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Original Article

Shear bond strength of composite to demineralized enamel conditioned with resin infiltration

Sebahat Melike Durukan[1], Burak Gümüştaş[2], Soner Şişmanoğlu[1,2]

[1] Altınbaş University, Faculty of Dentistry, Department of Restorative Dentistry, İstanbul, Turkey
[2] İstanbul University-Cerrahpaşa, Faculty of Dentistry, Department of Restorative Dentistry, İstanbul, Turkey

Correspondence:

Dr. Sebahat Melike DURUKAN
Altınbaş University, Faculty of Dentistry, Department of Restorative Dentistry, İstanbul, Turkey
E-mail: mlk.drknn@gmail.com

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Abstract

Aim: The aim of this in vitro study was to evaluate the influence of resin infiltration on bond-strength of composite resin to demineralized enamel.
Methodology: Thirty bovine incisors were used in this study. Buccal enamel surfaces of bovine incisors were wet polished and then were divided into three groups: sound enamel; demineralized enamel; demineralized enamel infiltrated with a low-viscosity resin (ICON, DMG, Hamburg, Germany). After acid-etching with 37% phosphoric acid for 20 seconds, a two-step, total-etch adhesive (Single Bond 2, 3M ESPE, St. Paul, MN, USA) was applied using a microbrush for 20 seconds, followed by gentle air-drying for 5 seconds. The adhesive was light-cured for 10 seconds. Following the adhesive application, flowable composite resin (Filtek Supreme Flowable, 3M ESPE, St. Paul, MN, USA) was gently placed into a microtubule and was photopolymerized using an LED curing unit (Elipar Deep Cure; 3M ESPE, St. Paul, MN, USA). The microshear bond strength (μSBS) tests were performed using a microshear testing machine at a cross-head speed of 0.5 mm/min. One-way ANOVA and Bonferroni tests were used to analyze the data (5%).
Results: Significant differences were found according to the ANOVA ($p < 0.05$). Pair-wise comparison results of μSBS (mean ± SD) were: sound enamel (25.16 ± 2.3); demineralized enamel (17.93 ± 2.1); demineralized enamel infiltrated with a low-viscosity resin (28.51 ± 3.76).
Conclusion: Resin infiltration applied to demineralized enamel before composite application increased the bond strength. No difference was found in the bond strength values obtained for sound enamel and resin infiltrated enamel.

Keywords: Enamel, bond strength, resin infiltration, demineralization, remineralization, composite resin

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Introduction

Dental caries is a progressive disease caused by a combination of bacteria, diet, and host-related factors (1). Under the influence of this combination, a cycle of remineralization and demineralization is constantly in progress on the surface of the tooth. A caries lesion is triggered by an imbalance in the demineralization and remineralization cycle, as well as a shift in the equilibrium (2). Initial enamel demineralization causes submicroscopic alterations, such as mineral loss from the lesion body, an increase in intercrystalline space, and a decrease in subsurface microhardness, although the tooth surface is still relatively strongly mineralized.

These changes in the porous enamel structure give a visible, distinct, chalky appearance, and further mineral removal through these mineral diffusion pathways is facilitated by the bacterial acid produced (3, 4).

Alterations at the submicroscopic and microscopic levels of the enamel surface become visible to the naked eye with the continuation of demineralization process. The initial whitish caries lesions that occur after tooth eruption are called white spot lesions. The majority of the enamel shows mineral loss in the early stages of white spot lesions, although the lesion surface is mostly unaffected. Transparency loss may occur over time when the lesion's surface layer becomes partly transparent (5). However, despite these changes, cavitation is not observed in the white spot lesion, and the caries is limited to the enamel tissue.

Saliva may contribute to the regression of initial caries lesions as a result of improvement in the patient's diet and oral hygiene habits. However, the effect of saliva is often not sufficient, and remineralization must be supported externally with remineralization therapies. Fluoride is an alternate therapy for remineralizing enamel since it increases enamel acid resistance and interferes with bacterial metabolism and enzymatic activity (6). Remineralization therapy can lead to regression or even healing of the initial enamel caries (2–4).

Although the progression of white spot lesions can be stopped by both saliva and externally applied remineralization therapies, sufficient success cannot be achieved in advanced WSL cases. Additionally, this whitish appearance in the surface of the anterior teeth may conflict with patients' aesthetic expectations and reduce their self-confidence. Traditionally, in these cases, the caries is removed by various methods and the cavitation is treated with invasive restorative procedures. Early enamel caries lesions have traditionally been controlled by less intrusive procedures (6, 7). In order to stop early enamel lesions and to fill the intercrystalline spaces, a resin infiltration procedure involving filling these spaces with a low-viscosity resin has been used (8). In contrast to composite fillings, the resin infiltration forms a diffusion barrier within the enamel lesion, strengthening the demineralized enamel structure with the resin matrix and limiting the development of new cavities (8, 9). Resin infiltration therapy for enamel lesions is a potential strategy to arrest lesion progression. The progression of demineralized enamel lesions has been halted or at least slowed using the low viscosity light-cured resin infiltration technique (10, 11). Furthermore, long-term research has demonstrated that a resin infiltrant with a low viscosity and a high penetration coefficient can completely penetrate demineralized enamel surfaces and early caries lesions.

Development on caries infiltration technologies has gained importance in recent years (12). However, limited information is available on the combined applications of resin infiltrants with conventional restorative materials and their bond strength to composite resins.

Therefore, this in vitro study aimed to investigate the influence of resin infiltration on the shear bond strength of composite resin to demineralized enamel treated with resin infiltration. The null hypothesis was that resin infiltration would not affect the bond strength.

Materials and Methods

Sample Preparation:

Thirty bovine incisors were used in the present study. Until the start of experiment, the samples were stored in water containing chloramine T at 4°C, and the solution was refreshed every week. The roots of all teeth were cut 2 mm above the dentin-enamel junction using a precision cutting device (IsoMet High Speed Pro; Buechler, Lake Bluff, IL, USA). The tooth fragments were embedded in cold-cure acrylic blocks with the enamel surface facing upwards and parallel to the block surface (n = 30). The buccal enamel surfaces of the bovine incisors were wet polished using silicon carbide abrasives with 600-, 800-, 1200-, 2500-, and 4000-grit under water. The specimens were then divided into three groups (n = 10): Group 1, sound enamel; Group 2, demineralized enamel; and Group 3, demineralized enamel infiltrated with a low-viscosity resin (Icon; DMG, Hamburg, Germany).

The demineralizing solution was made using distilled water, 2 mM Ca [$Ca(NO_3)_2$], 2 mM PO_4 (KH_2PO_4), and 75 mM acetate at 4.3 pH, as described by Ten Cate and Duijsters (13). For 72 hours, each sample was immersed in a 25 ml disposable plastic bottle containing 20 ml of a demineralizing solution. After removing the samples from the demineralization solution, they were washed with deionized water. The samples were viewed under a stereomicroscope to see if the surface had undergone a whitish change closely resembling opacity before the experiment began. Before and during the experimental tests, specimens were maintained in artificial saliva (distilled water, 1 mM $CaCl_2$, 50 mM KCl, 2 mM KH_2PO_4, and 0.01% NaN_3, with the pH adjusted to 7 with 1 M KOH) in the amount of 40 ml per tooth.

For the specimens in Group 1, no demineralization was applied, and specimens were stored in artificial saliva.

Resin infiltrant and restorative procedures:

The resin infiltrant (ICON) was applied to the demineralized enamel surface using the supplied agents with the resin infiltrant in accordance with the manufacturer's instructions for use (Table 1). The samples were treated with Icon-Etch (15% hydrochloric acid) for two minutes, followed by a water rinse and 30 seconds of air drying; Icon-Dry (ethanol) was applied for 30 seconds; Icon-Infiltrant was applied twice, once for 3 minutes and once for 1 minute. Both applications were light-cured with an LED curing unit (Elipar Deep Cure; 3M ESPE, St. Paul, MN, USA) for 40 seconds with an output of 1250 mW/cm^2.

Statistical analysis

Statistical analysis of the data obtained in our study was performed using the SPSS 21.0 program (IBM SPSS Inc., Armonk, NY, USA). Data (in MPa) were subjected to one-way ANOVA and Bonferroni tests. A *p-value* < 0.05 was considered statistically significant.

Results

The μSBS values of the study groups are shown in Figure 1. Group 3, which consists of resin infiltrated specimens, had an μSBS value that was noticeably higher (28.51 ± 3.76) than the other groups. The lowest bond strength values were observed for Group 2. The μSBS values (mean ± SD) were found as follows: sound enamel (25.16 ± 2.3) and demineralized enamel (17.93 ± 2.1).

In comparison to the demineralized (Group 2) samples with no resin infiltration, the bond strength of the enamel surfaces with resin infiltration had significantly higher values ($p < 0.05$). Additionally, there was no statistically significant difference between the group with resin infiltrated surfaces and the group with sound enamel surfaces (Group 1) ($p > 0.05$).

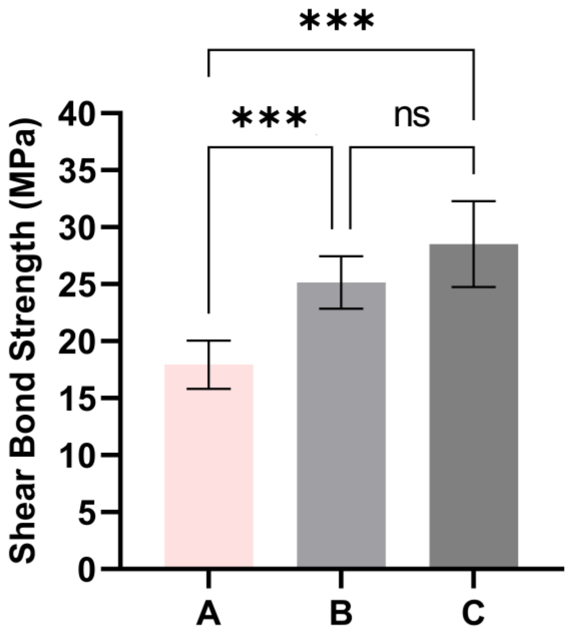

Figure 1. Comparative graph of the study groups: (A) Demineralized enamel (Group 2). (B) Sound enamel (Group 1). (C) Demineralized enamel with resin infiltration (Group 3). ***, $p < 0.001$; ns, not significant.

Table 1. ICON resin infiltrant application steps.

Resin infiltrant application steps	
Step 1	Icon-Etch (15% hydrochloric acid) applied for 2 minutes
Step 2	Water rinse; air dried for 30 seconds
Step 3	Icon-Dry (ethanol) applied on surface for 30 seconds
Step 4	Icon-Infiltrant (TEGDMA based) (applied twice): once for three minutes and once for one minute
Step 5	After both applications, surfaces were light-cured with an LED curing unit

Table 2. Composition of resin infiltrant, composite resin and adhesive systems.

Product	Composition	Batch Number	Manufacturer
ICON	Icon Etch: hydrochloric acid, pyrogenic silicic acid, surface active substances Icon Dry: %99 ethanol Icon Infiltrant: TEGDMA based resin matrix, initiators, additives	635703 633314 633139	DMG, Hamburg, Germany

Adper Single Bond 2	Bis-GMA, HEMA, dimethacrylates, ethanol, water, photoinitiator, polyalkenoic acid copolymer, 10vol% of 5 nm silica nanofiller	N508311	3M ESPE, St Paul, MN, USA
Filtek Supreme Flowable Composite	Matrix: Bis-GMA, Procrylat resins, TEGDMA Filler: YbF3 filler (0.1–5.0 µm), non-agglomerated/non-aggregated silica (20 nm, 75 nm), aggregated zirconia (4–11 nm) and silica (20 nm) cluster filler (average cluster particle size: 0.6–10 µm)	A2NA71771	3M ESPE, St Paul, MN, USA

[TEGDMA=triethylenglycol dimethacrylate, Bis-GMA=bisphenol-A-dimethacrylate, HEMA= 2- hydroxyethyl methacrylate]. Formulations according to the respective material safety data sheet and the literature (12,14).

Discussion

The aim of this research is to compare the µSBS of demineralized enamel treated with resin infiltrate with that of healthy and un-infiltrated enamel surfaces. The results show that the resin infiltration treatment of the demineralized enamel had no significant effect when compared to the sound enamel in terms of µSBS values. However, significantly higher µSBS values were observed in comparison to demineralized enamel with no resin infiltrate application ($p < 0.05$). Therefore, the null hypothesis that resin infiltration would not affect the bond strength was accepted. The null hypothesis of the study was rejected.

Collecting sound human teeth for in vitro testing of adhesive systems is becoming increasingly difficult because indicated extractions are decreasing significantly, which is among the main reasons for using bovine enamel in our research. As a result, several scientists use bovine teeth in place of human teeth to assess bond strength (15–17).

Researchers found that when compared to demineralized enamel that had not been infiltrated with resin, demineralized enamel treated with resin infiltrate did not degrade within the adhesive attachment area between the tissue and composite filling. On the other hand, it was found that the adhesive (Assure PLUS) employed in the study greatly enhanced the bond strength while having no effect on the other adhesive systems under investigation (18). According to the findings of the current study, variations in the monomer content of Assure PLUS and other adhesive systems (Scotchbond, Transbond XT Primer) are a significant contributor to the disparities in bond strengths found in resin-infiltrated groups. To be more precise, researchers used Assure PLUS and Scotchbond adhesive solutions, which are composed of MDP (10-Methacryloyloxydecyl dihydrogen phosphate) monomer and allow chemical bonding to dental tissues through ionic bonding to calcium in hydroxyapatite (19). Furthermore, the presence of ethanol improves the bond strength to dentin (20). Because the adherent in the current report was a resin base (ICON), the presence of MDP and ethanol is not viewed as a benefit of Assure PLUS and Scotchbond over Transbond XT.

According to previous research, preconditioning with a resin infiltrant improved the SBS of several bonding systems on simulated enamel lesions. This is likely because the infiltrant penetrated the lesion's body more deeply than primer or paste did (21). Monomer formulations produce a thick oxygen-inhibited layer with an improved triethylene glycol dimethacrylate (TEGDMA), such as ICON, which has a high penetrating capability (22) and likely results in the chemical attachment of the resin infiltrant to the primer's monomers (21). Research from the past has shown that high levels of TEGDMA and ethanol in resin infiltrants improve penetration by decreasing viscosity and contact angle to enamel, although a high BisGMA content reduced resin infiltrant penetration (8). On the contrary, it was demonstrated that adding more TEGDMA to BisGMA/TEGDMA composites increased polymerization shrinkage and polymerization stress, which may have a negative impact on bond strength (23). It was considered that the shrinkage of the polymer during polymerization was related to oxygen inhibition or incomplete solvent evaporation. The current findings support past studies (19, 20).

Tensile bond strength studies were used by the researcher to examine adhesion. Low-viscosity resin-infiltrated groups had similar values to sound enamel, which was adhered using Adper Easy Bond and/or Single Bond (1). According to the findings of the study, scientists believe that the affinity between the monomers present in the infiltrant and the monomers of the adhesive systems has been optimized, resulting in satisfactory µTBS values associated with infiltrated groups (24). Because resin infiltration does not negatively affect the composite's ability to adhere to enamel, restorative treatment can be recommended on tooth surfaces that have undergone resin infiltration, according to the findings of the current and prior studies. This is due to the stability of resin penetration with both total-etch and self-etch adhesives. In addition, previous studies have found that using an etch-and-rinse adhesive during resin penetration had no effect on the enamel µTBS (25).

Throughout the investigations, various adhesive systems were used on the samples. The existing study groups' data on bond strength (resin infiltration of intact enamel and demineralized enamel) are similar to our findings (12). Contrary to all of this, it has been shown that the self-etch adhesive systems that have been used display weaker bond strength than the etch-and-rinse adhesive systems, based on comparisons of the study groups' (sound enamel, demineralized enamel, resin-infiltrated demineralized enamel). As mentioned in the prior investigations, scientists predicted that the adhesive depth would degrade because the acidic monomers in the self-etch adhesive systems cannot properly abrade the surface (26).

One major limitation of the present investigation is collecting the data from immediate bond strength without using any ageing techniques. Whereas some literature has already called into question the appropriateness of the shear bond strength test for bovine dentin (27), other authors performing a microtensile bond strength test did not report significant differences between human and bovine teeth in this regard (28).

In this context, it was not possible to obtain the desired amount of data. The study has potential for improvement because it is restricted to in vitro circumstances and because necessary ageing techniques for evaluation were not used. Clinical research, studies using comparable methodologies, and studies using a variety of adhesive systems are required to shed more light on the impact of resin penetration on the bond strength of demineralized enamels.

Conclusions

Within the limitations of this in vitro study, it can be concluded that resin infiltrant did not adversely affect bond strength but rather improved it. No difference was found in the bond strength values obtained for sound enamel and resin infiltrated enamel.

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