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Clinical Evaluation and Interfacial Morphology Observation of Xeno III Self-etching Resin Bonding and Restorative System

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This study was a clinical trial of a one-step, total priming and bonding system, Xeno III. Thirty restorations were placed in minimally invasive V-shaped, Class V cervical cavities. Immediately after placement, baseline records were made after restorations were assessed using modified Ryge/USPHS criteria. Subsequently, restorations were evaluated at recall intervals up to 18 months. Additionally, the measurement of tensile bond strength and the FE-SEM observation of resin-tooth interface were performed *in vitro*. At 18 months, all restorations were classified as clinically satisfactory and assigned with an Alpha rating. Tensile bond strength of Xeno III was not significantly different from that of Clearfil SE Bond. Resin-enamel/dentin interface was very tight, with the presence of a very thin hybrid layer at the superficial dentin. Based on the results obtained, the Xeno III resin bonding system seemed promising as a one-step, self-etch adhesive.

Keywords: Clinical performance, Survival rate, Interfacial morphology

INTRODUCTION

With the advent of vastly improved adhesive restorative materials and an increased understanding of the caries process, the concept of “Minimal Intervention” (MI) was conceived and established — thereby enabling a decrease in surgical intervention of cavitated lesions[1]. In accordance with the MI concept, esthetic adhesive resin composite restorations have been widely performed, including areas such as the occlusal surface of posterior teeth. To date, it has seemed that resin composite restorations could be indicated for any cavity because of their highly esthetic property, increased mechanical properties, and strong bond strength to hard tooth tissues.

However, secondary caries sometimes develops around or under the resin composite restorations shortly after placement — even when proper cavity preparation and precise bonding procedure are duly conducted[2]. One proposed solution to overcoming this problem is to add a fluoride-releasing monomer to the restorative material. In light of this proposed anti-plaque approach, Xeno III was developed. It is a two-bottle, one-step total priming and bonding system, containing the fluoride-releasing phosphazene monomer that does not degrade after curing. In other words, the phosphazene monomer could be utilized to prevent plaque accumulation.

The purpose of the present study, therefore, was to evaluate the Xeno III system in these three-fold aspects: (1) clinical assessment of restorations using modified Ryge/USPHS criteria for a short-term period up to 18 months; (2) tensile bond strength measurement; (3) observation of interfacial ultrastructure between resin and tooth tissues by means of FE-SEM.

MATERIALS AND METHODS

Clinical performance of Xeno III resin bonding system

Thirty trial restorations were placed in a total of 30 patients using a one-step, self-etching resin bonding and restorative system, XENO III and XENO CF paste (Dentsply Sankin). Two operators in the Department of Dentistry, Toranomon Hospital, placed all the restorations. Baseline records were made by the practitioners immediately after placement. Patients were followed up routinely at recall intervals that were appropriate to their oral health status. At each recall, the restorations were evaluated for clinical acceptability.

1) Selection of patients and teeth

Trial patients were selected under these conditions: no serious diseases nor inferior oral environment. The mean age of the 30 patients — 13 females and 17 males — was 58.2±9.1 years. All the 30 restorations were placed to restore original cervical lesions. Half of the restorations were placed in the maxillary arch and the other half in the mandible. Three restorations were placed in incisors, eleven in canines, fifteen in premolars, and one in a molar. All teeth restored were reported to be vital and asymptomatic prior to restoration placement. By the very definition of a minimally invasive restoration, the cervical lesion requiring treatment should not be symptomatic.

2) Placement technique

The operators were all requested to use the simple dry field technique with cotton pellets during the placement of restorations. Rubber dam was not used in any placement of the 30 restorations. Standard water-cooled rotary instruments were used to prepare the cavities with minimally invasive approach. No linings were placed under any restoration, and all restorations were directly bonded to the exposed tooth surface.

The Xeno III system consisted of two liquid bonding agents and accessories. Mixed liquids of the Catalyst and the Universal had both conditioning and bonding effects. The Catalyst liquid comprised pyrophosphate ester (methacryloyloxyethyl acid phosphate), UDMA, fluoride-releasing phosphazene monomer, and photosensitizer. The Universal liquid comprised HEMA, water, ethanol, and microfiller particles.

The mixed liquid was applied to the cavity for 20 seconds, gently air-dried to evaporate alcohol solvent and water, and then light-cured for 10 seconds. There was no need to rinse off Xeno III. All Xeno CF resin composite restorations were placed following the manufacturer's directions and as outlined in the Directions for Use document as prepared by the manufacturer.

3) Evaluation

All restorations were examined immediately after placement. Restorations were assessed using modified Ryge/USPHS criteria[3] for retention, marginal discoloration, marginal adaptation, surface texture, abrasion, marginal fracture, and body fracture. Restored teeth were assessed for secondary caries, spontaneous pain, cold water pain, hot water pain, and occlusal pain. Soft tissue around restored teeth was also assessed for gingival irritation and soft tissue irritation.

Alpha rating was assigned when the restoration was performing satisfactorily in all categories of examination and showing no signs worthy of specific review at the next visit. Bravo rating was assigned when the restoration was performing satisfactorily, but a note was made of an aspect that required specific review at the next visit because it was outside acceptable parameters; nonetheless, the restoration had not failed nor was deemed to be so. Charlie rating was assigned when a restoration had failed in any of the assessed aspects and had to be replaced. The same assessment criteria were used at all subsequent examination visits up to 18 months.

Tensile bond strength measurement of Xeno III to tooth tissues

The tensile bond strengths of Xeno III to enamel and dentin were measured using extracted bovine teeth. Flat enamel and dentin surfaces were prepared by grinding the labial side of the bovine incisors, finished with #600 SiC paper under running water, and a bonding area of 4 mm in diameter was defined using vinyl tapes.

Tooth surfaces were treated with Xeno III mixed liquid for 20 seconds, gently air-dried, and light-cured for 10 seconds before Xeno CF resin paste was placed and light-cured. Stainless steel rods were then bonded to the set resin composite surfaces. After which, tensile bond strength was measured at a crosshead speed of 1.0 mm/min after 24 hours, and the results compared with Clearfil SE Bond (Kuraray Medical). For each material, the number of specimens was 10 to both enamel and dentin. Data were analyzed statistically by Student's t-test ($P<0.05$).

FE-SEM observation of Xeno III-tooth interface

Bonded assemblies were prepared according to the manufacturer's instructions by using freshly extracted human third molar teeth. After the resin paste had set, specimens were cut perpendicularly at the center of the teeth. Half of the cut specimen was embedded in an epoxy resi (Nissin EM; Epon 815: 2 g, Tohmide 245: 1 g, DMP-30: 0.12 g). After one week, the cut surfaces of the specimens were finished and polished with SiC papers #600, #800, #1000, #1200, #1500 and diamond pastes of 6 μm, 3 μm, 1 μm, 0.25 μm. The resin-enamel/dentin interface was then observed under a FE-SEM (ERA-8800FE, Elionix, Tokyo, Japan) after Ar ion beam etching (EIS-200ER, Elionix, Tokyo, Japan) for 35 seconds.

RESULTS

Clinical evaluation of Xeno III until 18 months

Data available for all assessment periods were 100 per cent. The recall response rate of each period was quite good because this study was a short-term one up to 18 months. The evaluation levels for each criterion are shown in Table 1. At the review, all restorations that were examined were rated Alpha. In other words, no restorations were rated Bravo or Charlie. A Kaplan Meier survival analysis was used to determine the survival probability of each restoration for each recall interval (Table 2). At the end of 18 months, the Kaplan–Meier's probability of survival for the experimental restorations was 1.00.

Tensile bond strengths of Xeno III

Figure 1 shows the tensile bond strengths of Xeno III and Clearfil SE Bond to bovine teeth. All data are expressed as mean ± SD. Bond strength to enamel using Xeno III was about 21.3 MPa, which was slightly higher than Clearfil SE Bond (19.4 MPa), but not significantly different ($P>0.05$). With dentin, the bond strength of Xeno III was about 15.4 MPa, which was also not statistically different from Clearfil SE Bond ($P>0.05$).

FE-SEM observation of Xeno III-tooth interface

The FE-SEM images of resin-enamel interface after Ar ion beam etching are shown in Fig. 2 (a: ×5,000; b: ×20,000). The superficial enamel layer was finely and densely etched, and the apatite crystals of

Table 1 Evaluation levels for all assessment criteria

		Month	1.5	3	6	12	18
Retention of restoration	(−)	Presence of restoration	30	30	30	30	30
	(+)	Loss of restoration	0	0	0	0	0
Marginal discoloration	(A)	None	30	30	30	30	30
	(B)	Moderate	0	0	0	0	0
	(C)	Severe	0	0	0	0	0
Marginal adaptation	(A)	Excellent	30	30	30	30	30
	(B)	Good	0	0	0	0	0
	(C)	Fair	0	0	0	0	0
	(D)	Poor	0	0	0	0	0
Abrasion	(A)	None	30	30	30	30	30
	(B)	Slight	0	0	0	0	0
	(C)	Severe	0	0	0	0	0
Surface texture	(A)	Smooth	30	30	30	30	30
	(B)	Slightly rough	0	0	0	0	0
	(C)	Rough	0	0	0	0	0
Marginal fracture	(−)	None	30	30	30	30	30
	(+)	Detected	0	0	0	0	0
Body fracture	(−)	None	30	30	30	30	30
	(+)	Detected	0	0	0	0	0
Secondary caries	(−)	None	30	30	30	30	30
	(+)	Detected	0	0	0	0	0
Spontaneous pain, Cold water pain, Hot water pain, Occlusal pain	(A)	None	30	30	30	30	30
	(B)	Slight	0	0	0	0	0
	(C)	Severe	0	0	0	0	0
Gingival irritation, Soft tissue irritation	(−)	None	30	30	30	30	30
	(+)	Detectd	0	0	0	0	0

Table 2 Kaplan−Meier survival analysis

Month	1.5	3	6	12	18
Total restorations	30	30	30	30	30
Satisfactory (Alpha)	30	30	30	30	30
Potential failure (Bravo)	0	0	0	0	0
Failure (Charlie)	0	0	0	0	0
Kaplan−Meier statistic	1	1	1	1	1

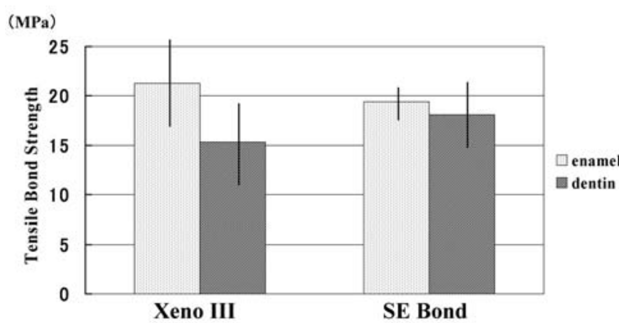

Fig. 1 Tensile bond strengths of Xeno III and Clearfil SE Bond to tooth tissues.

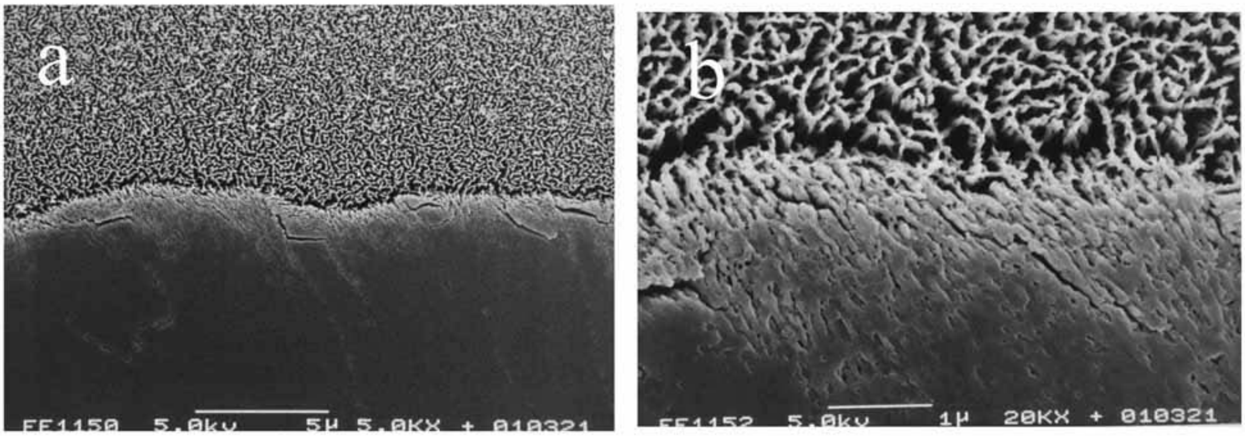

Fig. 2 SEM images of resin-enamel interface after Ar ion beam etching, where a: ×5000, b: ×20000. There was tight bonding interface between resin and enamel without any gap formation. Apatite crystals of superficial enamel prism were lightly decalcified.

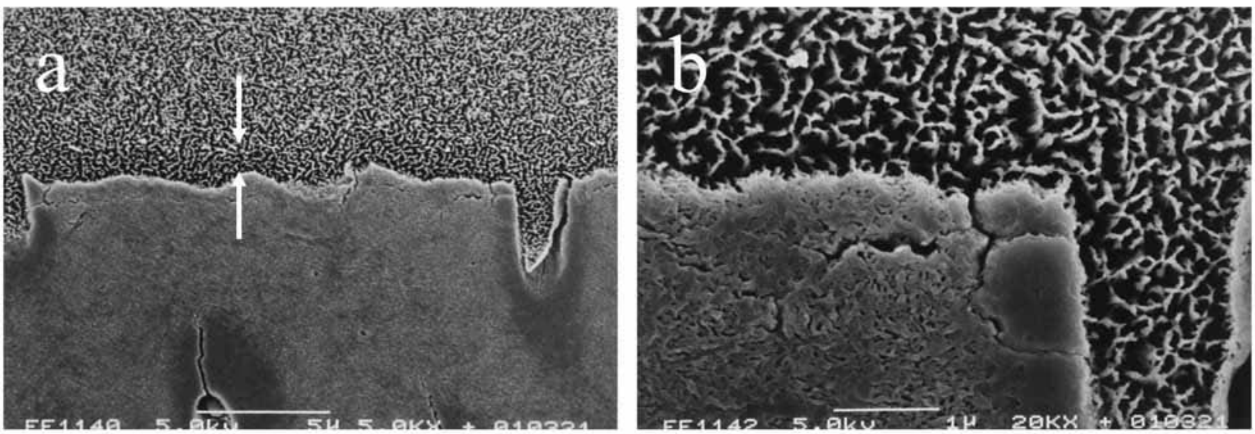

Fig. 3 SEM images of resin-dentin interface after Ar ion beam etching, where a: ×5000, b: ×20000. There was tight bonding interface between resin and dentin. When subjected to Ar ion beam etching, a hybrid layer of about 1 μm thickness clearly appeared.

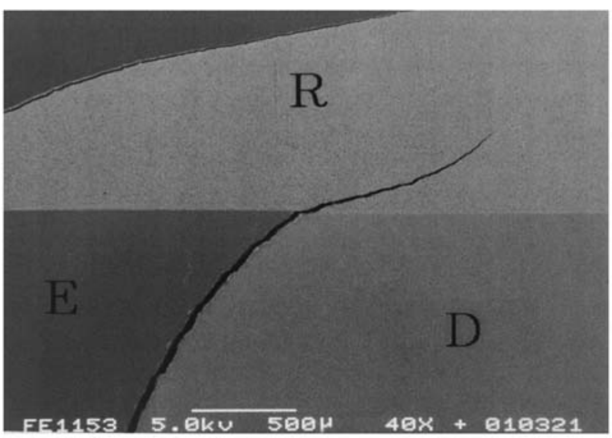

Fig. 4 SEM image of dentinoenamel junction (DEJ) adjacent to the interface after Ar ion beam etching (×40). A crack initiated from DEJ and propagated into the set resin composite, but not along the interface.

enamel prism were slightly decalcified. Nonetheless, this demineralization process was not so destructive when compared to conventional phosphoric acid etching, and there was a tight bonding interface between enamel and resin.

Figure 3 shows the FE-SEM images of resin-dentin interface after Ar ion etching (a: ×5,000; b: ×20,000). Similarly, there was a tight bonding interface between resin and dentin, and the hybrid layer produced was approximately 1 μm thick (Fig. 3a: indicated by arrows).

Figure 4 shows the FE-SEM image of dentinoenamel junction (DEJ) adjacent to the interface. It could be seen that a crack initiated from DEJ to the set resin composite, but not along the interface.

DISCUSSION

In operative dentistry, the principles of minimal intervention (MI) dentistry have been widely accepted largely due to the development and emergence of advanced adhesive restorative materials with strong bonding efficacy to tooth tissues. These materials have since made possible the maximum preservation of intact tooth tissues. Apart from restorative materials, the landscape of treatment technique has also changed during the last 15 years. Treatment focus is not only on less surgical intervention, but also with simplified clinical procedures.

Against this backdrop, two-step self-etching bonding systems — consisting of self-etching primer and bonding resin — have emerged fast and furious on the market[4]. In most of these systems, adhesive resin monomers could penetrate the decalcified dentin layer sufficiently, producing a thin but strong hybrid layer of high quality[5]. However, these two-step systems are thought to be still technically sensitive[6]. Recently, with a view to simplifying clinical procedures and reducing technical sensitivity, several one-step bonding systems have been developed[7].

Xeno CF Bond system[8], marketed in 1999, was the first one-step priming and bonding system in Japan. It was a two-liquid self-etching adhesive system containing pyrophosphate ester as an adhesion monomer, as well as fluoride-releasing phosphazene monomer (PEM-F) which was synthesized by Anzai *et al.*[9]. As for Xeno III, marketed in 2001, it was also a two-bottle one-step system — but which was improved in terms of storage stability by changing the loading ratio of filler particles[10].

PEM-F of the self-etching adhesive, which contains much fluoride, is very chemically stable and does not degrade after light curing. Conversely, an inorganic fluoride like sodium fluoride (NaF) in the bonding resin tends to hydrolyze in a short term and reduce the mechanical properties of the bonding resin. Anzai *et al.*[9] reported that the fluoride release amount of PEM-F was about 300 $\mu g/cm^2$ after two months, and that level was maintained over a period of one year. On the aspect of anti-plaque activity, this fluoride-releasing property of phosphazene seemed to be effective in preventing secondary caries around restorations.

On the aspect of simplified clinical procedures, a reliable restoration should be accomplished within a short time. To fulfill this objective, one-step systems which are so-called "all-in-one adhesives" have been recently developed and marketed[11]. However, clinical reports of such two-step or one-step adhesive systems are very scarce[12-14]. In light of such information scarcity, this study set out to examine the short-term clinical efficacy of the Xeno III bonding system — in a group of patients under conditions which represented its normal conditions of use. Additionally, FE-SEM observation of the interfacial ultrastructure between the resin and tooth tissues was performed *in vitro*.

In terms of clinical evaluation, the recall response rate of each period was quite favorable. This was chiefly because a six-month recall system was adopted, and that these patients also visited the medical departments of Toranomon Hospital periodically. All the 30 restorations were considered clinically satisfactory according to the assessment criteria, and thus graded "Alpha" during the period of 18 months after placement.

In terms of tensile bond strength, the present study showed that this resin bonding system yielded strength values which were quite high: 20 MPa to bovine enamel and 15 MPa to dentin. In this study, bovine teeth were used for conventional tensile bond strength measurement as a preliminary means to predict the adhesion properties with the results compared against Clearfil SE Bond.

In terms of FE-SEM examination, it was revealed that a tight bonding interface was obtained between the resin and enamel/dentin, whereby a thin hybrid layer was created at the dentin interface. Unlike the DEJ which is a natural, multilevel interface, the resin-enamel/dentin interface was created artificially by Xeno III resin bonding system — but which was noted to be clinically acceptable. During SEM sample preparation, the substantial shrinking of the dentin structure led to the destruction of DEJ, resulting in a wide and large crack along the junction. However, the resin-enamel/dentin interface maintained its marginal integrity. These findings thus suggested that the adhesion between the resin and tooth tissues was stronger than that of DEJ, thereby supporting the clinical evaluation results in this study.

At this juncture, it should be mentioned that some studies concluded that one-step self-etch adhesives like Xeno III might be inferior in terms of marginal adaptation when compared to etch-and-rinse or two-step self-etch adhesives after thermomechanical loading[15]. In view of contradictory and conflicting conclusions on Xeno III, further *in vivo* and *in vitro* laboratory and clinical studies might be necessary to estimate the longer-term clinical performance of this resin bonding system.

CONCLUSIONS

The Xeno III resin bonding system developed by Dentsply Sankin K.K. was a brand-new, one-step total priming and bonding system. Xeno III contained a fluoride-releasing monomer, phosphazene, that does not degrade after curing, and thus could be beneficial and effective in preventing secondary caries around restorations. Bonding interface between resin and dentin/enamel was tighter than that of DEJ, with the presence of a thin hybrid layer at the interface. Short-term clinical performance of Xeno III at 18 months after placement was considered to be clinically satisfactory. Based on the results obtained in this study, this resin bonding system was thought to be a very promising candidate for minimally invasive resin composite restorations.

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