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Effect of Tribochemical Coating on Composite Repair Strength

AV Ritter • T Sulaiman • A Altitinchi • F Baratto-Filho • CC Gonzaga • GM Correr

Clinical Relevance

Tribochemical treatment of existing composite surfaces is highly effective for composite repair. When repairing an old composite restoration, the clinician should try to use the same composite originally used for the restoration. If the information about the original restoration is not known, a composite with strong mechanical properties should be used for the repair restoration.

SUMMARY

This study evaluated the effect of tribochemical coating on composite-to-composite repair interfacial fracture toughness (iFT). Sixty beam-shaped specimens (21×4×3 ± 0.2 mm) were prepared with a nanofill composite (Filtek Supreme Ultra [FSU]) and a nanohybrid composite (Clearfil Majesty ES-2 [CME]) and aged for 50,000 thermocycles (5°C-55°C, 20-second dwell time) and then sectioned in half. The resulting 120 hemispecimens (60 for each composite) were randomly assigned to different repair methods (n=10): universal adhesive (Clearfil Universal Bond Quick [CUB]), sandblasting followed by CUB, or tribochemical coating (CoJet, CoJet sand, Espe-Sil, and Visio-Bond). The repair surface was prepared with a diamond bur (Midwest #471271), rinsed, and dried. Each aged composite brand (FSU, CME) was repaired with either the same composite or the opposite composite. All adhesives and composites were light cured with a high-irradiance LED curing light (Elipar DeepCure-S). After postrepair storage in 100% humidity and at 37°C for 24 hours, iFT was measured as K_{Ic} (MPa·m$^{1/2}$). Data were analyzed for statistical significance using two-way analysis of variance (ANOVA) and the Tukey honest significant difference *post hoc* test (α=0.05). Regardless of the substrate composite, ANOVA showed significant differences for surface treatment ($p<0.0001$) and repair composite ($p<0.0001$). Mean iFT values (SD) ranged from 0.91 (0.10) MPa·m$^{1/2}$ to 2.68 (0.12) MPa·m$^{1/2}$. Repairs made with FSU after CoJet resulted in significantly higher iFT (FSU: 2.68 MPa·m$^{1/2}$;

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CME: 2.21 MPa·m$^{1/2}$) when compared to the other experimental groups. The repair iFT was higher with CoJet treatment and when the nanofill composite FSU was used as the repair composite.

INTRODUCTION

When proper application technique is used, light-cured composite resins can be incrementally added to a tooth preparation, resulting in a unified restoration, with strong adhesion in between increments. This is possible because when the material is cured, the composite that is exposed to air is not fully cured since polymerization of composite resins is inhibited by oxygen.[1] This oxygen-inhibited layer, therefore, allows for the incremental placement of multiple layers of composite resin materials.[2,3] While this incremental technique has been recently challenged by bulk-fill techniques, particularly for posterior composite restorations, placing composites incrementally is still recommended when a layered effect is required for optimal esthetics in anterior restorations and when there are polymerization concerns (thoroughness of cure and/or polymerization shrinkage stresses) when light curing a large volume of composite in a single increment.[4-6]

However, when the oxygen-inhibited layer is absent, as when the cured composite has been resurfaced or when an old restoration is being repaired, achieving adequate adhesion between the aged composite and the new composite can be challenging. Repair of composite restorations may be recommended when a localized defect exists in an old (and large) composite restoration that is otherwise serviceable, a shade correction needs to be made, or a modification needs to be made in a restoration that has been placed recently.[7-10] A restoration repair can be advantageous when compared to complete restoration replacement because it can be accomplished in much less time, is less invasive and less prone to endodontic problems, is less costly to the patient, and can increase the longevity of certain restorations.[11]

Adhesion of newly placed composite material to an existing (either old or recently placed) composite restoration has been studied previously, and various surface treatment techniques have been recommended with different degrees of success.[12-15] Airborne particle abrasion with aluminum oxide particles coated with a silicon-dioxide layer has been referred to as tribochemical surface coating,[10,16,17] which results in the silicatization of the surface, enhancing the potential for composite bonding.[18,19] The most popular commercially available tribochemical system is the CoJet System (3M, St Paul, MN, USA), which was adapted from the laboratory-based Rocatec System (3M, formerly ESPE). When the system's sand particles strike the surface being treated, the impact energy generates high energy (triboplasma), and components of the abrasive are incorporated into the treated surface to a depth of 15 μm. The subsequent coating of this surface with a silane and adhesive allows a chemical bond between the treated surface and the repair material.[20,21] This surface treatment method has been recommended not only for composite repair but also for treatment of the intaglio surface of processed composite, ceramic, and alloy-based restorations to increase the adhesion potential for luting and bonding.[10] As it relates to intraoral repair of existing composite restorations, the effect of tribochemical coating on composite repair interfacial fracture toughness (iFT) has not been previously studied.

Adhesion of restorative materials to enamel and dentin and between restorative materials are typically assessed with mechanical tests, such as the microtensile and shear bond strength tests.[22] However, these mechanical tests rarely yield a truthful assessment of the adhesive interface.[23] To overcome this limitation, iFT has been suggested to be a more accurate and reproducible laboratory mechanical test to assess adhesive interfaces, as it allows more focused stress concentration at the interface when compared to microtensile and shear bond strength tests.[24,25]

Therefore, the purpose of this study was to evaluate the effect of tribochemical coating on composite-to-composite repair iFT using the single-edge V-notch beam method. We hypothesized that tribochemical surface treatment does not improve the iFT of composite repairs when compared to a 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) universal adhesive alone and to conventional sandblasting followed by the 10-MDP adhesive.

METHODS AND MATERIALS

Materials used in the study as well as their manufacturer information, composition, and instructions for use are listed in Table 1. A total of 60 beam-shaped specimens (21×4×3 ± 0.2 mm as per ISO 6872:2015 of the International Organization for Standardization) were prepared with two composite resin substrate materials—a nanofilled composite (Filtek Supreme Ultra [FSU], 3M) and a nanohybrid composite (Clearfil Majesty ES-2 [CME], Kuraray Noritake, New York, NY, USA) at room temperature

Table 1: *Materials Used in the Study*

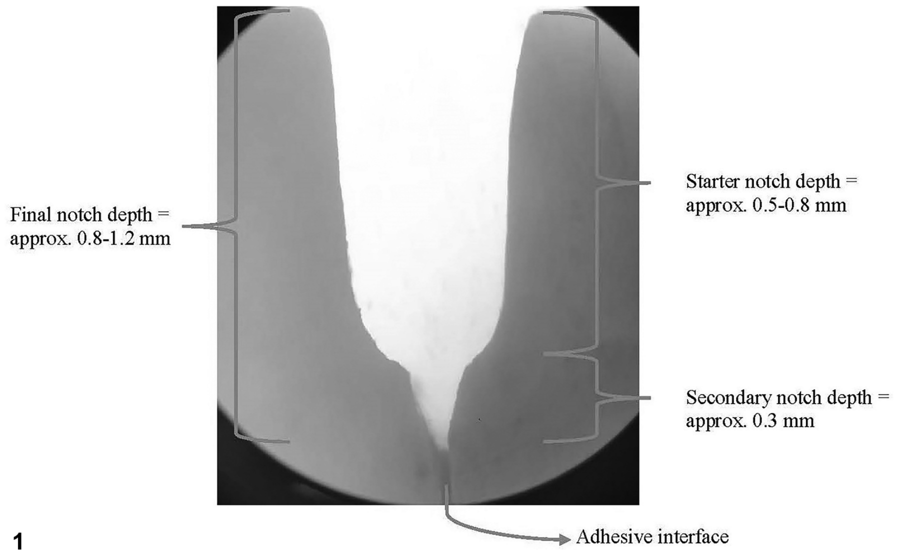
Material	Summarized Composition	Instructions for Use
Clearfil Universal Bond Quick (CUB) (Kuraray Noritake)	2-hydroxyethyl methacrylate, bisphenol A diglycidyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophilic amide monomers, colloidal silica, silane, sodium fluoride, and camphorquinone in ethanol and water	Apply with rubbing motion; air-dry for 5 s; light cure for 5 s
Sandblaster (MB11 MicronBlaster)	Sand: 30-μm aluminum oxide particles	Apply sand with 2-3 bar (30-42 psi) pressure for 10 s at ~10-mm distance
CoJet (3M)	CoJet sand: 30-μm silicatized aluminum oxide and synthetic amorphous silica particles; Espe-Sil: silane, ethanol; Visio-Bond: bisacrylate, aminodiol methacrylate, camphorquinone, benzyl dimethyl ketale, stabilizers	Apply CoJet sand with 2-3 bar (30-42 psi) pressure for 10 s at ~10-mm distance; apply Espe-Sil and let dry for 30 s; apply thin layer of Visio-Bond and light cure for 20 s
Clearfil Majesty ES-2 (CME) (Kuraray Noritake)	Nanohybrid composite resin, 78%/wt filled, shade A1	Apply incrementally; light cure for 20 s
Filtek Supreme Ultra (FSU) (3M)	Nanofill composite resin 72.5%/wt filled, 0.6-10 microns average cluster particle size, shade A1-b	Apply incrementally; light cure for 20 s

(23°C±1°C) using polyvinylsiloxane molds. This sample size was used based on previous studies by the authors.[26] The specimens were built incrementally and light cured through a 0.85-mm glass slab with an ELIPAR DeepCure-S LED Curing Unit (3M). The irradiance reaching the material (through the glass slab) averaged 1196 mW/cm^2. Three overlaps were performed for each beam. The center was cured first, followed by each end. Each section of the beam was cured for 20 seconds and then flipped over and cured for a total of six overlaps. The total energy of each overlap was calculated at 24.12 J/cm^2 under the glass slide and 25.16 J/cm^2 without the glass slide. All light-curing measurements were performed using the MarcLight Collector (BlueLight Analytics, Halifax, NS, Canada). Once cured from one side, each beam specimen was removed from the mold, light cured from the opposite side, and lightly wet polished manually with 600-grit silicon carbide abrasive paper (CarbiMet, Buehler Ltd, Lake Bluff, IL, USA) to eliminate composite flash and any residual matrix-rich composite from the specimen's surface. The composite resin substrate beam specimens were subject to accelerated aging (Thermocycling TC-4, SD Mechatronik, Feldkirchen-Westerham, Germany) for 50,000 cycles (5°C-55°C, 20-second dwell time) to simulate five years of intraoral use.[27] After thermocycling, the beams were cut into two halves using a precision saw (Isomet 4000, Buehler), resulting in a total number of 120 composite resin substrate beams (60 for each composite brand) with dimensions (10.5×3×4 ± 0.1 mm).

For all specimens, the surface to be repaired (one end of the aged composite beam) was roughened for five seconds with a coarse diamond bur (Midwest #471271, Midwest Dental, Wichita Falls, TX, USA) at high speed held manually, and specimens were randomly assigned to one of the following repair groups (n=10), according to 1) surface treatment (multifunctional 10-MDP adhesive, Clearfil Universal Bond Quick [CUB], Kuraray Noritake, and sandblasting followed by CUB or CoJet system) and 2) repair composite (FSU or CME).

When sandblasting was used, the substrate composite to be repaired was sandblasted from a 10-mm distance with 50-μm aluminum oxide particles at 2-bar pressure and rinsed. After sandblasting, CUB was applied according to the manufacturer's instructions (see Table 1) and light cured with the same light-curing unit and parameters described earlier. When the CoJet system was used, prior to bonding, the substrate composite to be repaired was sandblasted from a 10-mm distance at 2-bar pressure using the CoJet sand (30 μm); following sandblasting, the surface was coated with Espe-Sil and Visio-Bond, which are components of the CoJet system (see Table 1). The total energy from the light-curing unit to both adhesives was calculated at 12.92 J/cm^2 (10-second cure time at 1-mm distance).

Surface-treated hemispecimens were returned to the silicone molds and carefully seated to leave half of the mold empty. Fresh composite resin (either FSU or CME) was applied to the empty section of the mold and cured similarly to the method described



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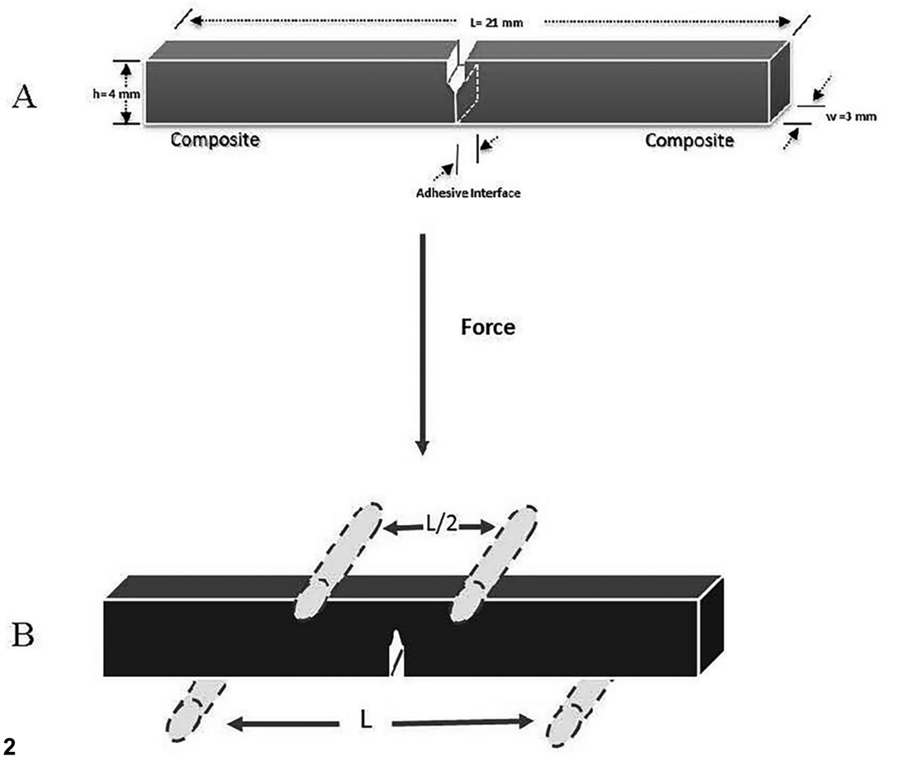

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Figure 1: *Representative stereomicroscopy cross-section of the notch placed at the adhesive interface.*

Figure 2: *Schematic representation of the specimen and test used for the interfacial fracture toughness test. A. Specimen geometry (upside down). B. Test set up (v-notch facing down). Please note the v-notch is not to scale and is magnified here for illustration purposes only.*

previously, using the same light-curing parameters and energy. All adhesives and composites were applied and light cured according to manufacturers' instructions (Table 1). Repaired specimens were lifted from the molds and once again lightly wet polished manually with 600-grit silicon carbide abrasive paper (CarbiMet, Buehler Ltd) to eliminate composite flash and any residual matrix-rich composite from the repair side of the specimen's surface.

Under a stereomicroscope (Olympus BX41, Olympus, Center Valley, PA, USA), the repaired interface was marked with a starter notch approximately 0.5 mm deep into each specimen using a 150-μm-thick diamond blade running in the precision saw (Isomet 4000, Buehler Ltd). Specimens were held manually for this step so as to exert control over this initial notch. Diamond polishing paste (3.5 μm, Kent Supplies, Quebec, QC, Canada) was then placed into the notch tip, and a new 0.12-mm-thick GEM Single Edge carbon steel blade (Ted Pella, Inc, Redding, CA, USA) was placed into the starter notch with light manual force (5-10 N) using a gentle back-and-forth motion as straight as possible. The force applied to create both the initial and the final notches was standardized via pilot studies using a custom-built pressure monitor device.[28] The Olympus BX41 stereomicroscope was again used to examine and measure both ends of the V-notch for evenness of depth and to ensure that a sharp crack was formed. The final notch was uniform and between 0.8 and 1.2 mm deep (Figure 1). A diagram of the complete specimen geometry is depicted in Figure 2A.

The notched specimens were tested for iFT using a four-point bending fixture (Figure 2B). The 3-mm-wide face with the V-notch was placed down (tensile side), and the specimens were loaded on a universal testing machine (Instron 4411, Instron, Norwood, MA, USA) with a crosshead speed of 0.5 mm/min at room temperature in air. The fracture load was recorded to three significant digits. The width (b) and thickness (w) of each specimen were recorded using a micrometer (Digimatic Micrometer, Mitutoyo Corporation, Kawasaki, Japan). The depths of the V-notches were measured using a calibrated microscope with magnification >50× to three significant digits. The iFT K_{Ic} (MPa·m$^{1/2}$) was calculated based on previously published methodology[29] using the following equation (ISO 6872:2015):

$$K_{Ic} = \frac{F}{b\sqrt{w}} \cdot \frac{S1 - S2}{w} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} \cdot Y$$

where K_{Ic} = fracture toughness;

F = fracture load;

b = specimen width;

w = specimen thickness;

$S1$ = support span;

$S2$ = inner four-point span;

α = relative notch depth, $\alpha = \alpha_1 + \alpha_2 + \alpha_3/3$ (the average of three notch depth measurements divided by the specimen thickness, measured with a stereomicroscope); and

Y = stress intensity shape factor, calculated as

$$Y = 1.9887 - 1.326\alpha - \frac{(3.49 - .68\alpha + 1.35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2};$$

where α = depth of sample/depth of notch;

For each substrate composite, data for iFT were statistically analyzed using two-way analysis of variance (ANOVA; surface treatment and repair composite). Tukey *post hoc* tests were used to further compare mean iFT between the various groups. All analyses were performed with a 0.05 significance level.

RESULTS

The mean iFT values ± SD ranged from 0.91 ± 0.10 to 2.68 ± 0.12 MPa·m$^{1/2}$ and are presented in Table 2A,B. When the nanofill composite FSU was the substrate composite (Table 2A), the ANOVA showed significant differences for surface treatment ($p<0.0001$) and repair composite ($p<0.0001$). The surface treatment × repair composite interaction was also statistically significant ($p<0.0001$). Tukey *post hoc* revealed a statistically significant difference in mean iFT values between adhesive only (0.96 MPa·m$^{1/2}$), sandblasting + adhesive (1.09 MPa·m$^{1/2}$), and CoJet (2.27 MPa·m$^{1/2}$). Regarding the repair composite, FSU showed statistically higher mean iFT (1.66 MPa·m$^{1/2}$) than CME (1.21 MPa·m$^{1/2}$) when FSU was the substrate composite. FSU substrate composite repaired with FSU composite after CoJet treatment resulted in statistically significantly higher iFT (2.68±0.12 MPa·m$^{1/2}$) when compared to all other groups (0.87±0.10 – 1.86±0.23 MPa·m$^{1/2}$).

For the nanohybrid substrate composite CME (Table 2B), the ANOVA also showed significant differences for surface treatment ($p<0.0001$) and repair composite ($p<0.0001$). However, the surface treatment × repair composite interaction was not statistically significant ($p=0.103$). Tukey *post hoc* revealed a statistically significant difference in mean iFT values between adhesive only (1.10 MPa·m$^{1/2}$) and CoJet (2.03 MPa·m$^{1/2}$) and between sandblasting + adhesive (1.05 MPa·m$^{1/2}$) and CoJet (2.03 MPa·m$^{1/2}$) but not between adhesive only and sandblasting + adhesive. Regarding the repair composite, the nanofill composite FSU showed statistically higher mean iFT (1.50 MPa·m$^{1/2}$) than CME (1.28 MPa·m$^{1/2}$) when CME was the substrate composite. CME substrate composite repaired with FSU composite after CoJet treatment resulted in statistically significantly higher iFT (2.21±0.36 MPa·m$^{1/2}$) when compared to all other groups (1.00±0.17 – 1.84±0.24 MPa·m$^{1/2}$).

DISCUSSION

Composite repair has experienced increased popularity with evidence published in the past decade demonstrating the benefits of composite repair over complete restoration replacement.[7,30-32] While there

Table 2A: *Interfacial Fracture Toughness (K_{Ic}, MPa·m$^{1/2}$ ± SD) Results for the Nanofill Composite Filtek Supreme Ultra (FSU) as a Substrate According to Surface Treatment and Repair Composite (n=10)*[a]

Substrate Composite	Surface Treatment	Repair Composite	Interfacial Fracture Toughness
FSU	Clearfil Universal Bond Quick (CUB)	FSU	1.05 ± 0.13 A
	Sandblast + CUB		1.26 ± 0.09 B
	CoJet		2.68 ± 0.12 C
	CUB	Clearfil Majesty ES-2	0.87 ± 0.10 A
	Sandblast + CUB		0.91 ± 0.10 A
	CoJet		1.86 ± 0.23 D

[a] *Letters indicate means that are statistically similar (p>0.05).*

Table 2B: *Interfacial Fracture Toughness (K_{Ic}, MPa·m$^{1/2}$ ± SD) Results for the Nanohybrid Composite Clearfil Majesty ES-2 (CME) as a Substrate According to Surface Treatment and Repair Composite (n=10)*[a]

Substrate Composite	Surface Treatment	Repair Composite	Interfacial Fracture Toughness
CME	Clearfil Universal Bond Quick (CUB)	Filtek Supreme Ultra	1.20 ± 0.13 A
	Sandblast + CUB		1.09 ± 0.11 A
	CoJet		2.21 ± 0.36 B
	CUB	CME	1.00 ± 0.17 A
	Sandblast + CUB		1.01 ± 0.13 A
	CoJet		1.84 ± 0.24 C

[a] *Letters indicate means that are statistically similar (p>0.05).*

is increased consensus about this technique, the literature is still not unanimous when it comes to what is the best surface treatment approach to repair existing composite restorations because the absence of the oxygen-inhibited layer prevents new composite material from completely adhering to an existing restoration.[3,33,34]

One of the limitations of laboratory measurement of adhesion strength is the inherent lack of clinical relevance of most mechanical tests, including shear and microtensile bond strength tests.[35,36] The iFT test has been proposed as a more relevant laboratory mechanical test to measure the strength of adhesive interfaces.[25,37] Since to the best of the authors' knowledge iFT has never been used to assess composite repair strength, this study evaluated the effect of surface treatment (sandblasting followed by adhesive application, adhesive application only, and CoJet), composite substrate type, and composite repair type on composite repair iFT utilizing the single-edge V-notch beam test setup. The null hypothesis advanced—that tribochemical surface treatment does not improve the iFT of composite repairs when compared to conventional sandblasting with a 10-MDP adhesive or with the adhesive alone—was not accepted, as tribochemical surface treatment resulted in significantly higher iFT mean values when compared to the other repair methods.

Several surface treatment techniques and adhesive systems have been recommended for composite repair, including roughening with a coarse diamond bur, acid etching, use of composite primers and silane agents, sandblasting, and using mechanical retention. Research also shows that tribochemical coating, which consists of airborne particle abrasion with aluminum oxide particles coated with a silicon-dioxide layer (CoJet), is effective in composite repair.[10] Our results are in agreement with other studies showing that tribochemical coating is indeed a very favorable surface treatment method for intraoral composite repair.[17,38,39] Although, as noted by Loomans and others,[17] composite repair rarely achieves the same strength as the cohesive strength of the composite material, the results of this study showed that significantly higher repair iFT were obtained when the substrate was treated with the CoJet system regardless of the substrate or repair composite used (Table 2). It is important to note, however, that the CoJet system was used in this study as a system; that is, CoJet sand was followed by Espe-Sil silane and Visio-Bond hydrophobic adhesive. It would be important for future studies to evaluate the separate contribution of each of these components, primarily the CoJet sand and Espe-Sil silane, to the results obtained.

Encouragingly, all iFT values obtained with the repair techniques tested were higher than the mean

Table 3: *Mechanical Properties of the Composites Used in the Study*[a]

	Filtek Supreme Ultra	Clearfil Majesty ES-2
Fracture toughness,[b] MPa·m	0.82	0.80
Compressive strength, MPa	360	356
Volume shrinkage, %	2	1.9
Flexural strength, MPa	165	118
Flexural modulus of elasticity, MPa	11,000	10,000
Vickers hardness, VHN	60	Not available

[a] *Information provided by the manufacturers.*
[b] *Data from pilot study by the authors, unpublished.*

fracture toughness values of the substrate composites (pilot study, unpublished data; FSU: 0.82±0.03 MPa·m$^{1/2}$; CME: 0.80±0.13 MPa·m$^{1/2}$), indicating that the 10-MDP adhesive with or without sandblasting and the CoJet treatment are effective in repairing these aged composite substrates. However, it is critical to note that these fracture toughness values were obtained in a pilot study by the authors (unpublished) and do not represent iFT since it is not possible to test iFT in specimens with no interface. Therefore, these data were not used as a control in the study reported here but rather are discussed in this context to illustrate the efficacy of the repair techniques tested.

Another technique that often is discussed in the context of composite repair is the use of a surface etchant (phosphoric acid) to clean the surface to be repaired. In a previous study by the authors, an additional phosphoric acid etching step had no positive (or negative) effect on the iFT of repaired specimens; therefore, for this study, this step was omitted.[26] Given that many of the modern dental adhesives, including the universal adhesive used in this study, are slightly acidic,[40,41] it is possible that their pH is already acidic enough to self-etch these surfaces, making a separate acid etch step with phosphoric acid unnecessary.

The adhesive used (Clearfil Universal Bond Quick) is a 10-MDP–based self-etch adhesive. It is a functional monomer that has potential to ionically interact with calcium in hydroxyapatite and form hydrolytically stable 10-MDP-calcium salts through a self-assembled nanolayered interaction.[42,43] Although this process is not applicable to composite-composite interfaces because of its applicability to dentin (and enamel) bonding, 10-MDP–based adhesives are commonly used clinically, and hence their effect on composite-composite bonding should be investigated. In a previous study by the authors, two other 10-MDP–based adhesives (Clearfil SE Bond, Kuraray; Scotchbond Universal, 3M) were shown to have improved composite repair iFT when compared to no-adhesive controls,[26] indicating that 10-MDP adhesives are effective in repairing aged composite substrates. Furthermore, 10-MDP is a solvating monomer that can penetrate into a cross-linked network and provide trapped C=C that may bond to the repair composite resin.

Regardless of the substrate composite used, the highest repair strength values were obtained with FSU composite (Table 2A,B), which, based on information provided by the manufacturers, has higher mechanical properties than CME (Table 3). Since the type of composite being repaired is not often known, this finding supports the notion that a standard repair adhesion protocol using a composite with robust mechanical properties can be followed regardless of the composite substrate being repaired. Polymerized composite resins are highly cross-linked, and after preparing the composite resin surface for repair, a smear layer will form, blocking any unreacted C=C bond that may be present at the surface. Therefore, vigorous efforts must be used to clear the surface from the smear layer in addition to using adhesives that contain solvents that may help penetrate the prepared surface to achieve adequate adhesion.

One limitation of the current study is that only two composite resins and one dental adhesive were tested. It is therefore not possible to generalize the results to the wide variety of composites and adhesives currently available. Additionally, rarely will clinically repaired interfaces be geometrically flat and completely independent of enamel and/or dentin adjacent interfaces. These adjacent surfaces in theory contribute to a stronger overall restoration repair strength, as these interfaces do not occur in isolation. Finally, although the crack propagation clearly followed the adhesive interface with no deviation, further studies using fractography and Weibull analyses could provide valuable information on specimen fabrication and help refine the mechanical test approach.

Despite these limitations, the results of this study strongly support the use of tribochemical surface treatment when aged composite restorations are being repaired. Additionally, the results further support the single-edge V-notch beam method as an appropriate model for testing of dental interfaces, as it allows stress concentration at the adhesive interface.

CONCLUSIONS

Under the limitations of this laboratory study, composite repair strength is surface treatment and composite dependent but was significantly higher when CoJet was used for surface treatment. Composite repair strength also was higher when FSU was the repair composite regardless of the substrate composite, particularly when CoJet is used as the surface treatment.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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