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

**ABSTRACT**

*Objective:* The aim of this study was to evaluate microtensile bond strength (-TBS) and failure mode of indirect composite restorations bonded to dentin using different combinations of Resin-Coating (RC) after thermal and load cycling. *Materials and Methods*: 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 -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. *Results:* Bond strength were significant 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 dentine exposure. *Conclusion:* The groups CSEB/PL and CS3/B showed the highest values of bond strength and the failure mode reveal good performance not exposing dentin tissue.

Key words: 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 the dental materials and 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 hypersensitivity 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 taking the impression.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. Second, 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 like 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 the RCT may influence in the success 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 tested hypothesis is that the different associations of materials used for the RCT do not influence in the bond strength and failure mode of the indirect restorations.

**MATERIALS AND METHODS**

*Sample preparation*

Thirty five extracted third molars were used in the study, under the Ethical approval was obtained by the local Research Ethics Committee. The periodontal ligament was simulated applying a layer of polyether (Impregum, 3M ESPE AG, Seefeld, Germany) over the roots.8 Than, the apical side of the teeth was embedded in epoxi resin leaving the crown and 2 mm of 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 I.

*Application Technique of the RC*

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

**Table 1**. Materials used in the study

|  |  |  |
| --- | --- | --- |
| Materials | Composition | Manufacturer/Batch # |
| Single Bond 2(SB2) | water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate, 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, Sílica, camphorquinone, nanofilller | 3M/ESPE. St. Paul, MN, USA#6031A2 |
| Clearfil S3(CS3) | MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, photoinitiator, ethanol. | Kuraray Medical, Tokio, Japan# 00001A |
| Protect Liner(PL) | Bis-GMA,TEGDMA, fluoride methil methacrylate, camphorquinone, silanized colloidal silica. | Kuraray Medical, Tokio, JapanF # 0046 |
| Rely X ARC | BisGMA 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 diglycidil ether dimethacrylate, HEMA: 2-hydroxyethyl metacrylate, MDP: 10 metacryloyloxydecyl dihidrogen phosphate, TEGDMA: triethylene glycol dimetacrylate

**Table 2**. Groups, materials and application technique.

|  |  |  |
| --- | --- | --- |
| Groups | Materials | Application Technique |
| **G1** | **SB2** | Single Bond 2 | a, b, c , d, e, d, e, k |
| **G2** | **SB2/B** | Single Bond 2Bond - Scotch Bond Multipurpose | a, b, c , d, e, d, e, kj, k |
| **G3** | **SB2/FL** | Single Bond 2Filtek flow | a, b, c , d, e, d, e, ki, k (20s) |
| **G4** | **CS3** | Clearfil S3 | h, e, k |
| **G5** | **CS3/B** | Clearfil S3Bond Clearfil SE Bond | h, e, kg, e, k |
| **G6** |  **SC3/PL** | Clearfil S3Protect Liner F | h, e, kk (20s) |
| **G7** | **CSEB/PL** | Clearfil SE BondProtect Liner F | f , e, g, e, kk (20s) |

Application technique: a: acid technique (15s); b: rinse surface (15s); c: dry with cotton-pellet; d: apply one layer total etch one step 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 self-etch one step adhesive (20s); i: apply one layer resin flow; j: apply bond three step etch and rise adhesive, K: light cure (10s).

*Indirect Restoration Technique*

 After RCT, impressions of the preparations were taken using putty and light (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. Than the stone were isolated with Isolacril (Asfer, São Paulo, SP, Brazil) and Indirect restorations were made with the composite 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 using the XL 2500 curing unit (ESPE, Germany; Norristow, PA, USA).

*Cementation Procedures*

The cavities were etched with 35% phosphoric acid (3M ESPE AG, Seefeld, Germany) for 15s, water rinsed and air blasted to remove the excess of water. Single Bond 2 (3M ESPE St. Paul MN, USA) was applied by dual-application,9 each coat was gently air dried (5s), and then light-cured with a quartz-tungsten-halogen light-curing unit (500mW/cm2) (XL2500, 3M ESPE, St. Paul MN, USA) 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, let 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 the restoration was inserted in the cavity preparation under digital pressure. The excess of luting cement was removed and light-cured for 40s through each surface of the tooth. Finishing was made with fine and extra-fine grit diamond burs (2135F and 2135FF, KG Sorensen, Barueri SP, Brasil) 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, under water at 37oC.

*Microtensile bond strength*

After the thermal and load cycling, the teeth were removed of the epoxy resin and the enamel tissue present on the proximal areas was cut off 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 will tested in dentin. To obtain the specimens, the restored teeth were sectioned occluso-gingivally in to serial slabs approximately 0.9mm thick using the same slow-speed water-cooled diamond saw. Each slab was then indented into resin composite and dentin beams, approximately 0.9 x 0.9mm in cross section. Each restoration yielded 2-3 beams for bond strength evaluation.

The beams were affixed to a Geraldelli device10 and tested to failure under tension in a universal testing machine Instron (Model 4411, Corona, Ca, USA) with a 50N load cell travelling 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 in a Scanning Electron Microscopy (JEOL-5600 LV, Japan) operated at 18 kV. Fracture modes were classified according to Table 3.

**Table 3** – Classification of mode fracture 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 significant higher in the CSEB/PL and CS3/B groups (p<0.05). Lowest bond strength was obtained with the 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)**

Fracture modes are summarized in the Table 3. 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 mixed failure at the coating materials or resin cement, and in the groups that not using a liner was observed adhesive or mixed failure in the interface between the resin coating and the hybrid layer and in some samples exposing dentin tissue.

**Graph 1**. 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.11 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 kind of restorative material and the cavity configuration might influence the bond strength results.12

A single adhesive application on the cavity preparation has been shown to protect the exposed dentin and prevent hypersensitivity.1 However, an additional application of a hydrophobic monomer or a low viscosity resin over the adhesive improves the bond strength of the restoration.13

The low bond strength exhibited in all groups can be explained by the fact that the most bond strength studies are usually conducted over a flat tooth surface, where presumably the C-factor (0.2)14 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.15 Box-like Class II cavities have four bonded walls and two unbonded surfaces (C-factor: 1.25). Some studies have observed that the bigger C-factor decreases the bond strength.14,16

 Another adverse influence on the bond strength values is the thermo-mechanical cycling, which associate to polymerization shrinkage stress on the resin luting cement, produces strain or even plastic deformation in the restoration17, probably creating microcracks on the adhesive layer specially on 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 the RCT. In this study the CSEB/PL combination exhibited high bond strength values, according 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.4,18,19 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.20,21

Another group that presented higher bond strength values was the CS3/B, and that was not expected because 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.22 More than half of the fractured specimens in both groups CSEB/PL and CS3/B, presented fracture mode between the RC materials and the resin luting cement, bespeaking the RCT efficacy since no specimens revealed expose dentin after fracture.

On the other hand, were expected higher or similar values to the group CS3/PL because the liner used in this group was a low viscosity resin, this composite has an elastic modulus (6-10GPa) greater than the hydrophobic monomer (3-4GPa),23 thus creates a thicker sealing film, which might 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, that might compromises the polymerization of the adhesive. The flowable composite resin contains hydrophobic monomers that may do 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, it was not observed when the hydrophobic bond of the two-step self-etching adhesive was the coating liner. This might be explained by the presence of hydrophilic monomers in the bond of Clearfil SE Bond (HEMA) that creates better materials compatibility.24 This is confirmed by the absence of premature failures unlike the CS3/PL 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.25 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.27 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.28 The most CS3 fracture happened between the RC material and resin luting cement, exposing dentin tissue in some specimens (Fig 5).

The total etching groups SB2; SB2/B and SB2/FL exhibited similar -TBS values. The Single Bond 2 adhesive is a combination of hydrophilic and hydrophobic monomers and an organic solvent like ethanol and water. Hence, the incomplete solvent volatilization compromises the adhesive polymerization29 and has a limited capacity of infiltration in the collagen network owing to the demineralization brought about the phosphoric acid, that may be larger than the depth of adhesive infiltration, becoming susceptible to degradation by metalloproteinases. However, SB2 not showed difference with or without liner, and these results are similar with the study of Nikaido et al 2003.30 It 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 tree Single Bond groups. The SB2 group present mixed failure between the RC and the hybrid layer and in some specimens was observed dentine tissue. In the 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.

 Better bond strength results were observed in the CSEB/PL and CS3/B groups. The presence of a liner in the RCT protected the dentin in all the groups, as revealed by the SEM images of the fracture modes involving the different coating materials without dentin exposure. However, since Clearfil S3 adhesive contains HEMA (hydrophilic monomer) and water, it is important to perform strong air drying to evaporate water and solvents. This procedure results in a viscous resin material with may entraps air bubbles remaining on the dentin surface and reduces the thickness of the layer, turning it more susceptible to the polymerization inhibition by oxygen,31 and may be the responsible for the presence of blisters in the adhesive layer.

The tested null hypothesis was rejected, since differences in bond strength and fracture modes were observed between the different combinations for resin coating.

**CONCLUSIONS**

1. The highest bond strength values on RCT for indirect restorations was observed using self-etch 2step / Flowable composite resin liner and self-etch 1step / hydrophobic monomer.
2. In all the experimental groups containing a liner, the SEM analysis of the fracture revealed an efficient bond performance after the thermal and load cycling, since dentin tissue was not exposed.

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

**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).