

**The Morphology Study of Self-
Etching Adhesive Systems**

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- **Abstract**

- **Objective:** To explore the morphology of self-etching adhesives at the composite-adhesive-dentin interface, and the etching patterns of dentin and enamel. **Method:** Four products, One-Up Bond F (OU) (Tokuyama), SE Bond (Kuraray), Prompt L-Pop (LP) (3M-ESPE) and Bisco experimental self-etching adhesive (ES) (Bisco), were evaluated, and One-Step (OS) (Bisco) with 32% H₃PO₄ was used as a control. SEM was used to reveal the resin layer and hybrid layer, the etching pattern of dentin and enamel, and resin tag formation of the four self-etching adhesive systems. Occlusal part of tooth specimens was ground off to expose flat dentin surface. Each adhesive system was applied according to its manufacturer's instructions and cured with light-cure composite on the top of it. The specimens of resin tag formation were then placed in 1% NaOCl solution for 10min. after soaked in 6N HCL solution over night. The interfacial specimens were sliced perpendicular to bonding interface, about 3mm thick, polished by 1500 Grit Sic paper, 1 μ m and 0.3 μ m alumina slurry, and soaked in 1% NaOCl solution for 20min. The etching time for etching pattern specimens of each adhesive was according to its manufacturer's instructions. The adhesive was rinsed off by acetone followed by water. **Result:** OU and SE Bond have short resin tags and thin hybrid layer, and LP and ES have relatively long resin tags and thicker hybrid layer. Tubules on etched dentin of four systems were all opened. Except ES, ground and ungrounded enamel of OU, SE and LP are not totally etched compare with 37% H₃PO₄. Debonded area in the interface of composite-adhesive-tooth exists in OU and LP. No debonded area was found in SE and ES interfaces. **Conclusion:** The morphology of the ES is the closest to the fifth generation adhesive system among the four products investigated.

- **Introduction**

- Recently, as more and more self-etching adhesive systems entered market, dentists could get the impression from most manufacture's advertisement and technical information, that most of these products can be bonded to tooth structure, especially to dentin, as well as conventional adhesives using phosphoric acid as an etchant. By looking at dentin shear bond strength and microtensile strength data of these products bonded to dentin, some of the products such as SE Bond from Kuraray, have the same high number as those conventional 5th generation adhesives. However, enamel bond strength data are always lower. Even when mechanical bond strength of the self-etching systems on dentin is close to conventional adhesives, we still need to be concerned about the microstructure or the morphology at the interface of composite-adhesive-dentin. Microstructure can help us to predict long-term bond strength since most mechanical data is reported after a short period of time of bonding.

- **Objective**

- **This purpose of this paper is to examine the microstructure of dentin-adhesive-composite of several self-etching adhesive systems**

- **Materials & Method**

- **Material**

	Source
• Uni-Etch® (32%H3PO4)	Bisco
• Aeliteflo 2A	Bisco
• 1500 Grit SiC sandpaper	3M
• 1.0 µm and 0.3 µm alumina slurry	Electron Microscopy Sciences
• Extracted human teeth	
• 1% NaOCl solution	
• 6N HCl solution	
• One-Up Bond	Tokuyama
• SE Bond	Kuraray
• Prompt L-Pop	3M-ESPE
• Experimental self-etching adhesive	Bisco

- **Equipment:**

• Scanning Electron Microscope(SM-510)	Topcon
• VIP curing light	Bisco

- **SEM specimens preparation:**

- Dentin and enamel etching pattern specimens of each system were created by applying the adhesive according to the manufacture's instructions, to dentin or enamel surface. The adhesive was rinsed off afterwards with acetone and water.
- Specimens of resin tag formation and composite-adhesive-dentin interface were made by removing occlusal part of tooth to expose flat dentin surface. Each adhesive system was applied according to its manufacturer's instructions and cured with light-cure composite Aeliteflo on the top of it. The specimens of resin tag formation were soaked in 6N HCL solution over night following with 1% NaOCl solution for 10min. The interfacial specimens were sliced perpendicular to bonding interface, about 3mm thick, polished by 1500 Grit Sic paper, 1µm and 0.3µm alumina slurry, and soaked in 1% NaOCl solution for 20min.

- **Results & Discussion**

- Etching with 37% H₃PO₄ for 15s was used as a control for the following six group SEM photographs
- Fig. 1 through Fig. 20 are SEM photographs of dentin and enamel etching patterns by 37% H₃PO₄ for 15s or each self-etching adhesive system. Enamel was treated with three different methods, such as pumiced only, sanded by 320 grit paper, and cut by a cross burr, before etching procedure. Fig.2, 3, 4 and 5 showed dentin tubules of all the systems investigated were opened, however, tubules of SE bond and One-Up Bond were less opened. Enamel surface treated with three different ways, was etched well by Bisco's experimental primer, less etched by Prompt L-Pop, and hardly etched by SE Bond Primer and One-Up Bond.
- It is said that the amount and length of adhesive resin tags are beneficial for increasing bond strength. Fig. 21 through Fig. 25 are the SEM photographs which showed resin tag formation of each adhesive system. The tag length and amount of each system were not the same. Bisco's experimental system showed the largest number and longest tags, SE bond and Prompt L-Pop showed intermediary, and One-Up Bond showed the least and shortest tag formation.
- Fig. 26 through Fig. 30 are the SEM photographs exhibiting the interface of composite-adhesive-dentin of each adhesive product. From the micrographs, no debonded area was found in the interface of Bisco experimental self-etching system and SE bond. However, a debonded area was found at the interface of One-Up Bond and Prompt L-Pop. The adhesive film thickness of SE bond is much higher than the other adhesive systems'. Hybrid layer formation of Bisco's experimental system and Prompt L-Pop was more obvious than SE bond and One-Up Bond.

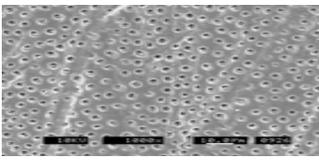


Fig 1. 37% H₃PO₄ etched dentin surface for 15s.

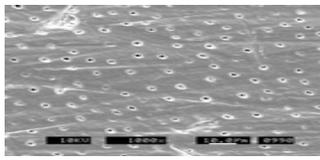


Fig 2. SE Bond Primer etched dentin surface.

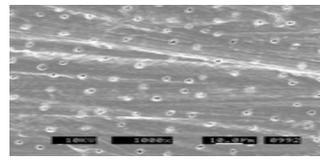


Fig 3. One-Up Bond etched dentin surface.

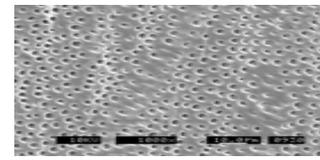


Fig 4. Bisco experimental system etched dentin surface.

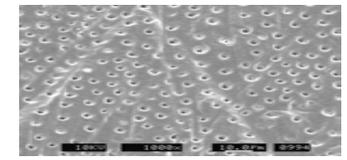


Fig 5. Prompt L-Pop etched dentin surface.

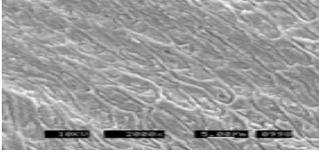


Fig 6. 37% H₃PO₄ etched enamel surface for 15s. after sanding with 320 grit paper

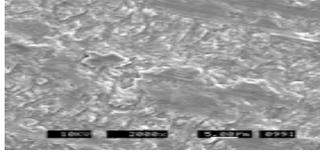


Fig 7. SE Bond Primer etched enamel surface for 15s. after sanding with 320 grit paper

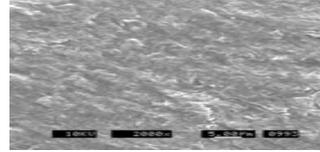


Fig 8. One-Up Bond etched enamel surface for 15s. after sanding with 320 grit paper

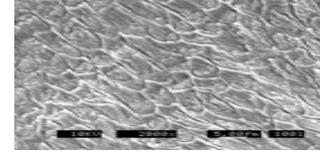


Fig 9. Bisco experimental primer etched enamel surface for 15s. after sanding with 320 grit paper

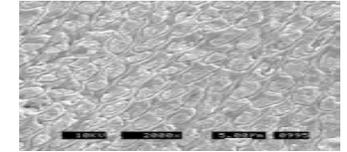


Fig 10. Prompt L-Pop etched enamel surface for 15s. after sanding with 320 grit paper

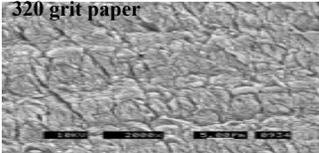


Fig 11. 37% H₃PO₄ etched enamel surface for 15s after cross burr cutting.

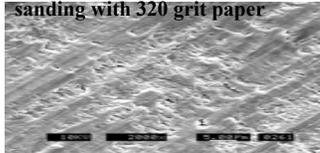


Fig 12. SE Bond Primer etched enamel surface after cross burr cutting.

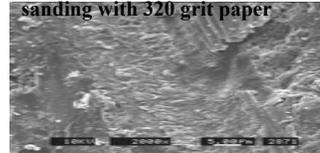


Fig 13. One-Up Bond etched enamel surface after cross burr cutting.

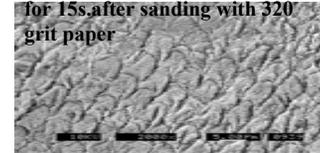


Fig 14. Bisco experimental primer etched enamel surface after cross burr cutting.

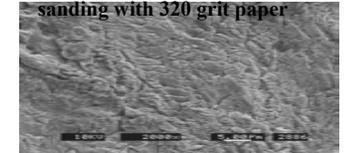


Fig 15. Prompt L-Pop etched enamel surface after cross burr cutting.

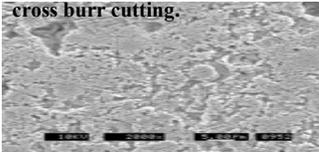


Fig 16. 37% H₃PO₄ etched enamel surface for 15s after pumicing .

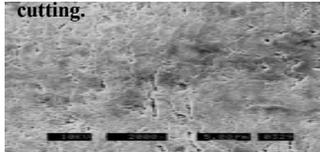


Fig 17. SE Bond Primer etched enamel surface after pumicing .

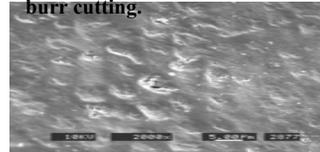


Fig 18. One-Up Bond etched enamel surface after pumicing

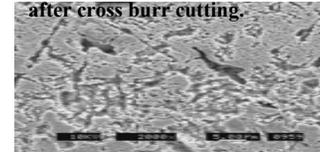


Fig 19. Bisco experimental primer etched enamel surface after pumicing .

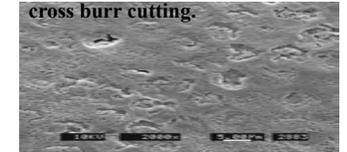


Fig 20. Prompt L-Pop etched enamel surface after pumicing

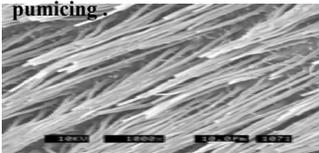


Fig 21. Resin tag formation of One-Step Plus after etching with 37% H₃PO₄ for 15s.

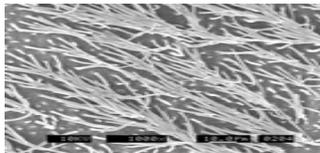


Fig 22. Resin tag formation of SE Bond.

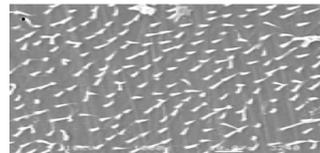


Fig 23. Resin tag formation of One-Up Bond.

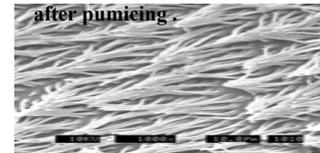


Fig 24. Resin tag formation of One-Step Plus with Bisco experimental primer.

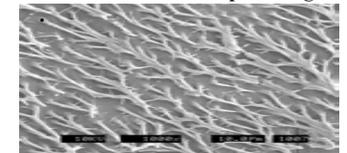


Fig 25. Resin tag formation of Prompt L-Pop.

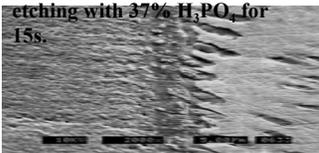


Fig 26. Composite-adhesive-dentin interface of One-Step Plus after etching with 37% H₃PO₄ for 15s.

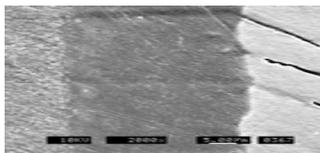


Fig 27. Composite-adhesive-dentin interface of SE Bond.

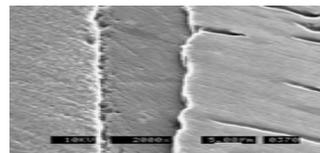


Fig 28. Composite-adhesive-dentin interface of One-Up Bond.

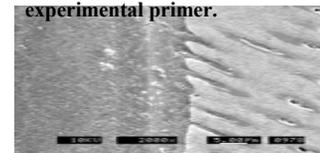


Fig 29. Composite-adhesive-dentin interface of One-Step Plus with Bisco experimental primer.

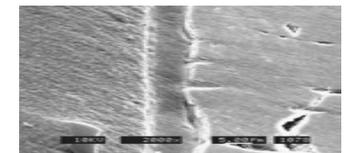


Fig 30. Composite-adhesive-dentin interface of Prompt L-Pop