Characterization of the mechanical response of pectin-based gels towards dispersing forces used in top down microgel formation processes

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Many authors have described different methods for synthesising microgel particles (Paques et al. 2014; Pravinata et al. 2016; Freitas et al. 2005). Microgel synthesis requires controlling the particle size and size distribution before or after the gelation process. Particle breakup and gelation compete during the microgel formation process. For this reason, the type of particle breakup and gelation used for microgel formation will determine the final microgel properties, including structure, size, and strength (Fernandez-Nieves et al. 2011). Microgels based on biopolymers are usually synthesised via controlled droplet gelation in an aqueous solution or in an oil based continuous phase, or by grinding macrogels into smaller units (Burey et al. 2008). Either method requires a dispersing step. This is the critical step to obtain microgel particles in suitable sizes and shapes (Wolf et al. 2001; Hamberg et al. 2003). Machines used conventionally for emulsification or dispersing, such as high-pressure homogenisers and rotor-stator systems can be employed for microgel formation.

However, specific flow types and forces that define how droplets or particles are broken up characterise each device, difficulting the choice of device for microgel preparation. During the dispersing process, forces present in the flow inside the emulsifying/dispersing device are acting on the gelled particles or polymer droplets. Droplet or particle breakup occur only if the forces acting onto the droplets/particles exceed a critical value and the material, either liquid or solid-like, yields and fails under the given conditions. The influence of a gel’s mechanical properties on the breakup result was studied, in order to be able to design the microgel preparation method so that particles of a specific size can be produced. In this regard, the rheological and mechanical properties of pectin gels were analysed. Gels were prepared with different pectin types (amidated pectin and citrus pectin) and pectinic acid, and a constant stoichiometric molar ratio. A dependency of storage modulus G’, Young’s modulus E, and breakup stress and strain on the pectin type used for gelation was shown. In order to assess the processability of the investigated gels to obtain microgel particles, the ability of a material to withstand deformations typically found in turbulent and laminar flow conditions was investigated. In this regard, gels were strained under compressive, shearing and tensile stress, which represent simplified forms of stress found in turbulent and laminar flow conditions. The gel response of the different types of gels towards the applied stress forms was compared. It was demonstrated that the type of strain applied to the gel and the gel recipe influenced the values for E, fracture stress, and strain. In addition, under compressive stress, the investigated gels reacted stiffer than under tensile stress. The findings of mechanical and rheological analyses were correlated with the particle size distribution of microgels produced from the investigated gels. The smallest microgel particles could be prepared from gels that were particular resilient towards tensile stress.