Shear bond strength of composite resin to dentin after application of cavity disinfectants – SEM study

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Abstract

Aim: The aim was to evaluate the effect of different cavity disinfectants on dentin bond strengths of composite resin applied with two different adhesive systems. Materials and Methods: Two-hundred mandibular molars were sectioned parallel to the occlusal surface to expose dentin in the midcoronal one-third. The dentinal surfaces were polished with waterproof-polishing papers. The specimens were randomly divided into five groups of 40 teeth each as follows: group 1(control) -- specimens were not treated with any cavity disinfectants. Groups 2--5 (experimental groups) -- dentin surfaces were treated with the following cavity disinfectants, respectively; 2% chlorhexidine solution, 0.1% benzalkonium chloride-based disinfectant, 1% chlorhexidine gel, and an iodine potassium iodide/copper sulfate-based disinfectant. The specimens were then randomly divided into two subgroups including 20 teeth each to evaluate the effect of different bonding systems. Dentin bonding systems were applied to the dentin surfaces and the composite buildups were done. After the specimens were stored in an incubator for 24 hours, the shear bond strength was measured at a crosshead speed of 1 mm/min. The specimens were then statistically analyzed. Statistical Analysis Used: One way analysis of variance and Tukey-HSD tests were used. Results: There was no significant difference between chlorhexidine gel and control groups regardless of the type of the bonding agent used (P>0.05). On the other hand, pretreatment with benzalkonium chloride-based, iodine potassium iodide/copper sulfate-based disinfectants or chlorhexidine solutions had a negative effect on the shear bond strength of self-etching bonding systems. Conclusions: The findings of this study suggest that when benzalkonium chloride-based, iodine potassium iodide/copper sulfate-based disinfectants or chlorhexidine solutions are used as a cavity disinfectant, an etch-and-rinse bonding system should be preferred.

Keywords: Benzalkonium chloride based disinfectant, composite resin, cavity disinfection, CHX gel and solution, iodine potassium iodide/copper sulfate based disinfectant

Introduction

Restorative procedures such as cavity preparation are used to remove the infected dentin and make space for the restorative materials. The successes of these procedures depend on the effective removal of infected dentin, prior to the placement of the restorative material.

Any leftover bacterial remnants during and after the cavity

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preparation pose one of the major problem in restorative dentistry.^[1] Failure to mechanically remove the infected tooth structure and achieve complete sterilization of the cavity preparation can lead not only to microleakage, increased pulp sensitivity, and pulpal inflammation, but also to secondary caries, necessitating replacement of the restoration.^[2,3]

Therefore, after removal of the carious dentin, it is important to eliminate any remaining bacteria that may be present on the cavity walls, in the smear layer, at the enamel-dentin junction, or in the dentinal tubules. [4] Histological and bacteriologic studies have shown that only a small proportion of the teeth are sterile after cavity preparation and that bacteria left in the cavity preparation could survive for longer than a year.

Today, the application of disinfectants after cavity preparation and before tooth restoration is gaining acceptance as they are considered to eliminate potential risks due to bacterial activity.^[5,6] For two decades, many chemicals have been tested as cavity disinfectants, including chlorhexidine digluconate (CHX), benzalkonium chloride, and iodine potassium iodide/copper sulfate, etc.^[7,8] However, there is concern about the use of cavity disinfectants with dentin bonding agents, since they may have an adverse effect on the bond strength of the composite resins. The objective of this study was to compare the effect of the most commonly used cavity disinfection materials on the shear bond strength of composite with either

a two-step self-etching or an etch-and-rinse bonding system.

Materials and Methods

Two hundred human permanent mandibular molars, free of cracks, caries, and restorations on visual inspection, were used for the study. The teeth were scraped of any residual tissue tags, kept in a 2.6% sodium hypochlorite solution and rinsed under running water for 15 minutes each. Later, they were cleaned with pumice and stored in normal saline at 40°C until use.

The teeth were sectioned with a low-speed diamond disk saw (Markus Inc., Michigan, USA) under water coolant to expose mid-coronal dentin. The sections of the teeth including the roots were embedded in autopolymerizing acrylic resin to form cylinders 2.5 cm in diameter and 5 cm high. Dentin surfaces were flattened using 600-800 and 1200 grit waterproof polishing papers.

The teeth were randomly divided into five main groups of 40 teeth each. In group 1, the specimens were not treated with any cavity disinfectant and served as control. The teeth in experimental groups were treated with one of the following cavity disinfectants: 2 % CHX solution (Consepsis, Ultradent USA), 0.1% benzalkonium chloride-based disinfectant (Tubulicid Red, Dental Therapeutics AB, Sweden), 1% CHX gel (Hibitane Dental), or an iodine potassium iodide/copper sulfate-based disinfectant (Ora-5, McHenry Labs, Texas, USA) for 20 seconds. The dentin surfaces of the teeth were then dried with air for 10 seconds. Each group was then randomly divided into two subgroups of 20 teeth each according to the bonding agent used, either Clearfil SE Bond (Clearfil SE bond, Kuraray, Osaka, Japan) or Prime & Bond NT (Dentsply Caulk, Milford, DE, USA).

For the Clearfil SE Bond system (Clearfil SE Bond, Kuraray), primer was applied to the dentin surface using a sponge supplied by the manufacturer and rubbed for 20 seconds. The dentin surface was then dried with oil-free compressed air. After this etching and priming step, the SE bonding agent was applied to dentin surfaces and light cured for 20 seconds (Spectrum 800, Confident India Pvt. Ltd.).

For Prime & Bond NT (PBNT) (Dentsply Caulk), the dentin surface was etched with 34% phosphoric acid gel (Dentsply Caulk) for 20 seconds, rinsed for 20 seconds with an air--water syringe and dried with compressed air. Then, Prime & Bond NT was applied, left undisturbed for 30 seconds, lightly air dried for 2 seconds, and light cured for 20 seconds. After adhesive application, the specimens were clamped in the Ultradent Bonding Jig (Ultradent Products; South Jordan, UT, USA). A hybrid restorative composite (Clearfil APX Shade A2, Kuraray) was carefully inserted into the surface by packing the material into cylindrical-shaped plastic matrices with an internal diameter of 2.30 mm and a height of 3 mm. Excess

composite was carefully removed from the periphery of the matrix with an explorer. The composite was incrementally cured with a quartz halogen curing light (Spectrum 800, Confident India Pvt. Ltd.) for 60 seconds.

After storing in an incubator at 37°C in 100% humidity for 24 hours, the specimens were placed in a universal testing machine and the shear bond strength was measured at a crosshead speed of 1 mm/min. The shear bond strength of composite resin to dentin was recorded in Newtons (N) and calculated in MPa taking into account the cross-sectional area of the composite buildup. The mean and standard deviation were calculated for the different groups. One way analysis of variance and Tukey HSD tests was performed to determine significant differences in bond strengths between the groups. After the testing procedure, the fractured surfaces were observed with a dissecting microscope (SZ-TP Olympus; Tokyo, Japan) at a magnification of 20× to determine failure modes and classified as adhesive failures, cohesive failures within the composite or cohesive failures within the tooth. One specimen from each group was sputter coated with gold after fracture and prepared for SEM examination. Coated specimens were then observed under the SEM (JMG-5600, KEOL; North Carolina, USA) with different magnifications.

Results

The mean and standard deviations of shear bond strengths for each group are presented in Table 1. Effect of cavity disinfectants on the shear bond strength of Clearfil SE Bond and Prime & Bond NT adhesive systems is summarized in Table 2.

When the results were analyzed, the mean shear bond strength values in the Clearfil SE Bond group which had been treated with CHX solution, benzalkonium chloride-based disinfectant and iodine potassium iodide/copper sulfate-(I₂KI/CuSO₄) based disinfectants were significantly lower than the other groups (P < 0.05). The shear bond strength was not adversely affected by any cavity disinfectants in groups in which Prime & Bond NT was applied after acid etching. Regardless of the bonding agent, the CHX gel used demonstrated no adverse effect on the shear bond strength of composite resin. The distribution of fracture modes observed with a dissecting microscope at a magnification $20 \times$ is shown for all groups in Table 3. The principle mode of failure in groups 2, 3, and 5 was adhesive. The other groups exhibited an equal distribution of the different failure modes. Figures 1 and 2 show the SEM micrographs of dentin surfaces after fracture in groups 2 and 3, respectively. Resin tags on dentin surfaces treated with the self-etching system were not clearly visible. However, in Figures 3 and 4, dentin surfaces which were treated with CHX solutions (group 7) or chlorhexidine gel (Group 9), respectively, showed clear resin tags when the etch-and-rinse system were used.

Table 1: Composition of cavity disinfectants used in the study

Product	Active ingredient	Manufacturer
Consepsis	2% Chlorhexidine gluconate	Ultradent, USA
Tubulicid red	0.1% Benzalkonium chloride,	Dental Therapeutics
	0.2% EDTA, 1% sodium flouride	AB, Sweden
CHX gel	1% Chlorhexidine gluconate	Hibitane Dental
Ora-5	0.3% lodine, 0.15% potassium iodide	McHenry Labs, Texas
	5.5% copper sulfate	USA

Table 2: Effect of cavity disinfectants on shear bond strength of Clearfil SE Bond and Prime & Bond NT adhesive systems.

Group	N	Clearfil SE bond mean (MPa±SD)	Prime N bond Mean (MPa±SD)
Control	40	20.99ª (4.94)	21.08ª (3.94)
2% CHX	40	16.89 ^b (2.47)	21.87ª (2.16)
0.1% Benzalkonium chloride based cavity disinfectant	40	15.67 ^b (2.41)	21.59a (2.73)
1% CHX gel	40	20.87ª (4.59)	21.69 ^a (3.60)
I2KI /CuSO⁴-based cavity disinfectant	40	15.44 ^b (2.89)	21.62a (2.36)

There was no statistically significant difference in the groups having the same superscript letters

Table 3: Failure modes of the all test groups

Description	Failure adhesive (%)	Cohesive (%)	Mixed (%)
Control/clearfil SE	20	40	40
2% CHX/clearfil SE	50	20	30
0.1% Benzalkonium chloride-based disinfectant/clearfil SE	60	20	20
1% CHX gel/clearfil SE	30	30	40
I2KICopper sulfate based disinfectant/clearfil SE	60	20	20
Control/PBNT	30	40	30
2% CHX/PBNT	40	30	30
0.1% Benzalkonuim chloride based disinfectant/PBNT	30	30	40
1% CHX gel/PBNT	40	40	20
I2KICopper sulfate/PBNT	40	30	30

Discussion

In the present study, the benzalkonium-based iodine–potassium iodide/copper sulfate (I₂KI/CuSO₄)-based disinfectants and CHX solution decreased the bond strength when used with self etching bonding system, whereas no adverse effect was observed when used with the etch-andrinse adhesive. However, irrespective of the bonding system used, the CHX gel had no adverse effect on the shear bond strength.

Benzalkonium chloride, and I₂KI/CuSO₄-based solutions have been commonly used as cavity disinfectants in clinical practice because of their disinfecting action and wettability property.^[9,10] It has been stated that after deproteinization dentin may exhibit a very porous structure with many irregularities and anastomoses and hence promote the bond strength, but controversial results still exist.^[11-14] From various

laboratory results, it has been observed that depending on the testing methodology and the adhesive system used, dentin surface treatment with these disinfecting solutions can increase, $^{[11,13,15]}$ decrease, $^{[10]}$ or have no effect $^{[16]}$ on the bond strength. This result is in line with the study by Gwinnet *et al.* $^{[17]}$ that these cavity disinfectants had no effect on the bond strength when the etch-and-rinse technique was used.

CHX, due to its excellent antibacterial activity, has also been used as a cavity disinfectant for many years.^[18,19] CHX has an excellent rewetting capacity and a strong affinity to tooth structure which is thought to improve the bond strengths of the adhesive to dentin.^[20]

However in the present study, the CHX solution exerted an adverse effect on the shear bond strength when used with the self-etching bonding system. This observation is in accordance with results of Gurgan *et al.*^[21] that using

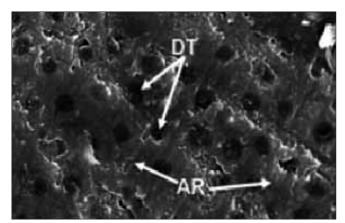


Figure 1: SEM micrograph of dentin surface treated with 2% CHX solution in group 2. Dentinal tubules were not clearly observed. DT: Dentinal tubule, AR: Adhesive resin

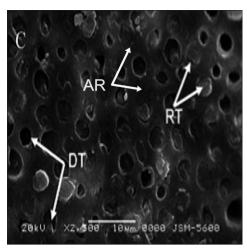


Figure 3: SEM micrograph of dentinal tubules filled with resin tags in group 7. Some dentinal tubules were left empty. DT: Dentinal tubule, RT: Resin tag, AR: Adhesive resin

chlorhexidine prior to or after etching the dentin without rinsing could adversely affect the shear bond strength of the dentin bonding agent.

Yet, few other studies showed that applying chlorhexidine before acid etching did not significantly affect the bond strength. Our study presented a similar result for specimens treated with the etch-and-rinse system and the CHX solution.

We did an extensive research of the concerned literature but did not discover any previous study using the CHX gel as a cavity disinfectant. The use of the CHX gel in our study did not affect the bond strength of composite regardless of which bonding system was used (self-etching or etch-andrinse). This finding can be attributed to the fact that CHX gel's lack of effect on the bond strength may be due to its limited penetration into the dentin structure due to its gel form. On the other hand, the gel form may also limit the

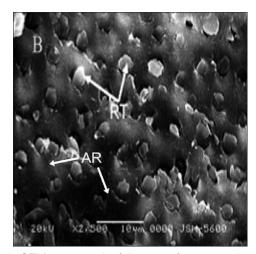


Figure 2: SEM micrograph of dentin surface treated with 0.1% benzalkonium chloride-based disinfectant in group 3. Some empty areas in dentinal tubules were observed. RT: Resin tag, AR: Adhesive resin

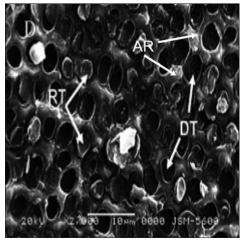


Figure 4: SEM micrograph of dentin surface treated with chlorhexidine gel and an etch-and-rinse bonding system (group 9). All dentinal tubules were completely filled with resin tags. DT: Dentinal tubule, RT: Resin tag, AR: Adhesive resin

affinity of the material to the tooth structure.

Conclusion

Within the limitations of this laboratory study, it may be stated that routinely used disinfectants, except the CHX gel, might have an adverse effect on the bond strength of selfetching adhesive systems. Thus, when clinicians decide to use benzalkonium chloride based, I₂KI/CuSO₄ based or CHX solutions as cavity disinfectants, they should prefer an etchand-rinse bonding system. However, the use of the CHX gel as a cavity disinfectant may be advisable, as it does not have any adverse effect on the bond strength of self-etching and etch-and-rinse systems. Further *in vitro* and *in vivo* studies are still needed to evaluate the effect of CHX gel for cavity disinfection.

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