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THE EFFECT OF HIGH TEMPERATURE HEATING ON WEIGHT AND COLOR CHANGES IN COMPOSITE RESIN AS FORENSIC IDENTIFICATION

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ABSTRACT
Background: *Fire disaster is a non-natural disaster that often occurs. The impact is that victims are difficult to identify. One of the identification methods that can be used is dental restorative materials with observing changes in color and weight that occur in dental restorative materials due to exposure to high temperatures. Composite resin is a dental restorative material that is often used because it has good aesthetics, physical and mechanical properties.* ***Purpose:*** *This study to determine the effect of high temperature on weight and color changes in nanohybrid and bulkfill composite resin as a reference for forensic identification.* ***Method:*** *This research is an experimental laboratory with a pre and post-test control group design. A total of 30 nanohybrid composite resin samples and 30 bulkfill composite resin samples were divided into 5 groups consisting of 4 treatment groups and 1 control group. The treatment group was heated at four temperature groups, 30°C-200°C, 30°C- 400°C, 30°C-800°C and 30°C-1100°C using a furnace (Thermo Scientific, USA). Weight changes were measured using a digital scale (Ohaus,China) and color changes were observed using the Shade Guide (Vitapan Classical, Germany).* ***Result:*** *A change in color and weight of the nanohybrid composite resin and bulkfill after being heated at high temperatures. Two-Way ANOVA test and Post Hoc LSD test showed that there was a significant difference in weight between nanohybrid composite resin and bulk fill composite resin at each temperature interval (p<0.001).* ***Conclusions:*** *High temperature heating affects the color and weight changes of nanohybrid and bulkfill composite resin.*

Keywords: *Bulkfill composite resin, High temperature heating, Nanohybrid composite resin*

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INTRODUCTION

Disasters are the result of a vast ecological breakdown in the physical and functional relations between man and environment, caused by nature or man, a serious and sudden event on such a scale that available resources cannot meet the requirements, and the stricken community needs extraordinary efforts to cope with the damaging situation, often with outside help or international aid.[1] Fire is classified as a non-natural disaster, which can be caused by various factors which in general can be caused by human or technical factors.[2]

Causes of fires such as fires that occur in building structures where victims can be exposed to high temperatures reaching 350-800^{0}C.[3,4] Transportation accidents involving gasoline such as cars and planes can be exposed to high temperatures of up to 1100^{0}C.[5] Other conditions such as fires caused by the ejection of volcanic eruptions can reach temperatures of 200^{0}C [6,7,] and bonfires that can reach temperatures of around 400^{0}C.[8] Deaths from fires, especially if exposed to high temperatures can cause tissue and organ damage, making it difficult to identify especially in mass fire.[9]

Identification is a step that can be used to detect the victim's body.[10] Identification using dental restorative materials has its own and different shapes for each individual so that it can be used as evidence to support the proper identification process in revealing the identity of the victim.[10] Dental restoration materials that are widely used in dentistry today are composite resins.[11] It is because they have good aesthetic, physical, and mechanical properties.[12] Nanohybrid composite resin is one of the composite resin innovations that have superior properties rather than the other types of composite resins. The nanohybrid composite resin contains a resin matrix of TEGDMA, UDMA, Bis-GMA, Bis-EMA, milled glass fillers, and nano particles (40-50 nm).[13]

In addition to nanohybrid composite resins, there are composite resin innovations for posterior dental restorations, namely the bulkfill type which was introduced in 2010. The composition of the bulkfill type composite resin includes a resin matrix consisting of TEGDMA, Bis-GMA, and Bis-EMA. In addition, there are inorganic materials, initiators, inhibitors, and pigments which have developed compared to other types.[14]

Composite resins exposed to high temperatures may result in physical and mechanical changes such as changes in weight, color, texture, cracks, fractures, and dimensional stability as a basis for comparison of ante-mortem and post-mortem data for the forensic identification process of victims who died burned or charred and estimate the temperature of the fire so that it can be used as medicolegal documentation.[15] This study aims to find out whether there were changes in weight and color in nanohybrid and bulkfill composite resin after being heated at high temperatures so that they could assist in determining the identity of burn victims.

MATERIALS AND METHODS

This research was conducted at the Dental Preclinical Laboratory, Biomedical Laboratory, and the Integrated Laboratory at Diponegoro University, Semarang (Ethics No. 59/EC/H/FK-UNDIP/VI/2021). This research is an experimental laboratory with Pre-test and Post-Test Control Group Design. This study used 60 samples of restorative materials consisting of 30 samples of nanohybrid composite resin and 30 samples of bulk fill composite resin where each material consisted of 4 treatment groups and 1 control group.

Composite resins were printed from metal molds with a diameter of 3 mm and a thickness of 2 mm, compressed with a load of 500 grams for 30 seconds and the excess filling was removed. The sample was light cured (LED B Woodpecker, China) for 20 seconds as close as possible to the sample surface and continued with finishing and polishing. The sample is removed from the metal moulds. The sample was divided into ten groups by simple random sampling technique.

The samples were measured by digital microscale (Ohaus, China) and each sample weight was limited to 43-46 mg. The samples were grouped into groups I, II, III, IV, and V for nanohybrid composite resins and VI, VII, VIII, IX, and X for bulk fill composite resins.

Groups I and IV were put into the furnace at a temperature of 30^0C, then the temperature was raised to 200^0C for 15 minutes, the temperature was lowered back to 30^0C, and cooled for 15 minutes.The furnace was set again at 30°C, then groups II and VII were included and given the same treatment up to a temperature of 400°C. Likewise with groups III and VIII (800°C) and IV and IX (1100°C). Groups V and X were control groups that were not given any heat treatment.

The samples were observed for color changes (before and after heating) using Shade Guide (Vitapan Classical, Germany) by 3 observers. Then, the sample was re-measured with a digital micro scale and compared before and after heating.

RESULTS

Research data on weight changes were analysed using a statistical program (IBM SPSS, v. 25, USA). The results of the change in weight and the results of the Two-Way ANOVA test (p value <0.05) obtained the following results in table 1 and table 2:

Table 1. Results of changes in weight of nanohybrid composite resin (in percent) and p value

Temperature	**Initial Weight (%)**	**Remaining Weight (%)**	**Weight Loss (%)**	**p *value***
$30^0 – 200^0$C	100	98.3	1.68	
$30^0 – 400^0$C	100	91.1	8.88	<0.001
$30^0 – 800^0$C	100	79.52	20.47	
$30^0 – 1100^0$C	100	74.75	25.25	

The Two-Way Anova Test showed a P significance value of <0.001. The results of the significance value of p<0.05 showed that there was a weight difference between nanohybrid composite resin at each temperature interval.

Table 2. Results of changes in weight of bulkfill composite resin (in percent) and p value

Temperature	**Initial Weight (%)**	**Remaining Weight (%)**	**Weight Loss (%)**	**p *value***
$30^0 – 200^0$C	100	96.21	3.79	
$30^0 – 400^0$C	100	87.27	12.76	
$30^0 – 800^0$C	100	76.73	23.27	<0.001
$30^0 – 1100^0$C	100	72.07	27.93	

The Two-Way Anova Test showed a P significance value of <0.001. The results of the significance value of p<0.05 showed that there was a weight difference between bulkfill composite resin at each temperature interval.

Table 3. Post Hoc LSD Test between Two Composite Resin

Sample Groups	*Bulkfill* 30^0 – 200^{0}C	*Bulkfill* 30^0 – 400^{0}C	*Bulkfill* 30^0 – 800^{0}C	*Bulkfill* 30^0 – 1100^{0}C	*Bulkfill* Kontrol
Nanohybrid 30^0 – 200^{0}C	<0.001	<0.001	<0.001	<0.001	<0.001
Nanohybrid 30^0 – 400^{0}C	<0.001	<0.001	<0.001	<0.001	<0.001
Nanohybrid 30^0 – 800^{0}C	<0.001	<0.001	<0.001	<0.001	<0.001
Nanohybrid 30^0–1100^{0}C	<0.001	<0.001	<0.001	<0.001	<0.001
Nanohybrid Control	<0.001	<0.001	<0.001	<0.001	-

The LSD Post Hoc Test showed a P significance value of <0.001. The results of the significance value of p<0.05 showed that there was a significant difference between the nanohybrid composite resin and bulkfill at each temperature interval.

The results of the color changes observed by three observers are as follows:

(1)

(2)

(3)

(4)

(5)

Fig. (1) Group V which was not heated (Shade Guide number A2 from brand Vitapan Classical) (2) Group I after heating at 200°C (3) Group II after heating at 400°C (4) Group III after heating at 800 °C (5) Group IV after heating to 1100°C.

(6)

(7)

(8)

(9)

(10)

Fig: (6) Group X which was not heated (Shade Guide number A2 brand Vitapan Classical) (7) Group VI after heating at 200°C (8) Group VII after heating at 400°C (9) Group VIII after heating at 800 °C (10) Group IX after heating to 1100°C.

DISCUSSION

The research that has been carried out aims to determine the changes that occur from composite resin restoration materials, especially changes in weight and color due to exposure to high temperatures, which can later complement post-mortem data so that it can help identify fire victims. High temperatures can cause changes in the restorative materials including composite resin.[16] In this study, treatment was carried out in the form of heating with different temperature levels on nanohybrid composite resin and bulkfill composite resin using a high temperature furnace with an exposure time of 15 minutes for each treatment group. The use of a high temperature furnace can be described as the oral cavity if exposed in the event of a fire, the first part that burns is the outer part of the body and the oral cavity such as the skin of the face, cheeks, lips, so that the teeth and restorative materials are still only exposed to the hot temperature of the fire.[16]

In the heating group at temperatures of 30–200^{0}C, nanohybrid composite resins experienced an average weight reduction of 1.68%, while bulkfill composite resins experienced an average weight reduction of 3.79%. The weight reduction can occur as a result of heat treatment, namely the monomer will be covalently bonded to the polymer network and then the monomer will be evaporated during the heating process.[16]

In the heating group at temperatures of 30–400^0C, nanohybrid composite resins experienced an average reduction of 8.88% and bulkfill composite resins experienced an average reduction of 12.76%. The weight loss that occurs on heating at this temperature is caused by the increased mobility of the polymer network so that it will cause further conversion of monomers, increase crosslink density, and also allow for some stress on the chemical structure of the composite resin, especially stress on the resin matrix.[16]

In the heating temperature group of 30-800^0 C, the nanohybrid composite resin experienced an average weight reduction of 20.47% and bulkfill composite resin of 23.27%. At a temperature of 30-1100^0C, the nanohybrid composite resin experienced a weight reduction of 25.25% and bulkfill composite resin by 27.93%. This is due to a complete degradation in the constituent components of each composite resin itself, such as the resin polymer matrix, coupling agent, water, activator, and initiator which melts at that temperature so that it only leaves residue, namely silica/zirconia filler which only melts at temperature 1710-2100^0C.[17]

The difference in weight reduction that occurs between nanohybrid and bulkfill composite resins can be caused by several factors. Differences in the organic matrix such as monomer type and photoinitiator may play a role in the observed results. Variations between monomers of different resins result in different properties and degrees of conversion. The less the amount of matrix in the composite resin, the greater the possibility of degradation. The number of matrix of bulkfill composite resin is less than that of nanohybrid composite resin, so the weight reduction is greater for bulkfill composite resin than nanohybrid. [13.16]

The nanohybrid composite resin contains a resin matrix consist of dimethacrylate monomers, namely triethylene dimethacrylate (TEGDMA), modified urethane dimetacrylate (UDMA), bisphenol-A-glycidyl methacrylate (Bis-GMA), and ethoxylated bisphenol-A dimethacrylate (Bis-EMA). Meanwhile, bulkfill composite resin contains a resin matrix consisting of triethylene dimetacrilate (TEGDMA), modified bisphenol-A-glycidylmetacrylate (Bis-GMA), and ethoxylated bisphenol-A dimethacrylate (Bis-EMA). The matrix degradation mechanism of Bis-GMA, Bis-EMA, UDMA and TEGDMA is different. Bis-GMA and bis-EMA exhibit a one-step degradation mechanism with maximal rates at 415^0 and 424^0C. In contrast, TEGDMA and UDMA showed two degradation steps with maximum levels at 357^0 and 444^0C for UDMA then 306^0 and 403^0C for TEGDMA.[13]

High temperature heating will also cause color changes in nanohybrid and bulkfill composite resins. After heating at a temperature of 30-200^0C, the nanohybrid composite resin changed to shade C1 from the initial color, shade A2. Meanwhile, for bulkfill composite resin, the color changes to shade A1 from the initial color shade A2. Bulkfill composite resin has a lighter color than nanohybrid composite resin. The color change that occurs at this temperature can be caused by surface burning associated with the persistence of free radicals of the inhibitory layer produced during polymerization. In addition, the chemical additive content of composite resins can cause discoloration when exposed to high temperatures. Photoinitiators and inhibitors can be the cause of color discoloration.[18]

The difference in color changes that occurs between the nanohybrid composite resin and bulkfill composite resin at this temperature is related to the difference in the number and types of constituent components used in each composite resin. The effect of discoloration to yellowish or whitish can be caused by the degree of degradation of champorquinone (photoinitiator). After heating, the bulkfill composite resin has a lighter color than the nanohybrid. This can be caused by the amount of champorquinone contained in the bulkfill composite resin is higher than the nanohybrid composite resin. If champorquinone is completely degraded, a yellowish color effect may occur.[16,18]

After heating at a temperature of 30–400^0 C, the nanohybrid composite resin and bulkfill composite resin turned black. This can be caused by chemical changes in the pigment or further oxidation that occurs in the resin polymer matrix, then the destruction of the pigment by heating. Opacity at a temperature of 30–400^0C can be related to the melting point of the material has been reached, which can cause carbonization and total darkening of the sample. Increasing the heating temperature on the nanohybrid and bulkfill composite resin removes impurity components such as water and mineral content in the composite resin which can increase the carbon content in the composite resin.[16,18]

After heating at temperatures of 30–800^0C and 30–1100^0C, the nanohybrid and bulkfill composite resin both changed to opaque white. It can be observed that the increase in opacity of the nanohybrid composite resin and bulkfill composite resin increased with thermal increase. The color change to white can caused by the melting of certain components in the composite resin such as the resin polymer matrix, coupling agent, water molecules, activator and initiator compounds, but silica components which have very high melting points do not melt at temperatures of 800^0 and 1100^0C. Causes a white color change in the two composite resins because the silica is pure white.[16,18]

The limitations of this study were the use of sample molds and not using the teeth. Then, after the specified temperature was reached the sample was removed from the furnace and allowed to cool at room temperature. In fact, many factors can complicate the heating effect on tissues and restorative materials such as exposure time and rate of temperature rise. In addition, the analysis of color change observations in this study is also a subjective method that depends on the individual criteria of each observer, so that it can produce different interpretations of the color changes that occur. High temperature heating affects the weight and color changes of nanohybrid and bulkfill composite resins. There was a statistically significant difference in weight change in the nanohybrid composite resin and bulkfill after a significant increase in temperatures of 30^0-200^0C, 30^0-400^0C, 30^0-800^0C, and 30^0-1100^0C. There are different changes in the nanohybrid composite resin and bulkfill after being added at temperatures of 30^0-200^0C, 30^0-400^0C, 30^0-800^0C, and 30^0-1100^0C.

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