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Confectionery gels: Effects of low calorie sweeteners on the rheological properties and microstructure of fish gelatin

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ABSTRACT

Low calorie sweeteners, such as xylitol and stevia, are being used for confectionery gels. Xylitol is digested using the human's glucose pathway and stevia is a natural low calorie sweetener. However, the microstructure and rheological properties of fish gelatin (FG) confectionery gels with these sweeteners has not been studied. Therefore, the effects of xylitol or stevia at 0, 1, 3, 5, 10 and 20% on the rheological properties and microstructure of FG (6.67% w/w) were studied. The addition of xylitol at low concentrations (i.e., 3–5%) increased the gel strength. However, the gel strength decreased upon further addition of xylitol, likely due to the xylitol preventing gel network formation. The gel strength decreased as stevia increased, which might be attributed to an antagonism that prevents fish gelatin from forming three dimensional network structures. Fourier transform infrared (FTIR) spectroscopy showed that the overall intensities of Amides A, B, I, II, III and the Fingerprint region increased with increasing concentrations of xylitol and stevia, suggesting decreased molecular order. Xylitol had more pronounced effects than stevia, which probably was related to its greater solubility and number of –OH groups.

1. Introduction

Gelatin is a water soluble, high molecular weight polypeptide derived from partial hydrolysis of collagen (Liu, Nikoo, Boran, Zhou, & Regenstein, 2015). Gelatin can be used as a thickener, stabilizer, emulsifier, foaming agent, and gelling agent (Karaman, Cengiz, Kayacier, & Dogan, 2016). Currently, most gelatin is obtained from mammalian by-products, such as cattle hides, beef bones and pork skins, but due to social, cultural and health-related concerns, there is an increasing demand for alternative sources (Karim & Bhat, 2009). With the decline in the harvest of saltwater fish species, more attention is being paid to using the by-products from freshwater fish processing as a potential alternative gelatin source. Fish gelatin (FG) can be obtained from fish by-products such as the skins, bones and scales (Zhang, Ma, Cai, Zhou, & Li, 2016).

Grass carp (*Ctenopharyngodon idellus*) is a widely grown freshwater fish. Grass carp is one of the most abundant freshwater fish in China, as well as other Asian countries (Hema, Shakila, Shanmugam, & Jawahar, 2016). Although grass carp is eaten regularly in Asia, due to its small, widely distributed fish bones, it has not been used in the United States (US) for other than gefilte fish (a Jewish-style fish ball) manufacture (Regenstein, personal communication). In the US it is rapidly increasing in number and spreading more widely, and is considered an exotic species with ecological/environmental concerns. So grass carp both in Asia and North America are readily available as a source of materials for fish gelatin extraction.

Most food gel products are composite gels with sweeteners, where all of the components contribute to the structure and physical properties of the food. The interaction of gelatin with other food components has been well studied (Kuan, Nafchi, Huda, Ariffin, & Karim, 2016; Sow & Yang, 2015). Both groups determined that low concentrations of sugars decreased the gel strength, while higher levels of sugars increased the gelling and melting

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temperatures. Sweeteners are also used to impart specific flow behaviors, textures, appearances, and where required mouth-feel properties. Because of their wide spread use in confectionery gels, these systems have been well studied (Kuan et al., 2016), but it is only recently that sugar-free or reduced-sugar foods (using low calorie or no calorie sweeteners) have become popular although these compounds have not been as well studied in confectionary gels (Shankar, Ahuja, & Sriram, 2013).

In confectionery gels, the sweetener is often the most critical factor for consumer acceptance due to its effects on both flavor and texture. Xylitol and stevia are two important low calorie sweeteners in the confectionery industry (Asif, 2015). Xylitol has been widely used in the food industry because of its numerous benefits such as fewer calories, lower viscosity in solution, good chemical stability and high sweetness. Stevia is also a natural low calorie sweetener with relative sweetness 250–300 times greater than sucrose (Alizadeh, Azizi-Lalabadi, & Kheirouri, 2014). Stevia has been used as an alternative sweetener and reported to have many benefits (Carbonell-Capella, Barba, Esteve, & Frígola, 2013; Kroyer, 2010). Among them are high sweetness as a natural low calorie sweetener which has attracted more attention. Xylitol and stevia are easily soluble in water at room temperature so their interaction with gelatin can be studied.

Over the last few years, the application of low calorie sweeteners in food is of great importance. Consumers are more interested in new healthier products with decreasing amounts of sugar. Thus, xylitol and stevia have been used in confectionery gels instead of sugar, such as in sugar-free candies, cookies, and chewing gum. As the sweetness and texture of these new food products should not be changed too much, a study of their impact in gelatin mixtures seemed appropriate. The objective of this research was to study the effects of low calorie sweeteners on the physiochemical properties and microstructure of confectionery gel products that use fish gelatin, and to better understand the interaction between low calorie sweeteners and fish gelatin in gels. In this paper the physicochemical properties studied included the texture properties, gel strength, rheological properties and gelatin secondary structure to begin to understand the potential applications of fish gelatin in confectionery products.

2. Materials and methods

2.1. Materials

Fresh grass carp (*Ctenopharyngodon idellus*) skins, which had been discarded after filleting, were obtained from a local aquaculture farm that processed on the farm in Jinzhou, Liaoning Province, China. The skins from recently harvested and processed fish were taken on ice to the Seafood Processing Laboratory of Bohai University within 0.5 h. On arriving at the laboratory, the visible fat was removed using a knife and the skins were washed with cold distilled water, then kept at 0 °C until the next step of the FG extraction. Xylitol and stevia were obtained from Shandong Longli Bio Technologies Inc. (Qingdao, Shandong, China). All other reagents used were at least of analytical grade.

2.2. Methods

2.2.1. Extraction of gelatin

Gelatin was extracted from clean skins after distilled water washing according to the methods of Songchotikunpan, Tattiyakul, and Supaphol (2008) with some modifications. The skins were cut with a scissor into small pieces (~2 × 2 cm). In the first step the small pieces of fish skins were treated with 0.1 M NaOH solution (1:30 w/v) and stirred using a magnetic stirrer at room temperature (~25 °C) for 4 h. The alkaline solution was changed every h. The samples were then washed with cold tap water until a neutral pH (<7.5) (pH-meter, Hangzhou Special Paper Co. Ltd., Zhejiang, China) of the wash water was obtained. In the second step, a 0.1 M HCl (1:30 w/v) pretreatment was done for 45 min with stirring at room temperature and again returned to a neutral pH (>6.5). The third step was heating skins in water (1:30 w/w) for 4 h at 50 °C in a water bath to extract FG. The extracted gelatin solutions were centrifuged at room temperature (Sorvall Stratos Centrifuge, Thermo Fisher Scientific, Waltham, MA, USA) at 8000 g for 10 min and the sediment was discarded. The viscous supernatant was concentrated using a rotary evaporator (RE-2000, YaRong Biochemical Instrument Co. Ltd., Shanghai, China) at 85 °C for 12 h and then freeze-dried overnight (FreeZone 2.5L, Labconco, Palo Alto, CA, USA). The dry matter was ground using a mortar and pestle. This gelatin powder was then stored in a desiccator at room temperature for up to 4 wks. The proximate compositions of the extracted gelatin were analyzed according to AOAC method (AOAC, 1997) using a Kjeldahl factor of 6.25.

2.2.2. Sample preparation

The required amount of gelatin powder was dissolved in deionized water (Milli-Q Ultra Pure Water System, Millipore Inc., Billerica, MA, USA) until completely swollen to prepare a 6.67% FG solution at room temperature. A series of xylitol and stevia stock solutions were prepared at 1, 3, 5, 10 and 20% (w/v) prior to gelatin hydration. Then the appropriate amounts of xylitol and stevia were added to the FG solutions. The mixture of gelatin with xylitol or stevia was kept for 1 h at 60 °C in a water bath (HH-6 Digital Display Thermostatic Bath, Changzhou Guohua Electric Appliance Co., Ltd., Jiangsu, China) with swirling until all of the FG was dissolved. After heating, the hot solutions (20 mL) were immediately poured into cylindrical-shaped flat bottom glass containers (21 mm diameter × 36 mm height). They were then cooled to room temperature and kept at 4 °C for about 18 h to form hydrogels, and then freeze-dried to obtain xerogels. Xerogels were individually packed in low O_2 permeable polyethylene pouches and kept in a vacuum dryer over P_2O_5 (MZ250 Vacuum Desiccators, Shanghai Experimental Instrument Company, Shanghai, China) for up to 2 wk. Hydrogels were used for rheological and gel strength measurements, and xerogels were used to evaluate the secondary structure.

2.2.3. Fourier infrared (FTIR) spectroscopy

The xerogels were ground into a powder using a mortar and pestle and mixed with KBr powder at a ratio of 1:50 (w/w) (Muyonga, Cole, & Duodu, 2004b). The spectra were automatically recorded using a near FT-IR spectrophotometer (Scimitar 2000, Madison, WI, USA) in the range of 4000 − 400 cm^{-1} at a data resolution of 2 cm^{-1} against a background spectrum recorded from the clean empty cell at room temperature (~25 °C). Spectrum acquisition for each sample was repeated three times and an average spectrum was obtained using the software that came with the instrument. Intensity measurements (areas under the peaks) were also obtained using the software's undefined algorithms.

2.2.4. Texture profile analysis and gel strength

The textural properties of samples were evaluated at room temperature using a TA-XT Plus texture analyzer (Stable Micro Systems Ltd., Godalming, UK) equipped with a 50 mm diameter aluminum cylindrical probe (P/50). To keep the temperature consistent between samples, the testing was done immediately after the sample was removed from the 4 °C refrigerator. Fish gelatin (6.67% w/w) with different xylitol and stevia concentrations were prepared as described previously. The samples were removed from the glass containers with a knife and a two cycle compression