IUFoST 2006
DOI: 10.1051/IUFoST:20060929

Characterizations of Collagen Fibers for Biodegradable Films Production

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ABSTRACT - Elaboration of films containing fibers of crude, insoluble, collagen is intended to be investigated, since collagen partial insolubility and fibrous structure could result in films with improved mechanical properties. The purpose of this work was to determine some chemical and physical-chemical characteristics of collagen fibers obtained from bovine hide. The material showed a high protein content of about 86%, and only moderate solubility at low pH. Protein denaturation was observed when collagen fibers dispersed in water were heated above 50°C. Denaturation temperature decreased with acidification of the dispersion, indicating lower thermal stability of the protein at low pH.
Keywords: biofilms, biopolymers, protein, composition, solubility, DSC

INTRODUCTION

Collagen is a fibrous protein responsible by structural sustaining of several animal tissues, being the main protein present in skin, bones, tendons, cartilages and teeth. It is also raw material for production of gelatin, cosmetics and foods, as well as an alternative for edible and/or biodegradable films manufacture. Collagen is practically insoluble in water, but lowering the pH of solution can increase solubility. Insoluble collagen is converted to soluble gelatin by acid or alkaline processing, but whereas products made from native collagen posses significant strength, this strength is lost when soluble collagen is used (Figueiró et al., 2004).

Biopolymers based films are a possible response to demanded environmentally friendly packaging materials, since along with biodegradability they are often derived from abundant and renewable sources, as in the case of collagen produced from bovines, which is available at large scale and feasible cost. Major inconvenient of films obtained from soluble proteins, such as gelatin, is their poor mechanical and water vapor barrier properties, and high water solubility (Fakirov et al., 1997). Elaboration of films containing collagen fibers in its crude form is intended to be investigated, since its partial insolubility and fibrous structure could result in films with improved mechanical properties. Collagen fibers has been used as an ingredient in a variety of meat products due to its water and fat binding abilities, but their physical-chemical properties and its potential use in biodegradable film for food packaging is still little explored. In this context, the purpose of this work was to determine some chemical and physical-chemical characteristics of collagen fibers produced from bovine hide.

MATERIAL AND METHODS

Chemical composition - Collagen fibers produced from bovine hide (Novaprom Food Ingredients Ltda., Lins, Brazil) were analyzed, in triplicate, to determine protein by the Kjeldhal method (N x 5.55); lipids by the method of Bligh and Dyer with cold extraction in chloroform-methanol-water; ash by incineration at 450° C; and moisture by the gravimetric method in a vacuum oven at 60° C for 48 hours (Cecchi, 1999).

Solubility - Suspensions of 2 g collagen in 30 ml of distilled water had their pH adjusted with glacial acetic acid or sodium hydroxide, to give different values between 2 and 11. Mass was completed to 100 g with distilled water, agitated at ambient temperature during 30 min., and centrifuged at 4°C by 20 min.. Protein content in supernatant was quantified, in triplicate, by Kjeldhal method (N x 5.55) and solubility was expressed as the ratio of soluble protein to total protein in suspension (% w/w) (Monterrey-Quintero and Sobral, 2000).

Thermal analysis - Aqueous suspensions with 30 % (w/w) collagen at natural pH (7.8) and at pH 3.0 (adjusted with glacial acetic acid) were analyzed, in triplicate, in a DSC TA2010 (TA Instruments, New Castle, USA), during heating at 10ºC/min, between 0 and 100ºC, in inert atmosphere (N_2). The reference was an empty aluminum pan.

RESULTS AND DISCUSSION

The collagen produced from bovine hide presented 85.84 ± 0.55 % (w/w) proteins, 1.60 ± 0.14 % (w/w) lipids, 2.34 ± 0.03 % (w/w) ash, and 9.76 ± 1.32 % (w/w) moisture. Olivo and Shimokomaki (2002), analyzing collagen isolated from bovine tendons obtained lower protein (78.84%) and higher lipid (9.28%) contents, whereas ash (3.05%) and moisture (8.31%) contents were similar to this work.

In the whole range of pH, solubility varied between 28.9% and 52.5% (Figure 1). Maximum solubility was observed at pH 2, whereas minimum solubility occurred from pH 6 to 11. The region of increasing solubility (pH 5 to 2) coincides with the range of increasing amount of proton combined with collagen fibers determined by Boki and Kawasaki (1994). When compared to Nile Tilapia myofibrillar proteins evaluated for film formation by Monterrey-Quintero and Sobral (2000), which showed solubility of 96.9% at pH 3, collagen proteins solubility can be considered low. Nevertheless, this may be a positive characteristic, since soluble collagen products present low strength (Figueiró at al., 2004).

Thermograms of collagen showed wide protein denaturation endotherms during heating. Denaturation temperature was considered as the peak temperature of endotherms (Monterrey-Quintero and Sobral, 2000). At natural pH, two peaks were detected, at 51.48 ± 3.04 ^{o}C and 63.42 ± 1.41 ^{o}C. Two peaks were also observed for collagen at pH 3.0, but peak temperatures were lower: 39.52 ± 1.84 ^{o}C and 52.54 ± 2.88 ^{o}C, respectively. At pH 3.0, peaks were less defined than at pH 7.8. The denaturation temperature decreasing indicates lower thermal stability of proteins, as a consequence of conformation change induced by interactions between proteins and hydrogen ions in the acid medium, which lead to higher hydration of collagen. This is in accordance with the high solubility observed at low pH´s. Friess and Lee (1996) observed denaturation of insoluble collagen from bovine tendons at pH 3.5 in the range of 35^{o}C to 43^{o}C, and verified experimentally that denaturation was complete and irreversible. Swollen collagen matrices showed a reduction in denaturation temperature, in agreement with results of the present work. Figueiró et al. (2004) measured a denaturation temperature of 52.2 ^{o}C in films made with collagen from bovine serosa at pH 3.5.

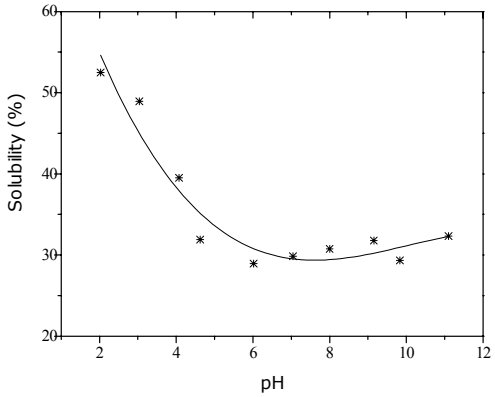

Figure 1. Solubility of collagen (g soluble protein/100 g total protein in suspension).

ACKNOWLEDGEMENTS - FAPESP and CNPq for financial support.

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