

Evaluation of the Bonding Procedure of Clearfil SE Bond in Primary Teeth: A High Resolution SEM Study

SUMMARY

The introduction of adhesive techniques and new materials gradually change operative dentistry. Self-etching primers are widely used in permanent teeth, as well as in primary teeth, under composite and compomer materials. The aim of the study was to investigate the resin-dentine interface of an adhesive system Clearfil SE Bond (Self-etching primer + bonding agent, KURARAY) in primary teeth using High Resolution Scanning Electron Microscope. Freshly extracted caries-free human deciduous molars were used in the study. Standard class V cavities were restored using Clearfil SE Bond according to the manufacturer's instructions. Adhesive was also applied upon total etching for 15s by using a 10% poly-acrylic acid, as a different protocol. Teeth were then restored with Compoglass F (VIVADENT) compomer restorative material.

The interface images of the specimens, micro-morphologically analyzed by SEM, showed that the Clearfil SE Bond, as a dentine adhesive system, had a homogenous hybrid layer, and also exhibited similar characteristics regarding resin penetration in deciduous teeth. When poly-acrylic acid conditioning was applied, the smear plugs were removed. The total etching technique revealed open tubules and resin tag formations. Micro-porosities were filled with resins and thus revealed a good adaptation of the compomer to enamel. It was concluded that Clearfil SE Bond as dentine adhesive system could be used successfully under compomer restorative materials in primary teeth. However, removal of the smear plugs by poly-acrylic acid etching could further improve the adhesion to dentin and enamel.

Key Words: Bonding; Primary Teeth; Clearfil SE Bond; Enamel; Dentin; SEM

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Introduction

Since Buonocore has introduced the acid-etching technique, which provides mechanical bonding between acrylic resin restorations and treated enamel surfaces via micro-mechanical retention, the use of adhesives in dentistry presented good results in the last decade^{1,2}. Successful clinical results of acid conditioning of enamel led to early effort of dentin bonding to include the use of acid-etching on dentin with the purpose to obtain adhesion. The resulting etch pattern is characterized by abundant micro-porosities, which allow the penetration of polymerizable monomers into those porosities to form resin tags that provide micro-mechanical retention³. Successful attempts of bonding to dentin, in a similar fashion, have been reported more recently^{2,4}. Due to the specific prop-

erties of human dentin, such as the tubular structure and its intrinsic wetness, bonding to dentin is more difficult to achieve than bonding to enamel. The bonding mechanism of recent dentin bonding agents is based on the penetration of ambiphilic molecules into acid-etched dentin. Water-chasing solvents, such as ethanol or acetone, are commonly utilized to facilitate penetration of the monomers and to obtain a direct contact of resin with the collagen fibers, which results in a mixed zone of polymerized resin and entangled collagen fibrils, the hybrid layer^{2,5}. Dentin bonding agents that rely on dentin hybridization have been reported to result in higher bond strengths particularly when the dentin had been left moist rather than desiccated with air⁵. Self-etching primers condition and prepare enamel and dentin simultaneously without rinsing. The mild acidity of these materials is responsible for their

inability to remove the smear plugs upon conditioning. These materials have the ability to dissolve hydroxyapatite partially, both within the smear layer and the dentin surface, resulting in a resin-infiltrated zone with entrapped minerals^{3,4}.

Modern adhesive restorative techniques may include the use of materials classified as polyacid-modified resin composites, known as compomers. Compomers are a combination of a matrix of acidic hydrophilic oligomers with glass particles filler (providing fluoride release) and dimethacrylate monomers that contain acidic and acrylic groups. An important parameter influencing the clinical outcome of restorative materials is the ability to bond the dentin. The compomer adhesive systems usually include the etching agent in the primer/adhesive component, thus generating a self-etching/priming/bonding solution^{1,6,7}. Self-etching/priming/bonding systems contain weak organic acids (such as maleic, oxalic, and citric acid, phenyl-p, MDP, amino-acid, iminodiacetic acid etc) that are used to condition the enamel/dentin substrate (instead of the 35-37% phosphoric acid, which is commonly used in the composite adhesive systems). The use of compomers in the absence of an acid pretreatment is controversial, and some studies reported a lower bonding strength to enamel¹.

Clearfil SE Bond® (Kuraray Co Ltd, Osaka, Japan) is a resin based dental adhesive system, which consists of a self-etching primer and a bonding agent. The primer offers simultaneous treatment of both dentin and enamel. The aim of this study was to investigate micro-morphologically the resin-dentin interface of Clearfil SE Bond as a self-etching primer-bonding system in primary teeth by using a high resolution scanning electron microscope.

Materials and Methods

Freshly extracted caries-free 10 human deciduous molars were used in the study. Teeth were kept in 0.1% tymol solution at room temperature for 1 week and washed under tap water. Teeth were randomly divided into 2 groups. Standard Class V cavities were prepared on the buccal surfaces by using diamond burs (Northbel FG Diamonds, 836/12, Italy).

In the group A, Clearfil SE Bond was applied according to the manufacturer's instructions and a compomer material was used (Compoglass F, Vivadent). Clearfil SE Bond was applied as follows: Primer was applied to the entire cavity wall with a disposable brush tip, left for 20 seconds, and evaporated the volatile ingredients with a mild oil-free air stream. Afterwards, bond was applied to the entire surfaces of the cavity with a disposable brush tip, and the bond film was made uniform by using a gentle oil-free air stream, light cured for 10 seconds with a vis-

ible light curing activator. Compomer was then placed to the cavity and light cured for 40 seconds.

In the group B, dentin conditioner (10% polyacrylic acid, Ketac Conditioner, ESPE, Germany) was applied to the cavity for 15 seconds, rinsed with water, gently dried for 10 seconds and Clearfil SE Bond was applied according to the manufacturer's instructions, as described in the group A, and compomer was then placed to the cavity and light cured for 40 seconds.

5 primary molars were used for each group, and after finishing of restorations, the teeth were stored in saline solution for 5 days at 37°C. 3 teeth were then divided mesio-distally for each group. The surfaces were smoothed and decalcified with 5% hydrochloric acid for 45 seconds, rinsed with water for 60 seconds to remove smear layer developed during the slicing and smoothing procedures and visualize resin tags and hybrid layer, as described by Macari et al⁸. Finally, the samples were washed for 2 min with distilled water and desiccated with 90% alcohol, stored overnight at room temperature, and mounted on stubs. They were then sputtered with gold-palladium (Magnetron Sputter Coater, VG Microtech) and finally examined in Philips XL Series High Resolution Scanning Electron Microscope (XL 305 FEC, Philips, The Netherlands).

Results

The SEM evaluation showed the presence of the hybrid layer and short resin tag formations when Clearfil SE Bond was applied according to the manufacturer's instructions in primary teeth (Fig. 1). The tubule orifices were obliterated by tubular plugs, and the smear layer, which was uniformly present on the dentin surface of the non-etched samples, was only partially dissolved. An adequate penetration of the material and compomer was present although the smear layer was slightly removed from the dentin surface (Figs. 2 and 3). The inter-tubular dentin showed no evidence of superficial demineralization. When polyacrylic acid conditioning was applied, tubular plugs and smear layer were totally removed, and an excellent adaptation with the compomer was observed (Figs. 4 and 5).

The findings of this study have also shown that polyacrylic acid conditioning had revealed open dentin tubules, and resin tag formations inside the dentinal tubules were more visible. Collagen fibres were also penetrating to tags, which promoted penetration of the material to dentin (Figs. 6 and 7).

After application of the Clearfil SE Bond in the group A, morphology of enamel crystals was similar in comparison with the group B, where polyacrylic acid conditioning was applied (Figs. 8 and 9). A good penetration between compomer and enamel was observed in both

groups. However, samples etched with 10% polyacrylic acid showed typical dissolution of enamel crystals revealing the crystal regular pattern in the enamel prisms. The enamel surface showed large areas of dissolution between the crystals, and high magnification images revealed crystal details and several small globular particles (Figs. 10-12).

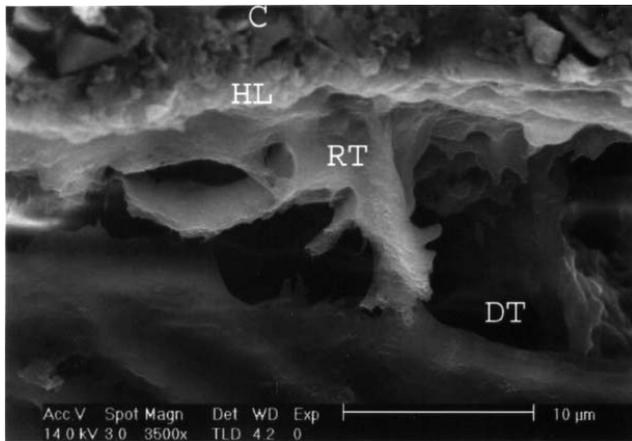
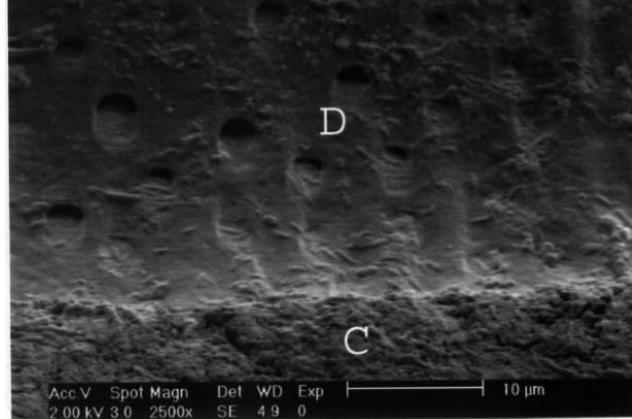
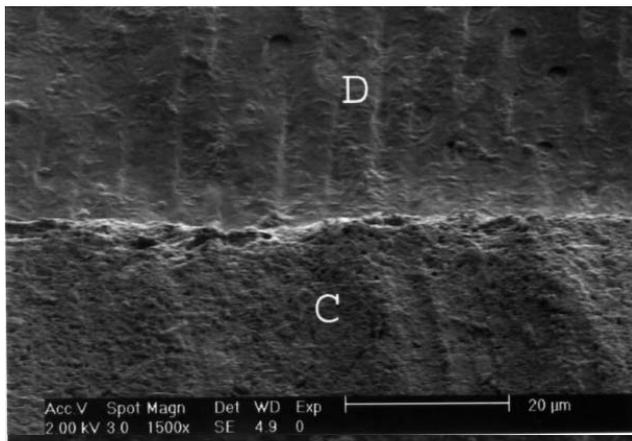
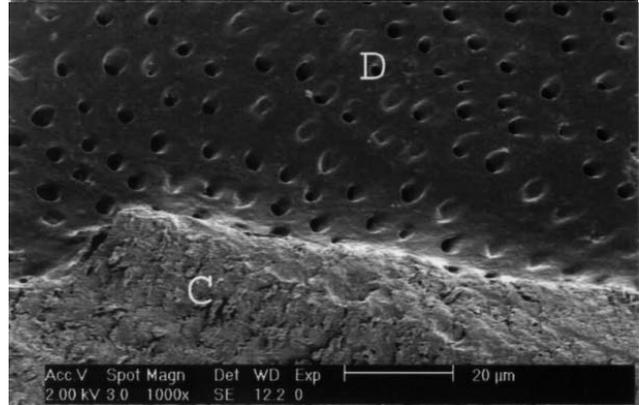
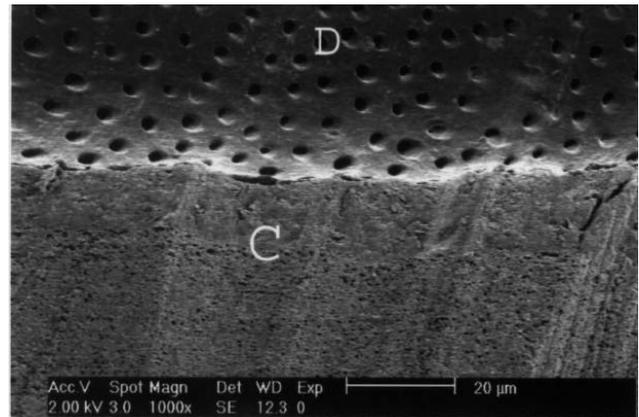


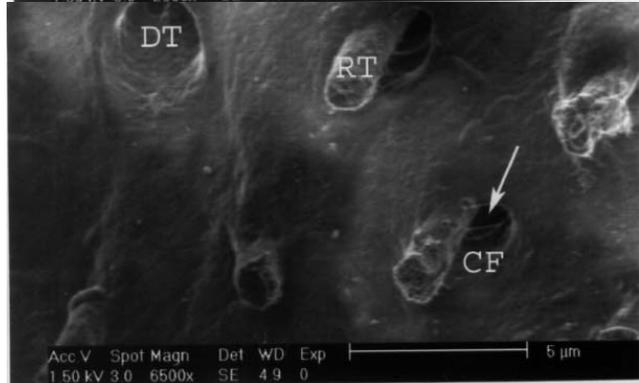
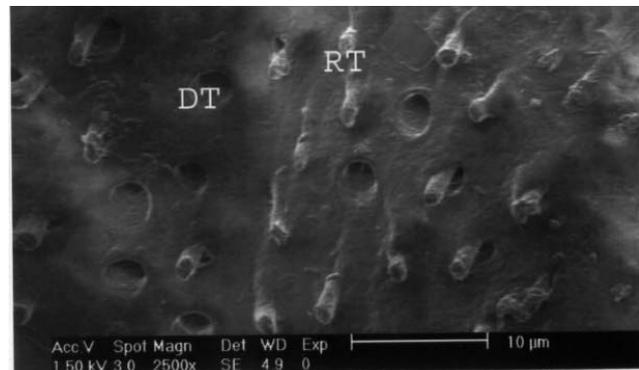
Figure 1. SEM resin-dentin interface formed by Clearfil SE Bond in group A. A homogenous hybrid layer (HL) is visible, about 3-4 μ thick, and short resin tags. Hybrid layer (HL), resin tag (RT), compomer (C), dentin tubules (DT)



Figures 2 and 3. SEM view of compomer-dentin penetration in group A. Note the adequate penetration with the material, although smear layer is slightly present on the dentin surface (C – compomer; D - dentin)



Figures 4 and 5. Polyacrylic acid conditioning has revealed open dentin tubules (DT) and perfect penetration between compomer (C) and dentin (D) in group B



Figures 6 and 7. Open dentinal tubules (DT) and more resin tag (RT) formations were present formed by Clearfil SE Bond after polyacrylic acid conditioning in group B. Collagen fibres (CF) penetrating to resin tags (arrowhead)

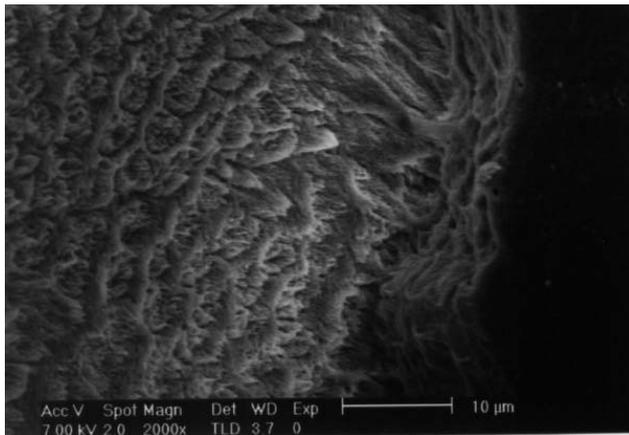


Figure 8. A good penetration between compomer and enamel was found in group A (without polyacrylic acid conditioning)

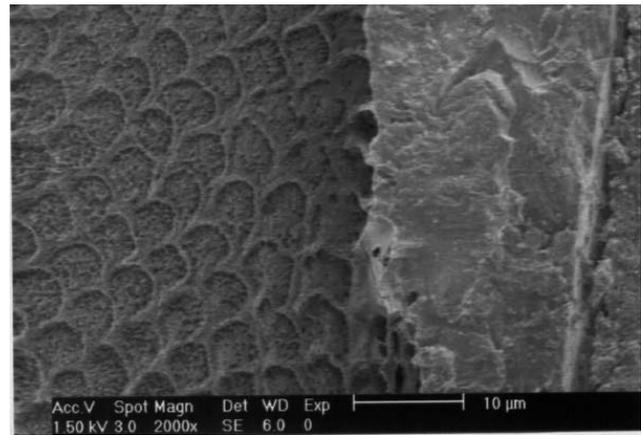
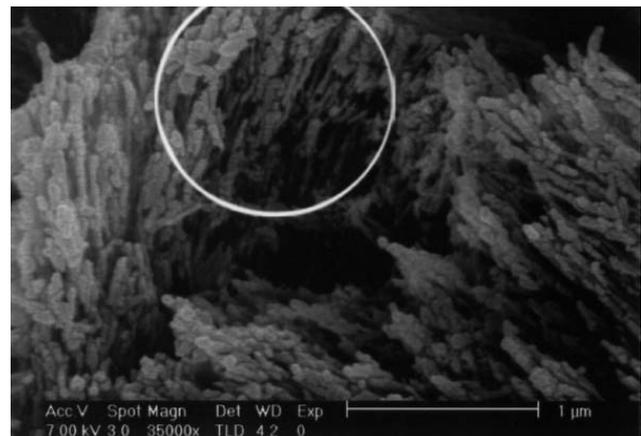
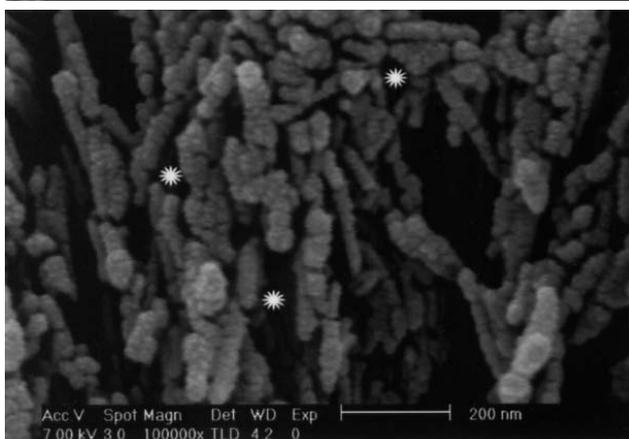
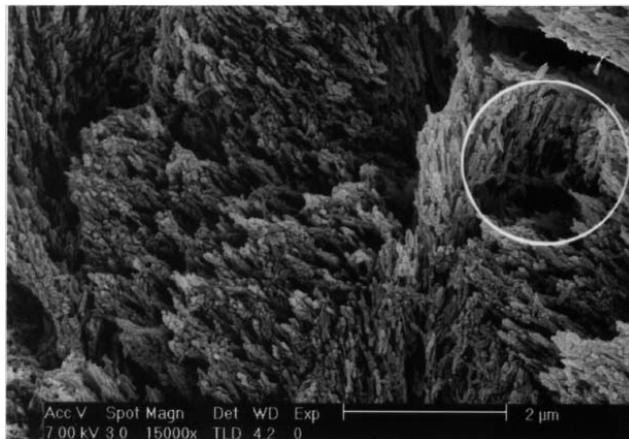


Figure 9. Samples conditioned with 10% polyacrylic acid (group B), showed typical dissolution of enamel crystals, revealing the crystal regular pattern in the enamel prisms. Note the perfect penetration of compomer to enamel



Figures 10, 11 and 12. Etching pattern after application of polyacrylic acid for 15 s on primary teeth enamel (group B). Higher magnification shows empty areas (*) between the crystals, available for fluid resin infiltration

Discussion

Many factors can influence the bonding performance of adhesive systems to dentine and enamel. The use of compomers in modern adhesive dentistry is well described and several trials revealed good clinical and laboratory performances of these materials. The polyacid modified

composite resin, or so-called compomer, consists of fluoro-aluminosilicate glass embedded in a polymeric matrix. The glass particles are partially silanized to bond the resin matrix, which is a light activated polymerization reaction of modified methacrylate monomers^{1,9,10}. Clinical trials on compomers (Dyract and Compoglass) demonstrated less micro-leakage and better clinical per-

formance when compomer primers were applied on a phosphoric acid-etched enamel surface (Prime and Bond 2.0, Dentsply DeTrey, Germany), and (Syntac Single Component, Vivadent, Liechtenstein) instead of using their own primer/adhesive agent. Self-etching primers, such as Clearfil SE Bond, involve the incorporation of smear plugs in the resin tags, thus forming a continuum in the substrate¹.

Although many different types of conditioners, primers and adhesive resins are used, the bonding mechanism of the various etched-dentin adhesive systems is remarkably similar. Acid etching removes the smear layer, opens the dentinal tubules, and decalcifies the inter-tubular and peri-tubular dentin. These phenomena were also present in this study when 10% polyacrylic acid conditioning was applied on primary dentin and enamel (Figs. 4-7).

Agostini et al¹¹ have reported that only Clearfil SE Bond had adequate bond strength to dentin of primary teeth, better than Prompt-L-Pop, Etch and Prime, and Prime and Bond NT. The maleic acid in the formulation of Syntac Single Component acts as the etchant of the system. It partially dissolves the smear layer and Prompt-L-Pop and Clearfil SE Bond have also the same effect on smear layer¹². However, the results of this study showed that removal of smear plugs with 10% polyacrylic acid etching could further improve the adhesion of the compomer material to dentine by resin tag formations (Figs. 6 and 7). A previous study suggested that resin tags formation might not contribute to the increase bond strength at all, thus only the hybrid layer formation, thickness and quality should be considered to explain the differences in bond strength¹³.

Toledano et al¹⁴ have reported that the use of Clearfil SE Bond in human permanent teeth resulted in significantly highest bond strengths on dentine in comparison with Etch& Prime and Scotchbond multipurpose. Pham et al¹⁵ tested the etching time effects on the bond strength of 2 adhesive systems in permanent and primary dentin and presented that shorter etching time (5 seconds, 35% phosphoric acid) did not adversely affect the bond strength to primary and permanent dentin with Single Bond (3M) and Self Etching Primer (Clearfil SE, Kuraray). Pecora et al¹⁶ have stated that Clearfil SE Bond has the ability to demineralise the smear layer and generate a strong bond comparable to one-step bonding agents. Smear layer was partly removed in this study from dentin surface when Clearfil SE Bond was applied (Figs. 2 and 3); however, total etching with polyacrylic acid removed the smear layer totally from dentin surface (Figs. 4 and 5).

In conclusion, Clearfil SE Bond, when used in accordance with the manufacturer's instructions under compomer material in primary teeth, showed a good

penetration into dentin as well as into enamel. 10 % polyacrylic acid conditioning for 15 seconds, as a modified application, has revealed open dentinal tubules, more resin tag formations and perfect adaptation of the compomer to dentin and enamel. So, it could be concluded that Clearfil SE Bond, as a self-etching primer bonding system, could be used successfully under compomer materials in primary teeth; however, removal of smear layer and plugs by polyacrylic acid conditioning could further improve adaptation of the material.

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