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The Effect of Different Surface Treatments of Shear Bond Strength of Repaired Polymer-infiltrated Ceramic Network Materials

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Abstract

Objectives: This study aimed to determine the alternative surface treatment method for repairing aged polymer-infiltrated ceramic network materials (PICNs) utilizing a shear bond strength (SBS) test.

Methods: A PICNs block (VITA Enamic®) was cut into 5x5x5 mm^3 followed by thermocycling for 10,000 cycles. The specimens were then randomly divided into four groups (n=12), based on different surface treatments. Group HF+Si: treated with a 9.5% hydrofluoric acid and silane application, Group HF+Si+He: treated with a 9.5% hydrofluoric acid and silane application followed by an application of a hydrophobic resin monomer, Group MEP: treated with a self-etching ceramic primer, Group MEP+He: treated with a self-etching ceramic primer followed by an application of a hydrophobic resin monomer. All specimens were repaired with a resin composite and underwent a thermocycling aging process for 10,000 cycles before measuring shear bond strength.

Results: One-way ANOVA revealed a significant difference in SBS among all groups. Group MEP exhibited a significantly lowest mean SBS value ($p<0.05$), while, mean SBS values from groups HF+Si, HF+Si+He, and MEP+He did not show statistically significant differences.

Conclusions: Treating aged PICNs with only self-etching ceramic primer group provided an insufficient shear bond strength. However, when a hydrophobic resin monomer was applied after conditioning with self-etching ceramic primer, shear bond strength was distinctly improved to a comparable level to those treated with 9.5% hydrofluoric acid and silane primer.

Keywords: hydrofluoric acid, hyphobic resin monomer, self-etching ceramic primer, silane, PICNs

Introduction

Advanced developments in digital technology and manufacturing processes have resulted in a dramatic paradigm shift in dentistry and the widespread use of computer-aided design/computer-aided manufacturing (CAD/CAM) in the fabrication of indirect dental restorations.[1,2] While various dental ceramics are currently improved and available for CAD/CAM fabrication[2,3], they are still brittle and susceptible to cracks and fractures. Such fractures are difficult to repair[4-6], impairing restoration longevity. As a result, the trend of development aims to reduce risk of fracture in indirect posterior restorations.[5]

Since 2013, the only one commercially available polymer-infiltrated ceramic materials (PICNs) with CAD/CAM technology, VITA Enamic® (VITA Zahnfabrik, Bad Säckingen, Germany), has been introduced to dental profession. VITA Enamic® is composed of a network of feldspathic ceramics infiltrated by a polymeric phase.[4,7-9] The polymer-infiltrated ceramic materials exhibited lower brittleness, rigidity, and hardness than glass ceramics. They also demonstrated increased flexibility and fracture toughness.[7,9] This may result in an improvement in stress distribution, particularly during the mastication process.[10] Nevertheless, several factors, including high masticatory forces, parafunctional habits, and internal defects within the material, critically impact the success of dental restorations in a long period of clinical service. Crack propagation induced by these factors may lead to fracture of restoration, significantly compromising longevity.[7-9] According to a three-year clinical research study conducted by Spitznagel *et al.*,[11] they discovered that fractures were the primary cause of failures in 103 PICNs CAD/CAM restorations which were unacceptable bulk fractures and chipping. The repairable failed restorations were repaired with resin composite and showed no failure up to three-year follow up.[11] Among those chipping, non-catastrophic fractures were repairable, extending esthetics and functional preservation of restorations.[12,13]

Previous studies have been reported that the most effective technique for repairing polymer-infiltrated ceramic materials was etching with hydrofluoric acid followed by silane application[14, 15] and re-restoring with resin composite.[13] However, hydrofluoric acid was considered a hazardous substance, especially when it was spilled on tissues. The aggressiveness of this acid can cause burns that frequently result in deep tissue necrosis.[13,16] The alternative less aggressive acid has been developed in order to reduce such complication during repairing procedure. A new self-etching glass-ceramic primer (Monobond Etch & Prime; Ivoclar Vivadent, Schaan, Liechtenstein) consists of four distinct compositions including an ammonium polyfluoride, phosphoric acid ester, solvent and silane enclosing in one bottle.[17] Murillo *et al.*,[18] compared the effects of the new self-etching primer with the contemporary technique on bonding to glass-ceramic and resin cement. The result indicated no statistically significant difference in microtensile bond strength between the two groups. However, no study has examined the effectiveness of either self-etching primer alone or combined with a hydrophobic resin on the repair of polymer-infiltrated ceramic network materials.

The application of bonding agents on old ceramic before repairing with resin composite has also been discussed.[19,20] According to certain studies, using bonding agents, ones containing hydrophobic resin monomer, improved the bond between glass ceramics and resin composites.[19,20] On the other hand, the systematic review and meta-analysis performed by Nogueira *et al.*,[21] showed insufficient evidence to encourage using an adhesive system as an adjunctive surface treatment before repairing. With this inconsistency, the study about the benefit of a hydrophobic resin application on repairing polymer-infiltrated ceramic network materials surface, especially when combined with self-etch ceramic primer is scarce.

Altogether, these raised the question whether the new self-etching ceramic primer combined with a hydrophobic resin monomer could effectively repair the polymer-infiltrated ceramic network materials (PICNs). Therefore, the purpose of this study was to determine the different surface treatment methods for repairing polymer-infiltrated ceramic network material (PICNs) using shear bond strength. Additionally, an application of a hydrophobic resin monomer before placement of resin composites was also investigated in the present study.

Materials and Methods

Specimen preparation

A total of 6 CAD-CAM PICNs block (VITA Enamic®, VITA Zahnfabrik, Bad Säckingen, Germany), size 12×14×18 mm^3, were cut into 5x5x5 mm^3 slices using

a low-speed diamond cutting (Isomet Low Speed Saw, Buehler, USA) under constantly running water followed by a thermocycling aging process was simulated using dwelling in water between temperatures of 5-55ºC for 10,000 cycles with a 60 s dwell time per bath (THE1400, SD Mechatronik GmbH). All specimens were then embedded in a self-curing acrylic resin. Each block was polished with five-step silicon carbide abrasive papers (200, 400, 600, 800, and 1,000 grit) using a polishing machine (NANO 2000, Pace Technologies, USA) to achieve standardized smooth surfaces before being cleaned with water for 5 minutes in an ultrasonic cleaner (Bransonic, Germany). All processes of specimen preparation are shown in Figure 1.

Surface treatment

The details of the material used in the study are shown in Table 1, and the experimental procedures are displayed in Figure 1. The specimens were randomly divided into four groups according to the surface treatment (n=12).

Group1 (HF+Si): etch with 20-µl of 9.5% hydrofluoric acid (PORCELAIN ETCHANT, Bisco, Schaumburg, USA) for 60 seconds, wash with air-water spray for 60 seconds, and air-dry for 10 seconds. Afterward, apply a 10-µl Silane Primer (Kerr, Brea, USA) in one direction with a 1.5-mm microbrush (Cotisen®, Huanghua promise dental, Hebei, China), wait 60 seconds, and drying with warm air from a 10-cm distance for 20 seconds. The warm air was calibrated to 60°C using a thermometer (Testo Saveris 2-T3, Testo SE & Co., Germany).

Group 2 (HF+Si+He): same as group 1, additionally, apply a 10-µl Heliobond (Ivoclar Vivadent, Schaan, Liechtenstein) in one direction with a 1.5-mm microbrush (Cotisen®, Huanghua promise dental, Hebei, China) and photopolymerized (Demi™Plus, Kerr, Orange, CA, USA) for 20 seconds

Group 3 (MEP): applying 20-µl Monobond Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein) using a 1.5-mm microbrush (Cotisen®, Huanghua promise dental, Hebei, China), agitate on the surface for 20 seconds, and wait for 40 seconds. Then, thoroughly rinse off with water for 20 seconds and drying the specimen with warm air with the same calibration as used in group 1.

Group4 (MEP+He): same as group 3, additionally, apply a 10-µl Heliobond (Ivoclar Vivadent, Schaan, Liechtenstein) in one direction with a 1.5-mm microbrush (Cotisen®, Huanghua promise dental, Hebei, China) and photopolymerized (Demi™Plus, Kerr, Orange, CA, USA) for 20 seconds

Repair method

A clear silicone mold with 3x3 mm^2 (wide x height) was placed at the center of each specimen to standardize the bonding area. Resin composite (Filtek™ Z350 XT, 3M ESPE, St. Paul, USA) with thickness of 1.5 mm. was applied in the mold and photopolymerized. The another increment with same thickness was then applied to provide 3 mm thickness of resin composite restoration. A light-polymerizing unit (Demi™Plus, Kerr, Orange, CA, USA), with a diameter size of 8 mm, was used to photopolymerize each increment for 40 seconds, the tip of the unit was touched to the mold at an intensity of 1,100 mW/cm^2, the device was calibrated with a radiometer (Optilux Radiometer, Kerr, Orange, CA, USA). After the polymerization, the silicone mold was carefully detached using a scalpel, and excess resin composite material was also gently removed. The specimens were then kept in

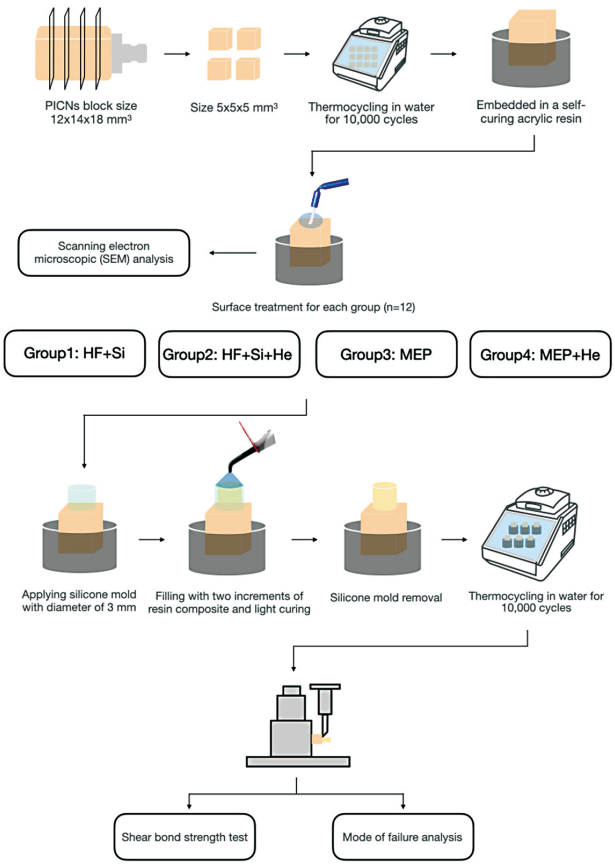

Figure 1: Flow chart of the experimental procedures.

Table 1: Material compositions.

Material	Type	Composition	Lot No.
VITA Enamic® (VITA Zahnfabrik, Bad Säckingen, Germany)	Polymer-infiltrated ceramic network materials (PICNs)	Ceramic content (86% wt, 75% vol): SiO_2 (58-63%), Al_2O_3 (20-23%), Na_2O (9-11%), K_2O (4-6%), B_2O_3 (0.5-2%), ZrO_2<1%, CaO<1% Polymer content (14% wt, 25% vol): UDMA, TEGDMA	99280
Filtek™ Z350 XT (3M ESPE, St. Paul, USA)	Nanofill resin composite	Filler: silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler. Resin: Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA	9783163
PORCELAIN ETCHANT (Bisco, Schaumburg, USA)	Hydrofluoric acid	9.5% Buffered hydrofluoric acid gel	2300001967
Monobond Etch & Prime (Ivoclar Vivadent, Schaan, Liechtenstein)	Self-etching ceramic primer	Butanol, tetrabutylammonium dihydrogen trifluoride, methacrylate phosphoric acid ester, bis(triethoxysilyl) ethane, silane methacrylate, colourant, ethanol, water	Z03CD9
Silane Primer (Kerr, Brea, USA)	Silane coupling agent	Ethanol, (1-methylethylidene)bis[4,1-phenyleneoxy(2- hydroxy-3,1-propanediyl)] bis-methacrylate, Poly(oxy-1,2-ethanediyl), α,α'-[(1- methylethylidene)di-4,1-phenylene] bis[ω-[(2- methyl-1-oxo-2-propen-1-yl)oxy]-, 2,2'-ethylenedioxydiethyl dimethacrylate 3-trimethoxysilylpropyl methacrylate	9730905
Heliobond (Ivoclar Vivadent, Schaan, Liechtenstein)	Light-curing, single-component hydrophobic resin monomer	Bis-GMA, TEGDMA, photoinitiator	Z02TZ2

distilled water at 37°C for 24 hours before thermocycling in water between 5 and 55°C for 10,000 cycles with a 60-s dwell time per bath (THE1400, SD Mechatronik GmbH).

Shear bond strength test

After thermocycling, shear bond strength test was conducted using a universal testing machine (EZ-S500N, SHIMADZU, JAPAN). Each specimen was attached to a metal mold. And was loaded with a crosshead speed of 1 mm per minute and applied at the bonding interface until failure. The bond strength was recorded and calculated by Trapezium 2 program. The mean and standard deviation of shear bond strength in each group were analyzed.

Mode of failure analysis

Fractured specimens were examined under a stereomicroscope (SZ 61, OLYMPUS, JAPAN) to evaluate the failure mode at a magnification of 15X. Modes of failure were classified into 4 types as following: adhesive, cohesive in either the PICNs or resin composite, and mixed failure. The percentage of each mode was calculated based on the total specimens of each group.

Scanning electron microscopic (SEM) analysis

The two representative specimens were subjected to SEM analysis to evaluate topographic change after surface treatment by rinsing with deionized water, drying with oil-free air, sputter coating with a conductive 6-nm gold layer, and analyzing the surface structure with an SEM (JSM-6610LV Scanning Electron Microscope JEOL, USA) at an acceleration voltage 20 kV. Moreover, the two specimens of each group were selected after the shear bond strength test to display the failure surface.

Statistical analysis

Statistical analysis was performed using SPSS

software (IBM SPSS statistics version 29.0.1.0, IBM; Armonk, NY, USA). All data was analyzed with a Shapiro-Wilk to test the normality of data distribution. The One-way ANOVA was used to compare the effect of surface treatment between groups in which the level of confident at 95% was considered to be statistically significant.

Result

Shear bond strength (SBS) and failure mode analysis

SBS values were normally distributed. Mean SBS values and standard deviations each group are shown in Table 2. One-way ANOVA demonstrated a statistically significant difference ($p<0.05$) of mean SBS values among all groups. Group 3 showed the lowest mean SBS values comparing to others. However, groups 1, 2, and 4 did no statistical difference.

The percentage of mode of failure is presented in Figure 2. Adhesive failure was the predominant mode observed exclusively in Group 3. In contrast, Groups 1, 2, and 4 primarily exhibited a mixed mode, although adhesive failure was also presented.

Scanning electron microscopic (SEM) analysis

Representative images of surface topography after each experimental protocol using SEM analysis are displayed in Figures 3. Abundant microporosity between the intact interpolymer network was observed in Group 1 (Figure 3B). Whereas slight surface roughness was observed in Group 3 (Figure 3D) which was similar to thermocyled PICNs surface (Figure 3A). However, a flat and smooth surface was found in either Group 2 (Figure 3C) or Group 4 (Figure 3E). Additionally, surface morphology of representative fracture surfaces was investigated and are presented in Figures 4. Fracture surface at adhesive interface was seen in adhesive failure mode (Figure 4A). For mixed mode of failure, partial fracture of either PICNs or resin composite could be seen as shown in Figure 4B and Figure 4C respectively.

Discussion

During the repair process, surface conditioning of the repaired substrate is the most critical factor determining success. Moreover, different surface conditioning methods can induce distinct topographic changes in various ceramic materials, leading to variations in bond strength.[22] The present study investigated different surface treatment protocols for repairing aged PICNs using shear bond strength test. The result showed that aged PICNs with different surface treatments exhibited different bond performances. The primary finding of the study was that the repairing PICNs with a self-etching ceramic primer alone was insufficient compared to conventional techniques, whereas the additional step of application of hydrophobic resin provided the effective surface treatment before repairing aged PICNs.

In line with the present study, previous research demonstrated that hydrofluoric acid treatment followed by the application of a silane coupling agent was the most effective method for surface treatment of the PICNs, including aged PICNs.[14,15] Eighty percent of PICNs consists of a feldspathic network, which is acid-labile. Hydrofluoric acid partially dissolves the glass-ceramic network, creating a distinct "honeycomb" pattern on material surface, as observed in the SEM image (Figure 3B).[12,23,24] When pre-hydrolyzed silane is applied, its inorganic component reacts with silicon dioxide on the etched glass surface forming siloxane bonds, while the methoxy groups bond with methacrylate-based resins. This silane-treated porosity allows micromechanical interlocking when resin cement is polymerized, resulting in a strong bond (Group HF+Si).[25-27]

Meanwhile, twenty percent of PICNs consists of a patented high-temperature and high-pressure polymerized resin, which resulted in a high degree of conversion of polymer-infiltration.[28] This property may reduce the potential for chemical copolymerization between free

Table 2: Means ± SD of the shear bond strength values (MPa) in each group.

Surface treatment (n=12)	Group 1 (HF+Si)	Group 2 (HF+Si+He)	Group 3 (MEP)	Group 4 (MEP+He)
Shear bond strength (MPa)	21.44±3.58^{A}	21.48±1.64^{A}	10.28±1.87^{B}	19.60±2.12^{A}

Means ± SD in MPa. Different capital letters in each row mean significantly different at $p<0.05$.
HF: Hydrofluoric acid; Si: Silane primer; He: Heliobond; MEP: Monobond Etch & Prime

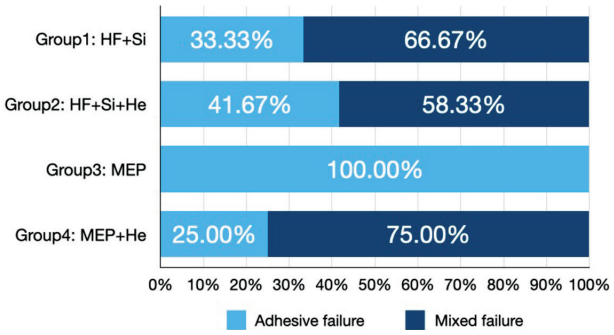

Figure 2: Mode of failure in each group.

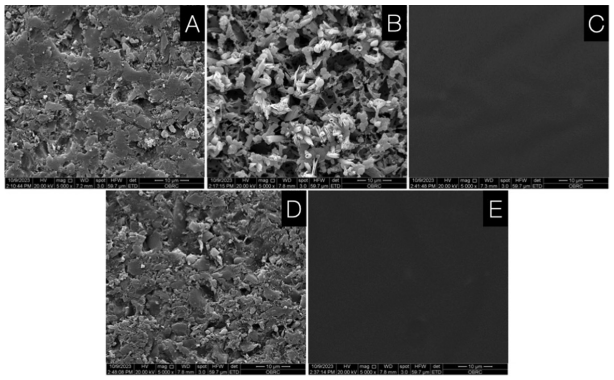

Figure 3: Representative SEM images after surface treatment at 5000X magnification. (A), Surface of aged PICNs presented roughness and narrow valley: (B), Aged PICNs, treated with 9.5% hydrofluoric acid and silane, exhibited numerous microporosities between the intact polymer phase: (C), Aged PICNs, treated with 9.5% hydrofluoric acid and silane followed by resin monomer application, revealed a flat and smooth surface: (D), Aged PICNs, treated with a self-etching ceramic primer, displayed slight surface roughness: (E), Aged PICNs treated with a self-etching ceramic primer followed by resin monomer application showed a smooth surface.

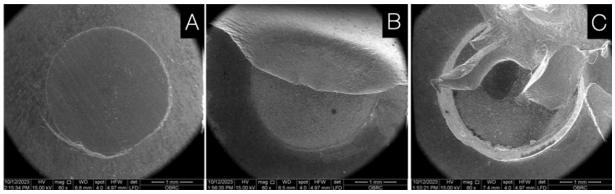

Figure 4: Representative SEM images of specimens after the shear bond strength test reveal different failure modes. (A), Adhesive failure, all the failure occurred only at the materials interface: (B), Mixed failure, partial fracture of the interface was shown and involved in PICNs: (C), Mixed failure, partial fracture at the interface was shown and involved in resin composite.

monomer in the PICNs and the resin-based materials. Additionally, this phase is resistant to hydrofluoric acid as it remained intact shown in Figure 3B, forming etching pattern that effectively facilitate bonding. Therefore, this micromechanical interlocking can be concluded that it has an even greater influence on the adhesive interface's performance compared to chemical reaction.[26]

The creation of sufficient space following glassy dissolution is essential for enhancing surface wettability and ensuring secure micromechanical interlocking between PICNs and resin-based materials. Unlike the HF+Si group, the porosities on PICNs treated with a self-etching ceramic primer (Group MEP) presented minimal surface modification, resembling the untreated surface as seen in Figure 3A and 3D. The main active ingredient in a self-etching ceramic primer responsible for glass-ceramic dissolution is Tetrabutylammonium dihydrogen trifluoride (TADF), which has lower acidic aggressiveness compared to hydrofluoric acid. Due to this milder etching effect, the removal of the glassy phase is limited, resulting in lower surface roughness, as reported in a previous study.[29] The small spaces created by this treatment may hinder the penetration of conventional resin composites, which are highly viscous. This limitation likely explains the significantly lower shear bond strength (SBS) observed in PICNs treated with MEP alone, which was consistent with previous studies showing that ceramic materials treated with MEP exhibited a lower bond strength compared to those treated with HF and silane.[16,30,31]

Numerous studies proposed applying hydrophobic resin monomer coating on silanated ceramic surfaces before repairing them with resin composite to improve the bond between resin composite and ceramic interface.[19,20] The viscosity of hydrophobic resin monomer was less than that of resin composite, providing improved flowability on silanated ceramic surface, filling in small pores and irregularities on surface, resulting in a close adaptation and preventing any defects.[19,20] In recent years, universal adhesives containing silane applied on etched ceramic surfaces have been introduced, claiming their simplified application procedure and fewer clinical steps.[32] However, various studies observed a negative effect on bond durability when multicomponent ceramic primers or bonding agents containing hydrophilic monomers were used to repair ceramic.[4,32-34] A previous study demonstrated that the hydrophilic component in dental adhesive applied to silanated feldspathic ceramics led to a decrease in microtensile bond strength over time, despite initially high values.[33] Therefore, the low-viscosity, hydrophobic resin, Heliobond, which lacks hydro-

philic components, was chosen as an adjunctive surface treatment before repairing the aged PICNs.

The results from HF+Si+He group showed no significant difference from the HF+Si group, indicating that the hydrophobic resin monomer was not necessary for surface treatment of aged PICNs in the scenario when PICNs was treated with HF and silane. This can be explained by the distinctly greater surface roughness on PICNs.[28] The deep and large valley on the PICNs surface may allow the viscous resin composite to adapt closely to the prepared surface, even without a low-viscosity hydrophobic resin layer. However, recent study was reported that the application of a universal adhesive can achieve similar SBS in HF-treated ceramic, despite no additional silane application.[35] Therefore, when conventional hydrofluoric acid was used as a surface treatment, the necessity of an additional hydrophobic coating in ceramic repair remained inconclusive and was required further study.

A self-etching ceramic primer contains not only TADF but also silanes as a single-component system, designed to simultaneously promote siloxane activity on the prepared surface in one application.[16,24,36] However, the acidic nature of the MEP solution raised concerns about the hydrolytic stability of organosilane, potentially reducing its effectiveness.[37] In addition to acidity, rinsing the surface with water after allowing the solution to react may interfere with silanol activity. Despite these concerns, the silane in MEP has been reported to retain silanol activity after immersion in hot water or thermocycling, as demonstrated using micro MIR-FTIR.[29] This stability was likely attributed to the specific component bis(triethoxysilyl) ethane (BTSE), which is more hydrophobic due to the presence of an ethane group in its structure.[38] BTSE enhanced hydrolytic stability and facilitated the effective performance of organosilane.[39] The results from MEP+He group in the present study also proved the retained activity of silane. The low-viscosity hydrophobic resin was able to flow intimately into the material structure, effectively wetting the MEP-treated surface and creating a well-prepared bonding interface for copolymerization with conventional resin composite materials. This led to SBS values from this group comparable to the HF+Si group, indicating the potential of MEP+He in adhesive performance for repairing aged PICNs.

The composition of the additional resin layer should also be considered. Fillers in adhesive agents enhanced mechanical properties[40,41], probably improving bond between the adhesive and the ceramic substrate. However, an increase in filler size and volume raised viscosity[40,41], negatively affecting wettability and limiting resin penetration into micro-porosities, which may compromise bond strength. Therefore, further studies are needed to investigate the impact of filler composition on the bond strength of repaired PICNs.

When compared to a newly restored material, aged material exhibited a significant decrease in bonding performance.[16,18] Thermocycling is the most commonly used method for accelerating aging simulation, particularly for assessing the thermal effect on the bond interface, which could induce material fatigue due to thermal fluctuations.[15,23,24,26,42-44] Several studies have reported differences in bond strength between immediate and 5000-thermocycled PICNs.[42,45] Additionally, a clinical study indicated that the first instance of chipping in PICNs restoration was observed approximately 11.4 months post-insertion.[11] To simulate intraoral condition for one year[11], 10,000 thermal cycles were performed. The incompatibility of the thermal expansion coefficients of different materials may lead to failure in repaired restorations. Moreover, decrease in bond strength values observed after thermocycling could be attributed to water exposure, which negatively affects polymer stability, resulting in resin composite plasticization and, ultimately, hydrolytic degradation.[42] In contrast with the *in vitro* study, oral environmental conditions affected wear and degradation of dental restorations.[46,47] Moisture degraded the siloxane bond, resulting in silane hydrolysis and deteriorating the bond over time.[42,47,48] Therefore, a combination of different accelerating aging processes is suggested for further study.

The difference in mechanical properties between repaired ceramic restorations and the less-stiff resin composite used at the fracture site can generate high tensile stresses at the ceramic-composite interface beneath the loaded area.[46] Therefore, further clinical studies are needed to evaluate the long-term survival of repaired restorations. Additionally, newly developed resin composites, which claim to have higher strength than previous formulations, should be investigated for their potential in repairing hybrid ceramic materials.

Conclusions

Aged PICNs can be effectively repaired using either hydrofluoric acid and silane or a self-etching ceramic primer followed by the application of a hydrophobic resin. However, surface treatment with a self-etching ceramic primer alone may be insufficient for achieving optimal repair of aged PICNs.

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Conflicts of Interest

The authors declare no conflict of interest.

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