

Evaluation of Color Stability of CAD/CAM And Bulk-Fill Resin Materials

CAD/CAM ve Bulk-Fill Rezin Materyallerin Renk Stabilitelerinin Değerlendirilmesi

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ABSTRACT

Introduction: Aim of this study was to evaluate color stability of two different types of bulk-fill composite resin and CAD/CAM hybrid ceramic materials after thermal ageing.

Methods: 12 samples from Tetric-N-Ceram Bulk-Fill and Estelite Bulk-Fill Flow with 4-mm thickness 10-mm diameter were prepared in a teflon mould. Prepared samples were polished with discs from coarse-to-fine(10sec). 12 samples from Vita-Enamic and Shofu-Block-HC were prepared with a microtome device. CAD/CAM blocks were polished with silicone abrasive papers. CAD/CAM samples were standardised in 4.0±0.1mm thickness with digital micrometer. Initial color measurements were taken with a spectrophotometer according to CIE-L*a*b*color system. All samples were put in a thermal cycle device, aged with 10000 cycles. Final color measurements were recorded after the thermal cycle procedure. The color change(ΔE) was calculated. To state the relationship between color change and clinical environment, data were transformed to National-Bureau of Standards(NBS). For statistical analysis Mann-Whitney-U Test with Bonferroni Correction were used with 0.008 degree of significance.

Results: According to the statistical evaluation, Vita-Enamic CAD/CAM hybrid ceramic material showed the highest color-stability when compared with other materials. Tetric-N-Ceram Bulk-fill composite resin was evaluated the lowest color-stability material(p<0.008).

Discussion and Conclusion: In a matter of color-stability based materials, ceramic content, size and ratio of inorganic filler particles, structure of organic resin matrix could be concluded that determinative.

Keywords: CAD/CAM, bulk-fill, hybrid ceramic, color stability, thermal cycle

ÖZ

Giriş ve Amaç: Bu çalışmanın amacı; iki farklı bulk-fill kompozit rezin ve CAD/CAM hibrit seramik materyallerinin termal yaşlandırma sonrası renk stabilitelerinin değerlendirilmesidir.

Yöntem ve Gereçler: Tetric N Ceram Bulk Fill ve Estelite Bulk-Fill Flow materyallerinden 4 mm kalınlığında ve 10 mm çapında teflon kalıpla 12 adet örnek hazırlandı. Hazırlanan örnekler en kalından en inceye disklerle cilalandı(10sn). Vita Enamic ve Shofu Block HC materyallerinden hassas-kesme cihazıyla 12 adet örnek kesildi. CAD/CAM örnekleri abraziv silikon karbid kâğıtlarla cilalandı. CAD/CAM örneklerinin kalınlığı 4.0±0.1 mm kalınlıkta olacak şekilde dijital bir mikrometreyle ölçüldü. Tüm materyallerin başlangıç renk ölçümleri CIE L*a*b* renk sistemi kullanılarak spektrofotometreyle ölçüldü. Tüm örnekler termal sıklüs cihazında 10000 sıklüsle yaşlandırıldı. Termal sıklüs işlemi sonrasında son renk ölçümleri kaydedildi. Renk değişim miktarı(ΔE) hesaplandı. Klinik ortam ile renk değişim miktarı arasındaki ilişkiyi belirlemek için, veriler Ulusal Standartlar Bürosu(National-Bureau of Standards-NBS) sistemine göre düzenlendi. Verilerin istatistiksel analizinde; Bonferroni düzeltmeli Mann-Whitney-U test kullanıldı, anlamlılık derecesi 0.008 olarak kabul edildi.

Bulgular: İstatistiksel değerlendirmeye göre; Vita Enamic CAD/CAM hibrit seramik materyali diğer materyaller ile karşılaştırıldığında en az renk değişimi göstererek renk stabilitesi en yüksek materyal olarak belirlendi. En çok renklenme gösteren ve renk stabilitesi en düşük materyal ise Tetric N Ceram bulk-fill materyali olarak belirlendi(p<0.008).

Tartışma ve Sonuç: Rezin içerikli materyallerin renk stabilitesinde seramik içeriği, inorganik doldurucu partiküllerinin oranı ve boyutu ile organik rezin matris yapısının belirleyici olduğu söylenebilir.

Anahtar Kelimeler: CAD/CAM, bulk-fill, hibrit seramik, renk stabilitesi, termal sıklüs

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INTRODUCTION

Modern dentistry is constantly evolving, and together with the increasing demand of patients' esthetic expectations, esthetic dental practice is becoming more challenging every day. For this reason, the color stability of restorative materials which is an important esthetic property of esthetic materials plays a decisive role in the success of restorations.¹

It has been accepted that conventional composite resins should be placed in increments with a thickness of 2 mm to provide adequate polymerization with an effective light transmission.² However, the incremental technique has some disadvantages. These disadvantages include increased patient chair-side time due to the individual irradiation of each layer, the possibility of air bubbles between the increments, and the risk of moisture contamination.²

Recently, bulk-fill composite resin materials have been introduced for clinicians to eliminate the disadvantages of conventional composite resin materials. It has been reported that this newly developed composite resin material can be placed in a single layer up to a thickness of 4-5 mm which makes them convenient in facilitating restorative procedures in deep and wide cavities and thus saving time for both the clinician and the patient. Besides, bulk-fill composite resin materials are more successful in terms of polymerization shrinkage, degree of conversion, and cavity adaptation compared with conventional composite resins.³

Researchers have attempted to increase the degree of conversion of the composite resin material by modifying the physical and mechanical properties and the content of the resin matrix. As a result of these improvements, the use of blocks with CAD/CAM technology is presented.

These blocks are industrially polymerized and have improved physical properties under standardized parameters at high temperature and high pressure to provide optimal physical and mechanical properties.^{4,5}

CAD/CAM hybrid ceramics are presented as one of the current materials that hold both positive properties of ceramic and composite materials presented for clinical applications. These hybrid materials have the advantage of being fabricated and repaired more easily than restorations made from CAD/CAM ceramic materials.⁵

As restorative materials are prone to intrinsic discoloration depending on temperature changes and water absorption, successful restorations should not only have high resistance and durability but also esthetic features.^{6,7}

Therefore, the purpose of this study was to evaluate and compare the color change of two current CAD/CAM hybrid ceramics with different inorganic content and bulk-fill composite resin materials before and after the thermal aging procedure. The null hypothesis is that the color change values of both CAD/CAM hybrid ceramic and bulk-fill composite resin materials after long-term thermal aging will be clinically acceptable.

MATERIALS AND METHODS

In this study, a high viscosity (Tetric N Ceram Bulk Fill, Ivoclar Vivadent, AG, Lichtenstein), and a low viscosity (Estelite Bulk Fill Flow, Tokuyama Dental Corporation, Tokyo, Japan), bulk-fill composite resin material; and two CAD/CAM hybrid ceramic (Vita Enamic, Vita Zahnfabrik, Bad Säckingen, Germany and Shofu Block HC, Shofu Inc., Kyoto, Japan) restorative materials were used (Table 1).

Table 1. Materials used in the study

CLASSIFICATION	MATERIALS	COLOR	LOT NUMBER	MANUFACTURER	COMPOSITION
BULK-FILL MATERIALS	Bulk-Fill Composite Resins				
	ESTELITE BULK FILL FLOW (EBF)	A2	04E06	Tokuyama Dental Corporation, Tokyo, Japan	Bis-GMA, TEGDMA, Bis-MPEPP, Mequinol, Dibutyl hydroxyl toluene, UV absorber
	TETRIC N CERAM BULK FILL (TNCB)	IVB	V16483	Ivoclar Vivadent, AG, Lichtenstein	Bis-GMA, UDMA, Ba-Al-Si glass prepolymer filler (monomer, glass filler, ytterbium fluoride), spherical mixed oxide.
CAD/CAM MATERIALS	Resin Matrix Ceramic Blocks				
	VITA ENAMIC (VE)	2M2-HT	41951	Vita Zahnfabrik, Bad Säckingen, Germany	%86 feldspar ceramic, %14 wt polyester, UDMA, TEGDMA
	SHOFU BLOCK HC (SB)	A2-HT	0916055	Shofu Inc., Kyoto, Japan	UDMA, TEGDMA, %61 silica powder, microfumed silica, zirconium silicate

Preparation of Bulk-fill Composite Resin Samples

A Teflon mold (10 mm in diameter and 4 mm in height) (Figure 1) was used to prepare bulk-fill composite resin specimens. For each bulk-fill composite resin material group, twelve samples were prepared (n= 12). The molds were filled with uncured bulk-fill materials, covered with mylar strips to prevent the formation of an oxygen inhibition layer, and finally polymerized with a LED light-curing unit (Elipar S™ 10, 3M ESPE, Seefeld, Germany) from the top surface for 20 s according to the manufacturer's instructions. During polymerization, the curing unit was directly applied onto the sample surface, with the tip positioned perpendicular to the surface with glass to obtain a plane surface. After polymerization, the top surfaces of all samples were polished with polishing discs from coarse to fine (Sof-Lex, 3M ESPE; St. Paul, MN, USA) for 10 s each, using a low-speed handpiece. After each polishing step, the polishing disc was discarded, and the samples were thoroughly rinsed with water for 10 s and air-dried for 5 s. Then, all samples were kept in distilled water at 37°C for 24 h in an incubator (Star Dental 320S; İstanbul, Turkey).

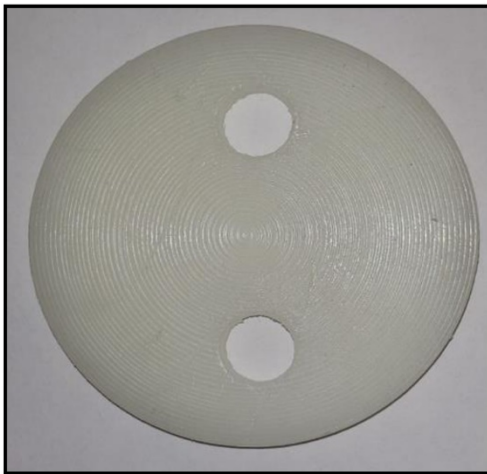


Figure 1.

Preparation of CAD/CAM Block Samples

CAD/CAM hybrid-ceramic blocks with dimensions of 14x12x18 mm were used (Table 1). Due to their fabrication characteristics, hybrid ceramic blocks do not need to be sintered. Each CAD/CAM block was sectioned into 4.3±0.2 mm thickness underwater with low-speed sectioning saw (IsoMet 1000, Buehler; Illinois, ITW, USA). Both sides of the CAD/CAM samples were polished using abrasive silicon carbide papers from coarse to fine (600, 800, 1000, 1200 grit) under water-cooling for 10 s each.⁸ After the polishing procedure was completed, CAD/CAM specimen thickness was confirmed to be 4.0±0.1 mm with a digital micrometer. All samples were kept in distilled water at 37°C for 24 h in an incubator (Star Dental 320S; İstanbul, Turkey).

Color Change Measurements

Initial L*a*b* values were evaluated using a spectrophotometer (Vita EasyShade® Advance 4.0; Vita Zahnfabrik, Bad Säckingen, Germany). Three measurements were conducted at the center of each specimen on a white background, and the mean values were calculated. Color measurements were recorded using the CIE L*a*b* color system.⁹ After initial color measurements, the specimens were thermocycled for 10.000 cycles between 5°C and 55°C with a dwell time of 30 s (Moddental, MTE100, Ankara, Turkey). Then final color measurements were recorded. Color change values (ΔE) were computed from the mean ΔL, Δa, and Δb values for each sample with the subsequent formula:⁹

$$\Delta E = [(L2^* - L1^*)^2 + (a2^* - a1^*)^2 + (b2^* - b1^*)^2]^{1/2}$$

According to this formula, ΔL, Δa, and Δb are the variations in the L, a, and b values, respectively, at baseline and after thermocycling (Table 2). To determine the correlation between the amount of color alteration recorded on a spectrophotometer and the clinical environment, data were converted to the National Bureau of Standards (NBS) system.¹⁰ The formula ΔEx0.92 was used to determine the correlation between the amount of color change and the clinical environment¹⁰.

Table 2- Evaluation of L*, a*, b* findings between and in groups

		TNCB	EBF	VE	SB	1p
		Mean±SD	Mean±SD	Mean±SD	Mean±SD	
L	Before TC	69,73±1,35	67,13±0,58	76,70±0,2	76,89±0,26	0,000*
	After TC	67,17±0,97	66,24±1,38	76,54±0,32	75,9±0,2	0,000*
	2p	0,002*	0,021*	0,167	0,002*	
a	Before TC	-2,97±0,32	-2,43±0,21	2,65±0,08	-0,72±0,1	0,000*
	After TC	-2,67±0,19	-2,08±0,39	2,61±0,1	-0,75±0,12	0,000*
	2p	0,020*	0,016*	0,025*	0,046*	
b	Before TC	6,8±1,08	19,9±1,35	21,78±0,19	16,47±0,33	0,000*
	After TC	5,22±0,9	18,7±1,1	21,34±0,25	16,16±0,3	0,000*
	2p	0,002*	0,002*	0,002*	0,002*	

¹Kruskal Wallis Test and ²Wilcoxon Sign Test * p<0.05 TC: Termal Cycle

Statistical Analysis

Statistical analysis was obtained using the IBM SPSS Statistics 22 (IBM SPSS, Turkey) program. The normal distribution of the parameters was evaluated by the Kolmogorov-Smirnov and Shfapiro Wilk Tests. It was found that the parameters did not show normal distribution. The Kruskal-Wallis test was used for the comparison between groups without normal distribution in the comparison of the quantitative data. The Mann-Whitney U test with Bonferroni Correction was used as the post hoc test. The Wilcoxon Sign Test was used for in-group evaluations. The overall significance in the study was $p < 0.05$, and the paired comparisons were assessed with a significance level of 0.008. The Mann-Whitney U test with Bonferroni Correction was used with 0.008 degrees of significance.

RESULTS

The materials used in this study were named Tetric N Ceram Bulk Fill "TNCB", Estelite Bulk Fill Flow "EBF", Vita Enamic "VE", and Shofu Block HC "SB".

There was a statistically significant difference between the groups in terms of ΔE values ($p < 0.05$) (Table 3, Figure 2). The ΔE value of TNCB was significantly higher than that of EBF, VE and SB. The ΔE value of EBF was significantly higher than that of VE and SB, and the ΔE value of SB was calculated to be significantly higher than that of VE ($p < 0.008$).

The equivalent of ΔE to NBS for TNCB was 2.852, EBF was 1.7204, VE was 0.46, and SB was 0.966.

Table 3. Evaluation of ΔE results between tested groups

GROUPS	ΔE
	Mean \pm SD/NBS ($\Delta E \times 0.92$)
TNCB	3,10 \pm 0,70 (2,9)
EBF	1,87 \pm 0,82 (1,7)
VE	0,50 \pm 0,22 (0,5)
SB	1,05 \pm 0,26 (1,0)
P	0,000*

Kruskal Wallis Test * $p < 0.05$

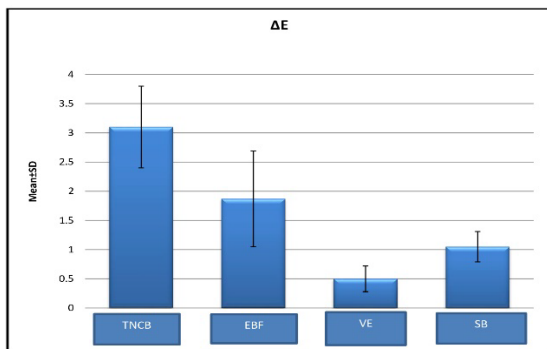


Figure 2.

DISCUSSION

In the color change experiments, ΔE gives the color change values on the tooth surface or dental materials before and after different procedures.¹¹ ΔE value between 1 and 3.3 can be recognized by an experienced human eye and is defined as 'clinically acceptable' color change.¹² In this study, ΔE values of the experimental groups according to NBS¹⁰ were TNCB:2.9> EBF:1.7> SB:1.0> VE:0.5. All of the materials exhibited clinically acceptable color change values after the long-term thermal aging procedure. Thus, the null hypothesis was accepted.

TNCB is packable, and high-viscosity bulk-fill composite, while EBF is flowable, and a low-viscosity bulk-fill composite. The manipulations and the contents of materials are different from each other. Additionally, the contents of the inorganic filler in the two hybrid ceramic blocks used in this study are different from each other. The inorganic filler content of the VE consists of a 14% polymer network diffused into an 86% feldspar ceramic matrix (weight).¹³ While the inorganic filler content of SB is 61% (weight) and contains silica, silicate, zirconium silicate, and micro ceramic fillers.¹⁴

In laboratory studies when evaluating dental materials, the use of the thermocycling test method is considered to be an important method for simulating *in vivo* conditions.¹⁵ Thermocycling is the process of subjecting samples to temperature changes that simulate intraoral conditions.¹⁶ Evaluation of the color stability of the materials after thermocycling could provide valuable information regarding the performance of the restorations. In *in vitro* studies, it has been reported that a thermocycling with 10.000 cycles corresponds to a one-year thermal aging.¹⁷ Hence, to evaluate and compare the long-term color change of current materials under 10.000 thermal cycles was preferred in this study.

A noticeable color change was observed in TNCB (2.9 to NBS) and EBF (1.7 to NBS) after the thermocycling aging procedure. It was emphasized that the hydrophilic nature of the resin matrix and the amount of water absorption are effective in color change.⁵ It can be concluded that the perceptible color change was seen in TNCB (2.9) and EBF (1.7) depends on the amount of water absorption of the organic matrix of these materials. It was shown that the water absorption potential of Bis-GMA is higher than that of UDMA, TEGDMA, and Bis-EMA.^{4,18} UDMA has a more hydrophobic structure than Bis-GMA.¹⁹ Also, Bis-GMA contains hydroxyl (OH) groups, which are more susceptible to water absorption. TNCB showed more color change than EBF. TNCB is composed of 21% organic resin matrices, which are Bis-GMA, Bis-EMA, and UDMA.²⁰ EBF contains Bis-GMA, Bis-MPEPP, and TEGDMA.²¹ The reason for the higher color change in TNCB than EBF can be explained as follows. The organic matrix percentage of EBF by weight

may be lower than TNCB. Hence, it was thought that the discoloration may be related to the organic matrix ratios. Although Bis-GMA is present in both materials; EBF has a different content, there is also a Bis-MPEPP monomer in it when compared with TNCB. Bis-MPEPP does not contain hydroxyl (OH⁻) groups that contribute to water absorption. Furthermore, the viscosity of Bis-MPEPP is lower than that of Bis-GMA.²² Therefore, the presence of Bis-MPEPP in EBF may already reduce the viscosity of the material; then, the proportion of TEGDMA in the structure may be kept low, which may explain the lower discoloration.

In this study, the thermocycling procedure resulted in a higher color change in bulk-fill composite resins than CAD/CAM hybrid ceramic materials. Acar *et al.*²³ found that hybrid ceramic materials exhibit better color stability than composite resins. The findings of the present study are in parallel with the findings of Acar *et al.* These researchers reported that discoloration of composite resins is related to monomer structure. The authors advocated that VE has less discoloration due to less Bis-GMA in the structure.^{23,24}

Although the organic matrix is a determinant constituent for the coloring of the composite resin, the size, type, and ratio of the filler particle are other constituents that affect the color stability of the composite resin.⁶ Smaller particle size means less surface roughness and less water retention.²¹ While the monomer structure absorbs water into the material, the filler particles promote water retention on the surface of the material.²⁵ The filler particle size of TNCB ranges from 40 to 3000 nm.²⁰ EBF contains supra-nano spherical filler particles containing SiO₂ and ZrO₂ at 200 nm size.²¹ Based on these data, it can be concluded that EBF has a uniform size and rounded filler particle type. These particles are smaller in size than TNCB material. As a result, EBF shows less discoloration due to the inorganic particles. In a study conducted by Shamszadeh *et al.* It was observed that TNCB exhibited discoloration in 1-month water storage,¹¹ that result also supports the data of this study. According to their study; it was explained that discoloration was due to the prepolymer shrinkage stress relievers contained in the material, to various sized inorganic fillers, and the light-sensitive system originating from the Ivocerin® molecule.¹²

It has been reported that SB material has a large and various particle size and that these particles are not evenly distributed in the resin matrix.²⁵ VE material, conversely, has a smaller filler particle size, and the ceramic resin material is in the form of an infiltrated and interlocked polymer network.²⁵ Due to this interlocked network structure, less surface roughness is expected in the material. SB material contains zirconium dioxide as an inorganic filler particle. It has been reported that hydrolysis occurs in the silane coupling agent interface due to water absorption in both composite and hybrid

ceramic materials containing zirconium dioxide.²⁶ This could explain why SB showed more discoloration than VE.

CAD/CAM hybrid ceramic materials are presented for clinical use by polymerization at high temperature and high pressure.⁵ When additional polymerization is applied to the materials, the monomer-to-polymer conversion ratio is increased, the polymerization efficiency is improved in the same manner, and the amount of residual monomer is decreased. Therefore, the fabricating method used for the production of CAD/CAM block polymerization improves the color resistance of the material.⁴ On the contrary, bulk-fill composites are only polymerized by light. Although additional techniques are applied, residual monomers can remain. Therefore; CAD/CAM hybrid ceramic materials showed better color stability than bulk-fill composite resins due to the polymerization that occurs under high temperature and high pressure.

CONCLUSIONS

The limitations of this study were as follows:

This study was performed under *in vitro* conditions which does not exactly mimic the oral environment. In this study, the intrinsic color change of tested materials was evaluated only after the thermal aging procedure. The effect of saliva and tooth brushing, chewing procedures, surface roughness, degree of conversion, and microhardness measurements could be added as investigational systems. Different colorant liquids like coffee, tea, wine could be tested to evaluate the discoloration. As a positive control group, at least one conventional composite resin material with 4-mm thickness could be used to compare the discoloration differences. Further studies should be performed including the above limitations to assess the long-term color stability of bulk-fill and CAD-CAM materials. It can be concluded that:

- 1- While the monomer ratios of Bis-GMA and TEGDMA had a negative effect on the color change of restorative materials, Bis-MPEPP and UDMA had a positive effect.
 - 2- Ceramic inorganic particles can be effective in the color stability of restorative material.
- Therefore, the type of ceramic used as an inorganic particle should be considered while planning a restoration in clinical practice.
- 3- Restorative materials prepared under additional polymerization techniques such as high temperature and pressure will be more successful in terms of color stability. The success in the color stability will be achieved with the maximum depth of cure, which decreases the amount of residual monomer and water sorption.

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