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Thermal Stability of Direct Dental Esthetic Restorative Materials at Elevated Temperatures*

ABSTRACT: With increasing use of direct esthetic restorative materials, the identity of a body may rely upon knowledge of temperature effects on this class of dental restorations. This research examined the effect of atmospheric gas on thermal decomposition and color change of a wide variety of direct esthetic restorative materials. Cured discs (4×1 and 8×1 mm) were made using manufacturer's directions: traditional glass ionomer (Fuji II), light-curable resonomer (Fuji II LC), compomer (Geristore™), and three types of resin composites—highly filled, urethane-based (Occlusin™), and two Bis-GMA/TEGDMA resins: hybrid (Herculite® XRV™) and microfill (Silux Plus™). Three replications of each material were heated at 5°C/min in a thermogravimetric analysis unit using either room air or nitrogen purge to simulate different thermal environments. First derivative values of percent weight loss with respect to temperature were obtained to determine temperatures associated with increased decomposition rates. Room-air heating showed greater numbers of decomposition events than did nitrogen-heated discs. The only material decomposing less than 200°C in either atmosphere was traditional glass ionomer. The majority of decomposition occurred between 200° and 500°C for all materials. Only products containing glass ionomer components decomposed between 600° and 800°C. Room-air heating resulted in ash white discs at 800°C and higher. Specimens heated in nitrogen were gray to black at 600°C and higher. Heating atmosphere greatly affected color, and some products demonstrated distinguishing color changes: glass ionomers, in particular, showed characteristic color features. An atlas was constructed from color change of specimens recovered after 200°, 400°, 600°, 800°, and 1000°C compared with non-heated controls.

KEYWORDS: forensic science/forensic odontology, thermogravimetric analysis, fire, esthetic restorative materials

Teeth are the most indestructible component of the human body (1). These structures have the highest resistance to most environmental effects: fire, desiccation, decomposition, and prolonged water immersion (2). Therefore, teeth survive most natural disasters and may provide the only means of positive, personal identification of an otherwise unrecognizable body.

Identification of human remains in mass disasters is a difficult task. Correlation of dental records to observed restorative treatment provides the best evidence for identification of unknown remains (1). Therefore, it is important to know what happens to teeth and their respective dental restorative materials as a result of disasters.

The effects of fire on teeth are influenced by the temperature applied and by its duration. Also, adjacent tissues as well as temperature alterations caused by substances used to quench the fire have been shown to affect the thermal impact on teeth and their restorations (3,4). Bodies may be subjected to various temperatures, depending on the origin of the fire and the conditions which promote continuation of the blaze. House fires seldom reach temperatures of 1200°F (649°C), whereas chemical fires can achieve temperatures exceeding several thousand degrees Fahrenheit (5). Cremation occurs at temperatures between 1600°F and 1800°F (871°C to 982°C) for one and one-half hours (5). Some dental materials can withstand extremely high temperatures. Porcelain can resist temperatures up to 2000°F (1100°C) while amalgam can resist temperatures up to 1600°F (870°C) (5).

There has been a tremendous increase in placement of resin-based restorative materials due to controversies centered around the use of dental amalgam (6). These materials are mostly classified as ''dental resin composites.'' Another restorative material which offers good esthetics is glass ionomer. Recently, a new group of direct esthetic materials has been introduced termed ''resonomers.'' These materials have components of both dental resin composites and glass ionomers. With the public demanding more esthetic restorations, chances are great that an individual who is treated in a contemporary dental practice will have at least one type of these materials in his/her mouth.

When properly placed, esthetic tooth-colored restorations are often difficult to identify in some teeth. However, this group of restorative materials may be more susceptible to physical changes resulting from high temperatures than traditional amalgam restorations. Because of fundamental differences among components used in this material class, the response of these materials to varying levels of heat may differ significantly. Such differences may be used as an aid in the identification of the exact type of restoration present in an unidentified person. Little information has been provided in the literature on the effects of high temperatures on ''direct esthetic restorative materials.'' The purpose of this investigation is to determine the thermal stability and visual differences of various direct esthetic restorative materials heated in room air or a non-oxygenated atmosphere.

Materials and Methods

Six discs (three each for heating in air or nitrogen), 4 mm in diameter and 1 mm thick, were fabricated according to manufacturer's instructions from resin composite (Silux Plus™, 3M Dental

Products, St. Paul, MN) (Herculite® XRV™, Kerr/Sybron Corporation, Romulus, MI) (Occlusin™, GC America Inc., Chicago, IL), glass ionomer (Fuji II, GC America Inc., Chicago, IL) (Fuji II LC, GC America Inc., Chicago, IL) and a resonomer (Geristore™, Den-Mat Corporation, Santa Maria, CA) for a total of 36 discs. Six additional discs, 8 mm in diameter and 1 mm thick, were fabricated from each of the previously stated materials (a total of 36 discs) to produce a color atlas of the observed visual changes. All specimens were cured in a metal mold sandwiched between two plastic, clear Mylar strips (DuPont, Wilmington, DE). Excess material was trimmed with an acrylic bur. For analysis, the typical specimen mass was 20 mg.

A thermogravimetric analytical (TGA) unit (TG-2950, TA Instruments, New Castle, DE) was utilized to accurately measure specimen mass as temperature was increased. The atmospheric conditions during heating were changed by applying different purge gases: room air or nitrogen. Triplicates of randomly selected specimens were individually heated in atmospheres of room air (20% oxygen, 80% nitrogen to simulate room-air heating) and a totally inert atmosphere (100% nitrogen, simulating heating by non-flame contact). Specimens were heated from room temperature to 1000°C at a rate of 5°C per minute.

The larger discs were subjected to individual heating and were removed at specific temperatures for photographic purposes: 200°, 400°, 600°, 800°, or 1000°C. A photographic profile of six discs was thus produced for each material in the presence or absence of oxygen. These heated specimens were visually compared with a control specimen that had not received any heating.

Results

Thermal decomposition profiles of the resin composite materials are shown in Figs. 1–3. In each of these figures, the weight loss profiles are presented for specimens heated in room air as well as in nitrogen. Also seen below each mass loss profile is the first derivative of the weight loss with respect to room temperature. Thus, temperatures associated with increased rates of weight loss become evident as peaks in this analytical procedure. The most obvious difference in weight loss profiles among the composite materials is the extent of material remaining after all organic component has been volatilized. This value reflects the weight proportion of inorganic filler present in each type restorative material. From these data, it can be seen that the microfilled product (Silux Plus™, Fig. 1) had the least filler content (approximately 58 wt%),
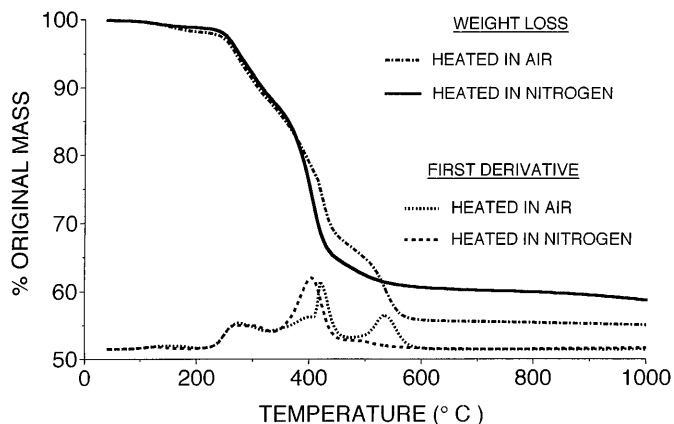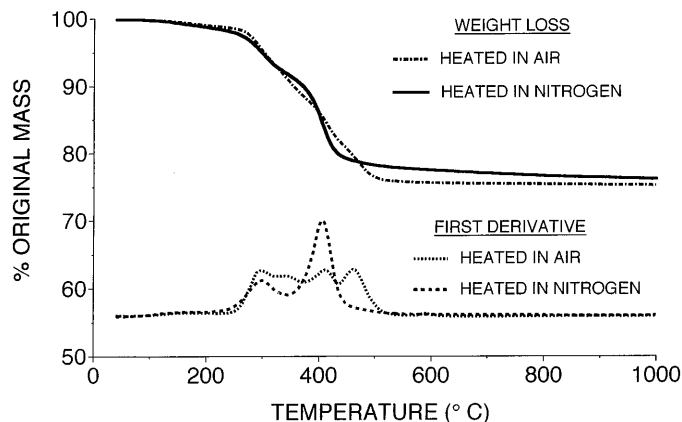

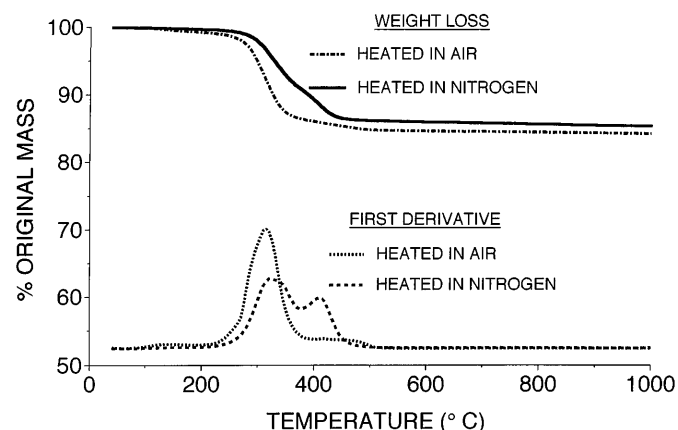FIG. 2—*Thermal decomposition profile of Herculite®XRV™.*

FIG. 3—*Thermal decomposition profile of Occlusin™.*

the medium particle hybrid material (Herculite® XRV™, Fig. 2) had a medium filler component (74 wt%), and the large particle hybrid material (Occlusion™, Fig. 3) had the most filler (about 87 wt%). Comparison of peak loss temperatures within this class of restorative material indicated that the urethane-based product (Occlusion™, Fig. 3) tended to decompose at lower temperatures than did the Bis-GMA-based resins (Silux Plus™, Fig. 1; and Herculite®XRV®™, Fig. 2). Heating specimens in a nitrogen atmosphere tended to result in higher decomposition temperatures for the urethane-based product (Fig. 3). However, heating in nitrogen for the Bis-GMA-based materials (Figs. 1 and 2) resulted in a decrease in decomposition event temperature.

Thermal decomposition profiles for materials containing a glass ionomer component were remarkably different from those of composite-based products. Decomposition of material having no resin component, totally glass ionomer-based (Fuji II, Fig. 4), indicated weight loss at relatively low temperature (near 100°C), whether the material was heated in room air or in nitrogen. Because water is required for the setting reaction of this material class, weight loss at this temperature may be associated with vaporization of trapped water molecules. The glass ionomer material containing a resin-based, photo-curable component (Fuji II LC, Fig. 5) does not display a similar weight loss in this temperature range. However, characteristic thermal events are seen between the two materials and follow trends for heating in either room air or nitrogen. At temperatures between 300° and 500°C, a single decomposition

FIG. 1—*Thermal decomposition profile of Silux Plus™.*

event is evident for both materials when heated in room air or in nitrogen. However, the temperatures associated with the light-cured material (Fig. 5) were about 40°C lower than those for the total glass ionomer product (Fig. 4). Within this temperature range, heating in nitrogen results in approximately 30°C higher values. Temperatures associated with minor decomposition events above 600°C are similar for both materials, and are not remarkably affected by heating atmosphere.

The product having a resin-based polymer matrix, but containing glass ionomer-like filler particles (Gerestore™, Fig. 6) demonstrated a different thermal decomposition profile from the other glass ionomer materials. For this material, the majority of weight loss occurred between 300° and 500°C, while for the glass ionomers, an additional 5 to 10% weight loss occurred above this temperature. This sudden weight loss pattern is similar to that seen with resin composite material, suggesting that these temperatures may be associated with volatilization of organic materials. Heating of Geristore™ in nitrogen resulted in a single decomposition event near 410°C, while room-air heating tended to spread the decomposition temperature between 340° and 430°C. It is interesting to note that a decomposition event between 750° and 800°C was noted for only those materials containing glass ionomer particles (Figs. 4–6).

Differences in temperatures associated with material decomposition for either heating atmosphere are more readily observed when simultaneously comparing first-derivative plots for similar atmospheric conditions. Figure 7 presents all first-derivative loss
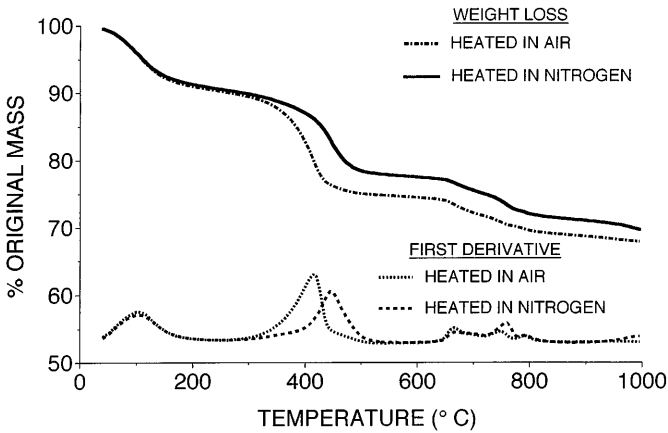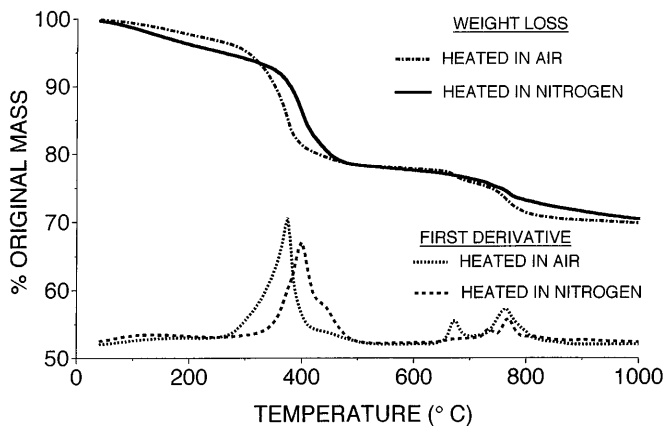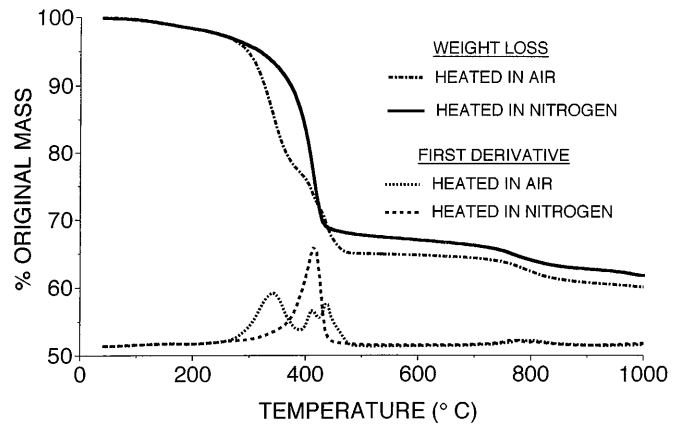

FIG. 6—*Thermal decomposition profile of Geristore™.*
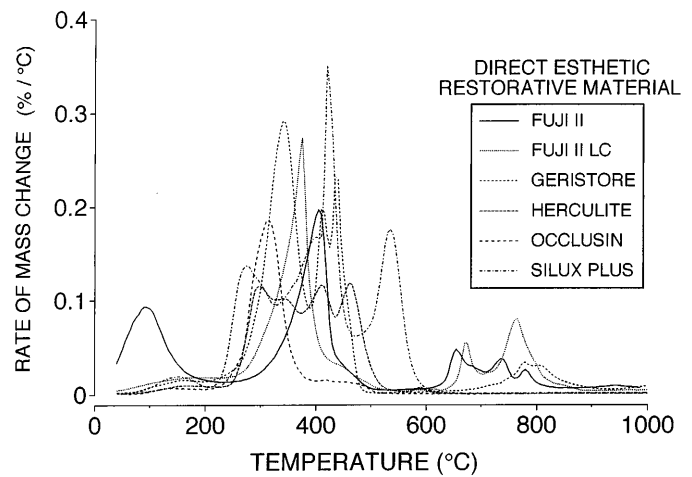
FIG. 7—*First derivative of weight loss for air-heated specimens.*

FIG. 4—*Thermal decomposition profile of Fuji II.*
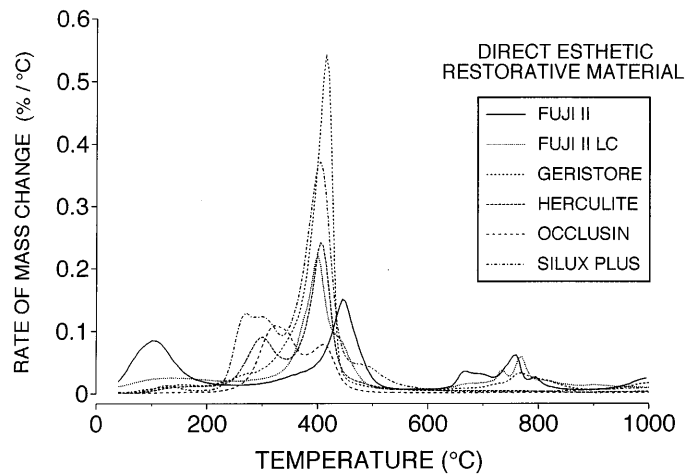

FIG. 8—*First derivative of weight loss for nitrogen-heated specimens.*

FIG. 5—*Thermal decomposition profile of Fuji II LC.*

profiles of the air-heated specimens, and Fig. 8 depicts the same information for the specimens heated in nitrogen atmosphere. As mentioned previously, the material undergoing only a glass ionomer reaction tends to be the only product demonstrating weight loss under 200°C in either atmosphere. Most decomposition for

materials heated in room air took place between 250° and 600°C, whereas the major decomposition temperature for nitrogen-heated specimens occurred between 250° and 350°C. Above 600°C, the only materials demonstrating weight loss in either heating atmosphere were those products containing a glass ionomer component.

Room-air heating for all materials resulted in ash white discs at 800°C. A distinctive red hue was noted for Fuji II when heated at 200°C. At 1000°C, Herculite®XRV™ and Occlusin™ appeared as vitrified masses whereas other materials appeared as ash white discs except for Fuji II, which appeared brown. Between 400° and 600°C, specimens displayed a wide array of shades: ash white, brown, gray, or black.

Nitrogen heating for all materials resulted in gray to black discs at 600°C and higher with the exception of Silux Plus™, which took on a unique light blue hue. Again, at 200°C, Fuji II was distinguishable from all other materials. At 400°C, a wide variety of shades appeared ranging from tan to brown to black.

(Color photographs showing the results discussed in the preceding two paragraphs are available from the corresponding author.)

Discussion

The results of this research should provide the forensic dentist with valuable information to discern differences among the thermal stability of classes of commonly used direct esthetic restorative materials. If ambient temperature does not exceed 200°C, the only material expected to decompose would be that which undergoes a conventional glass ionomer setting reaction. Visual data confirm the weight loss observations. The more glass ionomer reaction a material undergoes, the greater is the color change from room temperature, at either testing atmosphere. Thus, at 200°C, Fuji II (the conventional glass ionomer material) was greatly altered in color from other materials and quite distinguishable. The nitrogen atmosphere produced a lighter colored restoration than did that heated in room air. The only other material tested that underwent a glass ionomer setting reaction was Fuji II LC, which also had a secondary visible light curing mechanism. At 200°C, the color of this material was also different from the control as well as from other restorative materials, but not to the extent that the conventional glass ionomer material presented. Geristone is a resin-based compomer having glass ionomer particles as filler but not undergoing a glass ionomer setting reaction. This product tended to visually present color changes similar to those of the Bis-GMA-based, low-filled composite product, Silux Plus, when heated in room air.

The color of the resin composites was affected only at temperatures exceeding 200°C, for either heating atmosphere. The urethane-based composite (Occlusin™) demonstrated color-resistant changes when heated in nitrogen, but followed color changes of the Bis-GMA-based resins when heated in room air. The two Bis-GMA-based composite materials yielded similar color changes with temperatures and heating atmospheres with the exception of the red color of Herculite®XRV™ at 400° and the baby-blue color of Silux Plus at 1000°C in nitrogen. In room air, the only color difference noted was that of the darker color of Silux Plus at 600°C. It is also interesting to note that both Herculite® XRV™ and Occlusin™ produced vitrified masses when heated to 1000°C in room air. When heated in nitrogen, these specimens produced black (Herculite®XRV™) or amber glass-like residues (Occlusin™), while those heated in room air yielded pearl-like droplets. One distinguishing feature is the overt light blue color of Silux Plus™ when heated to 1000°C in nitrogen. No other materials demonstrated this particular hue change.

The heating atmosphere, in general, seemed to greatly affect the trend for color change among the different restorative products. When heated in an atmosphere containing oxygen, all materials tended to darken when heated to 400°C. However, they all showed a tendency to lighten again when exposed to temperatures of 800°C and above. An exception to this trend was Fuji II LC, which turned dark again when heated to 1000°C. In contrast, when heated in an atmosphere lacking oxygen, specimens tended to darken as temperature increased. The urethane-based composite (Occlusin™) tended to resist color change more than most of the materials tested in a nitrogen atmosphere. However, in room air, this product acted similarly to all other composite restorative materials.

The color atlas cannot be used as an absolute guide for identification of specific brands of direct esthetic restorative materials. However, trends observed in color changes among the various classes of this category of restorative materials should arm the clinician with additional means of narrowing the possibilities for positive determination of restorative material type after heating. From this information, it is hoped that the task of correlating dental restorations with existing record data will lead to greater numbers of correctly correlated identifications. It should be strongly emphasized that identification of human remains from dental records relying upon direct esthetic restorative materials to be identified implies that the dental profession is maintaining accurate, descriptive patient charts. To this end, the importance of accurate dental records that include the brand name and descriptive classification of all implanted restorative materials cannot be overstressed.

Summary and Conclusions

Based upon the conditions imposed in this study, the following conclusions may be made:

1. At a given temperature, the amount of material decomposition (mass loss) tended to differ between heating atmospheres, but was not consistent. Some materials decomposed to a greater extent in an atmosphere containing air, while others degraded quicker in a nitrogen-based atmosphere. Thus, the presence or absence of ambient oxygen cannot be used as a predictor for the decomposition of all direct esthetic dental restorative materials.
2. The only material decomposing at lower than 200°C in either atmosphere was traditional glass ionomer.
3. Room-air heating resulted in ash white discs at 800°C and higher.
4. Nitrogen heating resulted in gray to black discs at 600°C and higher.
5. Silux Plus™ had a unique blue hue after nitrogen heating at 1000°C.
6. Because there were distinctive features to the identification of direct esthetic restorative materials based upon their exposure to heat, the possibility arises that a more positive identification of remains could be made with knowledge of specific restorative materials used in patients' mouths. Thus, the dental profession is urged to diligently record all details of product identification in the patient record for possible future identification purposes.

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