1D10 Analysis of dehydrothermal treatment for improving collagen film properties

(Kyoto Inst. Tech.) ○Chen Xuefei, Zhou Lingling
(Textile Res.Inst.Gunma) Masaki Yamamoto
(Nitta Gelatin Inc.) Masaya Shinoda, Masanori Kishimoto
(Kyoto Inst. Tech.) Hideki Yamane

Introduction

Artificial collagen films can be produced from the collagen gel extracted from animal skin. The higher order structure and the superior mechanical property are lost during the extraction process of collagen from animal skin. Dehydrothermal treatment (DHT) is safe and effective for improving the mechanical property of the artificial collagen products. However, only few attempts have made to understand the effects of DHT treatment so far. In this study, the effect of the DHT condition on the properties of collagen films manufactured from collagen gel, such as mechanical property and the swelling ratio of water were examined.

Experiments

Collagen gel from bovine hide containing 4.7 wt% of collagen and 1.1 wt% of cellulose fiber in aq.HCl (pH 2), was utilized. The collagen gel film was prepared by casting the collagen gel on the glass plate. Then the gel film on the glass plate was neutralized and solidified by immersing in ammonia solution for 10 minutes. The solidified film was washed exhaustively with water for desalination and pH adjustment. The washed film was dried at room temperature for 24 h. Dehydrothermal treatment was applied to the dry collagen film by keeping the film in a vacuum oven at various temperatures for various time. Mechanical property of the films in wet state was measured by using a tensile tester. The swelling ratio after immersing films in urea aqueous (6 M) at 30ºC was used to focus on analyzing the crosslinking structure after DHT treatment.

Results and discussion

The tensile strength of collagen films with DHT treatments are shown in Fig. 1. The strength in the MD and TD direction increased with time of DHT at 105ºC up to 5 days and then decreased at a longer time (a). Similarly they increased with DHT temp. up to 125ºC for a day (b). DHT introduced the crosslinking in collagen film resulting in the improving of the mechanical property. However longer DHT at elevated temperatures caused the denaturation of collagen which deteriorated the mechanical property.

Previous studies reported that the dehydration from collagen during DHT results in the formation of ester and amide linkages after condensation reactions during DHT treatment. However, the nature of the crosslinking has not been given so far. Figs. 2 (a) and (b) show the swelling ratio of DHT treated collagen films in the aqueous urea solution for various periods of time. It has been known that urea denatures protein by disrupting the hydrogen bond. Swelling ration of the untreated collagen film is large and increased with immersion time in urea solution. That of the film treated for one day at 105ºC is still large and increased with immersion time. However, the swelling ratio of the films treated for long periods of time is rather low. Similar results were obtained when the DHT was applied at various temperatures. The film treated at elevated temperature showed a very low swelling ratio and it stayed unchanged with increasing immersion time in aqueous urea solution. These results indicate that the large part of crosslinking of collagen untreated film and DHT at a lower temperature for a short period of time is hydrogen bonding caused to higher swelling ration in aqueous urea solution. Covalent bonds were formed in the films treated at elevated temperatures for a long time.

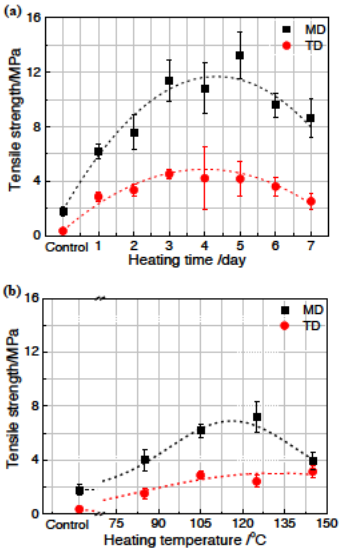

Fig. 1 Tensile strength of collagen films (a) treated for various periods of time at 105ºC and (b) at various temperatures for 24 hours.

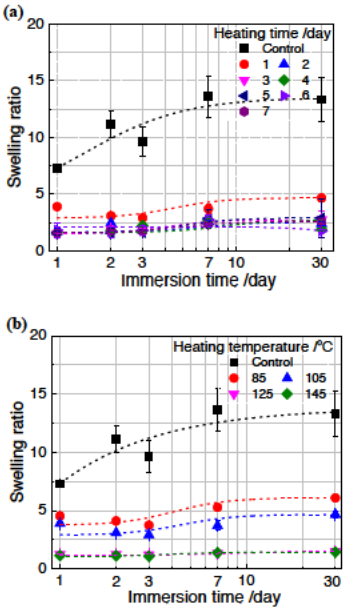

Fig.2 Swelling ratio of collagen films in aqueous urea solution. (a) Films treated for various periods of time at 105ºC and (b) those treated at various temperatures for 24 hours.

Analysis of dehydrothermal treatment for improving collagen film properties, Xuefei CHEN, Lingling ZHOU, Masaki YAMAMOTO, Masaya SHINODA, Masanori Kishimoto, Takashi AOKI, Hideki YAMANE, Kyoto Institute of Technology, Kyoto 606-8585 Japan, Tel: 075-724-7824, E-mail: hyamane@kit.ac.jp