EFFECT OF SURFACE TREATMENTS OF LABORATORY-FABRICATED COMPOSITES ON THE MICROTENSILE BOND STRENGTH TO A LUTING RESIN CEMENT

EFEITO DOS TRATAMENTOS DE SUPERFÍCIE DE RESINAS COMPOSTAS DE LABORATÓRIO NA RESISTÊNCIA A MICROTRAÇÃO DE UM AGENTE DE FIXAÇÃO RESINOSO

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The purpose of this study was to evaluate the influence of different surface treatments on composite resin on the microtensile bond strength to a luting resin cement. Two laboratory composites for indirect restorations, Solidex and Targis, and a conventional composite, Filtek Z250, were tested. Forty-eight composite resin blocks (5.0 x 5.0 x 5.0mm) were incrementally manufactured, which were randomly divided into six groups, according to the surface treatments: 1- control, 600-grit SiC paper (C); 2– silane priming (SI); 3– sandblasting with 50 µm Al_2O_3 for 10s (SA); 4- etching with 10% hydrofluoric acid for 60 s (HF); 5– HF + SI; 6 – SA + SI. Composite blocks submitted to similar surface treatments were bonded together with the resin adhesive Single Bond and Rely X luting composite. A 500-g load was applied for 5 minutes and the samples were light-cured for 40s. The bonded blocks were serially sectioned into 3 slabs with 0.9mm of thickness perpendicularly to the bonded interface (n = 12). Slabs were trimmed to a dumbbell shape and tested in tension at 0.5mm/min. For all composites tested, the application of a silane primer after sandblasting provided the highest bond strength means.

UNITERMS: Composite resins; Silane; Sandblasting; Hydrofluoric acid; Microtensile test.

INTRODUCTION

Adhesive dentistry associates the physical properties of the restorative materials to dental substrates by means of bonding, providing esthetics and function to previously damaged teeth. Indirect techniques are used in an attempt to overcome some shortcomings of direct composite resin restorations, such as polymerization shrinkage and conversion degree. Material manipulation out of the mouth allows better proximal contacts, morphology and adjustment of the occlusal surface[3, 9, 22, 23]. Moreover, extraoral polymerization allows higher conversion rate, influencing the composite mechanical properties[21, 24].

Clinical indications for indirect restorations are based on the evaluation of the remaining tooth structure, intraoral conditions and cost. Commercially available laboratory-fabricated composite resins have been introduced for the rehabilitation of anterior and posterior teeth, with improved

esthetic and handling properties. These materials present high percentage of inorganic fillers by volume, which improves the mechanical and physical properties[22, 23].

The clinical performance of composite indirect restorations depends on bonding of the luting agent to both the tooth and the restorative material. Thus, the long-term clinical performance is influenced by the surface treatment of the composites. The internal surface of indirect restorations can be treated with sandblasting, hydrofluoric acid or silane coupling agents, and with the combination of these treatments. The air-abrasion technique produces a rough surface[5, 8, 18, 19], while silane creates a chemical adhesion between the inorganic fillers and the organic matrix of the bonding agent[5, 10, 15, 25]. The hydrofluoric acid has been used to etch all-ceramic restorations[12, 20]; however, its effects on different filler particles of composite resins have not been effective in producing high bond strengths of resin cement bonded to indirect composite restorations[8, 18, 19, 20].

Shear and tensile bond conventional tests have been commonly employed to evaluate the bond strength of the resin luting cement to indirect restorative materials[2, 5, 8, 12, 19, 20, 25]. The microtensile methodology developed by Sano, et al. (1994) allows the evaluation of specimens with small cross-sectional areas, resulting in better and uniform stress distribution at the bonded interface and accurate results[16]. The purpose of this study was to investigate the effect of mechanical and chemical surface treatments of composite resin restorative materials on the microtensile bond strength to a resin luting cement.

MATERIALS AND METHODS

Two prosthetic composite resins, Targis (TA) (Ivoclar, Schann, Liechtenstein) and Solidex (SO) (Shofu Inc., Kyoto, Japan), and a composite resin for direct restorations, Filtek Z250 (Z250) (3M ESPE, St. Paul, MN, USA) light-cured in the laboratory unit were used in this study. Table 1 shows the composition of the restorative materials tested.

Forty-eight resin blocks were prepared for each restorative material in an acrylic transparent mold with four rectangular cavities (5.0 mm x 5.0 mm x 5.0mm) (Figure 1a). SO and Z250 were polymerized in a laboratory light-curing unit, Edglux (EDG Ltda, São Carlos, SP, Brazil). Each composite was inserted in 1-mm thick increments and each increment was light-cured for 3 minutes. A complementary polymerization was accomplished for 7 minutes. TA blocks were built in 1-mmm thick layers, initially polymerized in the intermediary Targis Quick unit (Ivoclar, Schann, Liechtenstein) for 10 seconds and an additional polymerization was accomplished in the Targis Power unit (Ivoclar, Schann, Liechtenstein) for 25 minutes at 95°C.

Composite block surfaces to be bonded were wet-ground with 600-grit silicon carbide abrasive paper for 15s. Samples of each composite were divided into 6 groups with 8 blocks each (4 pairs of composite resin blocks). Blocks were submitted to the following composite surface treatments: CO – 600-grit SiC paper treatment, SI – silanization (3M ESPE, St. Paul, MN, USA) for 30 s, SA – sandblasting (Danville Eng. Inc., San Ramon, CA, USA) with 50µm aluminum oxide particles for 10 s, HA- 10% hydrofluoric acid (Dentsply Caulk, Milford, DE, USA) etching for 30s and rinsed with water, HA + SI and SA + SI.

Two similarly treated surfaces (Figure 1b) were bonded together with Single Bond (3M ESPE, St. Paul, MN, USA) adhesive system and a fine layer of Rely X (3M ESPE, St. Paul, MN, USA) resin cement, according to the manufacturer's instructions. This set was submitted to a load of 500g for 5 minutes to standardize the luting agent thickness (Figure 1c). Visible-light activation of each surface of the bonded blocks was performed for 30 seconds, using a XL 3000 light-curing unit (3M ESPE, St. Paul, MN, USA). Specimens were stored in 100% relative humidity at 37°C for 24 h.

Bonded samples were positioned in a precision cutting machine 1000 Isomet (Buehler Ltd, Lake Bluff, IL, USA) and were serially sectioned perpendicular to the bonded interface to obtain three 0.9-mm thick slabs (Figure 1d) from each bonded samples (n = 12). Each slab was trimmed with a diamond bur to an hourglass shape with a cross-sectional area of approximately 0.8mm^2 (Figure 1e). Specimens were attached to the grips of a microtensile testing device and tested in tension in a universal testing machine model number 4411 (Instron Corp., Canton, MA, USA) at a cross-head speed of 0.5mm/min, until failure.

After testing, the cross-sectional areas of the specimen at the site of fracture were measured to the nearest 0.01mm with a digital caliper model number 727-6/150 (Starret, Sao Paulo, SP, Brazil) to calculate the tensile bond strength, expressed in MPa. Differences in tensile bond strengths were evaluated for statistical significance using two-way analysis of variance and Tukey test at the 0.05 level of significance.

RESULTS

Tensile bond strength means of composite resin to the luting material as function of the surface treatment are presented in Table 2. Two-way ANOVA showed that there were statistically significant differences for the factor "composite resin" ($p = 0.00127$), for the factor "surface treatment" ($p = 0.00001$) and for the factor "interactions" ($p = 0.01540$). The Tukey test showed significant differences among treatment surfaces with all composite resins tested ($p < 0.05$). Tensile bond strength to the luting agent was dependent on composite restorative materials and surface treatment ($p > 0.05$).

In general, higher tensile bond strength means were obtained when composite resin surfaces were treated with 50-µm aluminum oxide followed by application of silane coupling agent. For Targis indirect composite, sandblasting alone presented similar bond strength to the association of sandblasting and silanization, which was similar to the other surface treatments. Composite resins showed similar bond strength means for HA, HA + SI and SA + SI surface treatments.

DISCUSSION

Extra-orally cured composites produce a high degree of conversion of carbon double into covalent bonds, reducing the amount of residual unreacted metacrylic groups available for bonding[1, 5, 8, 18]. Thus, this study evaluated various surface treatments in an attempt to improve bonding of a luting composite to indirectly cured composite resin restorative materials. Resin-to-resin specimens were used to avoid bonding to tooth structure, which could interfere with the tensile bond strength values, due to dentin regional variability[4, 11, 16].

Sandblasted (air-abraded) and silanated Solidex and Z250 composites presented the highest tensile bond strengths to the resin cement. For Targis composite, sandblasting alone produced similar bond strength to air-abrasion treatment in conjunction with silanization, which was similar to the other surface treatments. Sandblasting was introduced to enhance bonding between resin and metallic alloys. Microabrasion with 50-µm aluminum oxide removes impurities, roughness and increases the surface energy of the inner surface of the metallic restoration. For composites, air abrasion promotes non-selective degradation and also roughness on the composite, creating an irregular surface and strong mechanical attachment of the adhesive resin to sandblasted composite. Moreover, it removes the resin matrix, exposing and maintaining filler particles on the composite surface for silanization[5, 8, 19, 20].

Silane is a coupling agent and its bifunctional molecule bonds to both the exposed composite filler particles and the bonding resin[10]. In its hydrolyzed state, silane contains silanol groups bonding to the filler surface by formation of siloxane bonds (Si-O-Si) with release of water. The resin-active vinyl group is responsible for adhesion to organic resin of bonding agent. This part of the silane molecule contains a double bond, which enables the molecules to react with the metacrylic groups in the bonding agent[14, 15]. The exposure of filler particles produced by sandblasting with 50-µm aluminum oxide particle abrasion facilitated bonding in the silanization step, because a large area of filler particles was present on the composite surface. Moreover, all composites tested present a high filler content that can improve bonding with the luting agent. Silanization without any surface pre-treatment did not produce high bond strength. Its mean value was similar to the control group, hydrofluoric acid etching, sandblasting and acid etching associated with silanization treatments for all composites tested.

All specimens were abraded with silicon carbide paper and control group specimens were not submitted to any other mechanical or chemical surface preparation. Wet abrasion produces a smear layer of composite resin that can interfere with bond strength. Hydrofluoric acid etching removes the smear layer of composite resin and etches the filler particles on the surface. Composite containing barium and strontium glass fillers are etched by hydrofluoric acid,

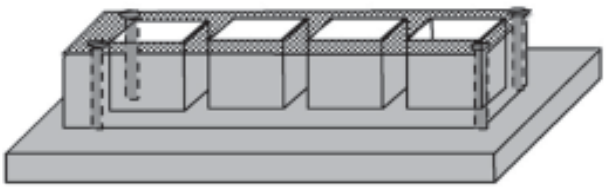

Fig. 1a

Fig. 1b

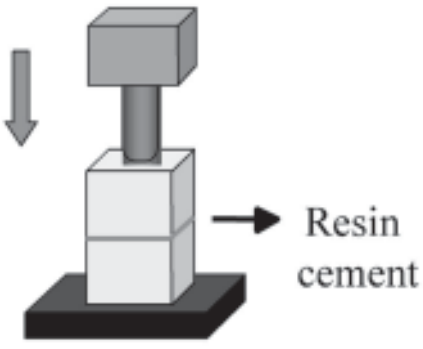

Fig. 1c

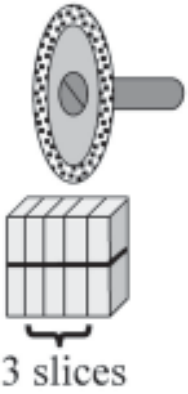

Fig. 1d

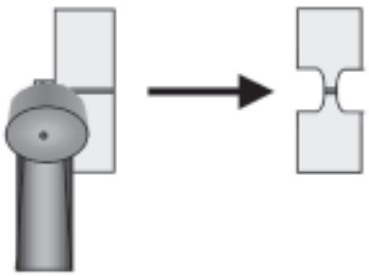

Fig. 1e

FIGURE 1- Schematic diagram of the specimen preparations

producing an irregular and retentive surface[6, 7, 13]. However, thirty seconds might be excessive acid conditioning and might have caused resin softening of the matrix due to total dissolution of the glass fillers on the composite surface.[18, 19] Thus, silanization had no effect after hydrofluoric acid etching, because conditioning removed the filler particles, leaving no glass particles available for bonding on the composite surface.

This study indicated that the surface pre-treatment of laboratory-cured composite resins with sandblasting followed by silanization provided the highest tensile bond strength values. Bonding of indirect composite resin restorations to resin cement was dependent on the micro-mechanically retentive surface provided by air-abrasion and on the silane chemical bond with exposed inorganic fillers.

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TABLE 1- Composition of restorative resin material used in this study

Composites	Composition	
	Matrix resin	**Inorganic fillers**
Z250	UDMA, Bis-EMA and Bis-GMA	66% in volume Zirconium glass and Coloidal silica
Targis	Bis-GMA, DDMA and UDMA	68% in volume Barium Glass, Mixed oxides and Coloidal silica
Solidex	Co-polimers of multi-functional and conventional resins	39% in volume Ceramic microfilaments

Abbreviations: UDMA- urethane dimethacrylate, Bis-EMA- bisphenol-A polyethylene glycol diether dimethacrylate and Bis-GMA- bisphenol-A glycidil ether dimethacrylate, DDMA- Decane dimethacrylate.

TABLE 2- Tensile bond strength means of resin composites to luting agent expressed in MPa (SD)

Composite Resins	Surface treatments					
	CO	**SI**	**SA**	**HA**	**HA + SI**	**SA + SI**
Z250	56.11 A b (10.64)	49.43 A b (13.90)	42.28 AB b (10.41)	39.38 A b (14.49)	31.50 A b (8.38)	105.17 A a (10.68)
Solidex	26.66 B b (14.91)	20.84 B b (6.95)	23.06 B b (4.73)	32.04 A b (4.46)	29.27 A b (1.71)	102.16 A a (10.50)
Targis	27.87 AB b (8.41)	38.32 AB b (2.78)	53.05 A ab (13.15)	33.97 A b (5.95)	32.90 A b (14,81)	86.61 A a (6.03)

Groups that are not significantly different ($p<0,05$) are marked with the same letters (lower case – horizontal and capital letter - vertical)

RESUMO

O objetivo deste estudo foi avaliar a influência de diferentes tratamentos de superfície na resistência de união de resinas compostas a um agente de fixação resinoso. Dois compósitos de laboratório, Solidex e Targis, e um compósito convencional, Filtek Z250, foram testados. Quarenta e oito blocos de resina composta (5.0 x 5.0 x 5.0mm) foram confeccionados através da técnica incremental, para cada compósito testado, e foram aleatoriamente divididos em 6 grupos. Os blocos foram submetidos a seis tratamentos de superfície: 1 - Controle, Lixa 600-SiC (C); 2 – Silanização (SI); 3 – Jateamento com Al_2O_3 50µm por 10 segundos (SA); 4 – Condicionamento com ácido fluorídrico por 60 segundos (HF); 5 – HF + SI; 6 – SA + SI. Blocos submetidos ao mesmo tratamento foram unidos com o agente de fixação resinoso Rely X. Uma carga de 500g foi aplicada por 5 minutos e as amostras foram fotoativadas por 40 segundos. Os blocos unidos foram seccionados em fatias de 0.9mm de espessura perpendicularmente à interface de união (n = 12). Foram realizadas constrições limitando a interface de união a 1mm e as amostras foram levadas para o ensaio de tração. As maiores médias de união foram obtidas para as amostras submetidas à aplicação do silano após o jateamento com Al_2O_3.

UNITERMOS: Compósito; Silano; Jateamento; Ácido fluorídrico; Microtração.

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