Original Article

Influence of commercial adhesive with/without silane on the bond strength of resin-based composite repaired within twenty-four hours

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Abstract Background/purpose: It is not clear whether the ground surface of resin-based composite (RBC) polymerized requires the application of an adhesive with/without a silane to improve bond strength. This study investigated the bond strength of RBC repaired within 24 h via the application of adhesive with/without a silane.  
Materials and methods: Seventy RBC blocks were prepared and assigned to either 0 or 24 h repair stage. Each stage was divided into seven groups: a control group with no surface roughening or applied adhesive, a surface-roughened group with no applied adhesive, two surface-roughened groups treated with a G-aenial Bond adhesive and a BeautiBond Multi adhesive, two surface-roughened groups treated with the previously-mentioned adhesives as well as silane coupling agents, and one group treated with a Single Bond Universal silane-containing adhesive. Microtensile bond strength (μTBS) measurements were performed after the repaired RBC blocks of each group (n = 5) had been immersed in a 37 °C water bath for 24 h. The failure mode of each sample was determined, and the data were analyzed via one-way analysis of variance and Dunnett’s test (p ≤ 0.05).  
Results: Regardless of the repair stage, the μTBS values of the adhesive-only and silane-adhesive groups did not differ significantly from those of the control group (p > 0.05). Only the no-adhesive groups exhibited a significantly time-dependent increase in adhesive failure rate.

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Introduction

Direct resin-based composite (RBC) restoration is a widely used treatment with many advantages, including the preservation of tooth structure, high esthetic appearance, and rapid and time-saving. Even though long-term studies have indicated that amalgam exhibits better longevity than an RBC, the tooth-coloration characteristics of RBCs and the superior bonding ability of adhesives have enabled the application of RBCs not only to anterior teeth, but also posterior teeth or stress-bearing areas with a satisfactory long-term survival rate in daily practice.

In clinics, filling defects, such as insufficient filling, voids, excessive reduction, spacing at the proximal surface, or color not matching the surrounding tooth substrate, may be detected immediately after the filling or finishing procedures on the same day or later after a short period. The ideal solution for such problems is to remove the RBC restoration completely and refill with RBC to obtain satisfactory results. Such a complete removal procedure is, however, time-consuming and detrimental to the tooth substrate. The RBC repair method, which is a technique performed by adding RBCs directly onto the previous RBC restoration or removing the defective portion of the previous RBC restoration and refilling, is considered an appropriate means to replace the traditional complete-replacement method and also adheres to the concept of minimal intervention.

It is widely known that the superficial layer of either RBC or an adhesive is a poorly polymerized resin-rich layer referred to as the oxygen-inhibited layer (OIL) that originates from exposure of the resin matrix to oxygen during polymerization. Several studies have mentioned that the existence of the OIL may promote the adhesion of newly added RBC onto a previous RBC restoration. However, it has also been reported that the bond strength of an RBC repair had no correlation with the existence of the OIL. Other studies have indicated that the immediate application of an adhesive can effectively improve the bonding ability of new RBC to existing RBC during the early repair period. Silane has been reported to improve wetting ability of RBCs and to react with the silica ingredients of fillers to form siloxane bonds during RBC repair. Its application is therefore recommended to provoke a chemical reaction with the filler of an existing RBC and promote the adhesion of new RBC. However, other studies have reported no improvements in bond strength after the application of silane.

Adhesive application has been simplified to a one-step procedure. However, it is still not clear whether the application of silane on the roughened surface of RBC polymerized for 24 h is necessary in this procedure to restore the bonding capacity to its original level. Surface roughening via sandblasting and/or the application of hydrofluoric acid is a frequently used method for generating irregularities and increasing the bonding area to achieve better bonding results. However, these methods are hazardous and difficult to perform intraorally despite their outstanding in vitro results.

This study investigated whether surface treatments involving the use of an adhesive with or without a silane, or a silane-containing adhesive, on the ground surface of RBC polymerized within 24 h, had any reinforcing effect on the repair bond strength. The null hypothesis of the present study was that RBC repair on RBC polymerized within 24 h would require no additional applications of adhesive or a combination of adhesive and a silane to restore the bond strength back to its original ungrounded level.

Materials and methods

Two different shades of RBC (AO2 and A2 shades, Beautifil II, Shofu, Kyoto, Japan) were used to distinguish the fracture site. Restored RBC blocks were prepared by inserting the opaque RBC (A2O) into a mold (4 mm × 4 mm × 4 mm) in two layers, with each layer of thickness 2 mm and sequentially light-cured for 40 s with a light-emitting diode (LED) light-curing machine (DEMI Plus, Kerr, Middleton, WI, USA). The second layer was covered with a polyester matrix strip and a glass slide to ensure that the upper and lower surfaces were parallel; it was also light-cured for 40 s. The LED light-curing machine operated with a light intensity of at least 1000 mW/cm². Seventy AO2-RBC blocks were fabricated and assigned randomly into two repair stages: (a) 0 h repair stage: immediate repair and (b) 24 h repair stage: repair after immersing the RBC block in a 37°C water bath for 24 h. The blocks in each repair stage were further divided into seven groups (n = 5), consisting of six roughened groups and one unroughened group, as explained below.

In the 0 h repair stage, the roughened blocks were prepared by removing 0.2 mm of the outermost layer of each AO2-RBC block with 600-grit sandpaper and cleaning the blocks ultrasonically for 5 min. The roughened AO2-RBC blocks were divided into six groups: (1) R-NA group: no adhesive and/or silane application; (2) R-GB group: adhesive (Gaenial Bond, GC, Tokyo, Japan) application for 10 s, air-blowing for 5 s, and silane application; (3) R-GB group: silane (Ceramic primer, GC, Tokyo, Japan) application for 10 s, air-blowing for 5 s, and silane application; (4) R-GB group: silane (Ceramic primer, GC, Tokyo, Japan) application for 10 s, air-blowing for 5 s, and silane application; (5) R-GB group: silane (Ceramic primer, GC, Tokyo, Japan) application for 10 s, air-blowing for 5 s, and silane application; (6) R-GB group: silane (Ceramic primer, GC, Tokyo, Japan) application for 10 s, air-blowing for 5 s, and silane application; (7) R-GB group: silane (Ceramic primer, GC, Tokyo, Japan) application for 10 s, air-blowing for 5 s, and silane application.

Conclusion: Our results suggest that the application of adhesives either with or without silane can significantly increase the bond strength of repairs to RBCs polymerized within 24 h.
GC, Tokyo, Japan) application for 60 s, air-blowing for 5 s, followed by the same procedures as for the R-GB group; (4) R-
BM group: adhesive (BeautiBond Multi, Shofu, Kyoto, Japan) application for 10 s, air-blowing for 5 s, and light-curing for 10 s; (5) R-BMP group: adhesive (BeautiBond Multi PR
Plus, Shofu, Kyoto, Japan) application for 10 s and silane (BeautiBond Multi PR
Plus, Shofu, Kyoto, Japan) application for 5 s, air-blowing for 5 s, and light-curing for 10 s; (6) R-SBU group: adhesive (Sin-
gleBond Universal, 3M-ESPE, Neuss, Germany) application for 20 s, air-blowing for 5 s, and light-curing for 10 s. A2-shade RBC was then applied onto the above AO2-RBC blocks using the
same manner as for the preparation of AO2-RBC blocks. The seventh experimental group with no roughening and no
application of adhesive or silane (NR-NA) was prepared by
adding the A2-shade RBC directly onto the freshly prepared
AO2-RBC blocks, served as the control group. These blocks
served as the 0 h repair RBC blocks.

In the 24 h repair stage, the 24 h-37°C water-immersed AO2-RBC blocks were also subdivided into seven subgroups (including an unroughened control group). Except the control
group, the 0.2 mm thickness of the superficial layer of AO2-
RBC blocks was reduced in the same manner as described for
the 0 h repair stage. Each group was treated in the same
manner as the respective groups of the 0 h repair stage. Finally, the bonded RBC blocks of the 24 h repair stage were immersed in a 37°C water bath for 24 h before the Micro-
tensile bond strength (μTBS) measurements. The outmost treated surface of AO2-RBC block in each group of 0 h repair
stage, ready for bonding with A2 RBC, were also prepared and
digen sputter (E-1045, Hitachi, Tokyo, Japan) coated with
platinum to observe the morphological diversity by a scan-
ing electron microscope (SU8010, Hitachi, Tokyo, Japan).

Microtensile bond strength measurements

After 24 h of water immersion, each bonded RBC block of the 0 and 24 h repair stages was sectioned perpendicularly
to the bonded surface into eight non-trimming (NT) stick specimens with a cross section of 1 mm \times 1 mm using a low-
speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA)
under water cooling. Those NT stick specimens of the same
group were subjected for the μTBS test. The NT stick
specimens of each group (n = 40) were fixed using cyano-
acrylate (Zapit, Dental Venture of America, Corona, CA,
USA) to measure the μTBS using a tensile test machine (AI-
3000, Gotech, Taichung, Taiwan) at a crosshead speed of
1.0 mm/s, as outlined by Chen et al.27

Failure mode

The fractured surface of each specimen in both the 0 and
24 h repair stages was examined using a stereomicroscope
(20X magnification, SMZ800N, Nikon, Tokyo, Japan) to
determine whether the fracture site was located at the
interface of the bonding surface (denoted as A for “adhe-
sive failure”), in any side of the RBC (denoted as C for
“cohesive failure”), or was partially adhesive and partially
cohesive (denoted as M for “mixed failure”).

Statistical analysis

One-way ANOVA followed by Dunnett’s tests at a confidence
level of 95% were carried out to analyze the measured μTBS
data to determine whether any significant difference existed
for the various parameters of the polymerization stage, the
application of an adhesive, or the combination of an adhesive
and a silane coupling agent. The statistical analyses were
performed using SPSS (version 19).

Results

The μTBS data of each group for the 0 and 24 h repair stages
are shown in Fig. 1 and the statistical results of all groups
are shown in Table 2.

### Table 1  Resin-based composites, silane coupling agents, and adhesives used.

| Materials Brands | Components | Lot number | Manufacturer |
|------------------|------------|------------|--------------|
| Resin-based composite | Beautifil II AO2 shade A2 shade | Bis-GMA, TEGDMA, UDMA, filler | A: 1309031 B: 1309051 | GC |
| Silane coupling agent | Ceramic primer | Primer A: Silane, ethanol; Primer B: methacryloxyalkyl acid phosphate, ethanol | 081205 | Shofu |
| Adhesive | BeautiBond Multi PR Plus | 4-MET, Phosphoric acid ester monomer, Dimethacrylate monomer, Distilled water, Acetone | 1211221 | GC |
| | Gaenial Bond | Carboxylic acid monomer, Phosphonic acid monomer (6-MHPA), Water, Acetone, Polymeric monomer, hotoinitiator, Others | 021211 | Shofu |
| | SingleBond Universal | MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Ethanol, Water, Silane, Initiator | 490325 | 3M ESPE |

The monomers having bifunctional group in each adhesive are shown in bold.
The 0 h NR-NA control group exhibited the highest mTBS value (97.5 ± 3.9 MPa) and the 24 h R-NA group exhibited the lowest mTBS value (52.2 ± 11.7 MPa). In the 0 h repair stage, the mean mTBS value for each group decreased in the following order: NR-NA group (97.5 MPa) > R-BMP group (95.3 MPa) > R-SBU group (93.6 MPa) > R-PGB group (92.0 MPa) > R-BM group (88.6 MPa) > R-NA group (75.0 MPa). In the 24 h repair stage, the mean mTBS value for each group decreased in the following order: R-BMP group (92.2 MPa) > R-SBU group (92.1 MPa) > R-PGB group (90.5 MPa) > R-BM group (89.2 MPa) > R-GB group (88.8 MPa) > NR-NA group (84.4 MPa) > R-NA group (52.2 MPa). The R-NA group exhibited statistical differences from the other groups in the same repair stage (p < 0.05) and the mTBS value significantly lowered to 76% or 62% of that of the control group in each respective repair stage respectively. This indicates that the removal of

The 0 h NR-NA control group exhibited the highest μTBS value among all groups in the 0 h repair stage and caused more adhesive failures in the 24 h repair stage. In the 0 h repair stage, the NR-NA and R-SBU groups exhibited the highest percentage of cohesive failure in the RBC. The NR-NA and R-NA groups exhibited a marked increase in their adhesive failure ratio in the 24 h repair stage. The groups treated with adhesive with or without silane exhibited a slight increase in their adhesive failure ratio in the 24 h repair stage. In both repair stages, the silane application groups (R-BMP and R-SBU groups) maintained high cohesive failure and low adhesive failure rates close to those of the 0 h repair NR-NA group.

**Discussion**

RBC is a widely used restorative material and is also considered as a suitable material for RBC repair, allowing for an alternative to the time-consuming method of complete RBC removal and refilling when a marked defect is found in an RBC restoration. To ensure satisfactory repair results, bond strength measurement is important for evaluating the adhesion capability. On the basis of the results, the μTBS value revealed a significant statistical decrease from NR-NA group to R-NA group in both the 0 h repair stage and 24 h repair stage groups and lowered to 76% and 62% of the μTBS value of the control group in each respective repair stage respectively. This indicates that the removal of
the OIL caused a negative effect on the bond strength of the R-NA group. The results of this study are in accordance with the previous reports. According to the adhesion principles, factors such as surface roughness, the surface free energy of the adherend, and the viscosity or surface tension of the adhesive have effects on wettability. Kubiak et al. reported that surface roughness was correlated with contact angle and had a strong effect on the wettability of the material surface. In this study, the removal of the OIL via grinding with 600-grit sandpaper produced an irregular surface that probably caused an increase in the contact angle. Aside from the loss of the OIL of RBC blocks, this contact angle increase could be another reason why the RBC blocks that had their OIL removed exhibited poor wettability, which in turn resulted in poor bonding capability with the new RBC.

The bond strength in this study showed a higher value in the R-SBU group compared with other previous studies in which unpolymerized resin monomers can still be detected at the end of the 24 h polymerization period and can last more than one week. This may explain our results, in which all adhesive-containing (R-GB, R-PGB, R-BM, R-MBP and R-SBU) groups for either of the two repair-stage times exhibited a bond strength comparable to that of the 0 h repair control group. It has been proven that the application of adhesive on the ground surface could increase the bond strength in the RBC repair by penetrating surface irregularities and bonding with unpolymerized resin monomers of the exposed RBC surface. This result was in accordance with other previous reports. Moreover, it has been reported that unpolymerized resin monomers can still be detected at the end of the 24 h polymerization period and can last more than one week. This may explain our results, in which all adhesive-containing (R-GB, R-PGB, R-BM, R-MBP and R-SBU) groups for either of the two repair-stage times exhibited a bond strength comparable to that of the 0 h NR-NA group. This indicated that the bonding characteristics of the adhesive could increase the wettability and enable reaction with the remaining monomers of the previous RBC within at least 24 h.

Silane is recommended for enhancing the bonding capacity between silica-based ceramics and resin through the existence of hydroxyl and organofunctional groups in silane, which respectively bond to silica and resin. However, silane can not only act as an adhesion promoter but also increasing the wettability and bond strength.

### Table 2

The mean values, the standard deviation, and the statistical significance of all groups in the 0 and 24 h repair stages.

| Repair stage | Group   | N  | Mean (MPa) | Standard deviation | Standard error | Statistical significance* |
|--------------|---------|----|------------|--------------------|----------------|---------------------------|
| 0 h          | NR-NA   | 5  | 97.5348    | 3.89960            | 1.59201        | a                         |
|              | R-NA    | 5  | 74.9590    | 6.37605            | 2.60301        | b                         |
|              | R-SBU   | 5  | 92.0706    | 4.18369            | 1.70798        | ac                        |
|              | R-PGB   | 5  | 92.2285    | 3.97797            | 1.62400        | a                         |
|              | R-GB    | 5  | 92.4602    | 2.55093            | 1.04141        | ac                        |
|              | R-MBP   | 5  | 89.8765    | 5.06892            | 2.06938        | ac                        |
|              | R-GB    | 5  | 92.2285    | 3.97797            | 1.62400        | a                         |
|              | R-SBU   | 5  | 92.0706    | 4.18369            | 1.70798        | a                         |

*: Groups labeled with different letters in the statistical significance column are statistically different (p < 0.05).

NR-NA: no roughened surface without adhesive and/silane application.
R-GB: roughened surface with adhesive (Gaenial Bond, GC) application.
R-PGB: roughened surface with silane (Ceramic primer, GC) and adhesive (Gaenial Bond, GC) application.
R-BM: roughened surface with adhesive (BeautiBond Multi, Shofu) application.
R-BMP: roughened surface with adhesive (BeautiBond Multi) and silane (BeautiBond Multi PR Plus, Shofu) application.
R-SBU: roughened surface with adhesive (SingleBond Universal, 3M-ESPE) application.
Beautifil II, composed of a multifunctional glass filler and S-PRG filler based on a nano-hybrid type fluoroborosilicate glass, at a filler loading of 83.3 wt%, was the target for chemical reaction with silane in ceramic restoration. According to the present study, the bonding mechanism on ground RBC can be assumed to be as follows: (1) The silane reacted with the exposed filler of the ground RBC and (2) the adhesives reacted with the unpolymerized monomers remaining in the matrix. This explains why the $\mu$TBS of the adhesive–silane groups (R-PGB, R-BMP, and R-SBU) tended to be higher in both the 0 and 24 h repair stages than those of the corresponding adhesive-only groups (R-GB and R-BM) even though no significant differences were observed among these groups ($p > 0.05$). These results are in accordance with Mamanee et al.’s report. As mentioned earlier, one of the two functional groups in silane, the organofunctional group, binds to methacrylate part in the adhesive and unpolymerized monomers, and the other one, hydroxyl group, binds with the exposed filler. Except the binding force gained from adhesive to the monomer of the previous RBC, the chemical reaction of silane with both of the exposed filler and adhesive that induces an increasing $\mu$TBS and leads to a reinforcing effect on RBC repair. Based on the above results, the null hypothesis of this study was rejected.

SingleBond Universal is marketed as a one-step self-etching adhesive incorporated with several constituents, including silane and MDP for multi-purpose use. Regardless of the repair stages, the R-SBU group exhibited $\mu$TBS values comparable to those of the R-PGB and R-BMP groups, indicating that silane-incorporated adhesives can not only simplify the repair steps, but also achieve satisfactory bond strength within a single application. From the results of this study, it should be noted that either the sequential application of an adhesive and a silane coupling agent, or the application of a silane-incorporated adhesive, can improve the bond strength of RBC repair. The failure mode of all silane-containing groups exhibited a greater ratio of cohesive failure in the RBC than the non-silane-containing groups, demonstrating the effect of silane on RBC repair.

The results of this study show that both the adhesive-only groups and the silane-adhesive application groups exhibited $\mu$TBS values significantly greater than those of the R-NA group for both repair-stage times, and no statistically significant differences with the NR-NA group were found for each respective repair-stage. However, the RBC is used in saliva-rich environments and is continuously exposed to thermal changes. Therefore, further studies on the bonding capacity of the repaired RBC under prolonged water immersion and thermocycling conditions are necessary to realize the long-term effects of the application of adhesive as well as adhesive–silane coupling agent in RBC repairs.

We conclude that the removal of the superficial layer within 24 h significantly lowers the bond strength of RBCs. The application of either an adhesive-only or an adhesive–silane combination in RBC repair within 24 h can restore the bond strength back to its original level. The addition of silane coupling agents has a reinforcing effect on the bond strength. The application of adhesives with or without a silane can significantly increase the bond strength of the repaired RBC polymerized within 24 h comparable to that of the control group.

**Figure 2** Fracture ratio of each group in the 0 h repair and 24 h repair stages. A: adhesive failure, M: mixed failure, R: cohesive failure in RBC.
Declaration of competing interest

The authors have no conflict of interest to declare.

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