

The effect of different bonding systems on shear bond strength of repaired composite resin

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ABSTRACT

The purpose of this study is to compare the shear bond strength of repaired composite resin with different bonding agents and evaluate the effect of bonding agents on composite repair strength. Forty composite specimens (Z-250) were prepared and aged for 1 week by thermocycling between 5 and 55°C with a dwell time of 30s. After air abrasion with 50 μ m aluminum oxide, following different bonding agents were applied (n = 10): SB group: Scotchbond multipurpose adhesive (3 step Total-Etch system); SE group: Clearfil SE bond (2 step Self-Etch system); XP group: XP bond (2 step Total-Etch system); XE group: Xeno III (1 step Self-Etch system). After bonding procedure was completed, new composite resin (Z-250) was applied to the mold and cured. For control group, 10 specimens were prepared. Seven days after repair, shear bond strength was measured. Data was statistically analyzed using one-way ANOVA and Tukey's test ($p < 0.05$). The means and standard deviations of shear bond strength ($\bar{m} \pm S.D.$) per group were as follows: SB group: 17.06; SE group: 19.10; XP group: 14.44; XE group: 13.57; Control Group: 19.40. No significant difference found in each group. Within the limit of this study, it was concluded that the different type of bonding system was not affect on the shear bond strength of repaired composite resin. [J Kor Acad Cons Dent 33(2):125-132, 2008]

Key words : Composite repair, Aluminum oxide sandblasting, Bonding system, Shear bond strength

- Received 2008.2.19., revised 2008.3.6., accepted 2008.3.7.-

I . INTRODUCTION

Composite resin restoration has been increasingly used in restorative dentistry with patient's demands, and also composite resin materials as well as bonding agents have been developed con-

tinuously. Despite of these developments, several factors, such as inadequate form, wear, color mismatch, chipping or bulk fracture may still present concerns¹⁾. In case of failure, the clinician must determine whether to replace the whole restoration or repair the part of restoration.

When the restoration is completely replaced, it is inevitable that further tooth loss lead to the weakening of tooth structure or pulp damage. Therefore, if we could obtain adequate bond strength between old and new composite resin, the repair instead of the replacement could be better treatment option from minimally interven-

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tion approach.

Gordan *et al.*²⁾ studied about two-year clinical evaluation of repair versus replacement of composite restoration. They concluded that resin-based composite restorations that present less-than-ideal marginal adaptation and stained margins were better off being repaired.

Many laboratory studies³⁻¹³⁾ reported that the aged composite resin restoration provided sufficient bond strength by adequate surface treatment and bonding agent. Various pre-treatment methods have been suggested to produce adequate bond strength between old and new composite^{3-8,10-12)}. These methods include the aluminum oxide sandblasting, roughening with diamond instrument, acid etching, silanization, application of bonding agent, etc. In several *in vitro* studies^{3,4,9-11)}, it was concluded that aluminum oxide sandblasting, followed by application of a bonding agent, proved to be a reliable method for enhancing composite repair strength.

Aluminum oxide sandblasting is surface treatment that causes microretentive feature and increasing the surface area available for wetting by adhesive resin³⁾. By penetrating the adhesive resin into the surface microirregularities, micro-mechanical interlocking is produced.

Some researchers^{13,14)} investigated about the effect of different bonding agents on bond strength between old and new composite resin. Cavalcanti *et al.*⁴⁾ reported that Clearfil SE Bond as a self-etching system showed higher repair bond strength than Single Bond as a 3 step total-etching system. They explained that it was because the Clearfil SE Bond contained the proprietary acid phosphate monomer 10-methacryloyloxydecyl dihydrogen phosphate and the acidic monomer might have a role in the higher capacity to wet the composite surface. Teixeira *et al.*¹⁴⁾ studied about shear bond strength of various self-etching bonding systems. In their study, Optibond Solo Plus SE showed the higher bond strength compared to some other self-etching systems. Shahdad *et al.*¹⁵⁾ said that long-established bonding agents based on chlorophosphate esters of

bis-GMA have proven to be acceptable choices as intermediate materials. Lucena *et al.*⁷⁾ described that low-viscosity filled adhesives have been demonstrated to have a high capacity to wet composite surfaces and penetrate the organic phase of the composite.

However, there were few studies about the difference of repair bond strength among various bonding systems. Currently available bonding systems include 3 step total-etch system, 2 step total-etch system, 2 step self-etch system and 1 step self-etch system. Presumably, 2 step total-etch system and 1 step self-system might have lower repair bond strength because of their priming components.

Therefore, the purpose of this study is to compare the shear bond strength of repaired composite resin with different bonding systems and evaluate the effect of bonding systems on composite repair.

II. MATERIALS AND METHODS

1. Specimen Preparation

The materials used in this study are listed in Table 1. Forty composite resin discs were made from hybrid composite resin (Z-250, Shade A3; 3M ESPE, St. Paul, MN, USA). Cylindrical teplon mold with an inner diameter of 8 mm and a height of 2 mm was used. Composite resins were inserted into the mold, and then they covered with glass cover plate placed perpendicular to the long axis of the cylinder and cured for 20 s at 90 degrees to the top surface with a light curing unit (Bluephase; Ivoclar Vivadent, Shann, Liechtenstein). After the specimens were removed from the mold, they were cured for a further 20 s on the portions of the specimens that were in contact with the mold, in order to ensure uniform and complete polymerization.

Aging was achieved by thermocycling for 1 week between 5°C and 55°C with a dwell time of 30 s.

Table 1. Materials used in this study

Material	Composition	Manufacturer
Z-250 (A3)	zirconia/silica filler (60 vol% without silane treatment, particle size range of 0.01 to 3.5 μm), BIS-GMA, UDMA, BIS-EMA	3M ESPE, St. Paul, MN, USA
Scotchbond Multipurpose	Adhesive: BIS-GMA, HEMA, photoinitiator	3M ESPE, St. Paul, MN, USA
Clearfil SE Bond	SE Bond: N,N-Diethanol-p-toluidine, MDP, BIS-GMA, HEMA, hydrophobic dimethacrylate, DL-camphorquinone, silanated colloidal silica	Kurary Co., Osaka, Japan
XP Bond	carboxylic acid modified dimethacrylate phosphoric acid modified acrylate resin (PENTA), UDMA, TEGDMA, HEMA, butylated benzendiol (stabilizer) Ethyl-4-dimethylaminobenzoate, camphorquinone Functionalized amorphous silica	Dentsply Caulk, Milford, DE, USA
Xeno III	Liquid A: HEMA, water, ethanol, silicon dioxide Liquid B: phosphoric acid modified methacrylate resin, UDMA, BHT, camphorquinone, ethyl-4-dimethylaminobenzoate	Dentsply Caulk, Milford, DE, USA

* Abbreviations: BIS-GMA = bisphenol-A-glycidyl ether dimethacrylate; UDMA = urethane dimethacrylate; BIS-EMA = ethoxylated bisphenol-A-dimethacrylate; HEMA = 2-hydroxyethyl methacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; PENTA = dipentaerythritol penta acrylate monophosphate, TEGDMA = triethylene glycol dimethacrylate; BHT = butylhydroxytoluene.

2. Surface Treatment

All specimens were air abraded for 10 s from a distance of approximately 5 mm perpendicular to the specimen surface using an intraoral air abrasion device (Danville Engineering Inc., Danville, CA, USA) filled with 50 μm aluminum oxide particles. Then they were rinsed with distilled water, and dried with oil-free compressed air.

The specimens were randomly assigned to four groups for different bonding agents ($n = 10$). Each group was following:

- ① SB group: Scotchbond Multi-Purpose (3M ESPE, St. Paul, MN, USA)
- ② SE group: Clearfil SE Bond (Kurary Co., Osaka, Japan)
- ③ XP group: XP Bond (Dentsply Caulk, Milford,

DE, USA)

- ④ XE group: Xeno III (Dentsply Caulk, Milford, DE, USA)

Directions for bonding procedure of each group are described in Table 2.

Ten more specimens were prepared for control group and these were not treated with bonding agent.

3. Application of the Repair Composite

After completion of bonding procedure as above, each specimen was positioned into the mold, and fresh resin, same as respective composite substrate (Z-250, Shade A3), was applied in 2 mm increments to the substrate surface using a polyethylene mold with a diameter of 3 mm. Then they

Table 2. Bonding procedures

Bonding agent	Procedures
Scotchbond Multi-Purpose (3 step total-etch system)	Scotchbond Multi-Purpose adhesive resin was applied, gently air dried and cured for 10 s.
Clearfil SE Bond (2 step self-etch system)	SE Bond was applied, gently air dried, and light cured for 10 s.
XP Bond (2 step total-etch system)	XP Bond was applied and leaved the surface undisturbed for 20 s. Solvent was evaporated by thoroughly blowing with air from an air syringe for at least 5 s. Light cured for 10 s.
Xeno III (1 step total-etch system)	Equal amounts of liquid A and B was dispensed into dispensing dish and mixed for approximately 5 s. They were applied for 20 s and dried by a gentle stream of air pressure for at least 2 s. Then light cured for 10 s.

**Figure 1.** Shear bond test machine.

were cured for 20 s.

The specimens were removed from the mold and thermocycled for 1 week between 5°C and 55°C with a dwell time of 30 s.

4. Shear Bond Strength Test

Following this aging process, shear bond test was performed using a shear bond test machine (R&B Inc., Daejeon, Korea) (Figure 1). The specimens were fixed in a mounting jig, and then they were loaded exactly at the intermaterial interface. The load at failure was recorded in newton (N)

and divided into cross section area (mm^2). Then, shear bond strength was expressed in MPa .

5. Fracture mode investigation

Fracture mode was also investigated under operating microscope (OPMI pico; Carl zeiss, Obercohen, Germany). It was classified as cohesive within the substrate or the repairing composite, adhesive within the bonding agent, or mixed failure.

6. Statistical analysis

Statistical analysis was performed with one-way ANOVA using SPSS 12.0 software (SPSS, Chicago, IL, USA). Tukey test was used for post-hoc multiple comparison. The level of significance was set at $p < 0.05$.

III. RESULTS

The mean and standard deviation of the shear bond strength of all tested group were presented in Table 3. Different bonding agents had no significant effect on shear bond strength between old and new resin. And all experimental groups were not significantly different with control group.

Fracture mode was presented in Table 4. Only in control group, 100% cohesive failure was

Table 3. Mean shear bond strength, each group n = 10

	Mean \pm SD (MPa)*
SB Group	17.06 \pm 6.33
SE Group	19.10 \pm 5.29
XP Group	14.44 \pm 3.53
XE Group	13.57 \pm 3.65
Control Group	19.40 \pm 4.06

*There was no significant difference between groups ($p > 0.05$).

occurred. In SB and XP group, only one specimen respectively presented adhesive failure, all other specimens showed cohesive or mixed failure.

IV. DISCUSSION

Repairing failed composite restorations could be considered as a minimal invasive and cost-effective treatment option. During composite resin repair, two critical factors must be considered: surface treatment and intermediate agent. While surface treatment promotes mechanical interlocking, the intermediate agent improves surface wetting and chemical bonding with the new composite.

Subject of adequate surface treatment was suggested by many investigators³⁻¹²⁾ in various methods, such as aluminum oxide sandblasting, roughening with diamond bur and silanization. It was demonstrated that aluminum oxide sandblasting of aged composite resin surface was most effective surface treatment method^{3,4)}. It was said that aluminum oxide sandblasting caused microretentive feature and increasing the surface area available for wetting by adhesive resin. So, in this study, aluminum oxide sandblasting was selected for surface roughening.

The primary focus of this study was to evaluate the effectiveness of different adhesive systems for repairing aged composites. Several previous studies^{3,4,9-11)} have recommended application of intermediate materials to improve bond strength for repairs. Bonding agents are used for penetrating into the microirregularities and increasing bond

Table 4. Failure mode

	Cohesive	Mixed	Adhesive
SB Group	4	5	1
SE Group	5	5	0
XP Group	0	9	1
XE Group	4	6	0
Control Group	10	0	0

strength. There are several bonding systems available in dentine bonding procedure. They are divided into 3 step, 2 step, and 1 step, and 2 step is subdivided into self-etching primer and self-priming adhesive. Generally, it have been known that the 3 step etch and rinse adhesives remained the 'gold standard' in terms of adhesion durability in dentine bonding and any kind of simplification in the clinical application procedure resulted in a loss of bonding effectiveness. To investigate that the general concepts about dentine bonding systems are also applicable to composite repair, we used four different adhesive systems for experimental group in this study. : Scotchbond Multipurpose (3 step total-etch system), Clearfil SE bond (2 step self-etch system), XP bond (2 step total-etch system), Xeno III (1 step self-etch system). In this study, it was concluded that different bonding systems had no significant effect in repair bond strength.

In XP and XE group, we assumed that they had lower shear bond strength than SB and SE group because of their priming components containing both hydrophilic and acidic resin monomer. Like our expectation, it was resulted that XP and XE group had lower shear bond strength than SB and SE group, but no significant difference found in each group. From this result, priming components of XP and XE group might not be critical factor in composite resin repair bond strength.

For measuring of repair bond strength, shear bond test was used in this study. Shear bond test is a method used in many studies to investigate the bond between adhesive materials and dentin

or enamel. In this study, large percentage of all groups showed a cohesive failure. And the cohesive shear strengths of the control group had not significant difference with the shear bond strength of the experimental groups. From this result, it is thought that the repairing procedure used in this study was adequate for aged composite resin repair. If a composite repair tends to fracture within the original composite (cohesive fracture), one can assume the selected protocol to be appropriate to bear the occlusal loads. The location of the repair failure within the repaired material itself, rather than at the adhesive surface, suggests a better bond¹⁶⁾. Nevertheless, shear bond strength tests may cause cohesive fractures of the substrate. This must be taken into account in the interpretation of the results.

Tezvergil *et al.*¹³⁾ reported that storage conditions did not show any significant effect. On the other hand, Ozcan *et al.*⁸⁾ reported that thermocycling seemed to be more effective in degradation of the composite tested. In addition to the weakening effect on physiochemical properties, temperature alterations could decrease the number of unreacted double bonds on the surface or within the composite which in turn may affect the composite-composite repair strength. Therefore, for producing the similar clinical condition, specimens were submitted to a themocycling in this study.

According to the Söderholm's study¹⁷⁾, it has been suggested that the greatest residual free radical activity of the substrate can be found on the surface of the substrate during the first 24h after polymerization. But, Boyer *et al.*¹⁸⁾ reported that the adhesion between aged composite resin and repairing composite resin gradually decreased up to 24 hours, and when comparing the adhesion after 1 day and 7 days, those after 7 days showed little decrease from the adhesion than those after 1 day. This decrease in adhesion is because of decrease in number of un-reacted monomers on the surface of restored resin as polymerization occurs¹⁸⁾. So, this experimental study was tested after 1 week thermal cycling. However, Sau *et al.*¹⁹⁾ reported that there were general increases in the repair shear bond strengths at 1 week and

general deteriorations at 4 weeks. Hence, further studies are required to address the effect of long-term storage in a moist environment on repair shear bond strength.

Consequently, repairing procedure selected in this study, air abrasion followed by the bonding agent, was provided the sufficient bond strength for aged composite resin repair. But, there was no significant difference between all experimental groups. From this result, bond strength of repaired composite resin seems to have no difference regardless of bonding system used.

For both dentine bonding and re-bonding for repairs, it is crucial that the intermediate system wet the substrate surface and employ a solvent that encourages bonding system monomer to penetrate into the substrate. Both of these key factors are influenced by the application protocol that might include multiple applications, longer dwell times and more careful solvent drying.¹⁴⁾ Therefore, it is required that the clinician should apply the bonding agent carefully according to the manufacturer's instruction.

From the result of this study, the bonding system had no significant effect on the composite repair bond strength, but clinically, it should be also considered the enamel and dentine bonding. It is because that enamel and dentine wall often might be contained with prepared cavity during the repair of restoration. Therefore, the bonding system should be selected considering enamel and dentine bonding as well as composite repair strength.

V. CONCLUSIONS

Under the condition of application of aluminum oxide sandblasting followed by the bonding agent, different bonding systems seem to have no effect on the shear bond strength of repaired composite resin.

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국문초록

접착 시스템이 수리된 복합 레진의 전단 결합 강도에 미치는 영향

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이 연구의 목적은 서로 다른 접착제들로 수리된 복합 레진의 전단 결합 강도를 비교하고, 접착 시스템이 복합레진의 수리 강도에 미치는 영향을 평가하기 위한 것이다. 40개의 복합 레진 시편 (Z-250)을 준비하였으며, 1주일 동안 열순환을 시행한 후, 50 μ m 알루미나 입자로 샌드 블라스팅 하였다. 이 시편들을 네 개의 그룹으로 나누었으며 (n = 10), 다음과 같은 서로 다른 종류의 접착제를 각각 적용하였다; SB 그룹 : Scotchbond multipurpose ; SE 그룹 : Clearfil SE bond ; XP 그룹 : XP bond ; XE 그룹 : XenoⅢ. 접착 과정을 완료한 후에, 새로운 레진 (Z-250, 3M ESPE)을 몰드에 적용하여, 광조사 하였다. 대조군으로, 접착 과정 없이 10개의 시편을 준비하였다. 7일 동안 열순환을 시행한 후 전단 결합 강도를 측정하였고, 파절 양상을 조사하였으며, 다음과 같은 결과를 얻었다.

1. 기존 복합 레진과 수리용 복합 레진 사이의 전단 결합 강도는 접착제 종류에 따른 유의한 차이를 보이지 않았다.
2. 재료의 응집 강도 (대조군)와 수리한 결합 강도 (실험군) 사이에 유의한 차이를 보이지 않았다.
3. 파절 양상은 대부분 응집성 파절 또는 혼합 파절을 보였다.

주요어: 복합레진 수리, 알루미나 샌드블라스팅, 접착 시스템, 전단 결합 강도