

## Effects of Different Aging Methods on Color Change of Bulk-Fill and Anterior Resin Composites

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### Abstract

The purpose of this study was to evaluate different aging method effects on color change of bulk-fill resin composite (BFRC) and anterior resin composite (ARC).

Sixty disc-shaped samples (8-mm in diameter and 4-mm thick) were prepared (30 samples in each composite). Samples were divided into 3 groups according to different aging procedures which were thermal cycling [TC], autoclave aging [AA] and water aging [WA] (n=10). Color measurements of the samples were recorded before and after aging. Color changes were calculated using the CIEDE2000 ( $\Delta E_{00}$ ) formula. Color measurements of the samples were recorded before aging. After aging, color measurements were repeated and color changes were calculated using the CIEDE2000 ( $\Delta E_{00}$ ) formula. Color differences were analyzed using a two-way analysis of variance and Holm-Sidak comparison. Significant differences were observed among the composite groups and aging methods ( $p < 0.05$ ).

All aging methods caused color change in composites. ARC showed less color change than BFRC. WA caused minimal color change and showed significantly difference than other aging methods. However no significant difference in color change were found between TC and AA.

The results provide information on the color stability of composite resins ARC and BFRC after different aging methods. In terms of esthetic dentistry, ARC composites should be used in the anterior region, where there is a high risk of caries due to coloration.

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### Introduction

Today, the expectations of patients are not only durable restorations, but also esthetically demanding restorations with satisfactory results. The increase in physico-mechanical and esthetic properties of composites enabled them to dominate a large area in esthetic dentistry. However, despite improvements in the monomer and filler composition, the color changing of composite resin restorations over time may still be a problem.

Flowable and high viscosity Bulk-Fill Resin Composites (BFRC), has gained popularity these days due to the convenience of application techniques. It has been proposed that

conventional composite resins should be layered with thicknesses of no more than 2 mm to achieve effective light transmission and adequate polymerization.<sup>1</sup> However, the layered application of the composite resin has several disadvantages; Separate light polymerization of each layer is time-consuming, since air bubble capture and isolation between layers must be achieved.<sup>2</sup> Unlike conventional composites with a maximum thickness of 2 mm, BFRC can be applied with a single layer of 4 mm.<sup>3,4</sup> These composites include monomers and filler compounds similar to conventional composite resins, except for the polymerization modulators and plasticizers used to modulate polymerization kinetics.<sup>5</sup> The new stress-decreasing monomers, more reactive photoinitiators and different types of fillers in nanosizes enable a novel formulation for the composite resins which allow the use of bulk-filling technique.<sup>6</sup> In these composites, the increase in particle dimension enhanced mechanical resistance while reducing the content to facilitate light infiltration to deeper layers.

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Researchers reported that BFRC showed a better adaption to the cavity walls and lower shrinkage stress.<sup>7</sup>

Oral cavity restorations; it is exposed to a wide range of factors such as different humidity, temperature changes, light, nutrients. In composite restorations, color changes occur as a result of external factors such as consumed foods, beverages and tobacco use.<sup>8,9</sup> The internal color change in the deeper layers of composite includes various factors related to the composition of the materials. More specifically, the type of photo-initiator, the structure of the resin matrix, filler content and particle size distribution are related to color stability.<sup>10,11</sup> The color stability is also influenced by the density and duration of the polymerization, and consequently by the degree of the conversion. Some physicochemical actions, like visible light and ultraviolet irradiation, temperature and humidity, cause internal color change in composite resins.<sup>12</sup>

Artificial aging methods have been used as tools for estimating the possible changes in the mechanical and optical properties of composites in a short time for this reason.<sup>13-15</sup> The main factor responsible for color change of the composite over time is the changes made in the organic matrix.

Accelerated aging methods used in the literature are; Water aging (WA), Artificial Accelerated Aging (AAA), UV-light aging (UVA), thermal cycling (TC) and aging in autoclaves (AA). AAA consists of exposing restorative materials to UV-B rays, humidity and sudden changes in temperature. There is no standardization of the aging time required to increase the color change in composites after AAA. Pires-de-Souza et al.<sup>16</sup> found that 300 hours of AAA corresponded to approximately 1 year of clinical use. In the same way, waiting for 30 days at 37 ° C in water, exposure to UV light for 120 hours at 37 ° C, 10,000 cycles of TC and 30 min AA corresponds to one year of clinical use. In our knowledge, there is no study that compare aging methods on color stability of composites.

The aim of this study was to compare the effect of different aging methods on the color stability of two different composites. The hypothesis of the study is that all in vitro aging methods and composites will cause similar color changes.

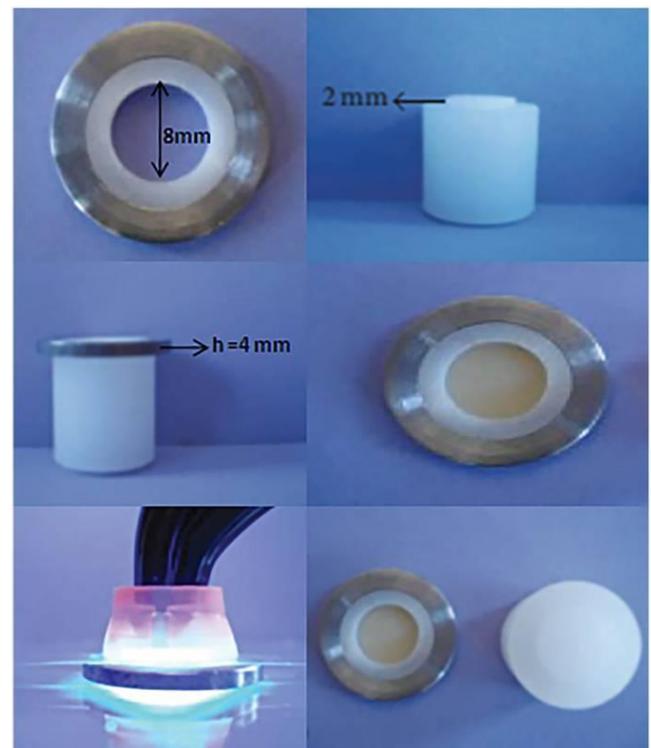
## Materials and methods

BFRC and Anterior Resin Composite (ARC) were used in this study. The compositions of the composites are shown in Table 1.

Resin composite	Main components	Manufacturer
Filtek Bulk Fill posterior restorative (Shade A2)	The inorganic filler (76,5% by weight-58,4% by volume); The filler (non-aggregated 20 nm silica filler, a non-agglomerated/non-aggregated 4-11 nm zirconia filler; an aggregated zirconia/silica cluster filler (20 nm silica and 4-11 nm zirconia particles); ytterbium trifluoride filler (agglomerate 100 nm particles); The monomer matrix contains AUDMA, UDMA, 1,12-dodecane-DMA.	3M ESPE
Estelite Σ Quick (Shade; A2)	Silica-zirconia filler and composite filler (Contains 82% by weight-71% by volume); Inorganic fillers contained are a spherical submicron filler (mean particle size: 0,2 µm, particle size range: 0,1 to 0,3 µm ); The monomer matrix contains Bis-GMA and Triethylene glycol dimethacrylate	Tokuyama Dental

**Table 1.** Materials used in this study.

Sixty disc-shaped specimens (8-mm in diameter and 4-mm thick) were prepared, with 30 specimens in each composite. Samples were divided into 3 groups according to different aging procedures (TC, WA, AA) (n=10).



**Figure 1.** Preperation of samples.

### Sample Preparation

ARC samples were prepared by 2 mm incremental technique. BFRC samples prepared in single 4 mm layer. Filled mold surface was covered with a Mylar film and the upper and

bottom surfaces of the mold were covered with glass slabs before polymerization to produce a smooth surface and finger pressure was applied to extrude excess composite. The composite disks were polymerized for 40 seconds with using a light-emitting diode (LED) curing unit (Elipar S10; 3M ESPE; St. Paul, MN, USA) at a light intensity of 1200 mW/cm<sup>2</sup> and a wavelength of 430–480 nm (wavelength peak 455 nm) (Figure-1). The tip of the LED was in contact with glass during polymerization. The surfaces of the samples were fixed with 800–1.200 silicon carbide grit. Afterwards, the samples were stored in distilled water for 24 hours at 37° C.

### Artificial Aging Methods

**TC:** The samples in the TC group were passed through a total of 10.000 cycles. For the TC test, the Universal-Testing Machine MTD-500 Plus thermal cyler was used in the Konya Necmettin Erbakan University Faculty of Dentistry Research Laboratory. During the TC test, the device was set to have a transfer time of 7 seconds and a standby time of 30 seconds in the baths of the samples at temperatures of 5 ° C and 55 ° C (± 2 ° C), respectively.

**WA:** Samples were immersed in 1 mL of water at 37 ° C for 30 days. Water was changed every 7 days to prevent biological contamination. This was chosen for 30 days to ensure sufficient time for the samples to be saturated with water.

**AA:** Samples placed in glass containers with distilled water, Konya Necmettin Erbakan University Faculty of Dentistry in the autoclave in the laboratory (Eryiğit Steam Sterilizer, Ankara, Turkey) 1 year aging to simulate 134 ° C at a pressure of 200 kPa for 30 min (ISO standard 6474-2; (ISO, 2008)).

### Color Measurement

Color measurements were performed with Lovibond RT Series Reflectance Tintometer UK spectrophotometer. The probe tip was then placed perpendicular to the center of each sample and each measurement was repeated 3 times to obtain accurate measurements. The color of each sample was measured and recorded before and after aging. All procedures were performed by the same operator.

The CIEDE2000 ( $\Delta E_{00}$ ) formula was used to calculate the total color change in composite resin samples:

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \left(\frac{\Delta C'}{k_C S_C}\right) \left(\frac{\Delta H'}{k_H S_H}\right)}$$

$\Delta L'$ ,  $\Delta C'$ , and  $\Delta H'$  define changes in lightness, chroma and hue between two samples compared.  $S_L$ ,  $S_C$ , and  $S_H$  are the weighing functions for the lightness, chroma, and hue parameters, respectively.  $K_L$ ,  $K_C$ , and  $K_H$  are the parametric factors to be regulated according to different viewing parameters. In this study,  $K_L$ ,  $K_C$ , and  $K_H$  were set to 1<sup>17</sup>.

### Statistical Analysis

A two-way analysis of variance test was used to determine the effect of the composites and aging methods on color changes. Holm-Sidak Comparison significant difference test was used to determine the differences between the groups at  $p < 0.05$  with using SPSS software (version 9.1).

### Results

The color change ( $\Delta E_{00}$ ), mean values and standard deviations observed after different aging methods are shown in Table 2.

There were statistical differences in color change between composites ( $p < 0.05$ ). ARC showed statistically less color change than BFRC ( $\Delta E_{00}$ : 1.80 - 2.36 respectively) ( $p < 0.05$ ).

	TC	WA	AAA	N
Bulk	2.5±0.8 <sup>ax</sup>	10 1,3±0,6 <sup>bx</sup>	10 3,2±0,2 <sup>cx</sup>	10
Anterior	2.1±1.5 <sup>ax</sup>	10 1,7±0,1 <sup>by</sup>	10 1,5±0,1 <sup>by</sup>	10
	2,3±0,8 <sup>a</sup>	20 1,5±0,2 <sup>b</sup>	20 2,3±0,9 <sup>a</sup>	20

**Table 2.** Color changes ( $\Delta E_{00}$ ) mean values and standard deviations after different aging methods.

There is no statistical difference between the same letters in the same row (a,b,c) and column (x,y) ( $p < 0.05$ ).  
 PT and AT values: 0.81 and 1.77 units ( $\Delta E_{00}$ )<sup>17</sup>

WA showed significantly difference in color change than other methods ( $\Delta E_{00}$ : 1.52) ( $p < 0.05$ ). WA caused minimal color change in both composites. However no significant difference in color change were found between TC and AA ( $\Delta E_{00}$ : 2.34 – 2.38) ( $p > 0.05$ ).

Statistically significant difference was found between all aging methods in BFRC. While there was a significant difference between TC-AA and TC-WA groups in the ARC, no significant difference was found between AA-WA groups.

There was no significant difference in color change between ARC and BFRC in the TC. There is a significant difference between the ARC and BFRC groups in WA and AA.

## Discussion

The hypothesis that composites and all in vitro aging methods would cause similar color changes in the experimental resin-based composite was rejected.

The CIEDE2000 uses the minds of chroma and hue, supporting the importance of the notional advancements of Munsell.<sup>17</sup>  $R_7$  defines that accounts for the interaction between chroma and hue differences in the blue area (the so called rotation term).<sup>18-19</sup>  $\Delta E_{00}$  represents the overall color change that can be reported by an observer. In recent years, CIEDE2000 color difference has been increasingly applied in color studies. Studies have shown that  $\Delta E_{00}$  correlates better with visual perception than  $\Delta E_{ab}$ . Therefore, CIEDE2000 color difference metric was used in this study.<sup>20,21</sup> In present study; the color differences were interpreted by comparisons with 50:50% perceptibility and 50:50% acceptability thresholds. The perceptibility (0.81 units) and acceptability (1.77 units) values for CIEDE2000 (1:1:1) were obtained from a actual study.<sup>22</sup> Aging produces an erosion on the surface of restorative materials, which exposes the components that contribute to the dyeing of composite resins, because the color changes are related to the porosity of the surface and the degradation of the components.<sup>23</sup>

The color change varies according to the type of monomer used for each material. It is claimed that the presence of urethane dimethacrylate (UDMA) may result in a better conversion and therefore the material will show a higher color stability. Researchers reported that Bisphenol a glycidyl methacrylate (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) reduced color stability of the material due to their higher tendency to absorb water<sup>24</sup>. According to the results of this study, despite the presence of UDMA in the content of BFRC, it showed lower color stability compared to the ARC with Bis-GMA and TEGDMA. The reason for this contradictory result may be that the difference filler contents and filler particle size.

According to some authors, the color stability of these materials is directly related to

the size, type and volume of charged particles, the type of matrix and monomer used, and the degree of dependence on the composite resin matrix and coloring agents.<sup>19,25-27</sup> Since the matrix content is associated with the water diffusivity and discoloration of the composites, therefore more color changes can be expected due to the higher matrix-fill rate.<sup>19</sup>

In addition to the matrix content and composition, the filler size and distribution affect the hydrolytic degradation of the matrix and filler interface and affect the color stability of the composites as it changes the scattering directions of the light at the filler-matrix interface.<sup>22,28</sup> Composites which have low water absorption, high amount of resin filler, reduced particle size, hardness and optimal fill- matrix bonding system are more resistant to color change.<sup>29</sup> ARC (82% by weight (71% by volume)) which is used in this study has a higher filler ratio than BFRC (76,5% by weight (58,4% by volume)). The filling particles are nano-sized in BFRC (4-100 nm) and micro-sized in ARC (0,2  $\mu\text{m}$ ). This study is in conflict with the results of Inokoshi at all, which states that a higher matrix-filler ratio will lead to a higher color change.<sup>30</sup> The results of the study showed less color change in ARC which had higher filler content. This result is in consistent with the study of Dietschi at all. They argued that lower particle size would provide better color stability.<sup>29</sup> Researchers also states that composite resins with large particles are more susceptible to water absorption and color changes.<sup>31</sup> However in present study, the BFRC, which has a lower particle size, showed more color change. The reason for the different results may be arise from the difference of contents and the aging methods.

When the aging methods were compared in ARC, the most change in color was observed in the TC. This method simulates the effect of hot and cold substances on the teeth, and shows the relationship between linear thermal expansion coefficient between tooth and restorative material.<sup>32</sup> Many studies use TC temperature changes (5°C-55°C), which are determined according to ISO standards. At rest, the temperature in the mouth was reported to be 36.4°C.<sup>33</sup> However, food and beverages are sometimes consumed cold, sometimes hot, and the temperature in the mouth changes. It has been reported that the temperature of consumed

foods and beverages varies between 50-60°C or 0-10°C.<sup>34</sup> These values are reported to be 85°C for coffee and -12°C for ice cream.<sup>35</sup> This may be due to the fact that the temperature of the water to which the composite samples are exposed in the TC is not stable. Increased temperature in short time intervals may cause more color changes in composites.

In the other two aging methods, while the temperature was constant. Color changes may attributed with the temperature differences in TC. AA samples showed more color changes, it may be due to the pressure exposure.

Most studies show that color change is not only dependent on the water immersion time, but also due to the composite formulation, differences in the refractive index and the hydrophobicity of the monomers.<sup>36-38</sup> Less color change in the WA samples may be related with these reasons. Further studies should be done to support this conclusion.

## Conclusions

Within the limitations of this study, the following conclusions can be drawn:

1-WA caused a significantly lower color change compared to TC and AA.

2- ARC showed statistically less color change than BFRC.

3- No significant difference in color change were found between TC and AA.

4- ARC can be used successfully in the anterior region without worrying about discoloration. BFRC should not be used in the anterior region, especially when the esthetic expectation is high.

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## Declaration of Interest

The authors report no conflict of interest.

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