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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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## KEYWORDS

Silane coupling agent;  
Oxygen-inhibited layer;  
Roughness;  
Resin-based composite repair;  
Microtensile bond strength

**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 ( $\mu$ TBS) 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  $\mu$ TBS 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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**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.

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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,<sup>1</sup> 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.<sup>2–4</sup>

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.<sup>5</sup> The ideal solution for such problems is to remove the RBC restoration completely and refill with RBC to obtain satisfactory results.<sup>6</sup> Such a complete removal procedure is, however, time-consuming<sup>7</sup> 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<sup>8–11</sup> and also adheres to the concept of minimal intervention.<sup>12</sup>

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.<sup>13–16</sup> Several studies have mentioned that the existence of the OIL may promote the adhesion of newly added RBC onto a previous RBC restoration.<sup>16–18</sup> However, it has also been reported that the bond strength of an RBC repair had no correlation with the existence of the OIL.<sup>15</sup> 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.<sup>19</sup> Silane has been reported to improve the wetting ability of RBCs and to react with the silica ingredients of fillers to form siloxane bonds during RBC repair.<sup>19–21</sup> Its application is therefore recommended to provoke a chemical reaction with the filler of an existing RBC and promote the adhesion of new RBC.<sup>5,22</sup> However, other studies have reported no improvements in bond strength after the application of silane.<sup>23,24</sup>

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.<sup>20,25,26</sup> 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/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 unground level.

## Materials and methods

**Table 1** lists the RBCs, silane coupling agents, and one-step self-etching adhesives used in this study.

### Specimen preparation

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/cm<sup>2</sup>. 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 light-curing for 10 s; (3) R-PGB group: silane (Ceramic primer,

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

Materials	Brands	Components	Lot number	Manufacturer
Resin-based composite	Beautifil II	Bis-GMA,	051514	Shofu
	AO2 shade	TEGDMA,	041505	
Silane coupling agent	A2 shade	UDMA, filler		
	Ceramic primer	Primer A: <b>Silane</b> , ethanol; Primer B: methacryloxyalkyl acid phosphate, ethanol	A: 1309031 B: 1309051	GC
Adhesive	BeautiBond Multi PR Plus	<b>Silane</b> , Ethanol, Others	081205	Shofu
	Gaenial Bond	<b>4-MET</b> , <b>Phosphoric acid ester monomer</b> , Dimethacrylate monomer, Distilled water, Acetone	1211221	GC
	BeautiBond Multi	<b>Carboxylic acid monomer</b> , <b>Phosphonic acid monomer (6-MHPA)</b> , Water, Acetone, Polymeric monomer, photoinitiator, Others	021211	Shofu
	SingleBond Universal	<b>MDP Phosphate Monomer</b> , Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Ethanol, Water, <b>Silane</b> , Initiator	490325	3M ESPE

The monomers having bifunctional group in each adhesive are shown in bold.

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, 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 (SingleBond 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 Microtensile bond strength ( $\mu$ TBS) 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 ion sputter (E-1045, Hitachi, Tokyo, Japan) coated with platinum to observe the morphological diversity by a scanning 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  $\mu$ TBS test. The NT stick specimens of each group ( $n = 40$ ) were fixed using cyanoacrylate (Zapit, Dental Venture of America, Corona, CA, USA) to measure the  $\mu$ TBS 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.<sup>27</sup>

### Failure mode

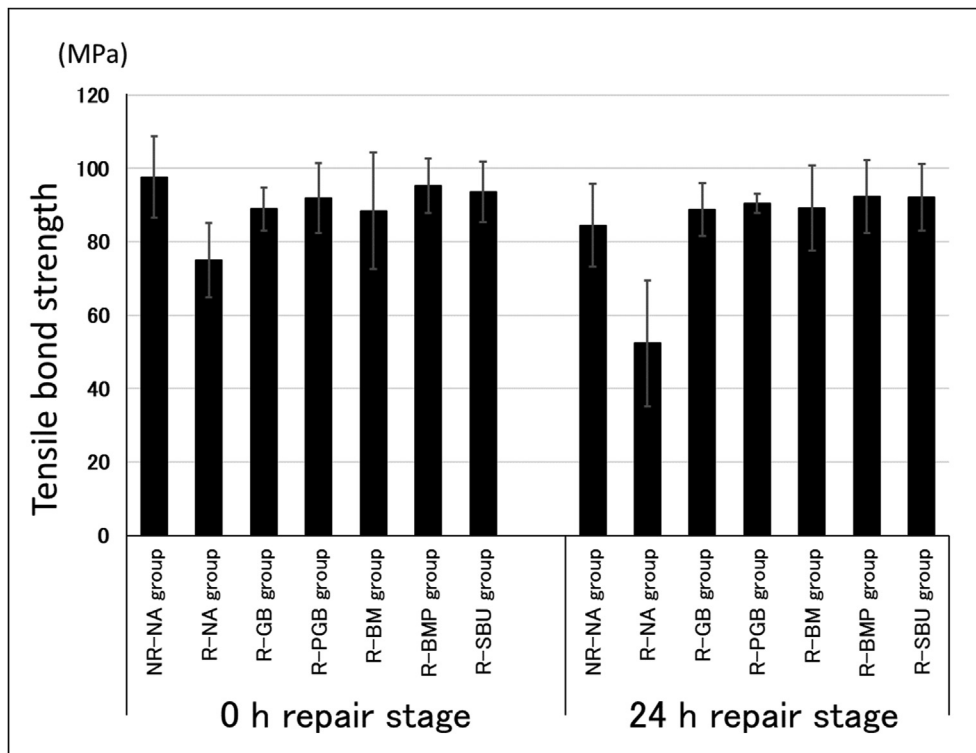
The fractured surface of each specimen in both the 0 and 24 h repair stages was examined using a stereomicroscope (20X magnification, SMZ800N, Nikon, Tokyo, Japan) to determine whether the fracture site was located at the interface of the bonding surface (denoted as A for “adhesive failure”), in any 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  $\mu$ TBS 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  $\mu$ TBS 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.



**Figure 1** Microtensile bond strength of each group in the 0 and 24 h repair stages. NR-NA: no roughened surface without adhesive and/silane application. R-NA: 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.

The 0 h NR-NA control group exhibited the highest  $\mu$ TBS value ( $97.5 \pm 3.9$  MPa) and the 24 h R-NA group exhibited the lowest  $\mu$ TBS value ( $52.2 \pm 11.7$  MPa). In the 0 h repair stage, the mean  $\mu$ TBS 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-GB group (88.9 MPa) > R-BM group (88.6 MPa) > R-NA group (75.0 MPa). In the 24 h repair stage, the mean  $\mu$ TBS 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  $\mu$ TBS value significantly lowered to 76% or 62% of that of the control group in 0 h or 24 h repair stage, respectively ( $p < 0.05$ ). Regardless of the repair stages, the adhesive–silane groups (R-PGB, R-BMP, and R-SBU) revealed a tendency to exhibit a higher  $\mu$ TBS values than their respective adhesive-only groups (R-GB and R-BM) without statistical differences ( $p > 0.05$ ). Any adhesive with/without silane application (R-GB, R-BM, R-BMP, R-PGB, and R-SBU) in the 24 h repair stage revealed a  $\mu$ TBS value comparable to that of each corresponding group and the NR-NA group in the 0 h repair stage ( $p > 0.05$ ). In the 24 h repair stage, the NR-NA group showed no statistical differences with the adhesive with/without silane application groups ( $p > 0.05$ ).

The failure mode of each group in the 0 and 24 h repair stages is shown in Fig. 2. The R-NA group exhibited the

highest adhesive failure ratio 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  $\mu$ TBS 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  $\mu$ TBS value of the control group in each respective repair stage respectively. This indicates that the removal of

**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-GB	5	88.8765	3.02073	1.23321	ac
	R-PGB	5	91.9787	2.55092	2.34164	ac
	R-BM	5	88.5915	9.31281	3.80194	ac
	R-BMP	5	95.2681	3.58735	1.46453	a
	R-SBU	5	93.5940	4.35543	1.77810	a
24 h	NR-NA	5	84.4129	3.95589	1.61498	c
	R-NA	5	52.1503	11.70550	4.77875	d
	R-GB	5	88.7815	6.29147	2.56848	ac
	R-PGB	5	90.4602	2.55093	1.04141	ac
	R-BM	5	89.2462	5.06892	2.06938	ac
	R-BMP	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-NA: 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.

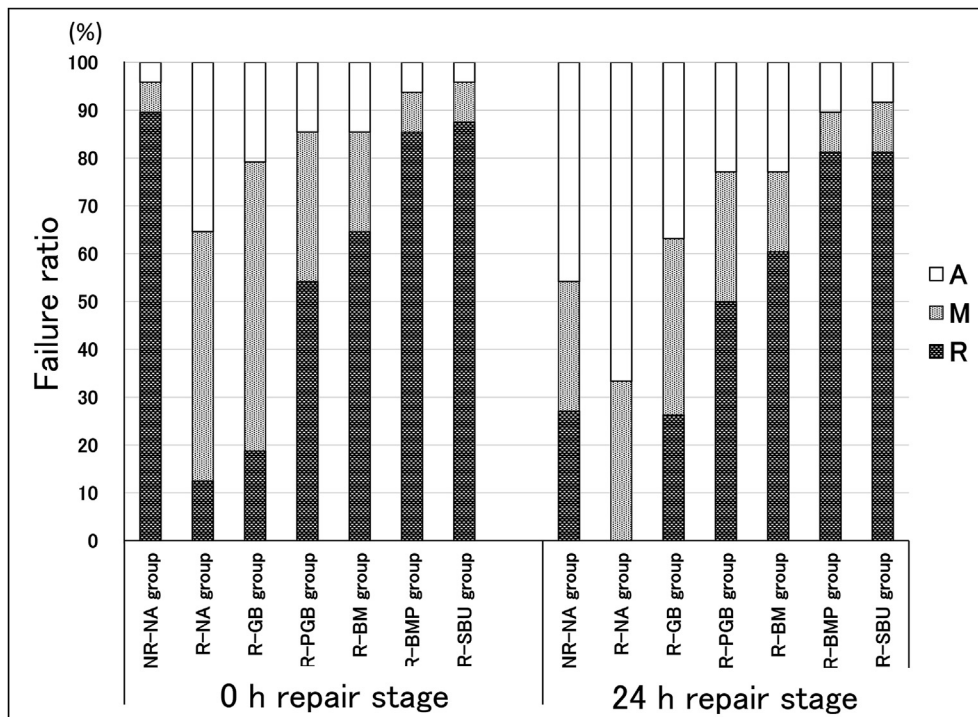
R-SBU: roughened surface with adhesive (SingleBond Universal, 3M-ESPE) application.

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.<sup>15–18</sup> 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.<sup>28</sup> Kubiak et al. reported that surface roughness was correlated with contact angle and had a strong effect on the wettability of the material surface.<sup>29</sup> 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 compared with other previous studies<sup>30,31</sup> in which measured by the non-trimming microtensile test method. In order to rule out the specificity of test machine prior this study, we have performed a preliminary study in advance to measure the cohesive bond strength of 8 mm thickness Beautifil II (A2) RBC block fabricated the same manner as those above-mentioned RBC blocks preparation procedure. The result of cohesive bond strength of Beautifil II (A2) revealed a value with no significant difference compared to that of the control group in 0 h repair stage. In fact, there are several factors that might influence the measurement values such as surface treatment technique, adhesive application time or light-curing time<sup>32</sup> and irradiation power of light-curing machine<sup>30</sup> etc. during the specimen preparation processes. Therefore, each study might reveal different bond strength values depending on the determined processes.

Several chemical or mechanical surface treatment methods, such as sandblasting using a microetcher, and the application of hydrofluoric acid, silane, adhesive, or a combination of these, have been recommended.<sup>25,33–35</sup> However, sandblasting and the application of hydrofluoric acid are not ideal methods to execute intraorally. Besides, it is more favorable to carry out a simple procedure rather than a complicated one in clinical settings. Therefore, the present study focused on the application of adhesive or a combination of silane and adhesive to assess the possibility of attaining 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.<sup>19</sup> This result was in accordance with other previous reports.<sup>26,30,31</sup> 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.<sup>30,36</sup> 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.<sup>37</sup> However, silane can not only act as an adhesion promoter but also increasing the wettability and bond strength.<sup>38</sup>



**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.

Beautifil II, composed of a multifunctional glass filler and S-PRG filler based on a nano-hybrid type fluoroaluminosilicate glass, at a filler loading of 83.3 wt%,<sup>39</sup> 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 Mamane et al.'s report.<sup>40</sup> 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.<sup>40</sup> 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.

## Declaration of competing interest

The authors have no conflict of interest to declare.

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