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PAPER

ODONTOLOGY

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Color Stability of Dental Restorative Materials Submitted to Heat Sources, for Forensic Purposes

ABSTRACT: During postmortem examination of the dental arches of carbonized victims, dental restorative materials may be found. The aim of this study was to evaluate the effect of heat source action on the color stability of composite resin (CR) and glass ionomer cement (GIC) restorations, to discriminate between them and compare with *antemortem* dental data. Sixty bovine teeth (30 CR and 30 GIC) were prepared (6 × 6 × 2 mm) and separated into groups (n = 10). The color readouts were taken by spectrophotometer, before and after heat action (100°C, 200°C, 300°C), in an oven for 15 min. There were color alterations for all coordinates (ΔE, ΔL^*, Δa^* $e\Delta b^*$) for both materials. GIC presented greater change. The authors concluded that it is possible to distinguish between the materials by the color changes analyzed by instrumental method, helping victim identification.

KEYWORDS: forensic science, forensic dentistry, human identification, dental materials, heat source, color

Accidents involving the action of heat, such as burns, explosions, automobile accidents, and aircraft crashes, among others, are frequent day-to-day occurrences. The fatal victims of these events might be totally or partially carbonized, and the victim identification processes are usually challenging to the forensic teams (1). The poor state of conservation of human bodies and incomplete presence of tissues remaining after carbonization can make forensic dentistry the most feasible method of identification, or the only possibility for analyses, because the teeth are the most heat-resistant tissues of the body (2,3) and may be better preserved than other body tissues (4). Moreover, forensic dentistry can decrease the working time and material costs and be highly reliable (5), as the work is carefully done and organized by a specialist professional in this field.

The dental method consists of detailed inspection of the dental arches and their respective dental elements and compares the *antemortem* and *postmortem* (6) examinations. During *postmortem* examination, restorations with esthetic materials can be detected, particularly composite resin (CR) and glass ionomer cement (GIC), due to their frequent indications (7).

Thus, discrimination between the two materials can help the correct identification of a victim, as this may be difficult to do by clinical inspection, mainly by visual analysis of teeth (1), a subjective method that depends on each observer's perception and may result in misinterpretations (8) about the real esthetic material in the victim's mouth.

The instrumental color analysis of esthetic materials may be an accurate and safe option for *postmortem* examination of carbonized victims and can therefore contribute effectively to the victim identification process (9).

Furthermore, instrumental color analysis can prove the action of a heat source on esthetic restorative materials by verifying the color changes that occurred after being exposed to high temperatures (10), mainly in case of human body examinations (5), when there are doubts concerning *causa mortis*, and suggest the approximate temperature to which these materials were submitted.

The aim of this study was to evaluate the action of a heat source on the color stability of CR and GIC, simulating the behavior of these materials when they are present in the teeth of carbonized victims, to contribute to forensic dentistry in the process of victim identification.

Materials and Methods

Sixty sound bovine incisive teeth were prepared (6 × 6 × 2 mm) in the central region of the buccal surface. After this, the teeth were randomly separated into two groups, according to the restorative material used (Table 1).

After restoring the teeth, initial color readouts were taken using a portable spectrophotometer (VITA Easyshade®, Bad Säckingen, Germany) that consisted of a digital tip 6 mm in diameter, with 19 optical fibers and spectrophotometric sensors that emit bands of light. The color readouts were taken by

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TABLE 1—*List of materials used, commercial brands, manufacturers, colors and restorative methods.*

Category	Commercial Name	Manufacturer	Color	Restorative Method (Clinical Steps)
Composite Resin	Filtek^{MR} Z250 XT	3M ESPE®, Sumare, SP, Brazil	A3	1. Acid etching (37% phosphoric acid, Alpha Etch DFL®, Rio de Janeiro, RJ, Brazil) for 15 sec, washing and drying; 2. Bonding system application (Adper Single Bond 2, 3M ESPE®, Sumare, SP, Brazil) and photo-activation (Ultralux EL Dabi Atlante®, Ribeirao Preto, SP, Brazil) for 10 sec; 3. Material was inserted in increments and photo-activated for 20 sec; 4. Finishing and polishing (File Discs Sof Lex™ Pop On, 3M ESPE®)
Glass Ionomer Cement	Ketac™ Fil Plus	3M ESPE®, Sumaré, SP, Brazil	A3	1. Powder/liquid (1:1) agglutination up to 1 min; 2. Material was applied in increments while it still had a humid shine, until the cavity was filled

measuring the reflected light from the restoration, according to the CIE $L^*a^*b^*$ (11) scale.

The CIE $L^*a^*b^*$ scale (Comission Internationale de L'Éclairage) consists of three Cartesian coordinate axes in which L^* (gray scale) indicates color luminosity (ranging from 0—black to 100—white); a^* indicates the amount of red (positive values) and green (negative values); and b^* indicates the amount of yellow (positive values) and blue (negative values) of color (11).

For the color readout, the restored teeth were placed on a standard white background (White Standard Sphere for 45°, 0° Reflectance and Color Gardner Laboratory Inc. Bethesda, Geretsried, Germany). Three measurements were taken in each tooth, and the average obtained was considered the initial color readout.

The teeth restored with each material were randomly separated into groups ($n = 10$) according to the temperature to which they were submitted (100°C, 200°C or 300°C) in an oven (ALUMINI—Sinter Press, EDG®, Sao Carlos, SP, Brazil), for 15 min. After this, new color readouts were taken.

The color changes (ΔE) of the materials were calculated by the formula (12):

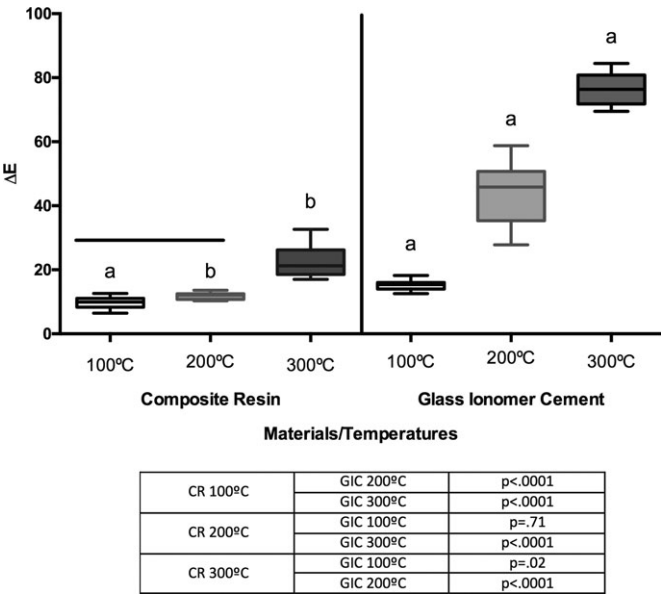
$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

where $\Delta L^* = L^*f - L^*i$; $\Delta a^* = a^*f - a^*i$, and $\Delta b^* = b^*f - b^*i$, being L^*i, a^*i, and b^*i referred to as the initial readouts, and L^*f, a^*f, and b^*f as final readouts for color coordinates. The color values (ΔE) and the changes in coordinates (ΔL^*, Δa^*, and Δb^*) were analyzed according to one-way ANOVA, Tukey, $p < 0.05$ to compare all the materials and the temperatures tested, as the materials could present similar alterations at different temperatures.

Results

The comparisons of ΔE readout mean values may be visualized in Fig. 1. There was color change (ΔE) for CR at the three temperatures evaluated. This change increased in intensity, as the temperature specimens were exposed to increased (to 300°C); a statistically different result ($p < 0.05$) in comparison with the other temperatures tested (100°C and 200°C) that showed no difference ($p > 0.05$) between them. For GIC, the higher the temperature to which specimens were submitted, the greater was the color change, with statistically significant difference ($p < 0.05$) between all the tested groups.

Comparing the materials, the authors verified that there was statistically significant difference ($p < 0.05$) between the temperatures tested, which was similar ($p > 0.05$) only for GIC after 100°C (Fig. 1).

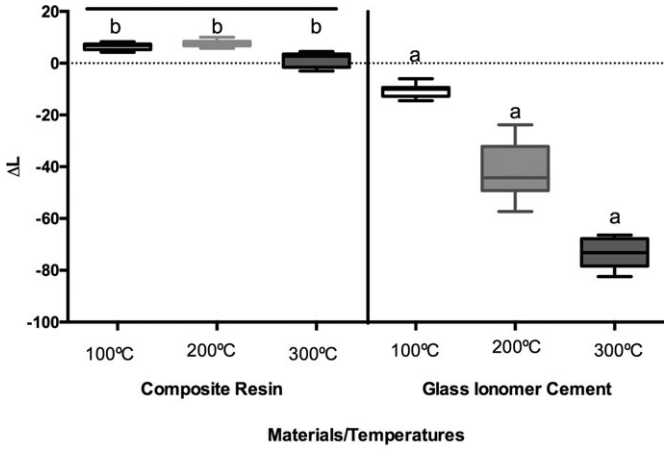


CR 100ºC	GIC 200ºC	p<.0001
	GIC 300ºC	p<.0001
CR 200ºC	GIC 100ºC	p=.71
	GIC 300ºC	p<.0001
CR 300ºC	GIC 100ºC	p=.02
	GIC 200ºC	p<.0001

FIG. 1—*Graphic representation of the comparison of ΔE changes of materials when submitted to different temperatures (one-way ANOVA, Tukey test, p < 0.05). Horizontal lines over bars indicate no statistically significant difference (p > 0.05) between groups. Different lowercase letters indicate significant difference (p < 0.05) between materials at the same temperature. CR, Composite Resin; GIC, Glass ionomer cement. Table below the graph indicates the differences between different materials submitted to different temperatures.*

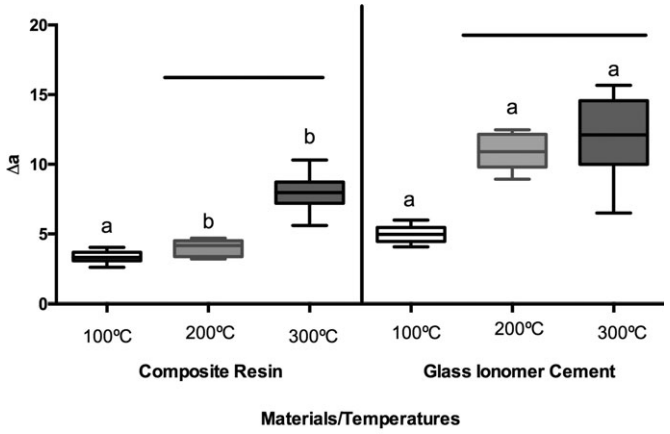
The comparisons of ΔL^* mean readouts may be visualized in Fig. 2. The results showed that the CR groups presented no significant changes ($p > 0.05$) in brightness at any of temperatures evaluated. For GIC, the higher the temperature specimens were exposed to, the greater were the L^* coordinate changes ($p < 0.05$), always with negative values, indicating darkening of the material. For GIC, the changes in ΔL^* were always greater than they were for CR, with statistically significant difference between the two materials ($p < 0.05$) for all the temperatures evaluated (Fig. 2).

As regards the a^* coordinate changes, this was greater as the temperature specimens were exposed to increased (Fig. 3). For CR at 300°C, there was a significant change ($p < 0.05$) in comparison with the other groups that showed no difference between them ($p > 0.05$). For GIC, the greatest change also occurred at 300°C, a result that was statistically similar ($p > 0.05$) at the temperature of 200°C and different ($p < 0.05$) at 100°C.



CR 100ºC	GIC 200ºC	p<.0001
	GIC 300ºC	p<.0001
CR 200ºC	GIC 100ºC	p<.0001
	GIC 300ºC	p<.0001
CR 300ºC	GIC 100ºC	p<.0001
	GIC 200ºC	p<.0001

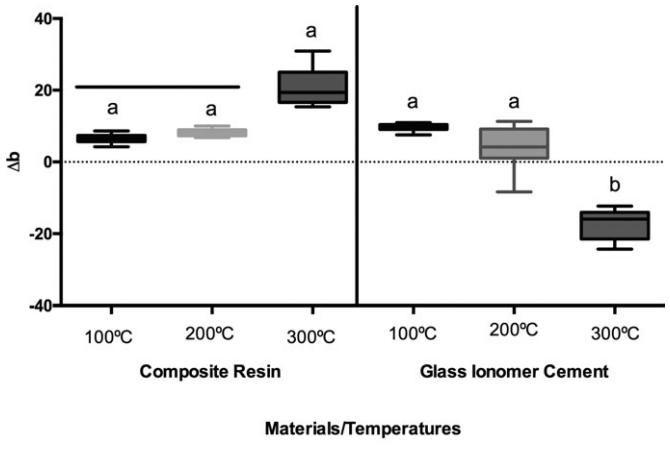
FIG. 2—*Graphic representation of the comparison of ΔL changes of materials when submitted to different temperatures (one-way ANOVA, Tukey test, p < 0.05). Horizontal lines over bars indicate no statistically significant difference (p > 0.05) between groups. Different lowercase letters indicate significant difference (p < 0.05) between materials at the same temperature. CR, Composite resin; GIC, Glass ionomer cement. Table below the graphic indicates the differences between different materials submitted to different temperatures.*



CR 100ºC	GIC 200ºC	p<.0001
	GIC 300ºC	p<.0001
CR 200ºC	GIC 100ºC	p=.66
	GIC 300ºC	p<.0001
CR 300ºC	GIC 100ºC	p=.0003
	GIC 200ºC	p=.0002

FIG. 3—*Graphic representation of the comparison of Δa changes of materials when submitted to different temperatures (one-way ANOVA, Tukey test, p < 0.05). Horizontal lines over bars indicate no statistically significant difference (p > 0.05) between groups. Different lowercase letters indicate significant difference (p < 0.05) between materials at the same temperature. CR, Composite resin; GIC, Glass ionomer cement. Table below the graphic indicates the differences between different materials submitted to different temperatures.*

Comparison between the materials showed that there was statistically significant difference ($p < 0.05$) between all the temperatures specimens were exposed to. But when compared with CR at 100°C and 200°C, and to GIC at 100°C, the comparisons showed no significant difference ($p > 0.05$) (Fig. 3).



CR 100ºC	GIC 200ºC	p.68
	GIC 300ºC	p<.0001
CR 200ºC	GIC 100ºC	p=.95
	GIC 300ºC	p<.0001
CR 300ºC	GIC 100ºC	p<.0001
	GIC 200ºC	p<.0001

FIG. 4—*Graphic representation of the comparison of Δb changes of materials when submitted to different temperatures (one-way ANOVA, Tukey test, p < 0.05). Horizontal lines over bars indicate no statistically significant difference (p > 0.05) between groups. Different lowercase letters indicate significant difference (p < 0.05) between materials at the same temperature. CR, Composite resin; GIC, Glass ionomer cement. Table below the graphic indicates the differences between different materials submitted to different temperatures.*

The action of heat promoted an increase in changes of the b* coordinate for CR at the three tested temperatures (Fig. 4). The greatest change occurred after the teeth were submitted to the temperature of 300°C; a statistically significant result ($p < 0.05$) in comparison with the other groups tested, that showed no difference between them ($p > 0.05$). With reference to GIC, all the groups showed statistically significant difference ($p < 0.05$) between them, the greatest change occurring at 100°C, a small increase at 200°C, and a decrease in the b* coordinate at 300°C.

The authors verified that the materials behaved differently ($p < 0.05$) at the temperature 300°C, in comparison with the other temperatures tested. The negative Δb^* of GIC was responsible for the statistically significant difference ($p < 0.05$) when compared with CR at 300°C. For the other temperatures tested, there was no statistically significant difference ($p > 0.05$) between the materials (Fig. 4).

Figure 5 shows representative images of the teeth before and after the submission to 300°C. The aspects of both material and teeth were found to be different at this temperature.

Discussion

The aim of this study was to evaluate the effects of heat action on the color stability of esthetic restorative materials in daily use in dental practice, by simulating the teeth of carbonized victims. The authors started with the null hypothesis that there would not be any difference in the properties of each material, irrespective of the high temperatures to which they were submitted. The results showed that the hypothesis tested could not be accepted, because the heat was able to produce a significant color change ($p < 0.05$) in the esthetic restorative materials, particularly at the higher temperatures.

The external examination of a corpse directly hit by fire or indirectly exposed to an intense heat, shows evidence of soft

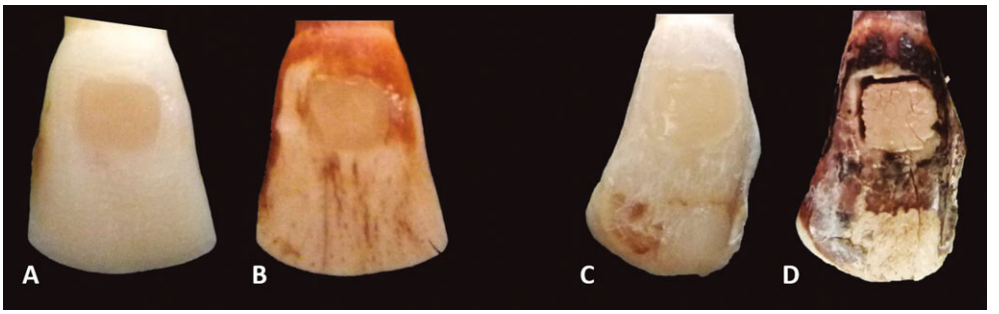


FIG. 5—*CR before (A) and after (B) submission to 300°C; GIC before (C) and after (D) submission to 300°C.*

tissue carbonization, and requires no further investigations to conclude that the victim was really exposed to heat, before or after death. However, in many accidents with carbonized victims, their bodies may be found many days after the event, in sufficient time for complete soft tissue putrefaction to begin, as seen in the decomposition of organic matter by bacteria and macroscopic fauna (13).

At the end of putrefaction stage, there will be few bodily remains, or only bone and teeth of these victims (1), leading to difficulties with: identification; definition of the *causa mortis*; and even doubts about whether or not there was exposure to high temperatures that had previously seemed obvious from external observation of the corpse. This occurs because the action of heat might not cause any evident injury to the dental and bone tissues, as these structures are protected by the skin, muscle, and fat tissues of the face (14), but might cause color change in the dental restorative materials found (15–17).

Therefore, the proposed methodology considered the temperatures of 100°C, 200°C, and 300°C as a parameter for submitting the restorations to high temperatures. The restorative materials would never reach temperatures as high as those prevailing externally. Furthermore, from a pilot test conducted previously, it was verified that as from 400°C for a period of 15 min, there was complete darkening of two esthetic restorative materials selected, making the color analysis impossible. For this reason, the authors evaluated temperatures of up to 300°C, so that the color analysis could be performed in all the sample groups. The exposure time of 15 min was selected by following a previously described methodology (16,18), in which the authors had concluded that the changes after 60 min did not differ from those that occurred after they had been submitted to the temperatures for 15 min.

Heat that reached a 400°C temperature in the inner part of the victim's mouth would only occur if the ambient temperature were around 800°C to 1000°C, and this would require a long period of fire action time, from 60 min or longer (19). Thus, considering the pilot test results, the authors found that the presence of darkened esthetic restorative material during the *postmortem* examination suggested that the temperature of exposure to heat in the mouth of carbonized victims would have been higher than 400°C, or that the time of exposure had been longer than 60 min.

As a substrate for the restorations, bovine teeth were used because they show a similarity to the human dental tissues, particularly with regard to enamel with: very similar orientation of the prisms; equivalent percentage by weight of calcium; and protein matrix composed of the same amino acids (20). With regard to dentin, there is a consensus in the literature that in the superficial cavities, 2-mm-deep, bovine dentin has been shown to be feasible for use in adhesion studies, providing adequate bond strength when compared with human dentin (21).

Several authors have analyzed color changes by visual inspection of teeth (4,14,22) and dental materials (2,15–17) resulting from the action of heat. However, there was still a lack of objective information on the color stability of esthetic restorative materials after they have been submitted to high temperatures, as the visual method is subjective and depends on many factors (7).

High temperatures lead to the appearance of intrinsic changes in composites, because the intrinsic color may be changed by many conditions in the deep portions of restorations (10,23), such as visible light, UV radiation, humidity, and temperature changes (24,25). The chemical additives of composites, mainly the initiators and ultraviolet filters, may degrade the color components (26) by the action of high temperatures. The inhibitor may also be the cause of discoloration, as the monomer containing Bis-GMA and benzoyl peroxide could be related to internal discoloration (27). Chemical changes such as the breakdown of polymer chains and oxidation of components, resulting from thermal degradation, release by-products such as formaldehyde and methacrylic acid, causing color instability (26).

The authors had expected that the materials analyzed would exhibit different behaviors toward the variable studied, because they are materials with different compositions, characteristics, and properties (28). With regard to GIC, the action of heat promoted matrix debonding, increase in porosity, and greater light dispersion within the material, making it more opaque and resulting in a greater color changes than those occurring in CR. Furthermore, GIC degraded more easily, making it more susceptible to color changes (29).

The L^* coordinate is important in the analysis of color change results because it represents the brightness of the samples. The human eye is more sensitive to variation in this axis than in the a^* axis and b^* axis, because the number of cells responsible for black and white vision (rods) is much higher than the number of cells responsible for color vision (cones). Therefore, any loss or gain in luminosity will be visualized and interpreted as a color change (11).

The material CR showed no significant change ($p > 0.05$) in luminosity after the teeth were submitted to the different temperatures. From the L^* analysis alone, the authors were unable to confirm whether the CR was subjected to heat, or at which temperature, up to 300°C, whereas for GIC, there was a decrease at the L^* coordinate values right after being submitted to 100°C. In other words, there was a loss of luminosity; the higher the temperature to which GIC was submitted, the greater was the darkening observed.

By comparison between the materials, the authors verified that up to 100°C, there was no difference ($p > 0.05$) between them,

due to the decrease in the L^* coordinate of GIC that led to its pattern of luminosity being really close to that of CR. At 200°C and 300°C, the great loss of luminosity in GIC made it distinguishable ($p < 0.05$) from CR. Thus, if the materials have been submitted to temperatures from 200°C to 300°C, it is only possible to discriminate between them by analysis of the L^* coordinate. The color changes in GIC might have been influenced by the water loss by evaporation due to the more intense heat from exposure to higher temperatures. The dryness of the material can result in a whiter and more opaque tonality (30), as demonstrated by the loss of luminosity in GIC by exposure to higher temperatures.

Changes in the a^* coordinate referred to the increase or decrease in the red matrix saturation (31). By the action of heat, the authors verified an increase in red saturation for RC that was more intense at the temperature of 300°C. The saturation of red remained stable between 100°C and 200°C. From 200°C to 300°C, the increase was really significant ($p < 0.05$), for both CR and GIC. The authors found that from analysis of the a^* coordinate only, there was no way to differentiate between the two materials.

Changes in the b^* coordinate values indicated changes in saturation of the yellow matrix (31). For CR, there was an increase in the b* coordinate values. In other words, there was an increase in yellow saturation for the three temperatures tested, and the "yellowness" was more intense at 300°C, which could result from degradation of the camphorquinone (photoinitiator) (25). For GIC, there was more "yellowness" in the material when it was exposed to 100°C. At 200°C, there was a less intense increase in the yellow tonality, whereas at 300°C, negative Δb^* values were found, indicating the "whitening" of GIC, probably due to the dryness of this material. Thus, it was not possible to distinguish between the materials when they were submitted to temperatures of up to 200°C, but the materials were easily distinguishable at 300°C.

Considering all the results, the authors were able to say that the color changes in CR and GIC could indicate whether these materials had really been exposed to heat, and this revealed how to distinguish between them in cases of identification of carbonized victims.

Conclusions

The authors concluded that heat was able to produce color changes in both studied esthetic materials; these changes were more intense, the higher the temperature to which they were submitted, up to 300°C. The color changes were more significant for GIC at all the temperatures analyzed. Furthermore, the tests proposed in this study were shown to be practicable and feasible and able to help forensic dentistry to discriminate between CR and GIC by means of instrumental color analysis up to 300°C, considering the overall color changes and changes in all the color coordinates involved. This can facilitate the process of victim identification.

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