Bond Strength between Luting Materials and a Fiber-reinforced Resin Composite for Indirect Restorations

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The purpose of the present study was to evaluate the bond strength between a fiber-reinforced composite (FRC) and six luting agents (Panavia F 2.0, Linkmax MC, Chemiace II, Multibond, Super-Bond C&B, and Fuji I). A prosthodontic resin composite material (RC-control) and a luting agent containing no functional monomer (MT) were used as controls. Shear bond strengths between alumina-blasted FRC and the luting agents were determined after 20,000 thermocycles. The FRC showed superior bond strength when compared with the RC-control. Highest bond strengths were achieved when FRC was bonded with Panavia F 2.0, Linkmax MC, Multibond, Super-Bond C&B, and MT, whereas Chemiace II trailed in the list of resin-based luting agents evaluated. Insufficient bonding was obtained with Fuji I. Results of the present study revealed that when fabricating restorations, the clinician should select an appropriate combination of resin composite material and luting agent so as to ensure the longevity of restorations.

Keywords: Adhesion, Cement, Glass fiber

INTRODUCTION

Light-polymerized indirect resin composites are one useful option for patients who desire esthetic dental treatment. However, clinicians occasionally encounter problems and difficulties when working with resin composite restorations, such as detachment, fracture, and wear. In the severe oral environment, proper bonding between a composite restoration and the luting material is thus of paramount importance to ensure long-term use of the restoration[1,2].

Fiber-reinforced resin composites have been studied in the context of fabricating laminate veneers[3,4], onlays[5], crowns[6,7], denture bases[8,9], fixed partial dentures[10-13], and root canal posts[14]. They were characterized by flexibility and fracture resistance[15,16]. The fiber materials used were glass, quartz, carbon, or polyethylene[17-20]. In particular, pre-impregnated fiber-reinforced resin composites showed better physical properties and easier handling than manually mixed fiber-reinforced resin composites[21,22]. Further, for some preimpregnated fiber-reinforced resin composites employing short glass fibers with high elastic modulus, tensile strength, toughness, and impact resistance have been improved[23,24]. Against this background, these materials are expected to be useful in constructing resin-bonded fixed partial dentures as well as resin composite crowns[25-27].

The bonding properties of a fiber-reinforced resin composite were evaluated based on bond strength after thermocycling[28]. The bond strengths of resin luting agents to fiber-reinforced composite posts were higher than those to zirconium oxide posts[29], and were significantly affected by luting agent[29,30]. It was found that the interfacial bonding between an experimental fiber-reinforced resin composite (FRC) containing milled glass fiber and a light-curing resin composite was strong enough to prevent delamination[25]. It was also reported that the FRC improved the toughness and impact resistance of the indirect resin composite. However, no information is available concerning the adhesive bonding between luting materials and this FRC material.

The purpose of the present study was to evaluate the bond strength between the FRC and six luting materials, and then compare the FRC to a conventional resin composite. When compared to conventional resin composites, the FRC is characterized by its higher rate of methacrylate monomers in addition to the unique mechanical properties mentioned above. Therefore, the tested hypothesis was that the luting materials bond to the FRC more strongly than to the conventional resin composite.

MATERIALS AND METHODS

Materials

The materials used in the present study are summarized in Table 1. FRC[25] (Sun Medical Co. Ltd., Moriyama, Japan) and a microfilled resin composite (RC-control; New Metacolor Infis Paste A3-B, Sun Medical Co. Ltd.) were used as the substrate materials. Seven luting agents were used: Panavia F 2.0 (PF; Kuraray Medical Inc., Tokyo, Japan), Linkmax MC (LM; GC Corp., Tokyo, Japan), Chemiace II (CA; Sun Medical Co. Ltd.), Multibond (MB; Tokuyama Dental Corp., Tokyo, Japan), Super-Bond C&B (SB; Sun Medical Co. Ltd.), an experimental resin luting agent (MT), and Fuji I (FI;

Table 1   Materials used in the present study

Material (Abbreviation)	Component ( )		Manufacturer	Irradiation time (seconds)	Lot No.
Substrate materials:					
Fiber-reinforced composite	UDPAC	41.73	Sun Medical Co. Ltd.	90	041027
(FRC)	TEGDMA	27.82	Moriyama, Japan		040803
	Silanized milled-glass fiber	24.84			
	Colloidal silica	4.97			
	Camphorquinone	0.30			
	Dimethyl aminobenzoic acid 2-*n*-butoxy ethyl	0.30			
	Hydroquinone monomethylether	0.04			
New Metacolor Infis	UDMA		Sun Medical Co. Ltd.	90	LS1
Paste A3-B (RC-control)	TEGDMA				
	Reactive prepolymerized filler	42*			
	Dimethyl aminobenzoic acid 2-*n*-butoxy ethyl				
	Hydroquinone monomethylether				
	Others				
Luting agents:					
Panavia F 2.0	Paste A: MDP, Methacrylate monomer,		Kuraray Medical Inc.	20	00095A
(PF)	Filler, Photo initiator, Chemical initiator		Tokyo, Japan		
	Brown Paste B: Methacrylate monomer,				00087A
	Filler, NaF, Photo initiator, Chemical initiator				
	Oxyguard : Poly(ethylene glycol), Accelerator				00491A
Linkmax MC	Paste A: Aluminosilicate glass powder,		GC Corp.	20	0409271
(LM)	Methacrylate		Tokyo, Japan		
	Paste B: Aluminosilicate glass powder,				0409271
	Methacrylate				
Chemiace	Liquid: Multifunctional methacrylate, HEMA		Sun Medical Co. Ltd.	0	LG-1
(CA)	4-META, BPO				
	Powder: Zirconia, Silica, Amine				LM1
Multibond	Liquid: MAC-10, MMA, Amine,		Tokuyama Dental Corp.	0	325
(MB)	Multifunctional methacrylate		Tokyo, Japan		
	Clear powder: PMMA, BPO				509
Super-Bond C B	Initiator: TBB		Sun Medical Co. Ltd.	0	LW62
(SB)	Monomer liquid: 4-META, MMA				KE3
	Clear powder: PMMA				KL1
Experimental	Initiator: TBB		Sun Medical Co. Ltd.	0	LW62
(MT)	Monomer liquid: MMA		Wako Pure Chemical		TWQ5264
			Industries Ltd., Osaka, Japan		
	Clear powder: PMMA		Sun Medical Co. Ltd.		KL1
Fuji	Powder: Fluoroalumino silicate glass,		GC Corp.	0	0410051
(FI)	Poly (acrylic acid)				
	Liquid: Poly (acrylic acid), Distilled water,				0401211
	Carboxylic acid derivative				

UDPAC: urethane dimethacrylate with poly-aliphatic carbonate segment, TEGDMA: triethyleneglycol dimethacrylate, UDMA: 2-methyl-2-propenoic acid 1,1'-[7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl] ester, MDP: 10-methacryloxydecyl dihydrogen phosphate, HEMA: 2-hydroxyethyl methacrylate, 4-META: 4-methacryloxyethyl trimellitate anhydride, BPO: benzoil peroxide, MAC-10: 11-methacryloxyundecan-1,1-dicarboxylic acid, TBB: tri-*n*-butylborane derivative, MMA: methyl methacrylate, PMMA: poly(methyl methacrylate).   *Fraction of inorganic filler.

GC Corp.).

The organic components of FRC were dissolved in acetone for 60 minutes, and the glass fibers were then air-dried and observed with a color 3D laser microscope (VK-8700, Keyence, Osaka, Japan) at a magnification of × 1,000 (Fig. 1). The FRC contained silanized milled glass fibers (average diameter: 11 μm; average length: 150 μm) and colloidal silica. The silane coupling agent used contained 3-trimethoxysilylpropyl methacrylate. On the other hand, RC-control contained a reactive prepolymerized filler[31] (TMPT filler). The TMPT filler was a composite filler copolymerized with poly (trimethylol propane trimethacrylate). An RC-control sample was burned out at 400 for 30 minutes (Ring Furnace, J. Morita Tokyo MFG. Corp., Saitama, Japan) in order to calculate the ratio of the inorganic components. Weight of the burned-out sample divided by the original weight indicated the percentage of inorganic components.
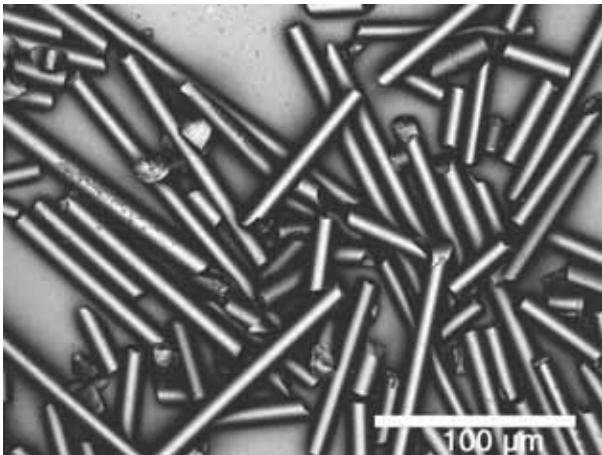

Fig. 1   3D laser micrograph of the milled glass fibers contained in the FRC.
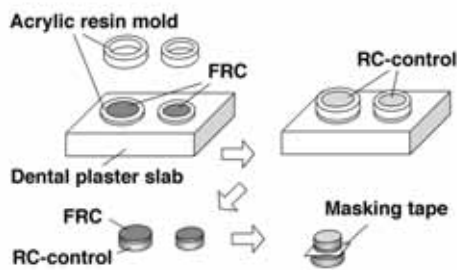

Fig. 2   Schematic illustration of specimen preparation.

Specimen preparation

An experimental separating agent consisting of -polyolefin, *n*-heptane, and pigment (Sun Medical Co. Ltd.) was applied to a dental plaster slab (New Fujirock Fast Set, GC Corp.). Two sizes of cylindrical acrylic resin mold (diameter: 8 mm, thickness: 0.5 mm and diameter: 7 mm, thickness 0.5 mm) were placed on the slab and filled with FRC (Fig. 2). The FRC was then polymerized with a photopolymerizing unit (-Light II, Morita Corp., Tokyo, Japan) for 90 seconds. Additional cylindrical acrylic resin molds (diameter: 8 mm, thickness: 2.5 mm and diameter: 7 mm, thickness: 2.5 mm) were placed concentric on the first molds. The space was filled with RC-control on the FRC paste and then polymerized with the photopolymerizing -Light II unit for 90 seconds. After removing the cylindrical acrylic resin molds, disk specimens of two sizes were obtained. Disk specimens of RC-control without FRC were fabricated as a control in the same manner.

All disks were air-abraded (Jet Blast III, Morita Corp.) with 50μm alumina (Hi-Aluminas, Shofu Inc., Kyoto, Japan) for five seconds. An air pressure of 0.2 MPa was applied, and the orifice was positioned approximately 10 mm from the specimen surface. A piece of 50μm-thick masking tape with a circular hole was positioned on the disk specimen to control the bonding area to 5 mm in diameter. FRC surfaces of the specimens were then bonded with a luting agent in accordance with the manufacturer's directions. As for LM in the present study, no composite primer was used in order to preclude the additional effect of adhesion promoting monomers and to simplify the bonding system.

Shear bond strength test

At 60 minutes after specimen preparation, a total of 168 bonded specimens were immersed in water at 37 for 24 hours. Half of the specimens were subjected to a thermocycling process (4 and 60 alternately, 60-second dwell time for 20,000 cycles) using a thermocycling apparatus (Rika Kogyo, Tokyo, Japan). Shear testing was carried out at a crosshead speed of 0.5 mm/minute on a universal testing machine (AGS-10kNG, Shimadzu, Kyoto, Japan). Shearing load was applied parallel to the bonded interface. Six specimens were tested for each condition.

Statistical analysis

Data were analyzed by three-way analysis of variance (ANOVA) to assess the significance of interaction among three factors thermocycling, substrate, and luting agent. Mean values for each condition were compared by a *post hoc* Fisher's Protected LSD test following one-way ANOVA. P-values of 0.05 were regarded as significant.

Fracture mode

The debonded surfaces of all specimens were observed through an optical microscope (SMZ-10, Nikon Corp., Tokyo, Japan) at a magnification of × 20. Failure modes were categorized into six groups: adhesive failure at the interface between the luting agent and resin composite (A), cohesive failure within the luting material (C), fracture within the resin composite (F), and three combinations of these modes (AC, CF, and ACF).

RESULTS

Figure 1 shows the 3D laser micrograph of the fibers contained in the FRC used in the present experiment. The fibers were confirmed to be straight and the lengths were varied.

Table 2 shows the ANOVA results for bond strength. Bond strength was significantly influenced by thermocycling, the substrate, and the luting agent. With the exception of the substrate/luting agent interaction, no significant interactions were found among the factors. The means and standard deviations of shear bond strength before and after thermocycling for 20,000 cycles are listed in Tables 3 and 4, respectively.

Before thermocycling (Table 3), the mean bond strength evaluated ranged from 3.9 to 28.4 MPa. FI showed the lowest bond strength in both FRC and RC-control. The FRC groups bonded with LM, MB, SB, and MT exhibited the highest bond strengths (24.2 28.4 MPa). Although the RC-control groups bonded with PF, CA, and MB were not significantly different from each other, LM, SB, and MT showed higher bond strengths than PF. When PF, LM, or MB was used, the bond strength with FRC was significantly higher than that with RC-control. Most FRC specimens were observed to suffer complete or partial cohesive failure. With the exception of FI, most RC-control specimens suffered complete or partial fracture at the substrate material.

After thermocycling for 20,000 cycles, the bond strength ranged from 0 to 22.1 MPa (Table 4). All samples bonded with FI failed after thermocycling. In the FRC groups, five luting agents (PF, LM, MB, SB, and MT) showed the highest bond strengths, and no statistically significant differences were found among these agents. In the RC-control groups, the bond strengths of three luting agents (MB, SB, and MT) were relatively high compared with the other luting agents (PF, LM, CA, and FI). When PF, LM,

Table 2 Results of analysis of variance for shear bond strength

Source of variation	d.f.	Sum of squares	Mean square	F-value	P-value
Thermocycling	1	954.9	954.9	59.3	0.0001
Substrate	2	1098.0	549.0	34.1	0.0001
Luting agent	6	4681.7	780.3	48.4	0.0001
Thermocycling/Substrate	2	25.1	12.5	0.8	0.5
Thermocycling/Luting agent	6	110.1	18.4	1.1	0.3
Substrate/Luting agent	5	246.6	49.3	3.1	0.01
Thermocycling/Substrate/Luting agent	5	66.7	13.3	0.8	0.5
Residual	140	2256.3	16.1		

Table 3 Shear bond strengths and types of bonding failure at 0 thermocycles

Luting agent	FRC				RC-control			
	Mean (SD)* (MPa)	Bonding failure** (number of specimens)			Mean (SD)* (MPa)	Bonding failure** (number of specimens)		
PF	21.8(3.5)[def]	AC(5),	C(1)		13.7(4.5)[b]	AC(1),	CF(1),	ACF(4)
LM	24.5(2.7)[fg]	AC(3),	C(3)		19.4(6.3)[cde]	F(4),	CF(2)	
CA	19.0(6.5)[cde]	AC(6)			17.0(3.1)[bc]	CF(3),	F(1),	ACF(1)
MB	28.4(4.7)[g]	AC(2),	C(3)	F(1)	17.9(3.8)[bcd]	F(5),	CF(1)	
SB	24.2(3.2)[fg]	AC(3),	CF(1)	ACF(2)	20.9(4.1)[cdef]	CF(4),	F(2)	
MT	25.5(3.1)[fg]	CF(3),	F(3)		23.1(4.8)[ef]	CF(2),	F(4)	
FI	3.9(0.9)[a]	A(1),	AC(5)		7.9(2.9)[a]	A(1),	AC(5)	

*Identical letters indicate that the values are not statistically different (p ≥ 0.05).
**A, adhesive failure at the luting agent-substrate material interface; C, cohesive failure within the luting agent; F, fracture at the substrate material; AC, CF, ACF, combined failure involving the above failure modes.

Table 4 Shear bond strengths and types of bonding failure at 20,000 thermocycles

Luting agent	FRC Mean (SD)* (MPa)	FRC Bonding failure** (number of specimens)		RC-control Mean (SD)* (MPa)	RC-control Bonding failure** (number of specimens)		
PF	19.7(2.9)[def]	AC(5),	ACF(1)	9.8(2.3)[a]	AC(3),	ACF(2),	CF(1)
LM	17.8(6.3)[cdef]	AC(5),	ACF(1)	10.1(3.0)[ab]	AC(5),	ACF(1)	
CA	14.9(5.7)[bc]	A(4),	AC(2)	13.1(2.6)[abc]	ACF(6)		
MB	22.1(5.4)[f]	AC(3),	ACF(3)	16.3(2.7)[cde]	AC(2),	CF(3),	ACF(1)
SB	20.2(3.0)[ef]	CF(5),	F(1)	17.4(4.8)[cdef]	CF(4),	F(2)	
MT	21.1(2.4)[ef]	CF(6)		15.0(6.1)[cd]	CF(1),	F(5)	
FI	0	A(6)		0	A(6)		

*Identical letters indicate that the values are not statistically different ($p \geq 0.05$).
**A, adhesive failure at the luting agent-substrate material interface; C, cohesive failure within the luting agent; F, fracture at the substrate material; AC, CF, ACF, combined failure involving the above failure modes.

MB, and MT were used, the bond strengths with FRC were significantly higher than those with RC-control. Furthermore, there were no cases where FRC showed a significantly lower bond strength than RC-control. Most specimens with PF, LM, CA, MB, and FI were observed to suffer complete or partial adhesive failure in both the FRC and RC-control groups. In contrast, SB and MT demonstrated no adhesive failures, but rather cohesive failure within the luting agent or in the substrate material.

DISCUSSION

The present study revealed that this experimental FRC could be successfully bonded with resin-based luting agents. Before thermocycling, FRC showed less substrate fracturing than RC-control despite the higher bond strength (Table 3). It has been reported that FRC is flexible but not tough, as compared to the RC-control material, according to bending tests[25]. The flexibility of FRC originates from both the incorporated fiber and the large amount of matrix resin. The majority of monomers used for the FRC matrix posses a soft polyaliphatic carbonate segment. Therefore, a possible explanation was that the FRC dispersed the shear stress at the bonding interface.

With regard to bonding durability, no definitive conclusions should be drawn without clinical evaluation. Thermal stress is one factor that weakens adhesive bonding. On this ground, the thermocycling test is considered as an expedient, *in vitro* experiment to accelerate aging with controlled thermal stresses in water. Thermal stress is mainly derived from the difference between the thermal expansion coefficients of the substrate materials and the luting agents used, and Young's modulus of the luting agent affects the relief of thermal stress[32,33].

It is known that FRC has a lower Young's modulus (1.6 ± 0.2 GPa) than RC-control (4.4 ± 0.2 GPa)[25]. Therefore, thermal stress was better relieved by the FRC layer.

In the present experiment, both FRC and RC-control were blasted with alumina. Once the oxygen-inhibited unpolymerized layer was removed, strong bonding between the resin-based luting agent and resin composite material was difficult to achieve[34]. FRC contained more methacrylate monomers than RC-control (Table 1). Accordingly, the authors speculated that the residual monomer existing in the FRC specimen contributed to the strong bonding with the resin-based luting agents, in addition to the oxygen-inhibited unpolymerized layer.

The resin-based luting agents evaluated in this study were classified into two categories: composite-type resin cements (PF, LM, and CA) and unfilled resins (MB, SB, and MT). All the unfilled resins contained methyl methacrylate (MMA), which is characterized by a smaller molecular weight than the other monomers used in the composite-type resin cements. Unfilled resins tended to exhibit higher bond strengths than the composite-type resin cements. In particular, in the cases of SB and MT, most specimens failed in substrate or cohesive failure mode. This indicated that the actual adhesive force generated at the bonding interface was superior to the obtained bond strength values.

As shown in Tables 3 and 4, the bond strengths of MT and SB remained at the same level after 20,000 thermocycles. The functional monomer used in SB was 4-methacryloxyethyl trimellitate anhydride (4-META), while MT contained no functional monomers. Apart from 4-META, SB and MT had similar chemical compositions: MMA monomer, poly (methyl methacrylate) powder, and tri-*n*-butylborane initiator for polymerization. These

results suggested that the effect of 4-META was not critical for resin composite bonding.

Shear bond strengths after 5,000 thermocycles between a composite-type resin cement containing 10-methacryloxydecyl dihydrogen phosphate and two commercially available fiber-reinforced resin composites were reported to be 20.1 23.7 MPa[28]. These values were comparable to those of PF in the present experiment.

With Fuji I, the glass-ionomer cement, the reduction in bond strength was the greatest. This result agreed with a previous report that a fiber-reinforced resin composite crown cemented with glass ionomer cement debonded after 10,000 thermocycles[2]. Taken together, these results collectively suggested that there were evident differences in the bonding mechanism to resin composites between resin-based luting agents and glass ionomer cements.

In light of the present findings, the hypothesis that luting materials bond to FRC containing milled glass fiber more strongly than to conventional indirect resin composites was confirmed for four luting agents: PF, LM, MB, and MT.

In conclusion, the use of FRC improved the bond durability between the indirect composite and resin-based luting agents. Greatest bond strength was achieved when FRC was bonded with PF, LM, MB, SB, and MT, followed by CA. Insufficient bonding was obtained with FI. In constructing indirect composite restorations, the clinician should select a proper luting agent in conjunction with FRC to ensure their long-term durability.

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