

ADHESIVE SYSTEM AFFECTS REPAIR BOND STRENGTH OF RESIN COMPOSITE

Adeziv Sistem Kompozit Rezinin Tamir Bağlanma Dayanımını Etkiler

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Received: 28/10/2016

Accepted: 01/02/2017

ABSTRACT

Purpose: This study evaluated the effects of different adhesive systems on repair bond strength of aged resin composites.

Materials and Methods: Ninety composite discs were built and half of them were subjected to thermal aging. Aged and non-aged specimens were repaired with resin composite using three different adhesive systems; a two-step self-etch adhesive, a two-step total-etch adhesive and a one-step self-etch adhesive; then they were subjected to shear forces. Data were analyzed statistically.

Results: Adhesive type and aging significantly affected the repair bond strengths ($p < 0.0001$). No statistical difference was found in aged composite groups repaired with two-step self-etch or two-step total-etch adhesive. One-step self-etch adhesive showed lower bond strength values in aged composite repair ($p < 0.0001$).

Conclusion: In the repair of aged resin composite, two-step self-etch and two-step total-etch adhesives exhibited higher shear bond strength values than that of one-step self-etch adhesive.

Keywords: Resin composite; repair; shear; adhesive; bond strength

ÖZ

Amaç: Bu çalışmada farklı adeziv sistemlerin yaşlandırılmış rezin kompozitlerin tamir bağlanma dayanımı üzerindeki etkileri incelenmiştir.

Gereç ve yöntem: Hazırlanan 90 adet kompozit diskin yarısına ısıl yaşlandırma uygulaması yapıldı. Yaşlandırılmış ve yaşlandırılmamış örnekler iki aşamalı kendinden asitli adeziv, iki aşamalı asitle-yıka adeziv ve tek aşamalı kendinden asitli adeziv olmak üzere üç farklı adeziv sistem kullanılarak kompozit rezinle tamir edildi. Örnekler makaslama kuvvetlerine maruz bırakıldı. Bağlanma dayanımı verileri istatistiksel olarak analiz edildi.

Bulgular: Adeziv tipinin yaşlandırma tamir bağlanma dayanımlarını belirgin olarak etkilediği belirlendi ($p < 0.0001$). İki aşamalı kendinden asitli adeziv ve iki aşamalı asitle-yıka tipi adeziv ile tamir edilen kompozit grupları arasında istatistiksel olarak anlamlı bir fark bulunmadı. Yaşlandırılmış kompozitlerin tamirinde tek aşamalı kendinden asitli adeziv daha düşük bağlanma dayanımı gösterdi ($p < 0.0001$).

Sonuç: Yaşlandırılmış kompozit rezinlerin tamirinde kullanılan iki aşamalı kendinden asitli ve iki aşamalı asitle-yıka tipi adezivler, tek aşamalı kendinden asitli adezive göre daha yüksek makaslama bağlanma dayanımı değerlerine sahiptir.

Anahtar kelimeler: Kompozit rezin; tamir; makaslama; adeziv; bağlanma dayanımı

Introduction

Resin composite restorations are widely used by many dentists mostly with respect to their esthetic properties. Besides, developments in adhesive systems help clinicians to restore teeth by using the minimally invasive approach and prolong the lifespan of both tooth and restoration. Due to their increased service life, resin composite restorations may eventually suffer from degradations, fractures, discolorations, marginal gaps and secondary caries. In these circumstances, clinician is faced with a choice of either replacement or repair of the defective composite restoration. Replacement of a defective restoration would lead to larger restorations, which would eventually fail and consequently would lead to more complex restorations, root canal therapy and even tooth loss. This restorative cycle of death of the tooth (1, 2) could be prevented by the repair of the composite restorations, since repair requires little intervention to the restoration and the tooth. Although some restorations would need replacement; in the case of repair, treatment times and costs are reduced and tooth tissue is conserved (3). Clinical studies showed that, repair of the composite restorations may be a feasible and an alternative treatment to replacement (4, 5). However, replacement or repair of the restoration often depends on the decision of the dentist. Eventually, the choice of repair versus replacement of defective restorations are based on patient-centered or tooth-specific criteria (3). Tooth-specific criteria include but not limited to, localized marginal defects and stains, small fractures of the restoration, discolorations, localized degradations and fractures of surrounding tooth. Difficulties in patient cooperation, patients with compromised medical conditions are suitable for restoration repairs. Several methods, alone or in combination, are suggested to provide a sufficient bond between the old and the repair composite as follows; roughening with coarse

diamond burs (6-9), phosphoric acid etching (6, 8, 10-14), sandblasting with aluminum oxide powder (6, 8, 15, 16) and application of silane coupling agent (17, 18). The use of an intermediate adhesive agent has also an impact on the bond strength of the repair restorations (9, 19). The purpose of this study was to evaluate the effects of different adhesive systems on the repair bond strength of aged resin composite. The null hypothesis tested was the type of the adhesive system would not have any effect on repair bond strength of the aged resin composite.

Materials and Methods

Experimental groups

This study was carried out in three main groups according to the adhesive system used. Each main group was further divided into two subgroups as aging and control (Table 1). In all groups the same resin composite resin material was applied (Table 2).

Specimen preparation

Ninety resin composite discs (8 mm diameter, 5 mm height) were incrementally prepared in a teflon mold using a resin composite (Clearfil Majesty Esthetic, Kuraray, Osaka, Japan). Each 2 mm-increment was light-cured for 20 s with a LED curing unit (Demetron II, Kerr, Orange, CA, USA) with an output intensity of 750 mW/cm². After polymerization, discs were removed from mold and the bottom surfaces of the discs were additionally cured for 60 s. To create a uniform smear layer, topmost surfaces of the specimens were abraded with 600 grit silicon carbide paper under running water for 30 s. All discs were cleaned in distilled water using an ultrasonic cleaner. Cleaned specimens were kept in distilled water at 37°C for 24 h.

Table 1. Experimental groups (2SE, two-step self-etch adhesive; 2TE, two-step total-etch adhesive; 1SE, one-step self-etch adhesive; TA, thermal aging).

Groups	Composite	Thermal Aging	Adhesive System	Repair Composite
2SE-control	Clearfil Majesty Esthetic	-	Clearfil SE Bond	Clearfil Majesty Esthetic
2SE-TA	Clearfil Majesty Esthetic	+	Clearfil SE Bond	Clearfil Majesty Esthetic
2TE-control	Clearfil Majesty Esthetic	-	XP Bond	Clearfil Majesty Esthetic
2TE-TA	Clearfil Majesty Esthetic	+	XP Bond	Clearfil Majesty Esthetic
1SE-control	Clearfil Majesty Esthetic	-	I Bond	Clearfil Majesty Esthetic
1SE-TA	Clearfil Majesty Esthetic	+	I Bond	Clearfil Majesty Esthetic

Table 2. Materials used in the study (4-META, 4-methacryloyloxyethyl trimellitate anhydride; 10-MDP, 10-methacryloyloxydecyl dihydrogenphosphate; Bis-GMA, bisphenol A diglycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; PENTA, dipentaerythritol pentaacrylate monophosphate; TCB, butan- 1,2,3,4-tetracarboxylic acid di-2-hydroxyethylmethacrylate ester; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate).

Brand	Type	Ingredients	Manufacturer
Clearfil Majesty Esthetic	Resin composite	Silanated barium glass filler (40%vol, 0.37-1.5 µm particle size), pre-polymerized organic filler, Bis-GMA, hydrophobic aromatic dimethacrylate, dl-camphorquinone	Kuraray Co., Ltd, Osaka, Japan
Clearfil SE Bond	2-step self-etch adhesive	Primer: MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, water Bond: MDP, HEMA, Bis-GMA, hydrophobic dimethacrylate, photo-initiator, silanated colloidal silica	Kuraray Co., Ltd, Osaka, Japan
XP Bond	2-step total-etch adhesive	PENTA, TCB resin, UDMA, TEGDMA, HEMA, nanofiller, camphorquinone, butylated benzenediol ,tertiary butanol	Dentsply Caulk, Milford, CT, USA
I Bond	1-step self-etch adhesive	UDMA, 4-META, glutaraldehyde, acetone, water, photo-initiator, stabilizer	Heraeus Kulzer GmbH, Hanau, Germany

All composite discs were roughened by a diamond bur (150µm grit size). A new diamond bur was used for each specimen. Each bur was used with five back and forth strokes for a total of 10 s using a high-speed hand-piece under air and water cooling. Specimens were thoroughly cleaned with water and air dried.

Testing procedures

Specimens were equally divided into 3 main groups according to the adhesive system used; two-step self-etch adhesive (2SE) (Clearfil SE Bond, Kuraray, Osaka, Japan), two-step total-etch adhesive (2TE) (XP Bond, Dentsply Caulk, Milford, CT, USA) and one-step self-etch adhesive (1SE) (I-Bond, Heraeus Kulzer, Hanau, Germany). Half of the specimens in each main group were subjected to thermal aging (TA) in distilled water for 50000 cycles (5-55°C, 60 s immersion time, 10 s transfer time) to simulate the aged, old composite. Other halves in each main group did not receive any aging protocol (Control) (n=15). Before the application

of the adhesive systems, all specimens were rinsed with water and thoroughly air-dried. In each main group, adhesive systems were applied according to manufacturer's instructions (Table 3).

After applying the adhesives, specimens were placed on a test jig (Bonding Assembly, Ultradent, South Jordan, UT, USA) for placement of the repair restoration. A tube with an inner diameter of 2.39 mm and 3 mm in height were used for composite placement.

Specimens were repaired with the same resin composite material with two increments. Each layer was polymerized for 20 s from the top surface. After removal from the sample holder, specimens were circumferentially polymerized for a total of 40 s. After polymerization, all specimens were stored in distilled water at 37°C for 24 h before bond strength test. Shear bond strength (SBS) values of each specimen after repair was measured by a testing device (Shear Bond Tester, Bisco, Schaumburg, USA) equipped with a non-bevelled chisel-shaped metallic tip with a crosshead speed of 1.0 mm/min (Figure 1).

Table 3. Application protocols of the adhesive systems.

Adhesive	Application Protocol
Clearfil SE Bond	Apply the primer to the surface with a brush, wait for 20s, air dry. Apply the bond to the surface with a brush, air dry. Light cure for 10s.
XP Bond	Apply the phosphoric acid gel for 15s, rinse with water for 15s, air dry lightly. Apply the adhesive to the surface, wait for 20s, air dry. Light cure for 20s.
I Bond Self-Etch	Apply with a brush by light rubbing for 20s. Air dry for 5s. Light cure for 20s.

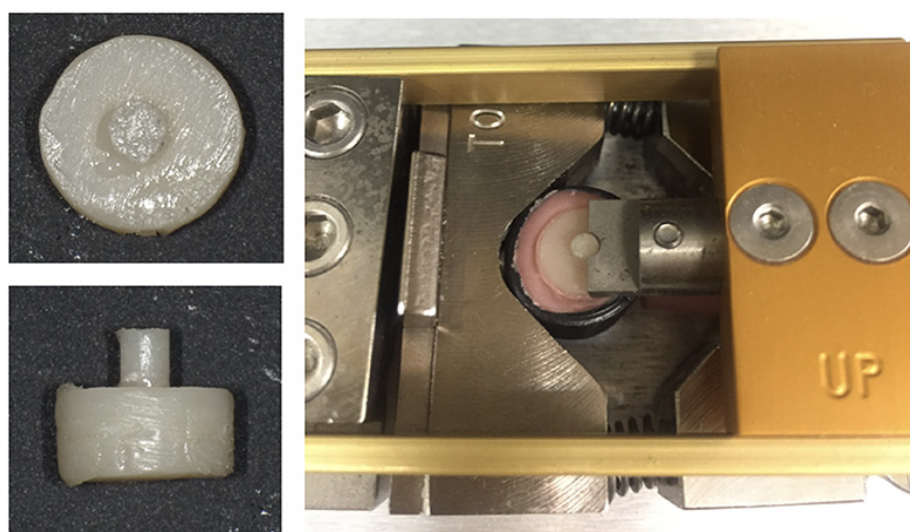


Figure 1. Images of the composite sample prepared for the shear bond strength test.

Statistical analysis

GraphPad Prism 6 statistical analysis software (Prism 6, GraphPad Software, CA, USA) was used in this study. As the data was completely numerical, distribution characteristics and equality of variances were checked with Kruskal-Wallis and Levene's tests, respectively. Since the normality assumptions have been met, two-way analysis of variance (ANOVA) test was selected for comparing multiple groups. Tukey's honestly significant difference (HSD) post-hoc test was used for subsequent pairwise comparisons. Confidence interval was set to 95% and probability (p) values less than 0.05 were considered as statistically significant.

Results

Two-way ANOVA results revealed no interaction between the adhesive type and thermal aging. However, adhesive type and thermal aging significantly affected SBS values ($p < 0.001$). The obtained SBS values and their respective standard deviations are shown in Table 4. No statistically significant difference was found between 2TE-TA and 2SE-TA. Bond strengths of 2TE-TA and 2SE-TA were significantly lower than their corresponding control groups (2TE-control, 2SE-control) ($p < 0.0001$ and $p = 0.005$ respectively). Bond strength values of 1SE-TA were not statistically different than 1SE-control.

Table 4. Shear bond strengths (MPa) and their standard deviations (Means sharing a letter are not significantly different, uppercase letters compare means in the same row, Lowercase letters compare means in the same column).

	2SE (SE Bond)	2TE (XP Bond)	1SE (I Bond)
Control	18.19 ± 3.20 Aa	19.15 ± 5.10 Aa	8.93 ± 1.90 Ba
Thermal Aging (TA)	13.66 ± 3.55 Ab	12.78 ± 3.46 Ab	5.63 ± 1.56 Ba

Discussion

This study evaluated the initial (24 h) repair bond strengths of thermally aged resin composite *in vitro* repaired with three different adhesive systems; two-step total-etch, two-step self-etch and one-step self-etch systems. Type of the adhesive significantly

affected repair bond strength of the aged resin composite ($p < 0.05$). Therefore, the null hypothesis postulating that the type of adhesive would not have any effect on repair bond strength of the resin composite was rejected. The two-step self-etch and two-step total-etch adhesives performed better when compared with the one-step self-etch adhesive in both

aged and non-aged groups ($p < 0.05$). When there is a need for repair on the composite restoration, macro-mechanical, micro-mechanical retention and chemical adhesion are equally important aspects to consider. It is possible to obtain a chemical bond between the old and fresh composite (20), since within two weeks free radicals inside the old resin composite may still be available for a chemical reaction (21). In this study, aging period was roughly equivalent to 5 years of clinical service (22), which was much higher than 2 weeks; thus, the possibility of the chemical bond between the composites was eliminated. Macro-mechanical retention can be obtained by creating retention holes and undercuts. Roughening the surface of the old composite with a coarse diamond bur, may provide better macro-mechanical retention (7-9, 14, 23). Most significant mechanism acting on the bond between the old and the fresh composite is micro-mechanical interlocking (24, 25). Use of a readily available diamond bur is a simple method to apply. However, the use of diamond burs with varying grit sizes does not yield different bond strength values (6, 8, 12, 26). Therefore, in the present study, a coarse diamond bur (150 μm grit size) was used to aid in micro-mechanical interlocking, as suggested by other studies (4, 5, 7, 9).

Use of an intermediate adhesive agent also plays a significant role in the repair process of resin composite restorations (9, 19). Bond strength of the repair restoration is significantly improved when the adhesives are applied after surface pretreatments (9, 19, 27). In this study, three different adhesive systems were evaluated, a two-step total-etch adhesive, a two-step self-etch adhesive and a one-step self-etch adhesive. Two-step total-etch adhesive, XP Bond, showed similar repair bond strength values in both aged and non-aged composite groups when compared with the two-step self-etch adhesive, the Clearfil SE Bond. Similar results have been previously reported (28). The one-step self-etch adhesive, I-Bond, showed significantly lower repair bond strength values in both aged and non-aged composite groups when compared to other adhesive systems which is in agreement with the findings reported by Teixeira *et al.* (29).

Resin composite restorations are constantly exposed to oral fluids and may eventually absorb water and swell. This may lead to degradation of the resin and leaching of the filler particles (30). Since self-etch adhesives are hydrophilic, self-etching primers may therefore effectively bind to the surface of the old composite through interaction of the phosphate groups

of the adhesive systems (9). Similar to Cavalcanti *et al.* (8); in the present study, repair of the aged composite with Clearfil SE Bond, which contains a functional monomer, 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP), showed higher bond strength values than those of other bonding agents. This may be attributed to the wetting ability of 10-MDP applied on composites. However, further research is still needed to test this hypothesis.

The use of total-etch systems requires etching with a phosphoric acid. Acid etching helps removal of the debris on the surface of the restoration to be repaired and could expose the underlying surface and filler (31). This would increase the surface area (23) and wettability (32) of the resin composite to be repaired. For the self-etch adhesives similar effect can be achieved by the acidic primer of the system (8). Although composites are mostly hydrophobic, they absorb some water during service (29). The ability of the adhesive systems to penetrate into the aged composite surface depends on their chemical affinity and the degree of hydration of the composites (29, 33). Application procedure of two-step self-etch adhesives involves etching of the surface with a hydrophilic self-etching primer followed by covering with a rather hydrophobic adhesive layer. One-step adhesives are generally more hydrophilic than their two-step counterparts and contain highly acidic monomers (34). Absence of the additional adhesive layer may therefore negatively influence their bonding ability to composite. Repair bond strengths of the aged composites were significantly lower than those of non-aged composites for the two-step self-etch and two-step total-etch adhesives. For the one-step self-etch adhesive, bond strength was not statistically different regardless of aging. However, bond strength values of the one-step self-etch adhesive (control, 8.93 ± 1.90 ; thermal aging, 5.63 ± 1.56) were significantly lower when compared to other adhesive systems and was below the clinically acceptable level which was reported to be a minimum of 18 MPa or between 20-25 MPa (19, 27, 35). All of the failure modes observed in this study was of adhesive type and this may indicate that the bond strength may be weaker than the cohesive strength of the resin composite used, regardless of aging, although cohesive strength of the resin composite was not measured. It was reported that the repair bond strength of the resin composites vary between 20% and 96% of the cohesive strength of the intact composite (9, 28, 36, 37). It is still not possible to offer a universal repair protocol for all composites,

since many of the studies have found different results (6, 19, 29, 38, 39). However, one should choose a protocol which is safer to use intraorally. Surface modification techniques such as sandblasting and application of hydrofluoric acid may have side effects (11). Therefore, the use of an intermediate adhesive system after pretreating the composite surface with a coarse diamond bur could be a method which is safer and easier to perform.

Conclusion

Within the limitations of this experimental study, repair of aged composites using a two-step self-etch or a two-step total-etch adhesive demonstrated higher bond strength values than repair with a one-step self-etch adhesive. Further studies are needed to assess the performance of these adhesive with different resin composites and in longer periods of aging.

Source of funding

None declared.

Conflict of interest

None declared.

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