Resin composite repair: Quantitative microleakage evaluation of resin-resin and resin-tooth interfaces with different surface treatments

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ABSTRACT

Objective: The aim was to evaluate the effect of different adhesive systems and surface treatments on the integrity of resin-resin and resin-tooth interfaces after partial removal of preexisting resin composites using quantitative image analysis for microleakage testing protocol. Materials and Methods: A total of 80 human molar teeth were restored with either of the resin composites (Filtek Z250/GrandoSO) occlusally. The teeth were thermocycled (1000×). Mesial and distal 1/3 parts of the restorations were removed out leaving only middle part. One side of the cavity was finished with course diamond bur and the other was air-abraded with 50 μm Al₂O₃. They were randomly divided into four groups (n = 10) to receive: Group 1: Adper Single Bond 2; Group 2: All Bond 3; Group 3: ClearfilSE; Group 4: BeautiBond, before being repaired with the same resin composite (Filtek Z250). The specimens were re-thermocycled (1000×), sealed with nail varnish, stained with 0.5% basic fuchsin, sectioned mesiodistally and photographed digitally. The extent of dye penetration was measured by image analysis software (ImageJ) for both bur-finished and air-abraded surfaces at resin-tooth and resin-resin interfaces. The data were analyzed statistically. Results: BeautiBond exhibited the most microleakage at every site. Irrespective of adhesive and initial composite type, air-abrasion showed less microleakage except for BeautiBond. The type of initial repaired restorative material did not affect the microleakage. BeautiBond adhesive may not be preferred in resin composite repair in terms of microleakage prevention. Conclusions: Surface treatment with air-abrasion produced the lowest microleakage scores, independent of the adhesive systems and the pre-existing resin composite type. Pre-existing composite type does not affect the microleakage issue. All-in-one adhesive resin (BeautiBond) may not be preferred in resin composite repair in terms of microleakage prevention. Key words: Adhesive System, composite repair, microleakage, surface treatment

INTRODUCTION

Despite ongoing advancement in adhesive technologies including improvements in mechanical and physical properties of the resin systems, polymerization systems, and the careful and conscientious techniques of dentists, most resin-based restorations’ service life is limited. In fact, the longevity of routine resin-based restorations in permanent teeth is limited to 6–10 years with a median survival time of 7 years.[1,2] The replacement of defective restorations has been reported as the most common approach, representing a major part of restorative dentistry in today’s general dental practice.[3] However, this approach may be considered as overtreatment when some portions of the restorations are clinically considered to be intact and still serviceable.[4] It should be considered that replacement of a restoration means widening of the cavity preparation...
and excessive tooth structure removal result in complex dental treatments, as endodontic and/or prosthetic treatment approaches which were not cost-effective and time-consuming. Although, conservative dentistry aims to avoid the patient from this dilemma by using one or more conservative alternative treatment options like repairing the localized defects.[5] However, a variety of repair techniques concerning the best one are found in the literature. The basic principle of the repair concept relies on satisfactory bonding between old and fresh composite layers in order to provide the best adaptation of the repaired composite to the old one.[6] Improvement of the bond strength between new and old composite usually requires increased surface roughness, to promote mechanical interlocking and the coating of old composite with unfilled resin bonding agents to advance surface wetting and chemical bonding.[7] The purposes of surface conditioning of an aged resin composite are removing the superficial layer to expose a clean composite surface with high energy and increasing irregularities of the surface.[8] In many cases, the composition of the restoration to be repaired is unknown; therefore the dependency of the microstructure and the composition of the repair material should be taken into consideration.[9] Likewise, the hydrophilicity of the resin-based adhesive systems for bonding may impair durability of the interfacial bond repair, since more hydrophilic adhesives tend to absorb more water over time.[10] Due to the limited amount of residual free-radicals available for reacting with new resin monomers on the old, contaminated, highly cross-linked resin matrix of composite substrate,[9] different chemical and micromechanical conditioning techniques, hydrofluoric and phosphoric acid etching, air abrasion and sandblasting with/without silane coupling agents or adhesive resin systems.[8,11-15] If adequate bonding between the old resin-based composite and freshly added bonding resin could be achieved, the repair of the existed old restorations becomes an attractive solution.[16]

The purpose of this study was to evaluate repair quality of different types of resin composites after partial removal of pre-existing resin composite restorations using different: (1) Surface finishing methods, and (2) adhesive systems with a microhybrid-resin composite using quantitative microleakage assessment. The null hypothesis was that the weakest zone of the repaired restoration complex is the interface between the existing and the new resin composite in terms of microleakage regardless of the conditioning methods.

**MATERIALS AND METHODS**

This study was approved by Baskent University Institutional Review Board (Project no: D-DA14/05) and supported by Baskent University Research Found. Eighty caries free and intact, anonymized human molars were collected and stored in distilled water. Test protocol of the study is schematically presented in Figure 1. Teeth were cleaned and polished with pumice and rubber cups for 10 s. Occlusal cavities were prepared on each tooth by a high-speed handpiece under water-cooling. A new bur (835R-012-4 ML, Diatech, Coltene/Whaledent AG, Switzerland) was used for every 5 teeth. The bucco-lingual width of the cavities was one-third of the intercuspal width, and the cavity depth was 3 mm. The cavities were restored with either of the universal resin composite
after application of a two-step etch-and-rinse adhesive system (Adper Single Bond 2): A. Grandio SO (Shade: A3) (Voco GmbH, Cuxhaven, Germany), or B. Filtek Z250 (Shade: D3) (3M ESPE, Seefeld, Germany). Resin composite was placed with incremental technique (2 mm-thick layers), adapted to the cavity walls with a flat faced or elliptical condenser and light cured using a halogen light of 500 mW/mm² intensity (Hi-Lux Ultra, Benlioglu, Turkey).

Then, they were finished with finishing burs (379-023-F, Diatech, Coltene/Whaledent AG, Switzerland) and rubber cups. A new bur was employed for each 5 teeth. The restorations were stored in distilled water for 24 h after, which, thermal cycling in deionized water was performed at 5°C ± 2°C to 55°C ± 2°C for 1000 cycles with a dwell time of 30 s and a transfer time of 10 s. Mesial and distal 1/3 parts of the resin composite restorations were removed, leaving only the middle 1/3 of the resin with a high-speed hand-piece under adequate water-cooling. One side of the cavity was finished with a coarse diamond bur (837R-012-8-ML, Diatech) while the other part of the cavity was air abraded with 50 μm Al₂O₃ particles (ProphyFLEX2, KaVo, Germany). Similarly, a new bur was employed for each 10 teeth. The samples were then randomly divided into 4 groups (n = 10/group) to receive the following adhesive systems [Table 1]:

- **Group 1:** AllBond 3 (Bisco, IL, USA) (dual-cure, etch and rinse, three-step, 2-hydroxyethyl methacrylate (HEMA)-free, ethanol-based, hydrophobic adhesive system)

- **Group 2:** Clearfil SE Bond (Kuraray, Okayama, Japan)

- **Group 3:** Beauty bond (Bisco, CA, USA) (single-cure, all-in-one, hydrophobic adhesive system)

**Table 1: Material descriptions and manufacturers of the materials used in the study**

| Material description | Material | Chemical composition | Manufacturer | Procedures |
|----------------------|----------|----------------------|--------------|------------|
| Self-etch, Two step Adper Single Bond 2 | Clearfil SE bond | Primer: HEMA (%25-45), MDP, hydrophilic aliphatic dimethacrylate, water, initiators, accelerators, dyes | Kuraray, Okayama, Japan | Apply primer and leave for 20 s |
| Water-based Light-cured Adhesive system | All bond3 | Ethanol (part A) >50, NTG-GMA salt (part A) >1 | Bisco, IL, USA | Etch for 15 s and rinse thoroughly |
| Etch and rinse Three-step HEMA-free Ethanol based Dual-cured hydrophobic Adhesive system | Adper single bond 2 | Bis-GMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids 10% w/w 5 nm diameter silica filler | 3M, ESPE, Seefeld, Germany | Etch enamel and dentin for 15 s |
| Etch and rinse Two-step Water-based Light-cured Adhesive system | Beauty bond | Bis-GMA, TEGDMA, phosphonic acid monomer, carboxylic acid monomer, acetone, water | Shofu, CA, USA | Apply bond, leave undisturbed for 10 s |
| All-in-one Self-etch HEMA free Light-cured Adhesive system | Grandio SO | Bis-GMA 2.5-5%, TEGDMA 2.5-5%, 89% w/w inorganic fillers | Voco GmbH, Cuxhaven, Germany | Apply the selected shade in layers that are a maximum of 2 mm thick Light-cure afterwards |
| Universal nanohybrid restorative | Filtek™ Z250 | TEGDMA, UDMA, Bis-EMA | 3M, ESPE, Seefeld, Germany | Place in increments <2.5 mm |
| Universal microhybrid restorative | UDMA, Bis-EMA | 0.01 μm to 3.5 μm with an average particle size of 0.6 μm zirconia/silica | Light cure each increment for 20 s |

HEMA: 2-hydroxyethyl methacrylate, TEGDMA: Triethylenglycol dimethacrylate, Bis-GMA: Bisphenol a glycidyl methacrylate, MDP: Methacryloyloxydecol dihydrogen phosphate, UDMA: Urethane dimethacrylate, Bis-EMA: Ethoxylated bisphenol A-methacrylate, NTG-GMA: N-tolyglycine-glycidyl methacrylate.
All of the restorations were repaired with Filtek Z250 resin composite (Shade: C2) and light-cured as mentioned above. They were all finished and polished like the previous restorations. The specimens were again thermocycled as previously mentioned (1000×) and immersed in 0.5% basic fuchsin solution (Wako Pure Chemical Industry, Japan) for 24 h. After a thorough rinsing with distilled water, the samples were air dried and embedded in epoxy resin (Struers, Copenhagen, Denmark). The specimens were sectioned mesiodistally with a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL) and digitally photographed at 20× magnification (1280 × 1024 resolution) under a stereo-microscope (Olympus, Tokyo, Japan). Photographs were saved as TIFF images and processed with a MacBook device. The extent of dye penetration was measured (in mm) using an open-source image analysis software (ImageJ, V.1.42, National Institutes of Health, Bethesda, MD) for both the bur-finished and air-abraded surfaces at both the resin-tooth and resin-repair interfaces.

The data were statistically analyzed with Krukal–Wallis, Mann–Whitney U with Bon Ferroni correction and Wilcoxon Signed Rank Tests by Statistical Package for Social Sciences (SPSS) 11.5 software (SPSS Inc., Chicago, IL, United States).

**RESULTS**

A total of 160 sections were examined for the quantitative evaluation of dye penetration. Table 2 presents the descriptive statistics of the dye penetration of both groups, with respect to different regions of the cavities and the adhesive systems.

The Kruskal–Wallis test revealed that the difference between the experimental regions (resin-tooth or resin-resin interface) with respect to adhesive systems was statistically significant ($P < 0.05$). Multiple comparisons with the Mann–Whitney U-test with Bon Ferroni correction revealed that only BeautiBond showed statistically significant more microleakage when compared with other adhesive systems at every site of the cavity, irrespective of the utilized resin composite type ($P < 0.0166$) [Table 3]. Figures 2 and 3 show representative Grandio or Z250 restorations repaired with BeautiBond adhesive system. Figure 4 presents a sealed resin-resin and tooth-resin interface after repairing.

The Mann–Whitney U-test with Bon Ferroni correction analyzed the differences between the effects of pre-existing resin composite types. The results revealed no statistically significant differences between each resin composite for all adhesive system types at every region of the cavity ($P > 0.048$).

Table 4 demonstrates statistically compared $P$ values when evaluating the effect of surface finishing type (air abrasion or bur-finishing) with respect to the regions of the cavity (tooth or repair site) when used with different adhesive systems and resin composites. The Wilcoxon Signed-Ranks Test revealed that surface finishing with air abrasion exhibited statistically less microleakage then bur-finishing irrespective of the type of pre-existing resin composite, cavity site (tooth or repair site), or the adhesive systems except BeautiBond ($P < 0.05$).

The effect of the surface finishing type on the repair site and the tooth site with respect to the pre-existing composite and the adhesive systems is evaluated.

| Restorative material | Adhesive system | $n$ | Air abrasion/tooth | Air abrasion/repair | Bur/tooth | Bur/repair |
|---------------------|-----------------|-----|---------------------|--------------------|-----------|-----------|
| Grandio SO          | All bond 3      | 20  | 0.151±0.020       | 0.056±0.006       | 0.180±0.016 | 0.082±0.004 |
|                     | CLSE            | 20  | 0.132±0.020       | 0.051±0.005       | 0.168±0.019 | 0.078±0.005 |
|                     | Single bond 2   | 20  | 0.148±0.017       | 0.055±0.005       | 0.171±0.014 | 0.080±0.004 |
|                     | Beauty          | 20  | 1.477±0.252       | 0.203±0.033       | 1.508±0.180 | 0.212±0.017 |
| Filtek Z250         | All bond 3      | 20  | 0.159±0.028       | 0.048±0.009       | 0.186±0.009 | 0.083±0.005 |
|                     | CLSE            | 20  | 0.137±0.019       | 0.053±0.005       | 0.169±0.016 | 0.079±0.006 |
|                     | Single          | 20  | 0.143±0.020       | 0.052±0.003       | 0.166±0.022 | 0.076±0.006 |
|                     | Beauty          | 20  | 1.574±0.191       | 0.212±0.032       | 1.641±0.171 | 0.217±0.021 |

SD: Standard deviation
The results of this study showed that air abrasion of the surface of the composites tested produced the lowest microleakage, independent of the adhesive systems and the pre-existing resin composite type. Besides, microleakage values at the repair site were found to be less than resin-tooth site. Thus the null hypothesis was rejected.

Table 3: P of the multiple comparisons of the adhesive systems by Mann-Whitney U-test with Bon Ferroni correction evaluating the effect of the adhesive systems with respect to the surface finishing type and the cavity site of each preexisting composite type

| Compared adhesive systems | P          |  |  |  |  |
|---------------------------|------------|---|---|---|---|
|                           | Air-abrasion/ | Air-abrasion/ | Bur/ | Bur/ |
|                           | tooth repair | repair tooth | repair | repair |
| Grandio SO                | 0.069       | 0.53         | 0.197 | 0.049 |
| All Bond-CLSE             | 0.544       | 0.425        | 0.139 | 0.361 |
| All Bond-single           | 0.000*      | 0.000*       | 0.000*| 0.000*|
| All Bond-beauty           | 0.173       | 0.343        | 0.704 | 0.343 |
| CLSE-single               | 0.000*      | 0.000*       | 0.000*| 0.000*|
| CLSE-beauty               | 0.000*      | 0.000*       | 0.000*| 0.000*|
| Single-beauty             | 0.090       | 0.149        | 0.195 | 0.050 |
| Filtek Z250               | 0.049       | 0.223        | 0.040 | 0.150 |
| All Bond-CLSE             | 0.472       | 0.270        | 0.095 | 0.028 |
| All Bond-single           | 0.000*      | 0.000*       | 0.000*| 0.000*|
| All Bond-beauty           | 0.791       | 0.909        | 0.879 | 0.272 |
| CLSE-single               | 0.000*      | 0.000*       | 0.000*| 0.000*|
| CLSE-beauty               | 0.000*      | 0.000*       | 0.000*| 0.000*|
| Single-beauty             | 0.000*      | 0.000*       | 0.000*| 0.000*|

*Statistical significance (P<0.0166)

Table 4: P values of the multiple comparisons evaluating the effect of surface finishing type (air-abrasion or bur-finishing) with respect to the regions of the cavity (tooth or repair site) when used with different adhesive systems and resin composites

| Restorative material | Adhesive system | n | P       |  |  |
|---------------------|-----------------|---|---------|---|---|
|                     |                 |   | Air abrasion/ | Air abrasion/ |
|                     |                 |   | tooth         | repair         |
|                     |                 |   | Bur/tooth     | Bur/repair     |
| Grandio SO          | All bond 3      | 20| 0.009*      | 0.005*        |
|                     | CLSE            | 20| 0.022*      | 0.005*        |
|                     | Single          | 20| 0.009*      | 0.005*        |
|                     | Beauty          | 20| 0.114       | 0.283         |
| Filtek Z250         | All bond 3      | 20| 0.037*      | 0.005*        |
|                     | CLSE            | 20| 0.022*      | 0.005*        |
|                     | Single          | 20| 0.047*      | 0.005*        |
|                     | Beauty          | 20| 0.241       | 0.444         |

*Statistical significance (P<0.05)

DISCUSSION

The results of this study showed that air abrasion of the surface of the composites tested produced the lowest microleakage, independent of the adhesive systems and the pre-existing resin composite type. Besides, microleakage values at the repair site were found to be less than resin-tooth site. Thus the null hypothesis was rejected.

Treatment strategies of resin composite repair have some potential problems, as the adhesion of two different resin composite types and the microstructure of the preexisting composite, which is not usually determined before the procedure. Adhesion between two composite layers is achieved in the presence of an oxygen-inhibited layer of unpolymerized resin.[17] Although, pre-polymerized or aged resin restorations contains no or less unreacted monomers on the surface layer and the resin-resin interface was supposed to be the weakest zone of the repaired restoration, the results of the current study revealed that this was not the case. In addition, in many cases, dissimilar resin composite materials were used. However, the restoration repair may be an acceptable treatment option with minimal stress on the pulp since the clinician removes and restores only the defective portion of the restoration.[5,18,19]

Micromechanical interlocking is the basis of the composite repair process.[8,13] Thus, the clinicians should attempt to increase the surface area of the pre-existing restoration prior to the repair procedure.[12] Furthermore, the application of a bonding agent as an intermediate agent is advisable to enhance substrate wetting.[20] Thus, it seems reasonable to suggest a surface treatment technique and an efficient adhesive system to optimize the repair procedure.[21]

Likewise, surface roughness is a common property known to improve the general adhesion potential of the material by promoting micromechanical retention between different composites.[22] The results of the current study emphasize that surface finishing with air abrasion exhibited statistically less microleakage then bur-finishing irrespective of the type of pre-existing resin composite, cavity site (tooth or repair site), or adhesive systems, except BeautiBond. Earlier studies showed that aluminum oxide sandblasting is able to produce more micro retentive features, increasing the surface area available for wetting and bonding by adhesive resin.[8,9,12] However, most previous studies investigated the interfacial bond strength of the repair system, and divergent results have been reported with the use of diamond burs for preparing composite surfaces for bonding.[9,14] On the other hand, a more even surface topography was found using aluminum by the Wilcoxon Signed-Rank test at a significance of 0.005. The multiple comparisons revealed that all of the adhesive systems produced statistically significant less microleakage at the repair site than the tooth region irrespective of the surface finishing type (bur-finishing or air abrasion) and the pre-existing resin composite (P = 0.05 for each multiple comparison).
oxide sandblast and silica coating in comparison with the bur abrasion, resulting in greater mean bond strength values and suggesting a more effective pattern for mechanical retention.\cite{12,23,24-26}

In the repair process, the chemical structure of the adhesive resin may have an important role than the fresh resin composite.\cite{22} Most of the dental adhesives contain HEMA which is an effective hydrophilic methacrylate monomer that plays a role in the wetting enhancement and diffusion promoter of co-monomers in the formation of the hybrid layer.\cite{24-26} High HEMA contents promote water uptake and subsequent hydrolytic degradation of the polymers, swelling and staining leading to accelerated microleakage.\cite{27} Thus, the aforementioned disadvantages of the HEMA led the researches to seek for less hydrophilic adhesives that compensates up for the degradation issue. Besides, all-in-one adhesives are may act as semipermeable membranes, permitting water movement through the layer even after polymerization.\cite{28}

The results of the current study demonstrated that hydrophilicity of the intermediate agent did not affect the microleakage of the repaired resin composite restorations at every site of the cavity irrespective of the pre-existing composite type and surface finishing time. The HEMA free 3-step etch and rinse adhesive system (AllBond3), and HEMA containing two step self-etching system (Clearfil SE Bond) and HEMA containing two-step etch-and-rinse adhesive system (Single Bond2) presented statistically similar microleakage values. And BeautiBond revealed the highest statistically significant microleakage values although it is HEMA free.

It should also be kept in mind that a higher concentration of solvent is necessary to omit HEMA from the adhesive. Therefore, air-drying has a significant role on the removal of solvents included in all-in-one adhesives. When HEMA-free self-etching adhesives are not air dried strong and long enough to remove droplets, which result by the phase separation between water and other adhesive ingredients, this will result in lower polymerization and mechanical properties.\cite{29,30}

The similar results between etch and rinse and two-step self-etching systems may be attributed to the finding that acidic primer of the self-etching system was able to promote an adequate surface cleansing.\cite{31} Previous bond strength studies show that the phosphoric acid cleansing of the surface to be repaired demonstrated no significant influence on the bond strength of the repair mechanism.\cite{8,9,13} Nevertheless, this issue requires further research.

To the authors’ knowledge, only one research exists that investigates the effect of hydrophilic adhesive systems on the repair strength of resin composites revealing that hydrophilicity is not an effective

[Figure 2: Grandio restoration repaired with BeautiBond adhesive system]

[Figure 3: Z250 restoration repaired with BeautiBond adhesive system]

[Figure 4: A sealed resin-resin and tooth-resin interface after repairing]
parameter on the repair bond strength.\[12\] Although the specimens were thermocycled twice before and after the repair process, the long-term performance of hydrophilic adhesive systems at the resin-resin or resin-tooth interfaces apart from the current data may be questionable as adhesive interfaces absorb water after long-term water storage, and its amount is positively correlated with hydrophilicity of the adhesive system.\[10,32\] Thus, hydrophobic adhesives may be expected to be more durable than current formulas in the market.\[21\]

When a resin composite restoration fails, it is not always possible to find out which composite material was previously used. In such situations, since the clinician cannot know which material was previously used, often, dissimilar composite materials adhere to each other during repair. The results revealed that the pre-existing resin composite type did not statistically affect the microleakage irrespective of the adhesive system or the surface finishing type. Likewise, the design of the current research aims to simulate clinical conditions in which the practitioner is not able to clinically identify the substrate resin composite’s matrix chemistry to be repaired by using two different resin composite types.

Microleakage tests are useful methods for evaluating the sealing performance of adhesive systems. Among different methods employed, dye penetration measurement on sections of restored teeth is the most commonly used technique. Thus, dye penetration technique may present an easy and fast laboratory test to compare the predicted performance of dental materials in lack of adequate clinical data.\[33-35\] The present study performed image analysis to obtain quantitative results instead of a conventional subjective scoring. The relative merit of this objective approach, when compared with a subjective scoring system, was to discard the need for scoring by separate evaluators and for consensus scoring in borderline cases, as well as to reduce the need of statistical procedures regarding inter-examiner reliability.\[36\]

This research measured both halves of the specimens for dye penetration with 40 specimens and 80 sections. This would have almost doubled the number of mutually exclusive specimens in the sample, and the statistical analysis was improved to emphasise the differences. However, the quantitative Image J analysis showed that the two sections for the same tooth demonstrated different microleakage values. The first intention was to investigate only one section, but after realizing that the two sections show different extents of dye penetration, it was decided to include both sections. This difference might be attributed to the thickness of the low-speed blade.\[21\]

**CONCLUSIONS**

Within the limitations of our study, the treatments based on air abrasion of the surface of the composites tested produced the lowest microleakage scores, independent of the adhesive systems and the pre-existing resin composite type.

The pre-existing resin composite type may not affect the microleakage of the repaired restoration independent of the adhesive system and the surface finishing type.

BeautiBond adhesive may not be preferred in resin composite repair in terms of microleakage prevention.

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