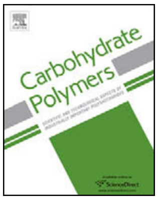
Carbohydrate Polymers 155 (2017) 128–135

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol



Tribology of swollen starch granule suspensions from maize and potato

Bin Zhang[a,b,1], Nichola Selway[c], Kinnari J. Shelat[a,b,c], Sushil Dhital[a,b], Jason R. Stokes[b,c], Michael J. Gidley[a,b,*]

[a] *Centre for Nutrition and Food Sciences, Queensland Alliance for Agriculture and Food Innovation, The University of Queensland, St. Lucia, Brisbane, QLD 4072, Australia*
[b] *ARC Centre of Excellence in Plant Cell Walls, The University of Queensland, St. Lucia, Brisbane, QLD 4072, Australia*
[c] *School of Chemical Engineering, The University of Queensland, St. Lucia, Brisbane, QLD 4072, Australia*

ARTICLE INFO

Article history:
Received 19 April 2016
Received in revised form 17 August 2016
Accepted 19 August 2016
Available online 21 August 2016

Keywords:
Starch
Granule ghosts
Maize
Potato
Tribology
Rheology

ABSTRACT

The tribological properties of suspensions of cooked swollen starch granules are characterised for systems based on maize starch and potato starch. These systems are known as granule 'ghosts' due to the release (and removal) of polymer from their structure during cooking. Maize starch ghosts are less swollen than potato starch ghosts, resulting in a higher packing concentration and greater mechanical stability. In a soft-tribological contact, maize ghost suspensions reduce friction compared to the solvent (water), generate bell-shaped tribological profiles characteristic of particle entrainment and show a marked concentration dependence, whereas potato ghost suspensions exhibit lubrication behaviour similar to water. Microscopy analysis of the samples following tribological testing suggests that this is due to the rapid break-up of potato ghosts under the shear and rolling conditions within the tribological contact. A reduction in the small deformation moduli (associated with a weak gel structure) is also observed when the potato ghost suspensions are subjected to steady shear using parallel plate rheometry; both microscopy and particle size analysis show that this is accompanied by the partial shear-induced breakage of ghost particles. This interplay between particle microstructure and the resultant rheological and lubrication dynamics of starch ghost suspensions contributes to an enhanced mechanistic understanding of textural and other functional properties of cooked starches in food and other applications.

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1. Introduction

Although most carbohydrate energy in higher plants is stored as semi-crystalline starch granules, the desirable physical properties of starch in food and industrial applications occur following a granule gelatinization process associated with loss of crystalline order. After heating in excess water with limited shear, starch granules swell to several times their initial size and release some low molecular weight soluble polymers particularly amylose (an essentially linear glucose polymer). However, the granules do not dissolve completely and can persist in a highly swollen state that is effectively a 'ghost' of its original swollen form (termed granule ghosts) (Prentice, Stark, & Gidley, 1992). The major difference between a "gelatinized starch granule" and a "granule ghost" is that solubilised polymers are absent from the granule ghost. This difference is not often recognized in the starch literature. Whilst granule ghosts are not commonly referred to, a majority of studies on the rheology-structure of gelatinized starch pastes are in fact on starch ghost pastes (Evans & Haisman, 1980; Evans & Lips, 1992; Lagarrigue & Alvarez, 2001). Generally, these highly deformable ghost particles are thought to play an important role in many of the characteristic physical properties of starch pastes, solutions, or gel networks such as viscosity, texture, and rheology (Evans & Lips, 1992; Lagarrigue & Alvarez, 2001; Steeneken, 1989). For example, the presence of dilute or highly packed granule ghosts in some semi-solid starch-containing foods such as soups, dressing, custards and sauces leads to 'short' texture, thick appearance and sometimes creamy mouthfeel (Stokes, 2011).

Abbreviations: MG, maize ghost suspension; PG, potato ghost suspension.

[*] Corresponding author at: Centre for Nutrition and Food Sciences, Queensland Alliance for Agriculture and Food Innovation, The University of Queensland, St. Lucia, Brisbane, QLD 4072, Australia.

E-mail addresses: zhangb@scut.edu.cn (B. Zhang), m.gidley@uq.edu.au (M.J. Gidley).

[1] Current address: School of Food Science and Engineering, South China University of Technology, Guangzhou 510640, China.

http://dx.doi.org/10.1016/j.carbpol.2016.08.064
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Granule ghosts isolated from normal starches such as maize and potato are enriched in amylopectin (a highly branched glucose polymer) with less than 10% of amylose (Zhang, Dhital, Flanagan, & Gidley, 2014). Recently, we found that the ghost remnants after amylase digestion contain less than 1% of single/double helices, and concluded that the ghost 'skin' originates from physical entanglements of highly branched and large molecular size amylopectin molecules (Zhang et al., 2014). Fisher, Carrington, and Odell (1997) reported that the potato ghost skin could support about 4000 mN/m tensile stress, approximately 1000 times higher than the yield stress of a red blood cell membrane. Starch components other than amylopectin (e.g., amylose, surface lipids and proteins, minerals) also play a role in restricting the extent of swelling (Debet & Gidley, 2006; Han & Hamaker, 2002), which varies depending on the botanical origins of the starch (Obanni & BeMiller, 1996). Shear and heat stability of ghost particles can be modified through certain chemical/physical methods, e.g., chemical cross-linking (to strengthen the wall structure and achieve high shear resistance) and pre-gelatinization (to increase the heat sensitivity).

Starch granule pastes/gels subjected to gelatinization and/or retrogradation exhibit a typical non-Newtonian and viscoelastic behavior, with a low yield stress and shear thinning behavior (Bagley & Christianson, 1982). The size, integrity and concentration (phase volume) of ghost particles within the matrix are important parameters which determine the viscosity and viscoelastic properties. Desse, Fraiseau, Mitchell, and Budtova (2010) reported strong deformation and solvent loss of individual swollen starch granules subjected to shear stress with the aid of a rheo-optical set-up. The morphological structure (e.g., size, shape and integrity) of starch ghost particles is influenced by their botanical origin, modification methods and processing conditions such as shear, cooking/storage temperature and time (Bagley & Christianson, 1982; Debet & Gidley, 2006). The viscosity of starch pastes is governed by the volume fraction of ghost particles in the dilute regime, whereas the particle rigidity (size, shape and deformability) is a decisive factor in the closely packed regime (Steeneken, 1989). Steeneken (1989) further suggested that both ghost particle rigidity and volume fraction within starch pastes are important in a broad concentration range between these two limiting behaviors.

While the rheological behavior of starch pastes/gels has been extensively investigated both experimentally and theoretically (Evans & Haisman, 1980; Evans & Lips, 1992; Lagarrigue & Alvarez, 2001), the lubrication properties of granule ghost suspensions have not been investigated. Lubrication has long been considered to play a critical role in oral perception of liquid and semi-solid foods, including textural and mouthfeel attributes such as smoothness and creaminess (Stokes, Boehm & Baier, 2013). However, only during the past decade have researchers attempted to quantify oral lubrication using soft-tribology as an in vitro technique, where elastomeric surfaces (e.g. polydimethylsiloxane, PDMS) are typically employed to mimic the low pressure contact between compliant oral surfaces (Bongaerts, Fourtouni, et al., 2007). Lubrication behavior is inherently dependent on relative motion between the soft-contacts of the tribometer, which is classically presented as a Stribeck curve with three different regimes namely boundary, mixed and hydrodynamic lubrication. In the hydrodynamic regime, the high fluid (or hydrodynamic) pressure can fully support the applied load and separate the contacts. This normally occurs at higher speeds with increased friction coefficients and shear force, although not all fluids have a hydrodynamic regime. Boundary lubrication occurs at lower speeds, higher load or with a poor lubrication system, as the fluid is excluded from the contact area, resulting in insufficient fluid pressure to support the applied load. In the mixed lubrication regime, the load can be partially supported by fluid pressure and partially by contacting asperities, i.e. intermediate between boundary and hydrodynamic lubrication. The friction coefficient under boundary and mixed regime conditions is more associated with surface characteristics, whereas the hydrodynamic regime is controlled by bulk rheological properties (Stokes, Boehm, & Baier, 2013).

Using these soft-tribological contacts, Selway and Stokes (2013) found that semi-solid foods (yogurt and custard) with similar viscoelasticity and flow behavior exhibit different frictional responses; hence probing the physical dynamics of complex soft systems at multiple length scales may provide better insights into texture and mouthfeel perception. Textural attributes such as the grittiness and smoothness of microparticulate dispersions have been shown to depend on particle size, shape and elasticity (Guinard & Mazzucchelli, 1996; Singer & Dunn, 1990; Tyle, 1993). The lubrication behaviour of such systems is also strongly dependent on the particle size relative to the film thickness between tribological contacts (Wilson, Sakaguchi, & Schmid, 1994). It has been shown that soft hydrogel particles smaller than the film thickness or surface asperity height are entrained between the surfaces, whereas larger particles tend to be excluded from the contact zone (de Vicente, Stokes, & Spikes, 2006; Garrec & Norton, 2012). Particle elasticity and phase volume have also been shown to have a profound influence on the tribological properties of dispersions, where stiffer (less deformable) gelled particles and higher phase volumes generate lower friction coefficients due to a reduction in surface-surface contact (Garrec & Norton, 2013).

Using maize and potato starches as exemplars, the first objective of the present study is to probe the lubrication properties of starch ghost suspensions over a range of concentrations to determine the influence of particle mechanics. The second objective of this study is to understand the viscosity and viscoelastic properties of starch ghost suspensions in both dilute and concentrated regimes. We report the small deformation oscillatory rheological behavior before and after repeated large deformation steady shear tests, combined with light microscopy of ghost particles before and after the test. The particle properties are discussed in terms of the observed differences in soft-tribological response between maize and potato ghost suspensions.

2. Materials and methods

2.1. Materials

Maize starch was purchased from Penford Australia Ltd. (Lane Cove, NSW, Australia), and potato starch was from Sigma-Aldrich. (St. Louis, MO, USA). Other chemicals used were obtained from Sigma-Aldrich. All water used was deionized.

2.2. Preparation of granule ghosts

Granule ghosts were prepared by following a method reported previously (Debet & Gidley, 2007; Zhang et al., 2014). Starch (200 mg) was suspended in a small amount of cold water and then poured into hot water (95 °C, 40 mL) with gentle mixing (250 rpm with magnetic stirrer bar) to prevent sedimentation. The dilute suspension (0.5% w/v starch) was cooked at 95 °C for 30 min to ensure the maximum swelling capacity was achieved and then centrifuged (30 °C, 2000*g* for 15 min). The supernatant was removed, and the spun ghosts were washed twice by resuspension in hot water (90 °C, 100 mL) with gentle manual stirring followed by centrifugation. The fresh ghost particles were finally resuspended in water (room temperature) at weight concentrations of 0.01%, 0.1%; and 0.87% (close packing limit, recovered directly from centrifugation) for potato ghost (PG) suspensions, and 0.01%, 0.1%, 1%, 2%, and 3% (close packing limit, recovered directly from centrifugation) for maize ghost (MG) suspensions, for tribological and rheological

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