BOND STRENGTH AND MICROLEAKAGE IN RESIN BONDING TO TOOTH STRUCTURE

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ABSTRACT

치질접착에서 접착강도와 변연누출

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치질에 대한 접착수복은 건전한 치질을 보존하면서 유지력을 증가시킬 뿐 아니라 치질과 수복물 계면에서의 미세누출을 방지할 수 있다는 측면에서 임상에서 그 사용빈도가 높아지고 있다. 그러나 복합레진충전후 중합과정에서의 수축현상이 여전히 해결되지 않는 문제로 남아 있으며 이로 인한 변연누출은 술후 지각과민, 변연변색이나 이차우식과 아울러 치수병변의 유발인자로 작용할 수 있다.

치질과 레진수복물간의 접착강도를 강화시킴으로써 변연부위의 누출현상을 극복할 수 있다는 인식 하에 많은 종류의 상아질 접착제에 대한 접착강도 검사와 미세누출 검사가 행해졌으나 접착강도와 변연누출간의 관계에 대한 직접적인 연구는 미흡한 상태이다. 본 연구에서는 우치를 이용한 동일시편에서 접착강도와 미세누출의 정도를 측정 후 상호관계를 알아보고자 하였다.

소의 하악 전치를 이용하여 법랑질 또는 상아질 시편을 각 군마다 30개씩 준비하였다. 32% 인산으로 15초 처리 및 20초 수세 후 치질처리 방법에 따라 법랑질/완전 건조시킨 군, 법랑질/습윤상태의 군, 상아질/완전 건조시킨 군, 상아질/습윤상태의 군으로 나누었다. 각 군의 시편을 One-Step™ adhesive를 적용 후 Charisma 복합레진으로 충전하였다. 그 후 2% methylene blue에 1주일 간 침잠시킨 후 24시간 흐르는 물에 수세한 뒤 인장접착 강도를 측정하였다. 인장접착 강도 측정 후 컴퓨터 프로그램 상에서 색소의 침투 면적을 계산하였으며 그 결과는 다음과 같이 나타났다.

1. 법랑질 및 상아질 군 공히, 접착강도와 미세누출을 비교시 유의한 음의 상관관계를 나타내어 접착강도가 높을수록 미세변연누출은 적게 나타내는 경향을 나타내었다 ($r=-0.50$, $p<0.05$).
2. 법랑질 군 및 상아질 군 공히, 습윤상태의 시편이 완전히 건조된 시편에 비해 높은 접착강도를 나타내었다 ($p<0.05$).
3. 상아질에서는 습윤상태의 군이 건조시킨 군 사이에 유의하게 적은 미세변연 누출을 나타낸 반면($p<0.05$), 법랑질에서는 습윤상태의 군과 건조시킨 군 사이에 미세변연 누출에 있어 유의한 차이가 나타나지 않았다 ($p>0.05$).

I. INTRODUCTION

The public has an increasing concern for esthetic dentistry, and as a result, the dental profession has increased the use of composite restorations and continual improvement has been made in their properties.

Bonded restorations have a number of advantages over traditional, nonadhesive methods. Traditionally, retention and stabilization of restorations often required the removal of sound tooth structure. This is not necessary, in many cases, when bonding technique is used. Bonded restorations better transmit and distribute functional stresses to the tooth across the bonding interface and have potential to reinforce weakened tooth structure. Bonding also reduces microleakage at the restoration-tooth interface.

Microleakage, however, still occupies a major focus of researchers trying to improve the longevity of composite restorations. Microleakage is defined as the clinically undetectable passage of bacteria, fluids, molecules, or ions between a cavity wall and the restorative material applied to it[1]. Microleakage is the results of breakdown of the tooth-restoration interface, which may cause discoloration, recurrent caries, percolation, hypersensitivity of restored teeth, and the development of pulpal pathology[2,3]. Microleakage has been attributed to many factors including differences in the coefficients of thermal expansion between the tooth structure and the restorative material, polymerization shrinkage of the resin materials, load stresses, and inadequate adhesion to tooth[4,5]. Microleakage at the enamel margins has been eliminated with phosphoric acid etching, a concept first proposed by Buonocore[6] and microleakage at the dentin (cementum) margins has been reduced significantly by the use of dentin bonding agents. Manufacturers have developed bonding agents in an effort to control leakage at the tooth-restoration interface by optimizing bonding through improved resin technology. However, clinically, microleakage at these interfaces still occurs.

An ideal dental adhesive would provide high bond strength and would eliminate microleakage. Marginal microleakage and bond strength tests have been frequently utilized to study the performance of dentin bonding agents. The micromechanical bonding mechanism was first described by Nakabayashi et al.[7] in 1982, as the formation of resin-reinforced zone. Hybridization is described that certain monomers can infiltrate the demineralized dentin and combine with the collagen and hydroxyapatite to form a new material that is part tooth and part resin. Maintaining a moist surface may be essential for optimal development of the hybrid layer when hydrophilic bonding systems are used. Desiccation of the conditioned dentin may cause collapse of the unsupported collagen network, inhibiting adequate wetting and penetration by the primer. When true hybridization occurs, bond strength increases dramatically. The hybrid layer forms an acid-resistant envelope that seals the dentin, preventing hypersensitivity and secondary caries. Therefore, strong unification between tooth structure and bonding resin is generally believed indispensible to not only enhancing longevity of the composite restoration but minimizing microleakage at tooth-restoration interface. However, the relationship between bond strength and microleakage performance is not clearly understood. Several investigators have attempted to correlate bond strength values with marginal gap size. Some authors reported a negative correlation between the bond strengths and the marginal contraction gaps[8,9]. While, another study[10] found no clear relationship between microleakage evaluation and shear bond strength with different bonding system.

Therefore, the correlation between dentin bonding strength and microleakage needs to be investigated in the same specimen.

The purpose of the present study was to evaluate the relationship between tensile bond strength and microleakage in the same restorations to understand the behavior of resin bonding to tooth structure.

Ⅱ. MATERIALS AND METHODS

1. Preparation of the specimens

One hundred and twenty freshly extracted bovine lower incisors were stored frozen until use. Crown segments were prepared by removal of the roots at the cementoenamel junction with a low-speed diamond disc. The pulp tissue was removed with a broach. For each tooth, the labial surface was wet ground with a model trimmer to expose a flat area of enamel or dentin 5mm in diameter. The exposed surfaces were hand-polished with wet 600- and 800-grit silicon carbide abrasive paper under sufficient water spray. The teeth were then divided into four equal groups and treated in the following manner:

Enamel/Dry Group : The enamel surfaces were dried with oil-free, dried compressed air for 10 seconds. 32% phosphoric acid gel was applied to the enamel surface for 15 seconds. The gel etchant was then rinsed off with an air-water spray for 20 seconds. The enamel surface was dried for 20 seconds at the maximal pressure of the dried air syringe (Hotman®, Japan) while the tip of the syringe was approximately 2cm from the enamel surface. A 5 mm diameter-hole was punched in the center of a strip of adhesive tape, which was gently burnished onto the ground surface to demarcate the bonding area.

Two consecutive coats of dentin-bonding agent (One-Step™ Adhesive, Bisco Inc., IL, U.S.A.) were applied using a saturated brush. The adhesive was thoroughly air-dried for 10 seconds using a compressed air syringe held at a distance of 2cm from the surface. The adhesive was light-cured for 10 seconds. Using the same brush tip without resaturating, an additional coat of the adhesive was applied to the surface, air-dried immediately, without light-curing. A silicone mold, 1.2mm in thickness and 6.5mm in diameter, was applied to the treated enamel surface. The mold was filled with a single increment of composite resin (Charisma®, Kulzer, Germany). Slight pressure was applied on top of the siliconemold with a slide glass and the composite resin was light cured for 40 seconds.

Enamel/Wet Group : The teeth in this group were prepared similarly to those in Enamel/Dry Group, except that, following rinsing of the etchant, the enamel was wiped with a damp facial tissue to remove the excess water. The surface of the enamel was shiny with moisture. Adhesives were applied and composite resin cylinders were bonded to the teeth as the same way in Enamel/Dry Group.

Dentin/Dry Group : Dentin surfaces of the teeth in this group were prepared with the same methods as in Enamel/Dry Group.

Dentin/Wet Group : Dentin surfaces of the teeth in this group were prepared with the same methods as in Enamel/Wet Group. Pulp chamber were sealed with utility wax and two coats of nail polish. Each specimen was then sealed with fingernail polish up to 1mm from the restoration margins. The specimens were immersed in 2% methylene blue solution for 7 days, washed in tap water for 24 hours, dried, and acrylic rods were attached to the specimen perpendicularly with cyanoacrylate.

Table 1. Chemical ingredients of the dentin bonding agent used in this study

Product	Chemical Ingredients	Manufacturer
One-Step™	Etchant : 32% phosphoric acid Adhesive : BPDM, HEMA, Bis-GMA in acetone	Bisco Inc, Itasca, Il, U.S.A.

2. Tensile bond strength measurement

The specimens were placed on the tensile jig which was attached to Instron universal testing machine (4202 Instron, Instron Co., U.S.A), and tensile bond strength were measured at a crosshead speed of 2 mm/min with load cell 50kgf. The bond strengths were expressed in MPa.

3. Microleakage test

The Degree of microleakage was determined with a dye penetration method. Each fractured surface was photographed. The photographs were later scanned and the dye penetration area was measured with the image analyzer (NIH image 1.60, Macintosh program, U.S.A.). Leakage value was calculated as the ratio of dye penetration area to the bonding area and expressed in percentage.

4. Statistical analysis

The data were analyzed by -test and statistical significance was defined as $p<0.05$. Relationships between bond strengths and microleakage were tested by regression analysis.

Table 2. Tensile bond strength value and degree of microleakage in each experimental group

(Mean ± S.D.)

Group	Tensile Bond Strength (MPa)	Microleakage (%)
Enamel/Dry	12.59±5.07 ┐*	18.80±24.48
Enamel/Wet	15.00±6.54 ┘	10.98±18.38
Dentin/Dry	3.54±2.11 ┐*	46.39±31.31 ┐*
Dentin/Wet	8 31±3.21 ┘	24 83±26.33 ┘

* Significant difference of the each group are noted at a $p<0.05$ level by student's t-test.

Ⅲ. RESULTS

1. Tensile bond strength

The mean bond strength of each group is shown in Table 2. The mean tensile bond strengths were 15.00 ±6.54MPa for the Enamel/Wet group, 12.59±5.07 MPa for the Enamel/Dry group, 8.31±3.21MPa for the Dentin/Wet group and 3.54±2.11MPa for the Dentin/Dry group. The Enamel/Wet group showed

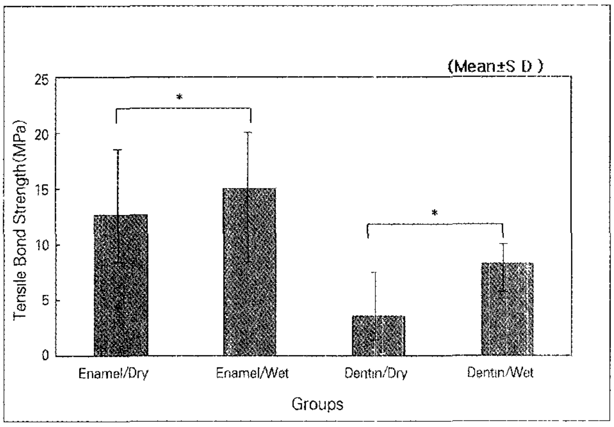

Fig. 1. Tensile bond strength of each experimental group
* Significantly different from each other group ($p<0.05$).

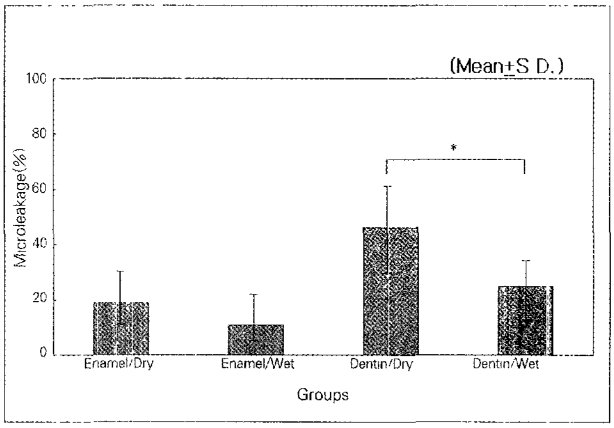

Fig. 2. Microleakage of each experimental group.
*Significantly different from each other($p<0.05$)

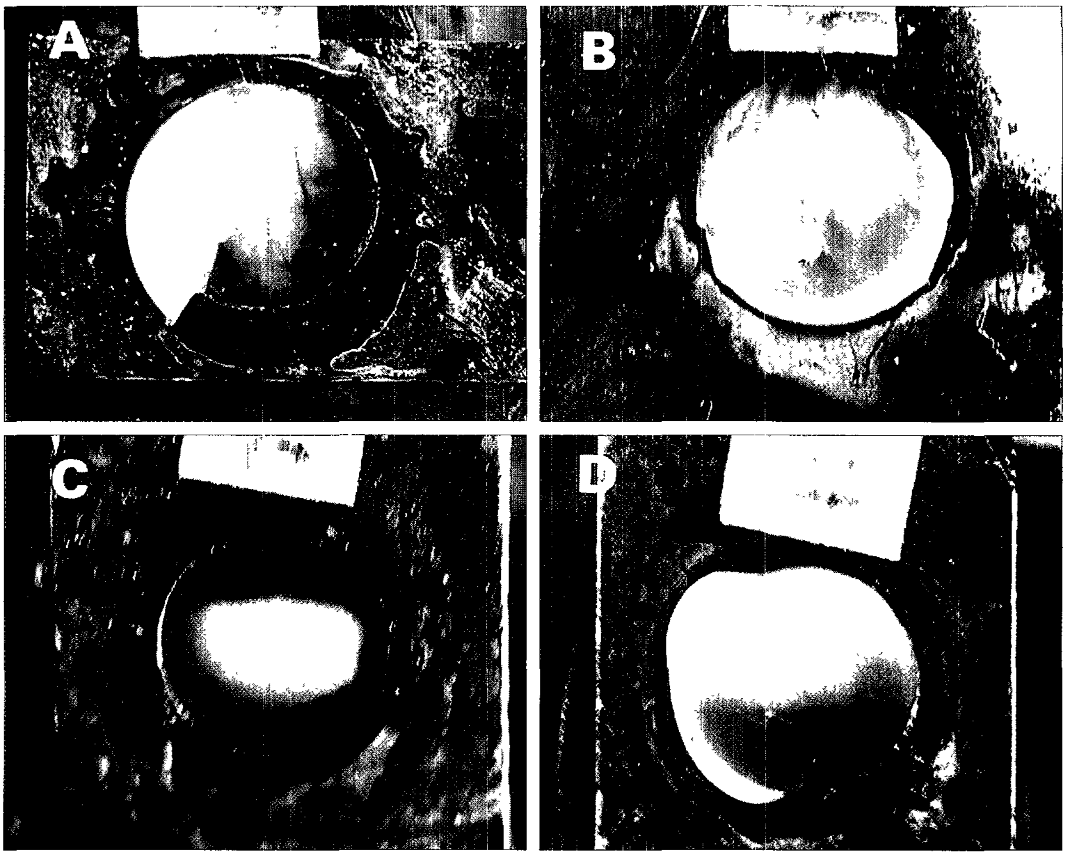

Fig. 3. Microleakage test. A. Enamel/Dry group: Representative specimen showed tensile bond strength of 15.45 MPa and microleakage value of 10.35% B. Enamel/Wet group: Representative specimen showed tensile bond strength of 18.11 MPa and microleakage value of 1.6% C. Dentin/Dry group: Representative specimen showed tensile bond strength of 3.49 MPa and microleakage value of 66.34%. D. Dentin/Wet group: Representative specimen showed tensile bond strength of 10.2 MPa and microleakage value of 15.52%

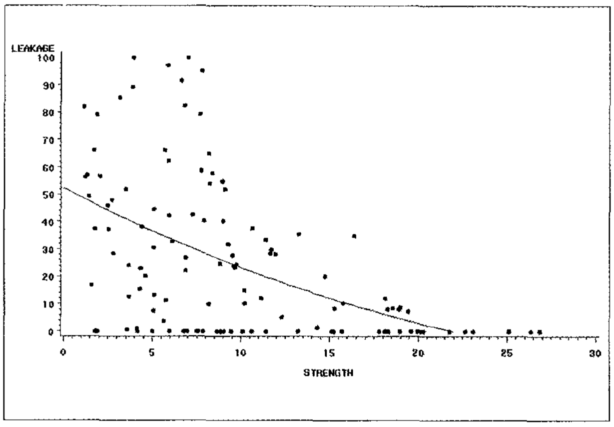

Fig. 4. Relationship between tensile bond strength value and the degree of microleakage.

significantly greater strength than that of Enamel/Dry group ($p<0.05$) and the mean bond strength of Dentin/Wet group was significantly greater than that of Dentin/Dry group ($p<0.05$)(Fig. 1).

2. Microleakage

The results of the microleakage testing are summarized in Table 2. The mean microleakage value were 18.80±24.48% for the Enamel/Dry group, 10.98±18.38% for the Enamel/Wet group, 46.39±31.31% for the Dentin/Dry group, and 24.83±26.33% for the Dentin/Wet group. The mean microleakage of Dentin/Dry group was significantly higher than Dentin/Wet group ($p<0.05$). However, there was no statistically significant difference in microleakage between Enamel/Wet and Enamel/Dry groups ($p>0.05$)(Fig. 2).

3. Relationship between bond strength value and the degree of microleakage

When the bond strength values and the corresponding microleakage values are compared, significant negative correlation was observed in entire data set ($r=-0.50$)(Fig. 4).

Ⅲ. DISCUSSION

Bond strength and marginal microleakage are the two characteristics of adhesives that are most frequently evaluated in vitro studies.

From a clinical point, even though loss of the composite restoration due to bonding failure is rare, microleakage is the main reason for marginal discoloration, secondary caries, hypersensitivity and pulpal pathosis. Hence, marginal integrity is of primary importance in maintaining the long-term function of composite restorations. In generally, the improvement of bonding at the resin-tooth interface would expected to have a positive influence in microleakage reduction. However, the direct relationship between these two is not clear.

Prati *et al.*[10] demonstrated that the same dentin bonding system used with different composites gave similar shear bond strengths and different microleakage values. Therefore, it has been suggested that the type of composite is a major factor responsible for the early gaps around restorations[11]; bond strength is not an adequate indication of the seal effectiveness of dentin bonding system.

However, in the present study there was a significant negative correlation between bond strength and microleakage. Hence, high bond strengths are associated with low microleakage and vice versa. These experiments were performed in a clinically relevant manner-that is, the microleakage tests were done, following measurements of tensile bond strength in the same samples[12].

Other researchers have obtained results, although not in the same sample, remarkably similar to those reported in this study. For example, Komatsu and Finger[9] obtained the linear relationship between the logarithm of the contraction gaps and the shear bond strength ($r=-0.88$). Tsai *et al.*[13] demonstrated an inverse relation between the bond strengths and microleakage of the original Scotchbond® (3M, U.S.A.), Scotchbond 2® (3M, U.S.A.), and Gluma® (Columbus, U.S.A.). Fortin *et al.*[14] showed some observable trends between shear bond strength and microleakage even though there was no statistically significant correlation. Adhesive systems that include a low-viscosity intermediate resin produced high bond strengths and low microleakage. Similarly, two materials with bond strengths in the intermediate range had significantly increased microleakage, and one material with a bond strength in the low end of the spectrum exhibited microleakage that was statistically greater.

Adhesive systems containing hydrophilic primers, dissolved in acetone or ethanol were found to produce higher bond strengths when acid-conditioned dentin was left visibly moist prior to bonding, a protocol commonly referred to as "wet bonding"[16,17]. In addition, Kanca[18] has reported that the bond strengths to etched and wet enamel using acetone based dentin-enamel bonding system were equal to or higher than bond strengths to etched and dry enamel.

Acetone has been found to be a better primer solvent than water for HEMA by virtue of its ability to displace water from the collagen network[19]. The addition of acetone to water raises the vapor pressure of the water and causes some of it to become volatile. In addition, acetone reduces the surface tension of the water. This has the effect of causing the acetone to appear to "chase" the water. This will continue until an equilibrium is established, carrying it with the resin primer. The remaining acetone is then dried, leaving a layer of primer on the dentinal substrate. This would account for the reduced microleakage in the wet bonded specimens.

A new dental bonding system One-Step™ Adhesive (Bisco Inc., Itasca, IL, U.S.A. was recently developed to provide clinical versatility with ease of use. It is a light-curable single-component primer/adhesive system, combining the two separate steps of primer and bonding resin applications into one. One-Step™ contains BPDM (biphenyl dimethacrylate), BIS-GMA, HEMA (hydroxyethylmethacrylate), and a photoinitiator in acetone. An excellent seal is achieved with One-Step™ adhesive on dentin substrate through the complete infiltration of adhesive resin into demineralized intertubular and peritubular dentin, and efficient in-situ polymerization[20].

Because acetone based adhesive system - One Step™ was used in this study, maintaining a moist surface is essential for the development of well-organized hybrid layer. In the Dentin/Wet group of this study, hydrophilic monomer may have infiltrated into the demineralized collagen matrix. Therefore, well-organized hybrid layer might be developed and increased the bond strength, forming an effective seal on the interface. The present study showed that the bond strengths to wet enamel surface was higher than to dry one. Probably, higher bond strengths to wet enamel are effected through better wetting of the surface - that is, acetone can displace moisture from the etched enamel and carry the resin monomers into close adaptation with the surface, resulting in high bond strengths. In addition, the bond strengths to wet dentin were higher than to dry dentin. In dentin, the presence of moisture appeared to play a crucial role in not only better wetting of the surface but also preventing the degeneration of surface collagen. Because water was crucial during the initial phase of inter-tubular infiltration in maintaining the structural integrity of the demineralized collagen fibrils, as well as keeping the interfibrillar spaces open so that resin could permeate these spaces through the water-displacing capability of the acetone[21,22].

Because composition and structure of enamel and dentin are substantially different, adhesion to the two tooth tissues will also be quite different. The inorganic content of mature enamel is 95% to 98% by weight (wt%); the primary component is hydroxyapatite. The remainder consists of water (4 wt%) and organic material (1 to 2 wt%)[23]. Adhesion to enamel is achieved through acid etching of this highly mineralized substrate, which substantially enlarges its surface area for bonding and tag-like resin extensions formed and micromechanically interlocked with enamel microporosities created by etching[24,25]. Unlike enamel, dentin contains a higher percentage of water (12 wt%) and organic material (18 wt%), mainly type I collagen, and hydroxyapatite (70 wt%). Structurally, numerous dentinal tubules radiate from the pulp throughout the entire thickness of dentin, making highly permeable tissue[23].

The difference in composition between the enamel and the dentin may have accounted for the fact that the dentin specimens showed more leakage than the enamel specimens. Besides, as dentin is more porous structure than enamel, the wet-bonding technique in dentin is more affected than in enamel.

The present study that showed wet bonding to dentin significantly reduced microleakage and the presence of moisture on enamel surface resulted in microleakage lower than that found in the dry enamel, although this difference was not statistically significant.

When bonding systems with primers and adhesives were applied separately, adhesive failure often occurred at the adhesive-hybrid layer interface[26]. This is most likely caused by inadequate wetting of the primed dentin surface with adhesive or by poor mixing of primers and adhesive. Gap or voids are formed between adhesive and hybrid layer (or primed dentin surface) and could be enlarged by the shrinkage stress exerted during the curing of the composite resin. Those gaps will act as stress concentrators and propagate preferentially along the adhesive/hybrid layer interface under stress, resulting in premature failure of the bonding of composite resin to tooth structure. However, for One-Step™ adhesive system, the primer and adhesive are combined in one-bottle and applied in a single step. A continuous gap-free link between the hybrid layer and adhesive layer should result, reducing the likelihood of adhesive failure.

One-Step™ is acetone based system. For that reason, this adhesive is rather sensitive to the air-drying of the etched dentin. In-vitro lab testing showed that even a brief 2 - 3 seconds of air-drying with an air-syringe would cause about 50% reduction in dentin bond strength[27].

Fluidity, modulus of elasticity and volumetric amount of filler can influence the photo-polymerization contraction of a composite and, consequently, the marginal sea[15,28]. The resin composite used in this study was a fine hybrid material, Charisma®. The use of this material may have had some effect on the microleakage values because of its relatively high elastic modulus and high shrinkage values compared with microfilled resin composite. These properties would tend to increase the microleakage potential of the restorations[10-1,29,30].

The bond strengths obtained in this study were somewhat lower than those reported in previous studies. One possible explanation for this is the difference of the testing methodology. Tensile bond strength was measured while some other reports[15,16,31] used shear bond strength. The second possible explanation is differences in the substrate. The bonding strength may be different from one material to the other. Another possible explanation may be related to the use of methylene blue solution in this study. Probably, as the specimens of this study were immersed in 2% methylene blue solution for 7 days, methylene blue may be penetrate into resin/tooth interface and cause hydrolysis. Therefore, the absolute values shown in a given study may not be significant.

In summary, significant negative correlation was found between the bond strengths and the microleakage values. Hence, higher bond strengths seem to be associated with lower microleakage, and vice versa. In addition, acetone-based bonding systems seem to work well in the presence of moisture. And further study is needed to investigate these relation in other bonding systems including water-based one.

Ⅳ. SUMMARY

Intuitively, higher bond strengths should result in less leakage. However, the relationship between bond strengths and microleakage value is complex and not clearly understood.

The purpose of this study was to evaluate the relationship between tensile bond strengths and microleakage values in the same restorations to understand the behavior of resin bonding to tooth structure.

One-hundred and twenty enamel or dentin specimens from freshly extracted bovine mandibular incisors were used. The specimen was treated with 32% phosphoric acid for 15 seconds and rinsed for 20 seconds, the teeth were divided into four groups by means of wet bonding technique or dry bonding. One-Step™ adhesive were applied to the specimen. The specimens were immersed in 2% methylene blue solution for 7 days, and tensile bond strength and microleakage were measured. The results were as follows:

1. Significant negative correlation was found between bond strengths and microleakage values. Hence, higher bond strengths seem to be associated with lower microleakage, and vice versa ($r = -0\ 50$, $p < 0.05$).
2. The Enamel/Wet group showed significantly higher bond strength than Enamel/Dry one, and Dentin/Wet group showed higher strength than

Dentin/Dry one (p<0.05).

3. Microleakage was significantly less in wet bonding than in dry one at dentin (p<0.05), however there was no significant difference between wet and dry bonding at enamel (p>0.05)

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