Encapsulation of konjac glucomannan in oil droplets to reduce viscosity of aqueous suspensions and gradually increase viscosity during simulated gastric digestion

^{1,2}Jin-E Wu and ²Qixin Zhong*

¹College of Food Science and Engineering, Wuhan Polytechnic University, Wuhan, 430023, China

²Department of Food Science and Technology, The University of Tennessee, Knoxville, TN 37996, USA

SHORT COMMUNICATION

Please send correspondence to

*Qixin Zhong

Department of Food Science and Technology

The University of Tennessee

2510 River Drive

Knoxville, TN 37996

Phone: (865) 974-6196; Fax: (865) 974-7332

E-mail: qzhong@utk.edu

Abstract

Konjac glucomannan (KGM) is a water-soluble polysaccharide with extraordinary ability to absorb water and increase viscosity and therefore a potential strategy to increase satiety and control food intake. However, it is difficult to incorporate KGM in aqueous foods without increasing viscosity significantly. In the present work, the objective was encapsulate KGM powder as the core of oil droplets in solid/oil/water emulsions to prevent the hydration of KGM and viscosity increase. Blending soy lecithin with soy oil enabled better mixing of KGM powder and oil and enabled the encapsulation of KGM powder at a sufficiently high concentration of whey protein isolate (WPI), as indicated by low viscosities. Preheating WPI reduced the amount of proteins required to encapsulate KGM. The gradual digestion of oil droplets by pepsin at simulated gastric conditions enabled the gradual hydration of encapsulated KGM powder to increase viscosity. These results suggest the feasibility of S/O/W emulsions to deliver KGM in aqueous foods without generating a high viscosity but enabling gradual increase of viscosity during gastric digestion to potentially impact satiety and food intake.

Keywords: konjac glucomannan, encapsulation, viscosity, solid/oil/water emulsion, gastric digestion

1. Introduction

Controlling satiety can possibly reduce food intake during a meal or between meals and therefore may be used as an intervention strategy to reduce issues caused by obesity (Marciani et al., 2001; Schroeder et al., 2013). High viscosity semi-solid foods have been shown to slow the eating and delay the gastric emptying rate which are correlated to stronger satiating properties than those less viscous (Zhu et al., 2013). Pre-meal consumption of whey proteins was shown to reduce food intake and post-meal blood glucose and insulin of subjects aged 20-27 y (Akhavan et al., 2010). Fermentable dietary fibers providing viscosities are other viable options because they provide negligible calories (Schroeder et al., 2013).

Konjac glucomannan (KGM) is such a fermentable dietary fiber that is available in the tuber of *Amorphophallus konjac* (Chen et al., 2014; Wang et al., 2015). KGM is composed of β -D-glucose and β -D-mannose monomers mainly linked by $\beta(1\rightarrow4)$ glycosidic bonds, with some side chains linked through $\beta(1\rightarrow3)$ glycosidic bonds (Chen et al., 2014; Tester and Al-Ghazzewi, 2013). Because human amylase cannot hydrolyze glycosidic bonds of KGM, KGM is a dietary fiber that can be hydrolyzed by β -mannase secreted by colonic bacteria (Wang et al., 2015). As a water-soluble polysaccharide with a molecular weight from 200 to 2000 kDa, KGM has an extraordinary capability of absorbing water and its solution is more viscous than any other biopolymer solutions (Wang et al., 2015). The high viscosity of KGM has been shown to inhibit appetite and slow intestinal absorption, which may be correlated to weight loss (Keithley and Swanson, 2005). In addition, KGM has been shown to reduce cholesterol, manage diabetes, beneficially impact gut microflora, among others (Tester and Al-Ghazzewi, 2013).

The strong ability to absorb water makes it challenging to incorporate KGM in moist foods and beverages without generating high viscosities. The objective of the present work was

to study the possibility of encapsulating KGM powder as the core of oil droplets in solid/oil/water (S/O/W) emulsions. The exclusion of water molecules contacting KGM powder enables the delivery of the water-soluble polysaccharide as colloidal particles to reduce viscosity. When oil droplets are emulsified by digestible surfactants such as whey protein isolate (WPI), the gradual digestion of WPI and therefore release of KGM may enable the gradual increase of viscosity during gastric digestion, which may eliminate effects due to sudden increase of viscosity and absorption of water by KGM.

2. Materials and methods

2.1. Materials

KGM powder was purchased from NOW Foods (Bloomington, IL, USA). WPI was obtained from Hilmar Ingredients (Hilmar, CA, USA). Other chemicals were purchased from Fisher Scientific Inc. (Pittsburgh, PA, USA).

2.2. Preparation of WPI solutions

Two sets of WPI solutions were studied. In the first set, WPI powder was hydrated at 1-15% w/v in distilled water overnight at 4 °C and adjusted to pH 7.0 using 1.0 M HCl and NaOH after warming to ambient temperature (21 °C). In the second set, the 5% WPI solution as prepared at pH 7.0 was heated at 80 °C for 5 min. After cooling in a room temperature water bath, the preheated WPI solution was spray-dried using a model B290 bench-top spray dryer (BÜCHI Corporation, Flawil, St. Gallen, Switzerland). Spray drying conditions (at a feed rate of 2.5 mL/min, an inlet air temperature of 160°C, air pressure of 600 kPa, and an outlet temperature of 110 °C) followed our previous work (Zhang and Zhong, 2015). The spray-dried powder was then hydrated to prepare solutions with 1-15% w/v preheated WPI as described previously.

2.3. Encapsulation of KGM powder

The encapsulation of KGM powder was carried out similar to our previous work (Zhang and Zhong, 2015) with modifications of powder preparation conditions. Briefly, 1.0 g of KGM powder was mixed with 5.0 mL corn oil and 2.33 g lecithin, followed by blending (Cyclone I.Q.² microprocessor homogenizer, VirTis Co., Gardiner, NY, USA) at 20,000 rpm for 3 min. Lecithin was used to facilitate the suspension of powder in corn oil because preliminary experiments showed unsuccessful encapsulation of KGM in treatments without lecithin. The 1 g S/O suspension was then added to 20 g of the WPI solution, and the mixture was homogenized at 10,000 rpm for 3 min using the same homogenizer. Emulsions as prepared had an overall KGM concentration of 0.63% w/w. Two independent S/O/W emulsions were prepared for following tests.

2.4. Viscosity of emulsions

The emulsions as prepared above were loaded onto the parallel plate geometry (40 mm in diameter and 1 mm gap) of a stress-controlled rheometer (model AR20000, TA Instruments, New Castle, DE, USA). Shear rate ramps were conducted from 0.01 to 100 s⁻¹ at 21 °C. A solvent trap was used to reduce moisture loss during tests.

2.5. Structural changes of emulsions during simulated gastric digestion

The emulsions prepared with an aqueous phase with 10% w/v WPI with and without preheating were studied. The simulated gastric juice at pH 2.0 contained 0.85% w/v NaCl and 0.32% w/v pepsin (Timchalk et al., 2010) and was mixed with the emulsion at a volume ratio of 20:1, followed by adjusting the mixture pH to 2.0 using 1.0 M HCl. Samples were loaded onto the above parallel plate geometry of the rheometer. The viscosity of samples was measured at 1.0 s⁻¹ for 2 h at 37 °C.

In another set of experiments, the emulsion sample prepared with preheated WPI was mixed with the simulated gastric juice containing pepsin and adjusted for pH as above. After incubation at 37 °C in a shaking water bath operating at 150 rpm (New Brunswick Scientific Co., Edison, NJ, USA) for 0, 15, 30, 60, 90, and 120 min, 0.1 mL aliquots were withdrawn and diluted 100 times before applying 10 µL of a diluted sample evenly on a freshly-cleaved mica sheet mounted on a sample disk (Bruker Corp., Santa Barbara, CA, USA). AFM imaging followed the same protocol in our previous work (Luo et al., 2014). The instrument was a Multimode VIII microscope (Bruker AXS, Billerica, MA, USA) and the rectangular cantilever with an aluminum reflective coating on the backside had a quoted force constant of 2.80 N/m (FESPA, Bruker Corp.). Topographical images were acquired at the tapping mode and a scanning speed of 1 Hz.

3. Results and discussion

Encapsulation properties

Because KGM absorbs water and increases viscosity quickly, it is difficult to exactly quantify the amount of unencapsulated KGM. Shear rate ramps were used as an indirect method to characterize encapsulation properties. For emulsions prepared with native WPI, most samples showed shear-rate thinning properties that are characteristics of water-soluble polysaccharide solutions (Williams, 2000). With the exception of the 5% WPI treatment, the emulsion viscosity at 21 °C generally decreased with an increase in WPI concentration, and the treatment of 15% WPI showed the least extent of shear-rate dependence (Figure 1A). The similar trend was observed for emulsions prepared with preheated WPI, and the samples prepared with 10% and 15% preheated WPI showed little shear-rate thinning properties (Figure 1B). Therefore, an

increasing amount of emulsifiers may facilitate the instantaneous stabilization of the O/W interface during shear homogenization to prevent the exposure of KGM powder to water, which increases encapsulation efficiency and reduces emulsion viscosity. The emulsion prepared with 5% WPI, with and without preheating, was an exception from this trend because these two treatments had the highest viscosity (Figure 1). When tested for the percentage of WPI remaining in the aqueous phase, the 5% WPI treatments had a significantly higher percentage of unadsorbed WPI (results not shown). However, we are unable to provide a rational explanation of this exception. Because of the extraordinary ability of KGM absorbing water, samples without successful encapsulation became gels or highly viscous after storage, while those with KGM successfully encapsulated in emulsions remained free-flowing after one-week storage at 5 °C. However, detailed characterizations on emulsion structures and stability were not attempted.

The much lower viscosity of the emulsion prepared 10% preheated WPI than that of 10% unheated WPI may have resulted from the improved emulsifying and stabilizing properties of WPI after thermal denaturation. It is well-established that heating denatures whey proteins to expose hydrophobic amino acids initially embedded internally (Ryan et al., 2013) and preheating whey proteins improves the percentage of proteins adsorbing onto soybean oil droplets irreversibly (Tcholakova et al., 2006). These properties may have resulted in the improved encapsulation of KGM using preheated WPI at 10% concentration.

Structures and properties of emulsions during simulated gastric digestion

Emulsions prepared with 10% WPI with and without preheating were mixed with 20 volumes of the simulated gastric juice and subjected to pepsin digestion at 37 °C. The viscosity during pepsin digestion was used as a parameter to monitor the kinetics. As shown in Figure 2, before digestion, the emulsion prepared with preheated WPI and diluted with the simulated

gastric juice had an apparent viscosity (~7 mPa) lower than that with native WPI (~9 mPa) at 1 s⁻¹. The viscosity increased gradually during the pepsin digestion and was similar for the two emulsions at the late stage of the 2 h treatment.

The structures of emulsions after pepsin digestion for different durations were studied using AFM, shown in Figure 3 for the emulsion prepared with 10% preheated WPI. Initially, discrete microparticles were observed. With the progression of digestion, the population and structure heterogeneity of particles appeared to have increased. After 2-h treatment, the structure became mostly amorphous, which likely indicated KGM structure after absorbing water. The AFM data in Figure 3 therefore support that the increase of viscosity (Figure 2) resulted from the digestion of proteins on droplet surface to cause the gradual hydration of initially encapsulated KGM.

4. Conclusions

Findings from the present work showed that KGM powder can be encapsulated in the core of oil droplets as S/O/W emulsions for incorporation in aqueous foods without generating a high viscosity. Lecithin in the oil phase facilitated the encapsulation, while preheating WPI enabled the encapsulation of KGM powder at a lowered protein concentration. The gradual digestion of emulsion droplets during the simulated gastric digestion gradually increased the viscosity. These features may enable the incorporation of KGM in a wide range of food products to maintain quality but increase viscosity and possibly satiety only after ingestion.

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References

- Akhavan, T., Luhovyy, B.L., Brown, P.H., Cho, C.E., Anderson, G.H., (2010). Effect of premeal consumption of whey protein and its hydrolysate on food intake and postmeal glycemia and insulin responses in young adults. *American Journal of Clinical Nutrition* 91(4), 966-975.
- Chen, X., Wang, S., Lu, M., Chen, Y., Zhao, L., Li, W., Yuan, Q., Norde, W., Li, Y., (2014). Formation and characterization of light-responsive TEMPO-oxidized konjac glucomannan microspheres. *Biomacromolecules* 15(6), 2166-2171.
- Keithley, J., Swanson, B., (2005). Glucomannan and obesity: a critical review. *Alternative Therapies* 11(6), 30-34.
- Luo, Y., Zhang, Y., Pan, K., Critzer, F., Davidson, P.M., Zhong, Q., (2014). Self-emulsification of alkaline-dissolved clove bud oil by whey protein, gum arabic, lecithin, and their combinations. *Journal of Agricultural and Food Chemistry* 62(19), 4417-4424
- Marciani, L., Gowland, P.A., Spiller, R.C., Manoj, P., Moore, R.J., Young, P., Fillery-Travis, A.J., (2001). Effect of meal viscosity and nutrients on satiety, intragastric dilution, and emptying assessed by MRI. *American Journal of Physiology - Gastrointestinal and Liver Physiology* 280(6), G1227-G1233.
- Ryan, K.N., Zhong, Q., Foegeding, E.A., (2013). Use of whey protein soluble aggregates for thermal stability - a hypothesis paper. *Journal of Food Science* 78(8), R1105-1115.

198 Schroeder, N., Marquart, L.F., Gallaher, D.D., (2013). The role of viscosity and fermentability of
 199 dietary fibers on satiety- and adiposity-related hormones in rats. *Nutrients* 5, 2093-2113.

200 Tcholakova, S., Denkov, N.D., Sidzhakova, D., Campbell, B., (2006). Effect of thermal
 201 treatment, ionic strength, and pH on the short-term and long-term coalescence stability of beta-
 202 lactoglobulin emulsions. *Langmuir* 22(14), 6042-6052.

203 Tester, R.F., Al-Ghazzewi, F.H., (2013). Mannans and health, with a special focus on
 204 glucomannans. *Food Research International* 50(1), 384-391.

205 Timchalk, C., Creim, J.A., Sukwarotwat, V., Wiacek, R., Addleman, R.S., Fryxell, G.E.,
 206 Yantasee, W., (2010). *In vitro* and *in vivo* evaluation of a novel ferrocyanide functionalized
 207 nanoporous silica decorporation agent for cesium in rats. *Health Physics* 99(3), 420-429.

208 Wang, Y., Liu, J., Li, Q., Wang, Y., Wang, C., (2015). Two natural glucomannan polymers, from
 209 Konjac and Bletilla, as bioactive materials for pharmaceutical applications. *Biotechnology*
 210 *Letters* 37(1), 1-8.

211 Williams, P.A., (2000). Chapter 1. Introduction, in: Williams, P.A. (Ed.), *Handbook of Industrial*
 212 *Water Soluble Polymers*. Blackwell Publishing Ltd, Oxford, UK.

213 Zhang, Y., Zhong, Q.X., (2015). Multiple-layered coatings on L-glutamine solid microparticles
 214 for the retention during storage and enteric delivery during *in vitro* digestions. *Food*
 215 *Hydrocolloids* 43, 584-592.

216 Zhu, Y., Hsu, W.H., Hollis, J.H., (2013). The impact of food viscosity on eating rate, subjective
 217 appetite, glycemic response and gastric emptying rate. *PLoS One* 8, e67482.

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Figure 1. Shear rate ramps at 21°C of emulsions prepared with an aqueous phase with 1-15% w/v WPI without (A) and with (B) preheating at 80 °C for 5 min. Data are averages from two independent replicates.

Figure 2. Viscosity of emulsions of prepared with 10% w/v WPI with and without preheating at 80 °C for 5 min during the simulated gastric digestion at 37 °C. The emulsions were mixed with a simulated gastric juice with pepsin at a volume ratio of 1:20 and adjusted to pH 2.0 before loading to the rheometer. Data are averages from two independent replicates.

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 232 Figure 3. AFM topographical images of emulsions prepared with 10% w/v preheated WPI after
 233 the simulated gastric digestion for 0 (A), 15 (B), 30 (C), 60 (D), 90 (E), and 120 (F) min.
 234 Emulsions were diluted with 20 volumes of a simulated gastric juice before digestion and further
 235 diluted 100 times before AFM imaging.