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Microtensile bond strength of indirect composite restorations using different combinations of resin-coating technique

Resistência da união por microtração de restaurações indiretas de resina, usando diferentes combinações na técnica de Resin-Coating

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Abstract

The aim of this study was to evaluate microtensile bond strength (μ-TBS) and failure mode of indirect composite restorations bonded to dentin using different combinations of Resin-Coating (RC) after thermal and load cycling. Thirty five extracted third molars were used in the study. Two box-like Class II cavities were prepared in each tooth (mesial and distal surface). The 70 cavities were distributed in 7 groups according to the coating materials: G1:Etch-rinse 2steps (SB2); G2:Etch-rinse 2steps/Hydrophobic-monomer (SB2/B); G3:Etch-rinse 2steps/Flowable composite resin (SB2/FL); G4:Self-etch 1step (CS3); G5:Self-etch 1step/Hydrophobic monomer (CS3/B); G6:Self-etch1step/Flowable composite resin liner (CS3/PL), G7:Self-etch 2step/Flowable composite resin liner (CSEB/PL). The cavities were molded with a vinyl polysiloxane impression material and the molds were poured with a stone plaster. The fillings were confectioned using the Sinfony composite system (3M/ESPE) and were cemented with resin luting cement (Rely X ARC system). After 24 hours, the teeth were submitted to thermocycling (2,000C/5-55°C) and load cycling (250,000C/30N). After, the restored teeth were sectioned in to beams and μ-TBS were measured. The data were analyzed with ANOVA and Tukey test ($p<0.05$). In addition failure mode pattern was determined by scanning electrical microscopy. Bond strength were significantly higher in the groups CSEB/PL and CS3/B ($p<0.05$). In the groups in which was not used a liner, the failure mode exhibited dentin exposure. The groups CSEB/PL and CS3/B showed the highest values of bond strength and the failure mode reveal CSEB/PL exhibited better performance since doesn't present any fracture kind A.

Keywords

Bond strength; resin coating; adhesives; indirect restorations.

Introduction

Esthetic indirect composite restorations have become widely accepted in extensive cavities, as a result of the improvement of both the dental materials and the restorative techniques; however, this type of restoration demands a more invasive cavity preparation and may lead to a postoperative sensitivity [1].

Attempting to minimize this sensitivity the Resin-Coating Technique (RCT) has been proposed [2]. This

technique consists in the hybridization of the exposed dentin followed by the application of a hydrophobic monomer or a low viscosity resin immediately after cavity preparation and prior to the impression step. [3]. The immediate dentin sealing technique offers several advantages. First, resin adhesion can be improved by bonding to freshly cut dentin and by polymerization of the resin adhesive without any stresses related to curing of the resin cement that will overlie it. Secondly, the adhesive provides a seal that reduces bacterial contamination, tooth sensitivity, and the need for anesthesia at the delivery appointment [4].

The effectiveness of this technique was reported evaluating bond strength [5], however, it was performed in flat surfaces, not considering factors as the cavity configuration, thermal variations and masticatory forces, which could influence the long-term durability of the restorations [6]. Moreover, the combination of an adhesive system and a liner (hydrophobic monomer or low viscosity resin) used for RCT may influence the longevity of the restoration[7].

Therefore, the aim of this study was to evaluate the microtensile bond strength and failure mode pattern of indirect composite restorations using different protocols of RCT after thermal and load cycling. The null hypothesis is that the different associations of materials used for RCT do not influence on the bond strength and failure mode of the indirect restorations.

Material and Methods

Sample preparation

Thirty-five extracted third molars were used in the study, after the approval obtained by the local Ethical Committee in Research . The periodontal ligament was simulated applying a layer of polyether (Impregum, 3M ESPE AG, Seefeld, Germany) over the roots [8]. Then, the apical side of the teeth was embedded in epoxy resin leaving the crown and 2 mm of the root exposed. Two box-like Class II cavities were prepared using diamond burs (#4137 KG Sorensen Barueri SP, Brazil). The cavities had the following dimensions: 4 mm of bucco-lingual width and 3 mm of proximal-axial width and the gingival margin of the cavity was located 1 mm above the cement-enamel junction. The dimensions and characteristics of the cavities are detailed in Figures 1A and 1B. The cavities were randomly distributed in 7 groups (n=10). The materials used in each group and composition are described in Table 1.

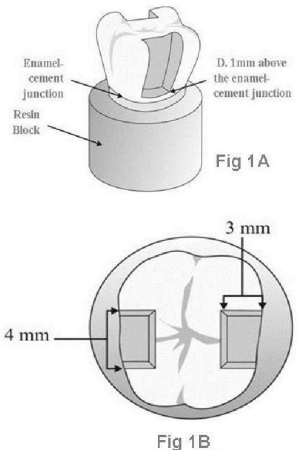

Figure 1 - A. Characteristics and measurements of class II cavity. B. Occlusal view of the cavity preparation.

Application Technique of the RC

The procedure for restoring the groups 1-7 was performed according to RTC technique. All restorative procedures and applied techniques are shown in Table 2.

Table 1 - Materials used in the study

Materials	Composition	Manufacturer/ Batch #
Single Bond 2 (SB2)	water, ethanol, Bis-GMA, HEMA, UDMA, polyalkenoic acid copolymer, dimethacrylate, nanofiller	3M/ESPE. St. Paul, MN, USA #5EP
Scotch Bond Multipurpose (B)	Bond SBMP: Bis-GMA, HEMA, photoinitiator	3M/ESPE. St. Paul, MN, USA #5HP
Filtek Flow (FL)	Bis-GMA, TEGDMA, Zirconia, Silica, camphorquinone, nanofiller	3M/ESPE. St. Paul, MN, USA #6031A2
Clearfil S3 (CS3)	MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, photoinitiator, ethanol.	Kuraray Medical, Tokyo, Japan # 00001A
Clearfil SE Bond (CSEB)	bisphenol A diglycidylmethacrylate, 2-hydroxyethyl methacrylate, 10-Methacryloyloxydecyl dihydrogen phosphate, Hydrophobic aliphatic methacrylate, Colloidal silica, dl-Camphorquinone, Initiators, Accelerators	Kuraray Medical, Tokyo, Japan # 00205B,
Protect Liner (PL)	Bis-GMA,TEGDMA, fluoride methyl methacrylate, camphorquinone, silanized colloidal silica.	Kuraray Medical, Tokyo, Japan F # 0046
Rely X ARC	Bis-GMA TEGDMA, Functionalized dimethacrylate polymer, silane treated Zirconia and Silica, 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol	3M/ESPE. St. Paul, MN, USA #5HP

Bis-GMA: bisphenol-A diglycidyl ether dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, MDP: 10 methacryloyloxydecyl dihydrogen phosphate, TEGDMA: triethylene glycol dimethacrylate.

Table 2 - Groups, materials and applied technique

Groups		Materials	Applied Technique
G1	**SB2**	Single Bond 2	a, b, c , d, e, d, e, k
G2	**SB2/B**	Single Bond 2 Bond - Scotch Bond Multipurpose	a, b, c , d, e, d, e, k j, k
G3	**SB2/FL**	Single Bond 2 Filtek flow	a, b, c , d, e, d, e, k i, l
G4	**CS3**	Clearfil S3	h, e, k
G5	**CS3/B**	Clearfil S3 Bond Clearfil SE Bond	h, e, k g, e, k
G6	**CS3/PL**	Clearfil S3 Protect Liner F	h, e, k l
G7	**CSEB/PL**	Clearfil SE Bond Protect Liner F	f , e, g, e, k l

Application technique: a: acid technique (15s); b: rinse surface (15s); c: dry with cotton-pellet; d: apply one layer one-step total-etch adhesive; e: gently air dry (5s); f: apply primer – two-step self-etch adhesive (20s); g: apply bond – two-step self-etch adhesive; h: apply one layer of one-step self-etch adhesive (20s); i: apply one layer of resin flow; j: apply bond – three-step etch and rise adhesive, k: light-curing (10s) , l: light-curing (20s).

Indirect Restoration Technique

After RCT, impressions of the preparations were taken using putty and light polyvinylsiloxane (Aquasil, Dentsply DeTrey, Konstanz, Germany). The PVC cylinder (12.5 mm) fixed to a metallic handle was used as an impression tray. After one hour, the casts were poured in stone (Durone IV, Dentsply, Petropolis, RJ, Brazil) and removed after 60 minutes. Then the stone were isolated with Isolacril (Asfer, São Paulo, SP, Brazil) and Indirect restorations were made with Sinfony System (3M ESPE AG, Seefeld, Germany) using the incremental technique, starting with the proximal box followed by the occlusal box. Each increment was light cured for 40 seconds with a quartz-tungsten-halogen light-curing unit (500mW/cm2) (XL2500, 3M ESPE, St. Paul MN, USA) (3M, ESPE, St. Paul MN, USA).

Cementation Procedures

The cavities were etched with 35% phosphoric acid (3M ESPE AG, Seefeld, Germany) for 15s, water rinsed and dried with cotton-pellet to remove the excess of water. Single Bond 2 (3M ESPE St. Paul MN, USA) was applied by twice, [9] each coat was gently air dried (5s), and then light-cured for 10s. The internal surface of the restorations were sandblasted with 50μm Al2O3 powder at 2-bar pressure and treated with 35% phosphoric acid (3M ESPE AG, Seefeld, Germany) for 1 min. Following, a silane drop (Ceramic Primer, 3M ESPE, St. Paul MN, USA) was applied, allowed to dry for 30s and air blasted. A layer of Single Bond 2 was applied, air dried and light-cured for 10s. The resin luting cement Rely X ARC (3M/ESPE, St. Paul MN, USA) was, then, applied in the internal surface of the restoration and it was inserted in the cavity preparation under digital pressure. The excess of luting cement was removed and light-cured for 40s through on each surface of the tooth. The restoration was finished with fine and extra-fine grit diamond burs (#2135F and $2135FF, KG Sorensen, Barueri SP, Brazil) and polished by a series of sandpaper disks (Sof-Lex, 3M/ESPE St. Paul MN, USA). The samples were stored at 37°C for 24 hours.

Thermal and Load Cycling

The specimens were subjected to 2,000 thermal cycles from 5o to 55oC, with bath time of 60s, using a thermo-cycling machine (MSCM, Marcelo Nucci ME Instrument, São Carlos, SP, Brazil). Following, the specimens were submitted to the mechanical load cycling, using an equipment (MSCT-3, Marcelo Nucci ME Instrument, São Carlos SP, Brazil) that consists of five stainless steel pistons with cylindrical tips of 8 mm of diameter and rounded extremities, these tips where kept in contact with the occlusal surface of the restorations. The equipment applies an intermittent axial force of 50N at a frequency of 2 Hz, totalizing 250,000 cycles, [10] under water at 37oC.

Microtensile bond strength

After the thermal and load cycling, the teeth were removed from the epoxy resin and the enamel tissue present on the proximal areas was cut off by using a slow-speed water cooled saw equipped with a diamond-impregnated disk (Isomet, 1000 – Buehler Ltd, Lake Bluff, IL, USA) to expose only the area to be tested in dentin. To obtain the specimens, the restored teeth were

sectioned occluso-gingivally into serial slabs of approximately0.9mm thick using the same slow-speed water-cooled diamond saw. Each slab was then indented into resin composite and dentin beams, of approximately 0.9 x 0.9mm cross-sectionally. Each restoration yielded 2-3 beams for bond strength evaluation.

The beams were fixed to a Geraldelli device [11] and tested to failure under tension in a universal testing machine (Instron - Model 4411, Corona, Ca, USA) with a 50N load cell at a crosshead speed of 0.5mm/min. Means and standard deviation were calculated and expressed in MPa. Statistical analysis was performing using ANOVA and Tukey test ($p<0.05$).

Fracture mode analysis

After that, all the specimens were mounted on stubs, gold sputter coated (Balzers model SCD 050 sputter coater, Balzers Union Aktiengesellschaft, Fürstentum Liechtenstein, FL-9496 - Germany) and examined on Scanning Electron Microscopy (JEOL-5600 LV, Japan) operated at 18 kV. Fracture modes were classified according to Table 3.

Table 3 – Classification of fracture mode after micro-tensile bond testing

Category	Fracture Mode
A	Mixed failure at the interface between Resin Coating material and Hybrid Layer
B	Adhesive failure between Resin Coating material and resin cement
C	Cohesive failure in the resin cement
D	Cohesive failure in the resin coating material
E	Mixed failure between Resin Coating material and resin cement
F	Failure at the interface between resin cement and the indirect composite

Results

Beams with premature failure during sectioning were recorded in the study by the "Zero" value. Statistically significant differences were observed between groups ($p<0.05$) as described in Table 4. Bond strength were significantly higher in CSEB/PL and CS3/B groups ($p<0.05$). Lowest bond strength was obtained with CS3 group ($p<0.05$). The others groups showed intermediate values.

Table 4 – Microtensile bond strength (MPa) according to the group

Group	Mean values Standard deviation
G1 (SB2)	11.24 (± 8.05) B
G2 (SB2/B)	12.59 (± 4.43) B
G3 (SB2/FL)	14.28 (± 5.28) AB
G4 (CS3)	6.50 (±10.34) C
G5 (CS3/B)	16.51 (± 5.23) A
G6 (CS3/PL)	9.48 (±6.89) BC
G7 (CSEB/PL)	16.42 (± 4.58) A

Mean values followed by the same letters were not statistically different ($p>0.05$)

Representative SEM photographs of the debonded specimens are present in the Figures 2-5. In all the groups containing a liner over the adhesive system, it was observed a mixed failure at either the coating materials or the resin cement. In the groups with no liner, it was observed an adhesive or mixed failure at the interface between the resin coating and the hybrid layer and in some samples dentin tissue was exposed.

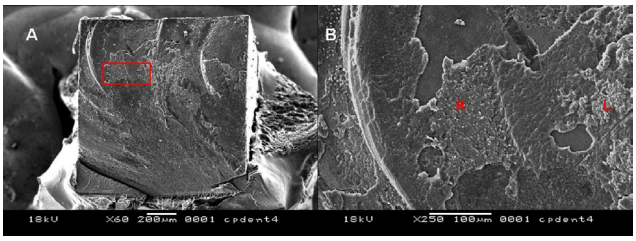

Figure 2 - Representative SEM photographs of the debonded CSEB/PL specimens. Mixed failure between resin coating material and resin cement. (L:Liner, R: Resin cement).

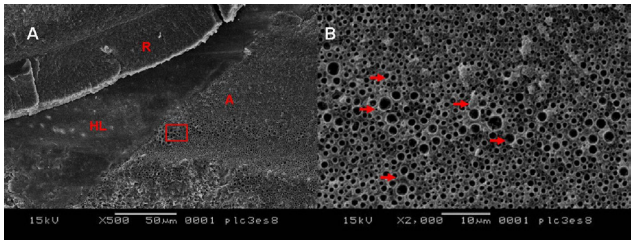

Figure 3 - Representative SEM photographs of the debonded CS3/PL specimens. Cohesive failure in the resin coating material. (HL: Hybrid layer; A: Adhesive; R: Resin cement; Arrows: blisters in the adhesive layer).

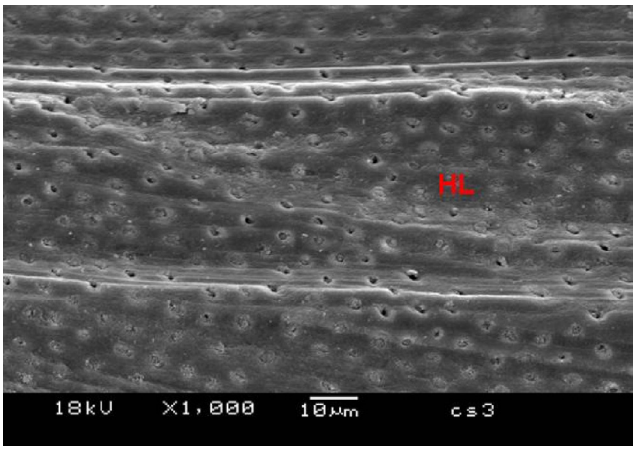

Figure 4 - Representative SEM photograph of the debonded CS3 specimens. Adhesive failure between adhesive and resin cement. (HL: Hybrid layer).

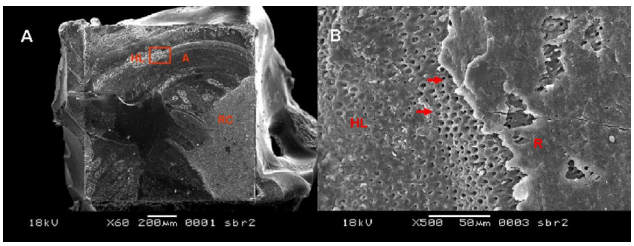

Figure 5 - Representative SEM photographs of the debonded SB2 specimens. Mixed failure at the resin coating material and hybrid layer interface. (HL: Hybrid layer; A: Adhesive; R: Resin cement; Arrows: Dentine tissue).

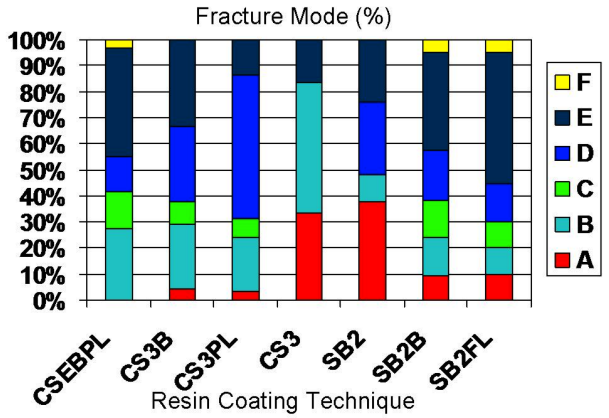

Figure 6 - Failure mode (according to Table 3) after microtensile bond strength test (%).

Discussion

The development of adhesive materials improved the cavity preparation design for indirect restorations, making it less invasive. Yet, the constant changes of these materials in the dental market jeopardize the execution of valid long-term clinical studies, demanding evidence from in vitro studies that simulate the oral conditions [12]. The use of the thermal and load cycling simulates the degradation undertaken by stresses on restorations, helping to better understand the dental materials performance. However, as previously shown, the amount and frequency of cycles, the type of the restorative material and the cavity configuration may influence the bond strength results [13].

A single adhesive application on the cavity preparation has been shown to protect the exposed dentin and prevent sensitivity [1]. However, an additional application of a hydrophobic monomer or a low viscosity resin over the adhesive improves the bond strength of the restoration [14].

Better bond strength results were observed in CSEB/PL and CS3/B groups. The presence of a liner in RCT protected against dentin exposure in all the groups, as revealed by the SEM images of the fracture modes involving the different coating materials. However, since Clearfil S3 adhesive contains HEMA (hydrophilic monomer) and water, it is important to perform a strong air drying to evaporate water and solvents. This procedure results in a viscous resin material with may entrap air bubbles remaining on the dentin surface and reduce the thickness of the layer. Thus, turning it more susceptible to the polymerization inhibition by oxygen, [15] which may account for the presence of blisters within the adhesive layer.

The low bond strength exhibited in all groups can be explained by the fact that most bond strength studies are usually conducted over a flat tooth surface, where presumably the C-factor (0.2) [16] has low adverse influence on bonding, probably overestimating the bond strengths on clinical situations that usually refer to complex cavity preparations restored under clinically relevant conditions [16]. Box-like Class II cavities have four bonded walls and two unbonded surfaces (C-factor: 1.25). Some studies have observed that the biggest C-factor decreases the bond strength [16-18].

Another adverse influence on the bond strength values is the thermo-mechanical cycling, which associated to polymerization shrinkage stress on the resin luting cement, produces strain or even plastic deformation in the restoration, [19] probably creating microcracks on the adhesive layer especially at the gingival wall. Additionally, difficulties to manufacture the beams for the microtensile test were observed, once the bond area is limited. Besides, the stress generated by the cutting procedure resulted in losses of beams in almost all the groups. The groups using only adhesive system without liner presented up to 35% of premature failures.

Selection of the adhesive system and liner is very important for RCT. In this study, CSEB/PL combination exhibited higher bond strength values, in agreement with previous studies that have shown the efficacy of

this combination [7]. The two-step self-etch adhesive Clearfil SE Bond contains an acidic primer such as MDP that solubilizes the smear layer and demineralizes the underlying dentin, resulting in mild surface etching, obtaining good results in several studies [4,10,20]. Moreover, the uncured resin on the oxygen inhibited layer will polymerize with free radicals diffusion from the low viscosity resin and this liner may diminish the adhesive system hydrolysis [21,22].

Another group that presented higher bond strength values was CS3/B, and that was not expected due to the highly hydrophilic characteristics of this adhesive (HEMA and water). Nevertheless, the adhesive coverage by a hydrophobic monomer acts as a physical barrier to the percolation of water through the adhesive layer and might increase the conversion degree, thus reduces the hydrophilic characteristic of the adhesive [23]. More than half of the fractured specimens in both CSEB/PL and CS3/B groups, presented a fracture mode between the RC materials and the resin luting cement, bespeaking RCT efficacy since no specimens revealed expose dentin after fracture.

On the other hand, it was expected higher or similar values to those of CS3/PL group since the liner used in this group was a low viscosity resin. This type of composite has a greater elastic modulus (6-10GPa) than the hydrophobic monomer (3-4GPa), [24] thus creating a thicker sealing film, which may be a better stress breaker than the hydrophobic monomer. A possible explanation for these results is based on the different composition of the materials. The CS3 adhesive is highly hydrophilic, containing water and HEMA, which may compromise the polymerization of the adhesive. The flowable composite resin contains hydrophobic monomers that may not react completely with the free monomers present on the adhesive surface, resulting in a structurally porous salt layer. The chemical incompatibility between the materials was reflected on the failure mode (cohesive failure in the resin coating material). Observations in high magnification revealed blisters in some areas of the adhesive layer when the flowable composite resin was applied as liner. On the other hand, this was not observed when the hydrophobic bonding agent of the two-step self-etching adhesive was the coating liner. This might be explained by the presence of hydrophilic monomers in the bonding agent of Clearfil SE Bond (HEMA) that creates better materials compatibility [25]. This is confirmed by the absence of premature failures unlikely to CS3/PL group that showed great number of premature failures (35%).

The lowest values were obtained with CS3 group that presents better performance when compared to other one-step self-etching adhesives [26].However, literature has shown that the hybrid layer formed by one-step self-etching adhesives presents microscopic channels through which water flows, compromising the adhesive polymerization, reducing the bond strength and accelerating the tooth/restoration interface degradation [24,26]. Besides, the adhesive layer is extremely thin due to the solvent volatilization. Therefore, its polymerization might be hindered by the contact with the oxygen [28].The manufacturer reports that this adhesive works based on molecular dispersion, meaning that the hydrophilic and hydrophobic components would remain in a homogeneous state, even after the solvent evaporation. Still, this adhesive cannot support the stress by itself and necessarily requires a liner to obtain better results in the RCT [29]. The most frequent CS3 fracture type occurred between the RC material and the resin luting cement, exposing dentin tissue in some specimens (Fig. 5).

The total etching groups SB2; SB2/B and SB2/FL exhibited similar μ-TBS values. The Single Bond 2 adhesive is a combination of hydrophilic and hydrophobic monomers and an organic solvent as ethanol and water. Hence, the incomplete solvent volatilization compromises the adhesive polymerization [30] and has a limited capacity of infiltration in the collagen network owing to the demineralization brought about the phosphoric acid, which may be larger than the depth of adhesive infiltration, becoming susceptible to degradation by metalloproteinases. However, SB2 did not show any difference with or without liner, and these results are similar to those of Nikaido et al. 2003 [31].This can be due to the adhesive viscosity, thus this material contains nanofillers that can be found within the hybrid layer. Therefore, these nanofillers will improve the mechanical properties of the adhesive, supporting the thermo-mechanical stress by itself not requiring a liner.

Although, the failure mode was not similar in the three Single Bond groups. SB2 group present mixed failure between the RC and the hybrid layer and some specimens presented dentin tissue. In SB2/B and SB2/FL groups the failure mode was mainly mixed between the RC and the resin luting cement, suggesting a better behavior when a liner is used.

The tested null hypothesis was rejected, since differences in bond strength and fracture modes were observed between the different combinations for resin coating. It was concluded that the combination of an adhesive system and a liner (hydrophobic monomer or low viscosity resin) influenced the longevity of the restoration.

Conclusions

1. The highest bond strength values of RCT for indirect restorations were observed using 2-step self-etch / Flowable composite resin liner and 1-step self-etch / hydrophobic monomer.

2. In all the experimental groups containing a liner, the SEM analysis of the fractures revealed an efficient bonding performance after the thermal and load cycling, since dentin tissue was not exposed.

Acknowledgements

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Resumo

O objetivo deste estudo foi avaliar a resistência da união, por microtração (μ-TBS), e o modo de fratura de restaurações indiretas de resina aderidas à dentina por diferentes combinações na técnica de Resin-Coating (RC), após ciclagem térmica e mecânica. Neste trabalho foram utilizados trinta e cinco terceiros molares extraídos. Em cada dente foram preparadas duas caixas Classe II (nas faces mesial e distal). As 70 cavidades foram divididas em 7 grupos, de acordo com os materiais de cobertura: G1: adesivo convencional de 2 passos (SB2); G2: adesivo convencional de 2 passos/monômero hidrófobo (SB2/B); G3: adesivo convencional de 2 passos /resina composta flow (SB2/FL); G4:adesivo auto-condicionante de 1 passo (CS3); G5: adesivo auto-condicionante de 1 passo/monômero hidrófobo (CS3/B); G6: adesivo auto-condicionante de 1 passo/resina composta flow forradora (CS3/PL), G7: adesivo auto-condicionante de 2 passos/resina composta flow forradora (CSEB/PL). As cavidades foram moldadas por materiais à base de polivinilsiloxano e os modelos foram obtidos em gesso pedra especial. As restaurações foram confeccionadas usando o sistema Sinfony de compósitos (3M/ESPE); e foram cimentadas com cimento resinoso (Rely X ARC system). Após 24 h, os dentes foram submetidos a ciclagem térmica (2.000C/ 5-55oC) e mecânica (250.000/30N). Foram então seccionadas em palitos para medidas de μ-TBS. Os dados foram analisados por ANOVA e teste Tukey ($p<0,05$). Além disso, os padrões de fratura foram determinados por meio de microscopia eletrônica de varredura. A resistência da união foi significativamente mais alta nos grupos CSEB/PL e CS3/B ($p<0.05$). Nos grupos onde não foi usado um agente forrador, os padrões de fratura exibiram exposição de dentina. Os grupos CSEB/PL and CS3/Bos maiores valores de resistência de união e o modo de fratura revelou que CSEB/PL exibiu melhor performance, considerando o fato de não apresentarem nenhuma fratura Tipo A.

Palavras-chave

Infiltração dentária; adesivos dentinários; restaurações intracoronárias.

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