



Effect of a silver diamine fluoride and potassium iodide-based desensitizing and cavity cleaning agent on bond strength to dentine

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ABSTRACT

Objectives: Recently a silver diamine fluoride and potassium iodide solution (Riva Star, SDI Australia) has been introduced for clinical use as a desensitizing agent or a cavity cleaner. Little is known whether the precipitate produced will affect adhesion to dentin. The aim of this study was to determine whether Riva Star influenced bond strengths to an etch-and-rinse (Optibond FL; Kerr, USA), 2-step self-etching (Clearfil Liner Bond F; Kuraray Noritake Dental, Japan) and all-in-one (Optibond Versa; Kerr) resin-based adhesives and a resin-modified GIC adhesive (Riva Bond LC; SDI Australia).

Methods: Human mid-coronal dentin was used; the adhesives were bonded according to the manufacturers' instructions or after the dentine surfaces had been treated with the Riva Star. Teeth were sectioned into 1 mm × 1 mm samples and subjected to a microtensile bond strength test. Results were analysed using univariate analysis of variance and Tukey HSD test.

Results: Adhesive strengths were adversely affected for all adhesives: manufacturer instructions: Optibond FL 32 MPa, SE Bond 28.5 MPa, Optibond Versa 35, Riva Bond LC 18.4 MPa; Riva Star treated groups: Optibond FL 22 MPa, SE Bond 10.9 MPa, Optibond Versa 9.6 MPa, Riva Bond LC 14.5 MPa. Only the GIC and etch-and-rinse adhesive were less effected by the Riva Star application. SEM analysis showed that Riva Star left a precipitate on the dentine surface.

Conclusions: It was concluded that if Riva Star is used as a desensitizing and cavity cleaning agent then tooth surfaces should be lightly roughened. Riva Star should not be used as a 'whole cavity' 'disinfecting' agent but may be used for spot application where a cavity floor approximates the pulp where caries-affected dentine may still exist, otherwise adhesion may be compromised.

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1. Introduction

Dentin hypersensitivity is an oral health problem that has been reported to affect from 4–57% of the population [1]. If uncontrolled, it can have a severe impact on the quality of life. Management is quite varied, ranging from the use of toothpaste [2], fluoride varnish [3] and other agents designed to occlude the open dentinal tubules to coating with resin-based adhesives and even restoration in the more extreme and intractable cases.

One agent that has been introduced as a desensitising agent and a cavity cleaner combines two solutions, silver diamine fluoride followed by potassium iodide [4]. The fluoride has the ability to remineralize early carious lesions and it is claimed that the solution has an antibacterial effect. This latter claim was demonstrated to be effective on *S. mutans* located in open dentinal tubules [5]. This antibacterial effect could be used for reducing bacterial numbers in deep carious lesions that approximate pulp prior to placing a definitive restoration.

Hence, the clinical applications of this combination solution are reasonably diverse. However, little is known about the surface effects of silver diamine fluoride in combination with potassium iodide and whether the adhesion of commonly used resin-based adhesives is affected in any way. The only paper that has investigated this factor assessed the use of a conventional glass ionomer cement (GIC) [6]. This study found that leaving the precipitate on the surface adversely affected GIC adhesion and that etching and rinsing was able to restore bond strength. Another study that

Abbreviations: GIC, glass ionomer cement; SDF/KI, silver diamine fluoride-potassium iodide; RMGIC, resin-modified GIC; PAA, polyacrylic acid; SEM, scanning electron microscopy; EDS, energy dispersive X-ray spectroscopy; CC, cohesive failure in restorative material; AD, adhesive failure; MI, mixed failure which was a combination of cohesive and adhesive failures; ANOVA, analysis of variance

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examined only the influence of silver diamine fluoride on the bonding of a self-etch or etch-and-rinse adhesive did not show any adverse effect [7]. Therefore, the aim of this work was to investigate whether application of the silver diamine fluoride-potassium iodide (SDF/KI) solution would have any effect on a 2-step etch-and-rinse system, two self-etching adhesives or a resin-modified glass ionomer cement adhesive. The null hypothesis tested was that the application of the SDF/KI solution does not affect adhesion to dentine.

2. Materials and methods

Eighty fresh non-carious human permanent molar teeth were obtained under a protocol (IRB ref. No. UW 11-355) approved by the Institutional Review Board at the University of Hong Kong. Before testing, the teeth were cleaned using an ultra-sonic cleaner and stored in 0.5% chloramine T solution at 4 °C.

2.1. Tooth specimen preparation

Human mid-coronal sound dentine was used as the bonding substrate. The occlusal enamel was removed using a low-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA). The dentine bonding surface was wet-ground with 600-grit silicon carbide (SiC) paper (Microcut; Buehler). The teeth were randomly divided into eight treatment groups.

2.2. Materials

The materials evaluated in this study are described in Table 1. Etch-and-rinse adhesive (Optibond FL; Kerr Corp., Orange, CA, USA), 2 step self-etching adhesive (Clearfil Liner Bond F; Kuraray Noritake Dental Inc., Tokyo, Japan and Optibond Versa; Kerr Corp.), and resin modified glass ionomer adhesive (Riva Bond LC; SDI Ltd., Victoria, Australia) were used in combination with the SDF/KI tooth desensitizing and cavity cleaning agent (Riva Star; SDI Ltd.). The application protocols for all the materials evaluated in this

study, as recommended by their manufacturer, are summarized in Table 2. Each adhesive system was assessed either with no Riva Star treatment (control) or in combination with Riva Star for a total of eight groups with a sample size of five teeth per group. After dentine pretreatment with or without Riva Star, the adhesives were applied and polymerized using a LED curing light (Elipar LED Curing light; 3M ESPE Dental Products, St. Paul, MN, USA) with intensity of 1200 mW/cm². After applying the adhesive, the bonded surface was incrementally covered (1 mm thick increments) with a direct micro-hybrid resin composite (Aura DC2; SDI Ltd.) restorative material or a resin-modified GIC (Riva LC; SDI Ltd.). Each increment was polymerized for 40 s using the LED curing light. The RMGIC specimens were coated with a protective varnish (Riva Coat; SDI Ltd.). All specimens were stored in distilled water at 37 °C for 24 h.

2.3. Micro tensile bond strength test

The bonded specimens were cut in the 'X' and 'Y' directions through the adhesive/tooth interface to obtain beams (0.9 mm × 0.9 mm width; 5–7 mm length) each consisting of restorative material, adhesive, and dentine. The beams were fixed in a micro tensile bond strength (MTBS) testing jig using a cyanoacrylate resin (Zapit; Dental Ventures of America Inc., Corona, CA, USA). The micro tensile bond strength was determined with a mechanical testing device (Type 4444; Instron Corp., Canton, MA, USA) at a crosshead speed of 1 mm/min. The tooth was the unit for evaluation; five teeth were tested for each group.

2.4. Micro-morphological analysis

Eight additional teeth were used to observe micro-morphological changes of the dentin surface and tubular occlusion on exposed dentin after the different procedures. The exposed dentine disks were finished with 600-grit SiC paper (Microcut; Buehler).

The dentine disks were randomly divided into four groups each containing two disks: 1) ground surface, 2) Riva Star treatment, 3)

Table 1
Materials assessed.

Material/trade name	Lot	Composition
Tooth desensitizing and cavity cleaning agent Riva Star (SDI Ltd., Victoria, Australia)	292792	Step 1: silver fluoride, ammonia solution Step 2: potassium iodide
Etch and rinse system Optibond FL (Kerr Corp., Orange, CA, USA)	5097479 5097480	Primer: HEMA, GPDM, PAMM, ethanol, water, photo initiator Adhesive: TEGDMA, UDMA, GPDM, HEMA, bis-GMA, filler, photo initiator
Gel etchant (Kerr Corp.) 2-step self-etching system Clearfil Liner Bond F (Kuraray Noritake Dental Inc., Tokyo, Japan)	5099702 AM0001	37.5% phosphoric acid, silica thickener Primer: MDP, HEMA, hydrophilic dimethacrylate, di-Camphorquinone, accelerators, water
Optibond Versa (Kerr Corp.)	AM0001 5027527 5024531	Adhesive: MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, di-Camphorquinone, accelerators, silanated colloidal silica, surface treated sodium fluoride Primer: HEMA, GPDM, hydrophilic co-monomers including mono and di-functional methacrylate monomers, ethanol, acetone, water, photo initiator Adhesive: Hydrophobic, structural, and cross-linking monomers, ethanol, photo initiator, 0.4-micro barium glass, nano-silica, sodium hexafluorosilicate
Conditioning of tooth surface Riva conditioner (SDI Ltd.) RMGI Riva Bond LC (SDI Ltd.)	292792 121131	25–30% polyacrylic acid Poly(acrylic acid), tartaric acid, 2-hydroxyethyl methacrylate, dimethacrylate cross-linker, acidic monomer, fluoroaluminosilicate glass powder
Restorative material Aura DC2 (SDI Ltd.)	132459	UDMA, TEGDMA, Bis-MEPP

HEMA: 2-hydroxyethyl methacrylate; GPDM: glycerol phosphate dimethacrylate; PAMM: phthalic acid monoethyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; bis-GMA: bisphenol-glycidyl methacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate; Bis-MEPP: bisphenol A ethoxylate dimethacrylate.

Table 2
Application protocol.

Material	Class	Application
Riva Star	Desensitizer and cavity cleaner	Silver fluoride and ammonia solution applied to dentine surface and then potassium iodide applied to dentine surface
Optibond FL	3-Step, etch-and-rinse adhesive	Application of gel etchant on dentine surface for 15 s, followed by rinsing for 15 s and gentle air-drying; Primer applied to dentine surface with light rubbing action for 15 s and 5 s air-drying; Application of the adhesive, 20 s light-curing
Optibond Versa	2-Step, self-etch adhesive	Primer applied to air dried dentine surface with rubbing action for 20 s and medium air pressure applied to surface for 5 s; Adhesive applied to primed surface with light brushing motion for 15 s and then air-thinned for 5 s; Primer/adhesive light-cured for 10 s
Clearfil liner Bond F	2-Step, self-etch adhesive	Primer applied to air dried dentine surface with rubbing action for 20 s and dried with mild air flow to surface; Adhesive applied to primed surface with light brushing motion and then created uniform bond film with a gentle air flow; Primer/adhesive light-cured for 10 s
Riva Bond LC	resin modified glass ionomer	Conditioner applied dentine surface for 10 s, followed by rinsing and air-drying; RMGIC applied to dentine surface; Light-cured for 20 s

Riva Star treatment, 37.5% phosphoric acid, and distilled water rinse, 4) Riva Star and Clearfil Liner Bond F primer. The specimens were dehydrated in a desiccator and then, sputter-coated with a thin layer of Pt–Pd alloy. The dentine surfaces from each group were then observed with a scanning electron microscope (SEM, S-3400N; Hitachi High Technologies America Inc., Schaumburg, IL, USA) using both back-scattered electron and secondary electron modes.

To verify the presence and distribution of silver fluoride on the treated dentine, energy dispersive X-ray spectroscopy (EDS, IXRF Model 550i; IXRF systems Inc., Austin, TX, USA) was utilized.

2.5. Evaluation of adhesive/tooth interface

The specimens were prepared following the same protocol for micro-tensile bond strength testing. Following storage in distilled water for 24 h at 37 °C, the bonded specimens were sectioned perpendicularly to the adhesive/tooth interface using a diamond saw (IsoMET; Buehler) under water coolant. The sectioned specimens were wet-ground with a series of SiC papers (600-, 800-, 1200-, 2400-, and 4000-grits, Microcut; Buehler) and polished using a felt and diamond suspensions (MetaDi; 3 and 1 µm, Buehler). The specimens were then ultrasonically cleaned for 1 min in distilled water. The bonding interface was subjected to an acid-base challenge using 10% orthophosphoric acid for 10 s, a distilled water wash, and 5% sodium hypochlorite solution for 5 min. The specimens were sputter coated with Pt–Pd alloy target for 90 s. The surfaces were then observed with SEM with an accelerating voltage of 15 kV.

2.6. Evaluation of failure pattern

Following microtensile bond strength testing, all of the fractured beams were observed through SEM to determine the mode of failure: cohesive failure in the restorative material (CC), adhesive failure (AD) between the tooth surface and adhesive, or mixed failure (MI), which is a combination of cohesive and adhesive failure.

2.7. Statistical analysis

Statistical analyses were performed with the application (GraphPad Prism 6 for Windows; GraphPad Software Inc., La Jolla, CA, USA). For the microtensile bond strength test, mean values and standard deviations of five specimens were calculated. The Kolmogorov–Smirnov test was primarily used for the micro tensile bond strength results. These results were analysed using the Bartlett test for evaluation of equality of variance. After checking

Table 3
Summary of two-way ANOVA for micro tensile bond strength conducted at each level of interacting factor.

Source of variation	% of total variation	P value	P value summary	Significant
Interaction	15.05	0.0009	***	Yes
"Riva Star"	48.73	<0.0001	****	Yes
"Treatments"	13.49	0.0017	**	Yes

ANOVA table	Sum of squares	df	Mean square	F (DFn, DFd)	P value
Interaction	638.7	3	212.9	F (3, 32)= 7.060	0.0009
"Riva Star"	2068	1	2068	F (1, 32)= 68.59	<0.0001
"Treatments"	572.6	3	190.9	F (3, 32)= 6.330	0.0017
Residual	965	32	30.16		

Table 4
Results of micro tensile bond strength (MPa).

	Optibond FL	Optibond Versa	Clearfil Liner Bond F	Riva Bond LC
Control	32.1 ^a (1.2)	35.0 ^a (3.9)	28.4 ^a (8.4)	18.4 ^b (5.6)
Riva Star	21.4 ^c (9.4)	9.6 ^d (2.0)	10.8 ^d (2.1)	14.5 ^{cd} (5.2)

Mean(SD); Identical lower case letters indicate values that are not significantly different (Sidak's multiple comparisons; $p > 0.05$).

Table 5
Results of pairwise comparison between Control and Riva Star.

Sidak's pairwise comparison test	Mean diff.	95% CI of diff.	Significant
Control vs Riva Star			
Optibond FL	10.67	1.501–19.83	Yes
Optibond Versa	25.38	16.21–34.55	Yes
Clearfil Liner Bond F	17.6	8.437–26.77	Yes
Riva Bond LC	3.878	–5.287 to 13.04	No

normality and equality of variance, individual two-way analysis of variance (ANOVA) and Sidak's post-hoc multiple comparisons were further applied. Two-way ANOVA was selected to analyse the data, with "Riva Star" and "Treatments" as factors. Sidak's post-hoc tests were applied for pairwise comparison between the control and Riva Star treatments and for the difference among the treatments. P -values less than 0.05 were considered statistically significant.

3. Results

3.1. Micro tensile strength

Two-way ANOVA demonstrated a significant interaction between the factors under study ($p=0.0009<0.05$) (Table 3). Therefore, two separate Sidak's tests were performed for pairwise comparison between the control and Riva Star treatments and the difference among the treatments.

The results of the microtensile bond strength tests are summarized in Table 4. The microtensile bond strengths without Riva Star treatment (Control) ranged from a minimum of 18.4 MPa to a maximum of 35.0 MPa, and they were categorized into two groups (categories a and b). Within the control group, three treatment groups showed the greatest bond strength (28.4–35.0 MPa,

category a), whereas the Riva Bond LC group showed the lowest bond strength (18.4 MPa, category b).

The bond strengths with Riva Star treatment ranged from a minimum of 9.6 to a maximum of 21.4 MPa, and they were also categorized into two groups (categories c and d). Among Riva Star treatments groups, the Optibond FL group showed the greatest bond strength (21.4 MPa, category c), whereas the self-etch adhesive groups (Optibond Versa and Clearfil Liner Bond F) showed the lowest bond strength (9.6 and 10.8 MPa, category d).

The results of pairwise comparison between the control and Riva Star treatments are summarized in Table 5. There were significant differences between the control group and the Riva Star treatments without Riva Bond LC (RMGIC) group.

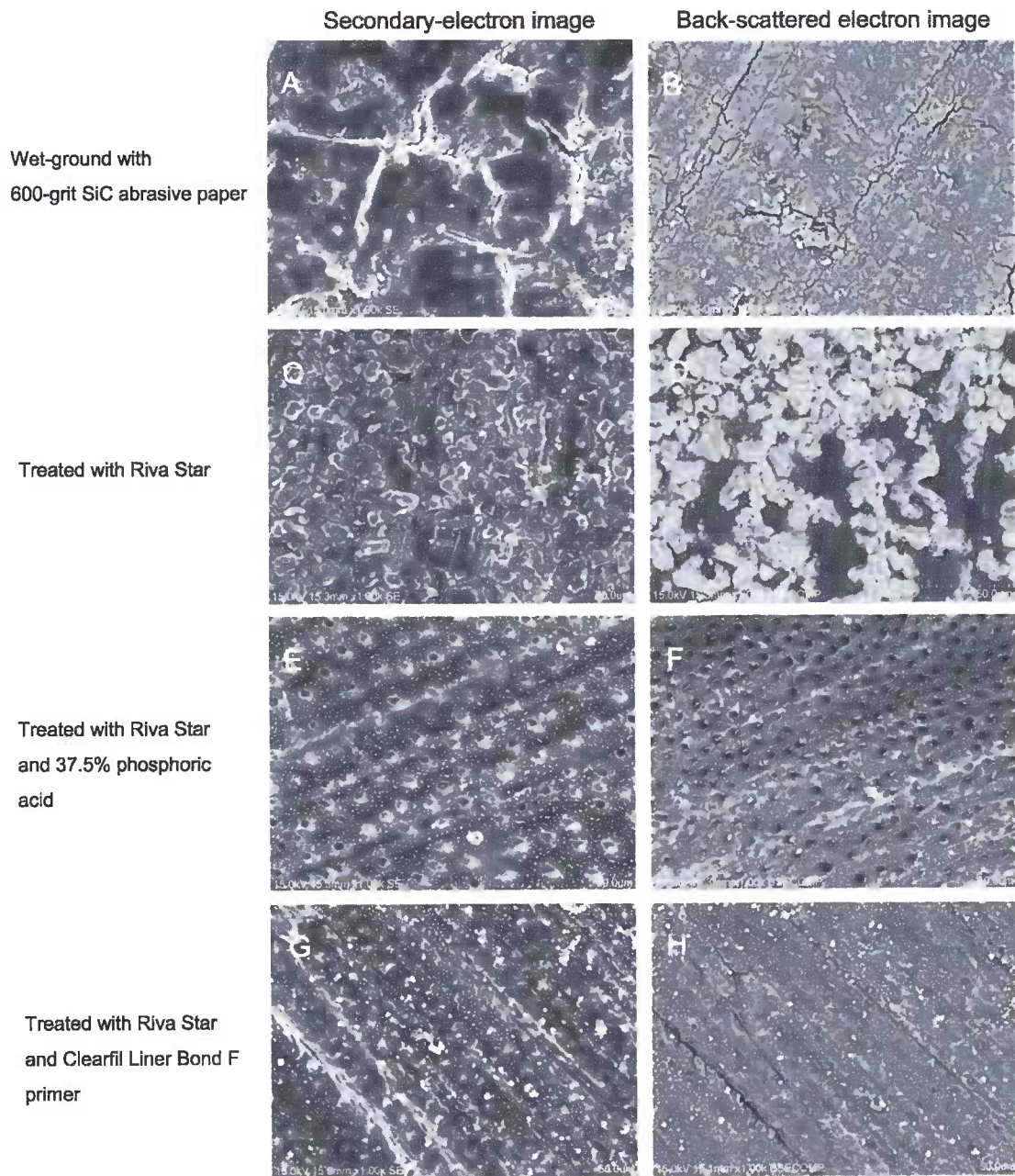


Fig. 1. SEM images of the dentine surface (A, B) wet-ground with 600-grit SiC abrasive paper, (C, D) treated with Riva Star, (E, F) treated with Riva Star and 37.5% phosphoric acid, (G, H) treated with Riva Star and Clearfil Liner Bond F primer.



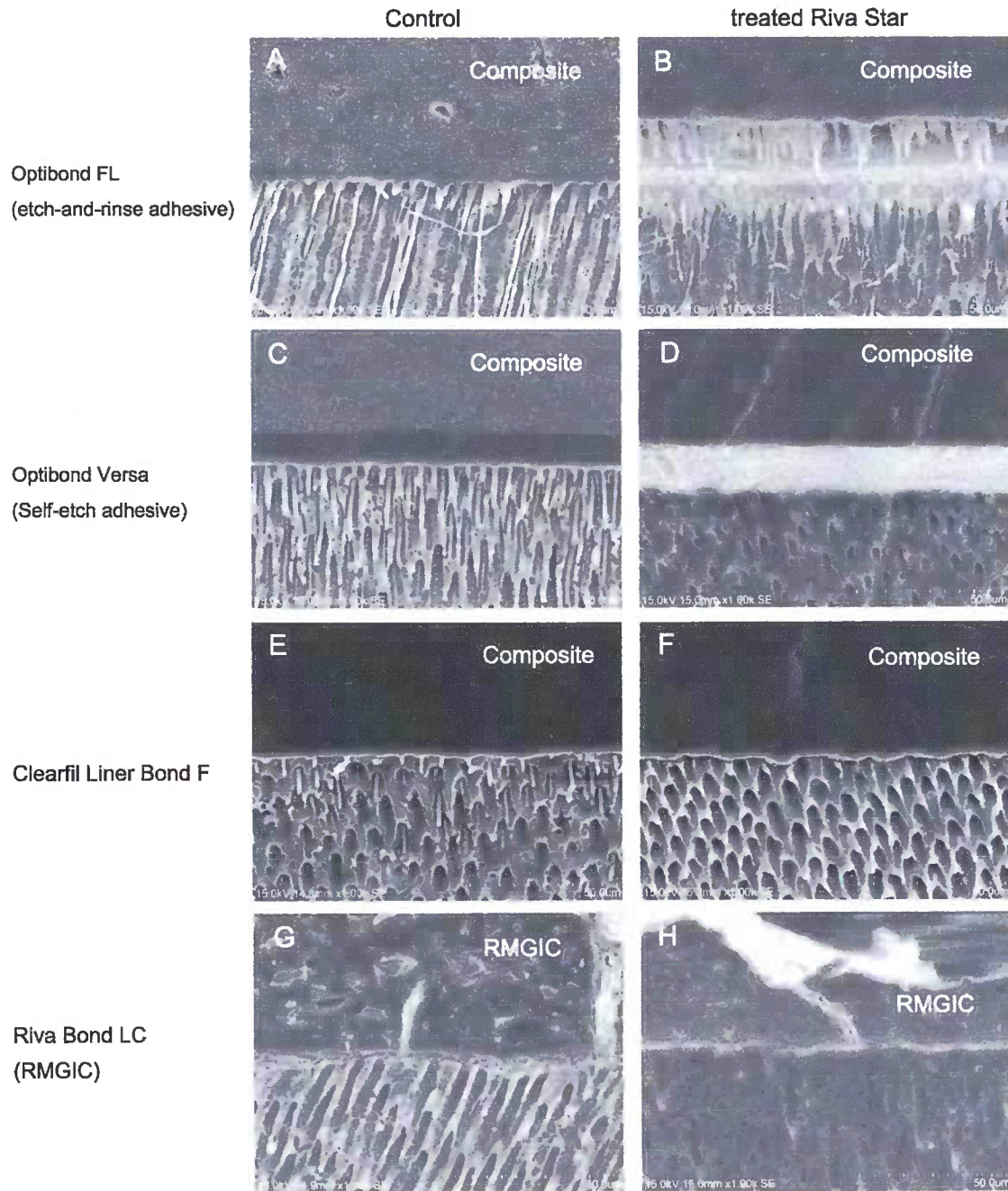


Fig. 3. SEM images of the dentine and restorative materials interface before testing; Control (first column) and treated Riva Star (second column).

interdiffusion zone seemed to be easily removed after the acid-base treatment.

For the 2-step self-etch adhesive, Optibond Versa (Fig. 3C and D) a thin resin-dentin interdiffusion zone was observed with numerous resin tags in the dentin. The SDF/KI treated dentine showed a dense precipitate on the dentin surface with no interdiffusion zone or tag formation. It appears that the SDF/KI precipitate may have interacted with the self-etching adhesive to form a dense layer on the dentin surface. For the other 2-step self-etching system (Clearfil Liner Bond F) the control surface did not show a distinct resin-dentin interdiffusion zone but there were fine resin tags noted. The SDF/KI treated surface was similar to the control however there was no resin tag formation. (Fig. 3E and F)

The RMGIC adhesive showed formation of a thin acid-base resistant layer on the control specimen however this was not

evident on the SDF/KI treated surface. The SDF/KI treated specimen showed an electron dense line at the interface suggesting that the AgF/AgI precipitate was not completely removed after the PAA conditioning.

3.3. Failure pattern

The failure mode distribution for fractured specimens with and without desensitizing treatment is summarized in Fig. 4. Representative SEM images showing the failure patterns are shown in Fig. 5. The failure patterns tended to show quite large variation between the control and SDF/KI treated surfaces. In general, all the resin-based adhesives showed much greater cohesive failure in the control groups and a marked increase in adhesive failure for the SDF/KI treated specimens. The SEM observations revealed that the

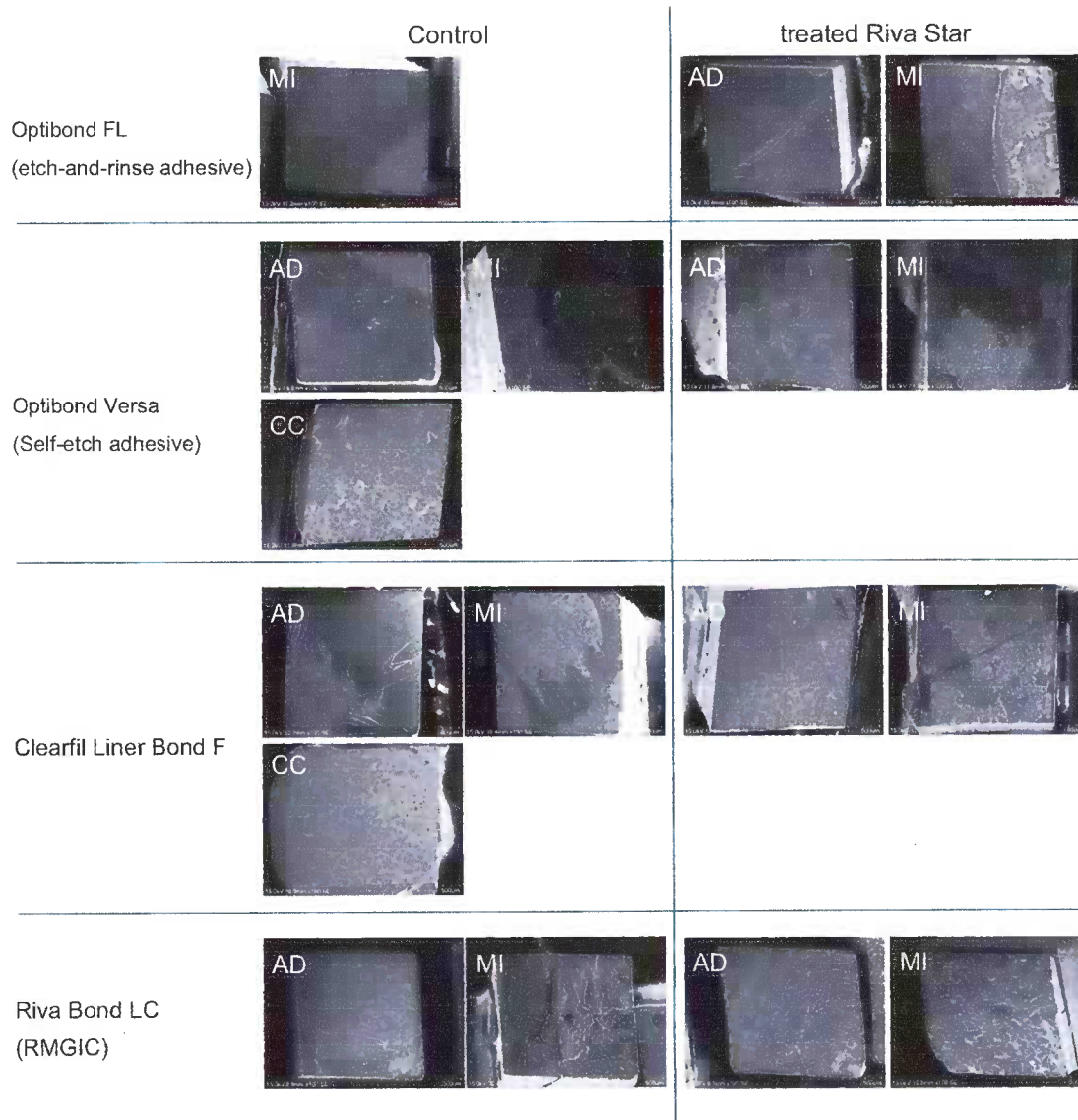


Fig. 4. SEM images of the dentine and restorative materials interface before testing; Control (first column) and treated Riva Star (second column).

cohesive failure was possibly a failure at the resin composite/-adhesive interface. This was still classified as cohesive failure as the 'bond' failed within the composite and not at the dentin-adhesive interface. For the RMGIC adhesive there was little variation between the control and SDF/KI treated surfaces.

4. Discussion

The benefit of using SDF/KI solution is that it does not stain the teeth black as occurs with SDF alone. In addition, it SDF/KI solution seems to have some antibacterial action, which may be useful for deep cavities that still contain infected carious tissue. The solution could be used as a spot application prior to placement of a temporary or definitive restoration. Based on the results of the current study, however, it is important that only the smallest area of dentine should be treated with the solution, otherwise the adhesive would most likely pull off the treated surface leaving a gap and a zone for potential fluid accumulation and continued progress of the caries. Total coverage of the bonded surface led to significant reductions in adhesive strength of all the materials. The

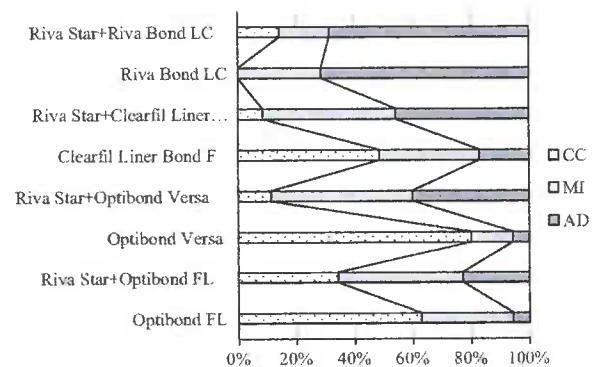


Fig. 5. Failure mode distribution for the four adhesive treatments with and without Riva Star. Modes of failure describe as cohesive failure in restorative material (CC), mixed failure (MI), or adhesive failure (AD).

groups that showed the least effect were the phosphoric acid etch group and the RMGIC group. Based on the SEM observations, it seems that phosphoric acid is able to remove most of the SDF/KI precipitate on the surface and possibly open the dentinal tubules.

However, there was still enough precipitate remaining on the tooth surface that it still adversely affected the adhesion. With regard to RMGIC, although the initial bond strength was lower than the resin-based adhesives, the change in bond strength was minimal. It would seem that the precipitate from the SDF/KI was either removed during conditioning or the PAA and RMGIC were still able to react with the underlying tooth surface or even the precipitate. The other two self-etching adhesives showed a marked reduction in adhesive strength. Although the SEM image for Clearfil Liner Bond F showed that most of the precipitate was removed by the self-etching primer, this was still not enough to allow good adhesion to occur. The pH of the SDF/KI solution is quite high, so it could also be that the residual pH on the tooth surface from the SDF/KI was so low that it prevented effective etching by the self-etching priming solution due to neutralization of the SE priming solution. For Optibond Versa, interface SEM (Fig. 3D) seemed to show an interaction of the precipitate, self-etching primer and resin with the formation of a more electron-dense layer in the back scatter image. This seemed to prevent intimate adaptation of the resin and dentin surface.

A similar phenomenon occurred with Clearfil Liner Bond F where few tags were formed in the SDF/KI treated group, again indicating that the tubules were occluded preventing resin penetration. It may also be that the SDF/KI solution prevented chemical adhesion of the 10-MDP in Clearfil Liner Bond F. It was also noted that the means increased markedly in the SDF/KI treated groups. This may lead to decreased reliability of bonding meaning that not only does the bond strength decrease overall, in the case of a larger cavity surface the bond in some locations might be good but in other areas it may be quite poor. It may also affect the clinician's ability to gain consistency in bonding.

The clinical implications of these findings are significant. It would seem that if the tooth surface has been treated with SDF/KI solution, a subsequent bonded restoration may debond quickly leading to more gap formation between the restoration and the tooth surface. Both scenarios could lead to premature restoration failure.

It is therefore recommended that the SDF/KI tooth surface should be lightly ground with a diamond bur to remove the precipitate layer. It may also be advantageous to undertake a brief etch of the surface if self-etching or universal bond systems are being used. However, this recommendation still remains to be tested. Alternatively, the surface could be conditioned with a polyacrylic acid conditioner, then glass ionomer cement or RMGIC could be placed as either a base/lining or definitive restoration. A paper by Knight et al. showed that if the surface was etched and then the SDF/KI solution was washed, the bond strength of glass ionomer cement was not adversely affected [6].

Due to the results of this study, it would be useful to determine whether the use of SDF alone leads to the same reduction in bond strength. A study investigating the adhesion of resin-based luting cements has shown that SDF solution did reduce the bond strength significantly compared with other desensitizing solutions [8]. Further work is needed on this finding.

5. Conclusions

The null hypothesis must be rejected. All dentin surfaces treated with SDF/KI and then bonded showed a significant deterioration in bond strength. Hence, care must be taken on teeth treated with this solution if a restoration needs to be subsequently placed.

Conflicts of interest

The authors declare that they have no conflicts of interest related to this study.

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