Title: Characterization of the thermal behavior and structural characteristics of gelatin in highsolids confectionary gels (HSGs) using differential scanning calorimetry (DSC), powder X-ray diffraction (PXRD) and total scattering pair distribution function (TSPDF)

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HSGs are widely used in confectionery products and as delivery systems for numerous dietary supplements and bioactive ingredients. Gelatin plays a critical role in controlling the texture of HSGs due to its unique thermal properties. However, the thermal properties of gelatin have not been investigated thoroughly in HSG systems. Thus, the objectives of this study are to determine the effect of heating rate and annealing temperature and time on the coil-helix transition of gelatin in a commercial gummy confection. The effect of heating rate was investigated using a heat-cool-heat cycle at 5, 10 and 25°C/min. The effect of annealing temperature and time was determined from 0°C to 38°C for 24h and for 4h to 72h at 22°C, respectively. The samples were first heated to 94°C to eliminate all structure then equilibrated to the desired temperature and held isothermally. The structural characteristics of the "as is" sample were determined using a Miniflex 600 PRXD over a 20 range of 0 to 100° and the PDF was measured with Mo-K α 1 radiation using a Stadi-P diffractometer. The gelatin helix-coil transition is characteristic of a kinetic event, with onset and peak temperature increasing as heating rate increases. The onset and peak temperature increased with annealing temperature, whereas the amount of triple-helix structure reformed increased with annealing temperature up to 20–22°C, then decreased. The reversibility of the triple-helix structure is time dependent. The amount of structure increased linearly with time up to 24h then leveled off and increased very slowly to the 72h end time. The XRD pattern is characteristic of an amorphous material as evidenced by the presence of a halo pattern. The TSPDF analysis showed no evidence of crystalline regions, the structural coherence length of the gelatin triple-helix ranged from 1.2 to 1.5 nm. The helix-coil transition of gelatin occurs over a range of temperatures, which depends on the product's thermal history. These results support the potential development of gummies with increased thermal stability. Characterization of the thermal behavior and structural characteristics of gelatin is a prerequisite to understanding, and subsequently, finding a suitable replacement for this popular, but costly, non-vegan material.

Keywords: high-solid confectionery gels, Triple-helix structure, gelatin, DSC