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Effect of Argon Plasma Surface Treatment on Repair of Resin Composite Aged Two Years

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Clinical Relevance

After one year of water-storage, the composite repair technique was improved with the combination of non-thermal plasma surface treatment with silanization and hydrophobic adhesive application on a sandblasted resin composite aged two years.

SUMMARY

Objectives: To evaluate the effect of argon plasma treatment (PLA) when combined with sandblasting (SAN), silanization (SIL), and hydrophobic bonding resin (HBR) on the shear bond strength (SBS) of a two-year water-aged resin composite bonded to a newly placed composite after 24 hours and one year of water-storage.

Methods and Materials: Thirty-six light-cured composite plates (20mm x 20mm x 4mm thick) were obtained and stored at 37°C in distilled water for 2 years. These aged plates were distributed into 6 groups (n=6) according to the surface treatment: no treatment (Negative Control); SAN+SIL+HBR (Positive Control); SAN+PLA+SIL+HBR; PLA+SIL+HBR; PLA+SIL; PLA+HBR. Fresh resin composite cylinders were built up using silicone molds (hole: 1.5 mm high x 1.5 mm diameter) positioned over the aged plates. Half of the SBS samples were stored in distilled water for 24 hours and loaded until failure, while the other half were stored for 1 year before being tested. Data were submitted to two-way analysis of variance and post-hoc Tukey Test (preset alpha of 0.05).

Results: Positive Control, SAN+PLA+SIL+HBR and PLA+SIL+HBR groups presented higher SBS means at the 24 hour evaluation. After 1 year of

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water storage, all groups demonstrated significant SBS reduction, with the SAN+PLA+SIL+HBR group presenting the highest SBS.

Conclusions: Resin plasma treatment in combination with other surface treatments can improve the SBS of composite repairs after one year of water storage. The SBS of the composite repair was not stable over time regardless of the surface treatment.

INTRODUCTION

The complete removal of an old composite restoration is frequently not necessary or desirable. Techniques such as simple composite repair preserve tooth structure, reduce the cost of clinical procedures and prevent the potentially harmful effects of tooth preparation on the pulp.[1,2] Studies have shown that the behavior and success of adhesion of the repaired area depend on the chemical composition of the composite restoration,[3] its wettability and roughness,[4,5] and the surface treatment protocol applied.[3,6,7,8,9]

It has been demonstrated that the bond strength between old and new composites decreases after prolonged water storage.[10] In addition, for older composite restorations, there are fewer chemical bonds available for bonding with a fresh composite, since unreacted monomers have leached out over time. To overcome this undesirable situation, surface treatments are recommended to increase the free surface energy of the composite restoration to be repaired. Sandblasting with aluminum oxide has been indicated to increase superficial roughness and create micromechanical retention at the composite surface.[11,12,13,14] In addition, a chemical bond may be established between resin and silica glass filler particles by application of a silane-coupling agent.[15,16,17,18,19]

An alternative method for treating the composite resin surface is the application of atmospheric pressure argon plasma (PLA), which increases the surface energy by surface chemical destabilization, making it possible for the composite resin surface to form new chemical bonds to another substrate.[20] Thus, PLA application to old composites may have the ability to enhance the composite restoration repair technique, improving its bond strength and longevity.[14,21,22] Several studies have evaluated composite repair bond strength using composite samples aged for a short time; however, none tested the effect of these treatments in repair techniques using two-year-aged composites.

The aim of this study was to evaluate the effect of PLA combined with sandblasting (SAN), silanization (SIL), and hydrophobic bonding resin (HBR) on the shear bond strength of a two-year water-aged restorative resin composite to a newly placed composite after 24 hours and one year of distilled water-storage. The hypotheses tested were that: 1) there is no difference among the surface treatments of old composites in their effects on repair bond strength; and 2) shear bond strength values are stable following water-storage for one year.

METHODS AND MATERIALS

Thirty-six standardized composite plates (20 mm x 20 mm x 4 mm thick) were obtained in the same way as in our previous study,[14] by placing composite resin (Charisma, Heraeus Kulzer, Hanau, Hesse, Germany, shade A2, lot number: 010611) in silicone molds. The composite surface was divided into four areas of 100 mm^2, which were light activated separately for 20 seconds; each area was fully covered by the light irradiation. The same light curing was repeated on the bottom surface. The light-curing unit used was a multiwavelength LED (Valo Cordless, Ultradent Products Inc, South Jordan, UT, USA) with 9.4-mm internal tip diameter and delivering 1,470 mW/cm^2 of irradiance (USB 4000, Ocean Optics, Dunedin, FL, USA) in standard mode. Composite plates were stored in distilled water at 37°C for 2 years. After aging, plates were polished with 600-grit SiC paper for 20 seconds (Norton, Vinhedo, SP, Brazil) to remove the outer surface and were submitted to ultrasonic cleaning for 5 minutes (USC 1400, Unique Industrio e Comercio de Produtos Eletronicos Ltda, Indaiatuba, SP, Brazil). Afterwards, plates were distributed into 6 groups (n=6) according to the following surface treatments:

1. Negative Control: old composite + new composite
2. Positive Control: old composite + SAN +SIL + HBR + new composite
3. SAN+PLA+SIL+HBR: old composite + SAN + PLA + SIL + HBR + new composite
4. PLA+SIL+HBR: old composite + PLA + SIL + HBR + new composite
5. PLA+SIL: old composite + PLA + SIL + new composite
6. PLA+HBR: old composite + PLA + HBR + new composite

The PLA (Surface Plasma Tool Model SAP - Lab applications; Surface - Engineering and Plasma Solution LTDA, Campinas, SP, Brazil) application time was 30 seconds,[14,23] using only argon gas (Praxair 4.8, White Martins Gases Ind. S.A., Rio de Janeiro, RJ, Brazil), with an output of 1.0 liter per minute.[24] Sandblasting (SAN) with 50-μm aluminum oxide particles (Microetcher, Danville Materials, San Ramon, CA, USA) was performed for 10 seconds, 10

mm distant from the plate surface at 60 psi, followed by ultrasonic cleaning (5 minutes) and air-drying for 30 seconds.

For SIL application, a drop of a silane-coupling agent (Ceramic Primer, 3M Oral Care, St Paul, MN, USA, lot number: N555194) was deposited on a mixing pad and collected by a disposable brush to be applied over the plates in a uniform thin layer. The layer was kept undisturbed for 15 minutes and air dried for 10 seconds for evaporation of water and other solvents. The HBR (Adhesive/Adper Scotchbond Multi-Purpose, 3M Oral Care, lot number: N515442) was applied in a uniform coating using a disposable brush, followed by 10 second light-activation with the same light-curing unit.

The silicone molds (Aquasil Ultra Putty, Dentsply Caulk, Milford, DE, USA) were positioned over the treated plates and a fresh composite (same brand and manufacturer - lot number: 010636A) was inserted into the mold. The fresh composite was light cured for 20 seconds and the silicon mold was carefully removed to expose a cured composite cylinder of fresh resin bonded to the aged composite plate (1.5 mm high x 1.5 mm diameter). Four composite cylinders were placed on each composite plate at four different locations on the plate, and the samples were immersed in distilled water at 37°C. Two composite cylinders on each plate were tested after 24 hours of water-storage, while the two remaining cylinders were tested after one year of water storage.

For the bond strength test, each plate was positioed on a device attached to a universal testing machine (EZ Test, Shimadzu Corp., Kyoto, Japan). A thin orthodontic wire (0.2 mm diameter) was looped around the cylinder, making contact with half of its circumference, and subjected to a shear force (crosshead speed of 0.5 mm/min). at the old-new composite bonding interface until failure occurred. Bond strength data were calculated using the peak of loading failure divided by the specimen surface area, and means were obtained in megapascals (MPa). The average value obtained from the two analyzed cylinders for each storage period was considered the mean value of each sample. Normality of the data was reached after transformation and subjected to two-way analysis of variance (ANOVA) and Tukey post hoc test ($p<0.05$), using SAS 9.3 software (SAS Institute, Cary, NC, USA).

After bond strength testing, the fracture surfaces were mounted onto brass stubs and gold coated (MED 010, Balzers, Balzer, Liechtenstein). Tested specimens were examined using a scanning electron microscope ([SEM] JSM-5600LV, JEOL Inc., Tokyo, Japan) at 35× magnification (voltage: 15 kV; beam width: 25-30 nm; working distance: 10-20 mm). The failure modes were classified as: 1- adhesive failure (at the old-new composite interface) or 2- cohesive failure within old composite.

RESULTS

Statistical testing indicated that both treatment ($p<0.001$) and evaluation-time factors ($p<0.001$) significantly influenced bond strength, with significant interaction between them ($p=0.003$) (Table 1). At 24 hours, the groups Positive Control (28.3 MPa), SAN+PLA+SIL+HBR (34.2 MPa), and PLA+SIL+HBR (28.2 MPa) presented the highest bond strength values, while Negative Control (8.8 MPa) and PLA+SIL (11.8 MPa) showed the lowest ones. Group PLA+HBR (18.4 MPa) presented an intermediate bond strength result. After one year, bond strength of all surface treatments reduced significantly and SAN+PLA+SIL+HBR (23.8 MPa) presented the highest mean, followed by Positive Control (17.6 MPa) and PLA+SIL+HBR (16.4 MPa). The other groups showed bond strength lower than 8 MPa.

Table 2 shows the results of failure modes, and Figures 1 and 2 are representative images of adhesive and cohesive failures, respectively. At 24 hours, groups Positive Control, SAN+PLA+SIL+HBR, and PLA+SIL+HBR had 100% cohesive failures, while Negative Control and PLA+SIL had 100% adhesive failures at the old-new composite interface. Group PLA+HBR presented 50% adhesive and 50% cohesive failures. At one year, an increase in the percentage of adhesive failures was observed for Positive Control (from 0% to 30%), SAN+PLA+SIL+HBR (from 0% to 20%), PLA+SIL+HBR (from 0% to 40%), and PLA+HBR (from 50% to 100%). Negative Control and PLA+SIL continued to have 100% adhesive failure.

Table 1: *Bond Strength Means (SD) for Experimental Groups (in MPa)*[a]

Group/Treatment	24 Hours	1 Year
Negative Control	8.8 (1.9) Ca	5.2 (0.8) Cb
Positive Control (SAN+SIL+HBR)	28.3 (1.8) Aa	17.6 (2.2) Bb
SAN+PLA+SIL +HBR	34.2 (2.8) Aa	23.8 (4.7) Ab
PLA+SIL+HBR	28.2 (3.0) Aa	16.4 (5.1) Bb
PLA+SIL	11.8 (1.7) Ca	2.7 (0.6) Db
PLA+HBR	18.4 (4.2) Ba	7.5 (1.5) Cb

Abbreviations: HBR, hydrophobic bonding resin; PLA, plasma; SAN, sandblasting; SIL, silanization.

[a] *Uppercase letters compare treatments within the same evaluation time and lowercase letters compare evaluation times within the same treatment (p<0.05, by Tukey test).*

Table 2: *Failure Modes (%) Among Experimental Groups*

Group/Treatment	24 Hours	1 Year
	AD/CO	AD/CO
Negative Control	100/0	100/0
Positive Control (SAN+SIL+HBR)	0/100	30/70
SAN+PLA+SIL +HBR	0/100	20/80
PLA+SIL+HBR	0/100	40/60
PLA+SIL	100/0	100/0
PLA+HBR	50/50	100/0

Abbreviations: AD, adhesive failure; CO, cohesive failure; HBR, hydrophobic bonding resin; PLA, plasma; SAN, sandblasting; SIL, silanization.

DISCUSSION

The first hypothesis—that the difference among the surface treatments of old composites would have no effect on repair bond strength—was rejected, because some groups (Negative Control, PLA+SIL and PLA+HBR) showed lower bond strength at both evaluation times than that obtained for Positive Control and SAN+PLA+SIL+HBR. The second hypothesis was also rejected, since the repair bond strength of all groups was reduced after one year, with reductions ranging from 30% (SAN+PLA+SIL+HBR) to 77.1% (PLA+SIL).

The same SAN+PLA+SIL+HBR group that showed the lowest percentage of bond strength reduction also resulted in the highest composite repair bond strength at one year (23.8 MPa). This group differed from Positive Control due to the PLA application after SAN. At 24 hours, the bond strength of these groups (Positive Control and SAN+PLA+SIL+HBR) did not show a statistical difference, but the higher percentage reduction seen with the Positive Control group (37.8%) after one year resulted in a lower bond strength mean compared with SAN+PLA+SIL+HBR. Thus, PLA positively influenced the results and showed an important role in the long-term evaluation of repair bond strength. SAN+ SIL+HBR was used as the Positive Control because a bibliographical survey showed that this technique obtained the best bond strength repair results.[11-13,19,25,26]

The correct method for the aging of composites is crucial for the analysis of their repair potential in a laboratory set-up that resembles the clinical environment. In spite of the fact that there is no aging protocol considered the gold standard for simulating the aging process that composites are subjected to in the oral environment, static storage in distilled water is the most used method for aging such composites (82.4%).[27] Besides the method, the time of aging is of utmost importance since, in clinical situations, composite repair can be expected to be necessary in the medium or long term.[28] However, only 22.2% of the studies in the current literature aged composites in water for 30 days or longer.[27] In this study, composite samples were aged by immersion in distilled water for 2 years before they were repaired and tested.

The storage of samples in composite repair studies is of great interest because new composites are more reactive than older ones. Free radicals and free monomers are still available in fresh restorations, improving their adhesive capability.[29] In contrast, over time hygroscopic

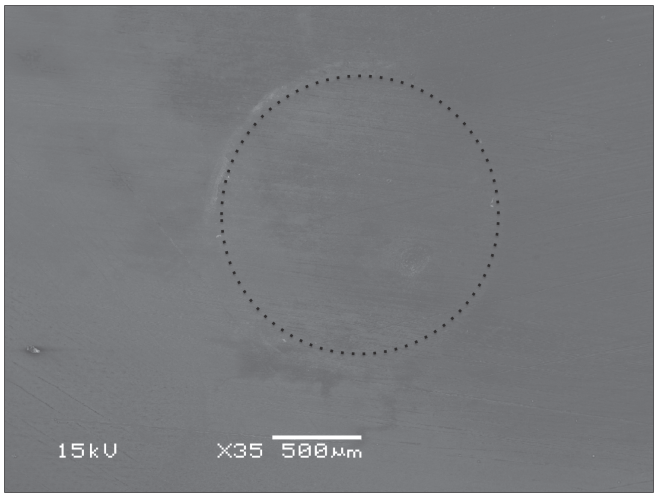

Figure 1. *Representative SEM image of adhesive failure (at the old-new composite interface). This failure occurred in a sample (at 24 hours) of the Group 5 (PLA+SIL). Original magnification 35×.*

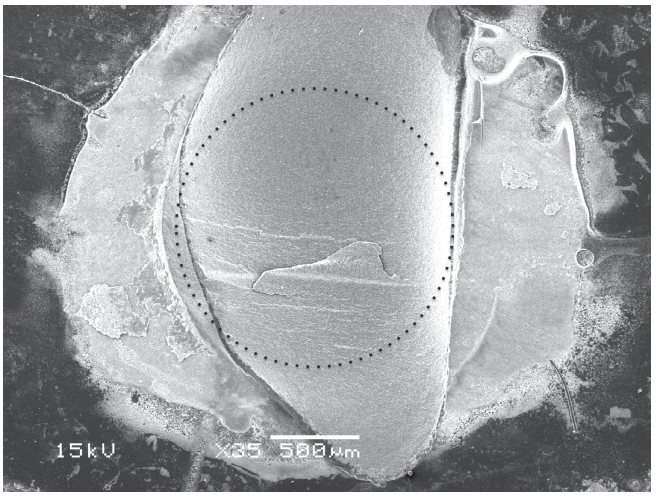

Figure 2. *Representative SEM image of cohesive failure within old composite. This failure occurred in a sample (at one year) of the positive control (SAN+SIL+HBR). Original magnification 35×.*

and hydrolytic effects of water aging take place within the cross-linked polymer structure, leading to water absorption and polymer swelling, which relax the physical bonds between polymer chains and allow free monomers and oligomers to be eluted.[27,30] Depending on the composition of old composite, the aging of a restoration can affect the materials differently. Thus, knowing the type of composite (such as microhybrid, nanohybrid, or nanofilled) to be repaired is important for the success of the procedure.[3,18,31] For example, the effects of PLA on various types of composites is not known, and further studies are needed to evaluate whether PLA can alter composite surface properties differently.

If a composite sample is aged, it has a low surface energy with poor wettability, and a surface treatment is required to improve these properties.[20] In this laboraotory study, the outer surface of old resin composite was removed using 600-grit SiC abrasive paper to flatten the surface and prepare it for bonding with fresh composite; in clinical practice, this is accomplished with carbide and diamond bur rotatory instruments. The exposed inner surface might have more residual monomers and pendant methacrylate groups for bonding to newly placed composite, potentially resulting in higher bond strength values.

PLA application is an option for increasing surface energy and making the polymeric structure more reactive to chemical bonding. PLA is a partially ionized gas consisting of a mixture of atomic and molecular reactive species.[14,20,32,33,34] In a high frequency electrical field, the free electrons become energized and collide with neutral gas molecules, and the energy transferred in the process dissociates molecules, forming numerous reactive species. The interaction of these excited species with solid surfaces increases their hydrophilicity and reactivity.[20] Argon gas is non-toxic and inert and is consumed during plasma generation. However, despite the fact that certain nonthermal plasma devices have already been approved as safe in some clinical trials, there are still many open issues with regard to the molecular and biophysical mechanisms of the procedure and the biological effects of plasma on mammalian cells and tissues.[35]

The application of argon plasma for 30 seconds has been used to treat dentin for bonding of direct composites,[36] for improving resin cement bonding to indirect composite materials[23] and zirconia ceramics,[37] and has been suggested for treating old composite restorations in repair techniques.[14] The PLA application time for 30 seconds is clinically adequate and produces the desired effect, as this is an additional step in the composite repair technique and because application times longer than 1 minute can be considered clinically long, no added benefits, and times less than 30 seconds do not have the positive effect to increase bond strength.

PLA effects may explain some outcomes of this study, such as that the group SAN+PLA+SIL+HBR presented the highest composite repair bond strength mean at one year. The ceramic primer (SIL) is a very fluid material and may not require PLA to increase wetting of the sandblasted, hydrophobic old composite, as can be seen when comparing the Positive Control and SAN+PLA+SIL+HBR groups at 24 hours. However, at one year, a statistical difference was observed between the Positive Control and the SAN+PLA+SIL+HBR group, which may be because the application of PLA results in reactive species being deposited on the composite surface, improving SIL chemical bonding to the fillers and HBR to the resin matrix of old composite. In addition, at both 24 hours and one year, the PLA+SIL+HBR group did not differ from the Positive Control (SAN+SIL+HBR), showing the potential of PLA to activate the surface and partially substitute for the SAN technique. These outcomes are different from those previously found, in which PLA did not improve bond strength when used alone or in combination with other surface treatments.[14] The same type of composite was used in the present study and in the article by Ayres and others [14]; however, the low aging time (six months) yielded amore reactive composite surface containing considerably more unreacted methacrylate groups,[38] which may have influenced the bond strength more significantly than the application of PLA. On the other hand, by aging the composite for two years, it was possible to verify the real effects of PLA application on improving the composite repair bond strength once unreacted monomers were leached out.

The air abrasion promoted by SAN is one type of roughening procedure that can provide higher composite repair bond strength than other mechanical methods. SAN produces three-dimensional roughness with variations in the peaks and valleys of the surface and thus provides more micro-retentive features than other mechanical treatments and more available area to interact with a bonding agent and the new composite increment.[11,12,14,25,26,39] Mechanical shocking by alumina particles and the non-selective removal of filler particles and portions of the polymer matrix promote retention by interpenetration of fluid material, forming a bonded interface after curing.[27]

The SAN technique contributes to the exposure of silica from the interior of the aged composite and also increases the surface area for adhesion. Old composite consists of inorganic filler particles that should be silanized to improve its bonding to organic monomers in the repair material.[19,40] One important

property of PLA application that may contribute to bond strength is its ability to change surface energy and enhance wetting of different surfaces,[24,36] possibly resulting in the resilanization of the filler particles at the sandblasted surface. This might explain why the PLA+SIL association (SAN+PLA+SIL+HBR group) demonstrated higher bond strength than SIL alone (Positive Control) in sandblasted composite after 1 year of water storage.

The groups that used SAN as surface treatment, along with the group PLA+SIL+HBR, showed higher means at 24 hours and one year, indicating the importance of SAN for immediate and long-term mechanical retention. In addition, the Negative Control and PLA+SIL groups, which did not use SAN, showed the lowest bond strength means at the 24-hour and one-year evaluation times. However, to safeguard the health and safety of patients and professionals during intraoral SAN, an aspirator device and rubber dam isolation must be used, thus limiting its clinical applicability.[41] Alternatively, to avoid SAN, rotary instruments with diamond burs may also be able to roughen restoration surfaces.[13,25,39]

Studies have suggested the use of a low-viscosity material for application after surface treatments, particularly SAN, because packable, high-viscosity composites do not penetrate into the micro-irregularities caused by air abrasion.[38,42,43] Thus, a bonding agent can improve composite repair bond strength;[44-46] this is attributed to the bonding agent's infiltration into and retention in the mechanical roughening created by air abrasion.[25] In this study, the Negative Control and PLA+SIL groups, which were not submitted to SAN and did not receive HBR, presented the lowest bond strength means at both evaluation times. Alternatively, some studies have used adhesives containing hydrophilic monomers instead of HBR.[47,48] However, highly hydrophilic bonding agents can result in the increase of water sorption and solubility on the adhesive layer, leading to early hydrolytic degradation and eventually to interfacial debonding.[25,47,48]

Despite the importance of the HBR layer, a previous study showed that an intermediate bonding agent did not improve the composite's immediate repair bond strength without SIL, which can improve repair bonding, and results in cohesive failures within composite structure after testing.[3] This fact could be seen in this study, since the PLA+HBR group did not show high bond strength means, especially after one year. Aged composites have low levels of unreacted monomers on their surfaces, and new reactions aiming to create a chemical bonding mechanism with filler particles are important for composite repair techniques. Silanes contain a silanol group (or alkoxy group), which can chemically react with the methacrylate group that copolymerizes with the resin matrix and with the hydroxyl groups from silica-based filler particles of the old composite.[49,50]

In this study, SIL combined with HBR and SAN yielded higher composite repair bond strength at 24 hours and one year. However, when used solely with PLA (PLA+SIL group) the bond strength was very low because silane chemical coupling with the old composite depends upon the availability of silica at the surface.[27] The two methods, mechanical (SAN) and physical (PLA), complement each other, and, according to a recent systematic review, the application of both physical and chemical surface treatments on aged dental composites improves the repair bond strength of methacrylate-based restorations.[27] In addition to the analysis of the influence of different types of surface treatments, the time-factor evaluation, with long-term water storage for one year, indicated that composite repair bond strength decreases over time regardless of the surface treatment.

The failure mode results in this study were in agreement with the bond-strength outcomes, since groups that presented the highest bond strength means had a low rate of adhesive failures and a high rate of cohesive failure within aged composite, showing better composite-to-composite bond strength repair quality. However, it is important to consider that the shear bond-strength test favors the occurrence of cohesive failures, mainly when there is a strong interaction between bonded materials.[51,52] The microtensile bond strength test is the preferred method of evaluating the bond strength of adhesive resins to enamel and dentin, because the stress concentration at the bonded interface is more severe in shear as compared to tension; the "macro" bond strength tests increase the rate of cohesive failure.[53] Moreover, among shear methods, the chisel as a loading device causes the most severe stress concentration at the tested interface,[53] while the orthodontic-looped wire can yield the highest shear bond strength values followed by chisel and stainless steel tape.[54] However, in studies of resin cement adhesion to ceramics and indirect resins, as well as in composite resin repair investigations, the shear strength test is still used.[3,5,7,10,13,14]

Groups that did not undergo any kind of mechanical surface treatment (Negative Control and PLA+SIL) showed 100% adhesive failure, while PLA+HBR showed an intermediate result, with 50% adhesive failure and 50% cohesive failure. The composite repair long-term bond-strength reduction was consistent with failure pattern results, since water-storage for one

year increased the number of adhesive failures for the Positive Control (30%), SAN+PLA+SIL+HBR (20%), and PLA+SIL+HBR (40%) groups.

CONCLUSIONS

Within the limitations of the shear bond strength method, PLA used in combination with other surface treatments (SAN, SIL and HBR) can improve the bond strength of composite repair after one year of water storage. When SAN was substituted by PLA, no difference was observed among the bond strength means of these groups, regardless of the evaluation time. The composite repair bond strength was not stable over time for any of the treatments evaluated. However, the best outcome was found when mechanical and chemical treatments were combined.

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Conflict of Interest

The authors have no financial interest in any of the companies or products mentioned in this article.

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