

EFFECT OF SURFACE SEALING ON STAIN RESISTANCE OF A NANO-HYBRID RESIN COMPOSITE*

Nano-Hibrit Bir Kompozitin Renkleşmesinde Yüzey Örtücülerin Etkisi

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Received: 10/12/2014

Accepted: 04/02/2015

ABSTRACT

Purpose: This study investigated the influence of sealant application on stain resistance of a nano-hybrid resin composite compared to the efficacy of a bonding agent used as a surface sealant on prolonging color stability of the resin composite.

Materials and Methods: 28 disc-shaped materials were prepared from a nano-hybrid resin composite Filtek Z550 and assigned to four groups: G1K: non-sealed; G2: Adper Single Bond; G3: Fortify ; G4: Biscover LV. After 24 h storage in distilled water at 37°C, all specimens were subjected to thermocycling and immersed into coffee solution. Color measurements were performed using spectrophotometer (VITA Easyshade; Vident) according to CIEL*a*b* system.

Results: Color change values were significantly different among the groups in each evaluation period except for after thermocycling ($p < 0.05$). For 7 days evaluation period, the difference between G3 and G4 group was statistically significant while G4 exhibited statistically significant differences ($p < 0.05$ and $p < 0.0001$ respectively) compared to control (G1) in 14 day whereas no significant difference was found between G1 and G2 in 28-day evaluation period. However, ΔE values of sealed specimens (G3, G4) differed significantly from non-sealed (G1) specimens after 28 days of immersion in coffee solution ($p < 0.05$ and $p < 0.0001$ respectively).

Conclusion: It may be concluded that using a bonding agent as a surface sealant does not increase stain resistance of resin composites of the sealants evaluated. Biscover LV showed the highest efficacy to prolong color stability of the resin composite.

Keywords: Color; surface sealant; discoloration; composite; coffee.

ÖZ

Amaç: Bu çalışmada nanohibrit yapıda bir reçine kompozit yüzeyine uygulanan yüzey örtücülerin ve dentin bağlayıcı ajan uygulanmasının; kompozitin (Filtek Z550) renkleşmeye karşı direnci üzerine etkisinin araştırılması amaçlanmıştır.

Gereç ve Yöntem: Çalışmada 28 adet Filtek Z550 nano hibrit kompozitten hazırlanan disk biçimindeki örnekler 4 gruba ayrılmıştır: G1K: yüzey örtücü uygulanmamış; G2: Adper Single Bond, G3: Fortify, G4: Biscover LV. 24 saat 37°C de distile suda bekletilip termosiklus uygulanan örnekler daha sonra günde 3 kere olmak üzere toplam 28 gün kahve solüsyonunda bekletilmişlerdir.

Bulgular: Başlangıç değerleri dışında, gruplar arası renk değişim değerleri, tüm zaman aralıklarında istatistiksel olarak anlamlı derecede farklı bulunmuştur ($p < 0.05$). 7 günün sonunda, gruplar karşılaştırıldığında G3 ve G4 grupları arasında istatistiksel olarak anlamlı derecede fark bulunmuştur. G4 grubu 14 günün sonunda kontrol grubuna göre istatistiksel olarak anlamlı derecede farklılık göstermiştir. Ancak 28 günlük deney sonucunda, G3 ($p < 0.05$) ve G4 ($p < 0.0001$) gruplarına ait ΔE değerleri istatistiksel olarak kontrol grubuna göre anlamlı bulunmuştur.

Sonuç: Bağlayıcı ajanın yüzey örtücü olarak kullanılmasının kompozitin renkleşmeye karşı direncini yeterli düzeyde arttırmadığı, Biscover LV yüzey örtücünün kompozitin renk stabilitesinin uzun dönem sağlanmasında en yüksek değerleri verdiği sonucuna varılmıştır.

Anahtar kelimeler: Renk; yüzey örtücü; renkleşme; kompozit; kahve.

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*This article has been presented as a poster at Conseuro Paris 2013 May 9-11.



Introduction

Tooth-colored restorations have been widely used in both posterior and anterior restorations since their first introduction (1). Currently, the rapid development of new materials and increasing esthetic demands make resin composites an inseparable part of dentists' routine practice. However, esthetic failures are still one of the most common reasons to replace the restorations (2). The color of the restoration should be matched with the color of the tooth and this combination must remain throughout the functional lifetime of the restoration. The discoloration of the resin composites may be caused by intrinsic or extrinsic factors (1, 3-5). Internal discolorations are permanent and can be caused by resin material itself. The resin matrix, the interface between the matrix and the fillers, type and amount of fillers and polymer quality have considerable influence on discoloration. Moreover, inadequate polymerization of light-cured composite resin may result in yellowish discoloration (1, 3, 5).

Extrinsic factors of discoloration may be superficial degradation and penetration or absorption of colorants from exogenous sources such as beverages and mouth rinses (1). Furthermore, surface roughness, surface integrity, and polishing procedures can affect the composite surface quality and therefore are related to the discoloration of restoration (1, 3, 5). In previous studies concerning color stability it was reported that beverages such as coffee (6-8), tea (9, 10), cola (6, 11), red wine (5, 12), whiskey (11), mouth rinses (13) and nicotine (14) may stain resin composites in different degrees. The staining potential of these drinks and solutions may vary according to their composition (5). In an attempt to overcome the color change in composites, low viscosity surface sealants were introduced. These materials are intended to penetrate into micro-porosities and micro-gaps to provide complete surface integrity. Moreover, absorption of colorants and pigments from the diet could be prevented and staining would be reduced by enhancing surface sealing (4, 12, 15). For the surface sealing, low viscosity dentin bonding agents could also be used even though they are not specifically formulated for such purpose. Although the performance of surface sealants have been investigated by many authors (4, 9, 16, 17) studies concerning dentin bonding agents are limited (8, 18, 19). Using dentin bonding agents to provide color stability is still controversial. The aim of the present study was to investigate the influence

of sealant application on stain resistance of a nano-hybrid resin composite and to evaluate the efficacy of a one-bottle total-etching bonding agent used as a surface sealant on prolonging color stability after thermal cycling. The null hypothesis was that the stain resistance capacity of the one-bottle total-etching dentin bonding agent does not differ from those of surface sealants.

Materials and Methods

Twenty-eight disc-shaped specimens (8 mm in diameter and 2 mm in thickness) were prepared from a novel nano-hybrid resin composite (Filtek Z550, 3M ESPE, St. Paul, MN, USA) using a custom-made stainless steel mold. The resin material was inserted into the mold standing on a glass plate with a transparent polyester strip in one increment. Subsequently, top-surface of the resin-filled mold was covered with another polyester strip and a glass plate was placed over it. Standard pressure (with a 1 kg weight) was applied on the glass plate for 15 seconds to let the excess resin out from the specimen surface to obtain a flat specimen surface without bubble formation. Following removal of the weight and the glass plate, the resin material was polymerized with a conventional halogen light curing unit (Hilux, Benlioglu Dental Inc., Ankara, Turkey) following the manufacturer's instructions with a mean irradiance of 550 ± 50 mW/cm² for 20 seconds. Tip of the light curing unit was positioned perpendicular to specimens' surfaces and the distance between the tip of the light source and specimen was standardized using a 1 mm thick glass microscope slide. Light intensity of the curing unit was measured by a radiometer (Hilux Dental Curing Light Meter, Benlioglu Dental Inc., Ankara, Turkey) before each use. Specimens were removed from the molds and numbers were scratched on their bottom surfaces with a scalpel. All specimens were stored in distilled water at $37 \pm 1^\circ\text{C}$ for 24 hours to finalize polymerization. Top surface of each specimen was polished with flexible aluminum oxide discs (Sof-Lex; 3M ESPE, St. Paul, MN, USA) under running tap water in the order of the manufacturer's instructions (for 30 seconds each step). Polishing was performed by the same operator in order to eliminate the operator dependent variables and the sofex discs were changed after 3 times of use. Specimens were then randomly assigned to four groups of 7 specimens each (G1, G2, G3 and G4 groups). G1 was unsealed for control; while G2,

G3 and G4 were sealed with different low-viscosity resins. Specimens in G2, G3 and G4 groups were conditioned for 15 seconds with 37% phosphoric acid gel (Etchant Gel, Prime Dent, Chicago, IL, USA). Following application of the etchant, the specimens were thoroughly rinsed with an air/water syringe ensuring that no acid gel residues were left on the specimen surfaces. Then, the specimens were gently air-dried. One-bottle bonding agent was applied to specimens in G2 group (Adper Single Bond 2, 3M ESPE, St. Paul, MN, USA) a surface sealant (Fortify, Bisco Inc. Schaumburg, USA) was applied to those in G3 group, a liquid polish (Biscover LV, Bisco, Schaumburg, IL, USA) was applied over the surfaces of the specimens in G4 group with a micro brush for 15 seconds using a light brushing motion. Then, top-surfaces of the specimens were gently air-thinned for 3 seconds and polymerized with the conventional halogen light curing unit with respective exposure times according to the manufacturers' instructions. Compositions and directions for use of all materials used in this study are shown in Table 1. The specimens were stored in distilled water at $37\pm 1^\circ\text{C}$ for another

24 hours to ensure complete polymerization of the sealing resins. Then, all specimens were subjected to ageing through 1500 thermal cycles at temperatures alternating between 5°C and 55°C with a dwell time of 30 seconds in each water bath (20). After thermocycling was completed, the specimens were exposed to immersion regimen (3, 4, 20-22). The specimens in each group were immersed into 30 ml non-sweetened black coffee (Nescafe, Nestlé, Vevey, Switzerland) solution prepared using a 3:10 powder: mass ratio as previously described (21, 22) three times a day 15 minutes each and kept at $37\pm 1^\circ\text{C}$. Between the immersion periods, the specimens were kept in distilled water at $37\pm 1^\circ\text{C}$. The immersion regimen was followed for four weeks. Color measurements were performed against a white background using a spectrophotometer (Vita Easyshade, Vita Zahnfabrik, Bad Säckingen, Germany) according to the CIEL*a*b system following thermocycling and at the end of one, two and four weeks.

Table 1. Compositions and application techniques of the materials which were used in this study.

Product	Composition	Lot Number	Application procedures
3M ESPE Adper Single Bond 2 Adhesive	ethyl alcohol, Bis-GMA, silicatreatednanoparticles, HEMA, glycerol 1, 3 dimethacrylate, acrylic acid, copolymer and itaconic acid, diurethane dimethacrylate	N121854	Composite surface was acid-etched with for 15 seconds. Acid was rinsed away; the surface was air dried and one thin coat of material was applied. This layer was air-thinned by blowing a gentle stream of air. Light-curing for 20 seconds with a halogen light.
Bisco Fortify Composite Surface Sealant	urethane dimethacrylate	0900010565	Composite surface was acid-etched with for 15 seconds. Acid was rinsed away; the surface was air dried and one thin coat of material was applied. This layer was air-thinned by blowing a gentle stream of air. Light-curing for 10 seconds with a halogen light.
Bisco BisCover LV Low viscosity liquid polish	dipentaerythritolpentaacrylate, ethanol	1100001424	Composite surface was acid-etched with for 15 seconds. Acid was rinsed away; the surface was air dried and one thin coat of material was applied. Waited for 15 seconds to allow evaporation. Light-curing for 30 seconds with a halogen light.

Before measurement, top-surface of each specimen was blotted dry using tissue paper and the contact guide of spectrophotometer was positioned on the center of the specimen surface. Three consecutive readings were obtained for each specimen and then averaged. The colour differences (ΔE) were calculated

using the following equation:

$$\Delta E: [(L_0 - L_i)^2 + (a_0 - a_i)^2 + (b_0 - b_i)^2]^{1/2}$$

ΔE = color change, L = luminance reflectance, a = red-green color coordinate, b = yellow-blue color coordinate, $_0$ = baseline, $_i$ = after treatment.

Statistical analysis

Statistical Package for Social Sciences (SPSS) software package version 15.0 (SPSS Inc., Chicago, IL, USA) was used in this study. The distributions of the variables were consistent with the assumptions of normal distribution. Therefore, one-way analysis of variance (ANOVA) and Bonferroni tests were used for multiple and pairwise comparisons to analyze color differences (ΔE), respectively. Confidence interval was set 95% and p values less than 0.05 were considered statistically significant.

Results

The mean and standard deviations (SD) of the color change values (ΔE) for the unsealed and sealed groups after thermal cycling and immersion in the coffee solution for 15 minutes three times a day at 1, 2 and 4 weeks are summarized in Table 2. There was no statistically significant alteration in color after the thermocycling procedure. The data analysis showed that the groups demonstrated statistically significant color change at 1, 2 and 4 weeks ($p < 0.05$).

For the first week evaluation, the mean values of color change of the groups showed clinically perceptible discoloration ($\Delta E > 3.3$) except for the Biscover LV group ($\Delta E = 2.99 \pm 1.77$). Even though there was a non-sealed control group, Fortify group (G3) had the highest discoloration overall. There was statistically significant differences between Fortify and Biscover LV group ($p = 0.048$) and no significant differences were observed between other treatment groups.

At two weeks, Fortify group still had the highest discoloration in all groups. Each group had the higher ΔE values than the first week. Unsealed control and Fortify groups had statistically significant discoloration when compared to other two groups ($p < 0.05$). No significant differences were detected among G2 and G4 groups ($p > 0.05$). At the end of 4 weeks, discoloration between groups did not follow the same pattern as before. Group G2 showed highest discoloration in sealed groups with no significant differences among unsealed control ($p = 0.494$) and group G3 ($p = 0.321$). Group G4 exhibited significantly lower discoloration in all group tested ($p < 0.05$) except for the group G3 ($p = 0.757$).

Table 2. Mean values of color differences (ΔE) with standard deviations (SD) for each group in each evaluation period. Different superscript letters in the same row indicate statistically significant difference.

Immersion period	G1-Control	G2-Sealed	G3-Sealed	G4-Sealed	p value
	(Unsealed)	(Single Bond)	(Fortify)	(BisCover LV)	
	$\Delta E (\pm SD)$	$\Delta E (\pm SD)$	$\Delta E (\pm SD)$	$\Delta E (\pm SD)$	
After thermal cycling	1.19(± 0.68) ^a	1.14(± 0.72) ^a	1.73(± 1.12) ^a	1.34(± 0.47) ^a	p=0.511
1 week	5.05 (± 1.70) ^{abc}	4.23 (± 1.02) ^{abc}	5.24 (± 1.15) ^b	2.99 (± 1.77) ^c	p=0.032
2 weeks	6.98 (± 1.06) ^a	5.08 (± 0.73) ^b	7.05 (± 1.27) ^a	4.00 (± 1.41) ^{bc}	p<0.001
4 weeks	9.70 (± 1.50) ^a	8.28 (± 1.52) ^{ab}	6.68 (± 1.75) ^{bc}	5.44 (± 0.97) ^c	p<0.001

Discussion

The null hypothesis of the study was rejected. Dentin bonding agent had the lowest stain resistance among surface sealant groups after 28 days. Hence, dentin bonding agents are not recommended using as surface sealants related to their lack of stain resistance according to present study. With the improvement of restorative materials and the demand for aesthetic restorations, the composite resins have become the material of choice for both anterior and posterior restorations (1). But despite the improvement of its physical and chemical properties, color stability is

still a drawback on the longevity of the restoration (23, 24). The discoloration of tooth colored resin-based composite materials could be a reason for the replacement of dental restorations in aesthetic regions. Not only patients but also dentists have concerns about the replacement procedure which is both time- and money-consuming. Besides, each removal of restorations would cause additional damage in the remaining tooth structure (25). Even short period of immersion in staining solutions (i.e. coffee, tea, cola) like 7 days, is sufficient to produce staining and color changes of composite resins (26-28). This study was conducted on aged nano-hybrid resin composite

discs to simulate thermal alterations that occur in oral environment. A recent study showed that the adequate evaluation of color stability in restorations could be carried out by either visual assessment with shade guides or use of a digital spectrophotometer (26). The perception of tooth color may be influenced by many factors such as lighting conditions, translucency, opacity, light scattering and human eye (29). Quantitative evaluation of minimal color change by means of visual assessment is not possible or even useful most of the times; besides presenting low reproducibility. However, standardized devices could be used for such measurements. Advancements in electronic optics and informatics could make the electronic techniques for color selection more adequate for daily usage (26, 28, 30). To minimize potential subjective errors in color assessment, the present study used a spectrophotometer for color measurements. For this reason, VITA Easyshade system (Vita Zahnfabrik, BadSackingen, Germany) was used. This spectrophotometer measures precise sections of the visible light spectrum, within 400 to 700 nm, expressing it in ΔE units. These systems are more precise, in comparison with colorimeters, if they are not influenced by the environment luminosity. ΔE values can be used to present color changes of restorative materials going through determined treatment or after certain periods of time (26, 28, 31). The parameter ΔE indicates the relative color changes that an investigator could use for the materials before and after treatment or between intervals (1). According to related clinical evaluations, values of $\Delta E < 1$ are regarded as not appreciable by the human eye (1). Values $1 < \Delta E < 3.3$ are considered appreciable by skilled operators (32), but clinically acceptable, whereas values of $\Delta E > 3.3$ are considered appreciable by non-skilled persons, and are clinically unacceptable (33). Therefore, color changes above a value of $\Delta E = 3.3$ were considered clinically unacceptable (10).

The staining ability of coffee was shown in many studies (10, 28, 34, 35). The staining potential of coffee had explained by authors (10, 35) colorants of coffee had both absorption and adsorption at the same time onto the surface of resin based materials. This absorption and penetration of yellow colorants were related to the compatibility of the materials' polymer phase (35). Therefore, we used coffee as the only staining solution in our study. The presence of more hydrophilic co-monomers in the resin sealant compared with the composite resin might also explain the decline in color stability (36). Application of a

resin surface sealant may have a detrimental effect by decreasing the color stability of composite restorations. However in one study (17), it was reported that the surface sealants could improve the stain resistance of a composite resin provisional material. Differences in the efficacy of surface sealants in providing staining resistance may be attributed to differences in the formulation of the materials. To reduce the discoloration of the resin composites resulted from surface roughness; polishing techniques have been widely recommended (9, 10, 28, 37). Previous studies (9, 28, 38) suggested that aluminum oxide flexible discs were the best instruments to generate low roughness in resin surfaces, and this finding was related to their capacity to reduce fillers and matrix evenly. Accordingly, aluminum oxide discs were used in the present study.

Accelerated ageing methods have been used in many studies evaluating discoloration of sealed composite resins (9, 12, 39, 40). Color changes in resin materials induced by UV irradiation have been correlated with chemical alterations in the initiator system, activators and the resin itself. The degradation of residual amines and oxidation of residual unreacted carbon-carbon double bonds culminate in the formation of yellowing compounds (9, 39). In addition, the physical and chemical properties of the monomers used in a resin matrix can influence resistance to staining (41). As the materials age, the water absorption characteristics of the resin monomers could contribute to differences in the degree of ΔE (9, 42). Composites kept in distilled water had color alterations over time because of the same absorption properties. It was previously shown (33, 43, 44) that the color changes in composite materials immersed in distilled water were imperceptible and clinically acceptable even after 1 month. Because of this reason, the specimens were kept in distilled water before immersion regimes and ΔE values had not measured shortly after polymerization in the present study. Baseline measurements were done after thermal cycling in which color alterations were thought to be clearly observed.

Catelan *et al.* (12) have found significant changes in micro-hardness values of both sealed and unsealed composites after accelerated ageing besides reporting no alterations in surface roughness. However, some of the studies (9, 40) have suggested ageing as the reason of the degradation and color changes in different composite resin materials. Thermal cycling has been used in studies that evaluates surface sealants

in terms of surface roughness and micro-leakage (19, 20). However, the behavior of surface sealants regarding color stability in thermal cycling procedures has not been systematically evaluated (45). For this reason; in the present study, the same cycle procedure (1500 thermal cycles) as Lima *et al.* (19) and Poggio *et al.* (20) was performed. As a result, aging resin composite with thermo-cycling did not affect the color change value irrespective of being sealed or non-sealed. However, different results could have been obtained if more than 1500 cycles were done. Further research is needed to elucidate this observation.

In this study, except for the Biscover LV group, the mean values of color changes of the groups after the first week was $\Delta E > 3.3$, which was considered visually perceptible. Even though there was a non-sealed control group, Fortify group had the lowest color stability overall. There was a statistically significant difference between Fortify and Biscover LV group. Fortify has no inorganic fillers and contains the most common resin material, the ethoxylatedbisphenol A dimethacrylate. This could be related to its lower stain resistance. The ethoxylatedbisphenol A is a dimethacrylate component, which is also present in many restorative resin composites (17) and may contribute to increase in staining. This background may explain the differences in the staining of Fortify specimens when compared to those covered with Biscover LV.

In the present study, Adper Single Bond had the lowest color stability compared with the other sealant groups after 4 weeks. However the difference was not statistically significant. This result was in agreement with a study in which Heliobond used as a dentin bonding agent showed no statistical difference when compared to Biscover LV (8).

Conclusion

Resin composites sealed with bonding agent showed increased stain resistance after 2 weeks of immersion regimen; but longer immersion periods caused higher color alteration as observed in non-sealed specimens. Even though specimens sealed with surface sealant materials showed higher stain resistance compared to control group; considering the adopted classification of ΔE values, the color change values in all groups are in the category of marked change. In addition, current knowledge on the effect of surface sealants on stain resistance of resin composites is limited. More studies are needed

before using or recommending surface sealants over resin composite restorations.

Source of funding

None declared

Conflict of interest

None declared

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