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Shear bond strength to sound and caries-affected dentin of simplified "etch-and-rinse" and "self-etch" adhesives and the hybrid layer micromorphology

Mikromorfologija hibridnog sloja i otpornost na silu smicanja adhezivne veze formirane između pojednostavljenih potpuno nagrizajućih i samonagrizajućih adheziva sa zdravim i karijesno izmenjenim dentinom

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Abstract

Background/Aim. After removal of caries-infected dentin, a considerable area of the cavity floor comprising caries-affected dentin. Bonding to caries-affected dentin is characterized by lower bound strength and inferior hybrid layer guality compared to bonding to sound dentin. The purpose of study was to compare shear bond strength (SBS) of currently available adhesive systems to sound dentin (SD) and caries-affected dentin (CAD) and elucidate the hybrid layer micromorphology. **Methods.** Sixty extracted human molars with coronal carious lesions formed the experimental sample while additional sixty extracted intact human molars (impacted third molars) served as controls. Identification of a carious-affected dentin was carried out using visual identification (North Carolina Dentin Sclerosis Scale). Teeth from both the experimental and the control sample were allocated to one of the following three groups: Adper Single Bond Plus/Filtek Supreme XT (ASB/FS) (3M ESPE), AdheSE One/Tetric EvoCeram (AO/TEC) (IvoclarVivadent), and Prime&Bond NT/CeramX Mono (PB/CXM) (Dentsply). Bonding procedures utilized in this work were in line with the manufacturers' instructions. The SBS was measured using a universal testing apparatus. Hybrid layer micromorphology was observed under scanning electron microscope (SEM). The mean SBS values (MPa), and hybrid layer thickness (in μm) were statistically analyzed using the *t*-test, Mann-Whitney U-test, ANOVA, and Holm's test. **Results.** Mean SBS ± standard deviation were: ASB/FS to SD = 10.56 ± 3.49; ASB/FS to CAD = 10.06 ± 2.55; AO/TEC to SD = 7.01 ± 2.05; AO/TEC to CAD = 6.73 ± 1.66; PB/CXM to SD = 9.01 ± 2.47; PB/CXM to CAD = 7.83 ± 1.42. A statistically significant difference was found between the bonding strength of ASB/FS and AO/TEC to both SD and CAD, and between ASB/FS and PB/CXM to CAD. Hybrid layer thickness was statistically significantly greater for ASB/FS than for PB/CXM. For the ASB/FS system, a statistically significantly thicker hybrid layer was formed on CAD than on SD. No hybrid layer could be observed for AO/TEC. **Conclusion.** All tested compsite systems bond equally well on sound and caries-affected dentin. The etch-and-rinse adhesives achieved stronger bond strengths. The Adper single Bond Plus-Filtek Supreme XT system formed a statistically significantly thicker hybrid layer on both type 1 of dentin than the Primu 8 Bond NT-CeramX Mono system.

Key words:
dental caries; dentin; dentin bonding agents; adhesives; shear strength; materials testing.

Apstrakt

Uvod/Cilj. Uklanjanjem karijesno inficiranog dentina tokom preparacije kaviteta, značajne površine na njegovim zidovima ostaju pokrivene karijesno izmenjenim dentinom. Povezivanje sa karijesno izmenjenim dentinom karakteriše manja jačina veze i slabiji kvalitet gradnje hibridnog sloja. Cilj rada je poređenje otpornosti adhezivne veze na silu smicanja (SBS) aktuelnih adhezivnih sistema sa zdravim (SD) i karijesno izmenjenim dentinom (CAD), kao i analiza mikromorfologije hibridnog sloja. **Metode.** Šezdeset ekstrahovanih humanih molara sa karijesom okluzalne površine izabrani su kao eksperimentalna grupa, a šezdeset ekstrahovanih intaktnih humanih molara (impaktirani treći molari) izabrani su kao kontrolna grupa. Karijesno izmenjen dentin prepoznat je upotrebom vizuelne identifikacije (North Ca-

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rolina Dentin Sclerosis Scale). Zubi obe grupe podeljeni su u tri podgrupe zavisno od primenjenog kompozitnog sistema: Adper Single Bond Plus/Filtek Supreme XT (ASB/FS) (3M ESPE), AdheSE One/Tetric EvoCeram (AO/TEC) (IvoclarVivadent), Prime&Bond NT/CeramX Mono (PB/CXM) (Dentsply). Adhezivna procedura sprovedena je u skladu sa uputstvima proizvođača. SBS je merena na univerzalnoj kidalici. Mikromorfologija hibridnog sloja analizirana je upotrebom skening elektronskog mikroskopa (SEM). Otpornost adhezivne veze na silu smicanja u srednjim vrednostima (MPa) i debljina hibridnog sloja u μm statistički su analizirani pomoću *t*-testa, Mann-Whitney U-testa, ANOVA i Holmovog testa. **Rezultati.** Srednje vrednosti SBS ± standardna devijacija iznosile su: ASB/FS na SD = 10,56 ± 3,49; ASB/FS na CAD = 10,06 ± 2,55; AO/TEC na SD = 7,01 ± 2,05; AO/TEC na CAD = 6,73 ± 1,66; PB/CXM na SD = 9,01 ± 2,47; PB/CXM na CAD = 7,83 ± 1,42. Statistički značajna razlika uočena je između ASB/FS i AO/TEC na obe forme dentina: na zdravom i na karijesno izmenjenom dentinu, a između ASB/FS i PB/CXM samo na karijesno izmenjenom dentinu. Debljina hibridnog sloja je bila statistički značajno veća za ASB/FS nego za PB/CXM sistem. Kod sistema ASB/FS debljina hibridnog sloja je bila statistički značajno veća na CAD nego na SD. Nije uočeno postojanje hibridnog sloja kod AO/TEC sistema. **Zaključak.** Svi testirani kompozitni sistemi se jednako dobro vezuju sa zdravim i karijesno-izmenjenim dentinom. Tolhesivi sa potpunim nagrizanjem ostvaruju veće vrednosti jačine veze. Adper Single Bond Plus-Filtek Supreme XT system formira statistički značajno tanji hibridni sloj na oba tipa dentina u odnosu na Prime&Bond NT-CeramX Mono sistem.

Ključne reči:
zub, karijes; dentin; dentin, vezivna sredstva; adhesivi; smicanje; materijali, testiranje.

Introduction

After removal of caries-infected dentin, a considerable area of the cavity floor comprising caries-affected (CAD) dentin with partially demineralized collagen remains [1]. Since caries-affected dentin has different mechanical, physical, and chemical properties from those characterizing sound dentin (SD), achieving intimate adaptation of the resin composite and tooth tissue is significantly harder in the former case [2, 3].

For the purpose of implementing composite restoration adhesion to tooth tissue, it is important both for the primer and the resin to penetrate as deeply into demineralized dentin as possible, creating a structure known as a hybrid layer [4]. Hybrid layer quality depends on the adhesive chemical composition and the application technique used as well as the tooth region and presence of caries-affected dentin [5].

Contemporary adhesive systems may be classified as self-etch and etch-and-rinse (total-etch), according to the use of phosphoric acid as a surface etchant. The etch-and-rinse adhesives completely remove the dentin smear layer and smear plugs during acid conditioning. In the next step, resin penetrates into the demineralized zone and provides required adhesion [6]. However, this technique is very sensitive, potentially resulting in contamination due to inconsistencies in executing each step [7].

The self-etch adhesives contain acidic monomers that provide simultaneous conditioning and priming of tooth tissue [8]. They enhance adhesive interdiffusion through the smear layer. Therefore, this method is deemed user friendly (as it requires fewer steps) and less sensitive (as it does not involve wet-bonding) [9]. In terms of their pH, self-etch adhesives can be classified as: (a) 'ultra mild'(pH > 2.5), (b) 'mild'(pH ≈ 2), (c) 'intermediately strong'(1 < pH <2), and (d) 'strong'(pH ≤1) self-etch approach [8].

Adhesives can also be classified according to the number of steps required for their application, where by etch-and-rinse adhesives are divided into 3-step (separate application of acid, primer and adhesive resin) and 2-step (separate application of acid and mixture of primer and adhesive resin). Similarly, self-etching adhesives can be 2-step (separate application of self-etching primer and adhesive resin) and all-in-one (application of self-etching primer and adhesive resin in one solution).

Bonding to CAD is characterized by lower bond strength and inferior hybrid layer quality compared to bonding to SD, irrespective of the type of adhesive systemsemployed [10]. For the etch-and-rinse adhesives, discrepancies between the demineralization level and the extent of resin monomer infiltration have been reported. As conditioning with phosphoric acid cannot completely remove mineral deposits inside dentinal tubules, the resin infiltration depth can be compromised. The aforementioned issues contribute to lower bond strength [1]. The etch-and-rinse adhesives form a thicker hybrid layer on CAD relative to that on SD [11], with a greater prevalence of porous zones [3]. Owing to the complete removal of the smear layer and smear plugs by etch-and-rinse adhesives, a large number of resin tags is produced [6]. Self-etch adhesive systems have also demonstrated lower bond strengths to CAD compared to SD. During the application of self-etch adhesives, demineralization by acidic monomers and adhesive infiltration occur simultaneously. This approach results in fewer discrepancies in the level of demineralization and the resin monomer infiltration. The hybrid layer formed by self-etch adhesives on CAD is usually thicker than that formed on SD [1], with the less pronounced resin tag formation. Only strong self-etch adhesives form the typical resin tags in dentin [8].

The purpose of this study was to compare shear bond strength (SBS) of current adhesive systems to SD and CAD, as well as examine hybrid layer micromorphology. The null hypotheses were: 1) the tested composite systems bond equally well to sound and caries-affected dentin; 2) there is no difference between etch-and-rinse and self-etch adhesives in composite system in bonding to these respective substrates; 3) no important differences in the microstructure of the interfaces between the tested composite systems on respective substrates exist.

Methods

Tooth selection

Sixty caries-free and sixty molars with occlusal carious lesion were collected after obtaining the informed consent from the patients, as approved by the Committee of Ethics of the University of Novi Sad. Upon extraction, the teeth were cleaned with scalers and polished with pumice before being stored in 0.5% aqueous chloramine solution at 4 °C. After the seven-day storage period, the teeth were rinsed and transferred to distilled unionized water at 4 °C and were used within one month from extraction [12].

The caries-affected dentin identification procedure and macroshear bond testing

Using self-curing polyester resin, the roots of thirty molars with occlusal carious lesion (Group A) and thirty caries-free molars (Group B) were individually embedded in polyvinyl chloride (PVC) cylinders of 22 mm diameter and 20 mm height. The tooth crown of every specimen protruded from the cylinder. Enamel of all occlusal surfaces was then removed using a diamond bur (No:806 314110524014 NTI-Kahla, Gmbh, Germany) inserted in a high speed hand-piece under copious air-water spray. The exposed dentin surfaces of each specimen in the Group A were ground with silicon carbide abrasive papers (SiC 600-grit paper,3M) under running water using a custom made grinding cylinder (Figure 1) in order to obtain flat dentin surface perpendicular to the long axis of the tooth/PVC cylinder.

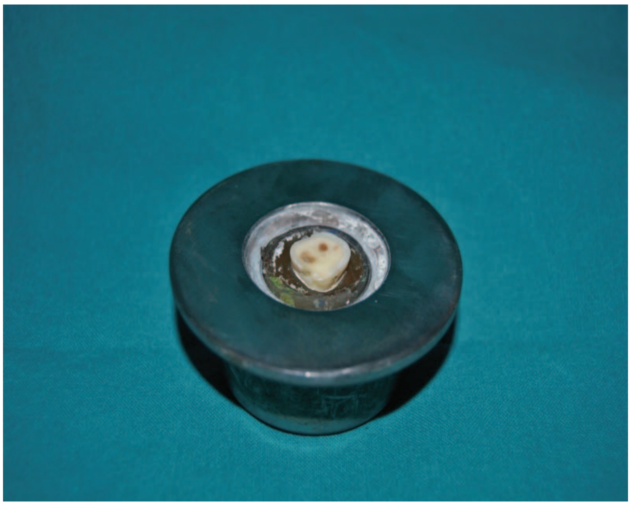

Fig.1 Custom made grinding cylinder.

To obtain caries-affected dentin, we ground the samples forming the Group B using the visual examination criteria set forth by the North Carolina Dentin Sclerosis Scale [13].

In each ground tooth, either SD or CAD was exposed, without revealing the pulp. Caries-affected dentin was identified according to the clinical (visual and tactile) examination guidelines, in accordance with the North Carolina Dentin Sclerosis Scale [13]. Opaque, light yellow, or whitish dentin was classified as sound dentin. Glassy dentin, dark yellow, or slightly brownish was classified as caries-affected dentin (Figure 2).

Fig. 2 – Caries-affected dentin.

The specimens from both groups were divided into three subgroups (n = 10), according to the adhesive/resin composite system used: Subgroup 1 – Adper Single Bond Plus/Filtek Supreme XT (3M-ESPE); Subgroup 2 – AdheSE One/Tetric EvoCeram (Ivoclar-Vivadent); Subgroup 3 – Prime&Bond NT/CeramX Mono (Dentsply).

Chemical composition and manufacturers' instructions for tested adhesives are shown in Table 1.

All implemented bonding procedures followed the manufacturers' instructions and the pertinent test protocol guidelines, based on the ISO/TS 11405 specification of the bonding area limitation [14]. The specimens were placed in a custom made tool–specimen bracket, comprising of a metal cube into which a cylindrical hole corresponding to the specimen dimensions (22 × 20 mm) was bored, and a transparent PVC cylinder of 4 × 4 mm dimensions, for placing the resin composite build-up (Figure 3). Assembling the afore mentioned two components in the described manner allowed forming the resin composite build-up perpendicular to the bonding dentin surfaces. These resin composite structures were layered gradually (in 2 mm increments), using the proprietary restorative resin composite of each adhesive. Each successive composite layer was light-cured using a LED curing device (SmarliteIQ2, Dentsply, Caulk, DE Milford, Serial No. B 21581).

Table 1

Chemical composition and manufacturers' instructions for tested adhesives

Adhesive	Chemical composition	Application
Adper Single Bond 2 (3M-ESPE, St Paul, MN,USA) LOT N172405	BisGMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system, methacrylate functional copolymer of polyacrylic and polyitaconic acids	1. Apply Scotchbond Etchant (35%) to dentin. Wait 15 s and rinse for 10 s. Blot excess water using cotton pellet. 2. Immediately after blotting, apply 2–3 consecutive coats of adhesive for 15 s with gentle agitation using fully saturated applicator. Gently air thin for 5 s to evaporate solvent. Light-polymerize for 10 s.
AdheSE One (Ivoclar-Vivadent, Schaan, Liechtenstein) LOT M50900	Derivatives of bis-acrylamide, water, bis-methacrylamide dihydrogen phosphate, amino acid acrylamide, hydroxy alkyl methacrylamide, silicon dioxide, catalysts, stabilizers	Application and agitation for 30 s, followed by air dispersion until there is no water movement, and finally light-curing for 10 s.
Prime&Bond NT (Dentsply, Caulk, Milford, DE, USA) LOT 0905000886	Di- and trimethacrylate resins, PENTA (dipentaerythritolpenta acrylate monophosphate), nanofillers-amorphous silicon dioxide, photoinitiators, stabilizers, cetylaminehydrofluoride, acetone	Apply Conditioner 36 etch for 15 s. Rinse with water spray for 10s. applay soft blow of air, and ensuring that the surface remains moist. Saturate the surface with ample amounts of the adhesive, reapply if necessary. Leave the surface undisturbed for 20s. Air blow gently for 5s. Light cure for 10s.

Bis-GMA – BisphenolA-glycidyl methacrylate; HEMA – hydroxylethyl methacrylate.

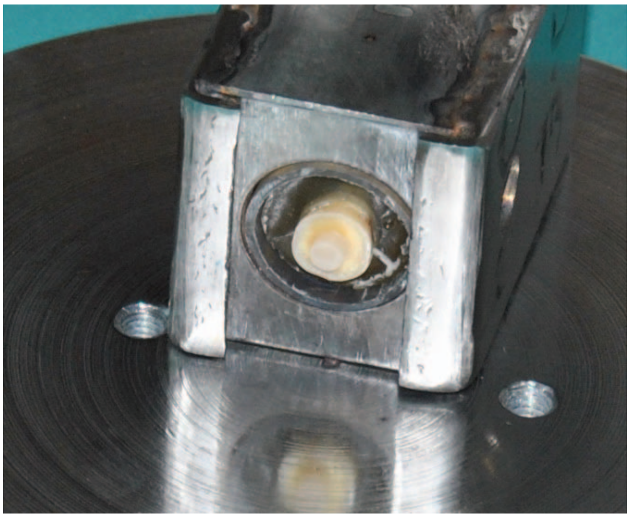

Fig. 3 – Specimen backet.

The prepared specimens were stored in distilled water at 37 °C for 24 h. Each specimen was used to determine the shear bond strength using the universal testing machine (Instron Testing Machine Model 1122, Instron, Norwood, Massachusetts, US). Prior to testing, the specimens were secured in a specimen bracket fixed to the universal testing apparatus. A straight-edge guillotine was positioned as close as possible to the bonding interface and was aligned with the loading axis of the testing construction. A crosshead speed of 1 mm per minute was used. The shear stress was recorded in Newton (N), and the bond strength was calculated using the following equation: P (MPa) = F [N/S (mm^2)] [15, 16].

The shear bond strengths were analyzed using the *t*-test, Mann-Whitney U-test, two-way ANOVA, and Holm's test, available in the Primer of Biostatistics Statistical Software Program. In the two-way ANOVA analysis, the adhesive/composite group and dentin substrate were the tested factors and the level of significance was set to $p < 0.05$.

Scanning electron microscopy (SEM) evaluation

The scanning electron microscopy evaluation was conducted on 60 extracted human molars, divided into two groups according to the previously described criteria. Bonding substrates were prepared using the same procedure as that adopted shear bond strength testing. Once again, bonding procedures were in line with the manufacturers' instructions (Table 1), and composite structures were constructed in bulk, in one 2 mm increments, applying the proprietary restorative resin composite of each adhesive. The teeth were then stored in distilled water at 37 °C for 24 h. The bonded teeth with the composite build-ups were sectioned parallel to the bonded surface to expose the dentin-adhesive interface. The specimens were polished under running water using silicon carbide (SiC) grinding papers of increasingly finer grit (600, 1000 and 1200). For each polished specimen, the CAD area was marked with a #11 scalpel under magnifying glass. The specimens were treated with 32% silica-free phosphoric acid gel (Uni-Etch, Silica gel free, Bisco, Schaumburg, IL, LOT 0800012148) for 60 s, followed by immersion in 2% sodium hypochlorite for 60 s to expose dentin-adhesive interface [17]. After rinsing with distilled water, the specimens were prepared under the Environmental scaning electron microscope (E-SEM) protocol, under the low vacuum and in wet conditions [18]. The specimens were examined by the SEM (JEOL, JSM-6460 Low Vacuum, Tokyo, Japan) at 1000 × magnification [17].

Table 2

The mean shear bond strength (SBS) ± standard deviation (sd) values, presence of statistical significance between the groups

Adhesive	Mean ± sd (min, max) in MPa CAD	Mean ± sd (min, max) in MPa SD	Significance CAD vs. SD
1. ASB-FS	10.06 ± 2.55 (5.09, 12.89)	10.56 ± 3.50 (6.37, 16.51)	non-significant
Significance 1 vs. 2	$p < 0.01$	$p < 0.02$	
2. AO-TEC	6.73 ± 1.66 (4.93, 10.34)	7.00 ± 2.05 (4.61, 11.3)	non-significant
Significance 2 vs. 3	non-significant	non-significant	
3. PB-CXM	7.83 ± 1.42 (5.25, 10.19)	9.0 ± 2.47 (4.46, 12.41)	non-significant
Significance 1 vs. 3	$p < 0.03$	non-significant	

SD – sound dentin; CAD – caries affected dentin; ASB-FS – Adper Single Bond Plus-Filtek Supreme XT.

Results

Shear bond strength (SBS) testing

Descriptive statistics pertaining to the shear bond strengths and the statistical significance of between-group differences are presented in Table 2.

The results yielded by our investigation showed that the adhesive system was a significant factor in determining shear bond strength. Greater bond strengths were achieved using the etch-and-rinse adhesives. The lowest bond strengths were noted for the AdheSE One-Tetric EvoCeram combination on CAD. In addition, the obtained value was statistically significantly lower than that measured for the Adper Single Bond Plus-Filtek Supreme XT combination applied to both CAD ($p < 0.01$) and SD ($p < 0.02$) specimens. Prime&Bond NT-CeramX Mono exhibited statistically significantly lower bond strength on CAD than the Adper Single Bond Plus-Filtek Supreme XT ($p < 0.03$). On the other hand, the bond strengths of Prime&Bond NT CeramX Mono and AdheSE One Tetric EvoCeram were not statistically significantly different. Finally, none of the examined adhesives exhibited statistically significantly different shear bond strengths to sound dentin relative to caries-affected dentin ($p > 0.05$).

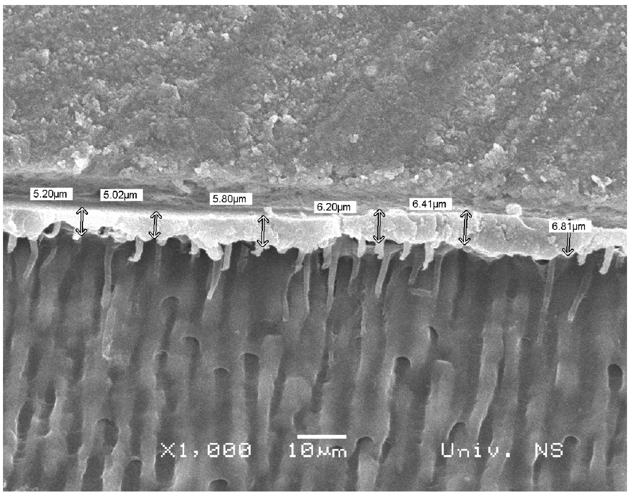

Fig. 4 – Scaninning electron micrograph at adhesive bond between Adpersingle Bond2/Filtek supremeXT to caries affected dentin (1000x magnification).

SEM analysis

The hybrid layer thickness was measured using the SEM device (NIH Image Analyser) proprietary software (Figures 4–7).

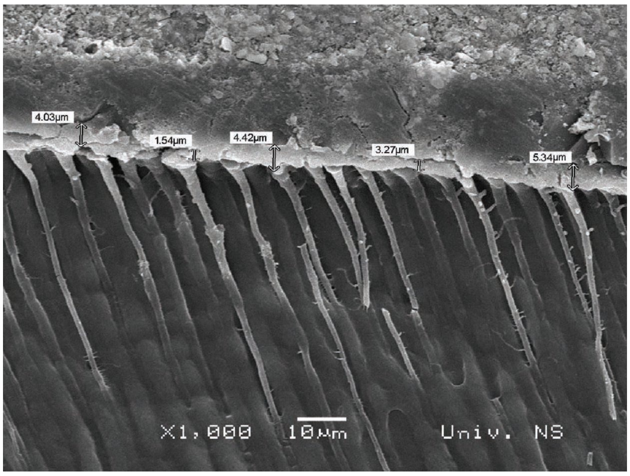

Fig. 5 – Scanning electron micrograph of adhesive bond bectween AdperSingle Bond2/Filtec supremeXT to sound dentin (SD) (1000x magnification).

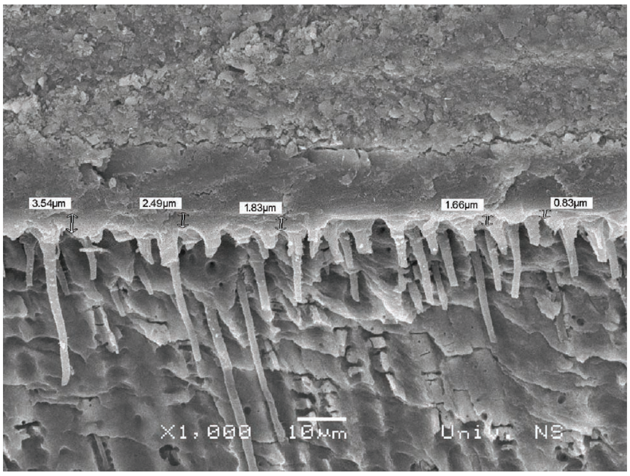

Fig. 6 – Scanning electron microgoph (SEMg) of adhesive bond between Prime&BoundNT/CeramX Mono to caries affected dentin (CAD) (1000x magnification).

Table 3

Basic statistical parameters characterizing hybrid layer thickness (HLT) and presence of statistical significance

Adhesive	Mean HLT ± sd (min, max) in µm CAD	Mean HLT ± sd (min, max) in µm SD	Significance CAD vs. SD
1. ASB-FS	5.32 ± 1.54 (2.89, 8.16)	3.39 ± 0.51 (2.23, 3.84)	$p < 0.01$
Significance 1 vs. 2	$p < 0.05$	$p < 0.05$	
2. PB-CXM	3.21 ± 2.68 (1.67, 4.54)	2.68 ± 0.91 (1.61, 4.44)	non-significant

sd – standard deviation; CAD – caries affected dentin; SD – sound dentin; ASB-FS – Adper Single Bound Plus-Filtek Supreme XT; AD-TEC – AdhesEOne-Tetric Evo Cerani; PB-CXM – Prime&Boudnt-CeramX Mono.

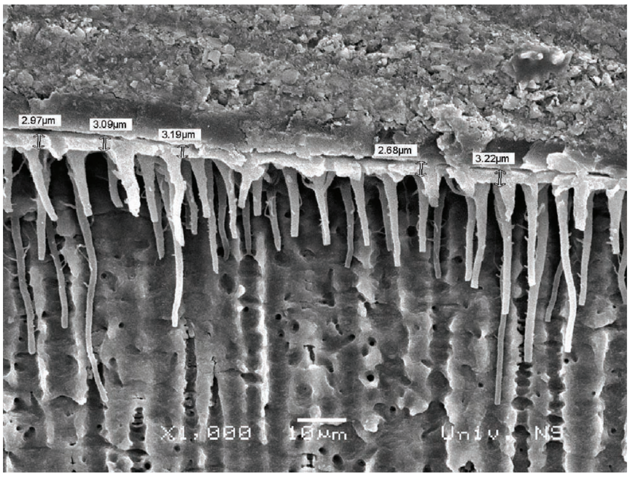

Fig. 7 – Scanning electron micrograph of adhesive bond between Prime&BondNT/CeranXMono to sound dentin (SD) (1000x magnification).

SEM images revealed that AdheSE One Tetric EvoCeram did not form the hybrid layer on the examined specimens.

The main statistical parameters pertaining to the hybrid layer thickness are presented in Table 3, along with the statistical significance of between-group differences. As can be seen from the tabulated findings, the greater hybrid layer thickness was formed on the CAD specimens.

The Adper Single Bond Plus-Filtek Supreme XT system produced a statistically significantly thicker hybrid layer on both types of dentin than the Prime&Bond NT – CeramX Mono system. On the SD specimens, the hybrid layers were thinner and the difference was statistically significant for the Adper Single Bond Plus-Filtek Supreme XT system only. No hybrid layer could be observed on the specimens with AdheSE One-Tetric EvoCeram.

Discussion

Adhesion to dentin is an important step in placing composite restoration. Organic nature of dentin and its higher humidity relative to enamel makes bonding to this hard tissue very difficult[2]. Compared to SD, bonding to CAD is characterized by the lower bond strength and inferior quality of the hybrid layer [10]. Owing to these discrepancies, the bonding efficacy of different adhesive systems with SD and CAD was extensively studied [18].

Development of new adhesive systems requires valid laboratory methods for testing material properties, such as the shear bond strength test technique [19]. To standardize the test protocol, ISO/TS 11405 specification was established in 2003. The device recommended by this protocol is referred to as a "guillotine" [14]. In our study, the adhesives that were compared in terms of their bond strength included the corresponding composite provided by the same company. Consequently, conclusions can only be drawn at the level of the adhesive/composite combination [20]. According to Salz and Bock [21], the "comparative bond strength tests are possible only at the level of identical adhesive/composite combinations, and certainly not at the level of the adhesive alone". This assertion justifies the approach adopted in our investigation. Indeed, the goal of testing adhesives incorporating composites produced the same company is to inform their application in everyday practice.

In our study, the comparison of the shear bond strengths of each adhesive to different substrates failed to reveal statistically significant differences. Consequently, the first null hypothesis was accepted. The shear bond strengths to SD and CAD were comparable, as the noted differences were not statistically significantly different. These results are in accordance with those reported by other authors [13, 20]. For example, Pereira et al. [22] posited that the absence of statistically significant differences between the bond strengths of Adper Single Bond Plus adhesive with SD and with CAD can be attributed to the large standard deviations, operator variability and/or technique sensitivity of this adhesive. Sonoda et al. [23] also reported the absence of statistically significant differences in the bond strength value of Prime&Bond NT to SD and CAD. These authors suggested that the caries retained after excavation, rather than the adhesive bond interface itself and this is potentially the weakest part of the adhesive bond. The low bond strength values were reported for AdheSE One to SD and CAD in other investigations, in which statistically significant differences between the two could not be established [17, 24]. AdheSE One has a pH of 1.5 and is classified as an "intermediately strong" self-etch approach (pH in the1−2 range) [8]. The functional monomer type in the adhesive composition plays a crucial role in the self-etch adhesive performance. In AdheSE, bis-methacrylamide dihydrogen phosphate serves as a functional monomer. This molecule is characterized by a short spacer chain of phosphate functional monomer, which induces formation of unstable monomer calcium salts. Consequently, chemical interactions are less pronounced, resulting in a lower dentin bond strength [25]. AdheSe is classified as a 2-hydroxylethyl methoacrylate (HEMA) free adhesive. Presence of HEMA in the self-etch adhesive composition increases the bond strength to dentin, as it provides good dentin wetting and hinders a phase separation between the hydrophobic components and dentin [25]. The low bond strength values of these adhesives can thus be attributed to the nature of their polymerization within dentin. Namely, a photoinitiator used in AdheSE One may contain acylphosphine oxides that do not react with many of the newer light emitting diode (LED) light curing units [23, 26].

A comparison of the SBS of the tested composite systems revealed some statistically significant differences (Table 2). Consequently, the second null hypothesis was rejected. Specifically, the bond strength of composite system with etch & rinse adhesive (Adper Single Bond Plus+Filtek Supreme XT) was statistically significantly higher than that measured for the composite system with self-etch adhesive (AdheSE One+Tetric EvoCeram). These results correspond to the findings yielded in an extensive study in which more than 16,000 SBS tests were examined [27]. However, as no statistically significant differences were noted between the bonding strength of the composite system with etch & rinse adhesive (Prime&Bond NT+ CeramX Mono) and that of the composite system with self-etch adhesive (AdheSE One+Tetric EvoCeram), our findings are not in line with those reported by Degrange and Lapostolle [27]. No differences between the shear bond strength of Prime&Bond NT and self-etch adhesives were found in the study conducted by Li et al. [28]. Prime&Bond NT is two-step acetone based etch & rinse adhesive characterized by high technique sensitivity. Acetone is unable to re-expand shrunken demineralized collagen [29]. High technique sensitivity and the chosen bond testing method (macro shear) may result in the low bond strength values [30, 31].

SEM is typically used when investigating the bonding mechanisms [18]. However, the ultrastructural data pertaining to adhesive interface cannot be directly interpreted in terms of bond strength to the tooth tissues. Micromorphological findings should always be carefully explained, as microscopic observations do not always correspond to the clinical findings [17].

Adhesion to dentin is a critical step in adhesive procedure. When using etch & rinse adhesives, the process commences with acid conditioning of dentin using phosphoric acid. This step results in a complete removal of the smear layer and smear plugs, leading to dentinal surface demineralization and exposure of the collagen fibrils [32]. The second step comprises of a resin monomer penetration into this demineralized dentinal surface. As a result, a resin-matrix reinforced by collagen fibrils, called hybrid layer, is formed [6]. Penetration of the resin monomer into opened dentinal tubules leads to the resin tag formation. The hybrid layers formed with CAD are thicker than those of SD [1–3, 5, 27, 33]. As CAD is partially demineralized, it is more susceptible to acid-etching. This leads to the formation of a deeper demineralized zone. For both CAD and SD, discrepancies the between demineralization depths and the resin monomer penetration extent are common [1]. The presence of highly acid resistant mineral deposits in dentinal tubules would interfere with the resin monomer infiltration as well as the resin tag formation. Thus, hybrid layer formed with CAD is thicker and the resin tags are less numerous, while the lateral branches are less pronounced and shorter.

Self-etch adhesives form a thicker hybrid layer with CAD compared to SD, but are thinner than those obtained when etch & rinse adhesives are utilized [3, 33]. The typical resin tags in dentin are produced by strong self-etching adhesives (pH $\leq$ 1) only, whereas they are rarely formed when mild and ultra-mild self-etching adhesives are employed [8]. Since the AdheSE One is classified as an "intermediately strong" self-etch approach, the typical resin tags are less numerous and are characterized by the poor lateral branches (Figure 8).

Due to their pH, self-etching adhesives cannot dissolve acid-resistant mineral deposits in dentinal tubules of CAD. However, the adhesive monomer penetration into CAD is hindered by a deeper mineralized zone, rather than adhesive pH [1]. With the exception of those based on strong self-etching adhesives, these systems cannot dissolve the smear layer and smear plugs, and they remain as a part of hybridized complex.

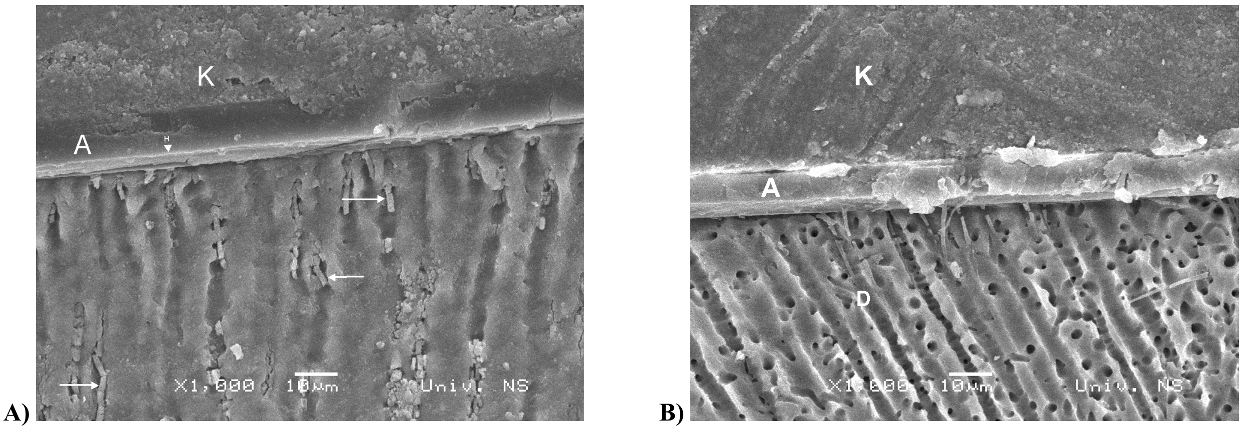

Fig. 8 – Scanning elecron microgroph (SEMg) of dentin specimens bonded with AdheSE One (1000x magnification). A) Caries affected dentin (CAD): barely observable hybrid layer, limited number of resin tags (arrows); B) Sound dentin (SD) – absence of hybrid layer, negligible presence of resin tags, many open dentin tubules without resin infiltration.
K – composite; A – adhesive layer; H – hybrid layer; D – dentin.

As the smear layer of CAD is enriched with disorganized collagen and mineral deposits, it may obstruct the resin monomer infiltration [34]. Thus, as we have found important differences in the microstructure of the interfaces between the tested composite systems on respective substrates, the third null hypothesis was rejected.

Conclusion

Noting the limitations of our study, the following conclusions can be drawn: 1) all tested composite systems bond equally well on sound and caries-affected dentin; 2) the etch-and-rinse adhesives achieved the stronger bond strengths; the Adper Single Bond Plus-Filtek Supreme XT system formed a statistically significantly thicker hybrid layer on both types of dentin than the Prime&Bond NT-CeramX Mono system.

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