

## Discoloration of CAD/CAM Blocks and Bulk-Fill Composite Resin Materials After Thermocycling

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### Abstract:

**Background:** Despite the recent developments in aesthetic materials, the coloring of dental materials poses a major problem. Color stability is an important criterion in restoration success. This study aimed to examine the effect of the thermocycling process on coloration on the newly available materials such as CAD/CAM blocks and bulk-fill resin composites.

**Materials and Methods:** The two CAD/CAM block (Shofu Block HC, Vita Enamic) and one bulk-fill resin (Tetric N Ceram) composite were tested. A total of 45 disks (30/block and 15/bulk-fill resin composites, 10 x 4 mm) were fabricated. Fifteen specimens/bulk-fill resin composites cured as the control. One side of the sample disks was polished according to the manufacturer's recommendations with the specially produced polish sets. The samples were subject to 5,500 cycles thermocycling (5-55°C). Color values were measured 3 times for each sample after and after thermocycling with spectrophotometer and their averages were. The color change ( $\Delta E$ ) of each sample was calculated. Data were analyzed with One-way ANOVA ( $\alpha=0.05$ ) and post hoc test (Tamhane's T2).

**Results:** Among the mean  $\Delta E$  values for the tested materials were statistically significant ( $p<0.05$ ). Tetric N Ceram bulk-fill resin composite had higher color change value ( $\Delta E_{TC}=2.99$ ) than Shofu Block HC ( $\Delta E_{SH}=1.07$ ) and Vita Enamic ( $\Delta E_{VE}=0.50$ ) CAD/CAM block materials. Vita Enamic block material showed better color stability than others.

**Conclusion:** CAD/CAM blocks and bulk-fill resin composite after thermocycling showed different color stability. The color stability of CAD/CAM hybrid blocks is higher than the bulk-fill resin composite. The color changes of the tested materials are considered clinically.

**Key Word:** CAD/CAM block; Discoloration; Bulk-fill resin composite; Thermocycling

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### I. Introduction

With the increase of aesthetic expectations, minimally invasive approaches have increased in cavity preparations, and more aesthetic composite resin restorations have begun to be used.[1] The use of composite resins in the restoration of posterior teeth has increased due to the reliability of the material.[2] Their bending strength and fracture resistance are similar to amalgam and porcelain. They have sufficient mechanical properties.[3]

There are disadvantages of polymerization shrinkage, difficulty in adaptation to cavity walls, microleakage, development of secondary caries, crack formation in the tooth or restoration, and eventually loss of restoration[4]. Therefore, to solve these problems, manufacturers made improvements in the organic and inorganic structure of the composite resin, the polymerization method of the material and the light devices.[5], [6]

The incremental technique used to prevent polymerization shrinkage, there is a gap between the composite layers, contamination, failure to bond, difficulty in adaptation to the cavity and long processing time caused the search for new materials[7], [8].

In recent years, a new type of composite resin called "bulk-fill" composite resin, which has improved physical and mechanical properties and can be applied in a single layer into the cavity, has been introduced. [9] This resin contains particles/filler like conventional nanohybrid composite resins. They also contain regulators and plasticizers (plasticizers) that regulate polymerization.[10] Filler content and particle size are very important in the distribution of the light beam.[11] Therefore, the transparency of the material has been increased, and the proportion of filler contents has been reduced to facilitate the transmission of light to deeper particles larger than 20  $\mu\text{m}$  were used to improve the mechanical strength of the material.[12], [13]

The clinician aims to restore the patient with a restorative material that has a feature similar to the lost tooth tissues. Restoration is identical to the morphological and surface features of the tooth, and there should be a good match between the remaining dental tissues and the color of the restoration. The optical properties of the

remaining dental tissue and the amount of missing tissue should be taken into account when performing the restoration.[14] The color of natural teeth emerges as a result of combining the optical properties of enamel and dentin.[15]

Despite the improvements in aesthetic materials, the coloring of dental materials poses a big problem. Color stability is an important criterion in restoration success.[16]–[18] The discolorations occurring in restorative materials are internal and external.[19]

External colorations occur depending on the coloring of the superficial layer of the composite-containing resin material, smoking and feeding habits, the accumulation of plaque, the pigmentation of the coloring agents, the time the material is exposed to the coloring agent, the surface roughness of the material as a result of finishing and polishing processes. [16], [18], [20] Intrinsic coloring occurs as a result of physicochemical reactions in the material. [21] The internal color change of composite-containing resin materials depends on dehydration, water absorption, oxidation of unreacted carbon double bonds, and the properties of the material (matrix structure, filler content, matrix-filler interface, polymerization degree.[16]–[18], [20], [22]–[25] Polymeric matrix, fill size, fill shape and silanization are effective in reducing polished surfaces. When the filler particles are much stiffer than the surrounding resin matrix, the resin matrix is particularly soft compared to the inorganic matrix tears.[26]

In cases where aesthetic expectations cannot be achieved through direct composite restorations, indirect techniques such as CAD/CAM are used for long-term restorations. With this technology, restorations are achieved by using metal-free dental materials with different mechanical and optical properties.[27]

CAD/CAM materials are generally divided into two groups. The first group represents more composite resin-like materials, while the second group represents more ceramic-like materials. The surface roughness and micromechanical properties of the materials in the first and second groups are different from each other.[28] Composite materials tend to wear out due to their soft structure. Ceramic materials are also rigid and fragile materials that cause the wear of the opposite tooth. To eliminate the disadvantages of both of these materials and to produce an ideal restorative material with physical and optical properties similar to the natural tooth, the manufacturers introduced new materials to the market.

A new polymer infiltrated ceramic network (PICN) materials have been developed. These materials include a ceramic and polymer network.[29] PICN materials are less fragile when compared to ceramics. Besides, with high flexibility, they show high fracture strength and can be processed well.[30] With the development of these materials, wear is observed in the enamel of the opposite tooth, similar to composite resins. The purpose of producing PICN materials is to expect to exhibit properties similar to natural tooth structures in the long run.[29] Vita Enamic is the first PICN material on the market.[31] Vita Enamic is a ceramic-based material reinforced by a polymer network, combining the advantages of both ceramic material and composite resin. Vita Enamic consists of 14% polymer mesh diffused into 86% by weight feldspathic ceramic matrix. This material shows high elasticity and fracture resistance.[26] SH resin ceramic material contains zirconium silicate micro-ceramic fillers. The manufacturer stated that the optical properties of this material have translucency and light transmission/distribution similar to natural teeth. Due to its good bending strength, the material can give off load forces. The inorganic filler content is 61% by weight.[32]

The literature on the color stability of Vita Enamic and Shofu Block HC materials is limited. Therefore, this study aimed to compare the optical properties of Shofu Block HC material which has the lowest inorganic filler ratio(61 %), and Vita Enamic material, which has the highest inorganic filler ratio(86 %).

For all these reasons, this study aimed to examine of discoloration of the bulk-fill composite resin and the CAD/CAM blocks. Our null hypothesis is "there would be no different discoloration among bulk-fill resin composite and CAD/CAM blocks materials".

## **II. Material And Methods**

Vita Enamic (VE), and Shofu Block HC (SH) CAD/CAM blocks (test material) and Tetric N Ceram (TC) bulk-fill resin composite (control grup) were selected for this study. Fifteen discs of 10 mm diameter and 4 mm thickness were prepared for each material. Teflon mold (10 x 4 mm) was used to prepare bulk-fill composite samples. The mold was filled with unpolymerized material and covered with transparent tape to prevent the formation of an oxygen inhibition layer. The tip of the light device was applied directly to the sample surface and then polymerized with the LED curing light (BluePhase G2, Ivoclar Vivadent, Schaan, Liechtenstein) for 20 seconds from the top surface of the composite material in accordance with the manufacturer's recommendations. After the samples were removed from the mold, they were also cured to other surfaces with 20 seconds of light.

Standard sizes of CAD/CAM ceramic blocks were used preparing of CAD/CAM samples. Each block was cut into sections (4.2±0.2 mm thickness) with a precision cutting machine (IsoMet 1000, Buehler; Illinois, ITW, USA). Discs with a diameter of 10 mm were obtained from these block sections with trephine bur (Hu-Friedy Mfg. Co., LLC, USA ).

One surface of composite samples was polished with especially kit (Super-Snap Mini-Kit, Shofu INC; San Marcos, California, USA) for 10 sec using low-speed contra-angle. Polished samples were washed with water for 10 sec and air-dried for 10 sec.

Prepared CAD/CAM block sections were polished with Vita Enamic set clinical diamond polishing system(Vita Zahnfabrik, Germany) for VE material and Shofu Cadmaster HP Kit (Shofu Dental GmbH, Germany) for SH material according to manufacturer's recommendations. After the polishing process was completed, the thickness of all samples was measured with a digital micrometre (MCM-25M, Tokyo, Japan), and the thicknesses were standardized to be 4.0±0.1 mm. After all samples in distilled water at 37° C in an incubator (Memmert Edestahl, Rostfrei, Germany) for 24 hours.

Color values (L\*, a\*, b\*) were measured before thermocycling as a baseline and after thermocycling, using a spectrophotometer(Vita EasyShade® Advance 4.0; Vita Zahnfabrik, Bad Säckingen, Germany) on the white background from the centre of polished surface for each sample. White calibration standard was used before each measurement. The color value was measured three times from each sample and the mean value was calculated.

The samples were subject to 5,500 cycles thermocycling (5-55°C) with 60 seconds dwell time. (Moddental, MTE100, Turkey). After the thermocycling procedure, color value measurements were performed.

$$\Delta E = [(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2]^{1/2}$$

Data were calculated and recorded. Clinically acceptable color difference values are reported as  $\Delta E \leq 2.0$ ,  $\Delta E \leq 3.3$  and  $\Delta E \leq 3.7$ . [33] Discoloration between  $1 < \Delta E < 3.3$  is considered clinically acceptable. [19] Clinical significance for CIELAB discoloration ( $\Delta E$ ) was evaluated according to the values in this study.

### Statistical analysis

Statistical software (SPSS 22; SPSS Inc, Chicago, USA) was used to perform the analysis. Data were first analyzed for normality of variables with the Kolmogorov Smirnov, and Shapiro-Wilk tests. Data were not normally distributed. One-way ANOVA and Tamhane's T2 post hoc test were used to compare differences between groups. All analyzes were evaluated according to 0.05 statistical confidence level.

### III. Result

A statistically significant difference was found between the groups in  $\Delta E$  values ( $p < 0.05$ ) (Table 1). The null hypothesis was rejected. Pairwise comparisons performed to determine of discoloration.  $\Delta E$  values of groups were found for TC ( $\Delta E_{TC} = 2.99 \pm 0.66$ ), VE ( $\Delta E_{VE} = 0.50 \pm 0.21$ ), and SH ( $\Delta E_{SH} = 1.07 \pm 0.24$ ).  $\Delta E_{SH}$  value was found to be significantly higher than  $\Delta E_{VE}$  ( $p < 0.05$ ).

**Table 1:** Shows statical difference within groups

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
$\Delta E$	Between Groups	50.77	2	25.38	137.93	.000
	Within Groups	7.73	42	0.18		
	Total	58.50	44			

**Table 2:** Shows statical values of discoloration of the three groups after thermocycling.

$\Delta E$		n	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum	Maximum
						Lower Bound	Upper Bound		
$\Delta E$	$\Delta E_{TC}$	15	2.99	0.66	0.17	2.61	3.36	2.42	4.61
	$\Delta E_{VE}$	15	0.50	0.21	0.05	0.39	0.62	0.30	1.10
	$\Delta E_{SH}$	15	1.07	0.24	0.06	0.93	1.20	0.40	1.32
	Total	45	1.52	1.15	0.17	1.17	1.87	0.30	4.61

### IV. Discussion

Thermocycling aging causes deterioration in the physical and chemical structure of composite-containing resin materials. While the deterioration of the physical structure causes wear and fatigue in the material; As a result of the deterioration of the chemical structure, enzymatic, hydrolytic, acidic and heat-related deterioration and dissolution in the material structure are observed. [34] In composite resin samples immersed in water for aging, it is observed that both the hydrolytic degradation of the filler particles and the reduction of the radical activity of functional monomers after the absorption of the organic resin matrix. [34], [35] A significant correlation between water absorption and  $\Delta E$  has been demonstrated in previous studies. [36]

A noticeable color change was found in TC after aging with thermocycling this study. Color change may be related to the water absorption amount of the material, in other words, the hydrophilic or hydrophobic structure of the resin matrix. [37] The hydrophilic property of the material and the amount of water absorption

affect the color change. If the resin matrix of material has hydrophilic properties, the water absorption level is high and it is open to color by absorbing not only water but also other colorant liquids.[38] For this reason, discoloration is expected to be low in materials with hydrophobic resin matrix properties.[39] Distilled water used in the thermal cycle does not contain any dyes. This situation is defined in the literature as an internal coloration that occurs due to water absorption of color change in composite resins.[40] Keeping composite resin materials in a wet or humid environment causes water absorption.[41]–[43] Water absorption also causes dissolution of composite resins and deterioration of mechanical properties.[42] Excessive water absorption has been reported to fluidize composite-containing resin materials, cause hydrolysis of the silane agent with the formation of microcracks, and expand the resin structure and shorten the life of the material. As a result of all this, the susceptibility of the material to color increases[44]–[46]The noticeable color change was seen in TC material can be said to depend on the water absorption amount of the organic matrix of the material. Because, after aging, the polymer structure softens by the absorption of water in the resin matrix part of the material.[47] To prevent this situation, the amount of conversion of the monomer in the material to the polymer can be increased, and the chemical properties of the material can be improved.[48] Properties related to the resin structure such as photoinitiators, activators, inhibitors and oxidation of unreacted monomers have also been reported to be effective in coloring composite resins.[40], [49]

Various resin matrixes used in composite materials have been shown to have important effects on coloration. [38], [49]–[51] Researchers have shown that the water absorption potential of the BisGMA monomer is a higher than the UDMA, TEGDMA and BisEMA monomers. [52]–[54] Comparing BisGMA and UDMA monomers, UDMA monomer has been found to have higher resistance to coloration, which has been linked to lower water absorption and water-solubility properties of the UDMA monomer. It has been shown to have a more hydrophobic structure than the BisGMA monomer. [45] The more coloration of the BisGMA monomer than other monomers has been linked to the fact that the hydroxyl (OH-) groups present in the monomer are more prone to water absorption. Llena et al. reported that the resin materials are more colored because the BisGMA based organic matrix is more hydrophilic.[55]

TC material showed more color change than CAD/ CAM block materials. Organic resin matrix constitutes 21% by weight of TC material, and monomers in this organic resin matrix are BisGMA, BisEMA and UDMA.[56]Barutçigil et al., examined color stability of different bulk composite resins. They found that bulk-fill composite resins containing BisGMA and TEGDMA monomers were more colored.[40] They explained that this is because BisGMA and TEGDMA monomers cause more water absorption when combined. These findings are the same as the findings in this study.

Although the organic matrix is a decisive factor in the coloring of the composite resin, the filler particle size, type and ratio are other factors that affect the color stability of the composite resin.[40], [57]–[59] During the polishing process, coloration occurs due to the roughness that occurs on the surface. [16], [59], [60] During finishing and polishing operations, a smaller size particle will be removed from the surface of a composite resin material with a small filler particle size.[61], [62] Accordingly, smaller particle size means less surface roughness and less water retention.[49] While the monomer structure absorbs water into the material, the filler particles absorb water on the surface of the material.[46] The filling particle sizes of TC material used in the study ranged between 40-160 nm.[63] Another factor responsible for the color change of the material is the depth of polymerization. Because the maximum polymerization of a material increases the conversion rate of the monomer to the polymer and hence the number of monomer decreases. This prevents the colorants from adhering to the material and coloration of the composite resin by reducing the surface roughness.[39], [49] As the amount of monomer release increases, this causes more monomer dissolution along with water absorption, which makes the material more susceptible to color by disrupting its mechanical properties.[64] Some studies reported that a second cure application for polymerization provided a significant increase in conversion rate, microhardness, surface roughness, flexural strength and finally color stability of the material.[65] Figueiredo said that when heat treatment is applied as an additional polymerization to composite resins, its physical properties increase, including color stability. [66] The stress at the interface of the resin matrix and filler particles is responsible for the deterioration of the substrate layers of the material. This makes it easier to form microcracks and voids in the material. If additional polymerization is applied along with the heat treatment, the stress or tension on the interface of the resin matrix and filler particles will be relieved by decreasing.[67] Expresses heat, pressure, and vacuum as contributing factors to polymerization efficiency.[68] Although additional techniques have been applied, monomer remains in composite resin materials. To prevent this, hybrid ceramics used with CAD/CAM systems (e.g. VE, SH) were produced.

The color stability of bulk-fill composite resin after aging with thermocycling was found lower than the color stability of CAD/CAM hybrid ceramic materials in this study. Acar et al. reported that hybrid ceramic materials showed higher color stability than composite resins. [69] The findings of this study are similar. Acar et al. Stated that excess coloration in composite resins was related to the monomer structure and that the color of

VE material was less due to less BisGMA in it.[69] In this study, the reason for the VE material to show less color change than all other materials may be due to the BisGMA ratio and different inorganic particles in it.

CAD-CAM hybrid ceramic materials have been polymerized at high temperature and pressure and presented for use.[70] When additional polymerization applied to the materials, the monomer-polymer conversion rate increases, the polymerization efficiency improves in the same way, and the amount of monomer reduced. Therefore, the processing method used for the production of the CAD/CAM blocks and the polymerization of the block improves the coloration resistance of the material.[27], [52]The reason for the bulk-fill resin to show more coloration than CAD/CAM blocks may be the lack of polymerization. Because additionally used polymerization methods, stress matrix and filler interface stresses are reduced. Due to the reduction of these stresses, microcrack formation that causes degradation in the lower surface is prevented. This prevents coloration by not allowing easy penetration of dyestuffs into the materials.[44], [67], [71]

Studies have demonstrated that the polymer structure softens as the result of the hybrid ceramic blocks absorbing water into the resin matrix. [47], [72] Both SH and VE have hydrophobic UDMA and hydrophilic TEGDMA monomers in the resin matrices.[73] Since the manufacturer companies do not share information about the monomer ratios for both materials. Less coloration of the VE block may be that the TEGDMA monomer ratio is lower than SH. It was reported that the TEGDMA monomer, which has a hydrophilic structure, shows more water absorption than UDMA, allowing dyes to enter the structure of the resin matrix.[74] Based on these results, we think that coloration originates from TEGDMA, a hydrophilic monomer.

The inorganic filler content of the VE material consists of 86% by weight of 14% polymer network diffused into the feldspathic ceramic matrix and is called the manufacturer's material as "hybrid ceramic". [75]; The inorganic filler content of SH material is 61% by weight and contains silica, silicate, zirconium silicate and micro-ceramic fillers.[76] In a study by Koizumi et al. included the SH material in Ferracane's composite classification, which is because the material was around 39% of the polymer network of the material. It was stated that the material was named as "hybrid composite".[26] Based on these explanations, we are linking the greater coloration of SH compared to VE to the less inorganic filler content and, accordingly, to the higher polymer composite structure.

The average size and shape of the fillers in the composite resin structure affect roughness and polish.[77] SH material has a large particle size of varying and these particles are not evenly distributed in the resin matrix. These particles, which are not evenly distributed, create more surface roughness in the SH material.[78] In addition, Koizumi et al. found high abrasion and surface roughness in their studies on the wear resistance of SH material. They reported that this was due to the irregular and large inorganic particle content in the structure of the material.[26] However, the VE material has a smaller filler particle size and the ceramic resin material is in an interlocking network.[69], [79], [80] Due to this interlocked web structure, less surface roughness is expected in the material. SH material contains zirconium dioxide as an inorganic filler particle. Hydrolysis occurs in the silane interface due to water absorption in both composite and hybrid ceramic materials containing zirconium dioxide.[81] VE material contains polymer-infiltrated ceramic network rich in aluminium as the inorganic particle, and this ceramic mesh forms a shape intertwined with the microstructure of the material.[75] This may explain the resistance of the resin-ceramic bond to hydrolytic dissolution. This resulting structure may be the reason for less coloring of the VE material. Because the zirconium dioxide particles in the content of SH create hydrolysis at the binder interface with water absorption and it is open to coloration with more water absorption in the material.[81] However, due to the interlocking network structure in the VE material, a material more resistant to water absorption emerges.[69], [79], [80] Therefore, the fact that SH material is colored more than VE material can be attributed to these reasons.

VE ( $\Delta E_{VE}=0.5$ ) material was found as the least colored material. SH( $\Delta E_{SH}=1.07$ ) is the second least colored material. The material with the most color change was determined as TC bulk-fill ( $\Delta E_{TC}=2.9$ ). A thermal cycling corresponding to a six months period was applied. However, in the mouth environment, restorative materials are exposed to various chewing forces, different contents and periods of food/drink, except for thermal changes. In addition, the person's tooth brushing habits and saliva buffering capacity should be taken into consideration. In this regard, the method we preferred to examine the color stability of CAD/CAM hybrid ceramic blocks and bulk-fill composite resins within the boundaries of the study was not able to simulate in vivo conditions exactly. For this reason, more information is needed about the color stability of the materials by adding other factors reflecting in vivo conditions.

## V. Conclusion

All of the restoration materials examined have clinically acceptable color stability. Color stability of CAD/ CAM prefabricated blocks is higher than bulk-fill resin composite. Due to their structural content, these CAD/CAM blocks also show different color changes.

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