Repair Bond Strength of Aged Resin Composite Using Sliane-containing Universal Adhesive System

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Abstract

To evaluate the effect of a silane-containing universal adhesive system to repair bond strength of aged resin composite, clear self-cured epoxy resin was poured into the prepared PVC and Flitek™ Z350 XT shade A1 was packed into 45 PVC tubes. The 45 specimens were thermocycled for 5,000 cycles between 5 and 55 °C. The specimens were ground, then, treated with Scotchbond™ etchant and divided into 3 groups. Group 1 was treated with Clearfil SE Bond (n=15). Group 2 was treated with Clearfil Ceramic Primer and Clearfil SE Bond (n=15). Group 3 was treated with Single Bond™ Universal Adhesive (n=15). Flitek™ Z350 XT shade A4 was packed in the split stainless steel mold to bond with the prepared specimens. All specimens were subjected to shear bond strength testing and record the result. The mean shear bond strength values of group 1, group 2, and group 3 were 10.21, 15.86, and 14.14 MPa, respectively. Group 2 and 3 showed a statistically significant higher shear bond strength than group 1 (Shapiro-Wilk test, One-way ANOVA and Turkey's test). However, there was no significant difference in shear bond strength between group 2 and 3. Silanization can improve the repair bond strength of aged resin composite. The application of a silane-containing universal adhesive was as effective as the use of silane and adhesive combination tested. The use of this material can be applied for nanofilled composite repair.

Keywords: *aged resin composite, repair bond strength, repair resin composite, shear bond strength, universal adhesive system*

1. Introduction

The common failures of resin composite restoration are poor marginal integrity, fracture, discoloration, and secondary caries. The treatment of choices would be replacement or repair. Resin composite replacement procedure is to remove all of the restorations and surrounding sound tooth structure. Even though the composite replacement is an invasive treatment, there is nothing to concern about the bond of the aged and new restoration. The preparation of resin composite restoration is less invasive treatment, however, there are still some questions about bonding between aged and new restoration (Blum et al., 2014).

The mechanism of bonding of composite repair is mechanical and chemical adhesion. The mechanical adhesion is the interlocking between adhesive and irregularities on the surface of substrates. To create a mechanical interlocking, roughening a surface of resin composite with diamond bur, carbide bur, silicon carbide paper or air abrasion with 50 μ aluminum oxide particles is recommended. The chemical adhesion is bonding between resin matrix and hydroxyl groups on silica filler surface by using silane coupling agents (Fornazari et al., 2017; Heymann et al., 2014). The methacrylate group of silane forms covalent bonds with repaired resin composite when it is polymerized. Silane also enhances the wetting ability of the adhesive over the irregularities created by surface roughening (Hemadri et al., 2014).

Reparation of resin composite often presents a different challenge; while there is no oxygen-inhibited layer if any, few unreacted double bonds remain in the aged composites for bonding to the new composite. Thus, the potential for chemical bonding between aged and new composite layers decreases over time. Improving the bond strength between new and aged composite requires to increase surface roughness to promote mechanical interlocking and coating with a silane coupling agent to enhance the surface wetting ability and produce chemical bonding (Nihei, 2016; Blum, 2014).

Recently, the universal adhesive was introduced to simplify clinical steps and for bonding to various substrates. The universal adhesive is a single-bottle of an adhesive system containing acidic primers and resin adhesives with or without silane coupling agents. The universal adhesive system can be used in total-etch, self-etch, or selective-etch mode depending on the specific clinical situation and personal preferences of the operator. Additionally, universal adhesives can be used for direct and indirect restorations and are compatible with self-cure, light-cure, and dual-cure resin-based cement. It is further stated that universal adhesives can be used not only to bond to dentin and enamel, but also be used on various substrates such as zirconia, noble and non-precious metals, resin composites, and various silica-based ceramics. In principle, this would enable bonding to these surfaces without the need for dedicated and separately placed primers such as silane and various products marketed as metal and zirconia primers (Alex, 2015).

One of the compositions of the universal adhesive system is a silane coupling agent which can enhance the chemical bond of composite repair. Moreover, minimal steps can reduce the clinical errors of the bonding procedure. Therefore, the repaired bond strength of composite through a silane-containing universal adhesive system is interesting. Thus, the main objective of this study was to evaluate the repair bond strength of aged resin composite using the Universal adhesive system (Wendler et al., 2016).

2. Objective

To evaluate the effect of a silane-containing universal adhesive system to repair bond strength of aged resin composite.

3. Materials and Methods

3.1. Specimen preparation

Cylindrical sample made of polyvinyl chloride tube (PVC) size 18(1/2”) with a diameter of 22 mm, 2.0-mm thickness, and 1.5-inch height was used as a base mold. The inner surface of the PVC was ground to increase retention. The PVC tube was placed into the cylindrical stainless steel mold. This mold was used for creating a cylindrical slot in the center with a diameter of 5 mm and 5-mm height. Clear self-cured epoxy resin was poured into the prepared PVC. Specimen preparation is shown in Figure 1.

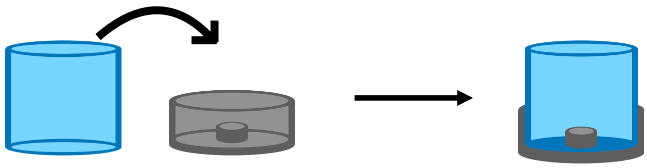

Figure 1 Preparation of specimen by putting PVC tube into stainless steel mold to create a cylindrical slot before pouring self-cured epoxy resin

Resin composite shade A1 (Flitek^{TM} Z350 XT, 3M ESPE, St Paul, MN, USA) was packed into 45 prepared PVC specimens using the incremental technique, 1 mm for the first increment and 2 mm for the second and third increment. Light curing using LED light curing unit (Demi^{TM} plus, Kerr Corp, Orange, CA, USA) for 40 seconds was done after each increment. Before initiation of light curing of the third increment, the surface of each composite specimen was flattened and leveled by covering with a mylar strip and compressing with a glass slab. The top surface of the resin composite was polished by NANO 2000 polishing machine (PACE TECHNOLOGY, USA) and equipped with 100-grit silicon carbide waterproof abrasive paper (TOA TDCC, Samutprakarn, Thailand) for 3 minutes. Composite preparation on PVC specimen is shown in Figure 2.

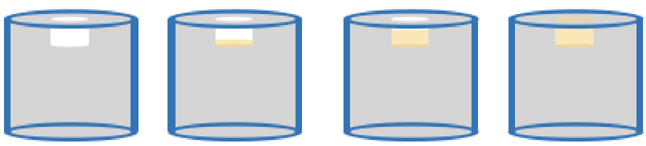

Figure 2 Resin composite shade A1 was packed into prepared PVC tube using incremental technique,1 mm for first increment, 2 mm for second and third increment.

3.2. Aging procedure

The prepared specimens were thermocycled for 5,000 cycles between 5 and 55 °C with a dwell time of 30 seconds and a transfer time of 15 seconds (TC301, King Mongkut's Institute of Technology Ladkrabang, Thailand) to accelerate composite aging for a half year period.

3.3. Surface treatment method

The 45 prepared specimens were ground using NANO2000 polishing machine (PACE TECHNOLOGY, USA) and equipped with 500-grit silicon carbide waterproof abrasive paper (TOA TDCC, Samutprakarn, Thailand) for 5 minutes to create micromechanical retention. The surface of all specimens was masked with plastic tape (OASIS® Floral Products, Australia) with a circular aperture of 2-mm diameter at the center and randomly assigned into 3 groups as shown in Table 1 according to the surface treatment. The composition of resin composite, silane coupling agent, and resin adhesive used in this study are shown in Table 2.

Table 1 Distribution of groups according to adhesives application

Group	Surface treatment
1	Scotchbond™ etchant (3M ESPE, Germany) and Clearfil SE Bond (Kuraray Noritake Dental Inc., USA)
2	Scotchbond™ etchant (3M ESPE, Germany), Clearfil Ceramic Primer (Kuraray Noritake Dental Inc., USA) and Clearfil SE Bond (Kuraray Noritake Dental Inc., USA)
3	Scotchbond™ etchant (3M ESPE, Germany) and Single Bond™ Universal Adhesive (3M ESPE, Germany)

Table 2 The composition of resin composite, silane coupling agent and resin adhesive

Material	Manufacturer	Chemical composition
Scotchbond™ etchant	3M ESPE, Germany	10% maleic and 35% phosphoric acid
Clearfil Ceramic Primer	Kuraray Noritake Dental Inc., USA	MDP, 3-Methacryloxypropyl trimethoxy silane, Ethanol
Clearfil SE Bond	Kuraray Noritake Dental Inc., USA	MDP, HEMA, Dimethacrylate monomer, Microfiller, Photoinitiator
Single Bond™ Universal Adhesive	3M ESPE, Germany	MDP phosphate monomer, HEMA, Dimethacrylate resins, Vitrebond copolymer, Filler, Ethanol, Water, Initiator, Silane
Filtek™ Z350 XT	3M ESPE, USA	Bis-GMA, UDMA, TEGDMA, Bis-EMA, non- aggregated 4 to 11 nm zirconia, non-aggregated 20 nm silica and aggregated zirconia/silica cluster filler (63.3 vol%)

Group 1 was treated with Scotchbond™ etchant (3M ESPE, Germany) and Clearfil SE Bond (Kuraray Noritake Dental Inc., USA) as a negative control group. First, Scotchbond™ etchant was applied to prepare resin composite surface for 30 seconds, then rinsed for 10 seconds and dried using a brief gentle air stream. Then, Clearfil SE Bond Adhesive was applied and light-cured for 10 seconds following the manufacturer's instruction.

Group 2 was treated with Scotchbond™ etchant (3M ESPE, Germany), Clearfil Ceramic Primer (Kuraray Noritake Dental Inc., USA) and Clearfil SE Bond (Kuraray Noritake Dental Inc., USA) as a positive control group. First, Scotchbond™ etchant was applied to the clean resin composite surface for 30 seconds, then rinsed for 10 seconds and dried using a brief gentle air stream. Second, Clearfil Ceramic Primer was applied for 5 seconds and air dried. Then, Clearfil SE Bond Adhesive was applied and light-cured for 10 seconds following the manufacturer's instruction.

Group 3 was treated with Scotchbond™ etchant (3M ESPE, Germany) and Single Bond™ Universal Adhesive (3M ESPE, Germany) as a test group. First, Scotchbond™ etchant was applied to the clean resin composite surface for 30 seconds, then rinsed for 10 seconds and dried using a brief gentle air stream. Then, Single Bond™ Universal Adhesive was applied and dried. Then, it was light-cured for 10 seconds following the manufacturer's instruction.

3.4. Resin composite repair

After placing the split stainless steel mold (3 mm in diameter and 2 mm in height) on the prepared resin composite surface, the resin composite shade A4 (Flitek™ Z350 XT, 3M ESPE, USA) was packed and light-cured using LED light curing unit (Demi™ plus, Kerr Corp, Orange, CA, USA) for 40 seconds.

3.5. Shear bond strength testing

All specimens' shear bond strength was measured using the Universal testing machine (EZ-S, SHIMADZU, Japan). The load was applied to the interface at a cross-head speed of 1 mm/min until failure and the stress-strain curve was analyzed with the machine's software program. The results were recorded. The procedure of shear bond strength testing is shown in Figure 3.

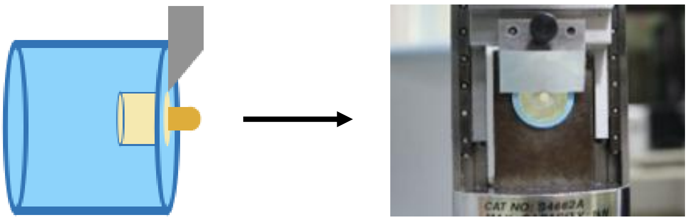

Figure 3 Apply shear force to the interface between aged and new resin composite and record the results calculated by the machine's software program.

3.6. Mode of failure

The fracture surface of all specimens was examined at X45 under a stereomicroscope (SZ 61, Olympus, Japan). The failure mode was classified as one of three categories; adhesive, cohesive, and mixed failure. Adhesive failure is the failure at the interface of the aged and new resin composite. Cohesive failure is the failure within aged or new resin composite. Mixed failure is a combination of adhesive and cohesive failure.

4. Results

The shear bond strength data showed a normal distribution (Shapiro-Wilk test). A significant interaction between groups was observed on the repair bond strength (one-way ANOVA, $p<0.05$). The mean shear bond strength of the 3 groups are shown in Table 3. The mean shear bond strength values of group 1, group 2 and group 3 were 10.21, 15.86 and 14.14 MPa, respectively. Group 2 showed a statistically significant higher shear bond strength than Group 1 ($p = 0.000$). Group 3 showed a statistically significant higher shear bond strength than Group 1 ($p = 0.001$). However, there was no significant difference in shear bond strength between group 2 and 3 ($p = 0.186$).

Table 3 The mean shear bond strength of each group (MPa)

Group	Mean Bond Strength (MPa) ± SD
Group 1 Scotchbond™ etchant + Clearfil SE Bond	10.21 ± 2.21[a]
Group 2 Scotchbond™ etchant + Clearfil Ceramic Primer + Clearfil SE Bond	15.86 ± 2.87[b]
Group 3 Scotchbond™ etchant + Single Bond™ Universal Adhesive	14.14 ± 2.80[b]

Group with the same letter do not show statistically significant differences ($p>0.05$)

Under the stereomicroscope with a magnification of 45X, 53.33% of the group 1 samples are failed adhesively and 46.67% are mixed adhesive. While group 2 and 3 samples showed an adhesive failure of 33.33% and a mixed mode of failure 66.67% as shown in Figure 4.

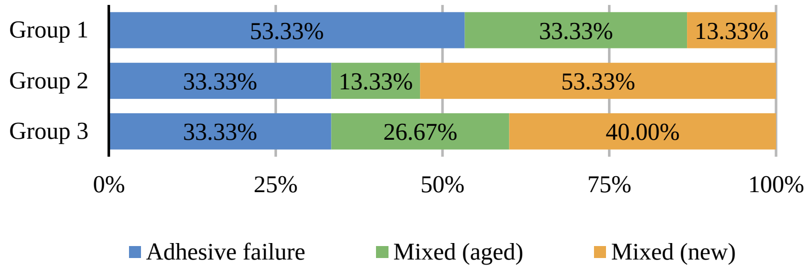

Figure 4 Mode of failure of each group; adhesive failure (blue color), mixed failure found in aged composite (green color) and mixed failure found in new composite (yellow color).

5. Discussion

Replacement of restorations may result in higher esthetics, in contrast, it may cause further destruction of sound tooth structure, pulpal injury, time-consuming and high expense. As the demands of restorative materials that combine esthetic and mechanical properties, resin composite filling materials have been increasingly used as direct restoration. Therefore, the repair of composite restorations would be a more suitable option since it saves time consumed, lowers cost, and preserves tooth structure compared to a replacement. Unfortunately, there are some topics to concern about the junction between aged and new resin composite. In general, the bond strength between aged and new composite depends on many factors such as aging conditions, time passed since the restoration of a tooth, type of composite, surface roughness, and type of bonding agent being used (Ghavam et al., 2018).

The aging process is one of the important factors that affect the longevity of a repaired restorations. Several techniques have been used for aging a composite including boiling, thermocycling, and storage in citric acid, sodium chloride, artificial saliva, and distilled water. Despite being the most common type of storage, it has been reported that water storage can cause water sorption by the resin matrix and subsequent hydrolysis and release of filler particles (Alex, 2015). In the thermocycling process, water sorption can decrease the structural and physical properties of composite resins and also cause aging of material. (Ghavam et al., 2018; Morresi et al., 2014). In this study, 5,000 cycles were performed between 5 to 55°C with a dwell time of 30 and 15 seconds corresponding to accelerate composite aging for a half year period.

Surface treatment of the reparation of the aged composite is needed for two purposes; to remove the superficial layer altered by the saliva, exposing a clean, higher energy composite surface and to increase the surface area through the creation of surface irregularities. Bonding between aged and new composite may occur by two distinct mechanisms: (1) chemical bonding with the organic matrix and the exposed filler particles, (2) micromechanical retention to the treated surface (Al smar, 2017). In this study, 500-grit silicon carbide waterproof abrasive paper was used to imitate micromechanical preparation and compared with a superfine diamond bur used in a clinical situation. Furthermore, the coarser the abrasive paper, the more the influence of micromechanical retention on the bond strength. Therefore, 500-grit silicon carbide waterproof abrasive paper was used to minimize the potential micro-mechanical interlocking. In this study, different surface treatments were performed to measure a chemical adhesion of bonding interface between aged and new composite.

Almost every type of organic polymer is compatible with silane coupling agents. The silane may be applied to the substrate as a pretreatment or, in many systems, the silane may be added to the resin where it migrates to the substrate during normal mixing and application procedures. The application of silane coupling agents to promote bonding has led to improving the physical properties of composite materials. Silane treatment during resin repair procedures basically aims to promote chemical bond by forming siloxane bonds between silicate-containing filler particles exposed on the repaired surface and the resin matrix of the fresh resin layer (Altinci et al., 2017; Lung et al, 2012). It was stated that surface wettability is also increased, which can improve the micro-mechanical bonding in the repair interface (Nihei, 2016; Blum, 2014).

Shear bond strength testing leads to non-homogeneous stress distribution in the bonded interface, which may eventually cause erroneous interpretation of the results due to a failure occurring in the substrate rather than the adhesive zone. This research study investigated a shear bond strength of repairing aged resin composite with resin composite. Repair bond strength was tested immediately after repairing of aged resin composite. The study of repair bond strength of aged repaired composite with universal adhesive has not been reported.

When conventional silane or silane-containing universal adhesive was applied for bonding procedure, the bond strength was higher than when the only adhesive was applied. Hydrolyzable alkoxy groups of silane coupling agent react with hydroxyl groups in the silica filler surface of resin composite forming oxygen bridges. Silane will be used in case of repairing the aged composite which contains silica as a filler (Fornazari et al., 2017). The bifunctional molecule of the silane coupling agent bonds the inorganic filler particles of the resin with the methacrylate of the adhesive system, and increases the wettability of the adhesive system to infiltrate into the irregularities of the treated composite surface (Al smar, 2017). In any case, it is clearly stated in the literature that the use of intermediate bonding agents and silanization could enhance bonding when repairing resin composite restorations. In this study, the combined treatment of surface roughness, separate application of silane and adhesive were the most efficient surface treatment method in our study. The result of this study confirmed the importance of silanization in composite repairs.

According to the surface of bonding, the researcher could not specify the accurate location of the bonding interface. Therefore, the new composite diameter should be more than 2 mm. to compensate for the error of the bonding area.

The composition of resin composite consists of a resin matrix and inorganic filler (Zimmerli et al., 2010). In this study, nanofilled resin composite (Filtek^{TM} Z350 XT) was used due to its chemical composition. Inorganic fillers are non-aggregated 20 nm silica and aggregated zirconia/silica cluster fillers. Organic resin matrices are Bis-GMA, UDMA, TEGDMA, Bis-EMA. When conventional silane or silane-containing universal adhesive was applied before the bonding, the bond strength was higher than only the bonding was applied. These results are probably due to increased wettability and consequent good chemical bond between resin matrix of new composite and exposed particles in the original resin matrix (Fornazari et al., 2017).

According to the mode of failure analysis, most of the negative control group failed adhesively due to only mechanical retention from surface treatment, while the other test groups showed lower adhesive failure due to chemical retention from applying of silane.

Altinci et al reported that combined treatment of silane along with universal adhesive provided the optimum repair bond strength. Yoshihara et al found Silanol-characteristic peaks were only detected for the immediately prepared adhesive/silane mixture. The result of this study evaluated the immediate repair bond strength which was correlated to previous studies. However, the study of a delayed repair bond strength of aged repaired resin composite with silane-containing universal adhesive has not been reported. Therefore, further study to evaluate bond stability of silane-containing universal adhesive used for repairing aged resin composite should be considered.

6. Conclusion

Silanization can improve the repair bond strength of aged resin composite. The application of a silane-containing universal adhesive was as effective as the use of silane and adhesive combination tested. The use of this material can be applied for nanofilled composite repair.

7. References

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