A16 Thermal Analyses of Property Changes and Weight Loss in Incinerated Bone and Their Implications for Forensic Anthropology

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After attending this presentation, attendees will understand how thermal analysis techniques such as Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) can be utilized to glean information about the properties and phase transitions undergone by bone when exposed to heating.

This presentation will impact the forensic science community by investigating how exposure to varying temperatures and differing heating regimes affects the rate of compositional changes of bone and by discussing the implications for further forensic analyses such as dating, isotope analyses, and genetic profiling.

Many studies have demonstrated that bone undergoes three stages of degradation and weight loss during heat exposure, which are attributed to a loss of water, the combustion of organic components, and finally, the loss of carbonate and crystal fusion. Not so well understood is to what extent these processes are influenced by the heating regime or the duration of exposure. This study utilized time- and temperature-resolved TGA and DSC to investigate the temperature regions in which the main structural changes occur and how these are influenced. TGA measures the weight changes in a sample as a function of a temperature profile and DSC allows for the monitoring of the heat flow in and out of a sample, which allows for the detection of phase transitions in the material.

Sheep (*Ovis aries*) rib bones were cut into cross sections of approximately 20mg and analyzed using an Evisa® STA 1500 Simultaneous Thermal Analyzer. Samples were heated from room temperature to 1,100°C, for which three different heating rates were employed, 6°C/min, 12°C/min, and 24°C/min. Additionally, two sets of samples were heated at a rate of 12°C up to a maximum of 300°C and 400°C and held at these temperatures for 45 minutes. All runs were carried out in triplicate.

Although weight loss as well as the heat flow curve of the three different heating regimes exhibit comparable patterns, an approximately 25°C delay in the temperature onset of phase changes with every doubling of heating rate was observed. In order to find the temperature regions of increased weight changes the first derivative of the TGA curve ($\Delta_{mass}/\Delta_{temp}$) was calculated. Distinct peaks were noted at about 125 +/- 25°C, 375 +/-25°C, 500 +/-°C, and 775 +/- 25°C at the heating rates of 6, 12, and 24°C/min, respectively. These observed weight changes correspond with the matrix phase alterations shown by the DSC. The initial heating phase corresponds to an evaporation of water, an endothermic process which peaks between 100°C and 150°C and continues up to a temperature of approximately 350°C, at which point the combustion of organic components commences, which introduces the exothermic process of the hydroxyapatite crystals becoming larger and more ordered. A brief endothermic phase attends the combustion of carbonate at about 450°C, which is followed by a strong exothermic phase as the bone's collagen continues to combust, spiking at approximately 500°C, and recrystallization of minerals proceeds. The process remains exothermic up to 750-800°C at which point the mineral begins to sinter and crystals melt in an endothermic event.

Not only did the increase in heating rate delay the temperature onset of the bone's phase transitions, but it also demonstrated an influence on the weight loss. At 1,100°C, the sample heated at 6°C min exhibited the gravest average weight loss with 59.6%, the one at 12°C lost 57.7%, and the sample heated at 24°C lost 51.1%. The observation that exposure time has an influence on the loss of mass is confirmed by the samples which were held at fixed temperatures for 45 minutes. Samples heated to 300°C, when initially reaching the temperature, lost 16.54% of their weight, which after 45 minutes increased to 33.32%. Samples held at 400°C started out with 35.2% mass loss which increased to 49.4%. The corresponding DSC curves indicate that although a steady loss of mass occurs, which is most likely attributed to the combustion of collagen, the bone does not undergo any further property changes if the temperature remains at the same level.

Being able to determine the point at which organic components in bone are lost is of crucial importance for forensic analyses, as successful collagen extraction is the basis to conduct isotope analyses, dating, or genetic profiling.

Burnt Bone, TGA, DSC