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Influence of Mechanochemical Treatment and Oxygen Inhibited Layer on the Adhesion of Self-Adhesive Resin Cement to Bulk-Fill Composite Resin

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ABSTRACT

Objectives: This study evaluated the shear bond strength (SBS) of self-adhesive resin cement (SARC) to bulk-fill composite resin (BFCR) following mechanical and chemical surface treatments.

Materials and Methods: The BFCR discs fabricated were divided into four groups, based on the presence or absence oxygen inhibited layer (OIL) and mechanical surface treatment, as follows; group I: OIL+no surface treatment (NT); group II: no OIL+NT; group III: no OIL+diamond abrasive (DA); and group IV: no OIL+air abrasion (AA). Each group was further divided into two subgroups based on chemical treatment using a silane agent. Following this, the SARC cylinders were bonded to the surfaces of the treated BFCR samples. SBS was evaluated for all the samples, and failure analysis was carried out. The data were analysed using an independent t-test, one-way ANOVA and post hoc Tukey test and a *p* value of <0.05 was considered to indicate statistical significance.

Results: The highest SBS was recorded in group IV (no OIL+AA) without silane application (25.66±4.49 MPa), while the lowest was observed in group I (OIL+NT) with silane treatment (0.4±0.24 MPa). Mechanical surface treatment succeeded in significantly improving the SBS, while chemical surface treatment using silane application failed to do so.

Conclusions: Mechanical surface treatment via abrasion enhanced the bonding ability of BFCR with SARC. However, OIL and chemical treatment using a silane agent did not improve the SBS.

Keywords: Air abrasion, Composite resin, Resin cement, Shear bond strength, Silane.

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Introduction

Composite resins are commonly used as core build-up materials. Incrementally cured composites suffer from void incorporation and interlayer contamination.[1] In an attempt to overcome these shortcomings, bulk-fill composite resins (BFCR) have been introduced with the claim of greater depths of curing in deeper increments.[2]

Dental composites light-cured in air possess a sticky superficial layer of unreacted monomers and oligomers known as the oxygen inhibition layer (OIL).[3] The OIL forms an interdiffusion zone where materials from both sides blend to copolymerize, producing chemical bonds.[4] Reports on the effect of this layer on bond strength have been inconsistent.[3,4]

Partial adhesive restorations aimed at preserving healthy tooth structures can be bonded to composite-restored teeth using self-adhesive resin cements (SARCs). Eliminating the need for a separate etching and bonding procedure, SARC has emerged as an economical alternative with regard to both time and chair-side costs. These materials retain the mechanical properties and bonding ability of conventional resin cements.[5]

For indirect restorations to be successful, a durable bond between the luting cement and the core composite is essential. Surface contamination of the core composite by saliva and temporary luting agents diminishes its ability to foster chemical changes.[6] Mechanical and chemical surface treatments restore some of these abilities prior to the luting of indirect restorations. Various techniques, such as air abrasion, roughening with a diamond abrasive point, and silanization, have been attempted but have produced inconsistent results.[7,8]

The bonding of SARC to restorative composite resin has been inadequately probed. This study, therefore, aimed to assess the shear bond strength of SARC adhered to BFCR subjected to various surface treatments. The null hypotheses investigated were that the presence or absence of OIL, as well as mechanical and chemical

surface treatments, would not impact the bond strength of SARC to the BFCR.

Materials and Methods

Study design and approval

The *in vitro* study was conducted after obtaining approval (IEC No. 21046) from the Institutional Ethics Committee of MCODS, Mangalore, on July 11, 2021.

Sample size calculation

Referring to a prior study conducted by Ghivari *et al.*[9], which compared composites cured with OIL to those cured without OIL, the essential parameters for assessing the role of OIL in shear bond strength included a 5% alpha error, a study power of 95%, and a clinically significant difference of 2 units. Based on these parameters, the required sample size in each group was determined to be 5.

Preparation of composite cylinders

Cubic acrylic moulds (3.5x1.5x1.5 cm) housing cylindrical slots were used to fabricate forty BFCR (Tetric N-Ceram Bulk Fill, Ivoclar Vivadent, Liechtenstein) cylindrical samples with dimensions of 6 mm in diameter and 2 mm in height. With a single increment of 2 mm in depth, the BFCR was filled into the moulds, leaving a single exposed surface.

In order to form OIL, ten samples were light-cured for 45 seconds in air at a light intensity of 800 mW/cm2 (Bluephase, Ivoclar Vivadent, Liechtenstein). The remaining 30 samples were light-cured in air for 30 seconds, followed by additional anaerobic curing for 15 seconds through the application of a glycerine gel to form the OIL.

Storage and aging

All the samples were stored in artificial saliva for 7 days at 37°C. Artificial saliva used composed of 0.4gram NaCl, 1.21gram KCl, 0.0005gram Na2S.9H2O, 0.7gram NaH2PO4.2H2O and 1gram CO(NH2), with all components being dissolved in 1000 ml deionized water with pH corrected to 6.75±0.75 using 0.1 N NaOH.

Grouping

The samples were randomly divided into four groups (n=10) based on the surface treatments. The details of the materials used are described in Table 1. Figure 1 summarizes the methodology.

Group I (OIL+NT): BFCR were cured in contact with air and not subjected to mechanical surface treatment to maintain OIL.

Group II (No OIL+NT): Anaerobically cured BFCR not subjected to mechanical surface treatment.

Group III (No OIL+DA): Anaerobically cured BFCR was roughened using a diamond bur (TF-12 Diamond Abrasive Point- medium grit, SS White, USA) with a slow-speed handpiece (NSK Ltd., Japan) for 10 seconds.

Group IV (No OIL+AA): Anaerobically cured BFCR treated with air abrasion and white alpha aluminium oxide particles for 15 seconds.

Each group was further divided into two subgroups (n=5): (A) no chemical treatment with a silane agent and (B) chemical treatment with a silane agent (Monobond N, Ivoclar Vivadent, Liechtenstein).

To obtain self-adhesive resin cement (SARC) cylinders, polyethylene moulds with a diameter and height of 3 mm and 4 mm, respectively, were positioned over the treated composite resin surfaces, into which the SARC (SpeedCem Plus, Ivoclar Vivadent, Liechtenstein) was injected and light-cured for 30 seconds.

The samples were stored a second time in artificial saliva for three days until shear bond strength (SBS) analysis commenced.

Shear bond strength testing

A universal testing machine (Instron Corporation, Canton, MA) at a crosshead speed of 0.5 mm/min was used to test samples for SBS after they were positioned into a jig. The strengths were calculated and converted to megapascal (MPa) by dividing the failure load expressed in Newtons (N) by the bonded area per square millimetre (mm^2).

Analysis of the failure modes

Fracture analysis of the adhesive surfaces was performed under a stereomicroscope at 20x magnification (Stereo Star Zoom-570, Reichert, New York, USA), and the failures were categorized as cohesive (fracture within the BFCR or SARC), adhesive (fracture at the adhesive interface between the BFCR and SARC) or mixed (simultaneous occurrence of adhesive and cohesive failures).

Statistical analysis

The SBS values obtained were tabulated and analysed using software (SPSS Version 20, IL, USA). Independent t-test was used to evaluate the effect of silane application in various subgroups. One-way ANOVA and post hoc Tukey tests were used to determine the level of significance among the experimental groups. A p value of <0.05 was considered to indicate statistical significance. Additionally, the chi-square test was performed to assess the significance of the difference in the type of failure among the various groups.

Table 1: *Materials used in the study and their description*

Materials	Manufacturer details	Composition	Usage
Air abrasion unit	PrepStart air abrasion system, Danville, San Ramon, CA, USA.	-27 micrometre white alpha aluminium oxide particles	-Operated at 80 psi pressure. -The composite surfaces to be bonded were air abraded with the alumina particles in circular sweeping motion for 15 seconds per sample.
Bulk-fill composite	Tetric N-Ceram Bulk Fill, Ivoclar Vivadent, Schaan, Liechtenstein.	-Dimethacrylates (21% by weight): Bis-GMA, Bis-EMA and UDMA -Polymer filler: 17.0 % by weight -Barium glass filler, Ytterbium trifluoride, Mixed oxide: 61.0 % by weight -Additive, Initiators (Ivocerin, Acyl phosphine oxide, Camphoroquinone), Stabilisers, Pigments: <1.0 % by weight	-Composite was light-cured in a single increment of 2 millimetres depth. (According to the manufacturer, can be cured in increments of up to 4mm.) -Light activation was performed for 40 seconds.
Silane	Monobond-N, Ivoclar Vivadent, Schaan, Liechtenstein.	-Alcohol solution of: -Silane methacrylate, -Phosphoric acid methacrylate, and -Sulphide methacrylate	-Composite surfaces were thoroughly rinsed with water spray and dried with water and oil-free air. -Monobond-N was applied onto the surfaces to be bonded using a microbrush and allowed to react for 60 seconds. -It was then be dispersed with a strong stream of air.
Self-adhesive resin cement	SpeedCem Plus, Ivoclar Vivadent, Schaan, Liechtenstein.	-Monomer matrix: Dimethacrylates and acidic monomers. -Inorganic fillers (40% by volume, size 0.1-7µm): barium glass, ytterbium trifluoride, co-polymer and highly dispersed silicon dioxide. -Additional contents (<1%): initiators, stabilisers and colour pigment	-For each application, a new automix tip was placed on the double syringe. -SpeedCem Plus was extruded from the automix syringe, the desired quantity applied directly onto the composite surface. -It was then light-cured for 30seconds with a light intensity of 800mW/cm².

Bis-GMA: bisphenol A-glycidyl methacrylate, Bis-EMA: Ethoxylated bisphenol A dimethacrylate, UDMA: Urethane Dimethacrylate

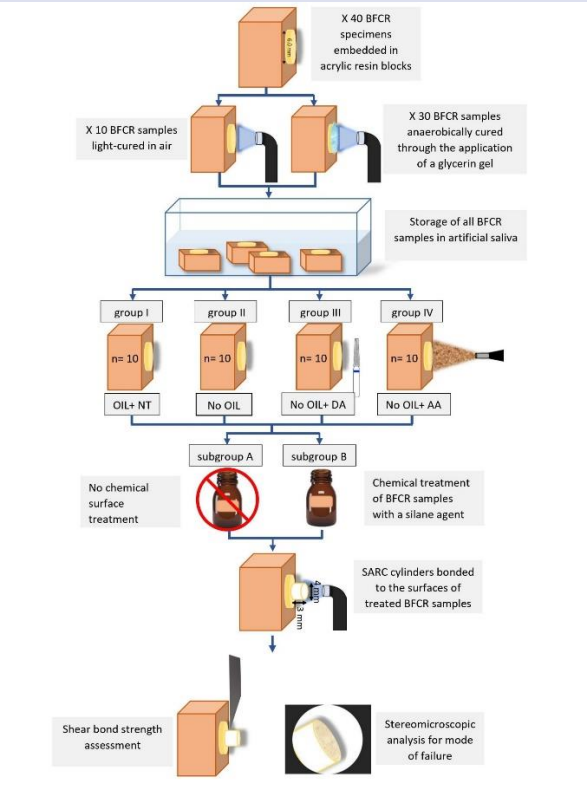

Figure 1: Schematic diagram of the methodology

Results

Shear bond strength test (SBS)

The highest SBS value was observed in Group IV-A (25.66±4.49 MPa), while the lowest was noted in Group I-B (0.4±0.24 MPa). A comparison of the SBS between the subgroups in each of the major groups by one sample t-test revealed statistically significant differences only for Group I (p = 0.038) (Table 2).

The SBS of all the subgroups, when compared using one-way ANOVA, demonstrated a statistically significant difference (p <0.001). A post hoc Tukey test revealed that the differences in SBS between groups IA, IB, IIA, and IIB were not statistically significant. Similarly, groups IIIA, IIIB, IVA, and IVB were not statistically significant (p >0.05). However, groups IA, IB, IIA, and IIB demonstrated significantly lower SBS than IIIA, IIIB, IVA, and IVB (p <0.001). Thus, irrespective of the subdivision, the mechanically treated samples (Group III and IV) revealed statistically significantly higher SBS than the groups where no surface mechanical alteration was done before SARC placement (Group I and II) (Table 2).

Analysis of the mode of failure

Kappa statistics demonstrated excellent inter-observer reliability (κ=0.911). The chi-square test demonstrated a significant difference in the mode of failure among the groups (χ^2=25.846, p =0.027). The samples in groups I and II exhibited exclusive adhesive failure. Groups III and IV displayed predominantly mixed failures. Cohesive failure was noted to be the least common occurrence. (Figure 2)

Table 2: Descriptive and analytical data of shear bond strength (MPa) values

Group	Subgroup	Range	Mean ± SD*	t	p value
Group I	A (No Silane)	1.71 to 8.95	4.59±3.09 ^{A}	3.025	0.038
(OIL+ NT)	B (Silane)	0.21 to 0.73	0.4±0.24 ^{A}		
Group II	A (No Silane)	0.78 to 5.27	3.11±1.62 ^{A}	0.45	0.666
(No OIL+NT)	B (Silane)	1.28 to 3.78	2.69±1.04 ^{A}		
Group III	A (No Silane)	15.45 to 25	20.64±3.92 ^{B}	1.475	0.178
(No OIL+DA)	B (Silane)	21.73 to 26.18	23.45±1.7 ^{B}		
Group IV	A (No Silane)	20.39 to 30.04	25.66±4.49 ^{B}	1.926	0.106
(No OIL+AA)	B (Silane)	18.39 to 23.58	21.41±2.06 ^{B}		

OIL: Oxygen inhibited layer, NT: no surface treatment, DA: diamond abrasive, AA: air abrasion, t= Independent t-test value; p= Probability value
* Different superscript letters indicate statistical significance within all the sub-groups (p <0.05).

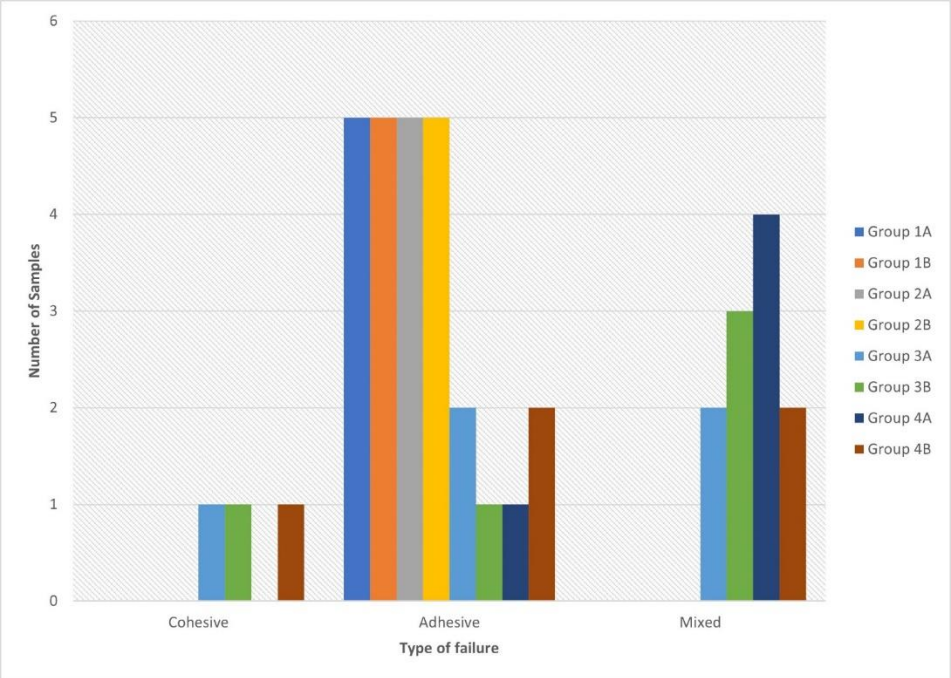

Figure 2: Distribution of failure modes in specimens from various groups

Discussion

The presence of unreacted acrylates in the oxygen inhibition layer (OIL) led to the assumption that this layer is a prerequisite for promoting bond formation between aged and fresh composite resins.[10] In this study, there were no significant differences between the bond strengths measured in the presence or absence of OIL, even without mechanical surface treatment of the BFCR. Hence, the first part of the null hypothesis had to be accepted. This finding implies that the presence of OIL is essential for the bonding of BFCR to SARC. The depletion of the photoinitiator camphoroquinone in the OIL may account for this difference, as may the presence of acidic monomers in SARC, rendering it incompatible with the OIL.[5,9,10]

The insufficient bond strengths observed in the groups not mechanically surface-treated may be an outcome of artificial saliva aging. Water sorption induces hydrolytic degradation in the resin matrix and eventual inactivation of the matrix. The presence of TEGDMA molecules in resin-based composites is largely associated with water sorption. These factors heighten the susceptibility of the BFCR to hydrolytic degradation, robbing its ability to form a clinically acceptable bond with the luting agent.[11,12] Feeble bonding between the BFCR and SARC in the samples of these two groups is also reflected in adhesive failure being the sole prevalent mode of failure here.

The deposition of a whitish precipitate on the BFCR samples may also have contributed to the decrease in bond strength. These fissured, semi-transparent precipitates appeared on the surface of the BFCR after a week of immersion in artificial saliva. Söderholm *et al.*[13] reported the presence of an unexpected whitish-yellow semi-transparent precipitate on the surface of composites stored in artificial saliva. A similar precipitate was detected by Gregson *et al.*[12] Using scanning electron microscopy and inductively coupled plasma atomic emission spectrometry, they concluded that the precipitate consisted of calcium and phosphate ion deposits. The precipitate detected in the present study is likely of a similar nature, and when left undisturbed, an immaculate bond between the BFCR and SARC was impeded.

Chemical bonding of composite resins to SARC occurs chiefly through the bonding of methacrylate monomers. The reduced availability of unreacted monomers on the cured composite surface may hinder the bonding mechanism.[14,15] Saliva contamination in the oral environment causes the leaching of unreacted monomers. Surface treatment of aged resin composites removes the saliva-altered superficial layer, creating surface irregularities to improve the available area for the cement to bond.[16]

Air abrasion, through nonselective degradation of the composite resin, creates an irregular surface enhancing micro-retentive features in the form of grooves and pits. This helps fortify the bond with the resin cement.[17] In the present study, air abrasion with 27 µ aluminium oxide particles without salinization of the composite surface yielded the highest bond strength. Previously conducted studies assessing the bond strength of repaired composites and that between a composite resin and luting cement corroborate these results.[9,16-19] Silane treatment of air-abraded composite surfaces, though insignificant, did not significantly degrade the bond strength. This result is consistent with that of a prior study, and this could be attributed to the lower inorganic content of the bulk-fill composite as well as the additional components of the multimode silane coupling agent.[20]

Treatment with a diamond abrasive point did not significantly differ between SBS from the air-abraded group, a finding supported by earlier studies.[8,15] This difference may be due to the similar micro-retentive features and mechanical interlocking produced by both techniques. Both treatments were highly effective at potentiating the bonding between the BFCR and SARC; hence, the second part of the null hypothesis was rejected. In the mechanically surface-treated groups, a rise in mixed fractures was observed at the interface of the two materials, accompanied by few cohesive failures. This finding supports the precedence of mechanically surface-treating the BFCR before it binds to the SARC.

Silane coupling agents are bifunctional molecules capable of promoting chemical adhesion between two dissimilar materials.[21] Studies exploring the efficacy of silane have offered no congruent verdicts. In the present study, no significant changes in bond strength could be discerned following surface treatment with a silane agent. The third aspect of the null hypothesis therefore cannot be rejected. Moreover, the OIL+NT group exhibited a significant reduction in bond strength following silane application. This result is concordant with reports from a prior study by Guiterrez *et al.*[22]

Silane coating and air drying of the composite surface create two distinct layers: the innermost chemisorbed layer, which is siloxane bonded to the composite surface, and the outer physiosorbed layer, which contains few siloxane bonds. Only the former contributes to the coupling mechanism, and the presence of the latter in excess may prove detrimental to the bonding procedure.[23] A thick, multiphase interfacial layer formed due to silane treatment impairs the intimate interaction of the methacrylate monomers of the SARC with the polymerized composite resin polymers.[22] The precipitate deposited on the composite surface during storage in artificial saliva might have consolidated this interfacial layer, further weakening the bond between the BFCR and SARC in mechanically surface-treated groups.[12,13] The present study demonstrated that chemical surface treatment alone fails to achieve satisfactory bond strength. Even as an adjunct to mechanical means, it does not prove beneficial and can be omitted.

Despite attempts to standardize all the laboratory techniques employed in this study, we cannot use duplicate the *in vivo* conditions. To predict the long-term behaviour of the materials tested, the approaches adopted should mimic oral conditions as closely as possible. Contamination of the BFCR samples using temporary luting agents prior to any surface treatment and adopting thermocycling as the aging procedure will help better align the results obtained with an actual clinical situation.

Conclusions

Within the limitations of the current study, the following conclusions can be drawn:

- The presence or absence of an oxygen inhibition layer does not impact the bond strength of bulk-fill

composite resins (BFCR) to self-adhesive resin cement (SARC).

- Mechanical surface treatment of the BFCR enhanced its bonding ability with the SARC.
- Chemical surface treatment of BFCR using a silane agent does not improve the bond strength of the material to the SARC.

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