hold the crystalline cellulose myofibrils in place, and may eventually serve as an energy reserve for the plant. Glucomannans do not have the extensive crystalline structure of cellulose, but several studies have shown that myofibrils and crystallinity exist *in situ* (Yui, Ogawa, & Sarko, 1992). Studies on stretched hydrated films show that KGM crystallizes in the mannan II polymorphic form (Millane & Hendrixson, 1994).

Increased crystallinity may be one factor that contributes to decreased water absorption and solubility indices of the KGM3 and KGM4 treatment groups. As seen with other measured properties, the shear conditions introduced by the die and increased barrel temperature most contributed to enhanced crystallinity of the extruded KGM samples. After drying and grinding into flour, crystallinity ranges from 13 to 37% (Li, Xia, Wang, & Xie, 2005) and extracted glucomannan can be even less. The degree of crystallinity depends on the degree of glucose substitution in the mannan, and the degree of acetylation of the side chains (Xiaoyan et al., 2010). Indeed, one suggested gelling mechanism is that hydrolysis of acetyl groups in alkali conditions allows more regions on the glucomannan backbone to interact or crosslink in junction zones.

The reason for the small increase in crystallinity in extrusion-modified KGM is uncertain. The decrease in molecular size may allow more chain interactions due to a lower degree of entanglement as chains approach each other. In addition, partial deacetylation of extruded KGM samples cannot be discounted, and this too would allow increased interactions amongst KGM chains.

3.4. Effect of extrusion conditions on flow behavior and molecular weight (M_w)

Fig. 3 shows the apparent viscosity as a function of shear rate for 0.5% KGM solutions. All datasets were well-fit by the Cross model ($r^2 > 0.98$). Fitted parameters for the model are presented in Table 4. The zero-shear viscosity (η_o) for the non-extruded KGMC was 3.66 Pa s. The apparent viscosity of all extruded KGM solutions were lower than that for non-extruded KGM solutions. Zero-shear viscosities for extruded KGM solutions ranged from 0.18 to 3.01 Pa s, with lowest values for solutions made from KGM3, and the highest for KGM1. As with other properties, solution viscosity was most affected for KGM modified at higher temperature,

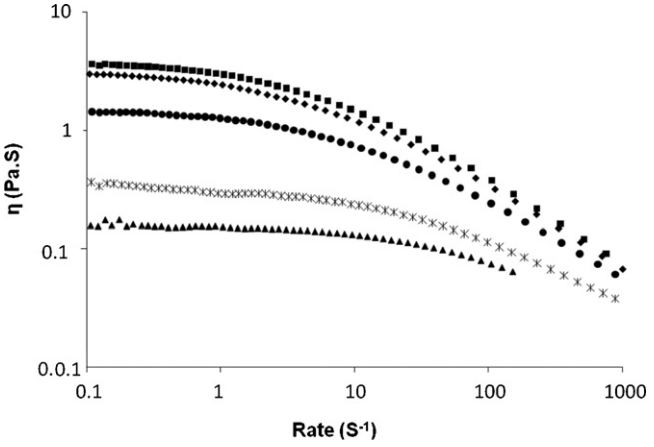

Fig. 3. Flow behavior of KGM dispersions (0.5%, w/w): (■) non-extruded (KGMC), (♦) KGM1, (●) KGM2, (▲) KGM3, and (*) KGM4.

Table 4
Constants for Cross model (Eq. (4)) fitted to shear dependence of viscosity.

Samples	η_0 (Pa s)	K (s)	n	r^2
KGMC	3.66d	0.17c	0.25a	1.00
KGM1	3.01c	0.18c	0.27ab	1.00
KGM2	1.51b	0.11b	0.31abc	1.00
KGM3	0.18a	0.01a	0.37c	0.98
KGM4	0.36a	0.03a	0.33bc	0.99

Values in a column followed by different letters are significantly different ($p < 0.05$).

and even more by inclusion of the extrusion die. In all cases, KGM dispersions exhibited shear thinning behavior, with power indices ranging from $n = 0.75$ (KGMC) to 0.63 (KGM3). Other researchers have measured pseudoplastic behavior for suspensions of konjac flour (Jacon, Rao, Cooley, & Walter, 1993). As noted previously, KGM solutions can be quite viscous and this can limit its use in some products (Yaseen et al., 2005). Thus, extrusion modification was found to be able to substantially reduce KGM solution viscosity in a controlled manner.

Intrinsic viscosity of KGM solutions was also decreased by extrusion modification, ranging from 6.7 to 17.3 dL/g as compared to 19.7 dL/g for the control. Data for each solution were fit with the Mark–Houwink equation (Eq. (5)), using constants determined previously for KGM (Kishida et al., 1978). The average molecular weight for non-extruded KGMC was 1.2×10^6 Da (Table 5). The molecular weights of all extruded KGM samples were decreased, ranging from 2.7×10^5 to 9.9×10^5 Da, indicating that extrusion could effectively disrupt chains of the KGM molecules. For starch, extrusion has also been found to reduce molecular weight through conversion of the starch polymers (Sriburi et al., 1999). The molecular weights of KGM produced at 90 °C and with no die were 9.9×10^5 Da and 6.9×10^5 Da, respectively, indicating that decreasing feed solids content had only a modest impact on the final KGM molecular weight. KGM samples produced with a die or at higher temperature (110 °C) had the greatest change in molecular weight, but did not differ significantly from each other (2.6×10^5 Da versus 2.7×10^5 Da). Again, this emphasizes that both adequately high temperature and inclusion of the extruder die caused the most pronounced effect on KGM properties.

Table 5
Intrinsic viscosity and M_w of samples.

Samples	Intrinsic viscosity [η] (dL/g)	M_w
KGMC	19.7d	1.2 × 106d
KGM1	17.3c	9.9 × 105c
KGM2	13.4b	6.9 × 105b
KGM3	6.4a	2.6 × 105a
KGM4	6.7a	2.7 × 105a

Values in a column followed by different letters are significantly different ($p < 0.05$).

4. Conclusion

Extrusion modification clearly influenced morphology, particle size, WAI, WSI, crystallinity, flow behavior, M_w and gel formation ability of KGM. Solids content in the feed, maximum extrusion temperature and inclusion of the die insert were important factors that affected these properties. Increased shear from die inclusion and increased temperature caused the greatest changes in morphology, particle size and surface roughness of the dry powder. Water absorption, as measured by WAI, was greatly reduced by extrusion modification. Crystallinity was somewhat increased by lower solids and higher temperature extrusion conditions. All solutions prepared from extrusion-modified KGM were less viscous than the non-extruded control, and maintained shear-thinning behavior. Presumably, the viscosity decrease was directly related to the decrease in molecular weight. For example, the molecular weight of KGM produced in extreme conditions was roughly one-fourth that of the control, and had a zero-shear viscosity about one-tenth of that of the control.

This study showed that critical properties of KGM could be modified by extrusion under various conditions. Four specific conditions were chosen, based on a preliminary study, to give a broad range of property values. However, intermediate properties could be attained by choosing other extruder conditions. Water absorption and viscosity are often the properties of most interest and concern, and these could be altered to a very useful range of values. In addition, modified KGM can still form good gels. The specific properties of extrusion-modified KGM gels will be the subject of a subsequent study.

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