

진동이 상아질 결합력에 미치는 영향에 관한 연구

이 진 · 김정욱 · 이상훈 · 김종철

서울대학교 치과대학 소아치과학교실 및 치학연구소

국문초록

초음파 진동을 치의학에 처음 이용한 것은 치석 제거기였으나, 최근에는 주조금속과 레진 인레이를 접착할 때, 인산아연시멘트나 레진시멘트의 점도를 낮추는데 이용되고 있다. 이러한 초음파 진동은 재료의 흐름성을 증가시킴으로써 피막도를 낮추고, 따라서 수복물 주위의 미세누출을 감소시킬 수 있는 장점이 있다. 그러나, 복합레진의 수복에 있어서 이러한 초음파 진동의 임상적 효과에 관한 연구가 아직까지 미흡한 실정이다. 이에 저자는 초음파 진동을 상아질 결합제에 적용하여 점도를 감소시킴으로써 상아세관으로의 레진 침투 정도의 변화와 결합력에 미치는 효과를 알아보고자 하였다.

발거 후 실온의 0.1% thymol 용액에 보관된 88 개의 건전한 사람의 대구치를 치관부 법랑질을 제거하고 아크릴 레진을 이용하여 직경 1-inch의 PVC 관에 매몰하였다. 각 시편의 교합면이 아크릴봉과 동일한 높이가 되도록 220-, 500-grit의 연마지로 순차적으로 연마하였고, 무작위로 추출하여 각 군당 22개씩 네 군으로 분류하였다. 1군과 2군은 Single Bond(3M-ESPE, St. Paul, USA)를 3군과 4군은 One-Step(Bisco Inc., Schaumburg, USA)을 제조사의 지시에 따라 치면을 산부식, 수세, 건조한 후 연속하여 2번을 도포하였다. 2군과 4군은 초음파 치석제거기를 이용하여 치면에 대고 10 초간 진동을 가한 후 광중합하였다. 이후 직경 2.3mm, 높이 3.5mm의 Teflon mold(Ultradent Products Inc., South Jordan, USA)를 이용하여 복합레진을 충전한 후 40 초씩 두 번에 나누어 광중합하였다. 모든 시편은 24시간 동안 실온의 수도물에 보관한 후 열순환을 시행하고, 만능측정기(Instron 4465, Canton, USA)로 전단결합강도를 측정하였으며 resin tag의 양상을 비교하기 위해 각 군의 시편의 치질을 탈회시킨 후에 표면을 주사전자현미경으로 관찰하여 다음과 같은 결과를 얻었다.

1. 초음파 진동을 가하지 않은 1군, 3군에 비해 초음파 진동을 가한 2군과 4군에서 전단결합강도가 유의하게 높게 나타났다($p < 0.05$).
2. Single Bond와 One-Step의 전단결합강도는 유의한 차이를 보이지 않았다($p > 0.05$).
3. 전자현미경 관찰에서 초음파 진동을 가한 군에서 resin tag의 길이가 길었고, lateral branch의 수도 많이 관찰되었다.

주요어 : 초음파 진동, 상아질 결합제, 점도, 전단결합강도, 레진 침투

I. Introduction

Dentin adhesive systems have been developed to obtain strong bonds between dental material and tooth substrate. It is important to achieve good marginal adaptation of the restorative material in order to reduce microleakage, staining, pulpal irritation, and recurrent caries¹⁾.

The adhesion of dental resin materials to enamel has been widely explained and is micro-mechanical in nature with the resin forming tag-like extensions into the etched enamel surface²⁾. However, the na-

ture of adhesion to dentin has been far more complex. Dentin contains more organic materials than enamel, which makes it an entirely different substrate for bonding³⁾. The target area for adhesion in dentin are the tubules, the intertubular dentin and peritubular dentin. More recently, manufacturers have replaced the bifunctional or multifunctional systems with new single-bottle systems, known as fifth generation dentin adhesive resins⁴⁾. These adhesives contain hydrophilic monomers that wet the dentin and penetrate into the dentinal tubules as well as the demineralized intertubular and peritubular

dentin⁴). Due to their hydrophilic nature, these adhesives seem to have greater affinity for wet rather than dry dentin⁵. The moisture pulls the monomer into and around the tubules, along with the adhesive ingredients. When cured, the dentin and the adhesive form a structural complex called the hybrid layer⁶. Formation of this hybrid layer of dentin and resin, which was first described by Nakabayashi et al.⁷ in 1982, is thought to be the primary bonding mechanism of most fourth generation adhesive systems³.

The use of vibration to alter thixotropic materials has long been recognized by the industry. However, its active use in dentistry has recently been documented⁸. Vibration was first utilized in dental scalers to aid in the removal of plaque and calculus from teeth. Furthermore, this phenomenon may be used to alter the viscosity of zinc phosphate cement⁹ or composite luting materials^{10,11} during the seating of cast metal and composite inlays. The technique requires the light placement of an ultrasonic scaler against the restoration for a few seconds. The vibration from the tip pass through the restoration into the underlying material. This changes the viscosity of the cement, and this in turn allows the restoration to slip into place easily⁸. Using vibration in this manner may have an added advantage: the improved flow characteristics of the material help reduce film thickness, thereby minimizing potential leakages that may occur around the restoration¹².

Regrettably, studies regarding the clinical applications of such vibration on composite restorations have been few. The objective of this study was to compare the bond strength and resin penetration into dentinal tubules achieved with those gained using the conventional technique and vibration technique.

II. Materials and Methods

1. Shear Bond Strength

Eighty-eight noncarious extracted human permanent molar teeth were stored in 0.1% thymol solution at room temperature after extraction. The teeth were cleaned by removing the remaining soft tissue and then stored in physiologic saline solution until use.

Each tooth was sectioned to remove the coronal enamel by using a slow-speed saw (Isomet, Buehler Ltd., Evanston, USA). Each specimen was embedded in 1-inch inner diameter PVC pipe with a cold-cure acrylic resin as mold. The occlusal surfaces were placed so that the tooth and the embedding medium were at the same level to form one flat surface. The samples were subsequently polished with wet 220-, and 500-grit silicon carbide abrasive papers. The samples were randomly assigned to 4 groups (n=22) (Table 1). Each adhesive system was applied according to its manufacturer's instructions.

Group I-Single Bond

Dentin was etched for 15 seconds with 35% phosphoric acid (Scotchbond Etching Gel, 3M-ESPE, St. Paul, USA) and rinsed with water for 15 seconds. After adjusting the surface moisture condition, two consecutive coats of Single Bond were applied with a saturated brush, gently air-dried with oil-free compressed air for 5 seconds to evaporate the solvent, and then light-cured for 10 seconds.

Group II-Single Bond with vibration

Dentin was etched, rinsed and dried in the same way as Group I. After application of Single Bond twice consecutively, the adhesive was gently air-dried for 5 seconds, and vibration was applied to the

Table 1. Materials used in this study

System	Composition	Manufacturer
Single Bond	Etchant: 35% phosphoric acid Adhesive: Bis-GMA, HEMA, dimethacrylates, polyalkenoic copolymer, ethanol, water	3M Dental Products, St. Paul, MN, USA
One-Step	Etchant: 32% phosphoric acid Adhesive: Bis-GMA, BPDM, acetone	Bisco Inc., Schaumburg, IL, USA

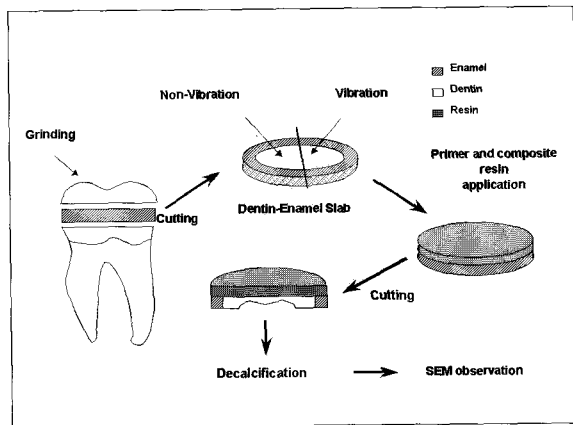


Fig. 1. Schematic representation of the experimental steps for SEM sample preparation.

enamel portion of the specimen using a commercially available ultrasonic scaler (Suprasson® P5 Booster, SATELEC, Merignat, France) with a sickle probe for 10 seconds. The adhesive was light-cured for 10 seconds.

Group III-One-Step

Dentin was etched for 15 seconds using 32% phosphoric acid gel. After rinsing and adjusting the surface moisture condition, two consecutive coats of One-Step adhesive were applied. The adhesive was gently air-dried for 5 seconds using a compressed air syringe and light-cured for 10 seconds.

Group IV-One-Step with vibration

Dentin was etched, rinsed and dried in the same way as Group III. After two consecutive applications of One-Step, the adhesive was gently air-dried for 5 seconds. Vibration was then applied with an ultrasonic scaler for 10 seconds the same as Group II. The adhesive was light-cured for 10 seconds.

Resin composite was condensed on to the prepared surface in two increments using a mold kit (Ultradent Products Inc., South Jordan, USA). Each increment was light cured for 40 seconds using a visible-light curing unit (Curing Light 2500, 3M-ESPE, St. Paul, USA).

After 24 hours in tap water at room temperature, the specimens were thermocycled for 500 cycles between 5°C and 55°C. The dwell time in each bath was 30 seconds and the transfer time was 10 seconds. Shear bond strengths were measured with a universal testing machine (Instron 4465, Canton, USA). A

knife-edged shearing rod with a crosshead speed of 1.0mm/min was used to load the specimens at the interface between composite and dental surface until fracture occurred. ANOVA and Student-Newman-Keuls test were used to evaluate the statistical significance of the results.

2. Examination of Debonded Specimens

The debonded dentin surfaces were coded and examined in a random sequence using a stereomicroscope at 40× magnification. The mode of bond failure was recorded as either "adhesive", meaning none or very little (<30%) composite still remaining on the dentin surface, or "mixed", meaning composite remaining on over 30% of the dentin surface, or "cohesive", meaning fracture occurring in the composite resin itself.

3. SEM examination

Infiltration patterns of the adhesive materials were investigated. Dentin discs, 3-4mm in thickness, were prepared from extracted teeth (Fig. 1). To avoid morphologic and structural variations of the dentin due to its depth, one-half of each specimen was vibrated in each group of each adhesive system, with the second half being kept without vibration. Subsequently, resin composite was placed and polymerized. Sectioned resin-tooth specimens immersed into 6 mol HCl for 24 hours to totally remove the calcified component, washed with distilled water for 5 minutes, then immersed in 5% NaOCl solutions for 20 minutes to remove the organic components. After dehydration procedure, the specimens were mounted to aluminum stubs with silver paint and sputter coated with gold-palladium, then examined with a scanning electron microscope (JSM-840A, JEOL Ltd., Tokyo, Japan).

III. Results

1. Shear bond strength

The mean and standard deviation values of shear bond strength were 18.10±5.83 MPa for Group I, 22.88±5.01 MPa for Group II, 16.32±4.58 MPa for Group III, and 20.16±3.52 MPa for Group IV (Table

Table 2. Results of shear bond strength tests(n=22)

	Mean(MPa)	SD	Difference
Group I	18.10	5.83	a
Group II	22.88	5.01	b
Group III	16.32	4.58	a
Group IV	20.16	3.52	b

*Different letters indicate significant difference(p<0.05).

Table 3. Mode of fractures

	Cohesive	Adhesive	Mixed
Group I	1	6	15
Group II	7	2	13
Group III	-	8	14
Group IV	3	4	15

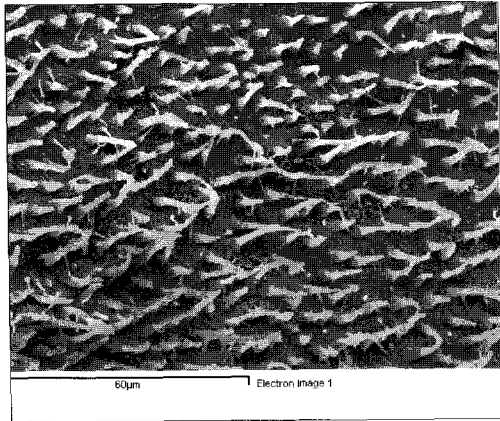


Fig. 2. SEM of demineralized non-vibration surface for Single Bond(×1000).

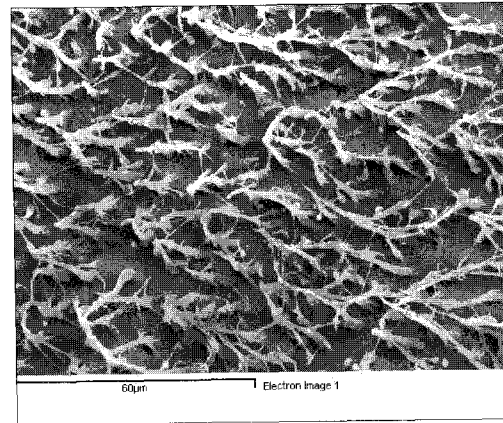


Fig. 3. SEM of demineralized vibration surface for Single Bond(×1000).

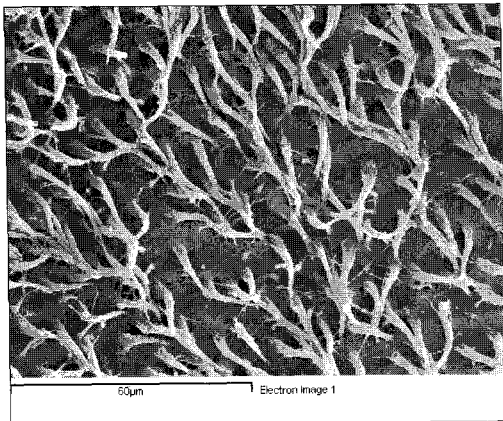


Fig. 4. SEM of demineralized non-vibration surface for One-Step(×1000).

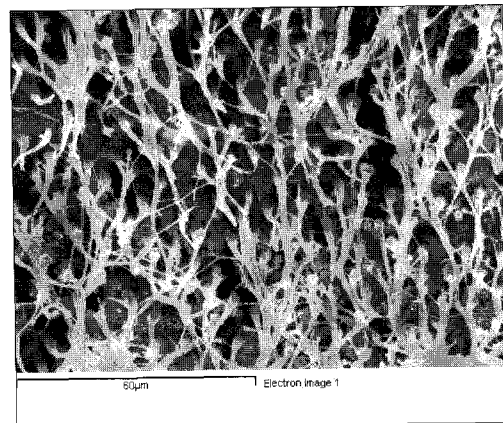


Fig. 5. SEM of demineralized vibration surface for One-Step(×1000).

2). The one-way ANOVA test revealed statistically significant differences between groups(p<0.0001). Student-Newman-Keuls test indicated that statistically significant differences existed between Groups I and II, as well as between Groups III and IV(Table 2).

Statistically significant increases in bond strength were observed in all one-bottle dentin bonding systems when vibration was used. The highest shear

bond strengths were attained at Single Bond with vibration. The shear bond strength of One-Step with or without vibration was slightly lesser than that of Single Bond, but the difference was not statistically significant.

2. Examination of debonded specimens

The fracture patterns of debonded specimens were

shown in Table 3. When applied with vibration or non-vibration, failure was mainly mixed type. More cohesive failures were appeared with specimens of vibration groups than those of non-vibration groups.

3. SEM examination

In the SEM examination, resin tags and lateral branches formed a fine resin network that completely infiltrated the intertubular dentin area. Each tubule tag featured an interconnecting network of lateral canals that contained polymerized adhesive. It was shown that there was variation in resin tag formation among adhesive materials and application methods. For vibration groups, resin tags were found to be greater in length and number of lateral branches. And more homogeneous appearance were showed for Single Bond than for One-Step(Fig. 2-5).

IV. Discussion

The single-bottle system incorporates the primers and the bonding resin into a single container. It represents an appreciable advance over previous systems and the fewer number of components and steps simplifies technique. This is especially valuable as the system allows the simultaneous and similar treatment of dentin and enamel¹³⁾.

One-Step is an acetone-based, and one-component bonding agent that relies on dentin and enamel etching with 32% phosphoric acid. It contains biphenyl dimethacrylate plus Bis-GMA and HEMA^{1,2,14,15)}. The mean bond strengths obtained at baseline in the present study were comparable to those obtained in other studies with similar surface conditions without any air drying^{15,16)}. When applied in vivo, One-Step provides a well-defined resin-dentin interdiffusion zone¹⁷⁾. Some areas of debonding may occur at the interface and alternate with areas without debonding between the hybrid layer and the adhesive¹⁷⁾.

For Single Bond, the presence of water in its composition might be beneficial¹⁴⁾. It has been suggested that the water present in the composition of some adhesives would be able to reopen the collapsed network of collagen fibers on dry spots left on the surface and prevent the formation of areas of "ghost" hybrid layer¹⁸⁾. Because water has a plasticizing effect

on collagen fibers, the shrunken collagen coagulate would reexpand and allow monomers to infiltrate the fibers¹⁹⁾.

There have been several reports dealing with the shear bond strengths of single-bottle dentin bonding adhesives used in the present study. Swift and Bayne²⁰⁾ obtained, with One-Step and Single Bond applied to moist dentin, each shear bond strength of 20.5 MPa and 19.2 MPa. Finger and Fritz²¹⁾ reported shear bond strength of 18.2 MPa for One-Step. Tjan et al.²²⁾ reported a value of 16.4 MPa for One-Step, while Abdalla and Davidson¹⁵⁾ reported 13.5 MPa in the same agent. For Single Bond, Prati et al.²³⁾ reported shear bond strength of 16.6 MPa. Swift and Bayne²⁰⁾ suggested that differences in bond strengths among the studies were most likely due to the technique-sensitive nature of the materials and manipulation differences among investigators. On the other hand, several investigators have offered the fact that dentin is a dynamic substrate with physiological changes as reason for variations in bond strengths. As such, bond strength to dentin is subjected to many variables²²⁾ such as pulpal fluid under pressure in vital dentin²³⁾, dentin depth²⁴⁾, tooth type²⁵⁾ and Ca²⁺ concentration of dentin²⁶⁾. McCabe and Rusby²⁴⁾ and Suzuki and Finger²⁷⁾ reported a tendency for bond strength to decrease with increasing depth of dentin.

Several reasons may account for this difference. Differences may exist in testing methodology. That is, these differences in results between studies may be due to variations in the interpretation which constitutes a wet dentin surface. The amount of moisture needed for wet bonding is therefore a critical factor in determining the bond strength of single-bottle adhesives. The fact remains that the application of dentin adhesive is a very technique-sensitive procedure. Inconsistency in the distance between dentin surface and pulp among the thinner dentin samples may have been responsible for the variations. As regards to this, Perdigao et al.²⁸⁾ have observed that relatively thin dentin substrates tended to exhibit cohesive failures and lower bond strengths. Previous researchers have described the dentin surface as wet, physiologically hydrated, or visibly moist. However, the degree of surface wetness is difficult to judge clinically, and the manufacturers are not specific

about just exactly how wet a moist dentin surface should be prior to application of the dentin bonding system. Excessive pooled water should probably not be allowed to remain on the dentin. Tay et al.²⁹⁾ described the overwet phenomenon, in which dentin is left too wet prior to bonding, with excess water adversely affecting measured bond strengths.

In addition, cross-sectional surface area can affect the bond strength between resin and dentin. Sano et al.³⁰⁾ and Phrukkanon et al.³¹⁾ reported that bond strengths were inversely related to the bonded surface area and that with small bonded surface areas, the bonds were predominantly of the adhesive type.

Composite resin thickness also affects shear bond strengths of dentin bonding agents. Price et al.³²⁾ reported that the bond strengths of 5mm-thick specimens were significantly lower than those of 2mm-thick samples. Rueggeberg et al.³³⁾ stated that there is a marked decrease in hardness and degree of conversion when the composite resin is more than 3mm thick.

Several reports have been conducted to study the relationship between the degree of resin infiltration and bond strength. Swift et al.³⁴⁾ suggested that the penetration of adhesive into etched dentin surface is critical for achieving high dentin bond strengths. In addition, Triolo and Swift³⁵⁾ indicated that lower monomer resin diffusion is directly associated with weak dentin bond strength. Amory and Yvon³⁶⁾, and Fanning et al.³⁷⁾ reported that adequate bonding of adhesive materials to dentin depends not only on adequate penetration of the adhesive into dentin, but also on the mechanical properties of the resin itself. That is, the nature of the resin can influence its shear bond strength³⁴⁾. According to the study by Swift et al.³⁾, bond strength of an etched-dentin adhesive may rely on its ability to completely replace dissolved hydroxyapatite with polymerized resin. However, the mechanism of adhesion is almost certainly multifactorial in nature. Therefore, mechanical attachment through penetration of bonding resins into dentinal tubules would not appear to be a prerequisite for high bond strengths^{24,38,39)}.

One of the keys in the field of dentin adhesion was the observation of the hybrid layer, resulting from resin penetration into the acid-demineralized dentin³⁹⁾. Due to low elastic modulus, this resin-

dentin interdiffusion zone may act as a stress-absorber between dentin and resin composite⁴⁰⁾. Some authors have suggested that the dimensions of the hybrid layer may be taken as an indicator of the strain-absorbing capacity of the corresponding interface⁴¹⁾. This elastic buffer could be of the utmost importance in absorbing the stresses originating from the polymerization shrinkage of composite resin. However, the thickness of the hybrid layer and its influence on bonding durability is still uncertain.

In the present study, Groups II and IV, to which vibration had been applied, showed significantly higher shear bond strengths, exhibiting greater number of resin tags and better lateral branch development under SEM examination(Fig. 2-5). This is considered to be the result of ultrasonic vibration used to diminish the viscosity of resin, which in turn aided resin penetration into dentinal tubules. Tavas and Watts⁴²⁾ reported that unfilled resin presented the rheological behavior, and therefore the consistency of composites was decreased by repeatedly loaded shear stress. This observation supports the findings of Chappell et al.⁴³⁾, and Ferrari and Davidson⁴⁴⁾, who observed resin tags in the lateral branches of the dentinal tubules. They suggested that the network of interconnected adhesive tags may be fundamental to the development of a stronger resin-dentin bond. In contrast, Jacobsen and S Iderholm⁴⁵⁾ observed the opposite results in their study to evaluate the shear bond strength of acetone- or water-based primers applied with or without agitation to either wet or dry dentin, and reported that the acetone-based primer gave the highest bond strength to wet dentin when agitation was not utilized. It was their explanation that such a decrease in bond strength may be related to a faster acetone evaporation caused by the agitation. As the acetone evaporates, the HEMA may form a more or less jelly-like structure, which may not diffuse as easily as a dilute HEMA solution⁴⁵⁾. However, in the present study, ultrasonic vibration, as opposed to manual agitation, was applied for short periods to optimize only the viscosity-decreasing effects of vibration.

To achieve good dental bonding, the adhesive solution should exhibit an ability to infiltrate the demineralized collagen network. That can be achieved not only by improving the wettability of the adhe-

sive, but also by retaining the collagen mesh expanded during adhesive infiltration. Ultrasonic vibration may be considered a useful method for optimizing the quality of interpenetrating network formation and maximizing the density of infiltrating adhesive. Examination of the form and thickness of the hybrid layer of vibrated and non-vibrated groups, not performed in this study, would be an area for further research efforts.

V. Conclusions

1. The shear bond strengths of vibration groups (Group 2, Group 4) were significantly greater than those of the non-vibration groups (Group 1, Group 3) ($p < 0.05$).
2. The shear bond strengths of Single Bond and One-Step were not significantly different ($p > 0.05$).
3. The vibration groups showed greater number of resin tags in tubules and lateral branches under SEM examination.

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Reprint request to:

Chong-Chul Kim, D.D.S., Ph.D.
 Department of Pediatric Dentistry, College of Dentistry, Seoul National University
 28-1, Yeongun-Dong, Chongno-Gu, Seoul, 110-749, Korea
 E-mail : kimcc@plaza.snu.ac.kr

Abstract

A STUDY OF ADDITIONAL VIBRATION EFFECT ON DENTIN BOND STRENGTH

Jin Lee, D.D.S. Jung-Wook Kim, D.D.S., Ph.D., Sang-Hoon Lee, D.D.S., Ph.D.,
Chong-Chul Kim, D.D.S., Ph.D.

*Department of Pediatric Dentistry and Dental Research Institute
College of Dentistry, Seoul National University*

The objective of the study was to apply the vibration technique to reduce the viscosity of bonding adhesives and thereby compare the bond strength and resin penetration into dentinal tubules achieved with those gained using the conventional technique.

Eighty-eight noncarious extracted human permanent molar teeth were sectioned to remove the coronal enamel and were embedded in 1-inch PVC pipe with acrylic resin. The occlusal surfaces were placed so that the tooth and the embedding medium were at the same level to form one flat surface, and the samples were subsequently polished with silicon carbide abrasive papers. The samples were randomly assigned to 4 groups(n=22). On Group 1 and 2, Single Bond(3M-ESPE, St. Paul, USA) was used, and on Group 3 and 4, One-Step(Bisco Inc., Schaumburg, USA) was used, and each was applied according to its manufacturer's instructions. For Group 2 and Group 4, vibration was applied with ultrasonic scaler for 10 seconds, and the adhesive was light-cured for 10 seconds. Resin composite was condensed on to the prepared surface in two increments using a mold kit(Ultradent Products Inc., USA) and each was light-cured for 40 seconds. After 24 hours in tap water at room temperature the specimens were thermocycled, and shear bond strengths were measured with a universal testing machine(Instron 4465, Canton, USA). To investigate infiltration patterns of the adhesive materials, the surface of specimen was examined with scanning electron microscope. The results were as follows.

1. The shear bond strengths of vibration groups(Group 2, Group 4) were significantly greater than those of the non-vibration groups(Group 1, Group 3)($p < 0.05$).
2. The shear bond strengths of Single Bond and One-Step were not significantly different($p > 0.05$).
3. The vibration groups showed greater number of resin tags in tubules and lateral branches under SEM.

Key words : Ultrasonic vibration, Bonding Adhesives, Viscosity, Shear bond strength, Resin infiltration