

RESIN TAG FORMATION OF SELF-ETCHING ADHESIVES

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국문초록

본 연구는 자가부식 접착제의 치질로의 침투와 레진 tag 형성을 알아보고 부가적인 산부식이 레진 tag 형성에 미치는 효과를 관찰하기 위하여 계획되었다.

3종의 자가부식 접착제(SE bond, AQ bond and L Pop)와 one bottle adhesive(Single bond)를 사용하였고 발거한 구치로 교합면 법랑질과 상아질 시편을 제작하였으며 각 군당 5개씩 네 개의 군으로 나눈 총 20개의 시편을 양분하여 각각 접착제를 적용하기 전 35% 인산으로 산부식하거나 산부식처리하지 않았으며 이후 레진을 적용시키고 중합하였다. 시편은 Silverstone microtome으로 절단한 후 HCl 용액과 NaOCl 용액으로 처리하고 건조시킨 후 이온으로 피복하였으며 주사전 자현미경으로 관찰하여 다음과 같은 결과를 얻었다.

1. 부가적 산부식 처리한 상아질은 모든 자가부식 접착제군에서 산부식하지 않은 상아질보다 resin tag의 길이와 두께가 증가하는 것이 관찰되었다.
2. 법랑질에서는 L Pop을 제외하고 부가적 산부식한 군에서 레진 tag의 두께가 산부식하지 않은 군보다 컸고 보다 명확한 부식양상을 관찰할 수 있었다. L Pop에서는 부식법랑질과 비부식법랑질간의 차이가 없었다.

본 연구의 결과는 자가부식형 접착제가 법랑질을 부식시키지 않고 상아세관으로 침투하는 정도가 산부식군에 미치지 못하다는 것을 의미한다. 이와 관련하여 접착강도와 미세누출 및 레진 tag 형태에 대한 부가적 연구가 필요할 것이다.

주요어 : 자가부식, 레진 tag, 주사전자현미경

I. Introduction

Since the concept of modifying the enamel structure with acid-etch technique was first introduced in dentistry¹⁾, the acid-etch technique with phosphoric acid has become a standard procedure for surface conditioning of enamel and dentin²⁾. Acid Etching creates a porous enamel surface layer ranging in depth from 5 to 50 μ m. The inflow of a low-viscosity bonding agent into the microporosities results in the formation of resin tag³⁾.

Adhesion to dentin, on the other hand, was achieved by the infiltration of resin monomers into the exposed collagen web and dentinal tubules following acid etching of dentin⁴⁻⁷⁾.

Recently, on the purpose of simplifying procedure and preserving the smear layer, self-etching primer was developed. By containing acidic functional monomers, self-etching primers has combined the

tooth surface etching and priming steps to simultaneously treat enamel and dentin. The rationale for these acidic monomers is the formation of continuum between the tooth surface and the adhesive material by the simultaneous demineralization and resin penetration of enamel and dentin surface with acidic molecules that can be polymerized in situ⁸⁾. However, there was a report that some self-etching primer resulted in less demineralization of enamel than do conventional acid etchants⁹⁾.

Especially in dentin, it allowed simultaneous demineralization and resin infiltration of the smear layer-covered dentin¹⁰⁾, self-etching primers with different degrees of aggressiveness may either completely dissolve or preserve the smear layer^{8,11)}. Despite the formation of thin hybrid layers of around 0.5 μ m with the use of the milder versions of self-etching primers, high initial bond strength has been reported for sound dentin^{12,13)}.

The bond strengths and sealing ability of dentin bonding systems seem to be unrelated to hybrid layer thickness but rather seem to correlate to dentin location and to the quality of dentin¹⁴⁻¹⁶. For this reason, resin tag morphology, numbers and lateral branches are more clinical and morphological importance than the thickness of the hybrid layer¹³.

It has not been well established if the new self-etching primer/adhesive systems are able to adequately infiltrate the dentin and enamel surface. The purpose of this study was to observe resin tag of the resin/enamel, dentin interface produced by self-etching adhesive systems and evaluate effect of additional acid etching on resin tag formation.

II. Materials and methods

Twenty recently extracted caries-free human molars were used in this study. The teeth were cleaned of gross debris and washed in distilled water.

Occlusal surface of each tooth ground with a water-cooled Surfmeter grinder(Buehler Ltd, IL, USA) to create a flat enamel/dentin surface. For the purpose of producing a standardized smear layer, the flattened occlusal surfaces were then polished with wet on 240-, 320-, 400-, and 600-grit silicon carbide paper(Buehler Ltd, IL, USA) on Handmet grinder (Buehler Ltd, IL, USA) subsequently. And then, grounded tooth were initially separated the crown from roots and cross-sectioned using a low speed diamond saw.

The sectioned tooth specimens were then distributed at random into four groups of 5 teeth each group.

Three representative commercially available self-etching primer(SE bond, L Pop and AQ bond) and a one bottle adhesive(Single bond) were used. The materials used are listed in Table 1 and the schematic representation of the experimental steps for sample preparation was shown in Fig. 1.

Table 1. Self-etching adhesives and control used.

Adhesive	Type	Batch Number	Manufacturer
AQ BOND	Self-etching	VK3-2003-03	Sun Medical Co(Japan)
Clearfil: SE BOND	Self-etching	61129-2001-06	Kuraray Co,Ltd.(Japan)
Prompt L-POP(LP2)	Self-etching	FW0056061	ESPE Dental AG(Germany)
Single Bond	One bottle	19970717	3M Dental(USA)

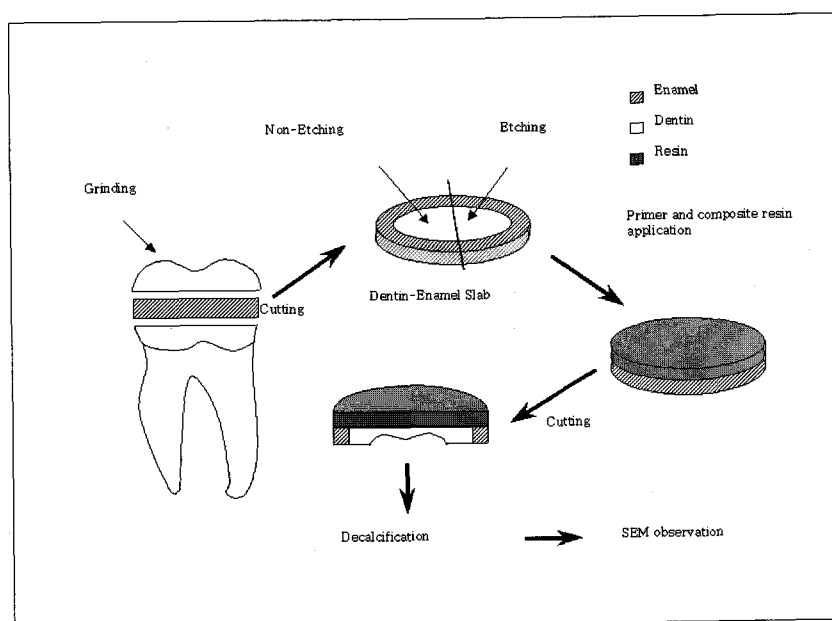


Fig. 1. Schematic representation of the experimental steps for sample preparation.

To avoid morphologic and structural variations of the dentin caused by different depth, one-half of each specimen in each group was etched with 35% phosphoric acid prior to the application of each adhesive system, with the second half being kept unetched. Subsequently, resin composite was placed and polymerized. Sectioned resin-tooth specimens immersed into 10N HCl for 24 hours to totally removed the calcified component, washed with distilled water for 5 minutes, then immersed in 5% NaOCl solutions for 20 minutes to removed the organic components. After dehydration procedure, the specimens were mounted on aluminum stubs with silver paint and sputter coated with gold-palladium, then examined with a scanning electron microscopy(JEOL, JSM-840A, Tokyo, Japan).

III. Result

The results are shown in Figs. 2-17. The specimens showed a resin replica of conditioned enamel/dentin created by resin penetration into the enamel microporosities and dentinal tubules exposed by etching.

Additional etching side dentin displayed longer and thicker resin tag than unetched side in all self-etching adhesive groups. Additional etching increased the number of resin tags and lateral branches also. Unetched dentin surfaces of SE Bond and AQ bond showed shorter and thinner resin tag than etched dentin surfaces. Dentin surface of L POP and Single bond showed very long resin tag with lateral branches in additional etching side, the other way, few resin tag in unetched dentin.

In enamel, additional etching side displayed deeper and more distinct etching pattern than unetched side in SE Bond and AQ bond. There is no difference between etched and unetched enamel in L Pop. Unetched enamel surface of Single bond showed replica of cut irregular enamel surface.

IV. Discussion

In dentin surface, the SEM observations of this investigation showed, in accordance with previous study¹⁷⁾, that characteristic reverse cone-shaped tags with their corresponding adhesive lateral branches

were evident in additional etching area and conventional adhesive control group. Self-etching primer caused funneling of the tubule in dentin surface. However, comparing with additional etching with phosphoric acid, resin tags found in samples of the self-etching primer were narrower at the tubule orifices, and adhesive lateral branches were scarce. The length of the resin tags observed in the samples of additional etching area and conventional adhesive control group were longer than those found in the self-etching primer. It could be explained that the smear plug was not removed in self-etching primer of the total-etch-technique, all smear layer removed and then penetrated the dentinal tubules with adhesive creating a hybrid layer. Also according to Milia et al¹⁸⁾, the use of a self-etching primer did not produce significant morphological changes in the moist dentin substrate. Adverse morphological conditions were observed when there was excess water on the dentin surface. Phosphoric acid altered the collagen more severely than the self-etching primer did. However, a recent study¹⁹⁾ about hybrid layer, showed that the resin cement did not penetrate the depth of the zone of demineralized dentin when the self-etch primer was used in combination with conventional acid etch treatment. Inadequate resin cement penetration left a substantial area of exposed protein at the dentin/cement interface. In contrast, there was complete resin cement diffusion throughout the demineralized dentin when the self-etch primer was used without acid etching.

The morphology and the role of the resin tags in dentin depend upon several factors such as dentin bonding agent used, orientation of the dentinal tubules, type of dentin surface, presence and density of branches of dentinal tubules, different in vivo conditions and dentin depth²⁰⁻²³⁾.

In contribution of resin tags to bonding, Gwinnett²⁴⁾ attempted to determine quantitatively, the role of resin infiltration in dentin bonding, it was concluded that resin infiltration can contribute approximately one third of the shear bond strength of this total etch system. Superficial dentin has few dentinal tubules and is composed largely of intertubular dentin²⁵⁾. Deep dentin near the pulp, especially after acid etching, is composed mainly of larger funnel shaped dentinal tubules with much less intertubular dentin.

Although hybrid layer refer to resin-infiltrated demineralized intertubular dentin, resin tags in each dentinal tubule usually penetrate the hybrid layer. These tags represent a minor fraction of superficial dentin sealed by resin, but they represent a significant fraction of bonded surfaces of dentin near the pulp chamber. Theoretically, polymer tags could contribute to resin retention if they are firmly bonded to the walls of the tubules. Their contribution to the total retentive strength of the bond should be proportional to the total cross-sectional area of the tags and the cohesive strength of the polymer^{26,27)}.

In enamel surface, the orientation of the enamel rods is important factor. The goals of enamel etching are to clean the enamel, to remove the enamel smear layer, to increase microscopic roughness by removal of prismatic and interprismatic mineral crystals, and to increase the surface free energy of enamel to produce enough monomer infiltration to seal enamel surfaces with resin and to contribute to the retention of resin composite restorations^{28,29)}.

The use of additional etchants resulted in the deepest etching patterns in accordance with previous study. Perdigao et al³⁰⁾ reported that the use of Clearfil Liner Bond 2, according to manufacturer's directions, resulted in a poorly-defined etching pattern. Regardless of the alternative etchant used, the use of the self-etching primer did not affect the mean enamel shear bond strength.

And Shintchi et al³¹⁾ also show that though the resin tag length of various acid concentrations was different significantly, the tensile bond strength of resin to enamel pre-treated with various acid concentrations did not vary significantly. However, further studies about relationship of etching pattern and bonding strength are needed.

Because the self-etching primer is not rinsed off, in contrast to conventional etchants, proper application of self-etching primer plays an important role in getting good bond strength. After the application of self-etching primer, the primed enamel and dentin surface should be air dried because the primer contains solvents like water, ethanol and acetone. It could be explained that these solvents may have an adverse effect on the polymerization of the bonding agent and resin tag formation^{32,33)}.

Miyazaki et al³³⁾ suggested that the enamel bond

strengths of self-etching systems can be influenced by the drying time of the primer applied prior to bond agent application.

In the present study, the results obtained suggest that the formation of resin tags and lateral branches could be related to the facts that the conditioner of the system has a relatively high concentration of adhesive bonding agent and the diffusion of monomers into the demineralized substrate can be promoted by the system as well. Although the self-etching primer dissolved the smear layer and produced resin tags with characteristic reverse cone-shaped and lateral adhesive branches, the self-etching adhesive did not etch enamel and penetrate into dentinal tubule as deeply as additional etching did. Further research should include the evaluation of the relationship of bonding strength, microleakage and resin tag morphology.

V. Conclusion

The resin tag formation of three self-etching primer(SE bond, AQ bond and L Pop) and an one bottle adhesive(Single bond) were examined. A total of 20 surfaces were collected and divided into four groups of 5 samples from human occlusal enamel and dentin disks. One-half of each specimen in each group was etched with 35% phosphoric acid prior to the application of each adhesive system, with the second half being kept unetched. Subsequently, resin composite was placed and polymerized. The samples were sliced and examined with a SEM.

The results were as follows:

1. Additional etching side of dentin displayed longer and thicker resin tag than unetched side in all self-etching adhesive groups.
2. In enamel, additional etching side displayed deeper and more distinct etching pattern than unetched side except L Pop. There is no difference between etched and unetched enamel in L Pop.

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사진부도 설명

- Fig. 2. SEM of SE Bond (unetched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed short and thin resin tag morphology.
- Fig. 3. SEM of SE Bond (etched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed longer and thicker resin tag than unetched side.
- Fig. 4. SEM of SE Bond (unetched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed shallow etching pattern.
- Fig. 5. SEM of SE Bond (etched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed deeper and more distinct than unetched side.
- Fig. 6. SEM of AQ bond (unetched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed short and thin resin tag morphology.
- Fig. 7. SEM of AQ bond (etched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed longer and thicker resin tag with more lateral branches than unetched side.
- Fig. 8. SEM of AQ bond (unetched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed shallow and irregular etching pattern.
- Fig. 9. SEM of AQ bond (etched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed deeper and more distinct than unetched side.
- Fig. 10. SEM of L Pop (unetched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed few resin tag and irregular dentin surface.
- Fig. 11. SEM of L Pop (etched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed very long resin tag with lateral branches.
- Fig. 12. SEM of L Pop (unetched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed typical etching pattern.
- Fig. 13. SEM of L Pop (etched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed deep and distinct etching pattern.
- Fig. 14. SEM of Single bond (unetched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed replica of prepared dentin surface and no resin tag.
- Fig. 15. SEM of Single bond (etched dentin), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed very long resin tag with lateral branches.
- Fig. 16. SEM of Single bond (unetched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed replica of prepared irregular enamel surface.
- Fig. 17. SEM of Single bond (etched enamel), treated with both HCL and NaOCl to remove mineral and protein, leaving resin tags($\times 2000$). It showed deep and distinct etching pattern.

사진부도 ①

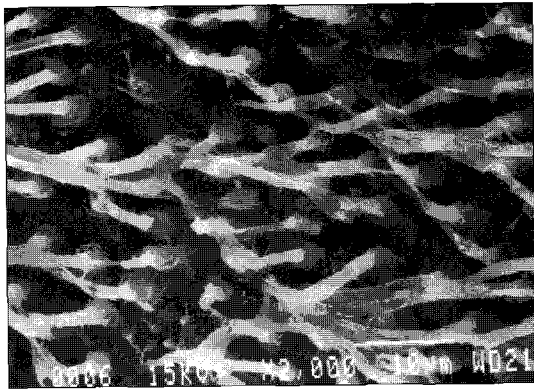


Fig. 2

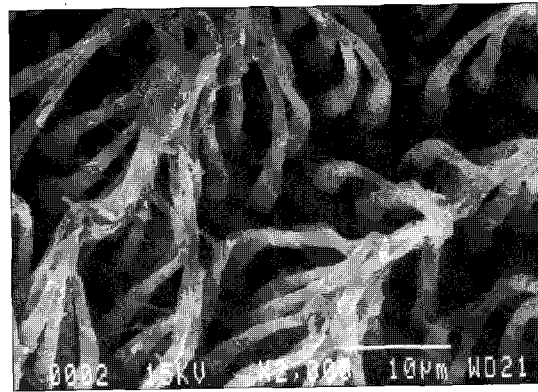


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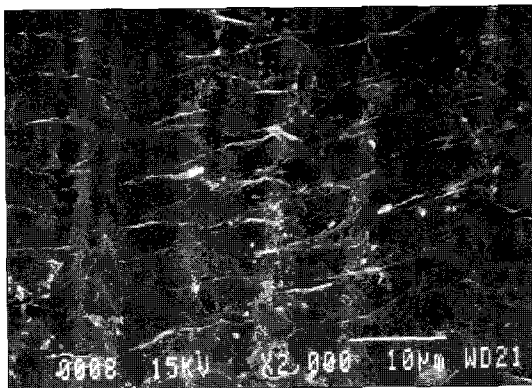


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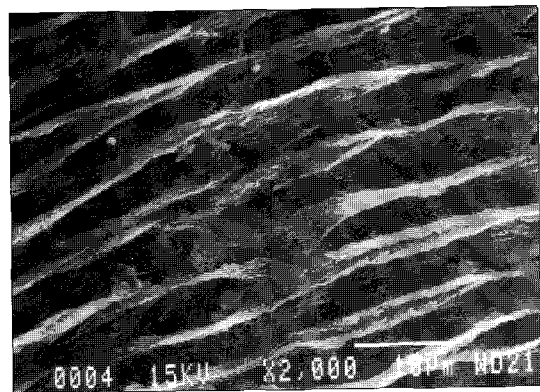


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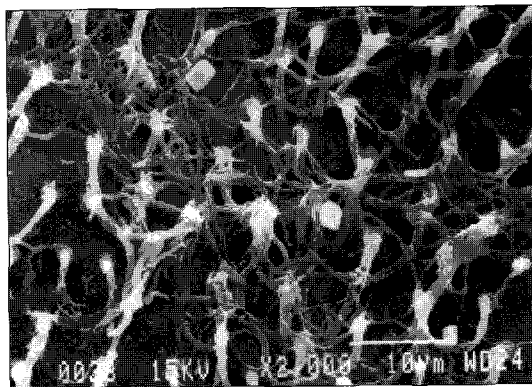


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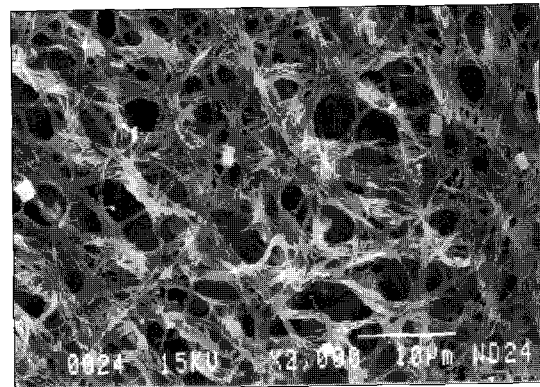


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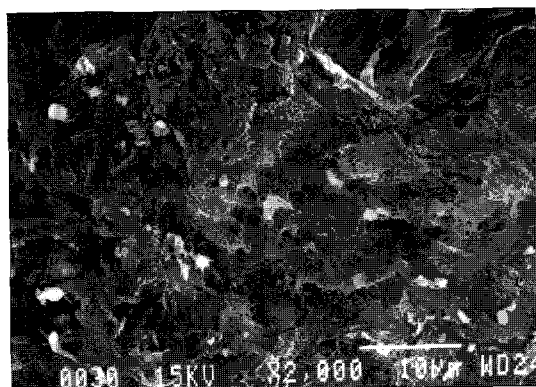


Fig. 8

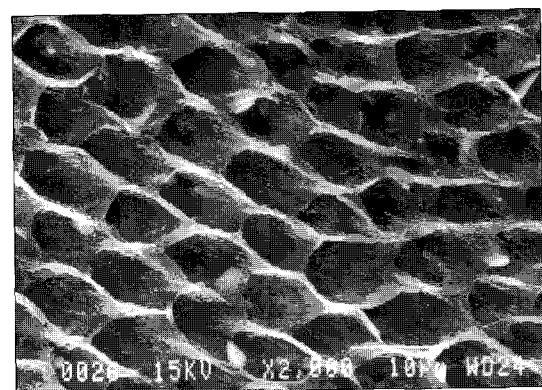


Fig. 9

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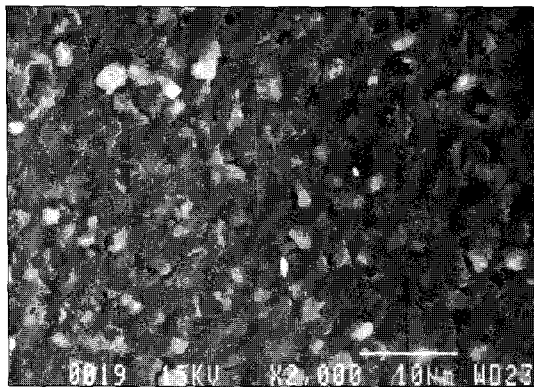


Fig. 10

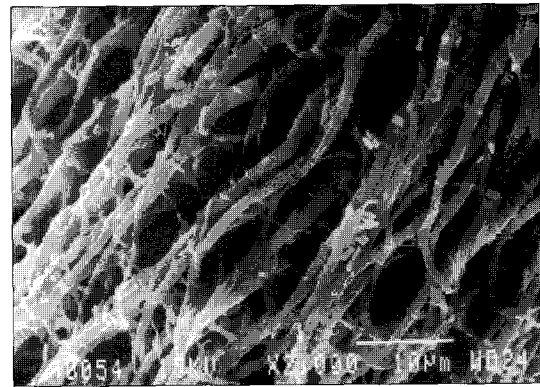


Fig. 11

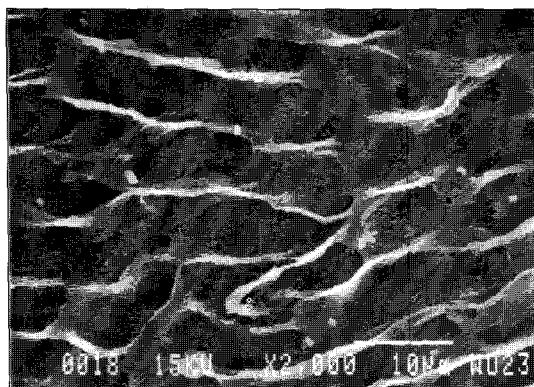


Fig. 12

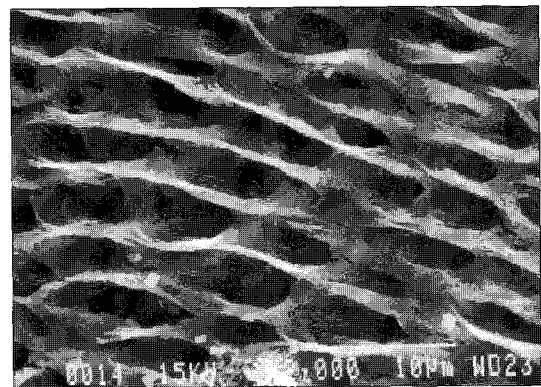


Fig. 13

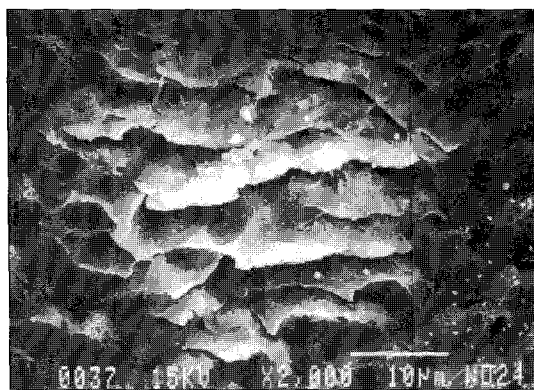


Fig. 14

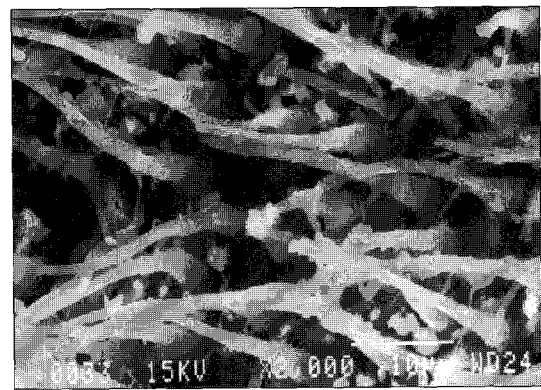


Fig. 15

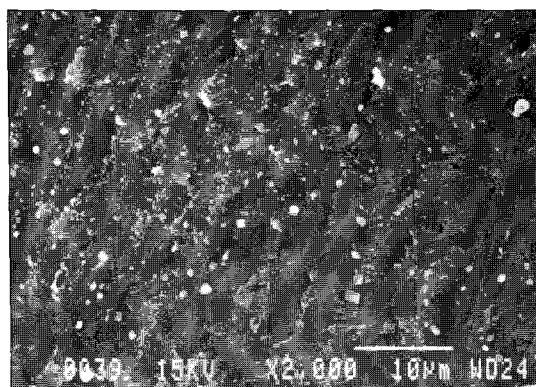


Fig. 16

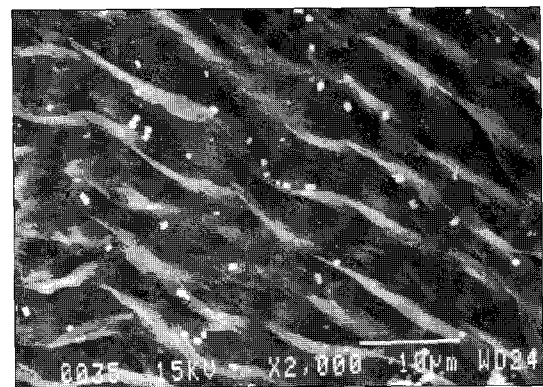


Fig. 17

Abstract

RESIN TAG FORMATION OF SELF-ETCHING ADHESIVES

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The aims of the present study was to observe resin tag of the resin/enamel, dentin interface produced by self-etching adhesive systems and evaluate effect of additional acid etching on resin tag formation

Three self-etching primer(SE bond, AQ bond and L Pop) and an one bottle adhesive(Single bond) were used. Flat occlusal enamel and dentin disks were obtained from extracted human molars. A total of 20 surfaces were collected and divided into four groups of 5 samples. One-half of each specimen in each group was etched with 35% phosphoric acid prior to the application of each adhesive system, with the second half being kept unetched. Subsequently, resin composite was placed and polymerized. The samples were sliced and immersed into HCl and NaOCl solutions, followed by drying and sputter coating for examination with a SEM.

The results were as follows:

1. Additional etching side of dentin displayed longer and thicker resin tag than unetched side in all self-etching adhesive groups.
2. In enamel, additional etching side displayed deeper and more distinct etching pattern than unetched side except L Pop. There is no difference between etched and unetched enamel in L Pop.

The results obtained suggest the self-etching adhesive did not etch enamel and penetrate into dentinal tubule as deeply as did additional etching. Further research should include the evaluation of the relationship of bonding strength, microleakage and resin tag morphology.

Key words : Self-etching, Resin tag, SEM