

The Bonding Effectiveness of a Multi-Mode Adhesive System before and after the Removal of Amalgam Restoration Corrosion Products

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Abstract

Background: This study aimed to evaluate the effects of the corrosion products of old amalgam fillings on the bond strength of a universal bonding agent and a composite replacement alternative.

Materials and Methods: Ninety occlusal cavities were provided on caries-free extracted molars. The specimens were restored with high-copper amalgam and stored in the corrosive solution of NaCl 1.0% for about one year. In the half of the specimens, the amalgam was removed carefully to prevent the cutting of the adjacent dentin, and the final layer of the amalgam was left out with an explorer. For the remaining specimens, 0.5mm of the adjacent dentin was removed using a diamond bur. Next, the restorative procedure was followed for 9 groups (n = 10), using two types of adhesives (Single Bond Universal [SBU], a universal bonding, in self-etch [SE], etch and rinse [ER] modes as well as Clearfil SE bond [CLSE], a 2-step self-etch adhesive), and also three types of dentin substrates (dentin with corrosion products [C-dentin], dentin with a 0.5mm extension [E-dentin], and normal dentin [N-dentin]). After storage in distilled water (37°C) for 24 hours, the specimens were subjected to microtensile tests. The Energy Dispersive X-Ray Spectroscopy evaluation (EDX) was carried out to examine the corrosion products in dentin. The bond strength data were analyzed using a two-way ANOVA, followed by the Tukey test for the pair-wise comparison, when ANOVA was found to be significant ($\alpha = 0.05$).

Results: A significant difference was observed in the bond strength values of the nine groups, with the lowest values identified in the groups with a discolored dentin substrate. In addition, SBU in the SE mode showed the lowest bond strength ($P < 0.001$).

Conclusion: The removal of approximately 0.5mm of adjacent dentin improved the bond strength of dentin adhesives to the tooth structure, with the universal bonding used in this study having showed the best results in the etch and rinse modes.

Keywords: Amalgam Replacement; Corrosion; Microtensile Bond Strength; Universal Bonding

Introduction

Dental amalgam is one of the most popular filling materials used in dentistry [1]. However, public demands for tooth-colored restorations have increased. Despite the clinical success of amalgam restorations, many of them are replaced with composite resin materials due to esthetic concerns, micro-leakage, bulk and marginal fractures, and the recurrent caries of amalgam [2]. In most cases of amalgam removal,

gray-black discoloration occurs in the exposed dentin, being attributed to the amalgam corrosion products [3]. As the discolored dentin is partially demineralized and the amalgam ions penetrate through dentinal tubules, the dentin remained should be considered a different substrate for clinical procedures from the sound dentin [4]. One of the most recent innovations in the adhesive dentistry has been the introduction of ‘universal’ or ‘multimode’ adhesives. These new products are one-step adhesives that can be used as self-etch (SE), etch-and-rinse (ER), or selective-etch systems. Universal adhesives contain all bonding components in a single package enabling them to bond to various types of substrates, such as enamel, dentin, glass ionomers, metals, ceramics, and the like [5,6]. Although it has been shown that the bond strength of adhesive resins to abnormal dentin, such as sclerotic or caries-affected dentin, is lower than to normal dentin [7], the effect of corrosion products on the adhesion of universal adhesives is still unknown. Harnirattisai, *et al.* reported that discolored dentin found underneath amalgam restorations had a lower bond strength than normal dentin, but no difference was found between ER and SE adhesives in the process of bonding to dark dentin [2]. Another study showed that the 0.5 mm extension of cavity walls after amalgam removal improved the marginal seal of composite restorations [8]. The purpose of this study was to evaluate the effect of corrosion products on the bond strength of adhesive systems. The null hypotheses, including 1) corrosion products would not influence the bond strength of adhesive systems, 2) the bonding efficacies of the two adhesive systems (SBU and CLSE) would be the same, and 3) the bond strength of the universal adhesive would not be influenced by the application mode (ER or SE), were tested.

Materials and Methods

Tooth selection and preparation

Ninety occlusal cavities were prepared by a single operator on caries-free extracted molars, using a high-speed handpiece with air-water coolant and a diamond fissure bur (Diatech, Coltene, Whaledent AG, Switzerland). The bur was replaced after every five cavity preparation. The average bucco-lingual extension of the cavities was approximately one-third of the intercusp width, with the depth having been 2 millimeters. The specimens were divided randomly into nine experimental groups (n = 10), resulting from the combination of the factors of the ‘corrosion product’ (maintaining or removing the corrosion products of dentin with corrosion products [C-dentin] or extending the cavity walls [E-dentin]), the ‘adhesive system’ (Clearfil SE Bond [CLSE, Kuraray, Okayama, Japan]), Scotchbond Universal Adhesive [SBU, 3M ESPE, St. Paul, MN, USA, also known as Single Bond Universal in some countries], and the ‘adhesive strategy’ (etch-and-rinse [ER] or self-etch [SE]). Groups 1 to 6 were restored with high copper amalgam (SDI; GS-80-Australia) and stored in the corrosive solution of NaCl 1.0% in an incubator (Thelco precision model 17) at 37°C for about six months [8].

Group 1: CLSE was applied without extending the cavity floor

The amalgam was removed carefully to prevent the cutting of the adjacent dentin. To achieve this purpose, the final layer of the amalgam was removed using an explorer. The bonding agent was applied according to the manufacturer’s directions, as shown in table 1. After the bonding procedure, the teeth were restored incrementally using a composite resin (Clearfil APX, Lot 0598, Kuraray Medical, Osaka, Japan) and light cured for 40 seconds per increment at 1200 W/cm², using a Bluephase C₈ (IvoclarVivadent, Schaan Liechtenstein).

Group 2: CLSE was applied after extending the cavity floor

After removing the original amalgam from the specimens, 0.5 mm of the adjacent dentin was removed carefully from the cavity floor using a diamond bur (Diatech, Coltene, Whaledent AG, Switzerland). To increase the measurement accuracy, a graded periodontal probe was utilized. Next, the restorative procedure was followed as done for group 1.

Group 3: SBU was applied in the SE mode without extending the cavity floor

In order to maintain the corrosion products, the cavity walls were prepared as done for group 1. The bonding agent (SBU) was applied according to the manufacturer’s instructions as shown in table 1. After the bonding agent was light cured, the cavities were restored incrementally using a composite resin (Filtek Z250 XT, 3M ESPE) as done for groups 1 and 2.

Group 4: SBU was applied in the SE mode after extending the cavity floor

After removing the original amalgam from the specimens, 0.5mm of the adjacent dentin was removed from the cavity floor. Next, the restorative procedure was followed as done for group 3.

Group 5: SBU was applied in the ER mode without extending the cavity floor

The specimens in this group were restored as done in Group 3, yet the bonding agent was applied in the ER mode, according to manufacturer’s instructions as shown in table 1.

Adhesive system/ (Lot Number)	Composition	SE Strategy	ER Strategy
Clearfil SE Bond (Primer: 00954A- Bond: 01416)	Primer: water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate Bonding: MDP, Bis-GMA, HEMA, camphorquinone, hydrophobic dimethacrylate, N,N-diethanol p-toluidine bond, colloidal silica	<ol style="list-style-type: none"> 1. Apply primer to tooth surface and leave in place for 20 s 2. Dry with gentle air flow to evaporate the volatile ingredients 3. Apply bond to the tooth surface and then create a uniform film using a mild air flow 4. Light-cure for 10 s at 1200mW/cm 	NA
Scotchbond Universal Adhesive (D-82229)	Etchant: 34% phosphoric acid (Scotchbond Universal Etchant) Adhesive: MDP phosphate monomer, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer, filler, ethanol, water, initiators, silane	<ol style="list-style-type: none"> 1. Apply the adhesive for 20 s and gently air-dry for 5s 2. Light-cure for 10s at 1200 mW/cm 	<ol style="list-style-type: none"> 1. Apply etchant for 15s rinse for 10s and air dry Apply adhesive as for the SE mode

Table 1: Adhesive System (Lot Number), Composition, and Application Mode of the Adhesive Systems Used According to the Manufacturer’s Instructions.

MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate;
Bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropyl)phenyl]-propane.

Group 6: SBU was applied in the ER mode after extending the cavity floor

After removing the amalgam from the specimens, 0.5 mm of the adjacent dentin was removed using a round diamond bur. Next, the restorative procedure was followed as done for group 5.

Group 7: CLSE was applied to normal dentin

The adhesive procedures and preparations in this group were performed through the same techniques used for groups 1 and 2.

Group 8: SBU was applied to normal dentin in the SE mode

The adhesive procedures and preparations in this group were performed through the same techniques used for groups 3 and 4.

Group 9: SBU was applied to normal dentin in the ER mode

The adhesive procedures and preparations in this group were performed through the same techniques used for groups 5 and 6. All specimens were mounted in an acrylic resin (Acropars, Iran) and stored in distilled water (37°C) for 24 hours.

The microtensile bond test

Five specimens from each group were selected randomly and sectioned using a low-speed computerized numerically controlled cutting machine (Nemo, Mashhad, Iran) under water cooling in two directions perpendicular to the bonded interface so as to provide beam-shaped specimens with the bonding area of approximately 1 mm². The specimens were analyzed using a stereomicroscope (Zoom;

Blue Light Industry USA Inc., La Habra, CA, USA) at 40x magnification. The samples with voids or cracks at the resin-dentin interface were excluded from the study. Twenty five sound beam specimens were selected randomly for each group [2]. The specimens were attached to a split holder using cyanoacrylate glue and subjected to μ TBS testing using a universal testing machine (STM20; Santam, Tehran, Iran) at the cross-head speed of 1mm/min until failure. The failure mode analysis was performed under the mentioned stereomicroscope at 100x magnification. The failure modes were classified as ‘adhesive’ (failure at the resin/dentin interface), ‘cohesive’ (failure within the composite layer or the dentin layer) and ‘mixed’ (the combination of adhesive and cohesive failures). The number of specimens with premature failures was recorded, yet the values were not included in the statistical analysis. The rationale behind this approach was that all premature failures occurred during the cutting procedure and were distributed similarly within the groups. Furthermore, the number of premature failures was not significant, being about 3% of the total number of the tested groups [5].

Energy dispersive x-ray spectroscopy and the scanning electron microscope (SEM)

The five teeth left in each group were sectioned mesio-distally using a KG Sorensen diamond disc (Industria e Comercio, Ltd, Sao Paulo, SP, Brazil), with each section having been coated with a palladium-gold layer and used for the surface analysis by means of scanning electron microscopy (SEM; XL 30; Philips, Eindhoven, Netherlands); in addition, an energy dispersive spectrometer operated at 20kV was utilized to analyze corrosion products in previously amalgam-restored specimens. The presence of tin (Sn), Chlorine (Cl), and copper (Cu) was determined at 2000x magnification after amalgam removal, with and without the extension of the dentinal floor.

Statistical analysis

The Kolmogorov–Smirnov test was used to assess if the data followed a normal distribution. After assuring the normality of distribution for each variable, the bond strength data were analyzed using the two-way ANOVA followed by a Tukey test for the pair-wise comparison if ANOVA was found to be statistically significant ($\alpha = 0.05$). SPSS version 16 (SPSS, Inc., Chicago, IL, USA) was used in the analysis.

Results

Bond strength test

The Kolmogorov-Smirnov test of normality showed that the variables followed a normal distribution. The mean bond strength values and standard deviation of all groups are shown in table 2. The bond strengths of CLSE and SBU to E-dentin and N-dentin were significantly higher than their bond strengths to C-dentin. There was no significant difference between the bond strength of E-dentin and that of N-dentin. The lowest mean microtensile bond strength (μ TBS) was obtained for group 3 ($P = 11.76 \pm 3.70$) (Table 2). The results of the present study showed that there were significant differences between different types of adhesive systems. Regardless of the type of the substrate, CLSE showed the highest bond strength, followed by SBU-ER and SBU-SE. The bond strength of SBU-ER was significantly higher than that of the same adhesive applied using the SE procedure ($p < 0.001$). The most frequent failure identified in each group was the adhesive failure (Table 3).

Adhesives	Dentin Type		
	C-dentin	E-dentin	N-dentin
CLSE	15.84 ^{Aa}	27.97 2.67 ^{Ab}	28.46 ^{Ab}
SBU-SE	11.76 ^{Ba}	18.02 ^{Bb}	18.36 ^{Bb}
SBU-ER	16.08 ^{Aa}	22.03 ^{Cb}	24.01 ^{Cb}

Table 2: Results of one-way ANOVA and Tukey test for pair-wise comparison of experimental groups.

^aGroups connected by different uppercase letters in columns and different lowercase letters in lines represent statistical differences ($p < 0.001$).

Groups	Mode of failure			Cohesive ^c in composite
	Adhesive ^a	Mixed ^b	Cohesive ^c in dentin	
Group 1	18	5	2	0
Group 2	20	1	4	0
Group 3	17	6	1	1
Group 4	21	2	1	1
Group 5	17	6	0	2
Group 6	20	1	3	1
Group 7	19	1	4	1
Group 8	20	4	1	0
Group 9	19	2	4	0

Table 3. Modes of failure distribution in experimental groups.

^a: Adhesive failure: failure at the resin/dentin interface.

^b: Mixed failure: combination of adhesive and cohesive failure.

^c: Cohesive failure: failure within the composite layer or the dentin layer.

The EDX analysis and SEM images

The energy dispersive x-ray microanalysis of the dentin adjacent to the restoration was used to analyze corrosion products. Tin, copper, and chloride were detected in groups with corrosion products (groups 1, 3, and 5). No mercury was found in the foregoing groups. After refreshing the dentinal floor, no metal element was found in groups 2, 4, and 6 (the same as control groups 7, 8, and 9).

The representative SEM images of resin-dentin interfaces are shown in figure 1 for all specimens. The hybrid layer was very thin in groups containing metal elements (groups 1, 3, and 5) and undetectable in some places. In contrast, the SEM images of the groups with E-dentin and N-dentin showed a typical hybrid layer. Open dentinal tubules were mostly observed in groups 6 and 9.

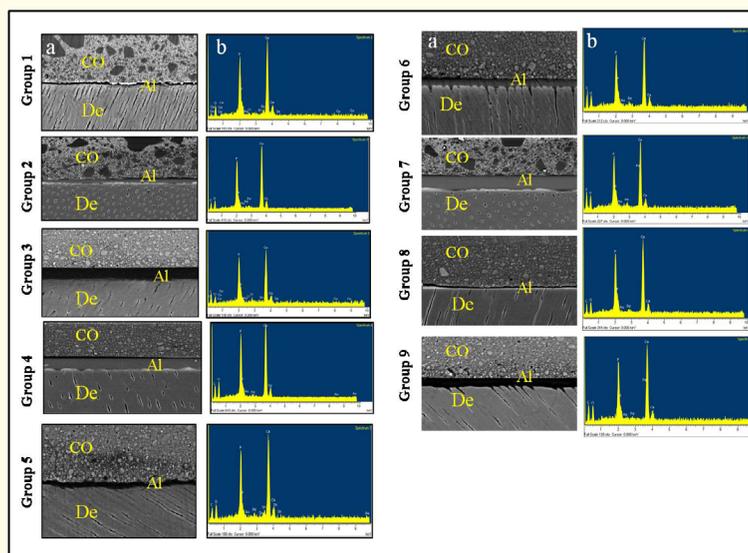


Figure 1: (a) SEM image of dentin (De)/composite (Co) interface and the adhesive layer (Al). (b) EDX microanalysis of the dentin adjacent to the restoration in experimental groups. Metal elements were detected in groups 1, 3 and 5 but no metal ions were found after extension of the cavity floor (groups 2, 4 and 6) the same as control groups (groups 7, 8 and 9).

Discussion

The results of this study rejected the first hypothesis that stated the μ TBS values of the two different adhesive systems to C-dentin were lower than those of N-dentin and E-dentin; however, there was no significant difference between the bond strength of N-dentin and that of E-dentin. This state could be attributed to the precipitation of metal ions in dentin tubules, which might affect the quality of adhesive resins penetrating into the dentin [1]. Amalgam restorations often corrode in the oral environment. In the present study, 1.0% NaCl was used as the test solution, which contained a higher Cl concentration, so it facilitated the generation of amalgam corrosion byproducts [9]. In the cases of amalgam removal, clinicians often encounter dark grayish black dentin on the cavity floor. Corrosion products from amalgam are responsible for the mentioned discoloration [4], especially Cu and Sn, which can penetrate easily into the underlying dentin [3]. The result of the energy dispersive x-ray microanalysis of the dentin adjacent to the amalgam restoration confirmed the presence of metal elements, such as Sn, Cl, and Cu (groups 1, 3, and 5). This finding was in accordance with that of other studies, for Sn was detected with the highest amount, followed by Cl and Cu [4,8]. After extending the cavity floor by about 0.5mm (E-dentin), no metal element was detected in the SEM analysis (groups 2, 4, and 6), being the same as the control groups (N-dentin) (groups 7, 8, and 9). This finding was fully in agreement with that of a previous study in which the specimens without extending the cavity walls showed the lowest bond strength of the composite restoration, yet the extension of the cavity preparation by about 0.5 mm resulted in a higher bond strength [10]. In another study, Ghavamnasiri, *et al.* reported that the 0.5 mm extension of cavity walls improved the marginal seal of the composite replacing amalgam restorations [7].

In the present study, the bonding performance of a two-step SE system (CLSE) was compared with that of a newly introduced one-step universal system (SBU) when applied, following a SE or an ER approach. SE adhesives are considered as simplified adhesive materials because they do not require a separate acid conditioning step [11]. The two adhesive systems were used on the three different substrates of 'N-dentin', 'C-dentin', and 'E-dentin'. According to the findings of the present study the second hypothesis could not be accepted that regardless of the type of the substrate, in a comparison between CLSE and SBU used with the SE mode or with the ER mode, CLSE revealed higher bond strength compared with a universal bonding adhesive [12,13]. CLSE is considered as the gold standard of two-step SE adhesives [13,14]. It contains MDP monomer (10-methacryloyloxydecyl dihydrogen phosphate) in its composition, which is capable of interacting chemically with hydroxyapatite, forming more strong ionic bonds with calcium than other monomers do (e.g. HEMA and Phenyl-P), thereby improving the micromechanical bond strength [12]. It is worth mentioning that both CLSE and SBU have MDP in their compositions. Nevertheless, the concentration of MDP in CLSE is higher than that of SBU because CLSE has MDP in both the primer and the bonding [12]. SBU is also compromised of polyalkenoic acid (also known as the Vitrebond copolymer), which bonds chemically to hydroxyapatite minerals [14]. The presence of the polyalkenoic acid in SBU may compete with MDP by bonding to the calcium of hydroxyapatite, thereby impairing the chemical bond of MDP to dentin [15]. In addition, the mixing of different components, such as acidic and non-acidic monomers, solvents, fillers, initiators, and silane in SBU could influence the bonding properties [6].

In this study, SBU was used in ER and SE modes on three different substrates. Similar to the past researches [16,17], the SEM observation of the resin-dentin interface in SBU-ER showed the complete penetration of the resin into dentinal tubules. However, SBU-SE showed shorter resin tags and a less retentive pattern. These findings were in accordance with those of other research reporting that the bond strength of SBU applied using the ER technique was significantly higher than that of the same adhesive applied using the SE mode [13,17,18]. Thus, the results of the study do not support the third hypothesis that irrespective of the type of the substrate, SBU in the ER mode presented higher bond strength than the SE mode [13,18]. Using phosphoric acid increased penetration of the adhesives and their better interlocking with the tooth structure by removing the smear layer and smear plugs, resulted in formation of a well-impregnated hybrid layer [18]. In addition, the etching pattern caused by SBU in the SE mode appeared to be less retentive than the one pretreated by phosphoric acid [16]. The results of the present study were not in agreement with the past research implying that 'the application mode would not affect the bond strength of the universal adhesive systems' [15,19].

It is worth noting that some adhesives, such as one-step SE adhesives, are significantly affected by the bur preparation [20]. Therefore, the way dentin is prepared should be considered as an important factor prior to bonding [21]. The bur-cut dentin is covered with a thick and compact smear layer that could prevent the impregnation of collagen fibers by adhesives of high pH [22]. SBU is considered as an ultra-mild SE adhesive, because of its pH being about 3.0 [23]. An adhesive with such a high pH level is not able to demineralize the superficial dentin [13], for the dentin or the smear layer could neutralize the etching primers of the relatively high pH [24]. Therefore, adhesives with higher pH levels are not able to demineralize the superficial dentin [13]. CLSE is classified as a mild adhesive with the pH of 1.9 - 2, which demineralizes dentin up to the depth of 1 μm [25]. It seems that the buffering effect of the smear layer is more noticeable in ultra-mild adhesives [26].

In the current study, adhesive failure was observed in most of the tested specimens. The mixed failure pattern as well as the combination of the adhesive and cohesive failures was more prevalent in the specimens containing corrosion products.

The present in vitro study evaluated the immediate (24h) bond strength of adhesive resins. In ER adhesives, there was a discrepancy between the etching depth and the adhesive impregnation of the network of exposed collagen fibrils [14]. The exposed collagen fibrils, at the bottom of the hybrid layer not fully encapsulated by the resin, would be vulnerable to degradation by collagen-bound matrix metalloproteinases (MMPs) after acid etching [27]. The enzyme activation process was the reason for the hydrolytic degradation of the hybrid layer and eventually the loss of the dentinal bond strength [28]. However, the simultaneous demineralization and infiltration of SE adhesives into the exposed collagen layer would decrease the risk of the discrepancy between the bottom of the collagen fibrils and resin infiltration [14]. Acidic environments, such as the acid components of total etch or self-etch adhesives could also increase the activity of matrix-bound MMPs [29]. Hence, further studies, especially clinical trials, need to be conducted to evaluate the effects of long-term storage on the bonding ability of other universal adhesives and restorative materials in discolored dentin and other substrates.

Conclusion

Given the limitations of this study, it could be concluded that the bonding effectiveness of the universal adhesives tested was significantly affected by corrosion products, with refreshing the dentinal floor for about 0.5mm having improved the bond strength.

Clinical Relevance

In the case of amalgam replacement by adhesive resin composites, refreshing the dentinal walls, and using two-step self-etch adhesives would provide the high bond strength.

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