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Composite-composite Adhesion as a Function of Adhesive-composite Material and Surface Treatment

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Clinical Relevance

When repairing aged (old) composite, etching with phosphoric acid for 20 seconds does not appear to improve composite-composite repair strength. Composite and adhesive types have a significant effect on composite-composite repair strength. When repairing aged (old) composite, matching the repair composite to the old composite that is being repaired appears to make no difference.

SUMMARY

Objective: To evaluate the composite-to-composite repair interfacial fracture toughness (iFT) as a function of adhesive and composite repair material.

Methods and Materials: Beam-shaped composite specimens (21×4×3±0.2 mm) were prepared for each substrate material (Filtek Supreme Ultra [FSU] or Clearfil Majesty ES-2 [CME]) and artificially aged for 50,000 thermocycles (5-55°C, 20-second dwell time). Aged specimens were sectioned in half, and the resulting hemispecimens were randomly assigned to one of the different repair methods (n=10) based on the following variables: type of substrate composite (FSU or CME), acid etch (yes or no), adhesive type (Scotchbond Universal or Clearfil SE Bond 2), and type of repair composite (FSU or CME). The repair surface was prepared with a course diamond bur (Midwest #471271). When used, 37% phosphoric acid was applied for 20 seconds, rinsed, and dried. All adhesives and composites were applied according to manufacturers' instructions. After postrepair storage (100% humidity, 37°C, 24 hours), iFT was measured and expressed as MPa. Data were analyzed for statistical signif-

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icance using a three-way analysis of variance and Tukey *post hoc* tests (α=0.05).

Results: iFT values ranged from 0.64 ± 0.19 MPa to 1.28 ± 013 MPa. Significantly higher iFT values were achieved when FSU was used as the repair composite resin regardless of the substrate composite resin ($p<0.001$). Clearfil SE Bond 2 adhesive was associated with significantly higher iFT values for FSU substrate ($p<0.001$). The etching procedure had no significant effect on the iFT values of the repair procedures ($p>0.05$).

Conclusions: Composite repair strength is adhesive and composite dependent. Repair strength appears to be higher when FSU is the repair composite regardless of the adhesive used.

INTRODUCTION

It has been extensively documented that between 40% and 70% of all restorations done in a general dental practice are replacements of failed restorations.[1] Although the longevity of dental restorations is dependent on many different factors, including material-, diagnostic-, patient-, and dentist-related factors,[2] secondary caries is the most common cause of restoration failure.[3-6] At the tooth level, secondary caries is defined as caries adjacent to an existing restoration,[7,8] and it can be found on the restorative interface, on the tooth surface immediately adjacent to the restoration, or both.[9-11]

Secondary caries is typically "treated" by replacement of the failed restoration, with very few resources being utilized to address its etiology and, importantly, to ensure that the new restoration has a better chance of survival. Given that disease prevalence is the best indicator of disease incidence, unless the patient's caries risk is addressed, secondary caries tends to be a recurring event.[12,13] The cycle of restoration replacement can then be extended throughout the life of the tooth, leading to increasingly larger restorations and eventually to the compromise of the restoration-tooth complex and ultimately tooth loss.[14]

The repair of failed restorations has been proposed as a conservative alternative to complete restoration replacement.[3,6,15,16] When the secondary caries fault is localized, which is commonly the case, careful excision of the compromised tooth structure adjacent to the existing restoration and repair of the site with a compatible restorative system present many advantages over the replacement of the entire restoration. Compared to complete restoration replacement, a repair can be accomplished in much less time, is less invasive and less prone to endodontic problems, is potentially less costly to the patient, and can increase the longevity of certain restorations.[17]

When repairing an existing direct composite resin restoration, the success of the repair will depend not only on the interaction of the repair material with the tooth structure but also on its interaction with the existing restoration. Current adhesive systems have been extensively tested relative to their interaction with enamel and dentin,[18-21] but their ability to predictably and successfully bond to aged composite, which is critical for a successful repair, has not been fully investigated.[22-24]

The microtensile bond strength test has been widely used as a standard method for testing bond strength to dentin.[25] However, modern dental adhesives have progressed significantly, leading to failures that are more cohesively within the composite or dentin rather than the actual interface, raising concerns about its methodology.[26] Various versions of interfacial fracture toughness (iFT) methods for *in vitro* testing of dentin bond strength have been reported in the literature, including short rod chevron notch, notchless triangular prism, chevron notch, and mini-interfacial fracture toughness. These tests appear to be more accurate and reproducible, revealing the interfacial properties better and being less test dependent.[27]

Therefore, the purpose of this project was to study the effect of composite substrate type, composite repair type, and adhesive protocol on the iFT (utilizing the Single-Edge V-Notch Beam method)[28] of composite-composite adhesion mediated by adhesives containing the functional monomer 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). We hypothesized that the composite type and adhesion protocol have no effect on the ability of 10-MDP–containing adhesives to repair aged composite resin. We also tested the null hypothesis of no association between each treatment and iFT, that is, that the regression coefficient corresponding to the treatment is equal to zero.

METHODS AND MATERIALS

This *in vitro* study measured and compared the composite-to-composite iFT as a function of composite substrate material, substrate treatment (etch,

Table 1: *Materials Used in the Study*

Clearfil SE Bond 2 (CSE) (Kuraray Noritake)	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photoinitiator, water Bond: 10-MDP, Bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller	Apply primer for 20 s; gently air-dry; apply bond; gently air-dry; light cure for 10 s
Scotchbond Universal (SU) (3M)	10-MDP, dimethacrylate resins, HEMA, Vitrebond copolymer, ethanol, water, filler, initiators, silane	Apply adhesive for 20 s; gently air-dry; light cure for 10 s
Phosphoric Acid Etch (Ultradent)	37% phosphoric acid viscous gel	Etch substrate for 15-20 s; rinse thoroughly; gently air-dry
Clearfil Majesty ES-2 (CME) (Kuraray Noritake)	Nanohybrid composite resin, 78%/wt filled	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	Apply incrementally; light cure for 20 s

adhesive), and composite repair material using the Single-Edge V-Notch Beam (SEVNB) method according to standard 23146 of the International Organization for Standardization. Materials used in the study as well as their manufacturer information, composition, and instructions for use are listed in Table 1.

A total of 120 beam-shaped specimens (21×4×3±0.2 mm) were prepared for each of the composite resin substrate materials (Filtek Supreme Ultra, 3M, St Paul, MN, USA; Clearfil Majesty ES-2, Kuraray Noritake, New York, NY, USA) at room temperature (23±1°C) using polyvinylsiloxane molds. The specimens were built incrementally and light cured with an ELIPAR DeepCure-S LED Curing Unit (irradiance 1420 mW/cm^2 and radiant output 989 mW; 3M). The composite resin substrate beam specimens were subject to accelerated aging (Thermocycling TC-4, SD Mechatronik, Feldkirchen-Westerham, Germany) for 50,000 cycles (5-55°C, 20-second dwell time) to simulate five years of intraoral use.[29] After thermocycling, the beams were cut into two halves, resulting in a total number of 240 composite resin substrate beams with dimensions (10.5×3×4 mm±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 course diamond bur (Midwest #471271) at high speed, and specimens randomly assigned to one of the repair groups (n=10). Independent variables tested were as follows:

1) Type of substrate composite (Filtek Supreme Ultra [FSU] or Clearfil Majesty ES-2 [CME])
2) Etching (or not) of the repair surface with 37% phosphoric acid gel (Ultradent, South Jordan, UT, USA)
3) Adhesive used for the repair procedure (Scotchbond Universal [SU] or Clearfil SE Bond 2 [CSE])
4) Type of repair composite (FSU or CME)

When used, phosphoric acid gel was applied for 20 seconds and thoroughly rinsed, and the etched surface was air-dried. All adhesives and composites were applied according to manufacturers' instructions and were light cured.

Repaired specimens were stored in 100% humidity and 37°C for 24 hours before testing. A notch approximately 0.5 mm deep was machined into each specimen at the repaired interface using a 150-μm-thick diamond blade. Diamond polishing paste (3.5 μm, Kent Supplies, Quebec, QC, Canada) was then placed into the notch tip, and a new razor blade was placed into the starter notch and a light force (5-10 N) applied using a gentle back-and-forth motion as straight as possible. A light microscope was used to examine both ends of the V-notch occasionally 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.

The notched specimens were tested for iFT using a four-point bending fixture. 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. Interfacial fracture toughness K_{Ic} (MPa * m$^{1/2}$) was calculated using the following

Table 2A: *Interfacial Fracture Toughness Results for Filtek Supreme Ultra (FSU) Substrate Composite According to Surface Treatment, Repair Adhesive, and Repair Composite (n=10)*[a]

Substrate Composite	Etch	Repair Adhesive	Repair Composite	Interfacial Fracture Toughness (MPa±SD)
FSU	Yes	None	FSU	0.98 ± 0.21 BC
FSU	Yes	SU	FSU	1.01 ± 0.15 BC
FSU	Yes	CSE	FSU	1.17 ± 0.18 AB
FSU	No	None	FSU	1.21 ± 0.18 A
FSU	No	SU	FSU	1.03 ± 0.09 ABC
FSU	No	CSE	FSU	1.15 ± 0.11 AB
FSU	Yes	None	CME	0.64 ± 0.19 D
FSU	Yes	SU	CME	0.82 ± 0.12 CD
FSU	Yes	CSE	CME	1.00 ± 0.07 BC
FSU	No	None	CME	0.72 ± 0.11 D
FSU	No	SU	CME	0.92 ± 0.07 C
FSU	No	CSE	CME	0.90 ± 0.11 C

Abbreviations: SU, Scotchbond Universal (3M); CSE, Clearfil SE Bond 2 (Kuraray Noritake); CME, Clearfil Majesty ES-2 (Kuraray Noritake).
[a] Means followed by same capital letter are not significantly different with a significance level of 0.05

Table 2B: *Interfacial Fracture Toughness Results for Clearfil Majesty ES (CME) Substrate Composite According to Surface Treatment, Repair Adhesive, and Repair Composite (n=10)*[a]

Substrate Composite	Etch	Repair Adhesive	Repair Composite	Interfacial Fracture Toughness (MPa±SD)
CME	Yes	None	FSU	0.86 ± 0.15 DE
CME	Yes	SU	FSU	1.07 ± 0.14 BC
CME	Yes	CSE	FSU	1.28 ± 0.13 A
CME	No	None	FSU	0.92 ± 0.19 CDE
CME	No	SU	FSU	1.07 ± 0.08 BC
CME	No	CSE	FSU	1.12 ± 0.10 AB
CME	Yes	None	CME	0.88 ± 0.12 DE
CME	Yes	SU	CME	0.97 ± 0.08 CD
CME	Yes	CSE	CME	0.88 ± 0.14 DE
CME	No	None	CME	0.80 ± 0.12 E
CME	No	SU	CME	0.93 ± 0.06 CDE
CME	No	CSE	CME	0.90 ± 0.11 CDE

Abbreviations: SU, Scotchbond Universal (3M); CSE, Clearfil SE Bond 2 (Kuraray Noritake); CME, Clearfil Majesty ES-2 (Kuraray Noritake).
[a] Means followed by same capital letter are not significantly different with a significance level of 0.05

equation:

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

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

where K_{IC} = fracture toughness, α = relative notch depth, Y = stress intensity shape factor, F = fracture load, b = specimen width, w = specimen thickness, $S1$ = support span, and $S2$ = inner four-point span. For each substrate composite, data for iFT were statistically analyzed using three-way analysis of variance (etching, adhesive, and repair composite). Tukey *post hoc* tests were used to further compare mean iFT between the various groups. All analyses were performed with a significance level of 0.05.

RESULTS

The mean iFT values (±SD) ranged from 0.64 (±0.19) MPa to 1.28 (±0.13) MPa and are presented in Tables 2A and 2B.

For FSU used as substrate composite (Table 2A), the analysis of variance showed significant differences for adhesive ($p<0.0001$) and repair composite ($p<0.0001$). The double interactions etching*adhesive ($p=0.0015$) and adhesive*repair composite ($p<0.0001$) were also significant. Etching ($p=0.1059$), the double interaction etching*repair ($p=0.1206$), and the triple interaction ($p=0.4403$) were not statistically significant. For the etching procedure, the mean iFT was similar for etching (0.94 MPa) and no etching (0.98 MPa). As for the adhesive used for the repair procedure, CSE showed higher mean iFT (1.31 MPa) than SU (0.95 MPa) and no adhesive (0.89 MPa), which were statistically similar. Regarding the repair composite, FSU (1.09 MPa) showed statistically higher mean iFT than CME (0.86 MPa) when FSU was the substrate composite.

For substrate composite CME (Table 2B), the analysis of variance showed significant differences for adhesive ($p<0.0001$) and repair composite ($p<0.0001$). The double interaction etching*repair ($p<0.0001$) and the triple interaction ($p=0.0021$) were also significant. Etching ($p=0.1018$), the double interactions etching*adhesive ($p=0.6438$), and etching*repair ($p=0.8943$) were not statically significant. For the etching procedure, mean iFT was similar for etching (0.99 MPa) and no etching (0.95 MPa). As for the adhesive used for the repair procedure, CSE (1.03 MPa) and SU (1.00 MPa) showed statistically similar mean iFT values, both higher than no adhesive (0.87 MPa). Regarding the repair composite, FSU (1.05 MPa) showed statistically higher mean iFT than CME (0.91 MPa).

DISCUSSION

Direct composite restorations frequently fail because of restoration fracture and/or secondary caries.[12,30-34] When either restoration fracture or secondary caries occurs, often a direct composite repair can be accomplished rather than the replacement of the entire restoration, increasing its longevity.[17] When repairing a composite restoration intraorally, the adhesion protocols may differ from those used on enamel and dentin. This study evaluated the effect of composite substrate type, composite repair type, and adhesive protocol on the iFT, utilizing the SEVNB method, of composite-composite adhesion mediated by adhesives containing the functional monomer 10-MDP. The hypothesis advanced, that is, that the composite type and adhesion protocol have no effect on the ability of 10-MDP containing adhesives to repair aged composite resin, was rejected, as composite type and adhesion protocol did significantly affect composite-to-composite iFT values. The iFT values were reproducible and consistent testing the bonding interface.

Both composite type and adhesive type had a significant effect on the outcome, while the use of phosphoric acid etching did not. The effects of phosphoric acid on enamel and dentin and its role in the adhesion process have been thoroughly documented.[22] However, phosphoric acid use in composite-to-composite adhesion and repair has not been fully studied. While it is expected that at the very minimum acid etching of the surface to be repaired would remove any smear layer that may have been generated by the surface preparation, we failed to demonstrate any beneficial effect of the phosphoric acid etching step. Polymerized composite resin may be etched with the use of hydrofluoric acid, which was not investigated in this study and should be further researched to determine any benefits for its use in repairing composite resin restorations. Although the adhesives tested are not nearly as acidic as phosphoric acid, it is possible that their pH is already acidic enough to self-etch these surfaces (phosphoric acid pH=0.1-0.4; CSE pH=2.3; SU pH=2.7), making a preliminary acid etch step unnecessary.

10-MDP is a functional monomer that can ionically interact with calcium in hydroxyapatite and form hydrolytically stable 10-MDP-calcium salts through a self-assembled nanolayered interaction.[21,35] 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. Most iFT values obtained with the repair techniques tested were similar or higher than the mean fracture toughness values of the substrate composites (FSU: 0.82±0.03 MPa; CME: 0.80±0.13 MPa; pilot study, unpublished data), 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.

It is important to note that the fracture toughness values reported above for the substrate composites were obtained in a pilot study by the authors (unpublished) and represent *fracture toughness*, not 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.

Interestingly, when aged FSU was repaired with FSU without acid etch or adhesive, the mean repair strength was not statistically different from the highest mean repair strength obtained in the study (1.21±0.18 MPa; Table 2). For this specific combination of substrate and repair composite (FSU-FSU), acid etching actually resulted in significantly lower mean repair strength values (0.98±0.21 MPa), which may indicate that etching (or insufficient rinse of the etching agent) may result in contamination of the surface and compromise the repair strength. Furthermore, the finding that an adhesive may not be needed when repairing an aged composite is not in full agreement with other similar studies.[16,36-39] This appears to be particularly true for FSU when the repair composite was the same as the substrate composite. We can only speculate on why this was the case, but it may be related to the chemistry of this particular material.

Regardless of the substrate composite and adhesive used, the highest repair strength values were obtained with FSU composite (Table 2). Clinicians often do not know the type of composite they are repairing; therefore, this finding supports the notion that a standard repair adhesion protocol can be followed regardless of the composite substrate being repaired. It is important to keep in mind that polymerized composite resins are highly cross-linked and that 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 fractured surface to achieve any sort of bonding.

An important challenge of this study is the fact that a positive control could not be easily tested. While one could hypothetically use a nonrepaired composite group as a positive control, as discussed above, it would not be entirely appropriate to compare the fracture toughness of a material with the iFT of the repaired specimens. Other possible positive control groups could have been included, such as silanization and air abrasion as surface treatment. These popular composite repair surface treatments, such as silanization with or without air abrasion, as shown by Loomans and others,[16] can also be very effective for composite repair, but they were not tested in this study because we wanted to test only the specific variables tested (substrate, etch, and adhesive). Additionally, these are interventional surface treatments and therefore not technically appropriate positive control groups. Another limitation of the study is that only two composite resins and two dental adhesives were tested. It is therefore not possible to generalize the results to the wide variety of composites and adhesives currently available. Finally, rarely will clinically repaired interfaces be geometrically flat and completely independent of enamel and/or dentin adjacent interfaces. These adjacent surfaces will contribute to a stronger overall restoration repair strength, as these interfaces do not occur in isolation.

CONCLUSIONS

Under the limitations of this *in vitro* study, composite repair strength is adhesive and composite dependent but appears to be higher when FSU is the repair composite regardless of the adhesive used. Phosphoric acid etch has no effect on composite repair strength. The SEVNB method introduced may be suggested as a valid method to test the interfacial fracture toughness of bonding interfaces.

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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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