

Influence of aging on direct resin composite repair bond

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Purpose: The objective of this study was to evaluate the effect of aging on repair bond strength of direct resin composites.

Materials and Methods: Total of 45 (D: 9 mm and L: 4 mm) composite specimens were prepared (Clearfil AP-X). Forty-two specimens were kept for storage in Milli-Q water and in human saliva at 37°C for 2, 4, and 12 weeks. Rest three non-aged specimens (L: 8 mm and D: 9 mm) were used as control. Each aged specimen was roughened by an abrasive point, washed with water jet and air dried. Then, silane was applied followed by the application of Clearfil SE bond. After surface treatment, fresh composite of same resin: SR (Clearfil AP-X) and different resin: DR (Estelite Σ) was added and light cured in each group. After 24 hours of storage in water at 37°C, all the repaired and non repaired control specimens were cut into 1 mm sticks with a cross-sectional area of 0.7 mm² at the bonding interface and microtensile bond strength test was performed. The fracture modes and surface alterations were inspected by using a scanning electron microscope. Data were analyzed by two-way and one-way ANOVA.

Results: Post-hoc comparison showed significantly reduced bond strength between 2 weeks saliva SR (43.97) and 12 weeks Milli-Q water DR (35.11) groups. When compared to control, Dunnett T3 test revealed significantly decreased bond strength in all groups of 12 weeks.

Conclusion: The composite repair bond strengths were influenced by the different types of storage solutions and resin composition. (Int Chin J Dent 2008; 8: 53-61.)

Key Words: aging, composite resin, human saliva, repair bond strength.

Introduction

Composite resins have been extensively used as direct restorations for their high-quality esthetic properties. To improve their properties, the incessant modifications in resin composites are being done continuously. Specifically, longevity or aging resistance of direct resin composites is a very important property that needs lot more effort to improve the quality of the tooth color restorations clinically. However, repair of the aged resin restorations are considered occasionally.

The degradation processes of dental composites are complex and possibly due either to mechanical degradation mechanisms¹ such as wear, abrasion, and fatigue, or chemical degradation mechanisms such as enzymatic, hydrolytic, and acidic action, or temperature-related breakdown.² These changes may result in discoloration, micro leakage, ditching at the margins, delamination, or simply fracture, which may affect the bonding properties when repair is considered as a treatment option. According to the principle of minimal intervention dentistry, repair rather than replacement of the defective restorations³ is the preferable treatment choice nowadays. Careful case selection and correct usage of surface treatment agents, followed by the use of a quality bonding system and restorative materials, can result in a repair that exhibits excellent retention and natural color blending.⁴

Several studies have evaluated the resin-resin union between aged resin surfaces and new resin increments, simulating a repair condition. Different surface treatments have been proposed, with variable results. These methods included chemo-mechanical preparation of the resin surface, such as abrasion, etching with hydrofluoric or phosphoric acid and use of intermediate bonding agents to enhance repair bond strength.⁵ Also, it has been indicated that roughening of surfaces promotes mechanical interlocking, the bonding agent improves

surface wetting and chemical bonding with the new composite.² Abrasion of the aged resin surface with diamond burs,⁶⁻⁸ carborundum,⁷ low-pressure silicate ceramic deposition⁷ or sandblasting⁶⁻⁸ provides satisfactory bond strength between the resin increments.⁹ Additionally, the application of silane with or without previous mechanical preparation has been advised in composite resin repair. In a study by Brosh et al.,⁷ silanization and unfilled resin slightly but not significantly improved the repair strength compared to unfilled resin alone. In another study, the use of silane combined with diamond bur roughening significantly improved the bond strength between repaired surfaces.⁷

In the laboratory, aging is simulated by storage of specimens in aqueous solutions for different lengths of time and maintaining the temperature usually at 37°C. In previous in vitro studies, commonly used storage media were water, deionized water, saline solution, ethanol or artificial saliva for different time periods. Thermocycling and water storage of bonded specimens are well-accepted methods to simulate aging and to stress interfacial bonds.¹⁰

In recent years, although increasing number of researchers have adopted the microtensile technique as a more accurate method for the assessment of the interfacial strength¹¹ but only a few studies have evaluated the composite-to-composite microtensile bond strength.¹² Eiriksson, et al.,¹³ recently reported the influence of salivary enzymes on wear.

To get optimum bond strength, the best choice is to use the same composite resin or at least select a material of the same family or manufacturer with the original composite resin. Repair composite resins from different manufacturer sometimes may have to be chosen because of certain unavoidable situations, e.g.; unavailability. The union between these resin composites is a very important factor for the success of the restoration which concerns function and aesthetics as well.¹² Therefore, the objective of this study was to evaluate the effect of different storage conditions on repair bond strength of composites.

Materials and Methods

Sample preparation

The materials used in this study are listed in Table 1. Total of 45 cylindrical composite specimens, 4 mm in height and 9 mm in diameter were prepared by layering 2 mm thick increments of Clearfil AP-X (Kuraray Medical Inc., Tokyo, Japan, shade A2) in transparent plastic mold according to manufacturer's instruction. Each increment was carefully condensed into the mold with a clean plastic spatula and light-polymerized for 20 s (600 mW/cm² power output halogen light source, Optilux 500, Demetron, Danbury, CT, USA). The last increment was covered with a plastic strip and a glass slide to achieve a flat surface of the specimen after light curing. Forty two specimens were kept for storage. Rest three specimens (L: 8 mm and D: 9 mm) were used as control.

Human saliva collection

Stimulated saliva (by chewing sugar free gum) was collected 2 hours after breakfast in a sterile centrifuge tube (TPP, Gerard Biotech, Oxford, OH, USA) followed by tooth brushing and embedded in ice from pre-informed healthy lab volunteer. All the collected saliva were then centrifuged (Kubota, High Speed Refrigerated Centrifuge, Model 6500, Tokyo, Japan) at a speed of 15,000 rpm (24,400xg) for 10 minutes at 4°C. Supernatant saliva from the centrifuge tube was then transferred into the upper chamber of a filter-top centrifuge tube (Corning Incorporated, CLS 430320, pore size 0.22 µm, Corning, NY, USA) and filtered by a Teflon dry vacuum pump (DTC-21, Diaphragm Type Teflon Vacuum Pumps, Ulvac Kiko, Inc., Chigasaki, Japan). The 50

mL reservoir was capped inside of a bio clean-bench (MCV-B91F, Sanyo, Osaka, Japan) discarding the filter. The reservoir containing saliva was then stored in a refrigerator at 4°C. Every time the saliva was collected from the same person.

Table 1. Composition, manufacturers, and lot numbers of the materials used.

Material	Composition	Manufacturer	Lot number
Clearfil SE-Bond	Primer: MDP, HEMA, Dimethacrylate monomer, Water, Catalyst Bond: MDP, HEMA, Dimethacrylate monomer, Microfiller, Catalyst	Kuraray Medical Inc., Tokyo, Japan	00721A 01034A
Clearfil Porcelain Bond Activator	Bisphenol A polyethoxy dimethacrylate, 3-methacryloxypropyl trimethoxysilane	Kuraray Medical Inc.	00209A
Clearfil AP-X	Barium glass, Silica colloidal silica, Silicon dioxide (71 vol%, 0.1-1 µm), Bis-GMA, TEGDMA, Photoinitiator	Kuraray Medical Inc.	A2: 0957AA A3.5: 0629AA
Palfique Estelite Σ	Silica zirconia, Prepolymerized organic filler (71 vol%, 0.2 µm), Bis-GMA, TEGDMA, Photoinitiator	Tokuyama Dental Corp., Tokyo, Japan	C2: W805

Aging the resin composite specimen in vitro

Seven specimens of composite resin were stored in each storage media (de-ionized/ Milli-Q water and human saliva) in an incubator (Sanyo, MIR-162, Osaka, Japan) at 37°C for 2, 4, and 12 weeks. The pH of all of the liquids was measured with a pH meter (Horiba, Kyoto, Japan) before the storage of the specimens [human saliva (pH 8.05), Milli-Q water (pH 6.5)]. Each specimen was stored in separate conical tube (Falcon, 35-2096, Blue Max Jr. 15 mL Polypropylene Conical Tube, Becton Dickinson, Franklin Lakes, NJ, USA). After 2, 4, and 12 weeks, six aged specimens from each media were removed and surface treatments were done. Control specimens were not aged.

Repairing the resin composite specimens

One side of the each specimen was roughened by an abrasive point (Shofu Inc., Kyoto, Japan) mounted on low-speed hand piece for 10 s, washed with water jet and dried with air for 20 s. After that, silane (mixture of equal amount of Clearfil SE bond primer and Clearfil Porcelain Bond Activator) was applied for 5 s followed by air dried and application of Clearfil SE bond. Bonding agent was air dried and light cured for 10 s. After surface treatment, each group of composites was again divided into two groups according to the composite resin used for repair (n=3). All of the samples were reassembled into the mold and the increments of fresh composite of same resin-SR (Clearfil AP-X, shade A3.5) and different resin-DR (Estelite Σ, shade C2) were added and light cured as mentioned above.

Measurement of micro-tensile bond strength (µTBS)

After 24 hours of storage in water at 37°C, all the repaired and the control specimens were cut into slabs of 1 mm thickness using a low speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water cooling. Each slab was again serially sectioned to multiple sticks at a depth of 1 mm. The sticks were trimmed to an hourglass shape at the bonding interface with a cross-sectional area of 0.7±1 mm² using a superfine diamond point (SF114, Shofu Inc.) under water cooling. The ends of the sticks were glued to a testing device in a table-top testing machine (EZ Test, Shimadzu Co., Kyoto, Japan) using cyanoacrylate glue (Zapit, Dental Venture of America, Anaheim, CA, USA), and subjected to a tensile force at a crosshead speed of 1 mm/minute.

The cross-sectional area of each stick was measured using a digital caliper (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan). The μ TBS were calculated by dividing the fracture load by the surface area, measured to the nearest 0.01 mm with the digital calipers.

Fracture mode analysis

After the micro-tensile bond test, surfaces of the fractured beams were mounted on brass tablets and gold sputter-coated. The fracture modes were observed using a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan). Mode of failure was classified as cohesive failure in resin composite (substrate or the repairing composite), adhesive (at the interface) or mixed failure (combination of adhesive and resin cohesive failure).

Topographical analysis of the aged sample surfaces

To compare and observe the surface changes, remaining aged specimen from each storage media and storage time were dried and kept in ambient condition in a desiccator and were gold sputter-coated and observed using a scanning electron microscope (JSM-5310) to evaluate the topographical changes.

Statistical analysis

Data were analyzed by separate two-way ANOVAs with bond strength as dependent variable, and storage solution and resin type as factors. Tukey's test was then performed for post-hoc comparison in between the groups. One-way ANOVA and Dunnett T3 test was also performed to compare the experimental groups with control. Data were analyzed using the Statistical Package for the Medical Science (SPSS Ver.15 for Windows, Chicago, IL, USA) for statistical procedures.

Results

Mean micro-tensile bond strength values with standard deviations are summarized in Table 2. The two-way ANOVA showed that composite repair bond strengths were influenced by the difference of the storage solutions and also by the composition of the resin. Post-hoc comparison showed significantly reduced bond strength between 2 weeks saliva SR (43.97±11.2 MPa) and 12 weeks Milli-Q water DR (35.11±3.17 MPa) groups. But no significant differences were found between the SR of saliva and Milli-Q water in between all groups until 12 weeks. SR of saliva always showed higher bond strengths than the SR of Milli-Q water and the DR of saliva on the other hand also showed higher bond strengths than the DR of Milli-Q water groups. When compared to control, Dunnett T3 test revealed significantly decreased bond strength in all groups of 12 weeks, DR (40.85±8.13 MPa) of 2 weeks Milli-Q water, DR (37.42±12.17 MPa) of 4 weeks Milli-Q water and DR (38.95±10.85 MPa) of 4 weeks saliva.

Table 2. Means and standard deviations of the micro-tensile bond strengths (μ TBSs) in MPa.

Storage media	Repaired resin	2 weeks	4 weeks	12 weeks
Human saliva	Same resin-SR	43.97±11.21 A	42.45±12.88	41.65±6.24 ab*
	Different resin-DR	41.04±9.57	38.95±10.85 *	36.96±8.16 a*
Milli-Q water	Same resin-SR	42.21±12.93	39.56±16.58	37.46±4.86 *
	Different resin-DR	40.85±8.13 *	37.42±12.17 *	35.11±3.17 Ab*

Different letters indicate statistically significant differences ($p < 0.05$). Asterisks (*) indicate the groups in which the repair μ TBS was significantly lower than the control cohesive strength (non aged, 47.67±6.32 MPa), (n=25).

Failure mode distribution in the tested groups is reported in Fig. 1. Highest adhesive failure was recorded in 12 weeks Milli-Q water DR group. In SR groups of both saliva and Milli-Q water, cohesive failures were between 36% and 42% and the mixed failures were ranged from 24-44%. Mixed failures were mostly observed in the saliva DR group (40%) at 2 weeks and 12 weeks showed 36%. Milli-Q SR groups at 4 weeks showed mainly mixed failures (36%). Fractured specimen stored in human saliva for 12 weeks, restored with same resin (SR) shows mixed failure where most of the area were resin composites (aged/repair) with some small area of partially exposed silane/adhesive bonding agent (Fig. 2a). DR group of specimen stored for 12 weeks in human saliva also shows mixed failure where silane/adhesive and resin were observed separately (Fig. 2b). Fractured specimen stored in Milli-Q water for 12 weeks (SR) shows mixed failure, small area of partially exposed silane/adhesive bonding agent were observed within the resin composite (Fig. 3a). DR group of 12 weeks MilliQ-water specimen shows complete adhesive failure (Fig. 3b).

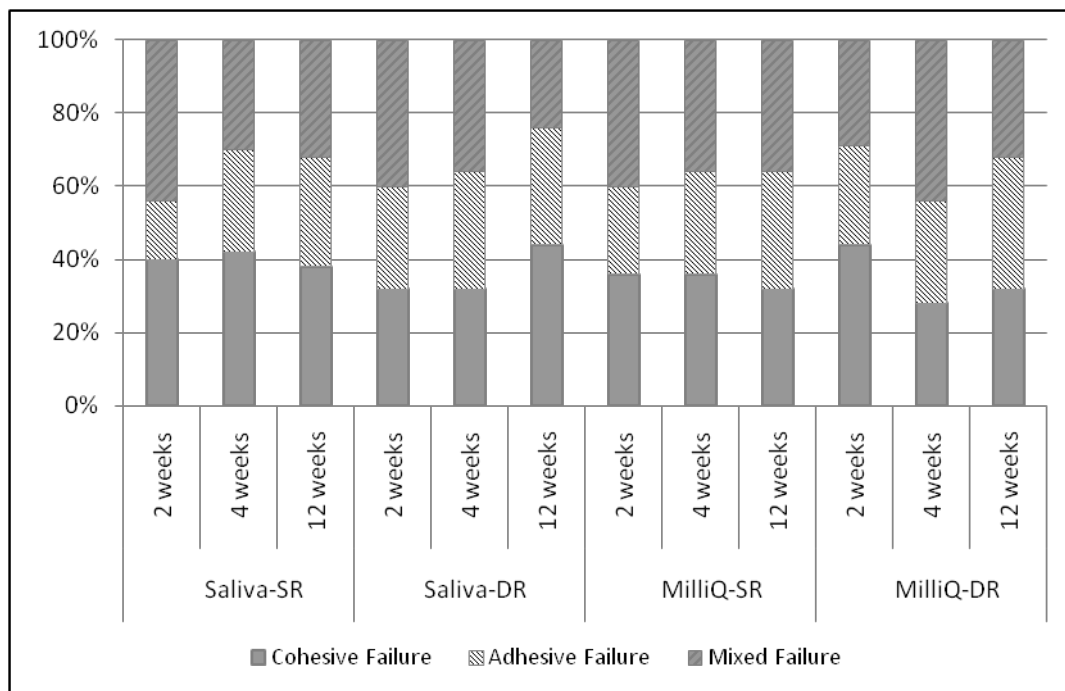


Fig. 1. Failure mode (%) distribution in the experimental groups.

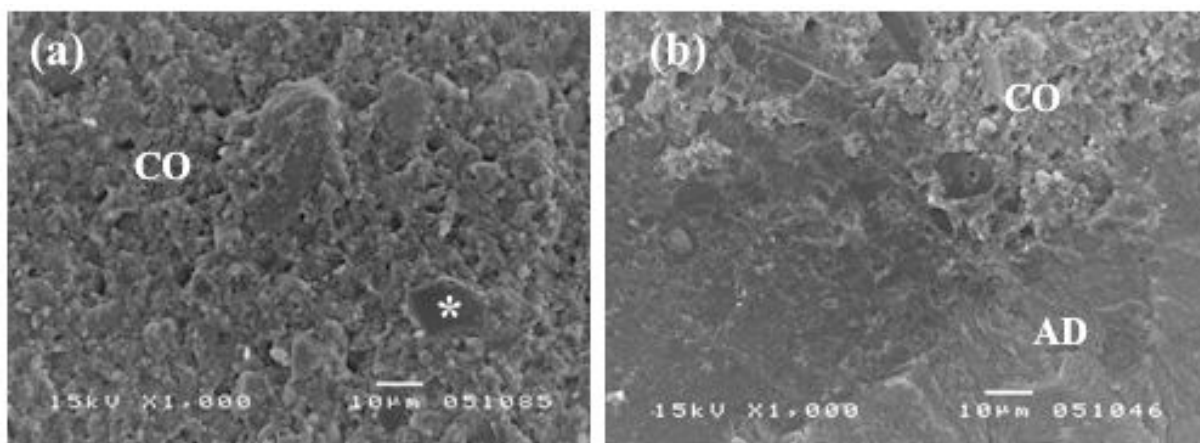


Fig. 2. Representative SEM images (1000 \times) of fractured specimens stored in human saliva for 12 weeks. (a) Restored with same resin (SR-APX) shows mixed failure, small area of partially exposed silane/adhesive (asterisk) bonding agent. (b) Restored with different resin (DR-Estelite Σ) shows mixed failure, silane/adhesive and resin are showing separately.

On SEM inspection surface deformity was observed on all composite resin specimens stored either in human saliva or in Milli-Q water (data not shown). However, in comparison the specimen aged in saliva revealed smoother surfaces than the Milli-Q water specimens (Fig. 4). Some debris and nano sized depositions were detected on the surface of 12 weeks saliva specimen (Fig. 4a). After 12 weeks, leaching out of fillers was more in Milli-Q water specimens (Fig. 4b) and structural deformity was less on the surface of the saliva specimen.

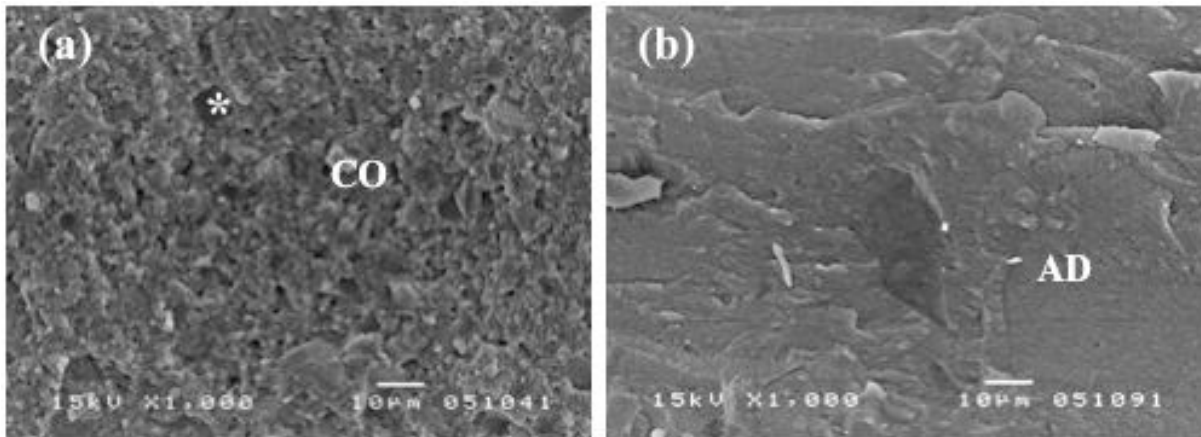


Fig. 3. Representative SEM images (1000×) of fractured specimens stored in Milli-Q water for 12 weeks. (a) Restored with same resin (SR-AP-X) shows mixed failure, small area of partially exposed silane/adhesive (asterisk) bonding agent. (b) Restored with different resin (DR-Estelite Σ) shows adhesive failure.

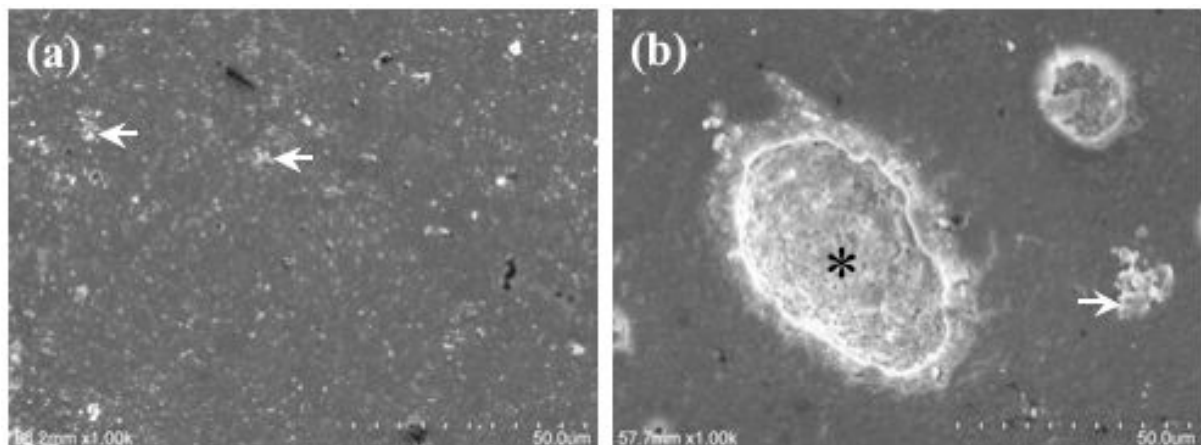


Fig. 4. Representative SEM image (1000×) of specimen stored for 12 weeks in human saliva (a) and MilliQ-water (b). Arrowheads indicate debris attached to the specimen surfaces. Black-asterisk indicates a large cavity-like deformity on the resin surface due to leaching of fillers during the storage in Milli-Q water for 12 weeks.

Discussion

The hydrolytic degradation of composite resin materials happens mainly because of accumulation of water between the filler-matrix interface that promotes the displacement of inorganic particles¹⁴ or due to the slow development of superficial flaws related to preexistent corrosive processes.¹⁵ It is believed that the water sorption causes resin softening by swelling of the polymer network and decreasing of the frictional forces between the polymeric chains.¹⁶ Söderholm et al.¹⁷ reported that filler leaching of composites was much higher in artificial saliva than in distilled water. The dissolution or elution of leachable components of composite resins, mainly inorganic ions or filler particles, may present, at short or long period, a deleterious effect in the polymeric network of the material, modifying its structure physically and chemically.

In the present study, stimulated whole saliva was used to observe the differences from deionized water as

water is frequently used as an *in vitro* aging medium. Ben-Aryeh et al.¹⁸ reported that no significant differences were found in salivary concentrations of sodium, potassium, calcium, magnesium and total protein between whole and parotid, resting and stimulated saliva. Saliva is mostly water 99.4% with 0.6% solids. The solid is composed of macromolecules like proteins, glycoprotein sugars and amylase, inorganic particles like calcium, sodium and chloride and organic particles like urea, amino acids, fatty acids and free glucose.¹⁸ Both the storage media used in this study were aqueous media. And these two aqueous media caused significant reduction of bond strength of barium glass filler containing Clearfil AP-X after 12 weeks. Usually the changes start from the composite surfaces. The mechanism of hydrolytic degradation is enhanced if the filler particles have metallic ions in their composition.^{17,19} An explanation of this effect is would be; some ions in the filler particles, such as zinc and barium, are electropositive and tend to react with water. With the loss of these elements into water, the charge balance inside the silica network is changed and reestablished with the penetration of hydrogen ions of the water in the spaces occupied by the zinc and barium. As a result of the increased concentration of hydroxyl ions, the siloxane (Si-O-Si) bonds of the silica network start to break, and there is formation of an autocatalytic cycle of surface degradation.^{17,20,21} This mechanism would explain the continuity of the superficial softening with aging time. Both the composite resins used for the present study have about similar compositions and same amount of fillers by volume. There are some dissimilarity among the size, shape and the type of fillers between these two composite resins. Due to these discrepancies and also for the different performances of the complex polymerization procedure, probably the bond strengths were lower when they repaired together. This explanation also supports the highest adhesive failure in 12 weeks Milli-Q water DR group. Repairing with the same resin composite had showed higher bond strengths in case of both storage media. But it was higher when composites stored in saliva. Okada et al.²² suggested that, when osmotic pressure will be taken into consideration, acrylic polymers in the hydrogel matrix immersed in distilled water should absorb more water than those in saliva. They also showed that composite resin did not react with Ca^{2+} and PO_4^{3-} ions from saliva. Specimens stored in Milli-Q water in the present study showed lower bond strengths very probably due to much water uptake which caused higher hydrolytic degradation.

Both the storage media showed significant reduction of bond strength and that of DR stored in Milli-Q water after 12 weeks reduced down to 20.15% compared to SR stored in Saliva for 2 weeks. After 12 weeks, DR group of Milli-Q water also showed significantly reduced (15.7%) bond strength from SR of saliva of same time. Saliva contamination significantly reduces the adhesive strength between resin increments⁹ and an effective decontamination of the surface must be performed to improve the adhesive strength between resin-resin increments. For this reason, a convenient and well accepted method was followed in the present study as several studies showed that abrasion or mechanical preparation of the contaminated surface followed by the application of silane and adhesive resulted higher resin-resin bond strength. Hisamatsu et al.²³ found the similar values of cohesive strength when silanes were employed in the repair procedure of composites. Mixing of adhesive resins with the ethanol-based PBA silane coupler results in a complex blend containing nonvolatile hydrophilic (HEMA and 10-MDP) and hydrophobic (Bis-GMA) resin monomers dissolved in volatile solvents, such as ethyl alcohol, water alone, or in combination.²⁴ Coating the previously air-dried silane/primer layer with a more hydrophobic bonding resin applied as a separate step may have accounted for superior adhesive potential of PBA/Clearfil SE Bond repair system.²⁵

SEM photomicrographs apparently showed that all the specimen surfaces aged in saliva and Milli-Q water

received some hydrolytic degradation. But it was prominent at 12 weeks Milli-Q water specimen. Specimens stored in saliva revealed less degradation all through the storage period. After 12 weeks, filler leached out from resin matrix were observed in Milli-Q water specimen. Some debris and nano sized depositions were detected on the surface of 12 weeks saliva specimen; these might be salivary proteins and inorganic particles that were deposited during long storage period.

In vitro studies do not reflect what exactly occurs in an oral cavity and offer inaccurate information in regards to the interaction between restorative materials and oral phenomena. When inserted in the oral environment, restorative materials are exposed to saliva, pH changes and other factors such as food and liquid. In the present study, repair bond strength of resin composites were affected more when aged in Milli-Q water and the repair resin was different compared to aged in human saliva. To get an optimum reflection of the oral environment, the use of human saliva as storage medium is not so much inconvenient though the solid contents of the saliva varies from individuals. With respect to the hydrolytic degradation process, as different types of restorative composite resins and polymerization methods exhibit different performances, further studies will need to be carried out with different resin composites and human saliva for longer period of time.

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