

Effect of surface sealant on the surface roughness of different composites and evaluation of their microhardness*

Purpose

The aim of this study was to evaluate the effect of a surface sealant on the surface roughness of different composites and compare their microhardness values.

Materials and Methods

Sixty disc-shaped specimens were prepared and assigned to 6 groups (n =10). Groups were prepared as follows; Group 1 (Herculite XRV Ultra), Group 2 (Beautiful Bulk Restorative) and Group 3 (Filtek Bulk Fill Posterior Restorative). Groups 4, 5, and 6 were prepared by applying a surface sealant (BisCover LV) on the specimens in groups 1, 2 and 3. Surface hardness of the discs in group 1, 2, and 3 and surface roughness of the discs in all groups were measured using the Vickers hardness test and a profilometer, respectively. One-way ANOVA was used to test for differences among the groups.

Results

No significant differences were observed in the microhardness and roughness between the experimental and control groups for each restorative materials. Group 3 showed the highest surface hardness and group 4 showed the lowest surface roughness values.

Conclusion

Using the BisCover LV resin after the polishing step has no significant effect on the surface roughness. The highest hardness values were obtained for the Filtek Bulk Fill Posterior Restorative after the polishing step. The smoothest surfaces were obtained for all experimental groups using the BisCover LV resin after the polishing step, Herculite XRV Ultra showed lower average roughness values than the other materials.

Keywords: *Roughness; microhardness; resin composite; surface sealant; mechanical properties*

Introduction

Dental composite resins are commonly used restorative materials for the replacement of defects in hard dental tissues (1,2). Despite of satisfactory mechanical and esthetic properties, they have some disadvantages too. Polymerization shrinkage causes some problems such as post-operative sensitivity, secondary caries and marginal leakage. To remove these problems, less than 2-mm- thick layering should be done and it is a time-consuming process. Thus, bulk fill composites have been produced that are claimed to have a low shrinkage stress (3).

Regardless of the cavity class, location and type of the composite material, a smooth surface finish is clinically important because it determines the esthetics and longevity of composite resin restorations (4). Proper finishing of restorations is desirable not only for esthetics but also for good oral health by preventing plaque retention (5). Surface roughness of dental materials can cause microtrauma to the oral tissues and enhance the

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retention of microorganisms, thereby contributing directly or indirectly to tissue injuries and possible oral diseases (6,7). Therefore, a smooth surface finish is important to maintain good oral health by reducing microorganism retention and plaque accumulation as well as for good esthetic appearance and less recurrent caries and gingival irritation (8-11). Moreover, average surface roughness (Ra) above the 0.2 µm threshold has been reported to increase the colonization and adhesion of bacteria on composite resin surfaces (12). The surface roughness of composite resin is usually determined by the size, hardness, and amount of the filler particles, flexibility of the material, and the hardness and grit size of the abrasive (13).

It has been shown that the surface micromorphology of composite resins after finishing and polishing steps can be influenced by the size, hardness and amount of the filler particles (14). During the polishing of hybrid composites, the harder filler particles are left protruding from the surface, whereas the softer resin matrix is preferentially removed. Therefore, the harder filler particles should be packed close together to protect the soft resin matrix from abrasives (15). The combination of reduced particle dimensions and wider size distribution allows the higher levels of filler loading, resulting in reduced polymerization shrinkage and improved mechanical properties (16). To achieve an effective finishing system for composite resins, the abrasive particles should be relatively harder than the filler materials to prevent the preferential removal of the soft resin matrix during polishing, leaving the harder filler particles protruding from the surface (13). According to earlier work, larger filler particles have resulted in greater Ra values (17,18). The composite resins with higher concentrations of small-sized filler particles have become popular in recent years due to the difficulties in producing smooth surfaces similar to the enamel surface using the composite resins that have larger filler particles. Typically, increased amounts of filler particles result in smoother surfaces because of the decreased particle size and better particle distribution within the resin matrix (13).

Surface sealants have been developed to preserve or improve the mechanical properties of direct restorative materials (19,20). Thus, application of the surface sealants after the polishing step has been recommended to increase the longevity of restorations (21, 22). Liquid polishing materials are low-viscosity, light-polymerized resin formulations with a low amount of filler particles that provide a smooth, sealed surface for interim and composite resin restorations (14,23,24). Surface penetrating sealants (SPS) are unfilled low-viscosity resins polymerized onto the composite surfaces to promote the filling of structural microdefects and microfissures by capillary action (25) for maintaining the

surface smoothness, improving the wear resistance (25, 26) and marginal sealing (27) of the restoration. Since various surface defects such as microcracks and irregularities are formed due to the removal of some of the surface particles during finishing, application of the liquid resin to the finished material surface has been

recommended to repair the structural microdefects and improve the abrasion resistance of posterior composite resins (28,29).

However, the effectiveness of sealants in improving the smoothness of composite surfaces is still controversial. Although some authors have suggested that sealants might be desirable to improve the surface finishing of composites (21,30), others have reported no significant reduction in the surface roughness of composites after the simulated abrasion test (31) and also in clinical evaluations after one and five years (25, 32).

Substantial surface hardness of the restoration is one of the main requirements in high stress-bearing areas such as posterior restorations (33) Materials which have reduced surface hardness are more susceptible to deformation (34). Microhardness can be influenced by monomer type, filler type, morphology, volume and weight (34-36). Moreover, finishing and polishing of the restoration can affect the hardness of the composite materials (34).

There are some studies that compare microhardness of different bulk fill composite materials and declare various results (37-41). It is stated that bulk fill composites with low filler content showed lower microhardness than with high filler contents (3,42,43). Despite of various results about the microhardness of different bulk fill composites, there has been no previously reported study that compare the microhardness of bulk fill composites chosen in our study each other.

The aim of this study was to evaluate the effect of the BisCover LV resin sealant on the roughness of different composites and compare their microhardness values. Based on this information, the following hypotheses were tested: (1) surface sealant reduces the roughness of composite materials (2) there were no significant difference between the microhardness of the composite materials used in this study.

Materials and Methods

Specimen preparation

Ten disc-shaped specimens with 10 mm in diameter and 2 mm in thickness were prepared in a teflon mold for each study group (Figure 1)(11). Total sixty disc-shaped specimens were prepared for the surface property tests and divided into 6 groups. Different study groups and materials used

Table 1. Groups and materials used

n=10	group 1	group 2	group 3	group 4	group 5	group 6
Composite restoration	Herculite XRV Ultra	Beautifil Bulk Restorative	Filtek BulkFill	Herculite XRV Ultra	Beautifil Bulk Restorative	Filtek BulkFill
BisCover application after restoration	-	-	-	+	+	+

Table 2. Characteristics of materials tested

	Composition	Manufacturer	Classification	Filler	Filler loading
Herculite XRV Ultra	Ethoxylated Bis-GMA, TEGDMA, BisEMA	Kerr, Orange, CA, USA	Nano-hybrid composite	SiO ₂ , Barium silicate glass, Prepolymerized filler with barium silicate glass and silica	71 wt% /54 vol%
Beautiful Bulk Restorative	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	Shofu Inc, Kyoto, Japan	Giomer based bulk fill resin composite	Surface modified prereacted glass (S-PRG) filler based on fluoroboroaluminosilicate glass, polymerization initiator	87 wt% /74.5 vol%
Filtek BulkFill	Bis-GMA, UDMA, Bis-EMA(6), procrylat resins	3M ESPE, St. Paul, MN, USA	Bulk-fill paste composite with glass microfibres	Zirconia/Silica, ytterbium trifluoride	76.5 wt%/58.5 vol%
Biscover LV	Dipentaerythritol pentaacrylate esters and Etanol	Bisco Inc, Schaumburg, IL, USA	Low-viscosity liquid polish		

in this study are outlined in Table 1, and the properties and type of the used materials are presented in Table 2. The composite resins were poured in a Teflon mold covered with a polyester strip and a glass slide (1 mm thick) was then placed over the polyester strip to flatten the surfaces according to the composite manufacturer's recommendation. The restorative materials were light-cured (Optilux Demetron, VLC 403, Danbury, CT, USA, 500 mW/cm²). Herculite XRV Ultra was applied into the mold and light-cured for 20 s. Beautiful Bulk Restorative and Filtek BulkFill were applied into the mold and light-cured for 20 s and 40 s, respectively. Afterwards, the surfaces of the specimens were polished for 30 s from extra-coarse grain size to extra-fine grain size with polishing discs (OptiDisc, Kerr Hawe, Karlsruhe, Germany). A new polishing disc was used for each specimen and then discarded after each use. Specimens in experimental groups 4, 5, and 6 were etched with 32% phosphoric acid (Uni-Etch, Bisco Inc.,

Schaumburg, IL, USA) for 15 s. Then, the etched specimens were rinsed with water and air dried before directly applying the BisCover LV resin (dipentaerythritol pentaacrylate in ethanol) (BisCover, Bisco Inc., Schaumburg, IL, USA) using a syringe and an applicator tip. After a 15 s wait for ethanol vaporization, specimens were light polymerized for 30 s with Optilux as the manufacturer's instruction. The light curing unit tip was positioned perpendicular to the specimens' surfaces, and the distance between the tip and the specimen was standardised using a glass microscope slide (1 mm in thickness). All samples were stored in distilled water at 37 °C for 24 hours. This research was conducted at Istanbul Medipol University and Istanbul University Laboratory.

Microhardness measurements

Surface hardness of different composite resins was measured using the Vicker's hardness test because of its ease-of-use and reliability of the measurements (44). The microhardness values for the samples in groups 1, 2, and 3 were obtained using an Innovatest Nexus 4503 hardness testing machine (Innovatest Europe, Maastricht, The Netherlands) for loads of 2.5 – 10 kgf (24.51 – 98.07 N) (Figure 2). The surface hardness measurements were performed using a microscope at 20x magnification under a load of 300 g for 15 s. The applied load and the hold time were kept constant for all samples throughout the study. The measurement was carried out three times in each sample at random locations and a mean value was calculated.

Surface roughness measurements

Surface roughness of different composite resins was determined using the roughness average (Ra) parameter, which represents the arithmetic average of absolute values

**Figure 1.** Teflon mold for composites specimens.

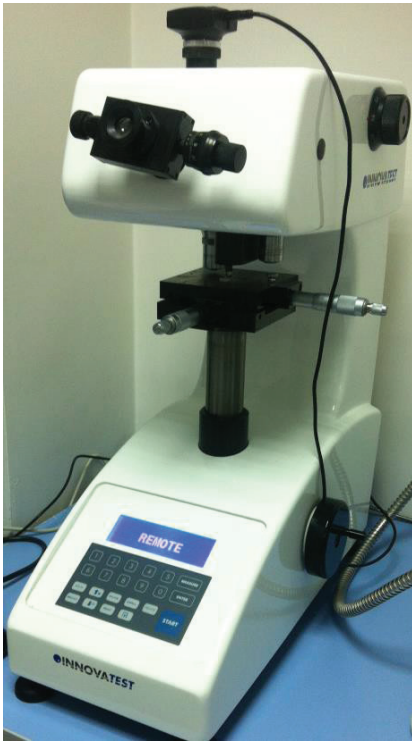


Figure 2. Hardness testing machine.

of the profile height deviations from the mean line recorded within the evaluation length (45). A profilometer (Taylor Hobson Surtronic 25, UK) was used for measuring the Ra values of groups 1, 2, 3, 4, 5, 6 with a cut-off value of 0.8 mm, a transverse length of 0.8 mm, and a stylus speed of 0.1 mm/seconds. For surface roughness test, readings were taken at three random locations on each surface and the average roughness value (Ra, μm) was obtained by using the arithmetic mean of these three readings.

Statistical analysis

Statistical analysis of the data was performed with Statistical Package for Social Sciences (SPSS) statistical software (SPSS PC, Vers.15.0; SPSS Inc.; Chicago, IL, USA). Descriptive statistics for continuous variables were calculated and reported in a mean \pm standard deviation format. To detect differences among Ra and microhardness values for different groups, a one-way analysis of variance (ANOVA) method was used at the 0.05 level of significance.

Results

Mean microhardness and roughness values for different groups are presented in Table 3 and Table 4. There were no significant differences in microhardness and roughness values between the experimental and control groups for each restorative material ($p > 0.05$). Based on the test results, group 3 showed the highest surface hardness and group 4 showed the lowest surface roughness values.

Discussion

The clinical significance of surface roughness and hardness is related to the esthetic restorations (discoloration and wear), the medical consequences of periodontal disease, and the development of secondary caries due to increased plaque accumulation. Wear and microleakage are the main limitations of the composite resins in mainly posterior restorations (46). Several research groups have studied the surface characteristics of different restorative materials (11,31,47-49) using contact profilometers, which detect surface irregularities using a stylus moving vertically across the surface. In addition, clinical studies have shown that the rough surfaces

Table 3. Mean microhardness values and differences within groups

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		p
					Lower bound	Upper Bound	
1	10	61,1370	10,10009	3,19393	53,9118	68,3622	0.105
2	10	58,4860	8,32322	2,63203	52,5319	64,4401	
3	10	66,6520	6,43766	2,03577	62,0468	71,2572	
Total	30	62,0917	8,83112	1,61233	58,7941	65,3893	

Table 4. Mean Roughness (Ra) values and differences within groups

	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		p
					Lower Bound	Upper Bound	
1	10	0,8540	0,23153	0,07322	0,6884	1,0196	0.370
2	10	0,7730	0,24459	0,07735	0,5980	0,9480	
3	10	0,7190	0,14579	0,04610	0,6147	0,8233	
4	10	0,7030	0,25347	0,08015	0,5217	0,8843	
5	10	0,7110	0,25723	0,08134	0,5270	0,8950	
6	10	0,8720	0,19921	0,06300	0,7295	1,0145	
Total	60	0,7720	0,22636	0,02922	0,7135	0,8305	

can promote plaque formation and reduce the efficiency of teeth cleaning procedures (50). Bollen et al. reported that a Ra of 0.2 μm or more could result in accumulation of bacterial plaque, thereby promoting the periodontal diseases and carious lesions (12). However, results from this study showed that the mean Ra and microhardness values obtained at the baseline for the experimental and control groups did not differ statistically from each other.

Effective surface sealants should have good surface wettability, a low contact angle, a low viscosity, and good penetration capability. Microgaps may occur between tooth/restoration interface depending on the polymerization shrinkage during restoring the tooth with resin composites. Surface sealants minimize the wear rates of the resins by filling the microdefects on the restorations (46). Therefore, the presence of low molecular weight monomers was found to be essential in dental sealants (47). It was assumed that a surface sealant containing Bis-GMA combined with low molecular weight monomers (TEGDMA and THFMA) would control its desirable characteristics such as viscosity and surface wettability (25). Also fillers were added to some sealants to increase their mechanical properties (46).

The wear resistance of composite resins can be enhanced with a surface sealant, as long as it is annually applied. In an *in vivo* study, the researchers found that the wear values of the sealed restorations after one year were approximately half of those found in the non-sealed restorations

(25). In addition, these low viscosity resins can increase the wear resistance of the tooth/restoration interface in luting indirect restorations (26).

Although relatively smoother surfaces were obtained with the polyester strips, the use of a glazing material after the polishing step resulted in significantly lower Ra values compared to that obtained with the use of polyester strips alone. The glazing material appeared to fill the structural microdefects, thereby providing a more uniform and smooth surface (30). However, some initial investigations demonstrated the degradation of the glazing materials over time, in spite of their resistance to toothbrushing and staining (31,46,51). Therefore, the limitations of this *in vitro* study have to be developed and improved in terms of aging.

The effectiveness of sealants in improving the smoothness of composite surfaces is still controversial. Although some authors have suggested that sealants might improve the surface finish (30), others have reported no significant reduction in the surface roughness of composites after the simulated toothbrushing abrasion test and also in clinical evaluations after one and five years (25). The complex structure of a surface cannot be fully characterized by the use of only surface roughness measurements (15).

According to Shintani and others, there were no noticeable differences in plaque accumulation among the surfaces polished using different methods, which resulted in Ra values within the range of 0.7-1 μm (52). Chung reported that restorations with less than 1 μm surface roughness appeared to be optically smooth (14).

The inherent surface roughness of composite resins should be equal or lower than the surface roughness of enamel-to-enamel occlusal contact areas (Ra = 0.64 μm). When comparing the roughness values of optimally polished surfaces, mostly the surface roughness values produced by

pressing the restorative materials against transparent matrices such as Mylar strips (53).

Thus, very smooth polished surfaces representative of the clinical situation can be obtained using clear matrices. Although the surface obtained with Mylar strip is perfectly smooth, it is rich in resin organic binder (53). Therefore polishing discs were used to mimic the clinic conditions before applying the surface sealant in this study.

It has been reported that a noticeable decrease in mean surface roughness could be achieved within first five seconds of polishing for practically all restorative materials, but a further decrease of the same magnitude could not be obtained with longer polishing times or the application of additional components (54). Thus, one-step polishing systems offer time saving benefit along with reduced roughness when polishing the composite restorations. Based on this fact one-step surface sealant was used when evaluating the resin surfaces in this study.

The first tested hypothesis was rejected because the surface sealant material decreased the surface roughness of composite resins, but there were no significant differences in the roughness values between the experimental and control groups for each restorative material. Different results may be obtained with different polishing techniques and composite resin materials.

Several factors related to the composite resin compositions were shown to affect the surface hardness of the composite restorative materials (54). It was observed that the mass fraction (55,56), size, and distribution of filler particles have significant effects on certain physical and mechanical properties, including surface hardness of the composite resins (57,58). Moreover, other parameters such as filler particle shape and density, monomer type and ratio, degree of crosslinking, and photoinitiators have also shown significant influence on the surface hardness of restorative materials (55,59).

A microhardness test gives information as to the mechanical properties of the material. A positive correlation has been determined between the hardness and inorganic filler content of composites. Increased organic filler levels result in increased hardness values (60,61).

In a study on filler particle size effect, significant high differences were noticed in the VHN (Vickers hardness number) mean values among bulk-fill and incrementally-fill composite resins, either for top or bottom surfaces. The highest VHN value was obtained for the incremental-fill nanohybrid composite (Grandio) compared to that of the two bulk-fill microhybrid composites (X-tra fil and QuiXfil) (62). According to Moszner et al. (16) and Thome et al. (63), the microhybrid composite resins exhibited higher microhardness values than that of the nanohybrid composite resin.

The second tested hypothesis that, there were no significant differences between the microhardness of the composite materials used was accepted. Despite there were no significant differences between the groups tested, group 3 had the highest microhardness values. This could be attributed to the filler particles of (glass microfibres, zirconia, silica and ytterbium trifluoride) the Filtek Bulk-fill. Also it can be speculated that bulk-fill resin composites allow more light to penetrate deep inside and which can result in more polymerized monomers. Our findings are in agreement with a previous study (64).

Therefore, further investigation is necessary to evaluate the surface roughness and microhardness values for different composite resins and polishing techniques.

Conclusion

In this study, the highest hardness values were obtained using Filtek Bulk Fill Posterior Restorative (silane treated ceramic, 3M-ESPE, Germany) after the polishing step. The smoothest surfaces were obtained using a surface sealant after the polishing step, Herculite

XRV Ultra showed lower Ra values compared with those of the other restorative materials. No significant differences were found in the surface roughness of selected composite resins sealed with BisCover LV. Similarly, the microhardness values showed no significant differences among different composite resin materials. Hardness value obtained for group 3 is higher but not significantly different compared to that of the groups 1 and 2. As a result, the glazing material showed a negligible effect on the surface roughness values of different polished composite resins. The current generation of composite resins focused on the filler particle size (nano-fill and bulk-fill) have improved the surface properties such as hardness and roughness of restorative materials. Therefore, the use of sealants to improve the smoothness and hardness of these composite restorations is questionable. Longitudinal clinical trials are necessary to validate this hypothesis and provide further insights into the design of composite resins for clinical use.

Türkçe Öz: Yüzey örtücü materyalin farklı kompozitlerin yüzey pürüzlülüğü ve mikrosertliği üzerine etkisinin değerlendirilmesi. Amaç: Bu çalışmanın amacı, bir yüzey örtücü materyalin, farklı kompozitlerin yüzey pürüzlülüğü üzerindeki etkisini değerlendirmek ve mikro sertlik değerlerini karşılaştırmaktır. Gereç ve yöntem: Altmış adet disk şeklindeki test numunesi hazırlanmış ve 6 gruba ayrılmıştır (n = 10). Gruplar aşağıdaki gibi hazırlanmıştır; Grup 1 (Herculite XRV Ultra), Grup 2 (Beautiful Bulk Restorative) ve Grup 3 (Filtek Bulk Fill Posterior Restorative). Grup 4, 5 ve 6 da sırasıyla grup 1, 2 ve 3'te kullanılan restoratif materyallere ek olarak bir yüzey örtücü materyal (BisCover LV) kullanılmıştır. Daha sonra Grup 1, 2 ve 3'teki disklerin yüzey sertliği ve tüm gruplardaki disklerin yüzey pürüzlülüğü sırasıyla Vickers sertlik testi ve bir profilometre kullanılarak ölçülmüştür. Gruplar arasındaki farklılıkları test etmek için tek yönlü ANOVA kullanılmıştır. Bulgular: Her bir restoratif materyal için deney ve kontrol grupları arasında mikro sertlik ve pürüzlülük açısından istatistiksel olarak anlamlı bir fark gözlenmemiştir. Grup 3 en yüksek yüzey sertliğini, grup 4 ise en düşük yüzey pürüzlülüğünü göstermiştir. Sonuç: Cila aşamasından sonra bir yüzey örtücü (BisCover LV) kullanılması farklı kompozitlerin yüzey pürüzlülüğü üzerinde istatistiksel olarak anlamlı bir etkiye sahip değildir. En yüksek sertlik değerleri, cila aşamasından sonra Filtek Bulk Fill Posterior Restorative için elde edilmiştir. Tüm deney grupları için cila aşamasından sonra yüzey örtücü (BisCover LV) kullanılarak en pürüzsüz yüzeyler elde edilmiştir. Herculite XRV Ultra diğer materyallere göre daha düşük bir ortalama pürüzlülük değeri vermiştir. Anahtar kelimeler: Pürüzlülük; mikro sertlik; rezin kompozit; yüzey örtücü materyal, mekanik özellikler.

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