

Original Article

Effect of Resin Coat Technique on Bond Strength of Indirect Restorations after Thermal and Load Cycling

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Abstract

Objective: To evaluate the ability of the resin-coat technique used in cavity preparation to protect dentin before impression taking and final cementation and its effect on the tensile bond strength of indirect restorations after thermal and load cycling. **Methods:** Occlusal enamel was removed from 25 third molars to expose flat dentin. Teeth were divided into 5 groups (n = 5): G1, receiving no dentin sealing (control group); G2, dentin was hybridized with all-in-one self-etch adhesive (Clearfil S3); G3, receiving combination of a one-step self-etch adhesive and low viscosity resin (Clearfil Protect Liner); G4, dentin was hybridized with “two-step” self-etch adhesive (Clearfil SE Bond); and G5, combination of a “two-step” adhesive system and low viscosity resin was applied. After dentin sealing, indirect restorations were performed with Sinfony system and cementation with dual-cure resin cement (Panavia F). Restored teeth were submitted to thermal (1,500 cycles) and mechanical cycling (200,000 cycles). After this they were sectioned into sticks (1 × 1 mm, approximately) and then subjected to microtensile bond strength testing. **Results:** all data were submitted to ANOVA and Tukey test (p < 0.05). Mean values (MPa) obtained were G1, 9.5; G2, 9.2; G3, 14.8; G4, 12.2; and G5, 17.4. Statistical analysis showed differences between groups, with G5 performance being higher than that of the other groups. **Conclusion:** when no resin coating-technique was used to protect dentin, lower bond strength values were obtained than those in the other groups. The combination of a “two-step” self-etch adhesive system and low viscosity resin promoted the best bond strength values.

Key words: Resin coat technique—Self-etch adhesive—Resin cement

Introduction

Nowadays there is increasing demand by patients for esthetic restorative procedures, partially justified by the significant improve-

ment in ceramic and polymeric materials. In cases of extensive cavities in teeth the indirect technique is the best choice, although it requires a wider preparation involving wear of sound dentin, which consequently increases

the frequency of post-operative sensitivity^{10,21}). In addition, other external factors, such as contamination by saliva, blood and temporary cements may interfere in the quality of bond strength and cementation between the tooth and restoration. The continual development of bonding agents has provided a significant increase in restorative options and the introduction of new techniques to control these factors. Thus, a new technique has been proposed, called the Resin Coating Technique (RCT)¹⁵, which protects the dentin right after cavity preparation, improving bonding quality between resin cement and dentin and better marginal adaptation¹¹). It consists of sealing the dentin with an adhesive system followed by a “liner” (hydrophobic monomer or low viscosity composite) after cavity preparation and should be applied before taking the impression. This technique allows avoidance of external contact with unprotected dentin and minimizes pulp irritation caused not only by both thermal and mechanical stimuli, but also by bacterial infiltration, which can occur while taking the impression, by the emplacement of a provisional crown and temporary cementation^{7,13}).

Use of self-etch adhesive systems in this technique is clinically more attractive because they can be applied on a dry dentin surface. In contrast, phosphoric acid and a non self-etch primer do not penetrate completely into the demineralized area, causing higher incidence of post-operative sensitivity. When an acidic primer is applied and air-dried, light curing is performed. Therefore the dentin does not need to be washed and this procedure becomes less critical due to the fewer number of steps involved. In this case, there is no need to control moisture on the dentin surface. Furthermore, etching with an acidic primer leads to a thinner demineralized dentin area and allows more filling with the adhesive in this area, reducing the risk of post-operative sensitivity⁸).

This technique has been shown to have many advantages. However, due to the continuing development of adhesive systems; there is no consensus on the appropriate

combination of adhesive system and “liner”.

Therefore, the objective of this study was to evaluate the influence of resin coating technique combinations on microtensile bond strength and fracture pattern of indirect restorations cemented with dual polymerized resin cement. The null hypothesis tested was that there would be no differences between the microtensile bond strength and fracture patterns of all the tested resin coat combinations.

Materials and Methods

1. Sample preparation

A total of 25 human third molars of similar size and shape obtained from patients between the ages of 18 and 30 years were used after approval from the Research Ethics Committee of the Dentistry School of Piracicaba—University of Campinas. The teeth were stored in a 0.1% (by weight) thymol suspension at a temperature of 4°C for a period not exceeding 4 months.

Afterwards, the occlusal surface of each tooth was removed using a diamond saw in a cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA) to expose a flat surface on the dentin. The exposed dentin was polished with #600 grit SiC abrasive paper and water for 30 sec to produce a standard smear layer.

Subsequently, the teeth were randomly divided into 5 groups ($n=5$) and the resin coating technique applied. The materials used in each group and their compositions are described in Table 1. The technique used is described in Table 2 according to type of treatment received. After treatment, the surfaces were covered with temporary material (Cavit—3M/ESPE AG, Seefeld, Germany) and the samples stored in distilled water at 37°C for 7 days.

All restorations were made using the indirect restorative system Sinfony (3M/ESPE AG). A circular silicone mold measuring 6mm in diameter and 4mm thick was used, which was filled in increments of approximately 1.5 to 2mm. Each increment was polymerized for

Table 1 Materials used in RCT and their composition

| Materials | Composition | Manufacturer |
|--|---|--|
| Clearfil SE Bond (Two-step self-etch adhesive system) | - Primer: MDP, HEMA, water, photoinitiator - Adhesive resin: MDP, BISGMA, HEMA, hydrophobics dimethacrylates, photoinitiator | Kuraray Medical Co. Ltd., Tokyo, Japan |
| Clearfil S3 Bond (One-step self-etch adhesive system) | MDP, BISGMA, HEMA, dimethacrylates, photoinitiator | Kuraray Medical Co. Ltd. |
| Protect Liner F (Low viscosity resin) | BISGMA, TEGDMA, micro filler, photoinitiator | Kuraray Medical Co. Ltd. |
| Panavia F (Dual-cured resin cement) | Paste A: silanized silica, MDP, dimethacrylates Paste B: silanized barium glass, dimethacrylates, chemical initiator | Kuraray Medical Co. Ltd. |

Table 2 Application technique used in RCT

| Groups/materials | Application technique |
|--|---|
| Group 1 Without RCT (Control) | No materials applied for RCT. |
| Group 2 RCT—only Clearfil S3 | Application on the surface and wait 20 sec, thorough drying for 5 sec and light curing for 10 sec. |
| Group 3 RCT—ClearfilS3 + Protect Liner F | ClearfilS3: Application on the surface and wait 20 sec, thorough drying for 5 sec and light curing for 10 sec. Protect Liner F: Application on the Clearfil S3 and light curing for 20 sec. |
| Group 4 RCT—only Clearfil SE Bond | Primer: Application on the surface for 5 sec, wait 20 sec and drying lightly with air for 5 sec. Bond: Application on the Primer for 5 sec, drying lightly for 5 sec and light curing for 10 sec. |
| Group 5 RCT—Clearfil SE Bond + Protect Liner F | Primer: Application on the surface for 5 sec, wait 20 sec and drying lightly with air for 5 sec. Bond: Application on the primer for 5 sec, drying lightly for 5 sec and light curing for 10 sec. Protect Liner F: Application on the Clearfil SE Bond and light curing for 20 sec. |

5 sec using Visio Alfa unit (3M/ESPE AG), and the restoration was finished by undergoing final polymerization of 15 min in the vacuum unit Visio Beta Vario (3M/ESPE AG).

After 7 days, the temporary material was removed with a dentin excavator and prophylaxis was performed with pumice stone and water for 20 sec followed by rinsing with water and drying for 10 sec. For final cementation the dual polymerized resin cement Panavia F (Kuraray Medical, Tokyo, Japan) was used according to the manufacturer's protocol. ED

Primer was applied on the dentin surface, rubbing it for 60 sec and drying gently for 5 sec. Next, the restoration surface was etched with 37% phosphoric acid, washed and dried. After this, silane (Ceramic Primer 3M/ESPE, St Paul MN, USA) was applied for 1 min and thoroughly dried. Equal amounts of resin cement (catalyst and base paste) were placed on the mixing paper and mixed for 20 sec to obtain a homogeneous mixture. The mixture was placed on the inner surface of the restoration and inserted into the cavity prepara-

tion under finger pressure. The excesses were removed and the material light polymerized on each side for 40 sec using the quartz-tungsten-halogen lamp XL-2500 (3M/ESPE). After this, the samples were stored in 100% humidity at 37°C for 24 hrs.

2. Thermal and load cycling

The roots of the teeth were covered with wax, and the wax thickness was adjusted to range between 0.2 to 0.3 mm. Then the teeth were embedded in plastic tubes (25 mm in height \times 20 mm in diameter) with chemically activated acrylic resin. After polymerization, the teeth were removed from the acrylic resin with hot water, creating spaces that constituted artificial alveoli. These spaces were filled with the polyether-based molding material (Impregum F—3M/ESPE AG), simulating artificial periodontal ligament.

The samples were submitted to thermal cycling in a thermal cycling machine (MSCM, Marcelo Nucci ME Instrument, São Carlos, SP, Brazil), and 1,500 cycles were performed at temperatures between 5°C and 55°C for 30 sec at each bath temperature.

After thermal cycling, load cycling was performed using the MSCT-3 appliance (Marcelo Nucci ME Instrument), which has a stainless steel tip 4 mm in diameter that comes into contact with the central part of the restoration. All the samples were submitted to 200,000 cycles under a 30 N load, at a rate of 2 Hz. After these procedures, the samples were immersed in water.

3. Microtensile bond strength test and statistical analysis

To obtain the beam specimens, the restored teeth were sectioned occluso-gingivally into serial slabs approximately 0.9 mm thick using the diamond saw in a water-cooled slow-speed cutting machine (ISOMET 1000, Buehler, Lake Bluff, IL, USA). Each slab was then sectioned by the same method into resin composite and dentin beams with a cross-sectional area of approximately 0.9 \times 0.9 mm. Each restored tooth (5 in each group, $n = 5$) yielded 12–13 beams for bond strength evaluation.

The beams were fixed to a Geraldeli's jig⁴⁾ with a cyanoacrylate glue (Super Bonder gel, Loctite, Henkel Corp., Rocky Hill, CT, USA) and tested to failure under tension in a universal testing machine Instron (Model 4411, Corona, CA, USA) with a 500-N load cell at a crosshead speed of 0.5 mm/min. Means and standard deviation were calculated and expressed in MPa. Statistical analysis was performed using a one-way ANOVA and the Tukey test ($p < 0.05$).

4. Fracture type analysis

After tensile testing, the fractured samples were collected and stored in distilled water for 24 hrs. Then the parts of the fractured samples were paired, mounted on aluminum stubs, gold sputtered (Balzers model SCD 050 sputter coater, Balzers Union Aktiengesellschaft, Fürstentum Lichtenstein, FL-9496, Germany) and examined by scanning electron microscopy JSM-5600LV (JEOL, Tokyo, Japan), operated at 15 kV to observe fracture type. The samples were classified as follows:

Fracture Type A: Adhesive failure at the interface between RCT material and dentin.

Fracture Type B: Adhesive failure between RCT material and resin cement.

Fracture Type C: Cohesive failure in resin cement.

Fracture Type D: Cohesive failure in the coating material.

Fracture Type E: Mixed failure.

Fracture Type F: Adhesive failure between cement and indirect restoration.

Results

Mean values (MPa) obtained in the microtensile test and standard deviation values are shown in Table 3. Group 5 in which the dentin was treated with the two-step self-etch adhesive (Clearfil SE Bond) and covered with a low viscosity resin (Protect Liner F) obtained the highest results among groups. Group 1, in which no resin coating was applied (control), had the lowest bond strength, along with Groups 2 and 4, in which only self-etch adhe-

Table 3 Mean bond strength (MPa) from different combinations of RCT

| Resin coating technique | G1—without RCT | G2—S3 | G3—S3 + PL | G4—SE | G5—SE + PL |
|-------------------------|----------------|-------------|--------------|---------------|--------------|
| | 9.5 ± 6.9 c | 9.2 ± 4.0 c | 14.8 ± 7.9 b | 12.2 ± 8.5 bc | 17.4 ± 6.8 a |

Values followed by same letter do not differ statistically ($p > 0.05$)

Table 4 Fracture type after microtensile bond strength test

| Fracture type | A | B | C | D | E | F |
|--------------------------|-----|-----|-----|-----|-----|-----|
| G1—No Coating | 60% | 0% | 30% | 0% | 0% | 10% |
| G2—Clearfil S3 | 50% | 0% | 20% | 0% | 25% | 5% |
| G3—Clearfil S3 + PL | 30% | 30% | 20% | 10% | 10% | 0% |
| G4—Clearfil SE Bond | 30% | 0% | 40% | 0% | 20% | 10% |
| G5—Clearfil SE Bond + PL | 20% | 40% | 5% | 15% | 20% | 0% |

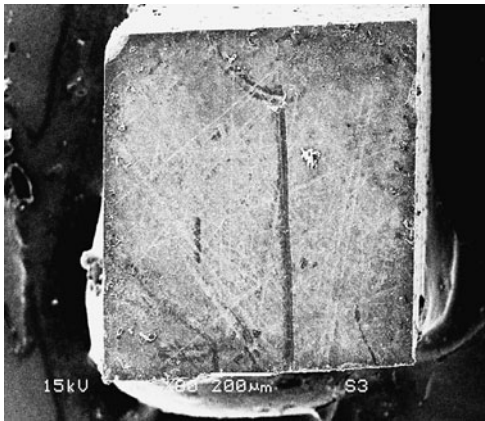


Fig. 1 SEM Image obtained of a fractured specimen ($\times 80$). It shows a fracture type A (predominantly adhesive failure between RCT material and dentin) on a dentin face from Group 2 (Clearfil S3).

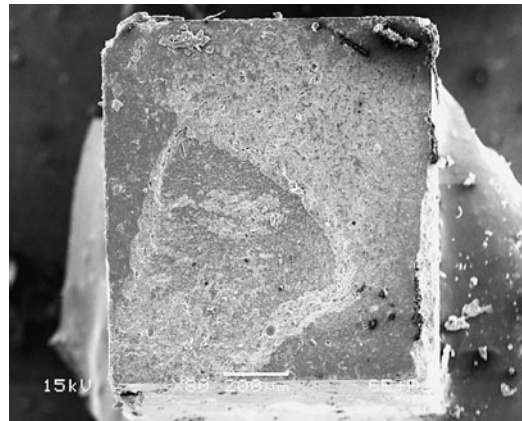


Fig. 2 SEM Image obtained of a fractured specimen ($\times 80$). It shows a fracture type C (cohesive in resin cement) on a restoration face in Group 4 (Clearfil SE Bond).

sive systems were applied. Group 3 (Clearfil S3 + Protect Liner F) showed the second best mean bond strength, differing from other groups, except for Group 4.

The fracture pattern was evaluated by Scanning Electron Microscopy (SEM) after microtensile bond strength testing and the results are shown in Table 4. Some representative images are shown in Figs. 1 and 2.

In Groups 1 and 2, the fracture pattern was predominantly type A (adhesive failure) and included dentin tissue exposure. In Group 3

there were mainly fracture types A and B. For Group 4, fracture type C (cohesive failure) was the most common and in Group 5, type B (mixed failure) was the predominant pattern.

Discussion

The constant development of adhesive systems makes it difficult to conduct long-term *in vivo* research. For this reason, in this study, tension generated by the thermo-mechanical

changes that occur in the oral environment were simulated by means of thermal cycling and load cycling devices. The aim was gain better knowledge of material behaviors under adverse conditions, since most studies perform the bond strength test 24hrs after final cementation and do not show what happens clinically. In an endeavor to achieve closer alignment with clinical conditions, the periodontal ligament was simulated with the use of polyether-based molding material for load cycling, since mastication forces applied on the restoration and tooth are transferred to the periodontal ligament and to adjacent bone inside the mouth, which was represented by the self-polymerizing acrylic resin. This artificial periodontal ligament works as a less rigid and resilient liner that absorbs part of the compressive forces, attenuating stress on the adhesive system and resin cement.

In this study, two self-etch adhesive systems were used. It is believed that application of a self-etch adhesive system has the advantage of reducing post-operative sensitivity in comparison with a conventional etch-and-rinse adhesive system¹⁵. Moreover, if a layer of low viscosity composite is added to an adhesive system, better absorption of the stress caused by the thermo-mechanical process is observed¹⁴. At the same time, better polymerization of the adhesive layer is achieved, since formation of the oxygen-inhibited layer in the adhesive is prevented, and consequently, higher microtensile bond strength is attained¹⁴. In the present research, this could be observed in the groups that used the RCT with the flowable composite (Groups 3 and 5) and which exhibited higher microtensile bond strength than the control group. In addition, Group 5 (CSE + PL) showed statistically higher microtensile bond strength values when compared with the other groups. However, all the bond strength means in this study were relatively lower when compared with other studies. A possible explanation is the fact that the samples were submitted to a high number of both thermal and mechanical stress cycles. Therefore, the null hypothesis that there was no difference between the

bond strengths of all the groups had to be rejected.

Christensen⁵ reported that a low viscosity resin layer on the adhesive system has been reported to be effective in dentin desensitization, avoiding postoperative sensitivity. This same study shows that use of two-step self-etch adhesive systems, such as Clearfil SE Bond, allows adequate bonding to dentin and enamel and ensures postoperative comfort.

The use of a low viscosity composite resin as an intermediate material was shown to be effective in reducing voids at the interface between the restoration and the tooth⁶, acting as an elastic layer to absorb the stress generated by the overlying layer of conventional resin-composite materials characterized by a higher elastic modulus^{3,20}. Extrapolating these findings to this study, they ratify the higher values observed in groups with this low viscosity resin layer (Groups 3 and 5), since all specimens underwent severe thermo-mechanical stress.

Another process that contributes to the values observed in Groups 3 and 5 is the high affinity between low viscosity resin monomers and resin cement monomers, parallel to the lower affinity between the monomers of self-etch adhesive and resin cement, especially with one-step self-etch adhesive systems, since in these systems, both hydrophobic and hydrophilic monomers and acidic and non-acidic monomers are found in the same liquid. This fact makes polymerization difficult and creates a lower quality hybrid layer, decreasing bonding between this type of adhesive and dentin¹⁸. This property can be confirmed in the fracture pattern analysis, which shows high adhesive and mixed failures in Group 2 (Clearfil S3). Composite resins such as Clearfil Protect Liner F are low viscosity resin-based materials, which differ from conventional composites resin only as regards their filler content, unlike conventional composites and resin cements. Flowable composites such as Clearfil Protect Liner F are low viscosity resin-based materials which differ from conventional resin-composites with regard to the quantity of their filler load

content and diluent monomers⁹). The same monomers are found in flowable composites, conventional resin composites and resin cements. Therefore, flowable composites contain the same type of filler particles as those in traditional hybrid composites¹ while they contain 20–25% less filler than non-flowable materials^{1,2}, giving the material a lower elastic modulus.

One-step self-etch adhesive systems are also reported to be permeable membranes¹⁷ and they are even more acidic in nature by this self-etch feature. There is evidence to suggest that there may be undesirable acid-base reactions between acidic monomers in self-etch adhesives and dual polymerized resin cement^{16,18,19}, which could jeopardize resin cement polymerization and bond quality. Thus, in the resin coating technique the adhesive system is applied much earlier than final cementation, right after the preparation and before taking the impression, so this incompatibility is not a significant clinical problem. Nevertheless, it could be meaningful if this type of adhesive system were chosen for the final cementation technique.

As regards the fracture pattern analysis, it was observed that in the group without RCT (control group) there was a predominance of type A fracture, exposing the dentin surface and consequently confirming that in case of a restoration fracture, dentin tissue would be fully exposed and vulnerable. This same fracture pattern occurred in the groups without low-viscosity composite resin (Fig. 1), confirming that the layer of adhesive system alone is not sufficient to protect the dentin tissue. In contrast, a different fracture pattern can be observed when the adhesive system is combined with low viscosity resin, in which there is a predominance of mixed failures between the coating materials, without the exposure of dentinal tissue. This would provide some security in case of a possible fracture or loss of restoration, because the dentin would be protected¹². Nevertheless, the use of only a two-step self-etch adhesive system (without the flowable composite) also showed low exposure of dentinal tissue, exhibiting higher

percentages (40%) of cohesive failure in resin cement (Fig. 2). But this group (G4) showed a statistically lower mean microtensile bond strength than Group 5, in which the flowable composite was used on the same adhesive system.

Therefore, use of RCT, and more specifically, the combination of a self-etch adhesive system/low viscosity resin, may be considered effective, which is in agreement with other studies^{12–14}, especially for the “two-step” self-etch adhesive system. However, it is important to emphasize that the technique used for indirect restorations consists of several steps, including cavity preparation, impression taking, manufacturing the prosthesis and cementation, and each of these steps is crucial to the success of the restoration.

Further *in vivo* and *in vitro* study using the resin coating technique with various combinations is necessary to clarify which is the best type of adhesive system in this technique.

Conclusions

1. The combination of a two-step self-etch adhesive/low viscosity resin yielded the highest tensile bond strength values.
2. The group that did not use the RCT yielded the lowest tensile bond strength values.
3. Fracture pattern analysis confirmed the effectiveness of this technique, particularly in the RCT group with the two-step self-etch adhesive system/low viscosity resin, with regard to not exposing dentin tissue.

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