Technical Report

TOKUYAMA EE-BOND

Light-Cured Dental Adhesive for Enamel-Etching Technique

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1. Introduction

Total-etching system and self-etching system are the two biggest categories in the dental adhesive market. Total-etching system has longer history and roughly more than half share of the world market. Which system is superior as a whole is a very difficult question to answer. Table 1 shows the advantages and disadvantages of self-etching and total-etching bonding systems. Since self-etching system does not involve phosphoric acid etching of the dentin having the danger of over-etching or over-drying, it significantly reduces the occurrence of post-operative sensitivity[1) 2)]. It also offers high durability when bonded to dentin [3)]. On the other hand, total-etching system provides higher bond strength when bonded to uncut enamel [4)] and excellent marginal integrity (fewer marginal defects and less marginal discoloration)[5)6)7)8)9)].

Table 1: Advantages and disadvantages of self-etching and total-etching systems

	Self-etching system	Total-etching system
Advantages	+ Simple procedures + Reduced post-operative sensitivity [1) 2)] + High bond durability to dentin [3)] + Less technique sensitivity (wet bonding is not required) + Esthetic (thin bonding layer)	+ High bond strength to uncut enamel [4)] + Excellent marginal integrity [5) 6) 7) 8) 9)]
Disadvantages	- Low bond strength to uncut enamel [4)] - Poor marginal integrity[5)6)7)8)9)] (marginal defect and discoloration)	- Complicated procedures - Higher risk of post-operative sensitivity[1)2)] - Low bond durability to dentin [3)] (decalsified dentin) - Higher technique sensitivity (wet bonding is required)

Tokuyama Dental Corp. develops and markets various adhesive compositions by applying 3D-SR (Self Reinforcing) technology [10) 11)]. The 3D-SR monomer readily penetrates into the tooth substance and forms a thin, strong, uniform bonding layer over the tooth surface through multi-point bonding with apatite calcium and three-dimensional cross-linking reactions, leading to superior bonding to the tooth substance [12)13)14)15)]. We believe combining 3D-SR technology and the enamel-etching technique will produce the highest quality dental adhesive providing superior bond strength and durability to enamel and dentin, simultaneously.

Accordingly, we marketed the TOKUYAMA EE-BOND enamel-etching system, which consists of EE-BOND, an application of 3D-SR technology, and TOKUYAMA ETCHING GEL HV, a new dental etching agent designed for enamel-etching technique.

Described below is the development concept underlying TOKUYAMA EE-BOND:

Development concept

The most ideal bonding system for enamel etching, offering:

+ Excellent bonding to both enamel and dentin
+ Long-term excellent marginal integrity and dentinal sealing
+ Reduced post-operative sensitivity
+ High viscous etching gel

2. TOKUYAMA EE-BOND: Composition, clinical procedures, and bonding mechanism

2.1 Composition

Table 2 shows the components of EE-BOND. It contains the phosphoric acid monomer, required to decalcify the tooth substance (the main component of the SR monomer); various monomers to form the bonding layer; alcohol; water; and camphorquinone as the photopolymerization initiator.

Table 2: Composition of EE-BOND

Basic components	Function
Phosphoric acid monomer	Decalcification of tooth substance, Formation of bonding layer
Bis-GMA	Formation of bonding layer
3G (TEGDMA)	Formation of bonding layer
HEMA	Penetration into the tooth substance, Formation of bonding layer
Alcohol	Solvent
Water	Decalcification of tooth substance
Glass filler	Reinforcement of bonding layer, fluoride release
Camphorquinone	Photopolymerization initiator

Table 3 shows the components of TOKUYAMA ETCHING GEL HV. The concentration of phosphoric acid is 39%, a typical value.

Table 3: Composition of TOKUYAMA ETCHING GEL HV

Basic components	Function
Phosphoric acid	Etching tooth substance
Purified water	Etching tooth substance
Thickener	Gelation of etching agent
Colorant	Coloring etching gel

2.2 Clinical procedures

Figure 1 shows the clinical procedures for TOKUYAMA EE-BOND. The treatment takes little time: The etching procedure takes 5 seconds, while the bonding procedure takes 10 seconds.

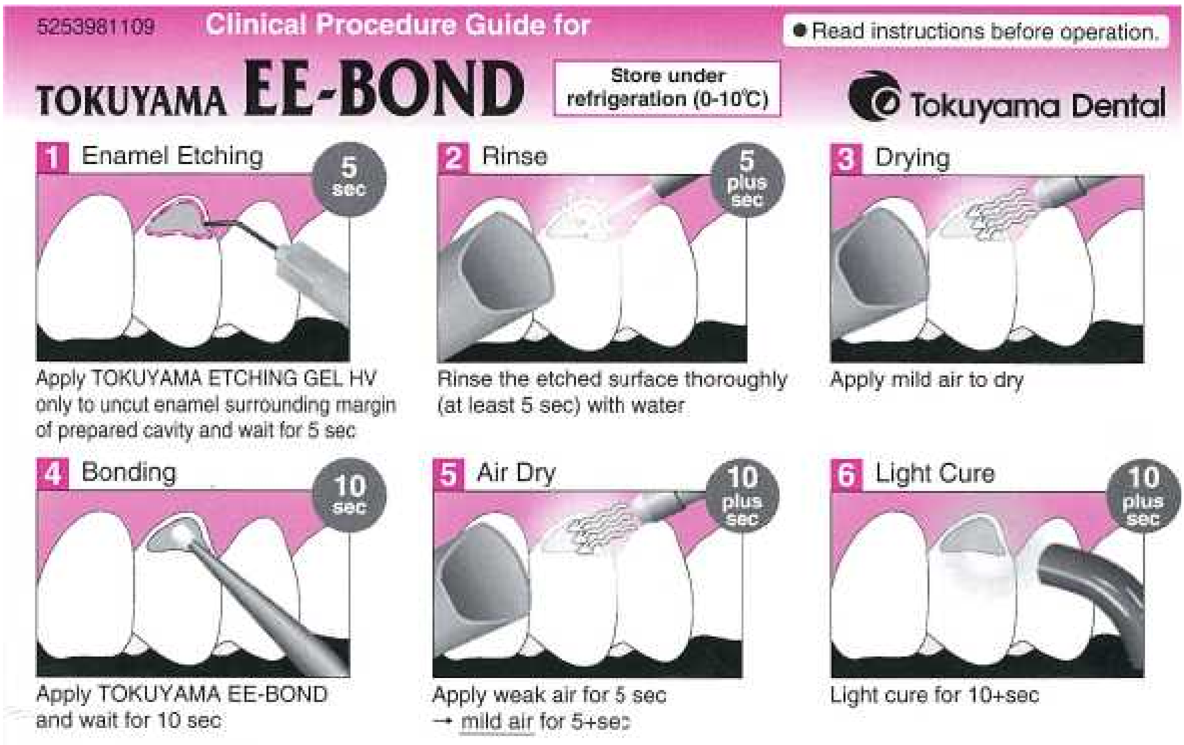


Figure 1: Clinical procedures for TOKUYAMA EE-BOND

As Figure 2 shows, only the uncut enamel surrounding the margin is etched with TOKUYAMA ETCHING GEL HV. The etching of the uncut enamel provides clinically satisfactory marginal quality. Since this does not require phosphoric acid etching of the dentin, it causes no mineral loss and reduces post-operative sensitivity.

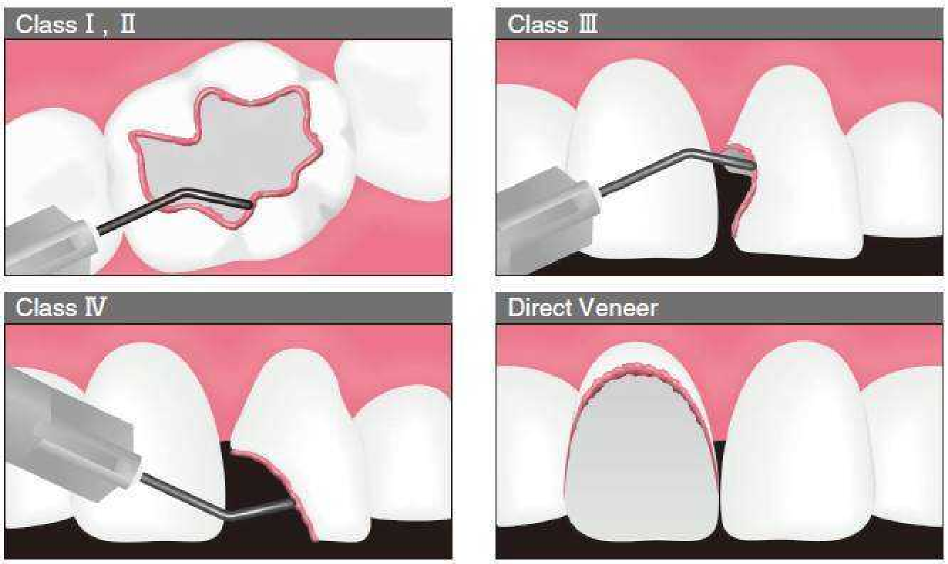
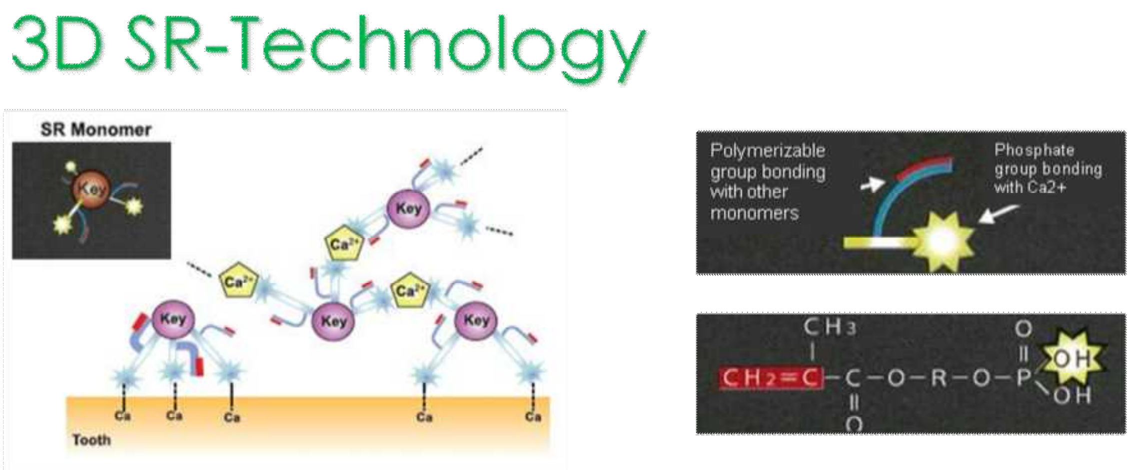


Figure 2: Examples of enamel etching with TOKUYAMA EE-BOND

2.3 Bonding mechanism

The bonding mechanism of TOKUYAMA EE-BOND is based on the formation of a thin, strong, uniform bonding layer on the tooth surface, resulting from penetration of Tokuyama's 3D-SR monomers into the tooth substance, multi-point bonding with apatite calcium, and three-dimensional cross-linking reactions between the 3D-SR monomers (Figure 3).



Adhesive 3D SR-Monomer enables excellent adhesion to the tooth structure because it includes multiple phosphate groups which is capable of forming an multi-point bond with apatite of the tooth and calcium ions freed during tooth demineralization. Besides, SR-Monomer includes multiple polymerizable groups which link together forming strong bonding layer when light cured.

Figure 3: 3D-SR technology

3. Features of TOKUYAMA EE-BOND

3.1 Bond strength

We evaluated the bond strength of TOKUYAMA EE-BOND with a micro-tensile test. Figure 4 shows the results for bonding to cut enamel. Figure 5 shows the results for bonding to uncut enamel. Figure 6 shows the results for bonding to cut dentin.

TOKUYAMA EE-BOND demonstrates satisfactory bond strength in all three cases: cut enamel, uncut enamel, and cut dentin. These results are attributable to a strong bonding layer on the tooth surface created by the curing of reaction products of the adhesive monomer (3D-SR monomer) in TOKUYAMA EE-BOND and the calcium of the tooth substance.

Test method:

(1) The cut enamel or dentin surface is prepared by polishing the labial surface of an extracted bovine primary anterior tooth with #600 SiC paper. The uncut surface is cleaned with PRESSAGE (Shofu) and rinsed with water.

(2) Each bonding material is applied according to manufacturer’s instructions.

(3) The cavity is filled with composite resin (Estelite Posterior, Tokuyama Dental) by incremental filling.

(4) After immersion in water for 24 hours or after 10 thousand thermal cycles (TC, between 5°C and 60°C, 1 minute), the sample is sectioned into a 1 mm × 1 mm sticks with a diamond cutter.

(5) With an universal tester (Ez Test, Shimadzu Corporation), the sample is subjected to a micro-tensile test (n = 9) at a crosshead speed of 1 mm/min.

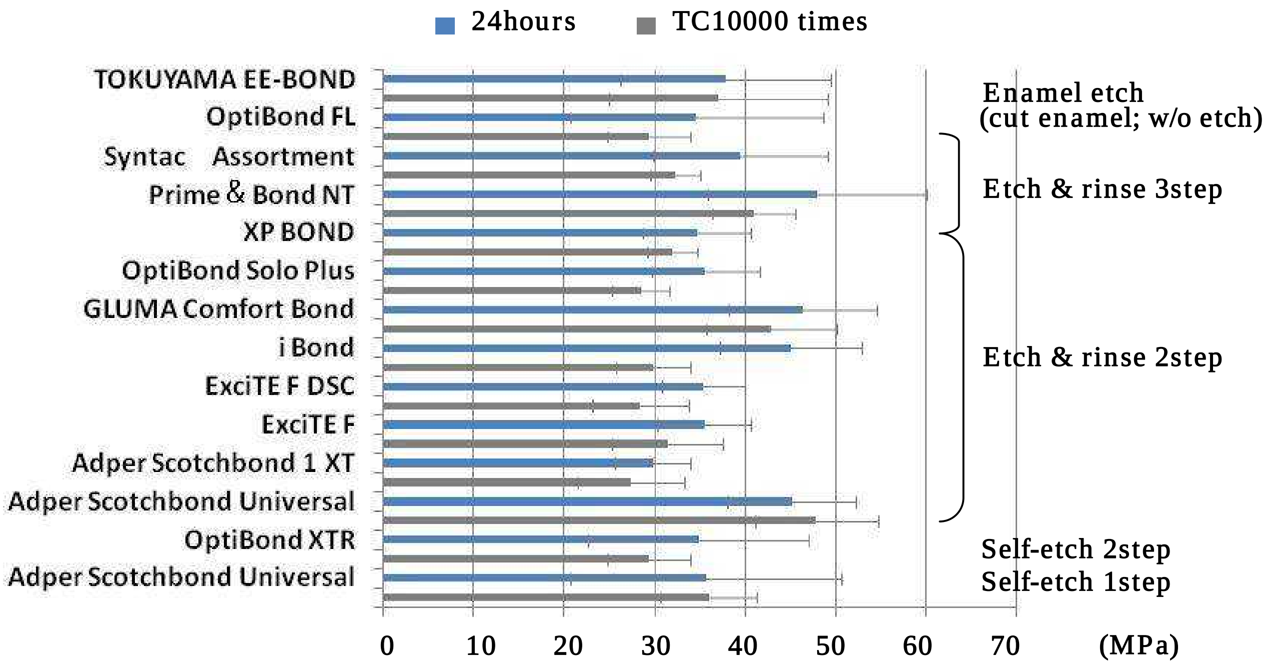


Figure 4: μTBS (Cut enamel)

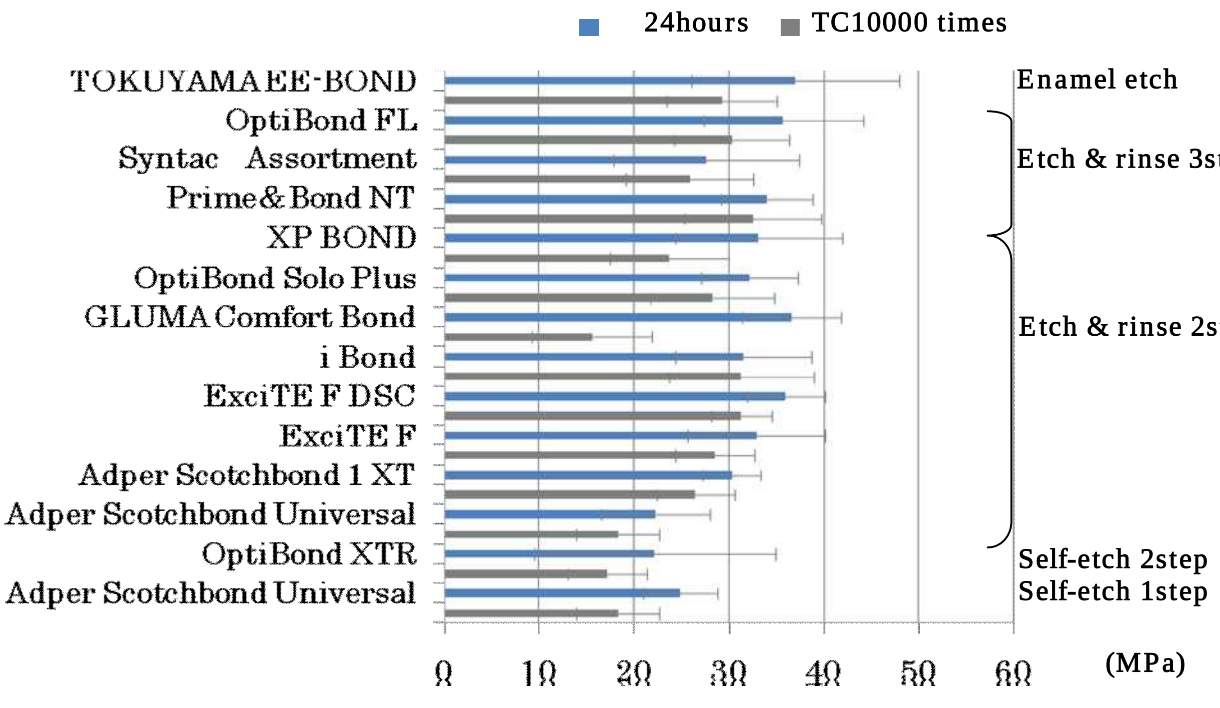


Figure 5: μTBS (Uncut enamel)

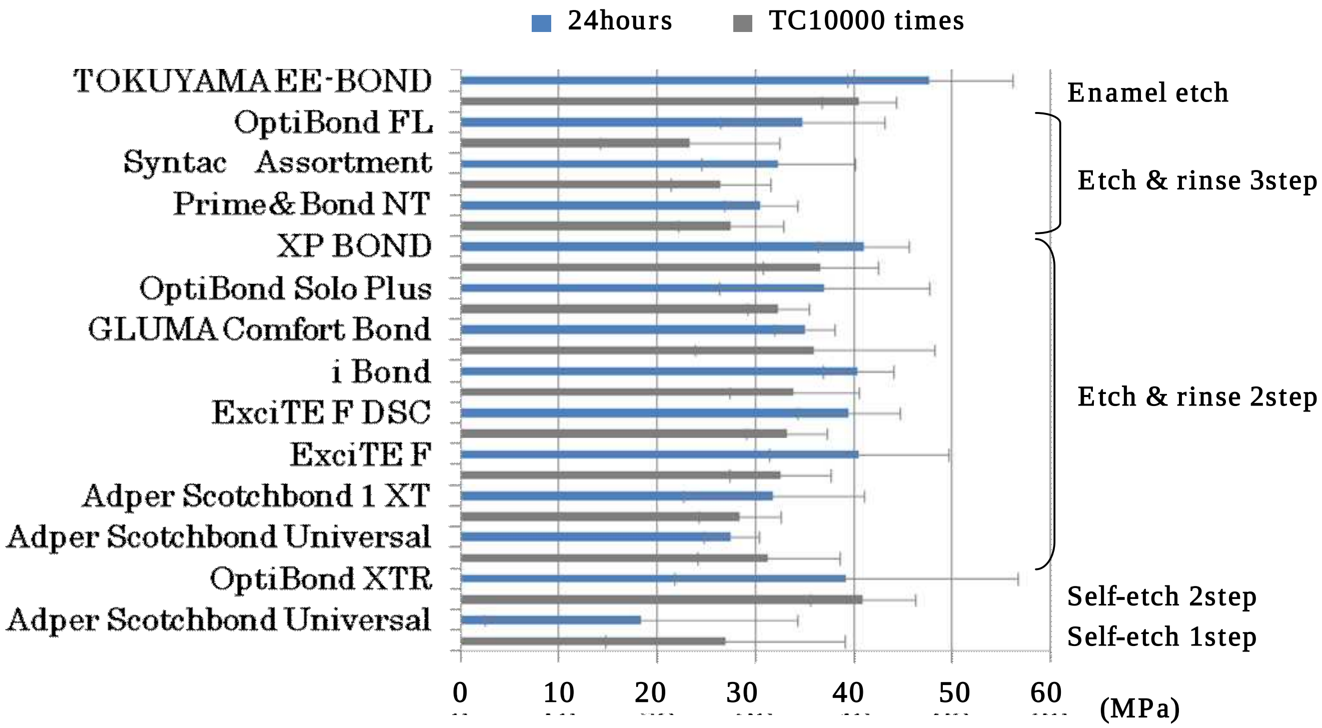
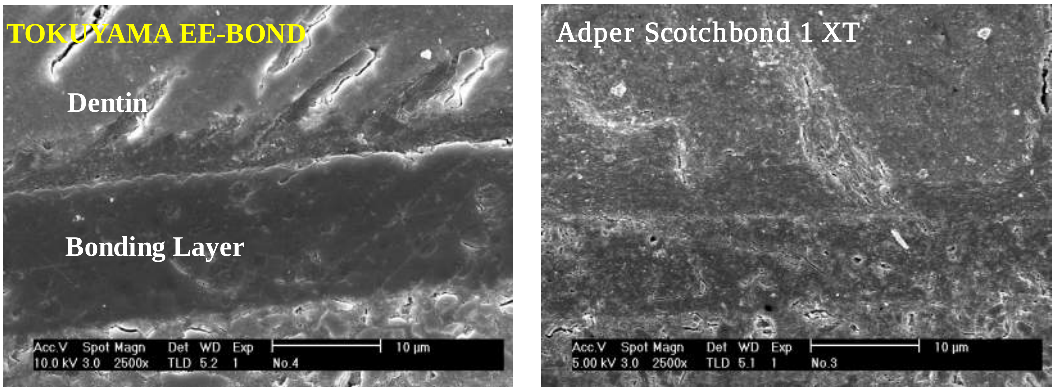


Figure 6: µTBS (Cut dentin)

3.2 Dentinal sealing

We inspected the adhesive/dentin interface by SEM (FE-SEM XL30SFEG, PHILIPS) after polishing with diamond paste (0.25 µm) (Figure 7). TOKUYAMA EE-BOND demonstrates satisfactory dentinal sealing. This result comes from the excellent penetration and adhesion to dentin structure of EE-BOND. The formation of the strong bonding layer achieves satisfactory dentin sealing and results in low post-operative sensitivity in clinical practice.



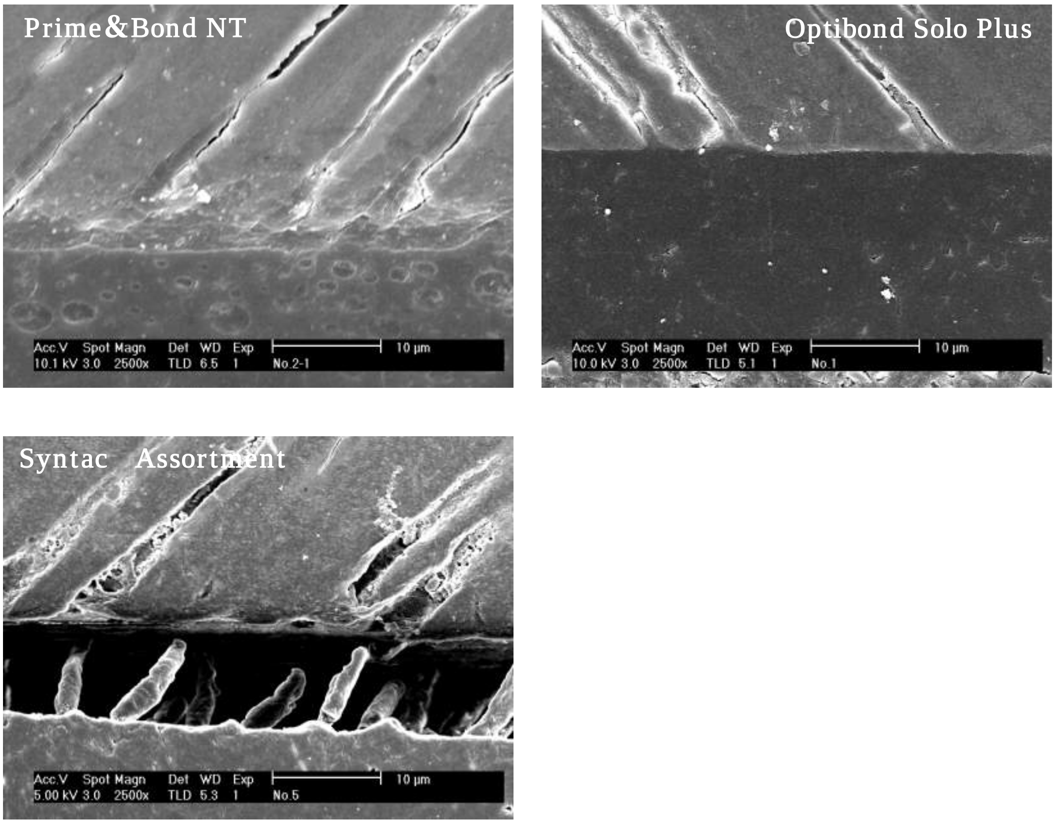


Figure 7: SEM observations of bonding interface on cut dentin

3.3 Cavity adaptation

We performed a cavity adaptation evaluation for TOKUYAMA EE-BOND and products from other manufacturers (Figures 8 to 12). TOKUYAMA EE-BOND shows a bonding layer with uniform thickness free of gaps over the entire tested surface, including uncut enamel (treated with TOKUYAMA ETCHING GEL HV for 5 seconds, then treated with EE-BOND for 10 seconds); cut enamel (treated with EE-BOND for 10 seconds); and cut dentin (treated with EE-BOND for 10 seconds). These results demonstrate satisfactory cavity adaptation of EE-BOND. One of what contributes to the exceptional cavity adaptation of EE-BOND is an unique characteristic that thin gelatinized bonding layer is formed on the tooth surface just after application (before light curing) through the chemical interaction of 3D-SR monomer in EE-BOND and the calcium of the tooth substance. Applied EE-BOND is polymerized by light irradiation forming thin but durable 3D matrix bonding layer. The thin layer is also expected to achieve attractive aesthetic results, especially on the anterior teeth.

Test method:

(1) A cylindrical simulated cavity of ∅4 mm × 4 mm is formed on the labial surface of an extracted

bovine primary anterior tooth.

(2) Each bonding material is applied according to manufacturer's instructions before incrementally building a resin composite.

(3) The cavity is filled with composite resin (Estelite LV, Tokuyama Dental) by incremental filling.

(4) The tooth is cut perpendicular to the tooth surface with a diamond cutter.

(5) The cut surface is polished with diamond paste (final: 0.1 μm, Buehler).

(6) The cut surface is observed with a laser microscope (VK9700, KEYENCE).

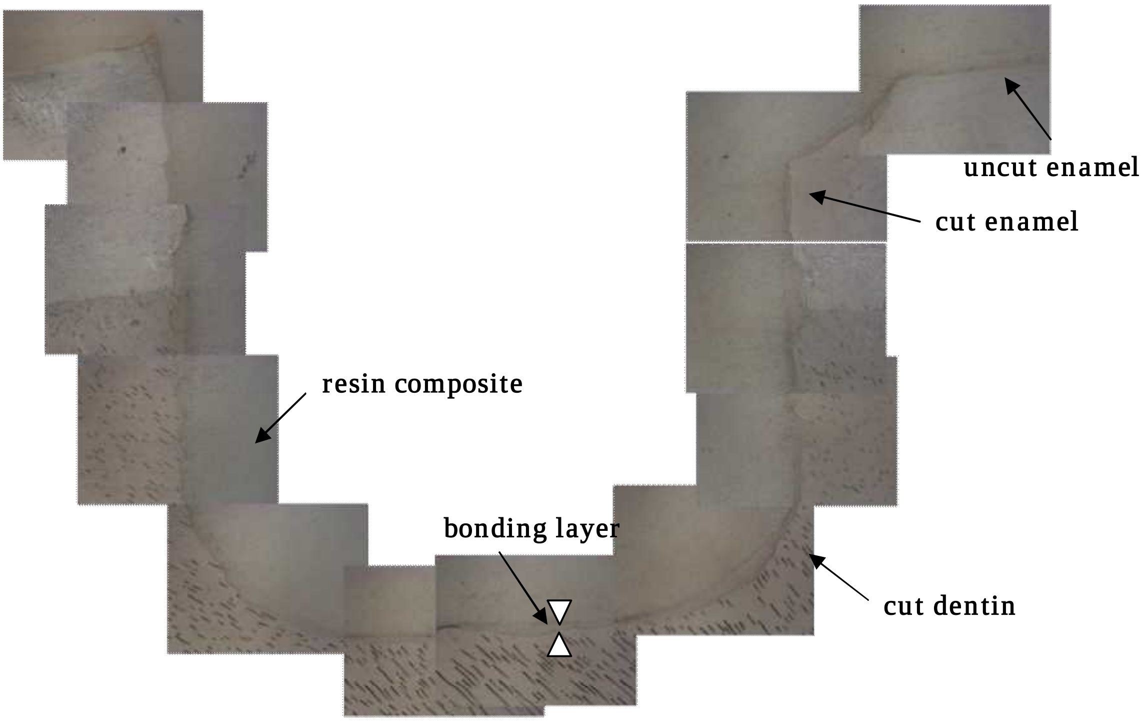


Figure 8: Cavity adaptation of TOKUYAMA EE-BOND

Adper Scotchbond 1 XT shows a gap at the corner of the cavity floor (indicated by the arrow in Figure 9).

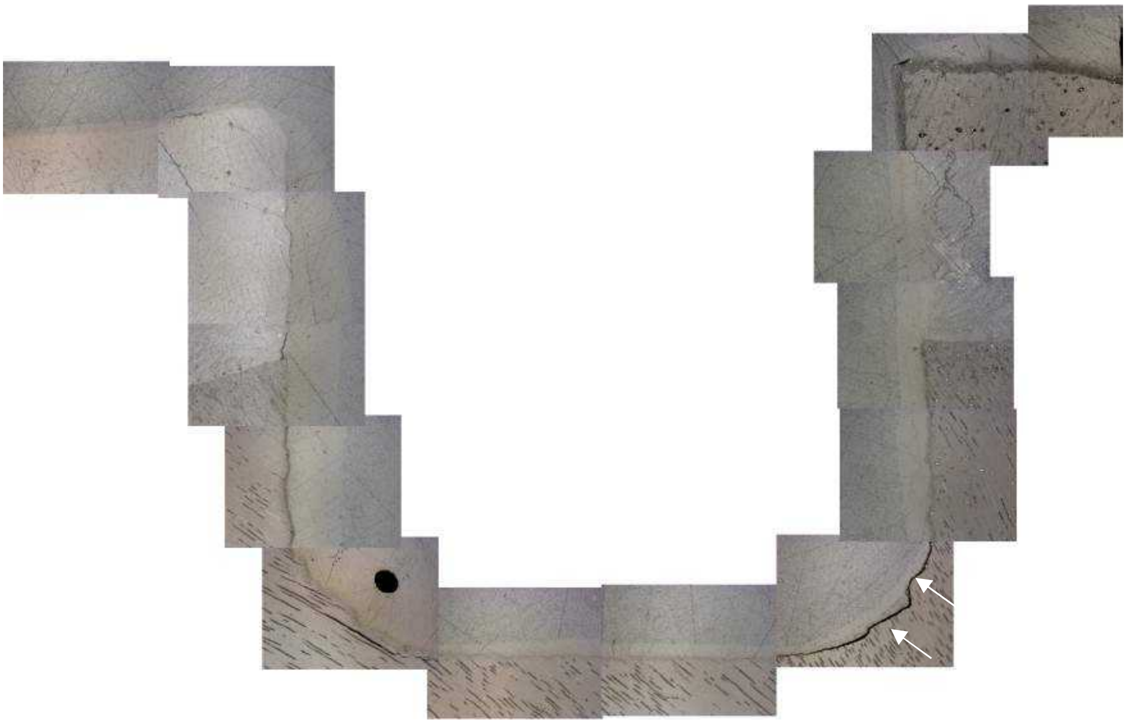


Figure 9: Cavity adaptation of Adper Scotchbond 1 XT

Prime Bond NT shows a slight gap at the cavity floor (indicated by the arrow in Figure 10).

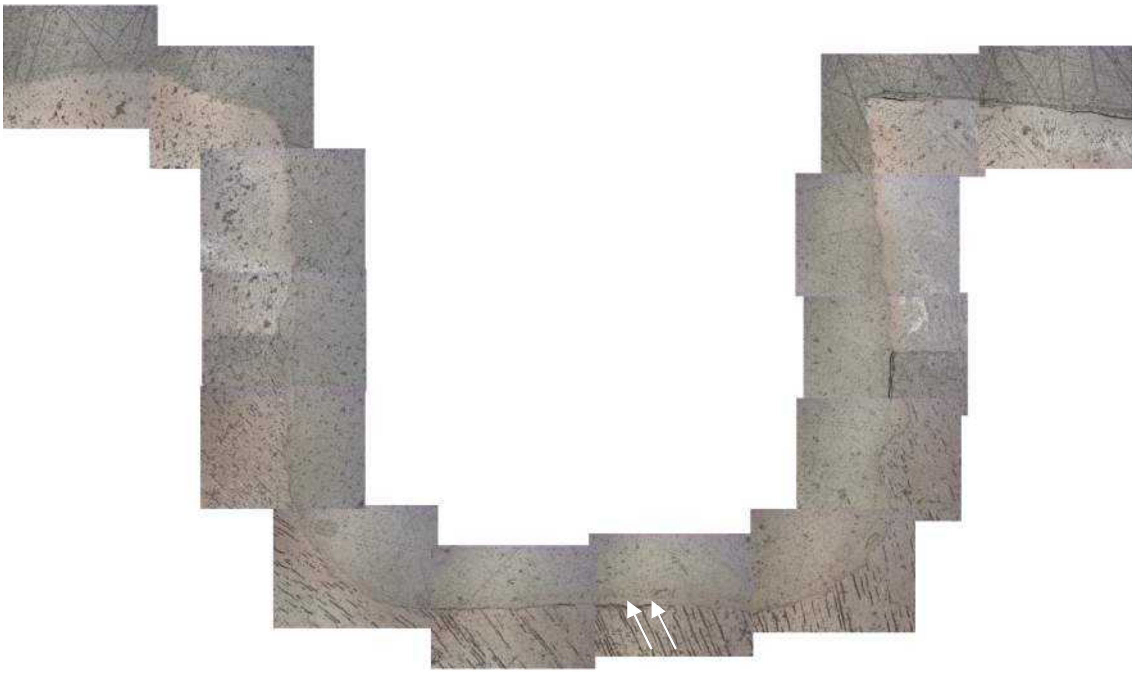


Figure 10: Cavity adaptation of Prime Bond NT

Optibond Solo Plus shows a gap at the corner of the cavity floor (indicated by the arrow in Figure 11).

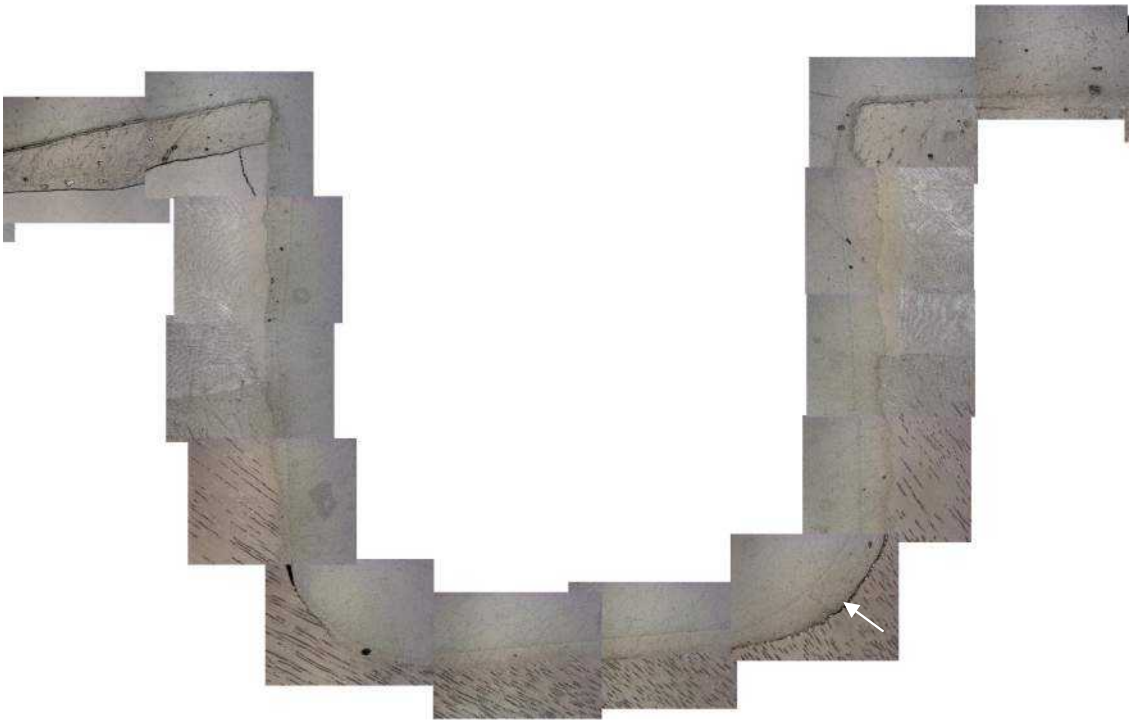
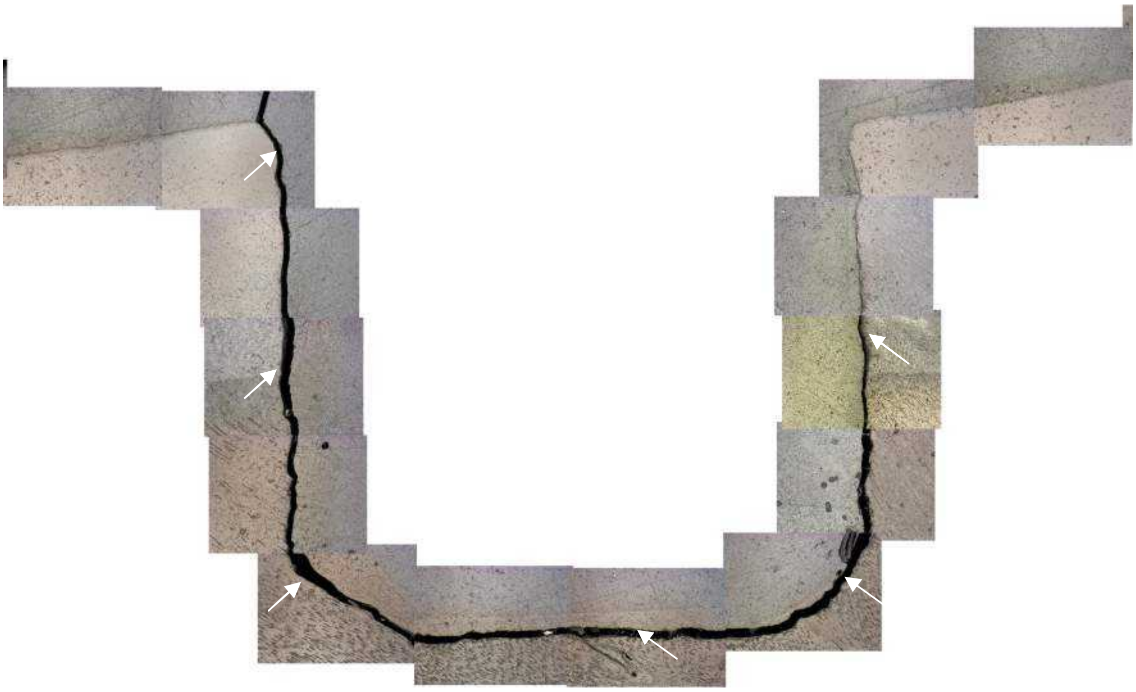


Figure 11: Cavity adaptation of Optibond Solo Plus

Syntac Assortment shows gaps over the entire cavity except the uncut enamel (indicated by the arrows in Figure 12).



Figre 12: Cavity adaptation of Syntac Assortment

3.4 Marginal sealing

We evaluated marginal sealing for TOKUYAMA EE-BOND and products from other manufacturers (Table 5 and Figures 13). The uncut enamel was treated with TOKUYAMA ETCHING GEL HV for 5 seconds, then treated with EE-BOND for 10 seconds. The cut enamel and cut dentin were treated with EE-BOND for 10 seconds. TOKUYAMA EE-BOND demonstrated satisfactory marginal sealing, with no dye penetration. In addition to superior adhesion to both dentin and enamel, acid etching to uncut enamel ensures high marginal bonding which prevents marginal micro-leakage and degradation.

Test method:

(1) A cylindrical simulated cavity of ∅4 mm × 4 mm is formed on the labial surface of an extracted bovine primary anterior tooth.

(2) Each bonding material is applied according to manufacturer's instructions before incrementally building a resin composite.

(3) The cavity is filled with composite resin (Estelite LV, Tokuyama Dental).

(4) After immersion in water at 37°C for 24 hours, the tooth is immersed in 1% fuchsin solution at 37°C for 24 hours.

(5) The tooth is cut perpendicular to the tooth surface with a diamond cutter.

(6) The cut surface is polished with #600 SiC paper (final: P3000).

(7) The cut surface is observed with an optical microscope for dye penetration.

Table 5: Evaluation of marginal sealing

Bonding Agent	Marginal sealing (n=4)
TOKUYAMA EE-BOND	−、−、−、−
Adper Scotchbond 1 XT	−、−、+、−
Prime&Bond NT	−、−、+、−
Optibond Solo Plus	+、−、−、−
Syntac Assortment	+、+、−、−

−: No dye penetration

+: Dye penetration into enamel

++: Dye penetration into dentin

+++: Dye penetration to the cavity floor

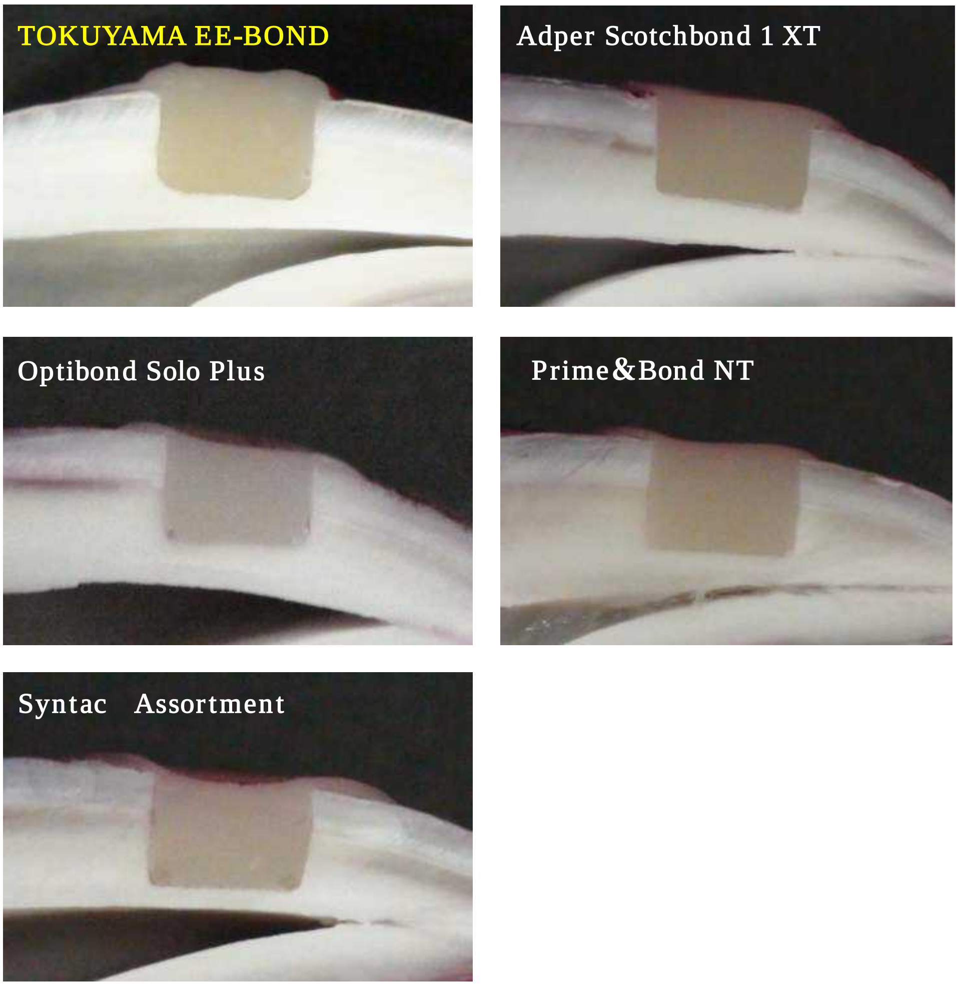


Figure 13: Evaluation of marginal sealing

3.5 Low technique sensitivity

We evaluated the effects of the wetness of the tooth substance on the bond strength of TOKUYAMA EE-BOND. Figure 14 shows the evaluation results of the strength of the bond to cut enamel. Figure 15 shows the evaluation results for the strength of the bond to cut dentin. The wetness of the tooth surface had no effect on the bond strength of TOKUYAMA EE-BOND.

Test method:

(1) The labial surface of an extracted bovine primary anterior tooth is polished with #600 SiC paper to prepare the bonding surface. The bonding area is specified by a piece of double-sided tape with a ∅3-mm hole. An ∅8-mm, 0.5-mm-thick wax sheet is attached to form a cylindrical simulated cavity.

Dry: The tooth is dried with a dental air syringe.

Moist: The tooth is allowed to stand for 1 hour in a constant temperature and humidity box set with temperature set at 37°C and humidity at 100%.

Wet: Water is applied with a dental micro-brush before bonding.

(2) The tooth is treated with EE-BOND for 10 seconds; air-dried; and light-cured for 10 seconds.

(3) The cavity is filled with composite resin (Estelite Posterior, Tokuyama Dental).

(4) After the tooth is immersed in water at 37°C for 24 hours, stainless steel attachments are bonded to cured resin composite, and the tooth is subjected to a tensile test (n = 4) with an autograph (AG-5000D, Shimadzu Corporation) at a crosshead speed of 2 mm/min.

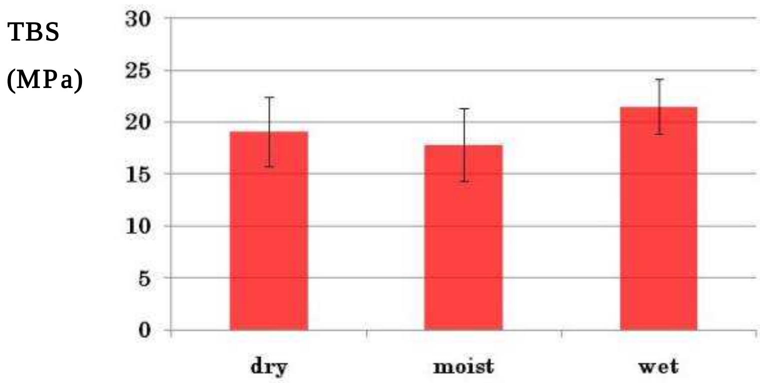


Figure 14: Evaluation of effects of wetness (Enamel)

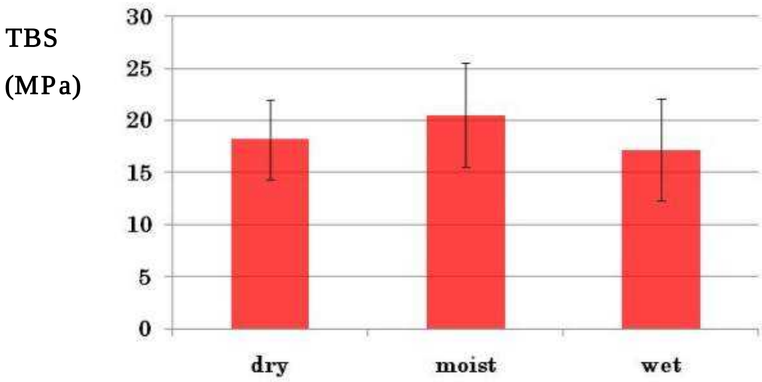


Figure 15: Evaluation of effects of wetness (Dentin)

3.6 Extended working time

Figure 16 shows the external appearance of EE-BOND 1 minute after dispensing. EE-BOND showed no signs of phase separation. The non-phase-separating property is expected to contribute to longer working time and stable bond strength.

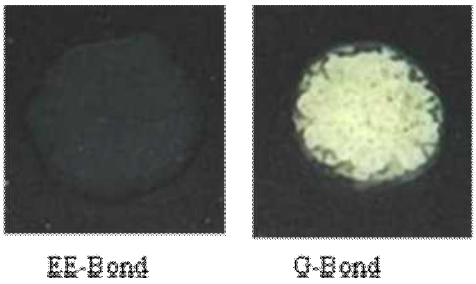


Figure 16: External appearance of TOKUYAMA EE-BOND after dispensing

3.7 Fluoride-releasing

TOKUYAMA EE-BOND releases fluoride gradually.

3.8 Features of TOKUYAMA ETCHING GEL HV

TOKUYAMA ETCHING GEL HV has properties suitable for etching just the uncut enamel surrounding the margin in the enamel etching technique.

First, the syringe tip provided with the product has a small diameter of 0.4 mm (outside) / 0.2 mm (inside), making it easy to apply the etching agent to just the targeted area.

Figure 17 shows the consistency of TOKUYAMA ETCHING GEL HV (test conditions: sample: 0.1 g; load: 50 g). The consistency of TOKUYAMA ETCHING GEL HV is relatively low, with a value of 18 mm, and resists spreading on the tooth substance. On the other hand, it is easily washed away simply by rinsing with water.

As shown above, TOKUYAMA ETCHING GEL HV makes it especially easy to etch just the specific targeted area. The high viscous etching agent is ideal for enamel etching (Figure 18).

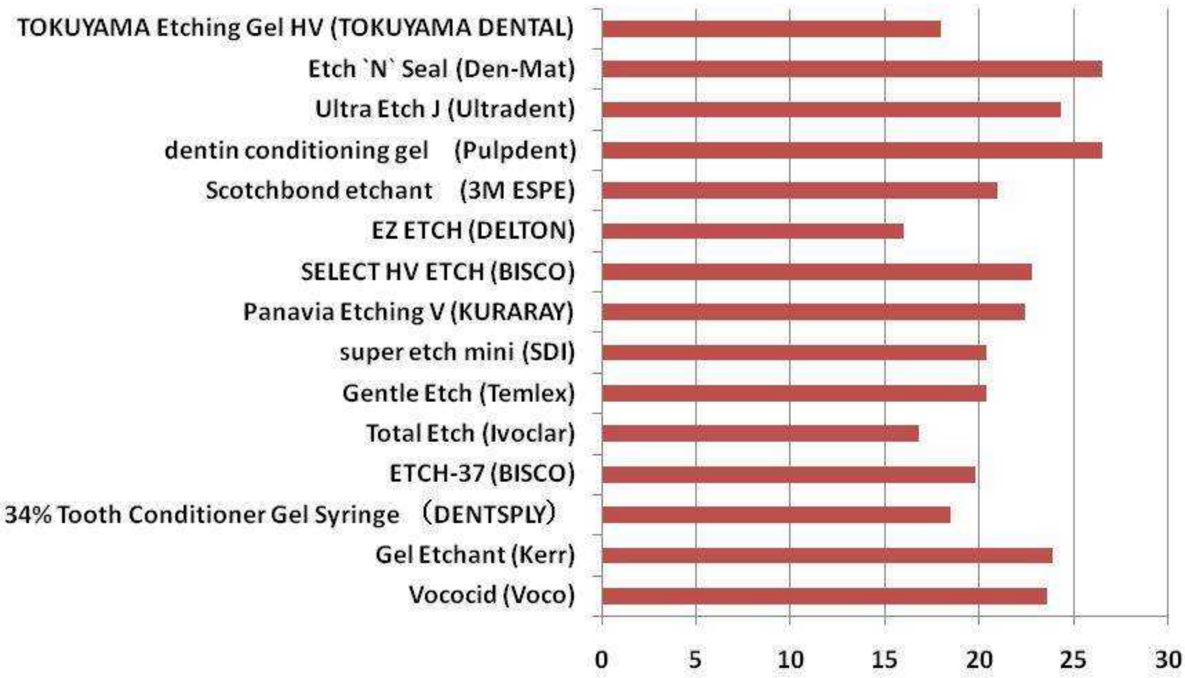


Figure 17: Consistency (mm) of TOKUYAMA ETCHING GEL HV

Figure 18: Example of clinical application of TOKUYAMA ETCHING GEL HV

4. Conclusion

TOKUYAMA EE-BOND (consisting of “EE-BOND” and “TOKUYAMA ETCHING GEL HV”) is a dental adhesive system designed for use with the enamel etching technique. It provides the following features. These high-performance characteristics make it the ideal dental adhesive for clinical applications.

Features of TOKUYAMA EE-BOND

- Outstanding adhesion performance
- Dependable marginal integrity (Minimized micro-leakage)
- Reduced post-operative sensitivity
- Perfect cavity sealing
- Less technique sensitivity
- Prolonged working time
- Fluoride releasing

Features of TOKUYAMA ETCHING GEL HV

- Extra fine dispensing tip
- High viscous gel staying on the spot
- Easy to visualize, easy to rinse

5. Frequently Asked Questions of TOKUYAMA EE-BOND

Q1. How many drops can I dispense per one bottle?

A1. Approximately 320 drops per one bottle (5mL)

Q2. Do I need to shake the bottle before use?

A2. No.

Q3.What is pH of EE-BOND?

A3. Around 2.3

Q4. How long can I use EE-BOND after dispensing on the dish?

A4. Complete the application within 5 minutes after dispensing because EE-BOND contains a volatile alcohol as a solvent.

Q5.What if EE-BOND dropped on the mucosal membrane?

A5. Wipe the affected area immediately. And thoroughly flush with water after restoration. Affected areas may whiten due to protein coagulation but will disappear within 24 hours.

Q6.What is shelf life of EE-BOND and TOKUYAMA ETCHING GEL HV?

A6. EE-BOND: 2 years after the date of production under refrigeration (0-10ºC /32-50ºF). TOKUYAMA ETCHING GEL HV: 3 years in room temperature (0-25ºC /32-77ºF)

Q7. Does EE-BOND have fluoride release?

A7. Yes.

Q8.Will the liquid of EE-BOND cause phase separation?

A8. No, EE-BOND will not cause phase separation even if the solvent (alcohol) is evaporated because EE-BOND contains hydroxyethyl methacrylate (HEMA) which is relatively hydrophilic and improve compatibility between water and monomer.

Q9. Is there any risk that HEMA may reduce water resistance of bonding layer, thereby cause less bonding strength and durability?

A9. That risk is minimized by the best balance of the composition contained in EE-BOND. Besides, SR monomer employed in EE-BOND, which forms cross-link resin matrix, contributes to enhance physical properties of bonding layer as well as adhesive strength and durability.

Q10. What if etching gel is applied to cut enamel or cut dentin by mistake?

A10. Etching cut enamel will neither improve nor impair the adhesive performance of EE-BOND to cut enamel. However, etching dentin may reduce bond strength of EE-BOND to dentin.

Q11. 5 seconds of enamel etching treatment is instructed in the instruction manual. Is it enough to obtain high bond strength to uncut enamel and marginal sealing of EE-BOND?

A11. Our internal test results indicate no big difference in its adhesive performance between 5 seconds and longer treatment time than that.

Q12. Why dentin etching is not required with TOKUYAMA EE-BOND?

A12. EE-BOND penetrates and bonds chemically/mechanically to the dentin surface as well as enamel while mildly demineralize the hydroxyapatite of the tooth and dissolve the smear layer on prepared dentin. Many recent articles reports that etching treatment and subsequent rinse and air drying step causes collagen collapse on the dentin surface, which inhibits penetration of resin monomer, forms the gap between bonding layer and dentin structure , and induces post-operative sensitivity. Employing a limited acid etch technique applied only to the uncut enamel margin, TOKUYAMA EE-BOND significantly reduces the risk of post-operative sensitivity.

Q13. Though EE-BOND itself demineralizes and bonds to tooth structure, why separated enamel-etching required for uncut enamel surrounding the cavity prepared?

A13. Compared to cut enamel and dentin, uncut enamel usually contains more factors inhibiting mild demineralization, well penetration and bonding/sealing performance of EE-BOND. Etching uncut enamel removes these inhibitors and enhances adhesive performance and long-term marginal integrity of the composite restorations.

Q14. Can I use other etching gel/agents with EE-BOND?

A14. Conventional etching gels containing Phosphoric Acid (25 ~ 60wt/%) can be used for enamel etching before applying EE-BOND. Gel type is recommended to etch only uncut enamel and avoid flowing into dentin.

6. References

1) Unemori M, Matsuya Y, Akashi A, Goto Y, Akamine A.
Self-etching adhesives and postoperative sensitivity. Am J Dent. 2004 Jun;17(3):191-5.

2) Swift EJ Jr.
Dentin/enamel adhesives: review of the literature. Pediatr Dent. 2002 Sep-Oct;24(5):456-61.

3) Hashimoto M.
In vivo Degradation of Resin-Dentin Bonds in Humans Over 1 to 3 years. J Dent Res, 79(6):1385-1391, 2000

4) Beloica M, Goracci C, Carvalho CA, Radovic I, Margvelashvili M, Vulicevic ZR, Ferrari M.
Microtensile vs microshear bond strength of all-in-one adhesives to unground enamel. J Adhes Dent. 2010 Dec;12(6):427-33. doi: 10.3290/j.jad.a18237.

5) Peumans M, De Munck J, Van Landuyt KL, Poitevin A, Lambrechts P, Van Meerbeek B.
Eight-year clinical evaluation of a 2-step self-etch adhesive with and without selective enamel etching. Dent Mater. 2010 Dec;26(12):1176-84. Epub 2010 Oct 13.

6) Blunck U, Zaslansky P.Enamel margin integrity of Class I one-bottle all-in-one adhesives-based restorations. J Adhes Dent. 2011 Feb;13(1):23-9. doi: 10.3290/j.jad.a18445.

7) Fron H, Vergnes JN, Moussally C, Cazier S, Simon AL, Chieze JB, Savard G, Tirlet G, Attal JP.
Effectiveness of a new one-step self-etch adhesive in the restoration of non-carious cervical lesions: 2-year results of a randomized controlled practice-based study. Dent Mater. 2011 Mar;27(3):304-12. Epub 2010 Nov 30.

8) Van Meerbeek B, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Peumans M.
A randomized controlled study evaluating the effectiveness of a two-step self-etch adhesive with and without selective phosphoric-acid etching of enamel. Dent Mater. 2005 Apr;21(4):375-83.

9) Frankenberger R, Lohbauer U, Roggendorf MJ, Naumann M, Taschner M.
Selective enamel etching reconsidered: better than etch-and-rinse and self-etch? J Adhes Dent. 2008 Oct;10(5):339-44.

10) Kawamoto C, Fukuoka A, Sano H.
Bonding performance of the new TOKUYAMA Bond Force bonding system, *The Quintessence*, vol. 26 no.3/2007-0614

11) Tagami J, Ito S, Okuma M, Nakajima M.
Performance and features of the new Bond Force adhesive resin, *The Nippon Dental Review*, vol. 67 (4)/Weekly No. 744, 163

12) Hosaka K, Nakajima M, Takahashi M, Ito S, Ikeda M, Tagami J, Pashley DH.
Relationship between mechanical properties of one-step self-etch adhesives and water sorption. Dent Mater. 2010 Apr;26(4):360-367

13) Mariam Margvelashvili, Cecilia Goracci, Milos Beloica, Federica Papacchini, Marco Ferrari.

In vitro evaluation of bonding effectiveness to dentin of all-in-one adhesives. Journal of Dentistry. 2010; 38:106-112
14) The Dental Advisor Vol.27, No.1, 2010
15) The Dental Advisor Vol.28, No.1, 2011