Effects of Heat Treatment on the Tensile Strength and Transition Temperature of Processed Collagen Devices

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Purpose: A study was conducted to assess the changes in transition temperature and tensile strength of processed lyophilized collagen strips undergoing heat treatment of 50°C, 60°C, 70°C, and 80°C. Transition temperature is characteristic of intermolecular cross-linking within the collagen fibers [1], while tensile strength is characteristic of gross cross-linking such as is in medical devices [2]. 13x26mm lyophilized collagen strips, with an approximately 1.3mm^2 cross-sectional area, were incubated at 50°C, 60°C, 70°C, and 80°C for 24 hours. Following incubation, they were assessed for tensile strength using a mechanical strength testing apparatus, then hydrated with deionized water and run through a differential scanning calorimeter to assess tensile strength and transition temperature, respectively. Results indicate that though the transition temperature of the collagen strips does not change significantly between 50°C and 80°C incubation for 24 hours, the tensile strength decreases significantly by 0.35±0.1 MPa between 50°C and 60°C incubation for 24 hours.

Methods: Purified bovine hide collagen was processed, lyophilized into strips, dehydrothermally treated, and sealed into Tyvek™ pouches. The sealed strips were incubated at either 50°C, 60°C, 70°C, or 80°C for 24 hours at relative humidity. After incubation, the strips were prepared into 13x26x0.1mm dimensions for mechanical tensile testing and differential scanning calorimetry. Using an automated mechanical strength testing apparatus (Shimadzu Autograph AGS-X, Shimadzu), the strips were clamped no more than 5mm on either side with stainless steel clamps. The collagen strips endured vertical forces pulling at 10mm/sec until torn. The peak force measured prior to tearing was calculated relative to cross-sectional area (MPa). 30-50mg samples of hydrated strips were placed in sealed aluminum crucibles. The crucibles were placed in a differential scanning calorimeter (Shimadzu DSC-60, Shimadzu) and heated from 25°C to 80°C at a rate of 5°C per minute. Tangential analysis was used to assess transition temperature. Differences in average tensile strength and transition temperature were assessed using One-way ANOVA.

Results: An n=3 samples per treatment group was assessed for tensile strength and transition temperatures. Results of heat treatment temperatures on tensile strength and collagen transition temperature are summarized in Table 1.

Treatment Temperature	Transition Temperature (°C)	Tensile Strength (MPa)
50°C	45.73 ± 1.68	0.88 ± 0.16
60°C	45.97 ± 0.64	0.53 ± 0.06
70°C	46.00 ± 1.31	0.44 ± 0.05
80°C	43.43 ± 1.60	0.57 ± 0.12

Table 1

MPa One-way ANOVA was run to determine significant differences. Results indicate that there were no significant differences in the transition temperature of the collagen in the products (Figure 1). However, tensile strength decreased significantly when increasing the incubation temperature from 50°C to 60°C, 70°C, and 80°C for 24 hours ($p<0.05$, Figure 2). The tensile strength did not differ significantly between the 60°C, 70°C, and 80°C 24 hour incubation groups.

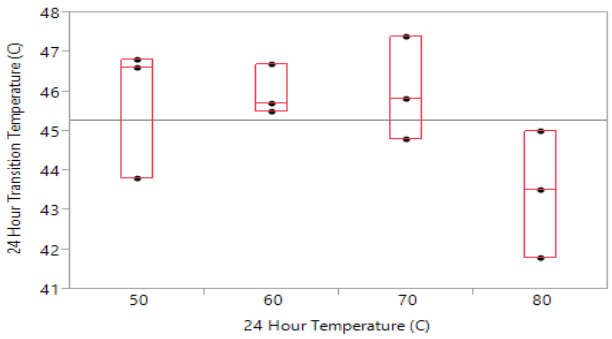

Figure 1

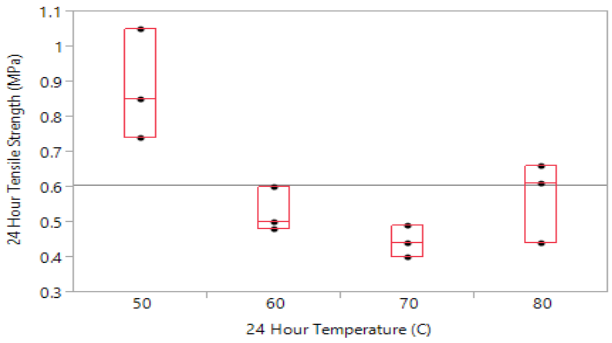

Figure 2

Conclusions: Medical devices composed of 100% purified type I collagen exhibit a relatively stable transition temperature after a 24 hour heat exposure of 50-80°C. However, temperatures at or above 60°C seem to affect the collagen's tensile strength. Transition temperature assessment through DSC has been used widely to make predictions about the intermolecular cross-linking of collagen fibers [1], and from the results of the study, the intermolecular cross-linking of the collagen fibers do not seem to be effected enough between 50°C and 80°C at 24 hours to change the transition temperature. A next step in the study will be to investigate a correlation between tensile strength, transition temperature, and to quantify the extent of collagen fiber denaturation due to heat in processed collagen products.

References:

[1] S. Skrzynski, A. Sionkowska, and A. Marciniak, Journal of Biophysics, vol. 2009, Article ID 819635.
[2] L. Olde Damink, Biomaterials, vol.17, 1996 765-773.