F5 Fluorescence and Structural Degradation in Composite Resins as a Function of Temperature: A Comprehensive Study

Christi N. Rattle, Jamie Kim, PhD, and Peter J. Bush, BS, State University of New York, SUNY at Buffalo, B1 Squire Hall, South Campus SUNY at Buffalo, Buffalo, NY 14214*

The goal of this presentation is to provide an understanding of the changes in properties of dental composite resins as a function of temperature. Comprehensive analysis was performed including measurement of fluorescence, radiopacity, chemical compositions, and microstructure. The analysis methods used were unequivocal in terms of the conclusion to be made from the data. In particular, fluorescence of composite resins can be used as an aid in location of resins in the human dentition during autopsies. Attendees will understand the temperature range at which these properties are lost and the resulting differences in contrast between tooth structure and restorative material.

This presentation will impact the forensic community and/or humanity in the determination of the conditions under which the chemical and physical properties of dental composite resins are present or disappear. This information could potentially be used to estimate incineration temperatures and conditions endured by burn victims.

The restorative dental work a person receives provides unique individual characteristics, as the likelihood of two dentitions being the same is very low. Therefore, it is important to be able to identify these restorations and also be able to analyze them using instrumental techniques. This may help to determine not only the identity of the victim, but the conditions and environment they were exposed to. With an increase in the public demand for esthetic restorations it is important to understand the characteristics of the materials used in modern dentistry. These characteristics can be measured by methods from which indisputable data can be obtained.

Composite resins consist of inorganic filler particles in an organic resin matrix. The organic component typically contains a mixture of methacrylates together with polymerization inhibitors, initiators and organic dyes for coloration. The methacrylates form the bulk of the resin, and the other components are present at low concentrations. The inorganic particles can form 70% or more by weight of the resin mass. Manufacturers for esthetic effect may also add organic fluorophores. The fluorophores are present in low concentration and therefore would be difficult to identify.

When subjected to increasing temperatures, polymers begin the process of pyrolysis. During pyrolysis, bonds in the long chain polymers are broken, leaving free radicals, which combine with oxygen. As the temperature rises, the organic material breaks into successively smaller fragments, leaving the ultimate reaction products CO2 and H2O.

As the fluorescing molecules undergo pyrolysis, the fluorescing property is lost. This can be measured by UV-VIS Spectrophotometry. This technique can determine without question whether any fluorescent properties remain. The breakdown of the bond structure can also be characterized by Fourier Transform Infrared Spectroscopy (FTIR).

Pyrolysis of the organic component of the resin does not affect the inorganic filler particles. Since the radiopacity of the resin is determined by the elemental content of the filler, temperature had no effect on the inorganic elements in the filler. This was measured by quantitative radiography.

As the temperature increases, the inorganic material eventually melts and fuses. This effect was examined by Scanning Electron Microscopy (SEM), which showed the microstructural changes. Although the SEM images are necessarily qualitative, they nonetheless indisputably characterize the structure. For each of the properties surveyed, a valid technique was used for analysis.

In this study, three different brands of resin were analyzed. Discs were prepared and exposed to temperatures from room temperature to 900°C in 100-degree increments for 30 minutes. For each condition, UV- VIS spectra, FTIR spectra, radiographs and SEM images were recorded. Images of the resins under UV illumination were also collected. This combination of techniques allowed characterization of the organic and inorganic components, as well as some interesting properties of the resins.

Visible inspection with UV illumination and also of UV-VIS spectra showed that the resins lost their fluorescence above 300°C. FTIR analysis showed that as the resins were exposed to increasing temperatures, bond degradation did occur, with peaks associated with organic bonds between 2800 to 3000 cm-1 diminishing in intensity. At 200°C and 300°C, a significant decrease in the peaks' intensity occurred, and by 400°C the peaks had completely disappeared, indicating that the organic bonds had been compromised due to the exposure to very high temperatures.

Secondary electron images taken by SEM revealed the structural changes that took place in the resins. Above 300°C, the organic matrix disappeared resulting in a friable assembly of filler particles. At

 ** Presenting Author*

700°C, the particles fused together in a single glassy mass. Above 700°C, porosity developed in this mass, indicating that there was a volatile component in the glass.

Quantitative radiography was performed using aluminum plates to generate a calibration curve. Radiography showed an apparent increase in radiographic density above 300°C, which coincided with the breakdown of the organic matrix.

The loss of fluorescence above 300°C can help to indicate the temperature of victim exposure. Inspection of the dentition using UV light would reveal to the eye whether or not that temperature had been reached. Radiography would also be a useful tool for resin location at any temperature.

As with any forensic evidence, the more tests one can run, the more reliable the conclusions will be. Some analytical techniques can return unequivocal results, producing evidence that cannot be refuted. In this study, four such techniques were used to analyze changes in restorative resin properties.

Composite Resin, Incineration, Fluorescence

