

The effect of adhesive thickness on microtensile bond strength to the cavity wall

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ABSTRACT

The purposes of this study were to examine the variability of adhesive thickness on the different site of the cavity wall when used total-etch system without filler and simplified self-etch system with filler and to evaluate the relationship between variable adhesive thickness and microtensile bond strength to the cavity wall.

A class I cavity in six human molars was prepared to expose all dentinal walls. Three teeth were bonded with a filled adhesive, Clearfil™ SE bond and the other three teeth were bonded with unfilled adhesives, Scotchbond™ Multi Purpose. Morphology and thickness of adhesive layer were examined using fluorescence microscope. Bonding agent thickness was measured at three points along the axial cavity wall, edge of cavity margin (**rim**), halfway down each cavity wall (**hlf**), internal angle of the cavity (**ang**). After reproducing the adhesive thickness at **rim**, **hlf** and **ang**, micro-tensile bond strength were evaluated.

For both bonding agents, adhesive thickness of **ang** was significantly thicker than that of **rim** and **hlf** ($P < 0.05$). As reproduced the adhesive thickness, microtensile bond strength was increased as adhesive thickness was increased in two bonding agents.

Adhesive thickness of internal angle of the cavity was significantly thicker than that of the cavity margin and the halfway cavity wall for both bonding agents. Microtensile bond strength of the thick adhesive layer at the internal angle of the cavity was higher than that of the thin adhesive layer at the cavity margin and the halfway cavity in the two bonding systems. (J Kor Acad Cons Dent 32(1):9-18, 2007)

Key words: Adhesive thickness, Microtensile bond strength, Cavity wall, Filled adhesive, Unfilled adhesive

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I . Introduction

Nowadays, dental composite resins are used increasingly by practitioners for esthetic qualities. As presently posterior composites are suited to be bonded Class I and Class II cavity preparations.

The mechanism of adhesive bonding is based on acid etching both enamel and dentin of the tooth

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cavity surface. Although adhesion to phosphoric acid-etched enamel is reliable and long-lasting, adhesion to dentin has been more challenging because of the complex mineral and organic components of dentin. The bonding mechanism of adhesive resins to dentin proposed by Nakabayashi¹⁾ was described as micromechanical bonding due to the impregnation and polymerization of monomers into the exposed collagen of demineralized dentin surface, creating a hybrid layer. The major role of the adhesive resin is the stabilization of the formed hybrid layer and the formation of resin tag in the unplugged dentinal tubules²⁾.

In the adhesive dentistry, total-etch technique has led to major improvements. However, the achievement of reliable bond to dental hard tissue without a separate acid etching step represented a major challenge in the past. Recently, self-etching adhesives were introduced. These systems use hydrophilic, acid monomers which are able to demineralize and penetrate enamel and dentin³⁾. Unlike total-etch adhesives, these systems does not completely resolve or remove the smear layer. Kaaden *et al.*³⁾ studied that the filled bonding agent Clearfil SE bond resulted in bond strengths higher than those of the unfilled adhesive systems. Perdigao and Lopes⁴⁾ suggested that self-etching adhesives should provide optimum bonding besides a simplification of the bonding procedure and a potential decrease in technique sensitivity.

Most of the cavity preparations clinically show not only areas of exposed enamel and superficial dentin but also deep dentinal areas. Then variable location of exposed dentin such as cavity wall and cavity floor are appeared in the cut cavity. Thus a layer of cured adhesive of variable thickness is present on the cavity surface inevitably⁵⁾. Adhesive thickness in the cavity wall is variable along the cavity depth by the gravity. In clinical situation, pooling of adhesive was apparent in the internal angle of the cavity and then decreased in thickness toward cavity margin. Peter *et al.*⁶⁾ suggested that pooling of dentin bonding agents at the internal angle of the cavity arises because of

the difference in viscosity between primers and unfilled adhesives. Perdigao *et al.*^{7,8)} noticed that air thinning had a tendency to cause pooling of the adhesive into irregularities on the dentin surface and at the internal angle of the cavity. During the application of the adhesive, the manufacturer recommends that it should be applied in a uniform coating and then excessive air thinning of the adhesive should be avoided⁹⁾.

Moreover, the variable adhesive thickness on the different area of the cavity would affect on the bond strength. Zheng *et al.*¹⁰⁾ suggested that the effect of the thickness of the adhesive layer on bond strength is material-dependent. In their study, the increase in bond strengths of Clearfil Liner Bond 2V was directly proportional to the thickness of the bonding layer and the bond strengths of Single Bond decreased significantly with increase adhesive resin thickness. They emphasized that care should be taken to avoid excess adhesive resin at line angle in cavities bonded with single bottle system that contain water and ethanol¹⁰⁾.

Recently, fillers have been added to single-bottle adhesive systems to reinforce the hybrid layer and increase bond strength. Increased filler loading increases viscosity of the bonding system and may reduce its flow¹¹⁾. In general, the variability of film thickness on the different site of the cavity is dependent on the type of bonding agents with or without filler because of the difference of their viscosity and flow. Grossman and Setzer⁵⁾ showed that the bonding layer was thinnest at the cavity margin and thickest at the internal angle of the cavity for both type of the bonding agents. In addition, they described that layer thickness for filled adhesive, Optibond showed a progressive increase down the cavity wall to the internal angle while unfilled adhesive, Scotchbond Multipurpose showed great film thickness variability⁵⁾. Therefore, the variability of the adhesive thickness using of the different type of the bonding agents, filled adhesive and unfilled adhesive, should be evaluated on the prepared cavity.

The purposes of this study were to examine the variability of adhesive thickness on the different

site of the cavity wall when used total-etch system without filler and simplified self-etch system with filler and to evaluate the relationship between variable adhesive thickness and microtensile bond strength to the cavity wall.

II . Materials and Methods

This study was performed in two parts. One part was to measure adhesive thickness on the cavity wall and the other part was to measure microtensile bond strength.

Part I . Adhesive thickness

Preparation of specimens

Six intact, non-carious, non-restored, human molars were used in this study. A class I cavity was prepared to expose all dentinal wall (mesiodistal width: 6 mm, buccolingual width: 4 mm, depth: 6 mm). After cavity preparation, three teeth were bonded with a filled adhesive (Clearfil™ SE bond; Kuraray, Medical Inc., Okayama, Japan) and the other three teeth were bonded with unfilled adhesives (Scotchbond™ Multi Purpose; 3M ESPE, St Paul, MN, USA) by Manufacture's recommendation. Rhodamine B was added in these adhesives. And then a hybrid light-activated resin composite (Z100; 3M ESPE, St. Paul, MN, USA) was filled in six cavities. The

compositions of dentin bonding systems used in the study are described in Table 1.

The bonded teeth were then stored in distilled water at 37°C for 24 hour prior to sectioning. Each tooth was sectioned bucco-lingually into a series of 1.0 mm thick slabs using a high-speed precision cut-off machine (Accutom-50; Struers, Ballerup, Denmark) with water coolant. Twelve specimens were prepared in each group (Table 2).

Fluorescence microscopy

These specimens were observed by fluorescence microscope (Axioskop; ZEISS, Oberkochen, Germany). Morphology and thickness of adhesive layer were founded through phase of diffusion with rhodamine B mixed adhesive resin. Bonding agent thickness was measured at three points along the cavity wall, edge of cavity margin (**rim**), halfway down each cavity wall (**hlf**), internal angle of the cavity (**ang**).

Statistical analysis

Statistical analysis of the collected data was performed by SPSS™ version 12.0 (SPSS Inc., Chicago, IL, USA). After calculating the means and standard deviations of the adhesive thickness for the specific points in each group, Student's *t*-test was used to compare adhesive thickness between two bonding systems. And one-way ANOVA and Scheffé's test for post-hoc compari-

Table 1. Dentin bonding systems used in this study

Dentin boning agent	Composition
Scotchbond™ Multi Purpose	Conditioner: 35% phosphoric acid Primer: HEMA, polyalkenoic acid, co-polymer, water Adhesive resin: Bis-GMA, HEMA
Clearfil™ SE bond	Primer: MDP, HEMA, hydrophilic dimethacrylate, dl-camphoroquinone, N,N-diethanol-p-toluidine, water Bonding: MDP, bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphoroquinone, N, N-diethanol-p-toluidine, silanated colloidal silica

Table 2. Application protocol for adhesive thickness measurement

Treatment group	Application protocol
Scotchbond™ Multi Purpose (SM)	Acid etching: 15 sec Washing: 15 sec; dry Primer application and gentle air dry Adhesive (rhodamine B added) and light curing: 20 sec
Clearfil™ SE bond (SE)	Primer: 20 sec and gentle air dry Bond (rhodamine B added) application and gentle air dry and light curing: 10 sec

Table 3. Application protocol for microtensile bond strength test

Treatment group	Application protocol
Scotchbond™ Multi Purpose 1 coat (SM 1)	Acid etching: 15 sec Washing: 15 sec; dry Primer application and gentle air dry Adhesive and light curing: 20 sec
Scotchbond™ Multi Purpose 7 coat (SM 7)	Apply one coat Apply consecutive coats without waiting between application and light curing: 20 sec Application and curing 5 times additionally
Clearfil™ SE bond 1 coat (SE 1)	Primer: 20 sec and gentle air dry Bond application and gentle air dry and light curing: 10 sec
Clearfil™ SE bond 6 coat (SE 6)	Apply one coat Apply consecutive coats without waiting between application and gentle air dry and light curing: 10 sec Application and gentle air dry and curing 4 times additionally

son was performed to evaluate the difference of adhesive thickness among three points in each group. Statistical significance was defined as $P < 0.05$.

Part II. Microtensile bond strength

Preparation of specimens

Adhesive thickness at each point was reproduced by multiple coating of adhesive. Adhesive

thickness at the *rim* and the *hlf* were same as one coat adhesive layer thickness in both Scotchbond™ Multi Purpose (SM group) and Clearfil™ SE bond (SE group), while adhesive thickness at the *ang* was same as seven coats in SM group and six coats in SE group.

Eight intact, non-carious, non-restored, human molars were used. A class I cavity was prepared to expose all dentinal wall. The teeth were sectioned longitudinally in mesiodistal direction

through the prepared cavity using a diamond disc attached low-speed handpiece with air-water cooling. Sectioned sixteen teeth were classified into four groups.

The four groups were etched and bonded in manner described in Table 3. After dentin bonding procedure, a hybrid light-activated resin composite (Z100; 3M ESPE, St. Paul, MN, USA) was built up free hand in three increments to an approximate height of 6 mm. Each increment was 2 mm and light-cured for 20 s.

The teeth were stored in water at 37°C for 24 hour. The 1 mm × 1 mm sticks were then sectioned mesiodistally and occlusogingivally using a high-speed precision cut-off machine under water coolant. Twenty specimens were prepared in each group.

Microtensile bond strength

The stick was fixed to the test bed using cyanoacrylate adhesive, Zapit (DVA Inc., Corona, CA, USA). The stick was pulled to failure under tension using a Micro Tensile Tester (Bisco inc., Schaumburg, IL, USA) at a crosshead speed of 1 mm/min. And then the microtensile bond strength was recorded in mpa.

Statistical analysis

Statistical analysis of the collected data was performed by SPSS™ version 12.0. After calculat-

ing the means and standard deviations of the microtensile bond strength for each group, Student's *t*-test was used to compare of microtensile bond strength for each group. Statistical significance was defined as $P < 0.05$.

III . Results

Part I . Adhesive thickness

Morphology of adhesive layer

In fluorescence microscopy observation, adhesive layer was pooled only at the internal angle of the cavity for both bonding agents (Figure 1). And for both bonding agents the bonding layer was thinnest at the cavity margin and thickest at the internal angle of the cavity.

Clearfil™ SE bond formed uniform layer over the cavity wall compared to Scotchbond™ Multi Purpose.

Adhesive layer thickness

Table 4 shows the results of adhesive thickness at the specific area in two dentin bonding systems.

In Scotchbond™ Multi Purpose, adhesive thickness of **ang** was significantly thicker than that of rim and **hlf** ($P < 0.05$). Adhesive thickness of **ang** was also significantly thicker than that of rim and **hlf** when Clearfil™ SE bond was used ($P < 0.05$).

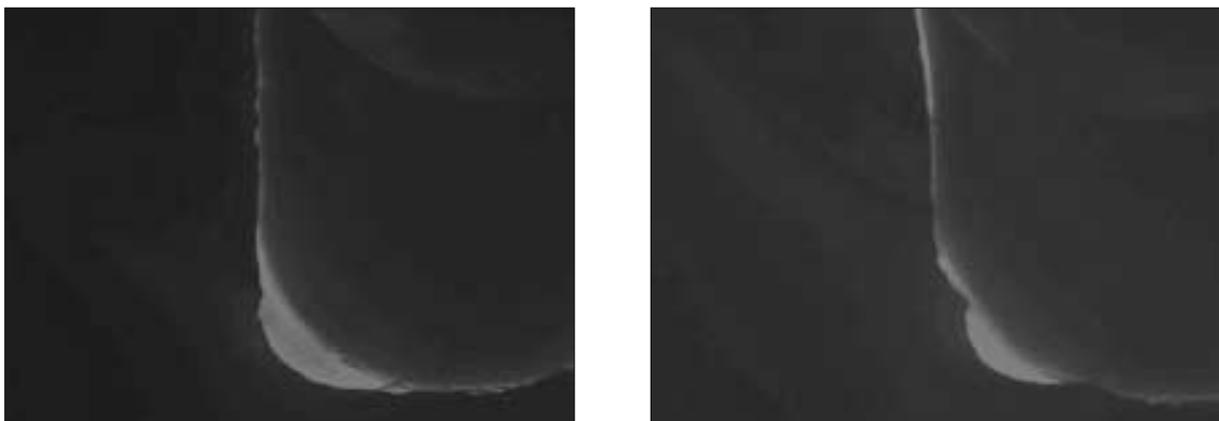


Figure 1. Fluorescence microscopic image of Scotchbond™ Multi Purpose (left) and Clearfil™ SE bond (right).

Table 4. Adhesive thickness (Mean ± SD, μm)

Location	Adhesive thickness		Student <i>t</i> -test
	SM	SE	<i>p</i> -value
rim (n = 20)	17.91 ± 3.09 ^a	35.77 ± 5.45 ^a	< 0.000
hlf (n = 20)	18.11 ± 3.20 ^a	36.12 ± 4.98 ^a	< 0.000
ang (n = 20)	109.24 ± 39.2 ^b	83.67 ± 6.78 ^b	0.009

^{ab}: Different letter indicates significant differences between the groups in vertical low (P < 0.05).

Table 5. Microtensile bond strength (Mean ± SD, MPa)

Groups	MTBS
SM 1 (n = 20)	12.60 ± 3.24
SM 7 (n = 20)	17.43 ± 3.52
SE 1 (n = 20)	11.19 ± 2.53
SE 6 (n = 20)	19.43 ± 3.68

Table 6. Statistical difference by Student *t*-test

	Dentin bonding agent		<i>p</i> -value
	SM1	SE1	
Adhesive thickness	SM7	SE6	0.132
<i>p</i> -value	< 0.000	< 0.000	0.086

In **rim** and **hlf**, adhesive layer thickness of Clearfil SE bond was significantly thicker than that of Scotchbond™ Multi Purpose (P < 0.05). In **ang**, however, adhesive layer thickness of Scotchbond™ Multi Purpose was significantly thicker than that of Clearfil™ SE bond (P < 0.05).

Part II. Microtensile bond strength

Table 5 and 6 show the results of microtensile bond strength for each group. Microtensile bond strength was highest in the SE 6 group (19.43 ± 3.68).

In Scotchbond™ Multi Purpose, seven coats group was significantly higher microtensile bond strength than one coat group (P < 0.05). In Clearfil™ SE bond, six coats group was also significantly higher microtensile bond strength than one coat group (P < 0.05). For both bonding agents, microtensile bond strength was increased as adhesive thickness was increased.

In one coat groups, microtensile bond strength for both adhesive was not significantly different (P > 0.05). Likewise, in multiple coats groups, microtensile bond strength for both adhesive was not significantly different (P > 0.05).

IV. Discussion

An ideal dental adhesive would provide high bond strengths. Bond strength is more predictive of a material's retentive potential¹²⁾. We observed the bonding agent within the cavity and assessed the film thickness of the adhesive layer along the cavity wall and measured microtensile bond strength at different sites of the cavity wall to better understand the bonding agents as an interfacial material in clinical situation.

Part I. Adhesive thickness

In fluorescence microscopy evaluation, Clearfil™ SE bond formed uniform layer over the cavity wall and the internal angle of the cavity compared to Scotchbond™ Multi Purpose. The highly significant difference between the coatings of the bonding agents on the cavity surface appears to be related to the type of bonding agent with or without filler. Clearfil™ SE bond with filler contents of about 10% has increased viscosity and reduced flow³⁾. Film thickness should be even along the entire composite resin-tooth interface to ensure consistent bonding and uniform stress distribution⁵⁾. The location of failure sites is often

interpreted as reflecting the weakest link in the restoration system⁵. It could be that the high proportion of mixed failures in tensile and shear bond test may be related to variations in bonding agent thickness^{4,13,14}. Therefore, an aspect of uniform stress distribution, Clearfil™ SE bond would be superior to Scotchbond™ Multi Purpose.

Because of an effect of the gravity, pooling of adhesive was apparent at the internal angle of the cavity and then decreased in thickness toward cavity margin. For both bonding agents, the bonding layer was thinnest at the cavity margin and thickest at the internal of the cavity. This result was agreement with Grossman's study⁵. And adhesive layer was pooled only at the internal angle of the cavity for both bonding agents. Peter *et al.*⁶ reported on film thickness reaching 254 μm in the line angle of cavity preparations. They suggested that the difference in viscosity between primers and adhesives cause the pooling of dentin bonding agents at the internal angle of the cavity in experiments used unfilled adhesives. And they proposed that air thinning is unable to drive the higher viscosity adhesive through the primed collagen network because of the damming effect of the preparation angle.

Filler should increase viscosity of the adhesives. It should be that the higher viscosity adhesive, Clearfil™ SE bond is less driven through the primed collagen network than lower viscosity adhesive, Scotchbond™ Multi Purpose. But in this study adhesive layer thickness of unfilled adhesive, Scotchbond™ Multi Purpose was significantly thicker than that of filled adhesive, Clearfil™ SE bond at the line angle. It seems that the reason of this result would be an effect of the gravity and the reduced flow of filled adhesive. Therefore, the higher viscosity, Clearfil™ SE bond would not flow to the bottom well though damming effect of internal angle of the cavity.

At the cavity margin and the halfway cavity wall, on the other hand, adhesive layer thickness of filled adhesive, Clearfil™ SE bond was significantly thicker than that of unfilled adhesive, Scotchbond™ Multi Purpose. In the case of the filled adhesive, the increased viscosity and the

role of the oxygen inhibition layer is a major cause of thick bonding layers⁶. Opdam *et al.*¹⁶ reported that thick adhesive layer seems to prevent the formation of interfacial gaps between tooth and restoration and act as a superior elastic buffer compared to thinner layers, whereas Hilton and Schwartz¹⁷ suggested that thick adhesive layer adversely affects bond strengths, increases crack propagation, elevates the thermal coefficient of expansion mismatch with the tooth and decreases the load bearing and wear component of the restoration.

Ultramorphological findings were able to demonstrate hydroxyl apatite crystals within the hybrid layer after the use of Clearfil™ SE bond. It was speculated that the bonding mechanism of Clearfil™ SE bond might depend upon interlocking with these crystals, possibly resulting in more rigid and compact interface¹⁸. Thin hybrid layer and thick adhesive layer may also be advantageous³.

Part II . Microtensile bond strength

Hybrid layer formation plays an important role in achieving maximum bond strengths between resin and dentin^{1,19,20}. Increased thickness of the adhesive resin film would result in higher bond strengths by improving stress distributions in the bonded assembly¹⁰. In this study, microtensile bond strength was increased as adhesive thickness was increased in two bonding agents. The increase in the tensile bond strength of both bonding agents with an increase in the thickness of the adhesive layer may be due to the improved stress distributions. The thicker adhesive layer may permit self-alignment of the specimen that corrects for minor deviations in specimens placement, thereby, improve stress distributions during testing, yield higher apparent bond strengths¹⁰. In this study, consequently, microtensile bond strength of the thick adhesive layer reproduced the adhesive thickness of internal angle of the cavity was higher than that of the thin adhesive layer reproduced the adhesive thickness at the cavity margin and the halfway cavity in the

two bonding systems. Therefore, on the assumption that other conditions are same, microtensile bond strength at the internal angle of the cavity is higher than that at the other sites of cavity wall.

Recently, fillers have been added to adhesive systems to reinforce the hybrid layer and increase bond strength. Some^{21,22)} have advocated the addition of filler to dentin adhesives because the filler might improve the mechanical properties of the material and act as elastic buffer beneath the restorative material. In this study, however, microtensile bond strength between two bonding systems were not significantly different on the specimens reproduced the each sites to the cavity wall. From the results of this study, more research is needed to know the relationship of adhesive thickness and microtensile bond strength in relation to the addition of filler and adhesion strategy.

In many other studies^{3,13,23)}, generally bond strengths are higher in superficial than deep dentin. Marshall *et al.*²³⁾ suggested that the nature of the substrate presented for bonding would vary with location.

For this reason, we support that high microtensile bond strength with thick adhesive layer at the internal angle of the cavity would compensate for the low bond strength due to dentinal structure in the deep dentin when used the two bonding systems. Therefore, the phenomenon of the pooling at the internal angle of the cavity would not be problem.

In clinical situation, additionally, simplified two step dentin bonding system, Clearfil™ SE bond has microtensile bond strength comparable with three steps, Scotchbond™ Multi Purpose. Accordingly, it would not be problem that Clearfil™ SE bond is applied to the cavity wall under influence of the gravity. Further studies on the bond strength of adhesive within the prepared cavity are needed considering other variables.

V. Conclusions

1. Adhesive layer was pooled only at the internal angle of the cavity for both bonding agents. Clearfil™ SE bond formed uniform layer over the cavity wall compared to Scotchbond™ Multi Purpose.
2. For both bonding agents, adhesive thickness of internal angle of the cavity was significantly thicker than that of the cavity margin and the halfway cavity wall ($P < 0.05$).
3. Microtensile bond strength of the thick adhesive layer at the internal angle of the cavity was higher than that of the thin adhesive layer at the cavity margin and the halfway cavity in the two bonding systems ($P < 0.05$).

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국문초록

와동벽에서 접착제의 두께가 미세인장 결합강도에 미치는 영향

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이 연구의 목적은 와동벽에서 다른 위치에서의 상아질 접착제의 두께를 평가하고, 이런 다양한 접착제의 두께와 미세 인장 강도 사이의 관계를 평가하기 위한 것이다.

여섯 개의 인간 대구치에 모든 상아질 면이 노출되도록 I 급 와동을 형성하였다. 3개의 치아는 filled adhesive (Clearfil™ SE bond)를 와동 내에 도포하였고, 다른 3개의 치아는 unfilled adhesives (Scotchbond™ Multi Purpose)를 도포하였다. 형광 현미경을 이용하여 접착층의 형태와 두께를 관찰하였다. 접착제의 두께는 수직 와동벽을 따라 와동 변연, 와동벽 1/2, 와동 내각의 세 지점에서 측정되었다. Scotchbond™ Multi Purpose와 Clearfil™ SE bond가 와동 변연과 와동벽 1/2, 와동 내각에서의 접착제의 두께를 재현하여 미세 인장 결합 강도를 측정하였다.

이 실험의 결과에서 두 가지 상아질 접착제 모두에서 와동 내각에서의 접착제의 두께가 와동 변연과 와동벽 1/2 위치에서의 두께보다 두꺼웠으며, 와동 내각의 두꺼운 접착제의 미세 인장 결합 강도는 와동 변연과 와동벽 1/2에서의 얇은 접착제 두께의 미세 인장 결합 강도보다 유의성 있게 높게 나타났다.

주요어: 접착제의 두께, 미세 인장 결합 강도, 와동벽, 필러가 포함된 접착제, 필러가 포함되지 않은 접착제