



Influence of Current Adhesive Systems on Color Stability of Resin Composite

Günümüz Adeziv Sistemlerinin Rezin Kompozitin Renk Stabilitesi Üzerindeki Etkisi

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ABSTRACT

Objective: The adhesive systems which have an important place in modern dentistry can affect the color properties of final resin composite restorations. Therefore, the aim of the present study is to evaluate the influence of current adhesive systems on color, color stability and translucency of resin composites.

Methods: Fifty disc-shaped microhybrid resin composite specimens were prepared. Resin composite discs were divided into five groups randomly according to adhesive system including a control group (n=10) (Clearfil Protect Bond, Adper EasyOne, Clearfil QuickBond and Tokuyama BondForce). Color and translucency measurements were performed with a clinical spectrophotometer at baseline, immediately after adhesive system application and after immersion in water for 30 days. For statistical analysis, paired-samples t-test, one-way analysis of variance (ANOVA) and Tukey's post-hoc comparisons were used (p<0.05).

Results: The adhesive application significantly changed the color (E*) of the resin composite discs regardless of the adhesive brand. Storage of the resin composite discs in water for 30 days caused significant color changes in the control group. Significant differences were found in color values of the Adper EasyOne and Clearfil Protect Bond groups after aging. All adhesive groups showed significant color changes in Δb values in comparison with the control group, indicating increasing bluing. The translucency of composite discs applied with BondForce and Adper EasyOne adhesives significantly decreased after aging.

ÖZ

Amaç: Modern diş hekimliğinde önemli bir yere sahip olan adeziv sistemler, nihai rezin kompozit restorasyonların renk özelliklerini etkileyebilmektedir. Bu nedenle, bu çalışmanın amacı, günümüz adeziv sistemlerin rezin kompozit materyalinin renk, renk stabilitesi ve translüensliği üzerindeki etkisini değerlendirmektir.

Yöntemler: Elli adet disk şekilli mikrohibrit rezin kompozit örneği hazırlandı. Rezin kompozit diskler, bir kontrol grubu olmak üzere rastgele olarak adeziv sisteme göre beş gruba ayrıldı (n=10) (Clearfil Protect Bond, Adper EasyOne, Clearfil QuickBond and Tokuyama BondForce). Renk ve translüenslik ölçümleri, başlangıçta, adeziv sistem uygulamasından hemen sonra ve 30 gün boyunca suya daldırıldıktan sonra klinik spektrofotometre ile gerçekleştirildi. İstatistiksel analiz için eşleştirilmiş örneklem t-testi, tek yönlü varyans analizi (ANOVA) ve Tukey'in post-hoc karşılaştırmaları kullanıldı (p<0,05).

Bulgular: Adeziv uygulaması, adeziv markasından bağımsız olarak rezin kompozit disklerin rengini (E*) anlamlı olarak değiştirdi. Kontrol grubunda, rezin kompozit disklerin 30 gün suda bekletilmesi anlamlı renk değişimine neden oldu. Adper EasyOne ve Clearfil Protect Bond gruplarının yaşlandırma sonrası renk değerlerinde anlamlı farklılıklar bulundu. Tüm adeziv grupları, kontrol grubuyla karşılaştırıldığında Δb değerlerinde anlamlı renk değişiklikleri gösterdi, bu da artan mavileşmeyi gösterir. BondForce ve Adper EasyOne adezivleri uygulanan kompozit disklerin translüensliği, yaşlandırma sonrasında anlamlı olarak azaldı.

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Conclusion: The adhesive systems may influence the final color of the restoration, however the adhesive systems tested in the study caused clinically acceptable discoloration at baseline and after aging.

Keywords: Resin composite, color stability, adhesive system, translucency

Sonuç: Adeziv sistemler restorasyonun son rengini etkileyebilir, ancak çalışmada test edilen adeziv sistemleri başlangıçta ve yaşlanma sonrasında klinik olarak kabul edilebilir renk değişikliğine neden olmuştur.

Anahtar Sözcükler: Resin kompozit, renk stabilitesi, adeziv sistem, translüsentlik

Introduction

The appearance of tooth-colored restorations in the aesthetic area is affected by several factors including the color and translucency of restorative materials and the underlying tooth tissues (1). Color matching and color stability have an important role in the short- and long-term success of any aesthetic restoration in dentistry (2,3). Polymeric materials, such as resin composites, become discolored over time when exposed to the intraoral environment (4). It is stated that the reason for change in color of resin restorations is due to changes in chemical structure of the materials and extrinsic staining over time (5,6).

Resin composites are semi-transparent materials. Due to this, color of tissues under it affects color of final restorations. This occurs especially when resin composite is applied in thin layers. Prior to placing resin composite restorations, an adhesive system should be applied to enamel and dentin surfaces in order to provide effective bond strength for resin composite with dental hard tissues. Therefore, the color properties of the adhesive system used for bonding may influence the final color of the restoration. In general, adhesive systems are highly complex mixtures containing polymerization initiators, solvents, hydrophilic monomers and hydrophobic monomers. In addition to these complex chemical contents of adhesive systems, chemical contents of each adhesive brand differ significantly (7). Similar to resin composites, adhesive systems are not full-transparent materials. Polymerization initiators such as camphorquinone play a major role in colorization of adhesive systems both before and after polymerization (8).

The amount of hydrophilic monomers and solvents have been increased in the content of recent adhesive systems. In this way, these adhesives are better infiltrated into microporosities on enamel and dentin surface (9). The increasing solvent and hydrophilic monomer content increase water absorption of the current adhesive systems (10). There is an inverse proportion between water absorption and color stability of polymeric materials (11). Therefore, the water absorption problem of adhesive systems may influence the color properties of the final resin composite restoration. In previous studies, it was stated that the adhesive brand affected both color matching and color stability of final resin composite restoration (12-14). However, such studies are not yet available in the literature for current adhesive systems that are new to the market and differ in their chemical contents. Thus, the aim of the present study is to investigate the effects of newly introduced adhesive systems on the initial colors and color stability of resin composites. The null hypothesis of the present study is that the initial color and

color stability of resin composites are not affected by the adhesive system.

Methods

Study Design

Four adhesive systems (Adper EasyOne, 3M ESPE, St. Paul, MN, USA; Clearfil QuickBond, Kuraray Noritake Dental Inc., Tokyo, Japan; BondForce, Tokuyama Inc., Tokyo; Japan Clearfil Protect Bond, Kuraray Noritake Dental Inc., Tokyo, Japan) were tested. Details of the materials used in the study are shown in Table 1.

Specimen Preparation

Fifty disc-shaped specimens from a microhybrid resin composite (shade A2, Herculite Classic, Kerr Italia, Scafati, Italy) were prepared. Resin composite discs were divided into five groups randomly according to adhesive system (n=10). One group without application of adhesive system served as a control group. The elastic mold, which was used to prepare the resin composite discs, was 8 mm in diameter and 1.25 mm in depth. A transparent polyester strip was placed on a glass slide. The elastic mold was fixed on the transparent polyester strip by using a piece of double-faced adhesive band. The resin composite was condensed into the mold. The top of the elastic mold was covered with another transparent polyester strip and a second glass slide was placed slightly over the composite to achieve flat surface. Polymerization was done on top surface of resin composite from 5 different areas each for 20 s with a LED curing unit (Elipar S10, 3M Espe, St. Paul, MN, USA) with an intensity setting of 1,200 mW/cm². The curing light intensity was checked by a radiometer. After the resin composite discs were left in the dark for 24 h to complete the polymerization, both surfaces of the discs were polished with 600-, 800-, 1,200-1,500-grit silicon carbide papers under water cooling. For distinction of the bottom and top sides of specimens, little notches were made with a dental bur on the edge of the bottom side of the composite discs. Final thicknesses of resin composite disks were measured by a digital caliper (1.2±0.1). Resin composite discs were kept in black bottles until color measurements were done.

Color Analysis

A clinical spectrophotometer (VITA Easyshade V, VITA Zahnfabrik, Bad Säckingen, Germany) was used for the color measurement. All the measurements were made from the top sides of the discs. The display of the clinical spectrophotometer shows CIE $L^*a^*b^*$ color system. CIE $L^*a^*b^*$ color system states color space by L^* , a^* , and b^* coordinates. L^* symbolizes the

Table 1. Materials used in the study

Material	Chemical composition	Application technique
Clearfil Protect Bond (Kuraray Noritake Dental Inc., Tokyo, Japan) Primer #8H0073 Bond #8J0069	Primer: MDP, MDPB, HEMA, water, hydrophilic dimethacrylate Bond: MDP, Bis-GMA, HEMA, microfiller, surface-treated sodium fluoride	Apply and leave for 20 sec, air dry gently for 5 s; apply bond and light cure for 10 sec
Adper EasyOne (3M ESPE, St. Paul, MN, USA) #4747900	HEMA, Bis-GMA, methacrylated phosphoric esters, 1,6 hexanediol dimethacrylate, methacrylate, functionalized polyalkenoic acid, silica filler, ethanol, water, initiators, stabilizers	Apply adhesive to tooth surface for a total of 20 s; air-dry the adhesive for 5 s; light cure for 10 s
Clearfil QuickBond (Kuraray Noritake Dental Inc., Tokyo, Japan) #700042	HEMA, Bis-GMA, MDP, Hydrophilic amide monomers, Colloidal silica, Silane, Sodium fluoride, Ethanol, Water	Apply with a rubbing motion then no waiting time after applying; dry for 5 s; light cure for 5 s
BondForce (Tokuyama Inc., Tokyo, Japan) #144	Methacryloyloxyalkyl acid phosphate, HEMA, Bis-GMA, TEGDMA, camphorquinone, purified water, alcohol	Apply adhesive and agitate for 20 s; air-dry gently until there is no water movement, then dry with strong air for 5 s; light-curing for 10s
Herculite Classic (Kerr Italia S.r.L., Scafati, Italy) #5517381	Bis-GMA, TEGDMA, camphorquinone, amine, iron oxide pigments, aluminum borosilicate glass, colloidal silica (SiO ₂) 79% of filler content	2 mm application max, gently adapt the product and light -cure for 20 s

MDP: 10-methacryloyloxydecyl dihydrogen phosphate, MDPB: 12-methacryloyloxydodecylpyridinium bromide, HEMA: 2-hydroxyethyl methacrylate, Bis-GMA: Bisphenol-A glycidyl dimethacrylate, TEGDMA: Triethyleneglycol dimethacrylate

lightness or black/white character of the color. The coordinates a^* and b^* define the chromatic features of the color. The a^* coordinate symbolizes the red-green axis and b^* coordinate symbolizes the yellow-blue axis.

Baseline color measurements (T_0) were performed in a custom-made light box with D65 illumination (KES 123 Led Bulb 12 W, K2 LED Systems, İstanbul, Turkey). Resin composite discs were placed on a white tile. The instrument was calibrated according to the manufacturer's instructions. Three consecutive measurements were made for each specimen and mean L^* , a^* , and b^* values were calculated (15). Color values (E^*) were calculated with the following formula:

$$E^* = (L^{*2} + a^{*2} + b^{*2})^{1/2}$$

Translucency was evaluated with translucency parameter (TP). Measurements were performed in the light box under D65 illumination with the Easyshade Compact spectrophotometer. Specimens were placed on a white tile. TP was obtained by calculating the color difference between the specimen on the white background and the black background with the following formula (15,16):

$$TP = ((L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2)^{1/2}$$

Subscript B refers to the color coordinates on the black background and subscript W refers to those on the white background. If the material is absolutely opaque, TP value is zero; if the material is totally transparent, TP value is 100. Thus, higher values for the TP value represent greater translucency (16).

Adhesive System Application and Water-Storage Aging Procedures

After baseline color (E_0) and translucency (TP_0) measurements, for second measurements (T_1), adhesive systems application

procedures were performed at the bottom surface of each resin composite discs according to respective manufacturer's instruction for each group (Table 1). Then, color (E_1) and translucency (TP_1) measurements were re-performed as in the first measurement from top surface of the resin composite discs. After the second measurements, resin composite discs were kept in distilled water for 30-day in separate dark bottles according to their groups. Distilled water in the dark bottles was changed every week. After aging process, resin composite discs were dried and color (E_2) and transparency (TP_2) measurements were made again (T_2). Color change values (ΔE) of resin composite discs between baseline and immediately after adhesive system application (ΔE_{0-1}) and between baseline and after aging procedure (ΔE_{0-2}) were calculated with the following formula:

$$\Delta E_{ab^*} (L^* a^* b^*) = ((\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2)^{1/2}$$

Statistical Analysis

Comparisons of E^* , L^* , a^* , b^* , and TP values among different time intervals (T_0 , T_1 , T_2) for each adhesive system were performed using paired-samples T-test. The effects of adhesive system on ΔE_{1-0} and ΔE_{2-0} values were analyzed by using one-way analysis of variance (ANOVA). Tukey tests were used for post-hoc comparisons among adhesive systems. All tests were performed by using SPSS 16 package program. Statistical significance was set at $p=0.05$.

Results

Mean and standard deviation of E^* , L^* , a^* , b^* and TP (translucency) values of adhesive bonded resin composite discs in different adhesive system groups and control group before and after artificially aging were presented in Figure 1. Paired t-test showed that adhesive application significantly changed the color (E^*) of the resin composite discs regardless of adhesive brand.

Storage of the resin composite discs in water for 30 days caused a significant change in color of the discs in the control group. Significant differences were found in the color values of Adper EasyOne and Clearfil Protect Bond groups after aging. On the other hand, there was no significant difference in color values of the discs in Clearfil QuickBond and Tokuyama BondForce groups between after and before aging. The translucency of composite discs applied with BondForce and Adper EasyOne adhesives significantly decreased after aging (Figure 1).

The findings of color changes (ΔE , ΔL , Δa and Δb) according to the adhesive system before and after artificially aging were summarized in Table 2. The application of Adper EasyOne (2.16 ± 0.3) and Clearfil Protect Bond (-2.16 ± 0.3) to the resin

composite discs resulted in the highest initial color changes without significant differences. Color changes in Adper EasyOne and Clearfil Protect Bond groups after aging were the highest, however, there was no significant difference among all groups. All adhesive groups showed significant color changes in Δb values in comparison with the control group, indicating increasing bluing (Table 2).

Discussion

Since the resin composite material is semi-translucent, the final color of resin composite restorations is affected by the transparency and thickness of material and background color. The clinical success of aesthetic restorations depends on color

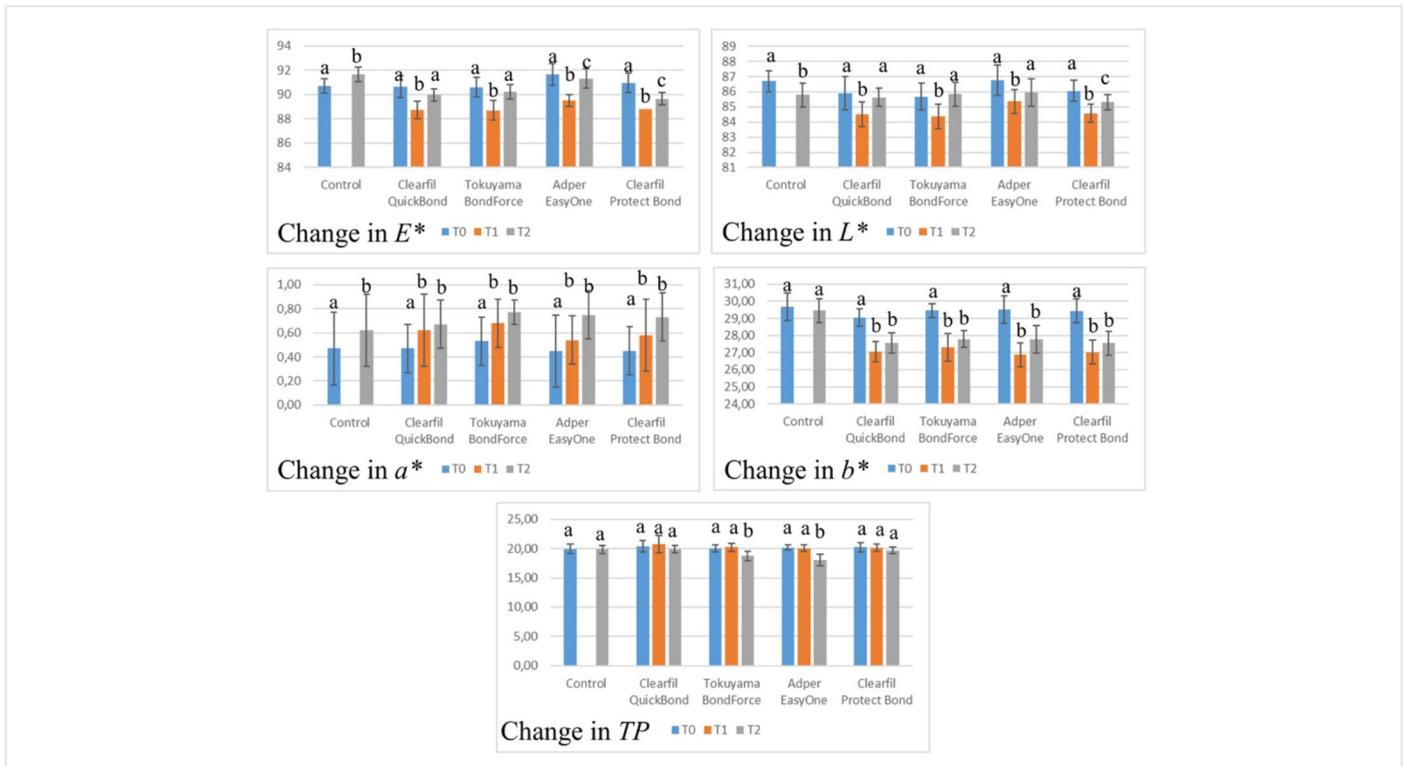


Figure 1. Bar graphs showing E*, L*, a*, b* and TP (translucency) values (means and standard deviations) of resin adhesive bonded resin composite discs according to adhesive brand and artificially aging. T₀: Time of color measurement before adhesive system application; T₁: Time of color measurement after adhesive system application; T₂: time of color measurement after artificial aging. Different superscripts on the bars of same adhesive brand indicate significant differences according to paired t-tests (p<0.05)

Table 2. ΔE , ΔL^* , Δa^* and Δb^* values (means and standard deviations) for all test groups

Adhesive system	Before artificial aging				After artificial aging			
	Color change							
	ΔE_{10}	ΔL_{10}	Δa_{10}	Δb_{10}	ΔE_{20}	ΔL_{20}	Δa_{20}	Δb_{20}
Control	-	-	-	-	-0.95 ± 0.8^a	-0.92 ± 0.7^a	0.72 ± 0.4^a	-0.24 ± 1.0^a
AEO	-2.16 ± 0.3^a	-1.41 ± 0.3^a	0.10 ± 0.1^a	-2.63 ± 0.3^{bc}	-1.34 ± 1.3^a	-0.83 ± 1.2^a	-0.74 ± 0.2^a	-1.76 ± 0.5^b
CQB	-1.93 ± 1.0^a	-1.38 ± 0.9^a	0.15 ± 0.1^a	-1.99 ± 0.5^{ac}	-0.71 ± 0.8^a	-0.26 ± 0.9^a	-1.04 ± 0.3^a	-1.47 ± 0.7^b
BF	-1.91 ± 0.6^a	-1.30 ± 0.5^a	0.15 ± 0.1^a	-2.14 ± 0.5^{ac}	-0.37 ± 0.6^a	0.16 ± 0.7^a	-1.03 ± 0.3^a	-1.67 ± 0.7^b
CPB	-2.16 ± 0.7^a	-1.41 ± 0.3^a	0.13 ± 0.1^a	-2.40 ± 0.5^{ac}	-1.30 ± 0.6^a	-0.75 ± 0.7^a	-0.84 ± 0.4^a	-1.88 ± 1.0^b

Different superscripts in the same column indicate significant differences according to Tukey test (p < 0.05). AEO: Adper EasyOne, CQB: Clearfil QuickBond, BF: BondForce, CPB: Clearfil protect bond

matching and color stability. As the adhesive system is a material used under resin composite material, it can be considered as a factor affecting the color of resin composite restorations. In particular, the possible influence of the color stability of hydrophilic adhesives on the color of the final resin composite restoration may be significant. Therefore, the effects of three single-bottle one-step self-etch adhesive systems and one two-bottle two-step self-etch adhesive system on the color of resin composite discs that were aged for 30-day by immersing in water and that were not aged were investigated in this study.

Under clinical conditions, there are dental tissues under resin composite and adhesive layer. If clinical conditions were tried to be established in this study, the effects of color and color stability of the adhesive system on the final color of the resin composite discs could not be revealed (12). Therefore, in this study, adhesive systems were applied to the bottom surfaces of composite discs, as in the study of Ritter et al. (13).

Application of adhesive systems on the bottom surface of the resin composite discs significantly changed the initial color values (E^*) of the resin composite discs in all groups. Initial color change (ΔE) values were between 1.91 and 2.16 units. After 30-day storage in water, there were significant differences in color matching in Adper EasyOne and Clearfil Protect Bond groups. The return of discoloration caused by other adhesives over time is a phenomenon that needs to be explained. Color change values (ΔE) of the resin composite discs changed between 1.47 and 1.88 units after aging. In the light of our findings, the null hypothesis, which is that the initial color and color stability of resin composites are not affected by the adhesive system, is rejected.

Considering the relationship between color change and clinical significance, there is no clear consensus in the literature on what extent of color change is visually detectable or acceptable. When the single-step self-etch and the total-etch adhesive systems are evaluated, even in the early stages, they exhibit significant color changes in water, so adhesives may be possible factors that can affect the color stability of resin composite restorations (12). However, in previous studies, $\Delta E = 1-1.2$ and $\Delta E = 2.7-3.7$ were specified as perceptible and acceptable color difference threshold values. (12). Alabdulwahhab et al. (14) stated that the color change range of 1.5-2.5 units could be recognized by an experienced clinician. Considering these threshold values, it could be concluded that the adhesives tested in this study caused an initial color change at a clinically acceptable level that could be detected by only experienced clinicians. Since some adhesives also cause perceptible color changes after aging, it can be concluded that color change due to adhesive in resin composite restoration after aging depends on the material. In a different *in vitro* study in which composite discs were kept in staining agents, it was found that the adhesives applied to the surface as modeling liquid did not affect the initial color and opacity (17).

From a clinical point of view, it may take a long time for adhesives to change color over time as a result of aging with water contact. Because the contact of the adhesive layer and water will be

through the dentinal tubules since the other surface is in contact with resin composite. For this reason, in this study, immersion of the adhesive applied resin composite discs directly in water for 30-day can be considered as an accelerated aging process. On the other hand, although it has a narrow surface, the adhesive layer is in direct contact with the oral environment through the resin-composite interface. For this reason, the color stability of the adhesive systems used with composite resin can play an important role in the aesthetic properties of the final restoration in the long term.

Study Limitations

There are some limitations of this study, since the storage conditions of the specimens are different from the oral cavity, it may not fully reflect the change of composite resins and adhesives as much as in clinical studies.

Conclusion

Within the limitations of this study, clinicians should keep in mind that although the effect of dental adhesive application on the initial and post-aging color changes of resin composites is not very perceptible, the color properties of the adhesive system can have some effect on the final resin composite restoration.

Ethics

Ethics Committee Approval: *In vitro* study.

Informed Consent: *In vitro* study.

Peer-review: Externally peer reviewed.

Authorship Contributions

Concept: M.K.A., Design: M.K.A., Data Collection or Processing: H.G.D., B.K., Analysis or Interpretation: M.K.A., Literature Search: M.K.A., Writing: H.D.B.

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