# **TECHNICAL NOTE**

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# Heat Induced Changes to Dental Resin Composites: A Reference in Forensic Investigations?\*

**ABSTRACT:** The objective was to investigate color change and surface damage in dental resin composites exposed to high temperatures over different time intervals for comparative purposes. Samples were prepared using two resins - Z100 $\otimes$  (R1) and Charisma (R2), heated at the following temperatures: 200°C, 400°C, 600°C, 1000°C, for 15, 30 and 45 min (n = 104 for each resin sample). Color ( $\Delta E^*$ ) and brightness ( $\Delta L^*$ ) changes were analyzed by spectrophotometry using the CIE  $L^*a^*b^*$  system and surface changes by Scanning Electron Microscopy (SEM). R1 showed more intense color changes after heat exposure than R2.  $\Delta L^*$  values were found to be the best parameter for evaluation of light and color change. A biphasic pattern after thermal exposure was detected, from dark brown to light white. SEM showed more intense alterations in R2 than in R1. These results indicate that the parameters observed in both resins are useful as a guide in forensic analyses.

**KEYWORDS:** forensic science, forensic odontology, composite resin, high temperature, color changes, spectrophotometer, scanning electron microscopy, human identification, dental identification

Human identification by forensic odontological analysis is a well-established and reliable method (1). Destruction of a body by various means—fire, desiccation, decomposition, prolonged water immersion—can make it unrecognizable, while teeth—the most indestructible components of the human body—may continue to provide a method for positive personal identification (2).

Investigation of the effects of peri- and postmortem processes affecting the dentition and dental materials is therefore essential. The development and application of novel dental materials necessitates a continual process of comparative analysis for forensic applications, which may vary geographically and culturally (3–5).

In those circumstances in which human remains have been exposed to high temperature fires for an extended interval of time, the comparison of dental evidence recovered from the scene with the records of putative victims often becomes the sole method of establishing positive identification (5). Although dental remains often retain a large degree of their anatomical configuration following burning, they are reduced in size and extremely fragile. As a result, they may be easily overlooked or inadvertently destroyed by untrained people (6).

The knowledge of the mechanical properties of teeth and dental materials commonly used in restorations following exposure to high temperatures can prevent unintentional destruction of the evidence as well as increase the chances of a positive identification.

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Some dental materials have already been studied with regard to their resistance to high temperatures. Amalgam resists up to 800°C (6–11), base metal alloys reach their melting points from 1275°C to 1500°C (6,9,11) and porcelains resist temperatures higher than 1000°C (6–9,11). There is not much information in the literature about the effects of high temperatures on composite resins, however (7).

Composite resins are the most prevalent materials used for aesthetic restorations (12,13), partly because there is a tendency for the substitution of metal alloys by other materials that can reproduce the closest appearance of the original teeth (13). Therefore, the chances are great that an individual who is treated within a contemporary dental practice will have at least one type of these materials in their mouth (7).

In different studies performed with resins exposed to high temperatures (7,9,11,14) the results were not consistent. Botha (11) and Robinson et al. (7) observed whitening of resins exposed at 900°C for 90 min. Rossouw et al. (14) obtained shades from grey to black when exposing resins to temperatures from 260°C to 500°C for between 5 and 30 min. Merlati et al. (9) observed changes from white to brown at 400°C, with no trace of the material detectable at 600°C.

In these investigations the range of time intervals used was limited and observations were those of a visual evaluation of color. Lack of statistical analyses or standardized methods and controls prevents meaningful comparison between studies.

A study with standardized methods and controls is necessary to obtain practical and verifiable results.

Spectrophotometry is a practical method that makes it possible to obtain numeric quantifications of color and brightness from different materials that can be compared to a standard scale. Investigation of color stability by spectrophotometry has been used in odontology for aesthetic quality control of resins (15–19).

Scanning electron microscopy (SEM) has been used in forensic dentistry to analyze severely burned and fragmented teeth as it allows magnification sufficient to confirm the presence of residues that cannot be detected by other means (6,9,10) and assist in their

identification. SEM accompanied by spectrophotometric analysis generates information with most potential value for use in comparison with the victim's dental records.

The aim of this study was to investigate color and brightness changes, and surface fragility in two of the most commonly used composite resins submitted to controlled high temperatures for determined time intervals in order to develop a comparative standard for forensic reference purposes.

#### Materials and Methods

Two different types of light-cured composite resins were used:  $Z100^{\$}$  (R1) and Charisma $^{\$}$  (R2). Both resins were selected in the A2 shade from the Scale of Vita.

These composite resins were chosen for the following reasons: (i) they are commonly used, (ii) they are significantly different in their composition—in both the organic matrix and inorganic component, (iii) they are reported on in other scientific publications and databases, and (iv) they offer a foundation upon which comparative analysis of other resins can be based.

Characteristics of the materials are given in Table 1.

Two hundred and eight cylindrical samples (104 of each resin) measuring 10.0 mm in diameter by 2.0 mm in height were prepared using a metallic split ring. A single layer of resin was put into each ring and pressed between pieces of glass to obtain a smooth surface. Preparations were light-cured for 40 sec (for R1) and 20 sec (for R2), according to manufacturer's directions, using a light-curing platform (Opt light 600; Gnatus, Ribeirão Preto, SP, Brazil), at light intensity of 500 mW/cm2, monitored by radiometer (Demetron, Danbury, MA). Finishing was done with extra-fine Sof-lex discs (3M Dental Products, St. Paul, MN).

Samples were randomly separated into different groups. Comparisons of color and brightness were made between samples submitted to high temperatures for different time intervals (n = 96 for each resin) and control samples that were not submitted to heating (n = 8 for each resin).

Heating was performed in a furnace (EDG, São Carlos, SP, Brazil) at temperatures of 200°C, 400°C, 600°C, and 1000°C for time intervals of 15, 30, and 45 min. Following heating, samples were stored in closed black plastic flasks, isolated from light and humidity. Spectrophotometric measurements were undertaken 10 h later.

A spectrophotometer (Color Guide 45/0; BYK-Gardner, PCB 6800, Geretsried, Germany), was used for color and brightness analysis (10° observation angle; D65 illuminant). All samples were scanned from 400 to 700 nm. This is an instrument with a 45°/0° geometry where the illumination (light source) shines on the sample at an angle 45° from the perpendicular to the sample surface and the viewer (detector) receives the reflected light at a location 0° from the perpendicular to (i.e. in line with) the sample. In 45°/0° and 0°/45° instruments, measurement is in reflectance-specular excluded (RSEX) mode only, and is optimal for measurements which include

TABLE 1—Characteristics of composite resins as manufacturer's instructions.

Products	Manufacturer	Batch Number	Туре	Filler Content %W	Mean Filler Size (μm)
Z100(R1)	3 M Co.,	3EN	Hybrid	71	4.5
Charisma(R2)	St Paul, MN Kulzer GmbH & Co., Wehrheim, Germany	010073	Microhybrid	64	0.02-2

a color component and a geometric component—i.e. gloss and texture (20,21). The full 10 mm circumference was analyzed in all samples.

Color measurements were obtained in the color shades system CIE L\*a\*b\*. Total color difference ( $\Delta E$ ) between two color stimuli was automatically calculated as follows:  $\Delta E = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2}.$ 

The CIE L\*a\*b\* system uses the three parameters L\*, a\*, b\* to define color: brightness, hue, and saturation (22). Brightness is the degree of lightness and darkness and corresponds to L\*, being L\* = 100 white and L\* = 0 black. The hue parameters a\* and b\* are on the chromatic scale and represent red as +a\* and green as -a\*; yellow corresponds to +b\* and blue to -b\* (19). The saturation is the intensity of a hue and it is given by the numeric value of a\* and b\*. The values of  $\Delta$ L\*,  $\Delta$ a\*,  $\Delta$ b\* correspond to the differences between the values of L\*, a\*, b\* in comparison to the standard sample.

Evaluation of surface damage was done by fixing the samples in aluminum stabs and observing at  $50\times$  magnification using Scanning Electron Microscopy (SEM - Zeiss, DSM 940, Oberkochen, Germany).

Data obtained from changes of color and brightness were analyzed statistically. As the data displayed a univariate normal distribution, a one-way ANOVA for samples of equal size was used. In order to distinguish the averages of the different experimental groups a Tukey test ( $p \le 0.05$  and  $p \le 0.001$ ) was employed to study their interaction. All statistics were performed using GMC Basic Software, version 8.1, FORP-USP, Ribeirão Preto, SP, Brazil.

### Results

The appearance of composite resins exposed to different temperatures and times of exposure are presented in Fig. 1. The results of spectrophotometric analysis of color ( $\Delta E^*$ ) and brightness ( $\Delta L^*$ ) changes are presented in Figs. 2 and 3, and Table 2. Results of SEM can be seen in Fig. 4.

Visual and spectrophotometric analyses are presented first—grouped by temperature—followed by SEM results.

Visual and Spectrophotometric Analysis

At 200°C

Changes in visual appearance were observed for both resins in comparison to controls, which increased with time interval. A grey-white speckling spread out over the surface of R1, while R2 presented a grey-white aspect just at the borders (Fig. 1).

Comparisons of  $\Delta L^*$  and  $\Delta E^*$  at different times of exposure of each resin showed no statistical difference ( $p \le 0.05$ ) at 15, 30 or 45 min. When R1 and R2 were compared, no statistical difference ( $p \le 0.05$ ) of  $\Delta L^*$  at 15, 30 or 45 min was observed. A statistically significant increase of  $\Delta E^*$  values ( $p \le 0.01$ ) was observed for R1 at 30 and 45 min (Figs. 2 and 3; Table 2).

*At 400°C* 

Intense changes in visual appearance were observed for both resins. A yellow-brown coloration developed after 15 min, followed by a brown-black coloration after 30 and 45 min. Graying at the margins were evident after 30 and 45 min, being greater in R2.

Comparisons of  $\Delta L^*$  and  $\Delta E^*$  for different times of exposure of each resin showed statistically significant differences when the 15 min group was compared to the 30 min or the 45 min groups ( $p \le 0.001$ ). This can be visualized in Figs. 2 and 3, and Table 2. No significant difference ( $p \le 0.05$ ) between the 30 min and 45 min groups was observed.

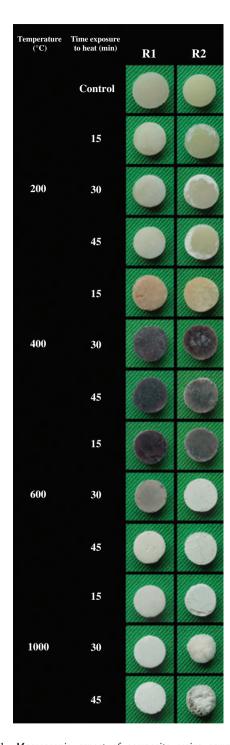


FIG. 1—Macroscopic aspect of composite resins samples and colors obtained at different time exposures to heat (200°C, 400°C, 600°C, 1000°C). From top to bottom it can be noticed the biphasic pattern of color changes for both resins.

When R1 and R2 were compared, no statistical difference in  $\Delta L^*$  and  $\Delta E^*$  at 15 and 30 min ( $p \le 0.05$ ) was observed, but at 45 min  $\Delta L^*$  and  $\Delta E^*$  presented significant differences with  $p \le 0.01$  and  $p \le 0.001$ , respectively (Table 2).

*At 600°C* 

Different patterns of change in visual appearance were observed for R1 and R2. R1 was a dark gray at 15 min, grey-yellow at 30 min and white at 45 min. R2 was gray at 15 min and white at 30 min, maintaining this color at 45 min (Fig. 1).

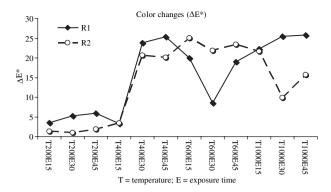


FIG. 2—Mean values of  $\Delta E^*$  for R1 and R2 at different temperatures (200°C, 400°C, 600°C, 1000°C) and time exposures (15, 30, and 45 min). See explanation in the text.

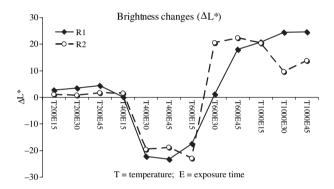


FIG. 3—Mean values of  $\Delta L^*$  for R1 and R2 at different temperatures (200°C, 400°C, 600°C, 1000°C) and time exposures (15, 30, and 45 min). See explanation in the text.

Changes in  $\Delta L^*$  and  $\Delta E^*$  at different times of exposure were distinct for each resin studied. For R1, significant differences  $(p \leq 0.001)$  among  $\Delta L^*$  values at 15, 30, and 45 min were observed. Significant differences for  $\Delta E^*$  values  $(p \leq 0.001)$ , however, were observed only between 15 and 30 min, and 30 and 45 min groups. No significant difference was detected  $(p \leq 0.05)$  between 15 and 45 min groups. For R1,  $\Delta L^*$  values (Fig. 3) corresponding to the color changes observed visually (Fig. 1) can be detected, while  $\Delta E^*$  values (Fig. 2) do not reflect the difference visually observed between the 15 and 45 min groups. The reasons for this will be discussed later.

The results for R2 differ in some respects from those of R1. Significant differences ( $p \le 0.001$ ) of  $\Delta L^*$  values were observed between the 15 and 30 min, and 15 and 45 min, but not the 30 and 45 min groups (Fig. 3; Table 2). For  $\Delta E^*$  values, a statistically significant difference was observed between the 15 and 30 min groups ( $p \le 0.05$ ), but not the 15 and 45 min or 30 and 45 min groups (Fig. 2; Table 2). Again, it is noticeable that while  $\Delta L^*$  values follow the visual alterations,  $\Delta E^*$  values do not (Fig. 1). Discussion of  $\Delta L^*$  and  $\Delta E^*$  as valid parameters for visual changes observed will be considered later.

When R1 and R2 were compared, significant statistical differences in values of  $\Delta L^*$  and  $\Delta E^*$  at 15 and 30 min with  $p \leq 0.001$  were observed. At 45 min, statistical differences were observed for  $\Delta L^*$  and  $\Delta E^*$  values, but with  $p \leq 0.01$  and  $p \leq 0.001$ , respectively.

Time (min)	15	30	45	15	30	45
$\Delta E^*$						
Temperature		200°C			400°C	
Rĺ	$3.5 \pm 0.79^{a,b,A}$	$5.3 \pm 2.46^{a,c}$	$5.9 \pm 1.27^{b,c}$	$3.32 \pm 1.45^{B}$	$23.87 \pm 3.37^{g,C}$	$25.35 \pm 2.40^{g}$
R2	$1.33 \pm 0.74^{d,e,A}$	$1.0 \pm 0.30^{d,f}$	$1.88 \pm 0.91^{e,f}$	$3.4 \pm 1.18^{B}$	$20.67 \pm 1.67^{h,C}$	$20.1 \pm 3.00^{h}$
Temperature		600°C			1000°C	
R1	$19.95 \pm 1.13^{i}$	$8.61 \pm 2.57$	$18.98 \pm 2.05^{i}$	$22.27 \pm 1.11^{l,D}$	$25.46 \pm 0.73^{-1,m}$	$25.82 \pm 0.55^{\text{m}}$
R2	$25.07 \pm 2.03^{j}$	$21.83 \pm 1.99^{k}$	$23.43 \pm 0.28^{j,k}$	$21.7 \pm 0.33^{D}$	$9.89 \pm 1.54$	$15.75 \pm 1.53$
$\Delta L^*$						
Temperature		200°C			400°C	
Rĺ	$2.73 \pm 0.73^{a,b,A}$	$3.49 \pm 1.87^{a,c,B}$	$4.37 \pm 0.97^{b,c,C}$	$0.08 \pm 1.01^{D}$	$-22.15 \pm 3.00^{g,E}$	$-23.24 \pm 2.18^{g}$
R2	$1.04 \pm 0.76^{d,e,A}$	$0.8 \pm 0.40^{\rm d,f,B}$	$1.71 \pm 0.96^{b,e,f,C}$	$1.57 \pm 1.20^{D}$	$-19.57 \pm 1.70^{h,E}$	$-18.78 \pm 2.74^{\text{h}}$
Temperature		600°C			1000°C	
RÎ	$-17.45 \pm 1.21$	$1.26 \pm 5.77$	$18.02 \pm 1.89$	$20.84 \pm 0.96^{\text{F}}$	$24.49 \pm 0.62^{j}$	$24.59 \pm 0.52^{j}$
R2	$-22.99 \pm 1.89$	$20.58 \pm 2.19^{i}$	$22.35 \pm 0.23^{i}$	$20.35 \pm 0.37^{\text{F}}$	$9.68 \pm 1.45$	$13.81 \pm 1.98$

TABLE 2—Mean values  $\pm$  standard deviation of color ( $\Delta E^*$ ) and brightness ( $\Delta L^*$ ) for different sample groups.

For comparison between time exposures (rows), means with same lower case letter are not statistically different at  $p \le 0.05$  using the Tukey test. For comparison between composite resins (columns), means with same capital letter are not statistically different at  $p \le 0.05$  using Tukey test

### At 1000°C

R1 appeared white at 15, 30, and 45 min, while R2 appeared white at 15 min, turning to light gray-yellow at 30 min and light gray at 45 min (Fig. 1).

Spectrophotometric analysis of  $\Delta L^*$  and  $\Delta E^*$  with different times of exposure generated some contrasting results in comparison with the visual analysis. For R1, significant differences of  $\Delta L^*$  values  $(p \le 0.05)$  were detected when 15 and 30 min, and 15 and 45 min groups were compared, but no statistical difference  $(p \le 0.05)$  was observed when 30 and 45 min groups were compared (Table 2). For  $\Delta E^*$  values, a statistically significant difference  $(p \le 0.05)$  was observed just when 15 and 45 min groups were compared (Figs. 2 and 3; Table 2). For R2, statistically significant differences for  $\Delta L^*$  and  $\Delta E^*$  values for all comparisons of 15, 30 and 45 min  $(p \le 0.001)$  groups were observed, with the exception of the 30 and 45 min  $(p \le 0.01)$  groups (Figs. 2 and 3, and Table 2).

When R1 and R2 were compared, no statistical difference  $(p \le 0.05)$  between them was observed at 15 min, but a significant difference  $(p \le 0.001)$  was detected in the 30 and 45 min groups (Figs. 2 and 3; Table 2).

## SEM Analysis

Scanning electron microscopy surface images from 45 min exposures are shown in Fig. 4, illustrating the greatest extent of physical changes. Images A and B (negative controls), respectively, show the original appearance of R1 and R2 with no exposure to high temperatures.

In the left column of Fig. 4, changes to surfaces of resin R1 can be seen. At 200°C, the resin surface became irregular in comparison to the control, presenting a thin powdered layer spread out all over the sample (Fig. 4c). This is compatible with the macroscopic appearance (see Fig. 1). At 400°C surface irregularities became more pronounced, with deep cracks that delimit areas of variable size (Fig. 4e). At 600°C the image suggests that the cracked surface observed at 400°C has flaked, leaving an underlying irregular surface exposed with new shallow lines of cracking on it (Fig. 4g). At 1000°C only a few shallow lines of cracking were seen, associated with smooth irregularities at the surface with a very thin powdered layer above it.

In the right column of Fig. 4, the development of R2 surfaces can be seen. At  $200^{\circ}$ C, the resin surface was smooth and spotted. Thin powdered layers of variable width and depth occurred adjacent to the areas of spotting (Fig. 4d). This appearance is compatible with the macroscopic one observed in Fig. 1. At  $400^{\circ}$ C the

surface irregularities were more pronounced, with deep cracks delimiting fragments of variable size with small splinters detaching from them (Fig. 4f). At 600°C the surface presented a mottled surface with less pronounced fracturing, suggesting that the fragments observed at 400°C had detached and exposed new fractures (Fig. 4h). At 1000°C the surface became disorganized presenting cavities of different sizes, with an appearance similar to that of Swiss cheese, suggesting that the resin had reached melting point (Fig. 4j).

### Discussion

Heat induced alterations to dental and restorative materials can be of great interest to forensic dentistry (10). Knowing the specific optical behavior of each dental material is important because recognition of changes induced by high temperatures can lead to the determination of which material was used in a dental restoration, facilitating identification of burned human remains. The objective of this study was to evaluate, for forensic purposes, color change and superficial damage to commonly encountered composite resins exposed to high temperatures.

Since their development in 1960, composite resins have become the main anterior dental restorative material (12). The main reason for their use is related to aesthetic qualities. Composite resins can have their colors selected as close as possible to the original colors of the teeth to be restored (23), but have the disadvantage of color instability (12,15,17,19,23–25). Discoloration of tooth-colored, resin-based materials may be caused by intrinsic and extrinsic factors. The intrinsic factors involve discoloration of the resin material itself, such as the alteration of the resin matrix and the interface of matrix and fillers (15,16,24,26). Extrinsic factors include staining by adsorption or absorption of colorants as a result of a combination of exogenous sources (12,13,15,19,26,27).

What is a disadvantage for aesthetic purposes becomes important for forensic purposes.

### Color and Brightness Changes

Color and brightness changes in composite resins can be evaluated visually and by instrumentation. Since instruments eliminate the subjective interpretation of visual appearance, spectrophotometry was used in this study to provide the empirical accuracy that may be required in forensic cases.

The use of the CIEL\*a\*b\* system of spectrophotometry allowed accurate and repeatable color and brightness comparisons. SEM

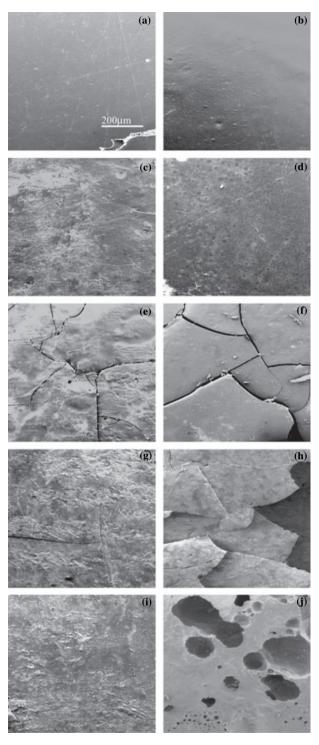


FIG. 4—SEM of composite resins surfaces (50× magnifications for all samples, white bar = 200 µm) exposed to different temperatures. Images of R1 are shown in the left column (a- control; c- at 200°C; e- at 400°C; g- at 600°C; i- at 1000°C) while in the right column images of R2 are presented (b- control; d- at 200°C; f- at 400°C; h- at 600°C; j- at 1000°C). Detailed description of surface alterations can be obtained in the text.

was used to record patterns of surface alteration related to exposure high temperatures and to offer a means of comparison with observations of macroscopic appearance—an approach impossible

The parameter most used for odontological aesthetic purposes in the CIEL\*a\*b\* system,  $\Delta E$ \*, was not found to be valuable in this study as it could not discriminate lightening or darkening of samples submitted to heat. The reason for this is based on the fact that  $\Delta E^*$  values are obtained from squared values of  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$ , which leads to the loss of positive or negative magnitudes of these values. Significant statistical differences were not detected even when materials became very light or very dark, because both materials had high values of ΔE\*—even when appearing completely different.

As a consequence, it was also necessary to analyze brightness changes,  $\Delta L^*$ . This parameter indicates variation from white to black in comparison with the original color, becoming positive if lighter or negative if darker—yielding more valuable information for forensic purposes.

The  $\Delta E^*$  non applicability, as a valid parameter in this study, is illustrated in Fig. 2 (ΔE\*). From 400°C (30 min) to 1000°C (15 min), R1 and R2 show very close values with no statistical difference in spectrophotometric analyses of  $\Delta E^*$ , but with completely distinct colors visually, i.e. 600°C (15 min) is dark gray-brown and 600°C (45 min) is white for both resins (Fig. 1).

Mean values of  $\Delta L^*$ , however, permitted more precise measurement of change in composite resin appearance. As can be seen in Figs. 1 and 3 (ΔL\*), negative values of ΔL\* corresponded to samples darkening, while positive values corresponded to lightening. From these results a biphasic behavior of composite resin relating to lightness after thermal exposure is perceptible, going from dark (brown) to light (white). Hence, the  $\Delta L^*$  values shown in Fig. 3 and Table 2 can be offered as the best instrumental parameters for the evaluation of the visual changes observed in the resin samples presented in Fig. 1.

A possible explanation for the whitish layer developed in samples at 200°C is the superficial debonding of the resin-filler interface (Figs. 4c and 4d). This process increases material opacity and has been observed after thermal and photochemical aging (12). The initial darkening observed in samples submitted to temperatures from 400 to 600°C may be due to chemical alteration of pigments or the oxidation of organic matrix compounds (12,25), and the destruction of these pigments by heating, as observed from 600°C (30 min) and higher, for R1 and R2.

R1 presented a more gradual increase in lightness, as can be seen in Figs. 1 and 3. This pattern may be related to differences in the quantity or type of pigments used in the resins. Manufacturers do not provide information on pigment concentration in these

Darkening observed for R2 at 1000°C (30 min) may be related to the melting point of this material having been reached. Longer exposures of the resins to heat beyond the limits used in this study could lead to carbonization and total darkening of the samples. Exposure to temperatures from 1000°C results in ignition and burning of the materials.

Generally, R1 presented higher values of color and brightness change than R2. This may related to the Bis-GMA (bisphenol A diglycidylether methacrylate) content. A study by Asmussen (25) showed that color stability of composite resins is related to higher amounts of Bis-GMA. R2 has higher concentrations of this monomer in its composition, which may explain the increased color stability of R2 in comparison to R1.

Our results are in agreement with Botha (11) who observed white coloration with high temperatures and with Merlati et al. (9), who observed brown coloration at 400°C. They are in contrast to those obtained by Rossouw et al. (14), who found colors from gray to black at a temperature range from 260 to 500°C after 5 to 30 min.

In our study no samples reached a black—rather than gray or gray-brown—shade. A gray-brown appearance is observed after 45 min at 400°C and after only 15 min at 600°C (Fig. 1). More prolonged periods of heat exposure or exposure to higher temperatures results in whitening of the material. These divergent results can be attributed to the use of different temperature stages and time exposures in the respective studies.

### Surface Changes

Scanning electron microscopy reveled that heating produces different kinds of changes to composite resin surfaces according to their composition. R1 is composed of a larger filler size and higher filler content than R2 (Table 1). Irregularities observed in resin surfaces may be a consequence of superficial erosion of the organic matrix. Larger filler size and content, and related electrostatic effects, may explain why the surface irregularities observed in R1 are more pronounced than those observed in R2.

Cracks observed at 400°C may be a consequence of surface drying and mechanical effects due to heating (Figs. 4e and 4f). At 600°C, a detachment of the cracked surface exposing inner fragmented layers of the material appears to have occurred. The intensity of this process was different for each resin. R1 has larger particles that increase bonding of the organic matrix, avoiding the loss of large plaques of the material (Fig. 4g). Conversely, R2 presented deeper cracks, releasing larger plaques layer-by-layer, creating a surface with irregularly sized fragments (Fig. 4h).

At 1000°C, the R2 surface seems to have melted, presenting large round empty cavities, which were not observed in R1 (Figs. 4*i* and 4*j*). A possible reason for this is that hybrid composite resins with high filler content, like R1, reach melting point at higher temperatures than microhybrid composite resins with lower filler content, like R2. Fillers added to the organic matrix of resins are commonly composed of glass particles, which have higher melting points (±1710°C) (28) and increased resistance to fire.

Merlati et al. (9) reported that in teeth restored with composite resins exposed to 600°C for 20 min, no restorative material was found. Both resins used in our study resisted to temperatures of 1000°C. This apparent discrepancy may be due to the fact that Merlati et al. (9) studied the restorative material directly on the teeth. Our spectrophotometric measurements and visual observations indicate that physico-chemical changes are occurring after 15-30 min exposure at 600°C. It is conceivable that a small specimen of altered composite resin could become fragmented or detached, and not been readily discernable as restorative material among other tooth fragments. A recent publication by Bush et al. (29) support this idea, once the authors describe that resin filled teeth exposed to 900°C for 30 min were found fragmented and color changed and resins that remained in the tooth were easily identifiable visually and with the use of a stereomicroscope. However, dislodged restorations from the teeth were difficult to distinguish from tooth fragments by visual inspection.

### Forensic Applications

Our results indicate that composite resins will provide a source of forensic evidence after exposure to temperatures of up to 1000°C, indicating that these materials can be used as means of comparison in human identification even for high temperature fire victims.

The color and brightness scale obtained in our study for each temperature/time exposure offers a practical comparative method for use in forensic investigations. Temperature and time exposure

of bodies to heat may be estimated using a spectrophotometric method, supported SEM and observation of visual appearance.

The fragility of composite resins exposed to high temperatures indicates considerable care is needed to avoid loss or damage to comparative material potentially useful in human identification. Modifications to scene protocols involving burnt human remains may assist in ensuring best practice.

### Further Work

Further investigation of color and brightness changes at temperatures greater than 1000°C and for periods over 45 min may establish the extent to which dental composite resins may have forensic value under the most extreme conditions.

In our study, exposure to high temperatures was rapid, approximately simulating the conditions of explosions or air crashes. Exposure to gradual increases in temperature may be more comparable to the conditions encountered in house fires.

These findings are of significance for forensic purposes, but an expansion in the database of resins and time exposures need to be done, as suggested by Bush et al. (29) to achieve its full utility by the forensic community.

### **Conclusions**

Our results are a novel description of color and brightness changes to two commonly used composite dental resins exposed to high temperatures for different time intervals, supported by SEM and visual analysis, and offer a potential forensic application in comparative identification of burnt human remains. Several specific conclusions can be drawn.

- Composite resins present a biphasic pattern of color and brightness change in comparison to the original, initially becoming darker and then lighter, when temperatures and exposures times increase.
- The biphasic pattern commences and develops at different rates in different resins.
- 3.  $\Delta L^*$  (brightness) is a more reliable parameter than  $\Delta E^*$  (color) in the study of heat induced changes to composite resins for forensic purposes.
- 4. The hybrid resin Z100<sup>®</sup> (R1) presented more intense color and brightness changes, but less intense superficial damage than the microhybrid resin Charisma<sup>®</sup> (R2), which reached melting point first.
- 5. Other standardized investigations of temperature and time exposure for dental restorative materials will provide a further resource for comparative forensic applications.
- Procedures for investigation of burnt human remains should incorporate protocols for the recovery and careful handling of fragile and fragmented dental materials.

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