Assessment of Bond Strength of Different Bonding Systems to Eroded Dentin Using Sonic Activation: In Vitro Study

Mustafa H. Mohammad[1] Samer A. Thyab[1]

[1] Department of Conservative and Aesthetic Dentistry, Baghdad College of Dentistry, University of Baghdad, Baghdad, Iraq

Eur J Gen Dent

Address for correspondence Mustafa H. Mohammad, B,D,S,
Department of Conservative and Aesthetic Dentistry, Baghdad
College of Dentistry, University of Baghdad, Baghdad, Iraq
(e-mail: altmemymostfa@gmail.com).

Abstract

Objective Bonding to dentin is a great challenge, and erosion makes it difficult. This study evaluated the effect of sonic agitation on bond strength to eroded dentin using total etch (TE) and self-etch (SE) adhesive systems.

Material and Methods A 96-sound human maxillary premolar teeth were prepared to obtain a flat dentin surface, randomly divided into two groups of 48 teeth each. One group was erosive demineralized with 0.05-M citric acid solution to produce an erodedlike dentin surface using de- and remineralization cycles, and the other group was considered a control with no erosive treatment. Each group was bonded with two different bonding strategies, TE (Single Bond 2) and SE (AdheSE One F). Each bonding system was applied with and without sonic activation, and the effect of bond agitation on shear bond strength (SBS) was tested after the application of composite to bonded dentin surface under shear load (0.5 mm/min). Two samples from each group were randomly selected for scanning electron microscopy (SEM) evaluation of the hybrid layer quality. An independent sample *t*-test was used to assess the effect of erosion, activation, and adhesive type on the SBS. Statistical significance was set at $p < 0.05$.

Result Concerning dentin substrate type, eroded dentin has lower SBS mean values than sound dentin, although it was not significant. Regarding the bonding strategy, TE produced higher SBS values than the SE bonding strategy; it was significant only in sound dentin; despite the bonding strategy and the substrate dentin type, sonic activation produced a significant rise in SBS values except when the SE bonding strategy was applied to eroded dentin where the rise was of no significant value ($p > 0.05$).

Conclusion Sonic agitation effectively improves the SBS value of composite bonded to eroded and sound dentin in both TE and SE strategies.

Keywords
- sonic activation
- eroded dentin
- adhesive system

Introduction

Erosion in dentin is a complex challenge in dentistry.[1] According to Bartlett and O'Toole, the prevalence of dental erosion has increased by approximately 30% among the global population, including the young adult population, emphasizing the importance of dental erosion in dental practice.[2] Enamel and dentin are highly mineralized tissues; dentin consists of the inorganic matrix surrounding and protecting organic content, predominantly type I collagen, making dentin a problematic substrate to bond to.[3] Dental erosion is a chemical process resulting in a cumulative loss of

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hard dental tissue not caused by bacteria.[4] This accumulative process primarily affects enamel, but when it reaches the dentin, it can remove dentinal plugs and organic intertubular dentin, resulting in increased tubule diameters.[5,6] The eroded dentin surfaces has more exposed collagen fibrils and reduced mineral content in the outermost layer.[5,6] This mineral dissolution often causes aesthetic and functional impairment and dentinal hypersensitivity.[7] That requires intervention, which mostly involves a direct composite resin, which is the most commonly used filling material.[8] The different characteristics of eroded dentin represent an additional challenge for dental adhesion, as resin monomers often inadequately infiltrate a superficial layer of exposed collagen in eroded dentin.[8] However, there has yet to be a consensus about which adhesive strategy achieves the best performance in eroded dentin[9]; currently, two-step etch-and-rinse TE and self-etch (SE) adhesives are the two popular adhesive approaches used mostly in restorative dentistry for composite placement to both sound and eroded dentine.[10,11] Higher bond strength results were observed for eroded dentin that received 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate) universal adhesives.[12] The functional monomer 10-MDP forms a stable nanolayer with the deposition of MDP calcium salts at the adhesive interface, increasing the mechanical resistance and minimizing hydrolysis.[13] To increase the bond strength, many studies tested the effect of adhesive application time[14] and the magnitude of rubbing force on bond strength.[15] Other studies tested the effect of agitation on TE and SE strategy to increase the bond strength.[16] Many studies show significant benefits of agitation, by either vigorous, active, or ultrasonic agitation, on the bond strength of SE adhesive,[17,18] whereas others report that ultrasonic agitation does not affect the SE bonding mode.[19–21] This study aimed to evaluate the effect of sonic activation on bond strength to eroded dentin with two different adhesive systems (Adhesive SE F one,7th generation) and (single bond 3M, 5th generation) and using scanning electron microscopy (SEM) analysis to evaluate the effect of sonic activation on hybrid layer thickness and resin tag length, so the null hypothesis of this study was that sonic agitation does not affect the bond strength of adhesive in sound and eroded dentin.

Materials and Methods

Sample Preparation

Ninety-six (96) intact noncarious human maxillary premolars extracted for orthodontic causes with an age range between 14 and 30 years were collected for this study. The teeth were stored in thymol solution (0.1%) for about 2 months till the time of the study to prevent bacterial and fungal growth; each tooth was cracked free and examined by a dental microscope (5X). After cleaning and polishing, they were placed in an acrylic mold with the aid of a dental surveyor; occlusal dentin was exposed by cutting the most coronal part of the crown 2 mm below the deepest point in the occlusal surface by a slow-speed straight headpiece with coolant, then the occlusal surface was grounded with a grinder polisher for 20 seconds.[22,23]

Sample Grouping

The sample was divided into the following:

- Group I (control): No erosion (NOER) was conducted to this sample.
  - Subgroup 1: 10 samples were bonded with a total-etch (TE) bonding strategy without sonic activation (NOACT): NOER_TE_NOACT.
  - Subgroup 2: 10 samples were bonded with a TE bonding strategy with sonic activation (ACT): NOER_TE_ACT.
  - Subgroup 3: 10 samples were bonded with a self-etch (SE) bonding strategy without sonic activation (NOER_SE_NOACT).
  - Subgroup 4: 10 samples were bonded with an SE bonding strategy with sonic activation (NOER_SE_ACT).
- Group II (eroded dentin): Erosion (ER) of the exposed dentin in this sample was made using the citric acid solution.
  - Subgroup 1: 10 samples were bonded with a TE bonding strategy without sonic activation after artificial erosion preparation (ER_TE_NOACT).
  - Subgroup 2: 10 samples were bonded with a TE bonding strategy with sonic activation after artificial erosion preparation (ER_TE_ACT).
  - Subgroup 3: 10 samples were bonded with an SE bonding strategy without sonic activation (ER_SE_NOACT).
  - Subgroup 4: 10 samples were bonded with an SE bonding strategy with sonic activation (ER_SE_ACT).

Ph Cycling Model

The specimens were subjected to cycles of demineralization and remineralization procedure over a total of 10 days (5 d/wk; 2 weeks). Specimens were erosively demineralized with 0.05-M citric acid solution (pH 2.3, anhydrous citric acid) six times per day for 5 minutes. The citric acid solution was renewed at each erosive challenge, and the remineralization solution was replaced daily. After each demineralization challenge, the specimens were rinsed with deionized water for 10 seconds and immersed in a remineralizing solution (4.08 mM H_3PO_4, 20.10 mM KCl, 11.90 mM Na_2CO_3, and 1.98 mM $CaCl_2$, pH of 6.7) for 60 minutes.[11] The pH of all solutions is monitored periodically with a pH meter.[13] In this study, an SEM image was used to ensure the erosion process in the dentin; the process was done after bonding and resin application to prevent further alteration to eroded dentin during the dryness process in the vacuum in the preparation procedure for SEM.[24]

Modified (Sonic) Micro-Brush Assembly

Sonic activation of the dentin surface after bonding system application was performed using a modified sonic endo-activator (Dentsply Sirona, Konstanz, Germany), in which a medium-sized bonding brush was installed on the top of the activator by a small-diameter rubber tube. The resultant assembly has two vibration frequencies of approximately 2 and 10 kHz, as shown in ►**Fig. 1**.

Fig. 1 (**a**) Modified sonic micro-brush assembly. (**b**) The components used to fabricate modified sonic micro-brush assembly, which include plastic tube, micro-brush, and endo-activator device.

Restorative Procedures

The bonding procedures of both adhesive strategies were performed as illustrated in ►**Table 1**. Bonding activation was performed by a modified sonic micro-brush assembly that vibrates at the lowest frequency (2 kHz) for 20 seconds, and the bonding procedure for the nonactivation group was made by manually rubbing the dentin surface with a medium-sized bonding brush for 20 seconds. The bonded surfaces for all groups were cured by a light-emitting diode light-curing unit (EliparTM DeepCure-L, 3M ESPE, Germany) which was used at a wavelength of 430 to 480 nm, 1,470 mW/cm^2 light intensity for 10 seconds according to the manufacturer's instructions. Flowable composite (Filtek Flow A2, 3M ESPE) was placed over the bonded dentin surfaces. A Teflon device was designed to standardize composite application on bonded dentin to produce a 4-mm-diameter and 2-mm-high composite disk that was cured with a light cure device for 10 seconds at a constant distance of 1 mm.[25,26]

Shear Bond Strength Test

An Instron Universal testing machine measured the shear bond strength (SBS) between the composite and dentin (Instron Corp, Canton, MA, United States). Instron's knife-edge stainless steel rod was gently adapted against the dentin–composite resin interface, and the loading head was set at a crosshead speed of 0.05 mm/min. Calculating SBS in megapascal involves dividing the peak load at failure by the surface area of the specimen.[22,27]

Table 1 Bonding adhesive systems, their composition, and their application technique according to the manufacture's instruction

Material	Manufacturer	Composition	Application technique
Single Bond 2	3M ESPE, St Paul, MN, United States	Etchant: 35% H_3PO_4 Adhesive: dimethacrylates, HEMA, colloidal silica, polyalkenoic acid copolymer, ethanol, water, and photoinitiator	Two-step etch-and-rinse system 1. Apply etchant for 15 s 2. Rinse and blot dry 3. Applying adhesive with manual bonding brush for 20 s for non activation groups + sonic activation for 20 s for activation groups 4. Gentle air stream 5. Light cure for 10 s
AdheSE One F	Ivoclar Vivadent, Schaan, Liechtenstein	Nonmethacrylate monomer, amino acid acrylamide, bis-methacrylamide dihydrogen phosphate, sulfonic acid acrylamide, water, alcohol, and potassium fluoride	One-step self-etching system 1. Dry surface 2. Applying adhesive with manual bonding brush for (20 s) for non activation groups + sonic activation for 20 s for activation groups 3. Gentle air stream 4. Light cure for 10 s

Scanning Electron Microscopic Analysis

Two specimens were randomly selected from each group for SEM analysis. To facilitate the study, the selected specimens, in their acrylic blocks, were sectioned longitudinally through the composite–dentin interface in a mesiodistal direction using a super-thin cutting disk mounted on an electrical saw with copious water coolant and at low speed to avoid heat generation. One of the obtained sections from each sample was randomly selected to be polished using fine silicon carbide papers graded from 400, 600, 800, and 1,000 grits under running water coolant to get a smooth and shiny surface.[28,29] After 24 hours of storage of specimens in distilled water to prevent dehydration, they were cleaned in 70% ethanol in an ultrasonic bath for 2 minutes to remove the polishing debris. The specimen preparation for SEM analysis included acid etching using 37% phosphoric acid for 15 seconds to remove cutting debris and smear layer; this was followed by dentin decalcification for 30 seconds using 6% normality hydrochloric acid and rinsing thoroughly with water. The sample was then deproteinized in 2.5% NaOCl for 10 minutes to dissolve the organic dentin matrix and enable examination of the adhesive interface. Finally, the samples were washed with 96% ethanol to remove the water before desiccating and prepared for SEM imaging.[27,28] After sample preparation, each sample was sputtered with gold nanoparticles. Then, the micro-morphological SEM analysis for the adhesive layer of each specimen was performed using SEM at successively higher magnifications.[30]

Statistical Analysis

The obtained SBS test data were analyzed using SPSS (software version 26). The Shapiro–Wilks test was used to assess the normality of data. An independent sample *t*-test was used to compare different groups to determine the effect of erosion, activation, and adhesive type on the SBS. Statistical significance was set at $p < 0.05$.

Results

The Shapiro–Wilk test revealed that the data were normally distributed ($p > 0.05$). The mean and standard deviation values of the SBS (MPa) for all the study groups are shown in ►**Table 2**. The TE bonding strategy with sonic activation resulted in the highest SBS mean value when bonded to sound dentin (17.2 ± 2.44 MPa). In contrast, the lowest mean value was recorded when eroded dentin bonded with SE bond with no activation (9 ± 3.37 MPa), as shown in ►**Fig. 2**.

Concerning dentin substrate type, an independent sample *t*-test at a significance level of 0.05 revealed a statistically significant difference between sound and eroded dentin within each bonding strategy whenever sonic activation was applied (as shown in ►**Table 3**).

When comparing the two bonding strategies, the differences were always significant, except in eroded dentin when no activation was applied, as shown in ►**Table 4**.

Despite the bonding strategy and the substrate dentin type, sonic activation produced significant differences in SBS values except when the SE bonding strategy was applied to eroded dentin, as shown in ►**Table 5**

SEM Interfacial Morphology Characterization

The SEM images of the activation group (►**Fig. 3b, d, f, h**) in both sound and erosion dentin show a well-defined hybrid layer and more resin tag infiltration. In the erosion non-activation group, the SEM images (►**Fig. 3e, g**) show cracks in the hybrid layer and empty spaces that represent the area where the resin fails to infiltrate; in the activation group (►**Fig. 3f, h**), SEM shows a decrease or absence of cracks and empty space as shown in ►**Fig. 3**.

Discussion

Bonding to eroded dentin is a great challenge and requires high knowledge and reasonable control of the bonding technique; both SE and TE bonding strategies that react differently to the smear layer are used for bonding eroded dentin, which shows significant histological change including reduced mineral content and great exposure of collagen fiber; the tubule becomes enlarged, and peritubular dentin becomes rough and pores form, making monomer

Table 2 Shear bond strength mean values and standard deviation (MPa) in all study groups

Groups			*N*	Mean ± SD (MPa)
Substrate dentin type	Bonding system	Sonic activation		
Erosion	TE	With	10	14.13 ± 2.83
	TE	Without	10	10.6 ± 3.24
No erosion	TE	With	10	17.2 ± 2.44
	TE	Without	10	12.5 ± 3.57
Erosion	SE	With	10	10.2 ± 2.74
	SE	Without	10	9 ± 3.37
No erosion	SE	With	10	13.1 ± 3.07
	SE	Without	10	9.3 ± 1.64

Abbreviations: SBS, shear bond strength; SD, standard deviation; SE, self-etch, TE, total etch.

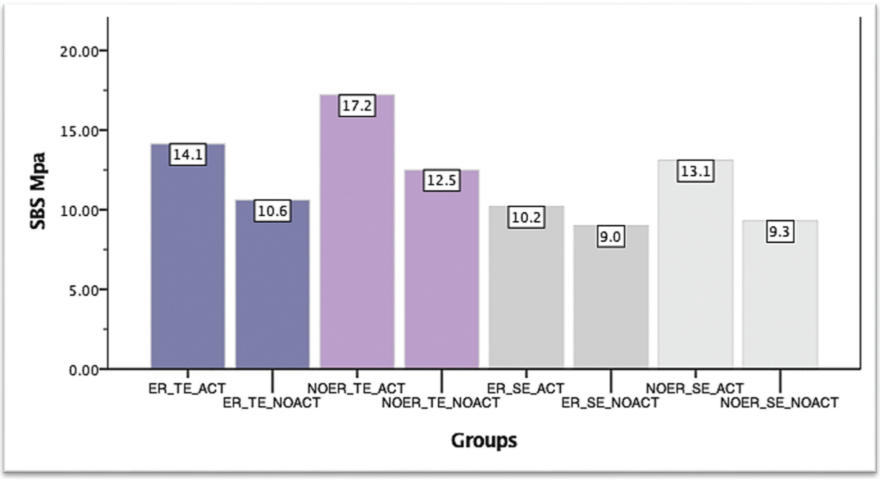

Fig. 2 The shear bond strength (SBS) mean values of all the groups.

Table 3 Independent sample *t*-test between different study groups concerning substrate dentin type

Groups	Substrate dentin type	*N*	Mean ± SD	*p*-value[a]
TE_ACTIVATION	Eroded	10	14.13 ± 2.83	0.018[a]
	Not eroded	10	17.2 ± 2.44	
TE_NOACTIVATION	Eroded	10	10.6 ± 3.24	0.228
	Not eroded	10	12.5 ± 3.57	
SE_ ACTIVATION	Eroded	10	10.2 ± 2.74	0.039[a]
	Not eroded	10	13.1 ± 3.07	
SE_ NO ACTIVATION	Eroded	10	9 ± 3.37	0.803
	Not eroded	10	9.3 ± 1.64	

Abbreviation: SD, standard deviation.
[a]The significance value.

Table 4 Independent sample *t*-test between different study groups concerning bond strategy

Groups	Adhesive type	*N*	Mean	*p*-value[a]
EROSION_ACTIVATION	TE	10	14.13 ± 2.83	0.006[a]
	SE	10	10.2 ± 2.74	
EROSION_NOACTIVATION	TE	10	10.6 ± 3.24	0.293
	SE	10	9 ± 3.37	
NOEROSION_ACTIVATION	TE	10	17.2 ± 2.44	0.004[a]
	SE	10	13.1 ± 3.07	
NOEROSION_NOACTIVATION	TE	10	12.5 ± 3.57	0.023[a]
	SE	10	9.3 ± 1.64	

Abbreviations: SE, self-etch; TE, total etch.
[a]The significance value.

infiltration and polymerization more challenging.[31] This alteration in eroded dentine morphology might jeopardize the quality of the hybrid layer since dentine bonding depends on the infiltration of resinous monomer to exposed tubules after the process of demineralization and the subsequent polymerization of monomer that fills spaces created in collagen mesh that makes resin tag and hybrid layer. The quality of infiltration of monomer determines the durability of this

Table 5 Independent sample *t*-test between different study groups concerning the bond activation method

Groups	Activation method	*N*	Mean	*p*-value[a]
TE_EROSION	Activation	10	14.13 ± 2.83	0.018[a]
	No activation	10	10.6 ± 3.24	
TE_NOEROSION	Activation	10	17.2 ± 2.44	0.003[a]
	No activation	10	12.5 ± 3.57	
SE_EROSION	Activation	10	10.2 ± 2.74	0.394
	No activation	10	9 ± 3.37	
SE_NOEROSION	Activation	10	13.1 ± 3.07	0.003[a]
	No activation	10	9.3 ± 1.64	

Abbreviations: SBS, shear bond strength; SE, self-etch.
[a] The significance value.

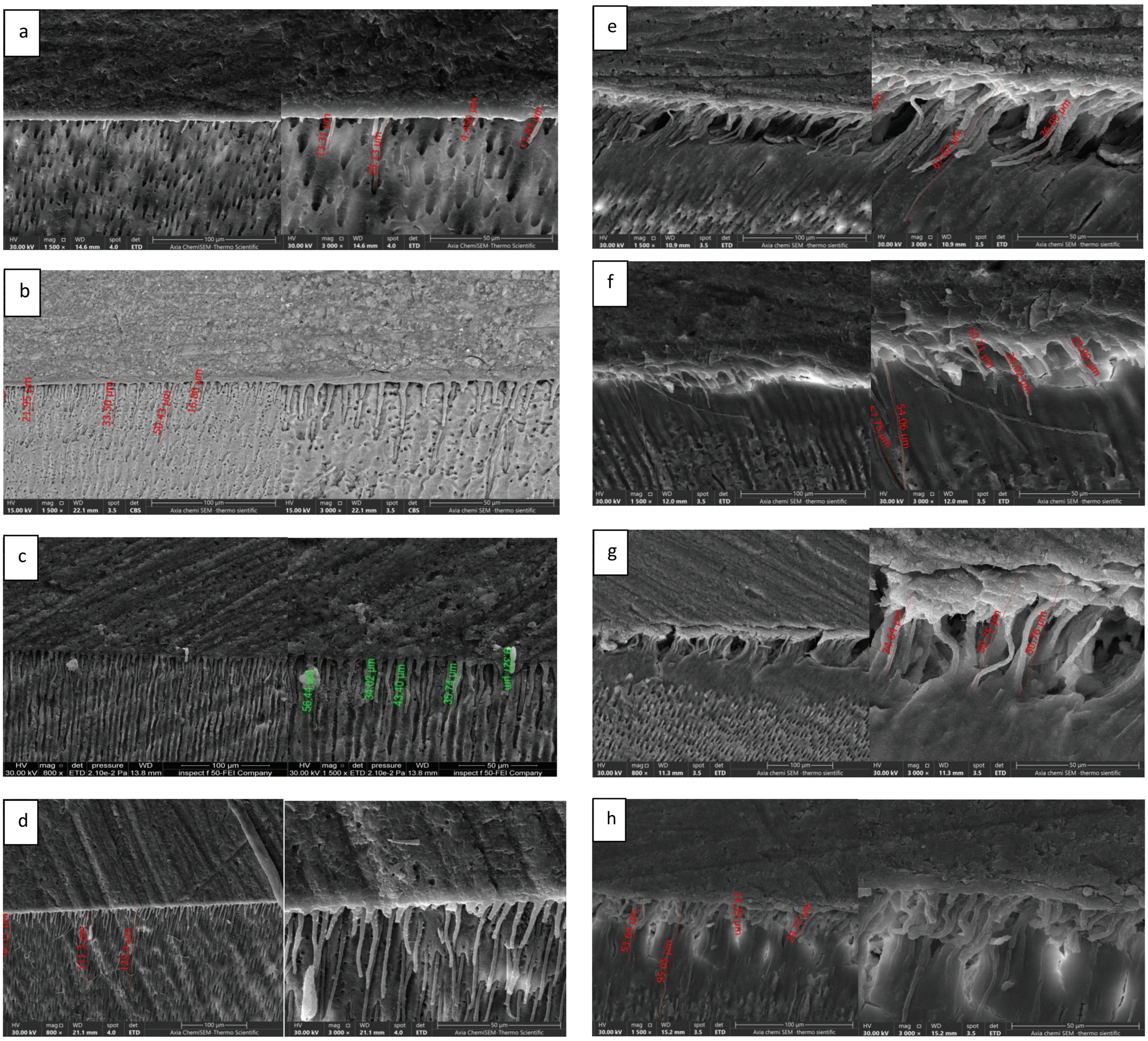

Fig. 3 Representative scanning electron microscopy (SEM) micrograph showing resin sound dentin interfacial morphology of the TE and SE systems with and without activation: (**a**) SE without activation, (**b**) SE with activation, (**c**) TE without activation, (**d**) E with activation, (**e**) SE eroded without activation, (**f**) SE eroded with activation, (**g**) TE eroded without activation, (**h**) TE eroded with activation.

layer.[31,32] This is demonstrated by the high bond strength for sound dentin when compared with eroded dentin.[10]

This study uses citric acid to mimic the erosion process because of its reproducibility, pH control, and simplicity.[33] In this study, eroded dentin has a bond strength lower than sound dentin, especially for the TE bonding strategy; however, it was nonsignificant statically, which may be attributed to the composition of the adhesive, which contains polyalkenoic acid copolymer copolymer, which can bind chemically with calcium that is left after dentin substrate dissolution.[13] This study also demonstrates the SE adhesive performs well in eroded dentin when compared with the TE strategy. This may be attributed to no discrepancy between the demineralization of low pH acid and penetration of resin monomer, which causes less exposure to collagen fibril and a more stable hybrid layer.[34] Furthermore, because these adhesives include functional monomers like 10-MDP that can chemically interact with calcium that is left after the erosion process. This may form that so-called nano layering phenomenon that increases bond strength. These confirm the findings of Jang et al, who reported that bond strength was material dependent and best for adhesives containing functional monomers like 10-MDP.[19] Regarding the adhesive system used in the current study, there was significantly higher bond strength with the TE bonding strategy than the SE strategy in sound dentin. In contrast, in the eroded dentin, there was no significant difference between these adhesive systems; this is consistent with the result of Zimmerli et al,[32] who demonstrated that the TE adhesive system has a superior bonding performance in both sound and eroded dentin, as compared with the SE adhesive system although the difference was not significant.

One of the reasons for decreasing bond strength in eroded dentin is the low or difficult infiltration of monomer to the dentin substrate.[10] There is agitation of the adhesives during their application to dentin surface as a modality to overcome the difficulty of bonding to eroded dentin; these attempts were made to meet the recommendations of the manufacturers.[35,36]

By sonic agitation, there was a significant improvement in bond strength in both TE and SE bonding systems with variable degrees. This achievement was explained by increased fluid dynamics of adhesive on dentin surface by pressure waves and imparting energy that increased the diffusion of the resin monomer into dentin in both sound and eroded dentin.[37] This can be ensured by a hybrid layer analysis with SEM. In all groups, a more uniform and thicker hybrid layer was seen, with a noticeable increase in the number and length of resin tags. The SEM images of the tested eroded dentin groups showed cracks and spots with no resin infiltrates. This area arises from the collagen fibril that dissolved by sodium hypochlorite or hydrochloride during sample preparation for SEM examination where the monomer failed to infiltrate. The application of sonic agitation resulted in more a uniform hybrid layer with much less empty spaces with no monomer infiltration. These results may be attributed to the increase in the mechanical displacement of the smear layer when compared with the nonactivation groups that results from increased penetration ability of the adhesives when applied on dentin. On the other hand, these improvements may be related to the enhancement of adhesive solvent evaporation during activation of adhesives by micro-brush. These findings also confirm the fact that solvent evaporation is necessary to increase the resin–dentin bond.[38] Our result may conform to the findings of Jang et al, who tested the effect of various agitation methods on 2-hydroxyethyl methacrylate (HEMA) free SE adhesive. Single-step adhesives are simple to use but have low bonding durability in comparison to other adhesives because of the hydrophilic monomers that increase the nano-leakage. To decrease this nano-leakage, there are clinical suggestions such as prolonged adhesive application time, active agitation, or ultrasonic agitation, which can enhance the chemical interaction between adhesive and dentin substrate by scattering the etching by-product into the hybrid layer that increases bond strength.[19] Jang et al also showed, in SEM image, entrapment of water blisters in the hybrid layer when there was no agitation to dentin, but decreasing or absence of water blister was found when there was active agitation or ultrasonic agitation.[19] Miyazaki et al showed that active agitation of the adhesive improves the bond strength of three-step TE adhesives compared with passive agitation.[39] do Amaral et al reported that when vigorous agitation was applied to a single-step SE adhesive, there was an increase in bond strength; this was attributed to the increased monomer diffusion and evaporation of the solvent. Ultrasonic agitation can also increase the bond strength of SE adhesive because ultrasonic agitation induces acoustic streaming that increases the infiltration of adhesive to the dentin substrate.[36] Contrary to our study, Jacobsen and Söderholm who tested the effect of agitation to HEMA, water, and acetone primer in wet and dry dentin before the application of adhesive in the three-step adhesive system found no difference in bond strength in HEMA and water-based primer with and without agitation. They also found a low bond strength when the acetone-based primer was agitated. They claimed that agitation caused an increase in the rate of evaporation of solvent, which turns the adhesive into a jelly structure and thus cannot infiltrate the spaces within the collagen fibril.[40]

A limitation of this study is that it is an in vitro study with no clinical trial. The study did not account for the effect of thermocycling on the SBS of composite to dentin under erosion stimulation, nor did it assess the pulp response, which represents a defense mechanism. In addition, the study did not investigate potential alterations in the oscillating frequency of the sonic micro-brush when applied on the dentin surface, which can reduce its efficiency. Also, the sonic agitation frequency and its effect on adhesive spattering during activation need further investigation.

Conclusion

Despite the limitations of the current study, sonic agitation can be recommended for adhesive application in sound and eroded dentin for both SE and TE bonding systems, as it significantly improves the bond strength, especially for the TE bonding system when used to bond eroded dentin. It improves the infiltration of resin, quality of the hybrid layer, and the length and number of resin tags.

Ethical Approval

The research ethics committee of the College of Dentistry, University of Baghdad, Baghdad, Iraq, approved this research project (Ref. no.: 912; date: January 19, 2023).

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Conflict of Interest

None declared.

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