



Review

Review of techniques to manufacture micro-hydrogel particles for the food industry and their applications



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ARTICLE INFO

Article history:

Received 22 March 2013
 Received in revised form 27 June 2013
 Accepted 28 June 2013
 Available online 9 July 2013

Keywords:

Microgel
 Nanogel
 Core-shell
 Microparticle
 Nanoparticle
 Biopolymer
 Protein
 Polysaccharide
 Emulsion
 Atomisation
 Microfluidics
 Phase separation
 Crosslink
 Rheology
 Encapsulation
 Satiety
 Targeted delivery
 Controlled delivery
 Spheroids
 Fibre

ABSTRACT

Microgels are 'soft' microscopic cross-linked polymeric particles that are being increasingly exploited in a variety of industries for rheology control, encapsulation and targeted delivery. They are valued because of the ability to tune their functionality to address specific applications in oil recovery, coatings, drug delivery, cosmetics, personal care and foods. Food microgels are typically biopolymer hydrogels in the form of microspheres, nanospheres (also called nanogels), spheroids and fibres. The utilisation of engineered microgels in foods has so far been limited, despite their great potential to address several needs in the food industry, including: satiety control, encapsulation of phytonutrients and prebiotics, texture control for healthier food formulations (e.g. reduced fat products), and targeting delivery to specific areas in the digestive tract. We review the scientific and patent literature on the utilisation and manufacturing methods for producing microgels with an emphasis on micro-hydrogels for food applications.

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Contents

1. Introduction	782
2. Microgel properties and applications	782
2.1. Flow behaviour, rheology and texture control	782
2.2. Encapsulation and targeted delivery	784
3. Manufacturing methodologies and approaches	784
3.1. Moving from precursor to microgel	785
3.1.1. Physical gelation	785
3.1.2. Chemical gelation	785
3.2. Physical formation of precursor droplets – emulsion route	785
3.2.1. Droplet deformation and breakup theory	786
3.2.2. Microgel formation from homogenisation	786
3.2.3. Microfluidics	786

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3.2.4.	Membrane	787
3.3.	Physical formation of precursor droplets – atomisation route	787
3.3.1.	Theory of drop break-up in air	787
3.3.2.	Spinning disk atomisation	787
3.3.3.	Spray nozzle	788
3.3.4.	Extrusion	788
3.3.5.	Spray drying and spray cooling	788
3.3.6.	Rehydration of spray-dried biopolymer solutions	789
3.4.	Physical formation of precursor droplets – microparticulation	789
3.4.1.	Shear gels	789
3.4.2.	Microparticulated proteins	789
3.5.	Physicochemical formation of precursor droplets	790
3.5.1.	Coacervation	790
3.5.2.	Phase separated polymers	790
4.	Conclusion and outlook	790
	References	791

1. Introduction

Micro hydrogels are ‘soft’ microscopic particles consisting of cross-linked polymeric molecules. They are valued for their functionality and ability to tune physical properties in industrial applications including oil recovery, paint and surface coating, controlled drug delivery, cosmetics, personal care, pharmaceuticals and foods (Tan et al., 2010; Stokes, 2011). The particles are swollen in solvent and possess a rich set of functionalities and suspension properties that can be exploited for rheological and texture control *as well as* encapsulation and/or targeted delivery. Exploitation of the unique properties of microgels in food and beverage applications are yet to be fully realised, which is partially due to a lack of awareness of their potential and the perceived limitations for their large scale manufacture. To address this issue and encourage further research and development in the field, we review routes to manufacture food-grade micro- and nano-hydrogel particles (which we simply refer to here as *microgels*) and highlight their potential benefits in an exciting variety of food applications.

The incorporation of solvent into the polymer network structure of microgels makes them unique and highly exploitable across many industries. The network structure is usually viscoelastic and responsive to variations in its environment (e.g. solvent quality) that allows the microgel to swell or de-swell accordingly. For example, adjusting solvent quality to cause de-swelling can be used to drive solid-to-liquid transitions in microgel suspensions while it can also be used to release encapsulated ingredients. Their rheological and encapsulation/release properties can be tuned for specific applications through variations in the composition, size, shape, cross-link density, and surface properties of the microgels.

While microgels are tuneable to obtain a range of different functionalities for non-food and food applications (for examples, see Fernandez-Neives et al., 2011), the degree of modifications to chemical structure and surface properties are more limited in food applications due to the need for the microgels to be safe to eat. The major consequence is that microgels for food applications are (typically) based on semi-rigid polymers that are present in nature, i.e. polysaccharides, while synthetic microgels are (typically) based on flexible polyelectrolytes. As a consequence, processes for producing food-grade microgels are very different from their synthetic counterparts and provide unique challenges, and there are many properties of food-grade microgels that are distinctly different to those exploited in other industries. Interestingly, the most widely used microgels are in fact food grade and naturally occurring: starches! While starch granules are hard in their natural state, upon heating they swell in what is referred to as gelatinization. In this state the soft granules can be considered to be microgels

since they are comprised of cross-linked carbohydrate polymers. Starches are universally used in food for texture and rheology control, and are a rich topic for review in their own right. Thus they are considered outside the scope of this review.

This review includes a brief presentation on microgel properties and applications followed by a more thorough evaluation of the scientific and patent literature on the main methods applicable to non-starch biopolymer microgel manufacture. The utilisation of engineered microgels in foods has been limited due to the difficulty in manufacturing food-grade biopolymer microgels using a consistent, cost effective and scalable process (Gouin, 2004). We review commercially viable processes for manufacturing food grade microgels at an industrial scale.

2. Microgel properties and applications

2.1. Flow behaviour, rheology and texture control

The rheology of colloidal microgel suspensions uniquely shares characteristics of both polymer solutions and hard-sphere suspensions (Wolfe and Scopazzi, 1989; Stokes, 2011). Microgel suspensions show increased viscosity and shear thinning at relatively low solids content like polymers, but they are inherently particles and are thus governed by many of the same factors as hard colloidal spheres. However, their specific volume can alter in response to changes in solvent quality in much the same way as polymers alter their conformation with solvent quality. We summarise here the key rheological-based properties of microgels noting that a more detailed review is provided in Stokes (2011).

The viscosity of hard sphere suspensions increases with increasing phase volume until it diverges towards infinity at a so-called maximum packing fraction governed by the particle size distribution, and corresponding to 0.64 for monodisperse hard spherical particles, as described by commonly used models of Krieger and Dougherty (1959) and Quemada (1977). The viscosity of non-attractive microgel suspensions follows a similar functional relationship to that of hard spheres, but the maximum packing fraction can exceed 0.64 and even go beyond unity. This is possible because microgels contain solvent in their network structure, and so their specific volume is defined under dilute concentrations where they are fully swollen. Large effective phase volumes can be obtained for the softer microgels because they deform and compress at high phase volumes. Fig. 1 depicts the viscosity and linear viscoelastic properties of microgel dispersions and their structural arrangement as either a gel or soft glass at high phase volume.

Colloidal scale microgel particles can be designed to possess long range attractive forces that cause aggregation and flocculation

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