UNIVERSIDADE ESTADUAL DE CAMPINAS
FACULDADE DE ODONTOLOGIA DE PIRACICABA

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Cirurgião - Dentista

AVALIAÇÃO DA ADAPTAÇÃO MARGINAL, RESISTÊNCIA DE UNIÃO E NANOINFILTRAÇÃO DE RESTAURAÇÕES INDIRETAS UTILIZANDO A TÉCNICA DE SELAMENTO DENTINARIO E CIMENTO RESINOSO

Tese apresentada à Faculdade de Odontologia de Piracicaba, da Universidade Estadual de Campinas, para obtenção do Titulo de Doutor em Materiais Dentários.

Orientador: Prof. Dr. Mário Alexandre Coelho Sinhoreti

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“O que o Mestre é, vale mais que os seus ensinamentos”

Karl Menninger

RESUMO

As restaurações estéticas indiretas têm sido a opção de escolha no caso de cavidades extensas. No entanto, este tipo de restauração demanda um desenho de preparo muito mais invasivo, podendo levar a sensibilidade pós-operatória, assim como também, certa incompatibilidade entre o cimento resinoso e o adesivo. Na tentativa de solucionar estas limitações tem sido sugerida uma técnica chamada de *"Resin Coating Technique"* ou Técnica de Selamento Dentinário (TSD), que consiste em proteger a superfície exposta após o preparo com um sistema adesivo e sobre este é aplicado ou nao um *"liner"* (monômero hidrófobo ou resina composta de baixa viscosidade). A utilização desta técnica vem oferecendo bons resultados, embora, pouco se sabe referente à combinação apropriada entre adesivo e *"liner"* e a interação desta técnica com os diferentes tipos de cimento resinoso. Portanto, este estudo teve como objetivos avaliar a influencia de diferentes combinações da TSD, na adaptação marginal, resistência de união, nanoinfiltração de restaurações indiretas, e após de ter identificado a combinação mais apropriada para a TSD, foi avaliada a interação desta com os cimentos resinosos através dos testes de adaptação marginal e resistência de união. Para estes estudos foram avaliados as combinações de sistemas adesivos que utilizam o condicionamento acido prévio de 2 e 3 passos e adesivos autocondicionantes de 1 e 2 passos seguido da aplicação ou não aplicaçao de uma camada de "liner" (monômero hidrófobo/resina composta de baixa viscosidade). Nas combinações avaliadas os grupos que obtiveram menores resultados tanto para adaptação marginal e resistência de união foram as combinações que não utilizaram *liner*. O grupo adesivo autocondicionante de 2 passos/resina composta de baixa viscosidade apresentou maior resistência de união e menor grau de nanoinfiltração. No entanto, todas as combinações avaliadas de TSD não conseguiram evitar a desadaptação marginal e nanoinfiltração das restaurações. Referente ao comportamento desta combinação com diferentes tipos de cimento, foi avaliada a adaptação marginal e resistência de união de diferentes cimentos resinosos (autocondicionante, autoadesivo e de ativação química) obtendo como resultados que o grupo que obteve melhores valores de resistência de união foi o grupo que utilizou cimento autocondicionante, embora não houve diferença entre os cimentos no que se refere à adaptação marginal. Por tanto, pelos achados destes estudos conclui-se que para obter melhor adaptação marginal utilizando a TSD é necessária a aplicação de um *liner*. A combinação de sistema autocondicionante 2 passos/resina de baixa viscosidade obteve maiores valores de resistência de união e menor padrão de nanoinfiltração. O cimento resinoso autocondicionante mostra-se ser o mais compatível com a TSD.

Palavras Chave: adesivos dentinarios, restaurações intracoronarias, materiais dentarios.

ABSTRACT

Indirect esthetic restorations have been considered an excellent restorative alternative for extensive cavities. However, this type of restoration demands a more invasive preparation, which may cause post-operative sensibility and incompatibility between resin cement and adhesive system. In attempt to minimize these limitations the Resin Coating Technique (RCT) has been proposed. This technique consists in protecting the exposed dentin after the cavity preparation with an adhesive, followed by the application or not application of a liner (hydrophobic monomer or low viscosity resin). The effectiveness of this technique was reported in several studies, however, little is known about the appropriate combination between adhesive and liner and the behavior of this RCT with different resin cements. Therefore, the aims of this study were: evaluate the influence of the combination of different materials used in the RCT on the marginal adaptation, bond strength and nanoleakage of indirect restorations and after identify the most appropriate combination for the RCT, was evaluated the behavior of this RCT with different resin cements using the marginal adaptation and bond strength tests. For these studies were evaluated etch and rise adhesive system (2 and 3 steps) and self-etch adhesives (2 and 1step) followed of the application of liner layer (hydrophobic monomer or low viscosity resin). In all evaluated combinations, the groups that showed low results in marginal adaptation and bond strength were the combinations that not used a liner. The group self etch 2 steps/flow composite resin, showed high bond strength and low nanoleakage. However, in all the restorations showed marginal disadaptation and nanoleakage, independent of the material combinations used for the resin coating. In the study about the interaction of the RCT with the type of resin cement (Self-etching, Self-Adhesive and Chemical Cure) evaluating marginal adaptation and bond strength, was obtained the highest bond strength for the Self-etch group, however, there is no difference between groups for marginal adaptation. So, within the limits of this study, it can be concluded that the use of a liner is necessary for a better marginal adaptation using the RCT. The combination of self etch 2steps/flow composite resin showed high bond strength and low nanoleakage. The self etch resin cement revealed high bond strength showing more compatibility with the RCT.

Key words: dentin bonding agent, inlays, dental materials.

SUMÁRIO

INTRODUÇÃO

Nos últimos anos, tem ocorrido maior demanda por restaurações estéticas de parte dos pacientes, o que em parte é justificado pelo avanço significativo dos materiais restauradores. No caso de cavidades extensas, as restaurações indiretas são a opção de escolha. No entanto, a técnica demanda de um desenho de preparo muito mais invasivo, sendo desgastada dentina sadia e por conseqüência, maior incidência de sensibilidade pós-operatória (Xie *et al*., 1993; Kaneshime *et al*., 2000).

Além disso, durante o processo de cimentação destas restaurações, pode existir certa incompatibilidade, de alguns sistemas adesivos (convencional-2passo e autocondicionante-1passo) com os cimentos resinosos (especificamente de ativação dual ou química). Isto é causado principalmente pela presença de monômeros que não reagiram na superfície do adesivo, que ao entrar em contato com o cimento resinoso reagiria com as aminas terciárias (que possuem caráter alcalino) consumindo estas e impedindo que estas atuem como ativadoras da reação de polimerização do cimento resinoso. (Ikemura, *et al*. 1999;Sanares *et al.*, 2001).

Não somente a incompatibilidade entre o cimento resinoso e o sistema adesivo pode influenciar na união das restaurações indiretas. Fatores clínicos como contaminação por saliva, sangue e a utilização de materiais provisórios podem interferir na união entre a restauração e o dente, levando a uma inadequada adaptação marginal e conseqüentemente, à formação de fenda ao redor da restauração, podendo levar à sensibilidade pós-operatória (Kaneshime *et al*., 2000).

A contínua evolução dos sistemas adesivos tem possibilitado um aumento significativo de alternativas restauradoras, permitindo assim, o desenvolvimento de novas técnicas para o controle dessas variáveis.

Nos últimos anos, tem sido proposta uma técnica que possibilita a proteção da dentina após o preparo cavitário, assim como, melhora na qualidade de união do cimento resinoso, sendo esta chamada de "Técnica de Selamento Dentinário" ou *Resin Coating Technique* (Satoh *et al*., 1994). Esta técnica consiste em hibridizar a dentina exposta utilizando um sistema adesivo, seguido de uma cobertura ou nao com adesivo de caráter hidrófobo ou com compósito de baixa viscosidade. Este procedimento é realizado imediatamente após a confecção do preparo, sendo que após esse selamento, seria realizada a moldagem para a confecção da peça protética. Deste modo, esta técnica minimizaria a irritação pulpar causada tanto por estímulos térmicos e mecânicos, assim como pela infiltração bacteriana, que pode ocorrer durante a realização da moldagem, colocação do material provisório ou durante a cimentação definitiva (Nikaido *et al*., 2003a).

Com esta técnica, também se eliminaria o problema de incompatibilidade, devido a que essa camada adicional de *liner* (monômero hidrófobo ou resina de baixa viscosidade) reagiria diretamente com os monômeros que não reagiram do adesivo, evitando assim a reação destes com as aminas terciárias do cimento resinoso.

Incluso, foi demonstrado, que utilizando esta técnica, pode-se conseguir maiores valores de resistência de união com o cimento resinoso, já que uma das limitações dos sistemas adesivos de caráter hidrófilo, diz respeito a possibilidade de que poderiam agir como uma membrana semipermeável após sua polimerização, permitindo a movimentação de fluidos dentinários através da camada de adesivo, prejudicando a união e a polimerização do cimento resinoso (Tay, et al 2002; Carvalho *et al*., 2004).

Por este motivo, a aplicação de uma camada de adesivo de característica hidrófoba ou de um compósito de baixa viscosidade sobre a superfície hibridizada, iria reduzir a permeabilidade da camada de adesivo e promover melhor união ao cimento resinoso. (Jayasooriya *et al*., 2003a; Nikaido *et al.,* 2003b).

O uso de sistemas adesivos autocondicionantes nessa técnica é clinicamente mais atraente, porque estes sistemas podem ser aplicados sobre a superfície da dentina recém preparada e seca. Quando o *primer* acídico é aplicado e removido com jato de ar, não precisa ser realizada a lavagem, sendo assim, este procedimento é menos crítico por ter menor número de passos e sem a necessidade de deixar a superfície de dentina úmida. Além disso, o condicionamento com o *primer* acídico leva a menor profundidade de desmineralização da dentina, possibilitando maior preenchimento pelo adesivo e diminuindo assim o problema de sensibilidade pós-operatória (De Munck *et al*., 2005).

Como pôde ser observado, inúmeras vantagens são proporcionadas quando se utiliza esta nova técnica. Por este motivo alguns estudos têm sido realizados avaliando esta técnica no que se refere à resistência adesiva e adaptação marginal, obtendo ótimos resultados(Jayasooriya *et al*., 2003a,b; Nikaido *et al*., 2003b; Nikaido *et al.,* 2008).

Não obstante, ainda se sabe muito pouco e não existe um consenso sobre qual combinação de materiais seria a mais adequada (adesivo/monômero hidrófobo ou adesivo/compósito de baixa viscosidade) e a interação desta técnica com os diferentes tipos de cimento resinoso. Outro fator importante a considerar é a longevidade clínica, que também é uma das principais preocupações tanto do clínico como do paciente. Por este motivo, este estudo tentou realizar *in vitro* a simulação mais próxima possível do que ocorre na cavidade bucal, ou seja, confeccionando-se preparos cavitários (a maioria dos estudos foram realizados em superfície plana) e simulando as oscilações térmicas e mecânicas que ocorrem na cavidade bucal.

Para tanto, este trabalho de tese foi confeccionado em formato alternativo dividido em quatro capítulos, tendo como objetivos: avaliar a adaptação marginal (capítulo 1), resistência adesiva (capítulo 2) e nanoinfiltração (capítulo 3) de restaurações indiretas utilizando diferentes combinações da "Técnica de Selamento Dentinário". Após ter identificado a combinação mais apropriada para esta técnica, foi avaliada a interação desta combinação com diferentes tipos de cimento resinosos, através dos testes de adaptação marginal e resistência de união (capítulo 4).

CAPÍTULO 1

Evaluation of the marginal adaptation of indirect restorations using different protocols of resin coating

Short Title: Marginal adaptation using resin coating

Clinical Relevance

For indirect restorations, the use of a liner for the Resin Coating Technique, can reduce marginal gap formation.

SUMMARY

The aim of this study was to evaluate the influence of the combination of different materials used in the resin coating technique (RCT) on the marginal adaptation of indirect restorations with margins in enamel and cement after thermal and load cycling. The tested hypothesis is that the different combinations of materials for the RCT influence the marginal adaptation of the indirect restorations. Eighty extracted third molars were used in this study. Two cavities were prepared in each tooth, one on the mesial surface (gingival margin in enamel-EM) and the other on the distal surface (gingival margin in cement-CM). The 160 cavities were distribuited into sixteen groups according to the adhesive system, liner material and cavity margins. Cavities with margins in enamel were restored with the following material combinations: G1: Single Bond 2 (Sb2), G2: Sb2 + Bond Scotchbond Multi-Purpose (Sb2B), G3: Sb2 + Filtek Flow Z350 (Sb2Fl), G4: Scotchbond Multi-Purpose (SBMP), G5: Clearfil S3 (CS3), G6: CS3 + Bond Clearfil SE Bond (CSE3B), G7: CS3 + Protect Liner F (CS3PL), G8: Clearfil SE Bond + Protect Liner F (CSEBPL),). The same combinations were applied to the cavities with margins in cement, corresponding to the following groups: G9, G10, G11, G12, G13, G14, G15 and G16, respectively. The cavities were molded with a vinyl polysiloxane impression material (Aquasil/Dentsply) and the molds were poured with a stone plaster (Velmix-Kerr). The fillings were confectioned using the Sinfony composite system (3M/ESPE) and were cemented with resin cement (Rely X ARC-3M/ESPE). After 24 hours, the teeth were submitted to thermocycling (2000 cycles / 5-55°C) and load cycling (250,000 cycles / 30N). Following, Caries Detector (Kuraray) was applied to the restoration margins and washed for 30 seconds. Images from the proximal view of the teeth were captured and evaluated using the software Image Tool 3.0 (University of Texas, USA) for the measurement and percentage calculation of the gap formation. The results were submitted to ANOVA and Tukey's test ($p<0.05$). The mean values (%) for the groups with EM were: G1=46.68, G2=15.53, G3=19.83, G4=27.53; G5=59.49, G6=25.13, G7=34.37, G8=15.20, and for CM: G9=38.38, G10=23.25, G11=26.97, G12=25.85, G13=37.81, G14=30.62, G15=29.17, G16=20.31. The statistical analysis showed significant differences between groups, for either enamel or dentin margins. The highest values of marginal disadaptation in enamel and cement margins were represented by the groups that not used a liner Sb2 and CS3. The other groups presented intermediate values. Within the limits of this study, it can be concluded that the more appropriate combination for the RCT are the groups that used a liner. However, all the restorations either with margin in enamel or cement, presents gap formation independent of the RCT combination..

Key words: marginal adaptation, resin coating, adhesives, indirect restorations

INTRODUCTION

Esthetic indirect composite restorations have been considered an excellent restorative alternative for extensive cavities. The indirect process of confection the restorations, allow the reproduction of a good anatomic form and proximal contact, in addition to the control of the polymerization shrinkage. On the other hand, this type of restoration demands a more invasive preparation, which may cause pulp irritation due to the exposure of deep dentin. Clinical studies evaluating indirect restorations have reported up to 30% of hypersensitivity after the treatment.[1] Attempting to minimize this hypersensitivity the Resin Coating Technique (RCT) has been proposed.[2] This technique consists on the hybridization of the exposed dentin followed by the application of a hydrophobic monomer or a low viscosity resin, protecting the dentine exposed after the cavity preparation.

The stress caused by the polymerization shrinkage in indirect restorations is limited to the resin cement layer. The presence of a resin coating is believed to act as an absorbent layer of the tensions originated by the polymerization shrinkage of the resin cement and the stress induced by the masticatory forces, increasing the possibilities of clinical success of the restorations. It was previously reported by Jayasooriya & others in 2003,[3] who observed a considerable reduction in the formation of microcracks in the interface tooth-restoration using the RCT. The effectiveness of this technique was reported in several studies [4,5] ; however, these studies were 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.[6,7] Thermal and mechanical stresses, undergone by restorations in the oral environment, may be simulated *in vitro* using thermal and load cycling.

One way to predict the clinical success of the restorations in vitro is evaluating the marginal adaptation. Studies have shown that improvements in marginal adaptation may be achieved using the Resin Coating Technique (RCT); however, little is known about the maintenance of the marginal adaptation when the restorations are submitted to stresses present in the clinical situation. Therefore the aim of this study was to evaluate the influence of the combination of different materials used in the RCT on the marginal adaptation of indirect restorations with margins in enamel and cement, after thermal and load cycling. The hypothesis tested is that the different associations of materials used for the resin coating, influence the marginal adaptation of the indirect restorations.

MATERIALS AND METHODS

Sample preparation

Eighty extracted third molars were used in the study, under the approval of the Committee of Ethics in Research from the Piracicaba School of Dentistry – University of Campinas (113/2005). The teeth were embedded in acrylic resin leaving 2 mm of root exposed. The periodontal ligament was simulated applying a layer of polyether (Impregum, 3M ESPE, AG Seefeld, Germany) over the roots.[8] Two Class II cavities were prepared using diamond burs (#4137 KG Sorensen, Barueri, SP, Brasil). The cavities had the following dimensions: 4 mm of bucco-lingual width and 3 mm of proximal-axial width. The gingival margin of the mesial cavity was located 1 mm below the cement-enamel junction (enamel margin – EM), while in the distal cavity the margin was located 1 mm above it (cement margin – CM). The dimensions and characteristics of the cavities are detailed in Figures 1a, 1b and 1c. Were randomly distributed in 16 groups (n=10). The materials used in each group, composition and the application techniques are described in Table I.

Table 1. Materials, brand (Lot#), composition, application technique, and manufactures materials used in this study (Bis-GMA:bisphenol-A diglycidil ether dimethacrylate, HEMA: 2-hydroxyethyl metacrylate, MDP: 10 metacryloyloxydecyl dihidrogen phosphate, TEGDMA: triethylene glycol dimetacrylate

Materials	Brand Lot #	Composition	Aplication
Groups1 and 9 **Etch and Rise 2 step** (Sb2)	Single Bond 2 Etchant #(5EN) Adhesive#(5EP)(3M/ESPE)	- Etchant: 35%H_3PO_4, gel sílica -Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate, nanofiller	a (15s), b (15s), c , d, e, d, e, l (10s)
Groups 2 and 10 **Etch and Rise 2 step + Hydrophobic Monomer**. (Sb2B)	Single Bond 2 Etchant#5EN Adhesive #5EP Bond SBMP #(5PH) (3M/ESPE)	- Etchant: 35%H_3PO_4, gel sílica - Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate -Bond SBMP: Bis-GMA, HEMA, photoinitiator	a (15s), b (15s), c , d, e, d, e, l (10s) k, l (10s)
Groups3 and 11 **Etch and Rise 2 step + Flow composite resin** (Sb2Fl)	Single Bond 2 Etchant#5EN Adhesive #5EP Filtek flow #6031A2 (3M/ESPE)	-Etchant: 35%H_3PO_4, gel sílica - Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate FiltekFlow: Bis-GMA, TEGDMA, Zirconia, Sílica, camphorquinone, nanofilller	a (15s), b (15s), c , d, e, d, e, l (10s) Filtek Flow: i, l (20s)
Groups 4 and 12 **Etch and rise 3 step** (SBMP)	Scotch Bond Multipurp. Etchant #5EN Primer #5BB Bond #5PH (3M/ESPE)	- Etchant 35%H_3PO_4, gel sílica - Primer: HEMA, polyalquenoic acid copolymer - Bond: Bis-GMA, HEMA, photinitiator	a (15s), b (15s), c j, e, k, l (10s)
Groups 5 and 13 **Self etch 1 step** (CS3)	Clearfil S3 # 00001A Kuraray	CS3: MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, photoinitiator, ethanol.	h (20s), e (5s), l (10s)
Groups 6 and 14 **Self etch 1 step + Hydrophobic Monomer** (CS3B)	Clearfil S3 # 00001A Bond Clearfil SE Bond Batch #(00773A) Kuraray	CS3: MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, photoinitiator, ethanol. Bond: MDP, Bis-GMA, HEMA, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine	h (20s), e (5s), l (10s) g, e (5s), l(10s)
Groups 7 and 15 **Self etch 1 step + Flow composite resin** (CS3PL)	Clearfil S3 # 00001A Protect Liner F # 0046 Kuraray	CS3: MDP, Bis-GMA, HEMA, dimetacrylates, fotoiniciador ProtectLiner:Bis-GMA,TEGDMA, fluoride methil methacrylate, camphorquinone, silanized colloidal silica.	h (20s), e (5s), l (10s) Protect Liner: l (20s)
Groups 8 and 16 **Self etch 2 step + Flow composite resin.** (CSEBPL)	Clearfil SE Bond Primer #00727A Bond #01044 Protect Liner F # 0046 Kuraray	-CSEB Primer: MDP, HEMA, water, ethanol, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine. -CSEB Bond: MDP, Bis-GMA, HEMA, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine ProtectLiner F:BISGMA,TEGDMA.	f (20s) , e , g, e (5s), l(10s) Protect Liner: l (20s)

Application technique: a: acid technique; b: rinse surface; c: dry with cotton-pellet; d: apply one layer total etch one step adhesive; e: gently air dry; f: apply primer two step self etch adhesive; g: apply bond two step self etch adhesive; h: apply one layer self etch one step adhesive; i: apply one layer resin flow; j: apply primer three step etch and rise adhesive, K: apply bond three step etch and rise adhesive l: light cure

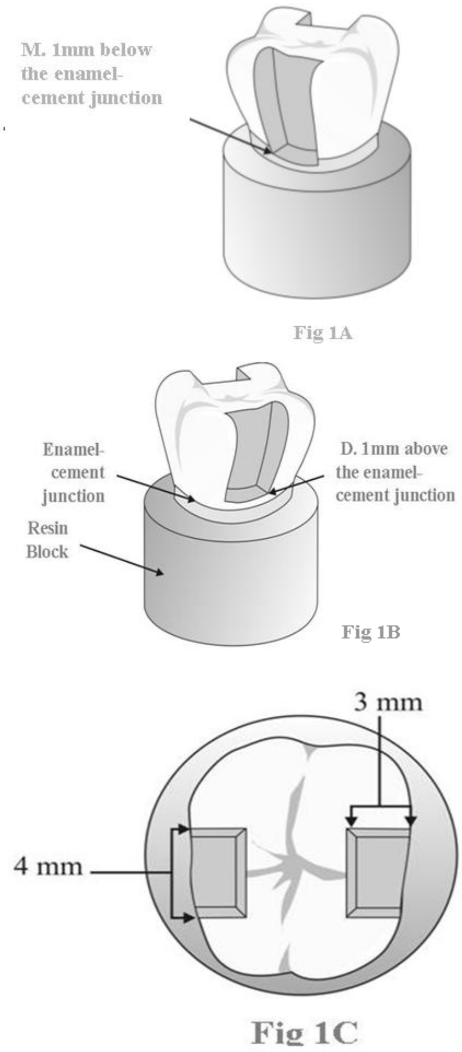

Figure 1 - Characteristics and measurements of class II cavity. a. Mesial view; b. Distal view; c. Occlusal view of the cavity preparation.

Following the application of the different RCT, the cavities were molded with vinyl polysiloxane impression material (Aquasil, Dentsply DeTrey, Konstanz, Germany), and the molds obtained were poured with die gypsum type IV, Velmix (Kerr Dental Laboratory

Products, Orange CA, USA). The restorations were made using the Sinfony System (3M ESPE AG, Seefeld, Germany).

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 in a single layer and light-cured with a quartz-tungsten-halogen light-curing unit (500mW/cm^2) (XL2500, 3M ESPE, St. Paul, MN, USA) for 10s. The internal surface of the restorations were sandblasted with 50µm Al_2O_3 powder at 2-bar pressure and treated with 35% phosphoric acid (3M ESPE AG, Seefeld, Germany) for 1 min. Following, a silane layer (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 and light-cured for 10s. The resin 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 cement was removed and restoration was light-cured for 40s from each surface of the tooth. Finishing was performed with fine and extra-fine grit diamond burs (2135F and 2135FF, KG Sorensen, Barueri, SP, Brasil) and polished with 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 5^o to 55^{o}C, 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 6 mm of diameter and rounded extremities, these tips keep in contact with the occlusal surface of the restorations. The equipment applied an intermittent axial force of 50N at a frequency of 2 Hz, totalizing 250,000 cycles. The test was performed under water.

Evaluation of the marginal adaptation

In order to evaluate the marginal adaptation of the restorations, a solution consisting of propylene-glycol and acid red 52 Caries Detector (Kuraray Medical Inc, Okayama, Japan) was applied on the proximal surface of each restoration during 10s. The solution was washed and dried with absorbent paper. Images of the proximal surface of the restorations were captured using a digital camera SLR Canon (Rebel XT, Lake Success, NY, USA) equipped with a Lens 105mm f2.8 Sigma Corporation (EXDG Macro, Kanagawa, Japan) and transferred to a computer equipped with Image Tool 3.0 software (Periodontology Departament, University Texas, Health Science Center, San Antonio, TX, USA). The software was used to measure the gap formation in the margin of the restoration (dentin or cement). The values were converted in percentage, based on the ratio of the gap formation length and the total length of the restoration margins. The values were submitted to statistical analysis (ANOVA and Tukey's test $p<0.05$).

RESULTS

All the groups presented gap formation, either in EM or CM. Table 2 summarize the results of dye penetration in EM. Groups Sb2B (15.53%) and CSEBPL (15.20%) showed significant reduces on gap formation when compared with the others groups. The groups that not used a liner Sb2 (36.68%) and CS3 (59.49%), showed the highest values of marginal disadaptation.

Table 2 - Mean values of gap formation (%) of indirect restorations with margin in enamel

Resin Coat	Mean values	Standard deviation
Sb2	46.68 C	± 12.29
Sb2B	15.53A	± 13.04
Sb2Fl	19.83AB	± 09.12
SBMP	27.53 B	± 16.09
CS3	59.49 C	± 09.18
CS3B	25.13 B	± 12.17
CS3PL	34.37 B	± 13.24
CSEBPL	15.20A	± 10.79

Mean values followed by different letters showed significant difference (Tukey5%).

Results of gap formation in CM are shown in Table 3. The highest gap formation was obtained with the groups that not used liner Sb2 (35.38%) and CS3 (37.81%). The others groups showed similar values.

Table 3 - Mean values of gap formation (%) of indirect restorations with margin in cement

Resin Coat	Mean values	Standard deviation
Sb2	38.38 B	± 08.32
Sb2 B	23.25 A	± 09.59
Sb2Fl	26.97 A	± 06.32
SBMP	25.85 A	± 15.83
CS3	37.81 B	± 07.43
CS3B	30.62 AB	± 13.83
CS3PL	29.17 A	± 12.38
CSEBPL	20.31 A	± 07.72

Mean values followed by different letters showed significant difference (Tukey 5%)

DISCUSSION

The results of the present study revealed significant differences between the combinations of materials used for the RCT, confirming the tested hypothesis. Marginal adaptation is one of the most important aspects in indirect restorations, because it influences their longevity. It is well known that the cement layer is considerably challenged by the chewing load.[9] A suitable alternative to reduce the stress produced by the masticatory forces and also by the polymerization shrinkage of the resin cement is the RCT, which distributes the stress that would concentrate in the cement layer to the hybridized dentin and the liner material.

The present study focused on the type of materials used for the RCT. Due to constant and fast evolution of the dental materials, that do not allow evaluations long-term clinical trials, mechanical and thermal cycling were carried out allowing the simulation *in* *vitro* of what would happen *in vivo*.[10,11] Tests of thermal and load cycling have been very used in an attempt to simulate the fatigue of the restorations in the oral environment. In theory, the fatigue resulting from the masticatory forces generates stresses in the interface tooth/restoration, increasing the damages in this area.[5,10, 12-19] Moreover, the temperature changes induce interfacial stresses due to differences of contraction and expansion between the tooth structures and the materials involved; these tensions may cause microcracks, which propagate throughout the adhesive interface and result in fluid percolation through the gap formed.[20] Therefore, the thermo-mechanical simulation might provide results closer to the clinical situation.

Resin cements produce high levels of stress during polymerization. Besides, the incorporation of air bubbles during the manipulation of the cement weakens it and makes it more susceptible to fractures. The marginal disadaptation observed in all groups might have resulted from these factors. Yet, these results presented higher gap formation than previous studies,[21] probably due to the stress promoted by the thermal and load cycling. Still, it is suggested that other factors might have influenced our results in comparison with other studies, such as the type of preparation, the number of cycles and the load applied during cycling.[3,7,16,18]

It can be assumed, that the sealing of the dentin with a layer of hydrophobic monomer or a low viscosity composite could have absorbed part of the stress caused by the polymerization shrinkage of the cement or the load cycling, promoting better marginal adaptation.[3, 14, 22] The marginal adaptation might have been also influenced by the mechanical properties of the materials used for sealing, especially by the modulus of elasticity. The adhesive layer without a liner may not be thick enough to support the stresses generated. [23] Previous studies have shown that the use of liner (hydrophobic monomer or low viscosity resins), might potentially absorb the stress caused by the polymerization shrinkage and the masticatory forces, improving the marginal adaptation of the restoration[24,25,26] the lower gap formation of the groups that used a liner, also confirm the influence of this property on the marginal adaptation of the indirect restorations.

However, if the modulus of elasticity of the liner is too low, it may deform under loading. This might explain the results observed in groups CS3 and Sb2, that only was applied a layer of adhesive without a liner.[16] Another important factor on the marginal adaption of restorations is the number of layers. The application of a liner avoid the inhibited layer by oxygen that occurs during the polymerization, improves the cure degree and consequently higher mechanical properties supporting better the stress, compared with the groups that used only a coat of adhesive.

A high percentage of gap formation was observed in group CS3. This adhesive presents low viscosity, resulting in a thin adhesive layer. The polymerization of this layer, in the presence of oxygen, may be limited, weakening the sealing of the tooth/restoration interface. Moreover, this adhesive is mainly constituted by hydrophilic monomers and solvent,[27,28] which generate low cohesive resistance. As a result, the adhesive is incapable of supporting the chewing stress.[29,30]

CONCLUSIONS

1. The groups that not used a liner presented highest marginal disadaptation.
2. All the indirect restorations showed marginal disadaptation, independent of the material combinations used for the resin coating.

Acknowledgements

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CAPÍTULO 2

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

ABSTRACT

Purpose: The aim of this study was to evaluate microtensile bond strength (u-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 Class II cavities were prepared in each tooth (mesial and distal surface). The 70 cavities were distributed in 7 groups, according to the materials used for resin coating: G1:Etch-rise 2steps(SB2);G2:Etch-rise 2steps/Hydrophobic-monomer(SB2B), G3:Etch-rise 2steps/Flow composite-resin(SB2Fl), G4:Self-etch 1step(CS3), G5:Self-etch 1step/Hydrophobic-monomer(CS3B), G6:Self-etch1step/Flow composite-resin(CS3/Fl), G7:Self-etch 2step/ Flow composite-resin (CSEB/Fl). 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 cement (Rely X ARC system). After 24 hours, the teeth were submitted to thermocycling (2000C/5-55°C) and load cycling (250,000C/30N). After, the restored teeth were sectioned in to serial slabs and u-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, CS3+B ($p<0,05$) compared with the other groups. In relation to the failure mode, in most samples of the groups that not were used a liner, was observed dentine exposed.

Conclusion: The groups CSEB+PL and CS3B showed the highest values of bond strength and in relation at the failure mode reveal good performance not expose dentin tissue.

CLINICAL SIGNIFICANCE

The indirect restorations that used the resin coating combination: 2-step self etch adhesive associated to a flowable composite resin or 1-step self etch adhesive associated to monomer hydrophobic, showed better performance than the other groups obtaining higher bond strength and not expose dentine tissue after fracture.

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, may lead to a postoperative sensitivity.[1, 2]

Attempting to minimize this hypersensitivity the Resin Coating Technique (RCT) has been proposed.[3] 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 a final impression.[4] The advantages of the RCT are to minimize pulp irritation and hypersensitivity caused by mechanical and thermal stimuli and bacterial infiltration, which can occur during impression taking, confection of the provisory restoration and final cementation.

The effectiveness of this technique was reported in several studies evaluating bond strength[5,6]; however, these studies were 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.[7,8] 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.[9]

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 tested hypothesis is that the different associations of materials used for the RCT, do not influence in the bond strength and failure modes of the indirect restorations.

MATERIALS AND METHODS

Sample preparation

Thirty five extracted third molars were used in the study, under the approval of the Committee of Ethics in Research from the Piracicaba School of Dentistry – University of Campinas (113/2005). The teeth were embedded in epoxi resin leaving 2 mm of root exposed. The periodontal ligament was simulated applying a layer of polyether (Impregum, 3M ESPE AG, Seefeld, Germany) over the roots.[10] Two Class II cavities were prepared using diamond burs (#4137 KG Sorensen Barueri SP, Brasil). The cavities had the following dimensions: 4 mm of bucco-lingual width and 3 mm of proximal-axial width. 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, composition and the application techniques are described in Table I.

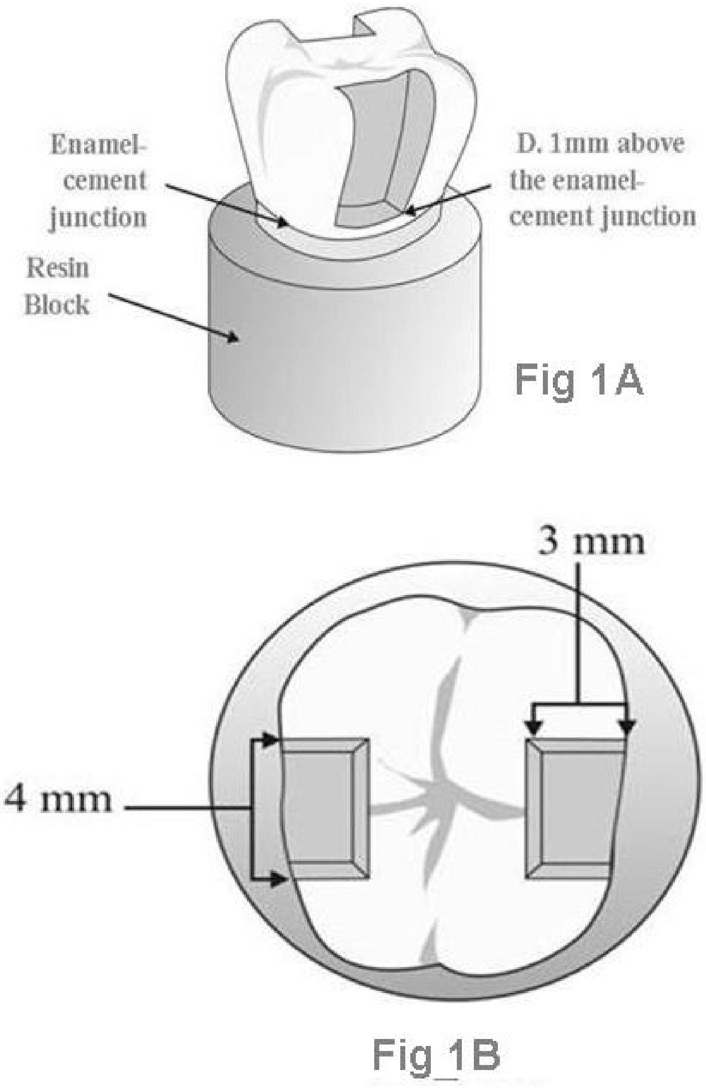

Figure 1 - a. Characteristics and measurements of class II cavity. b. Occlusal view of the cavity preparation.

Table 1. Materials, brand (Lot#), composition, application technique, and manufactures materials used in this study (Bis-GMA:bisphenol-A diglycidil ether dimethacrylate, HEMA: 2-hydroxyethyl metacrylate, MDP: 10 metacryloyloxydecyl dihidrogen phosphate, TEGDMA: triethylene glycol dimetacrylate

Materials	Brand Lot #	Composition	Application
Group SB2 **Etch and Rise 2 step**	Single Bond 2 Etchant #(5EN) Adhesive#(5EP)(3M/ESPE)	- Etchant: 35%H_3PO_4, gel sílica -Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate, nanofiller	a (15s), b (15s), c , d, e, d, e, l (10s)
Group SB2B **Etch and Rise 2 step + Hydrophobic Monomer**.	Single Bond 2 Etchant#5EN Adhesive #5EP Bond SBMP #(5PH) (3M/ESPE)	- Etchant: 35%H_3PO_4, gel sílica - Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate -Bond SBMP: Bis-GMA, HEMA, photoinitiator	a (15s), b (15s), c , d, e, d, e, l (10s) k, l (10s)
Group SB2Fl **Etch and Rise 2 step + Flow composite resin**	Single Bond 2 Etchant#5EN Adhesive #5EP Filtek flow #6031A2 (3M/ESPE)	-Etchant: 35%H_3PO_4, gel sílica - Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate FiltekFlow: Bis-GMA, TEGDMA, Zirconia, Sílica, camphorquinone, nanofilller	a (15s), b (15s), c , d, e, d, e, l (10s) Filtek Flow: i, l (20s)
Group CS3 **Self etch 1 step**	Clearfil S3 # 00001A Kuraray	CS3: MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, photoinitiator, ethanol.	h (20s), e (5s), l (10s)
Group CS3B **Self etch 1 step + Hydrophobic Monomer**	Clearfil S3 # 00001A Bond Clearfil SE Bond Batch #(00773A) Kuraray	CS3: MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, photoinitiator, ethanol. Bond: MDP, Bis-GMA, HEMA, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine	h (20s), e (5s), l (10s) g, e (5s), l(10s)
Group CS3PL **Self etch 1 step + Flow composite resin**	Clearfil S3 # 00001A Protect Liner F # 0046 Kuraray	CS3: MDP, Bis-GMA, HEMA, dimetacrylates, fotoiniciador ProtectLiner:Bis-GMA,TEGDMA, fluoride methil methacrylate, camphorquinone, silanized colloidal silica.	h (20s), e (5s), l (10s) Protect Liner: l (20s)
Group CSEBPL **Self etch 2 step + Flow composite resin.**	Clearfil SE Bond Primer #00727A Bond #01044 Protect Liner F # 0046 Kuraray	-CSEB Primer: MDP, HEMA, water, ethanol, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine. -CSEB Bond: MDP, Bis-GMA, HEMA, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine ProtectLiner F:BISGMA,TEGDMA.	f (20s) , e , g, e (5s), l(10s) Protect Liner: l (20s)

Application technique: a: acid technique; b: rinse surface; c: dry with cotton-pellet; d: apply one layer total etch one step adhesive; e: gently air dry; f: apply primer two step self etch adhesive; g: apply bond two step self etch adhesive; h: apply one layer self etch one step adhesive; i: apply one layer resin flow; j: apply primer three step etch and rise adhesive, K: apply bond three step etch and rise adhesive l: light cure

Following the application of the different RCTs, the cavities were molded with vinyl polysiloxane impression material (Aquasil, Dentsply DeTrey, Konstanz, Germany), and the molds obtained were poured with die gypsum type IV, Velmix (Kerr Dental Laboratory Products, Orange CA, USA). The restorations were confectioned using the Sinfony System (3M ESPE AG, Seefeld, Germany).

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 in a single layer and light-cured with a quartz-tungsten-halogen light-curing unit (500mW/cm^2) (XL2500, 3M ESPE, St. Paul MN, USA) for 10s. The internal surface of the restorations were sandblasted with 50µm Al_2O_3 powder at 2-bar pressure and treated with 35% phosphoric acid (3M ESPE AG, Seefeld, Germany) for 1 min. Following, a silane layer (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 and light-cured for 10s. The resin 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 cement was removed and restoration was light-cured for 40s from each surface of the tooth. Finishing was realized with fine and extra-fine grit diamond burs (2135F and 2135FF, KG Sorensen, Barueri SP, Brasil) and polished with 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 5^{o} to 55^{o}C, 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 6 mm of diameter and rounded extremities, these tips keep in contact with the occlusal surface of the restorations. The equipment applied an intermittent axial force of 50N at a frequency of 2 Hz, totalizing 250,000 cycles. The test was performed under water.

Microtensile bond strength

After the thermal and load cycling, the teeth were retired of the epoxy resin and was removed the enamel tissue present on the proximal areas 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 sectioned by the same method 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 device[11] and tested to failure under tension in a universal testing machine Instron (Model 4411, Corona, Ca, USA) with a 500-N 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 2.

Table 2 – 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 with the value of "Zero". Statistically significant differences were observed between groups ($p<0.05$) as described in Table 3. Bond strength were significant higher in the groups

CSEB+PL, CS3+B ($p<0,05$). Lowest bond strength was obtained with the group CS3 ($p<0.05$). The others groups showed intermediate values.

Table 3 – Microtensile bond strength (MPa) of indirect restorations using different combinations of resin coating

Resin Coating	Mean values Standard deviation
Sb2	$11.24^{B} \pm 8.05$
Sb2B	$12.59^{B} \pm 4.43$
Sb2Fl	$14.28^{AB} \pm 5.28$
CS3	$6.50^{C} \pm 10.34$
CS3B	$16.51^{A} \pm 5.23$
CS3PL	$9.48^{BC} \pm 6.89$
CSEBPL	$16.42^{A} \pm 4.58$

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

Fracture modes are summarized in the Figure 2, representative SEM photographs of the debonded specimens are showed in the Figures 3-6. In all the groups that using a liner over the adhesive system was observed mixed failure in 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.

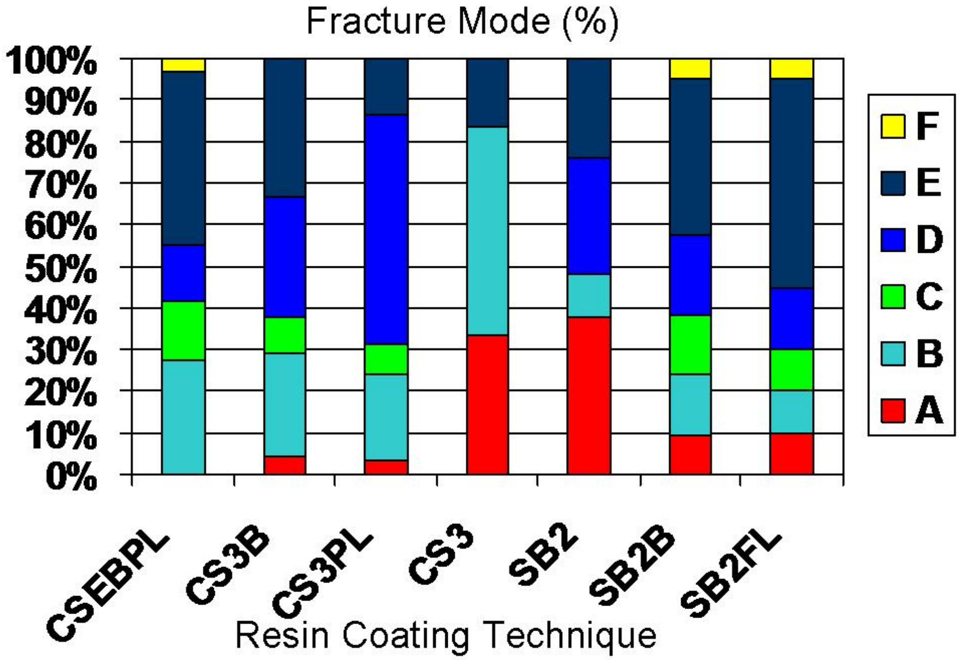

Figure 2. Failure mode after microtensile bond strength test (%).

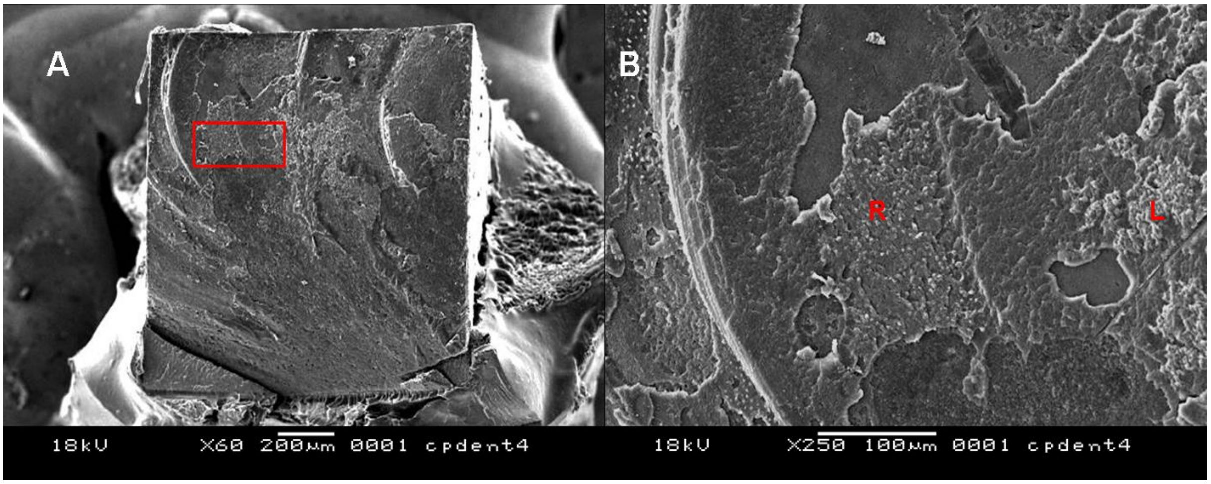

Figure 3. Representative SEM photographs of the debonded specimen that used CSEB/PL combination. Mixed failure between Resin Coating material and resin cement. Magnification: A: 60X, B: 250X. (L:Liner, R: Resin Cement).

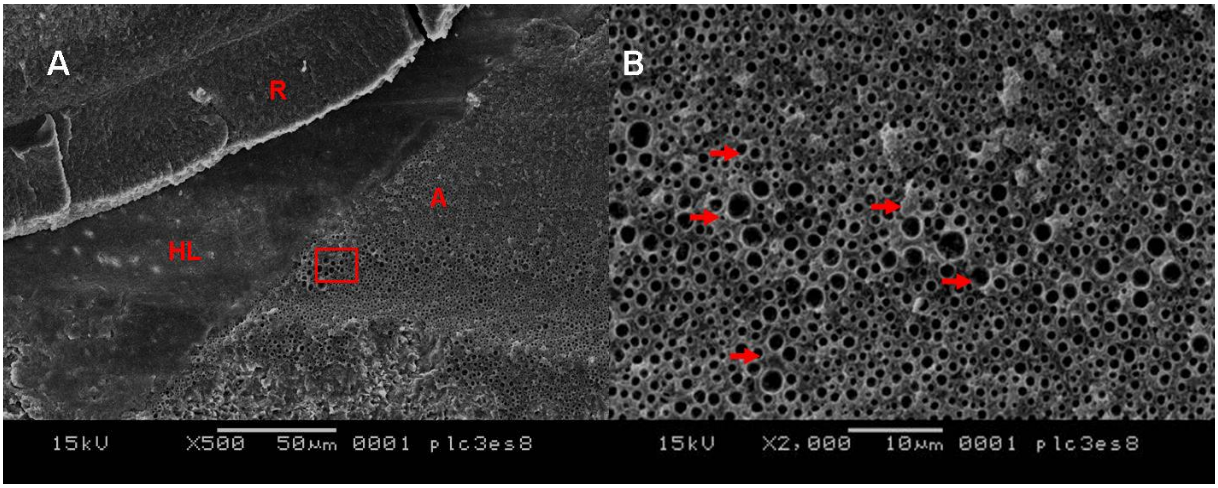

Figure 4. Representative SEM photographs of the debonded specimen that used CS3/PL combination. Cohesive failure in the resin coating material. Magnification: A:500X B: 2000X. (HL: Hybrid Layer, A: Adhesives, R: resin cement, arrows: small blisters in the adhesive layer).

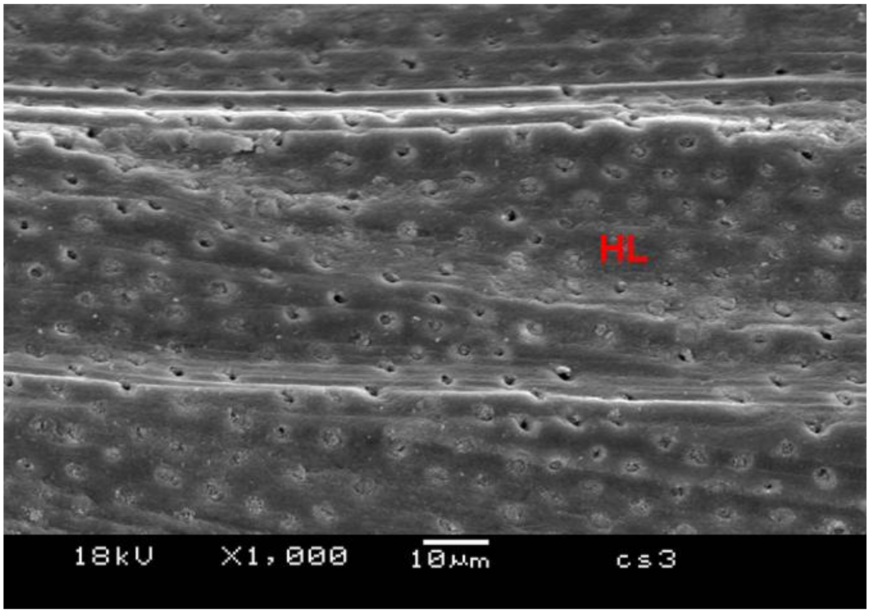

Figure 5. Representative SEM photograph of the debonded specimen that used CS3 without liner. Adhesive failure between Adhesive and resin cement. Magnification: 1000X (HL: Hybrid Layer).

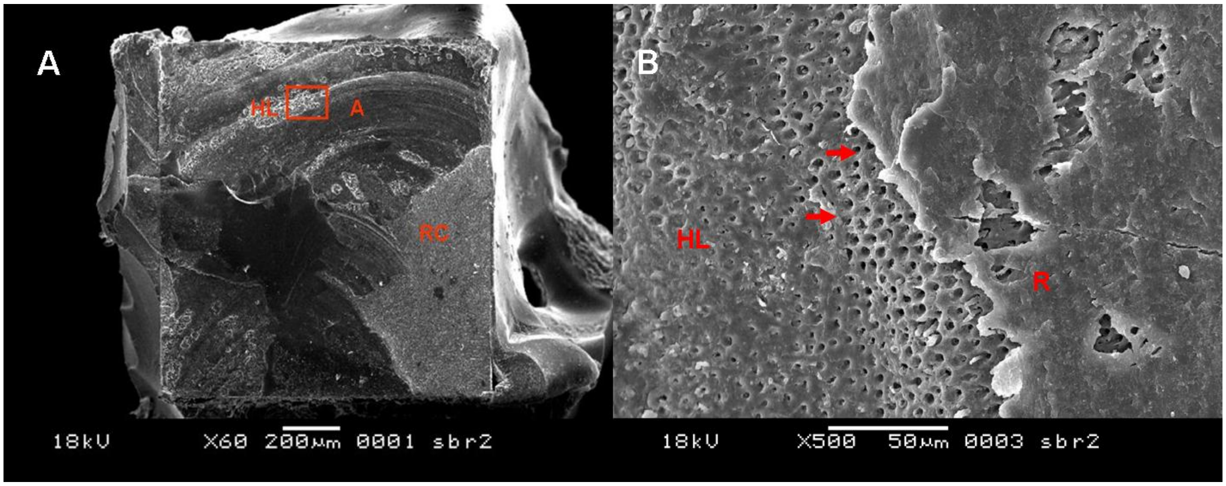

Figure 6. Representative SEM photographs of the debonded specimen that used SB2 without a liner. Mixed failure at the interface between Resin Coating material and Hybrid Layer. Magnification: A: 60X, B: 500X. (HL: Hybrid Layer, A: Adhesives, R: resin cement, arrows: dentine tissue).

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. The use of the thermal and load cycling simulate stresses undertaken by dental restorations helps to better understand the performance of the materials and the mechanisms of degradation. However, as previously shown, the amount and frequency of cycles, the type of restorative material and the cavity configuration might influence the results.[12, 13]

A single application of an adhesive to the cavity prepare has been shown to protect the exposed dentin and prevent hypersensitivity.[2] However, studies have shown that an additional application of a hydrophobic monomer or a low viscosity resin over the adhesive improved the bond strength of the restoration.[14, 15]

Lower bond strength results in all the groups were observed in comparison with previous studies [16-18]. This might be explained by the fact that bond strength studies are usually conducted over a flat tooth surface, where presumably the C-fator (0.2) [19] - has low influence on the results, different from a clinical situation (overestimating the bond strengths that can be achieved in complex cavities prepared and restored under clinically relevant conditions).[20] Box-like Class II cavities have four bonded walls and two unbonded surfaces (in this study C-factor: 1.25). Some studies have observed that increasing the C-factor, decrease the bond strength. [17,21,22]

Another factor that influences in the low bond strength values is determined by the thermo-mechanical cycling, which produces strain in the restoration, creating microcracks in the adhesive layer located in the gingival wall of the cavity, or even plastic deformation[23] associated to the stress produced by the polymerization shrinkage of the resin cement. Additionally, difficulties in obtaining the beams for the microtensile test were observed, since the bond area was limited. Besides, the stress generated by the cutting procedure resulted in losses of beams in almost all the groups. Groups where used only adhesive system without liner, presented up to 35% of premature failures.

Selection of the adhesive system is very important for the success of this technique, in this study the combination of CSEB+PL obtained high bond strength values according with previous studies have shown the efficacy of this combination.[9] The two-step self etch adhesive Clearfil SE Bond contains an acidic self etch primer such as MDP that solubilize the smear layer and demineralize the underlying dentin, resulting in mild surface etching, obtaining good results in several studies. Moreover, the uncured resin in the oxygen inhibited layer, will polymerize with the diffusion of free radicals from the low viscosity resin and this layer protect the adhesive system.

Another group that presented higher bond strength values was the CS3+B, and that was not expected because to the highly hydrophilic characteristics of the adhesive (composed by hydrophilic monomer -HEMA and water). Nevertheless, the coverage of the hydrophilic adhesive with a coat of hydrophobic monomer acted as a physical barrier to the percolation of water through the adhesive layer might have increased the degree of conversion and reduced the hydrophilic characteristic of the adhesive.[24] In relation to the fracture mode, in both groups CSEB+PL and CS3+B, more than half of the fractured specimens showed fracture at the interface between the RCT and the resin cement, showed the efficacy of the RCT 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)[25,26] and thus, creating a thicker sealing film, functioning as a better stress breaker than the monomer hydrophobic. A possible explanation for these results is based in the different composition of the materials. The CS3 adhesive is highly hydrophilic, presents water and HEMA in its constitution, compromising the polymerization of the adhesive. The flowable composite applied over the adhesive, present hydrophobic monomers that do not react completely with the free monomers present in the surface of the adhesive, resulting in a structurally porous salt layer. The chemical incompatibility between the materials was reflected in the type of failure mode (cohesive failure in the resin coating material). Observations in high magnification revealed small blisters in some areas of the adhesive layer when the flowable composite was used as liner. On the other hand, they were not observed when the hydrophobic bond of the two-step self-etching adhesive was used as liner. This might be explained by the presence of hydrophilic monomers, such HEMA, in the bond of Clearfil SE Bond, creating better compatibility, [27,28] confirmed by the absence of premature failures different than the CS3+PL that showed great number of premature failures (7/20).

The lowest values were obtained with CS3 group, this adhesive has been shown to present better performance when compared to other one-step self-etching adhesives.[29]However, literature has shown that the hybrid layer formed by one-step self-etching adhesives presents microscopic channels through which there is water flow, compromising the polymerization of the adhesive, reducing the bond strength and accelerating the degradation of the tooth/restoration interface.[24,30,31] Besides, the adhesive layer is extremely thin due to the volatilization of the solvent. Therefore, its polymerization might be hindered by the contact with the oxygen.[32] The manufacturer has reported that this adhesive works based on molecular dispersion, meaning that the hydrophilic and hydrophobic components of the material would remain in a homogeneous state, even after the solvent evaporation. Still, this adhesive can not support the stress by it self and necessarily requires a liner to obtain better results in the RCT. In relation to the fracture mode was observed failure between the RCT and resin cement and in some specimens exposed dentin tissue (Fig 5).

The total etching groups, Sb2; Sb2+B and Sb2+FL showed similar values. The Single Bond 2 adhesive is a combination of a hydrophilic primer, hydrophobic resin and an organic solvent like ethanol and water, consequently, the incomplete volatilization of the solvent also compromises the polymerization of the adhesive [33] and has a limited capacity of infiltration in the collagen network because the demineralization caused by the phosphoric acid is larger than the infiltration by the adhesive, becoming susceptible to degradation by metalloproteinases. However, not showed difference with or without liner, and this results are similar with the study of Nikaido et al 2003[34]. The reason could be in the viscosity of the adhesive, this material has nanofillers that can be found within the hybrid layer,[35] then, this nanofillers will improved the mechanical properties of the adhesive, supporting the thermo-mechanical stress by it self and not requiring a liner.

Although, the failure mode was different between them. For the SB2 group was observed mixed failure between the RCT and the hybrid layer and in some specimens was observed dentine tissue, in the other two groups SB2 + FL and SB2 + B, the mode failure was principally mixed failure between the RCT and the resin cement, suggesting a better behavior if a liner is used.

Better bond strength results were observed in the groups CSEB+PL and CS3+B. Add to that, 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 exposing dentin tissue. The tested hypothesis was rejected, since differences in bond strength and fracture modes were observed between the different combinations for resin coating.

CONCLUSIONS

1. The indirect restorations that used the combinations for resin coating: self-etch 2step / low viscosity resin and self-etch 1 step / monomer hydrophobic showed the highest values of bond strength.
2. SEM analysis of the fracture modes showed that, the groups that used a liner, revealed an efficient performance after the thermal and load cycling, since dentin tissue was not observed.

Acknowledgements

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CAPÍTULO 3

Nanoleakage in indirect composite restorations using different combinations of resin coating technique

ABSTRACT

Purpose: The aim of this study was to evaluate the nanoleakage patterns in indirect composite restorations bonding to dentin using different combinations of Resin-Coating (RC), after thermal and load cycling.

Materials and Methods: Twenty five extracted third molars were used in the study; two Class II cavities were prepared in each tooth (mesial and distal surface). The 50 cavities were distributed in 5 groups according to the RC materials combinations: G1:Etch-rise 2steps/Hydrophobic monomer (SB2/B);G2::Etch-rise 2steps/Flow composite-resin(SB2Fl), G3:Self-etch 1step(CS3), G4:Self-etch1step/Flow composite-resin(CS3/Pl), G5:Self-etch 2step/ Flow composite-resin (CSEB/Pl). The cavities were molded with a vinyl polysiloxane impression material and the molds were poured with stone plaster. The fillings were confectioned using the Sinfony composite system (3M/ESPE) and cemented with resin cement (Rely X ARC). After 24 hours, the teeth were submitted to thermocycling (2000C/5-55°C) and load cycling (250,000C/30N). Past 24 hours, the restored teeth were sectioned in to serial slabs and immersed in 50% ammoniacal silver nitrate for 24h, exposed to photo-developing solution for 8h, carbon coated and observed in SEM using backscattered electron mode.

Results: Diverse nanoleakage patterns were observed for the different RC combinations. Silver accumulation were observed in the entire thickness of the HL in the SB2B and SBFl groups, while in the “all in one” self-etch groups (CS3 and CS3Pl) silver accumulations similar to “water trees” within the adhesive layer were observed . In the CSEBPl group, less nanolekage than other groups was observed and this was limited to the hybrid layer.

Conclusion: The group CSEB+PL presented a superior behavior and revealed less nanoleakage compared to the other groups.

CLINICAL SIGNIFICANCE

The current RC combinations used in this study do not achieve perfect sealing at the interface of the restoration and may influence in the durability of the restorations. However, the combination of a 2 step self-etch adhesive and flow resin, was able to prevent nanoleakage with more efficiency than other RC combinations.

INTRODUCTION

One of the best alternatives for restoring extensive cavities is the indirect composite restoration; however, this type of restoration demands a more invasive cavity preparation and may lead to a post-operative sensitivity. [1,5]

The resin-coating technique (RCT) has been proposed as an attempting to avoid this hypersensitivity.[26] The technique consists in protect the exposed dentin with an adhesive system followed by the application of a liner (hydrophobic monomer or low viscosity composite resin) after the cavity preparation and prior to taking the final impression.[21]The advantages of the RCT are to minimize the pulp irritation and hypersensibility, caused by the mechanical and thermal stimuli and bacterial infiltration, which can occur during impression taking, temporary-restoration confection and final cementation. The effectiveness of this technique was reported in several studies, evaluating bond strength and marginal adaptation.[4,9,20,27] Still, little is known about the behavior of this technique in nanoleakage tests. The term “nanoleakage” has been used to describe microporous zones as the pathway for degradation of a bonded interface, either in completely cured adhesive resin, within the hybrid layer, and/or demineralized dentin, that allow tracer penetration to occur in the absence of interfacial gaps.[25]

Also, limited information about nanolekage in RCT exits. Most studies were performed on flat surfaces, not considering factors like cavity configuration, thermal variations and masticatory forces, which can influence in the long-term durability of the restorations.[2,6]Due to the constant and fast evolution of the dental materials, not exists a protocol about the proper combination between adhesive system and a liner, to be used with this technique.[19]

Therefore, the aim of this study was to evaluate the nanoleakage patterns in indirect composite restorations bonding to dentin using different combinations of resin-coating technique, after thermal and load cycling.

MATERIALS AND METHODS

Sample preparation

Twenty five extracted third molars were used in the study, under the approval of the Committee of Ethics in Research from the Piracicaba School of Dentistry – University of Campinas (113/2005). The teeth were embedded in epoxy resin leaving 2 mm of root exposed. The periodontal ligament was simulated applying a layer of polyether (Impregum, 3M ESPE AG, Seefeld, Germany) over the roots.[3]Two Class II cavities were prepared using diamond burs (#4137 KG Sorensen Barueri SP, Brasil). The cavities had the following dimensions: 4 mm of bucco-lingual width and 3 mm of proximal-axial width. The gingival margin of the cavity was located 1 mm below the cement-enamel junction. The dimensions and characteristics of the cavities are detailed in Figures 1A and 1B. The cavities were randomly distributed in 5 groups. The materials used in each group, composition and the application techniques are described in Table I.

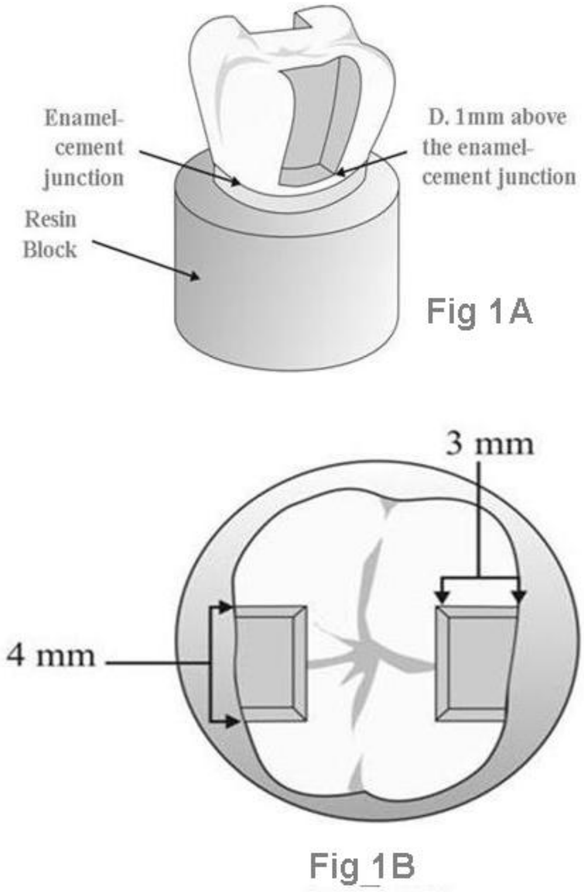

Figure 1 - a. Characteristics and measurements of class II cavity. b. Occlusal view of the cavity preparation.

Table 1. Materials, brand (Lot#), composition, application technique, and manufactures materials used in this study (Bis-GMA:bisphenol-A diglycidil ether dimethacrylate, HEMA: 2-hydroxyethyl metacrylate, MDP: 10 metacryloyloxydecyl dihidrogen phosphate, TEGDMA: triethylene glycol dimetacrylate

Materials	Brand Lot #	Composition	Application
Group SB2B **Etch and Rise 2 step + Hydrophobic Monomer**.	Single Bond 2 Etchant#5EN Adhesive #5EP Bond SBMP #(5PH) (3M/ESPE)	- Etchant: 35%H_3PO_4, gel sílica - Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate -Bond SBMP: Bis-GMA, HEMA, photoinitiator	a (15s), b (15s), c , d, e, d, e, l (10s) k, l (10s)
Group SB2Fl **Etch and Rise 2 step + Flow composite resin**	Single Bond 2 Etchant#5EN Adhesive #5EP Filtek flow #6031A2 (3M/ESPE)	-Etchant: 35%H_3PO_4, gel sílica - Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, Bisphenol A glyceralote, polyalquenoic acid copolymer, dimetacrylate FiltekFlow: Bis-GMA, TEGDMA, Zirconia, Sílica, camphorquinone, nanofillller	a (15s), b (15s), c , d, e, d, e, l (10s) Filtek Flow: i, l (20s)
Group CS3 **Self etch 1 step**	Clearfil S3 # 00001A Kuraray	CS3: MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, photoinitiator, ethanol.	h (20s), e (5s), l (10s)
Group CS3PL **Self etch 1 step + Flow composite resin**	Clearfil S3 # 00001A Protect Liner F # 0046 Kuraray	CS3: MDP, Bis-GMA, HEMA, dimetacrilatos, fotoiniciador ProtectLiner:Bis-GMA,TEGDMA, fluoride methil methacrylate, camphorquinone, silanized colloidal silica.	h (20s), e (5s), l (10s) Protect Liner: l (20s)
Group CSEBPL **Self etch 2 step + Flow composite resin.**	Clearfil SE Bond Primer #00727A Bond #01044 Protect Liner F # 0046 Kuraray	-CSEB Primer: MDP, HEMA, water, ethanol, dymetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine. -CSEB Bond: MDP, Bis-GMA, HEMA, dimetacrylate hidrophilic, camphorquinone, N,N-diethanol p-toluduine ProtectLiner F:BISGMA,TEGDMA (42%wt silanized colloidal sílica).	f (20s) , e , g, e (5s), l(10s) Protect Liner: l (20s)

Application technique: a: acid technique; b: rinse surface; c: dry with cotton-pellet; d: apply one layer total etch one step adhesive; e: gently air dry; f: apply primer two step self etch adhesive; g: apply bond two step self etch adhesive; h: apply one layer self etch one step adhesive; i: apply one layer resin flow; j: apply primer three step etch and rise adhesive, K: apply bond three step etch and rise adhesive l: light cure

Following the application of the different RCTs, the cavities were molded with vinyl polysiloxane impression material (Aquasil, Dentsply DeTrey, Konstanz, Germany), and the molds obtained were poured with die gypsum type IV, Velmix (Kerr Dental Laboratory Products, Orange CA, USA). The restorations were confectioned using the Sinfony System (3M ESPE AG, Seefeld, Germany).

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 in a single layer and light-cured with a quartz-tungsten-halogen light-curing unit (XL2500, 3M ESPE, St. Paul MN, USA) for 10s. The internal surface of the restorations were sandblasted with 50µm Al_2O_3 powder at 2-bar pressure and treated with 35% phosphoric acid (3M ESPE AG, Seefeld, Germany) for 1 min. Following, a silane layer (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 and light-cured for 10s. The resin 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 cement was removed and the restoration was light-cured for 40s from each surface of the tooth. Finishing was performed with fine and extra-fine grit diamond burs (2135F and 2135FF, KG Sorensen, Barueri SP, Brasil) and polished with 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 5^o to 55^oC, 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 a equipment (MSCT-3, Marcelo Nucci ME Instrument, São Carlos SP, Brazil) that consists of five stainless steel pistons with cylindrical tips of 6 mm of diameter and rounded extremities. The equipment applied an intermittent axial force of 50N at a frequency of 2 Hz, totalizing 250,000 cycles. The test was performed under water at 37 °C.

Nanoleakage evaluation

After thermal and load cycling, the teeth were sectioned into 1mm x 1mm thick slices using a low-speed diamond saw (Isomet Buehler Ltd. IL, USA) and the beams obtained were stored in distilled water at 37ºC for 24h. A total of 20 beams were obtained for each group. Following, all the beams were coated with two layers of acid resistant varnish (Revlon nail enamel, SP, Brasil), except for a 1mm width around the adhesive layer and immersed in 50% ammoniacal silver nitrate (pH=9.5) solution for 24h.[28] Specimens were then thoroughly rinsed in distilled water and immersed in a photo-developer solution (Kodak Rochester, NY, USA) for 8 hours under a fluorescent light. After that, embedded in epoxy resin (Buehler Ltd., Lake Bluff, IL, USA) and polished with silicon carbide papers of ascending grits # 600, 1200, 2000 and diamond pastes 1, 0.3, 0.05 *u*m (Arotec Ind. e Co. Granja Viana, SP, Brasil). Specimens were then ultrasonically cleaned after the use of each polishing paste.

All the specimens were dehydrated in increasing alcohol concentration (25, 50, 75, 100%) and immersed in HMDS (Hexamethyldisilazane) for 10 min.[22] Then mounted on aluminum stubs, an carbon coated (MED 010, Balzers Union, Balzers, Liechstenstein) and examined in a Scanning Electron Microscopy (JEOL-5600 LV, Japan), using backscattered electron mode.

RESULTS

All specimens in the five groups showed nanoleakage. Representative SEM backscattered electron images are represented in Figures 2-6. In Fig 2, SB2B group showed the presence of silver in the entire thickness of the hybrid layer and adhesive layer and in some parts of the liner. In Fig 3, the nanoleakage pattern for the group SBFl, showed the presence of silver in the entire hybrid layer with the difference that there was less leakage in the adhesive layer and no leakage in the liner. For the CS3 group, represented in Fig 4, a nanoleakage pattern within the adhesive layer similar to “water trees” can be observed. In Fig 5, the CS3PL group showed a nanoleakage pattern within the adhesive layer similar to “water trees” and in some parts silver deposits in the liner layer. For the group CSEBPL, showed in Fig 6, the presence of silver only in the hybrid layer can be observed

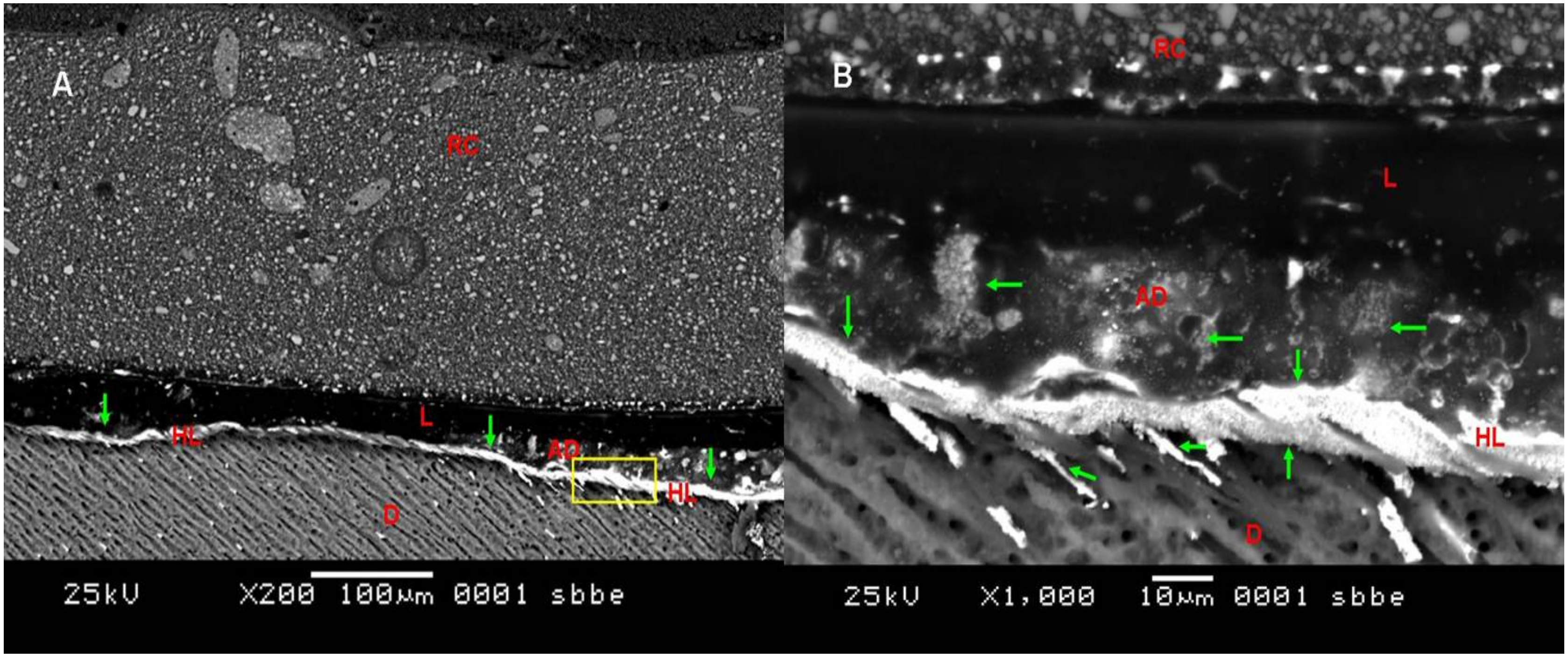

Figure 2. Backscattered images of the group SB2B. In (A) (200X) silver deposition (green narrows) was observed in the entire Hybrid Layer (HL) and adhesive layer. In (B) (1000X) Nanoleakage in the entire HL and adhesive layer (AD) and in parts of the Liner. (RC: Resin Cement, D: Dentin, L: Liner-Hydrofobic monomer).

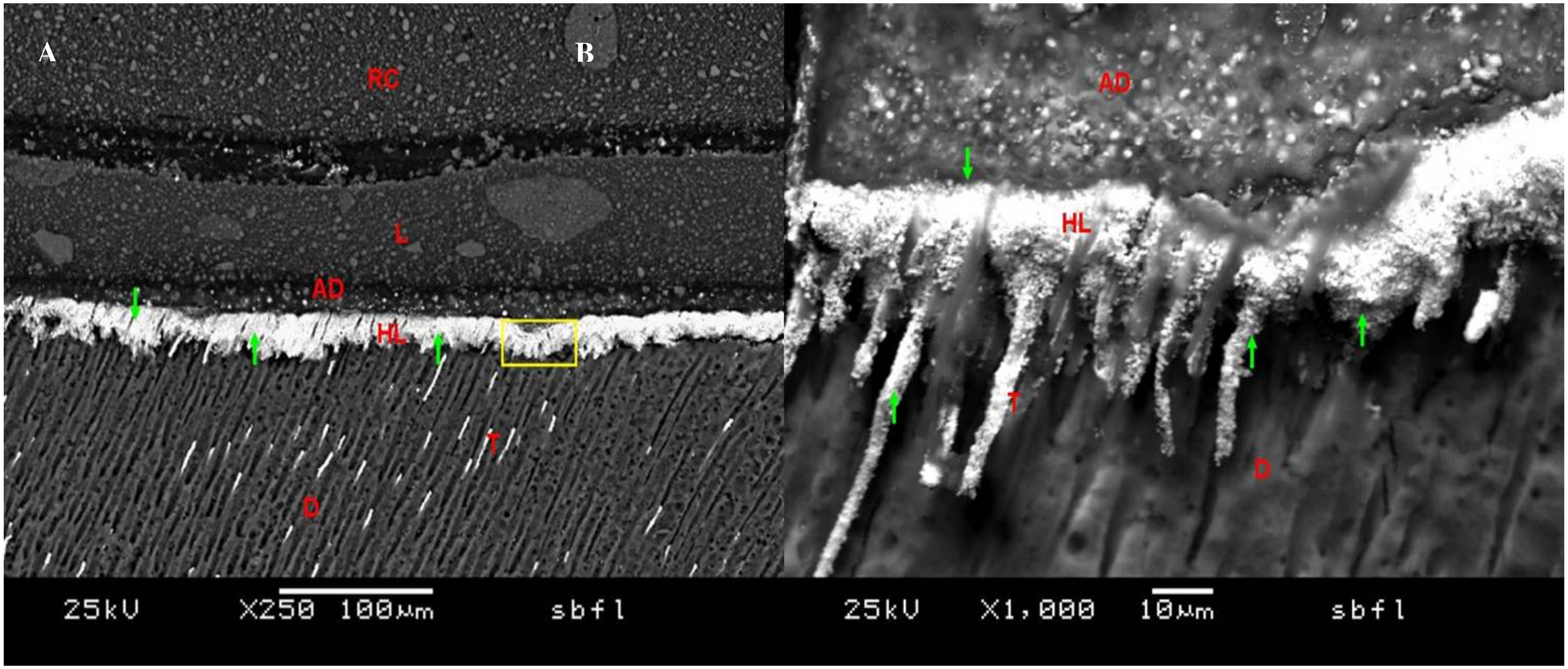

Figure 3. Backscattered images of the group SBFl. In (A) (250X) silver deposition (green narrows) was observed in the entire thickness of the Hybrid Layer (HL) and in parts of the adhesive layer. In (B) (1000X) Nanoleakage in the entire HL and in some parts of the adhesive layer (AD). (RC: Resin Cement, D: Dentin, L: Liner-Low viscosity resin, T: Tag resins).

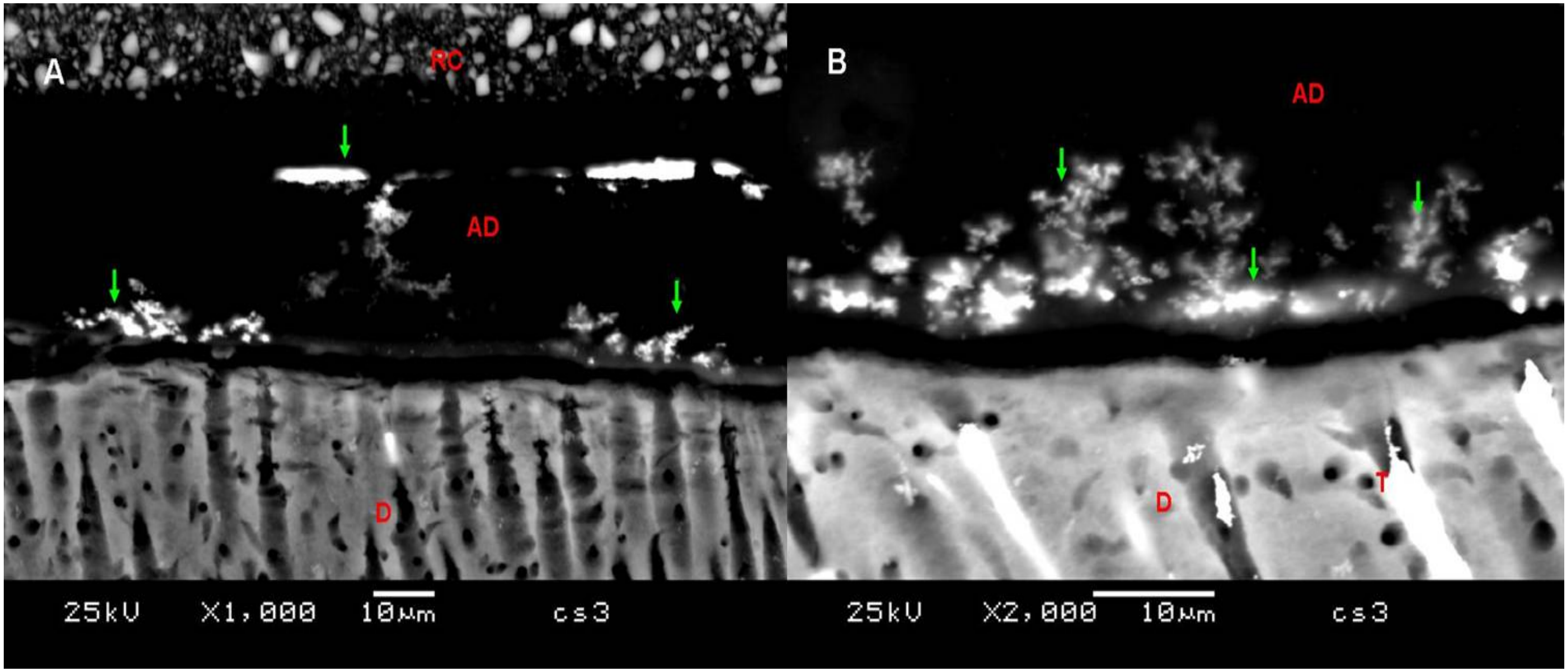

Figure 4. Backscattered images of the group CS3. In (A) (1000X) nanoleakage (green narrows) was observed in the Adhesive Layer “water tree”. (B) (2000X). (RC: Resin Cement, D: Dentin, T: Tag resins).

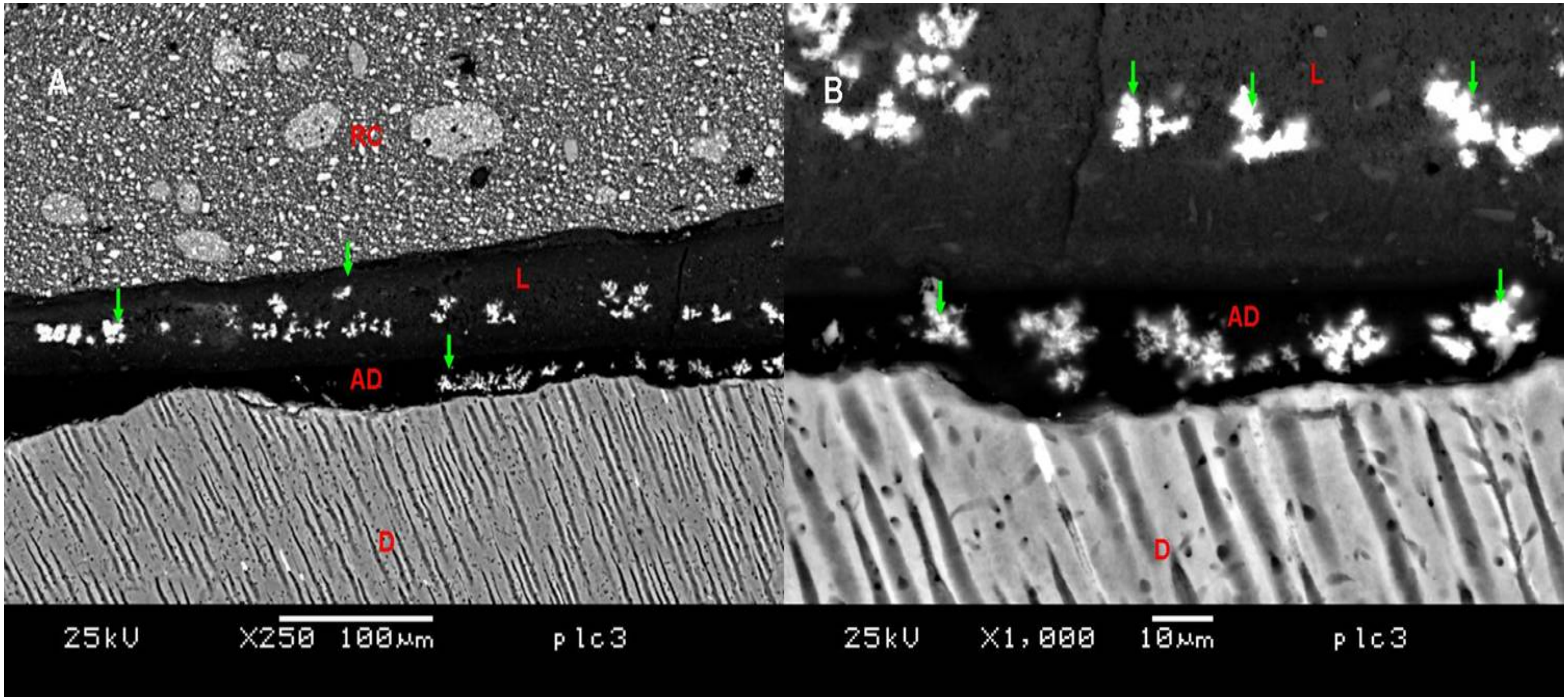

Figure 5. Backscattered images of the group CS3Pl. In (A) (250X) water tree (green narrows) was observed in the Adhesive Layer (AD) and silver depositions in the Liner layer (L). In (B) (2000X) nanoleakage in the adhesive layer and in the liner similar to water tree (AD). (RC: Resin Cement, D: Dentin, L: Liner-Low viscosity resin).

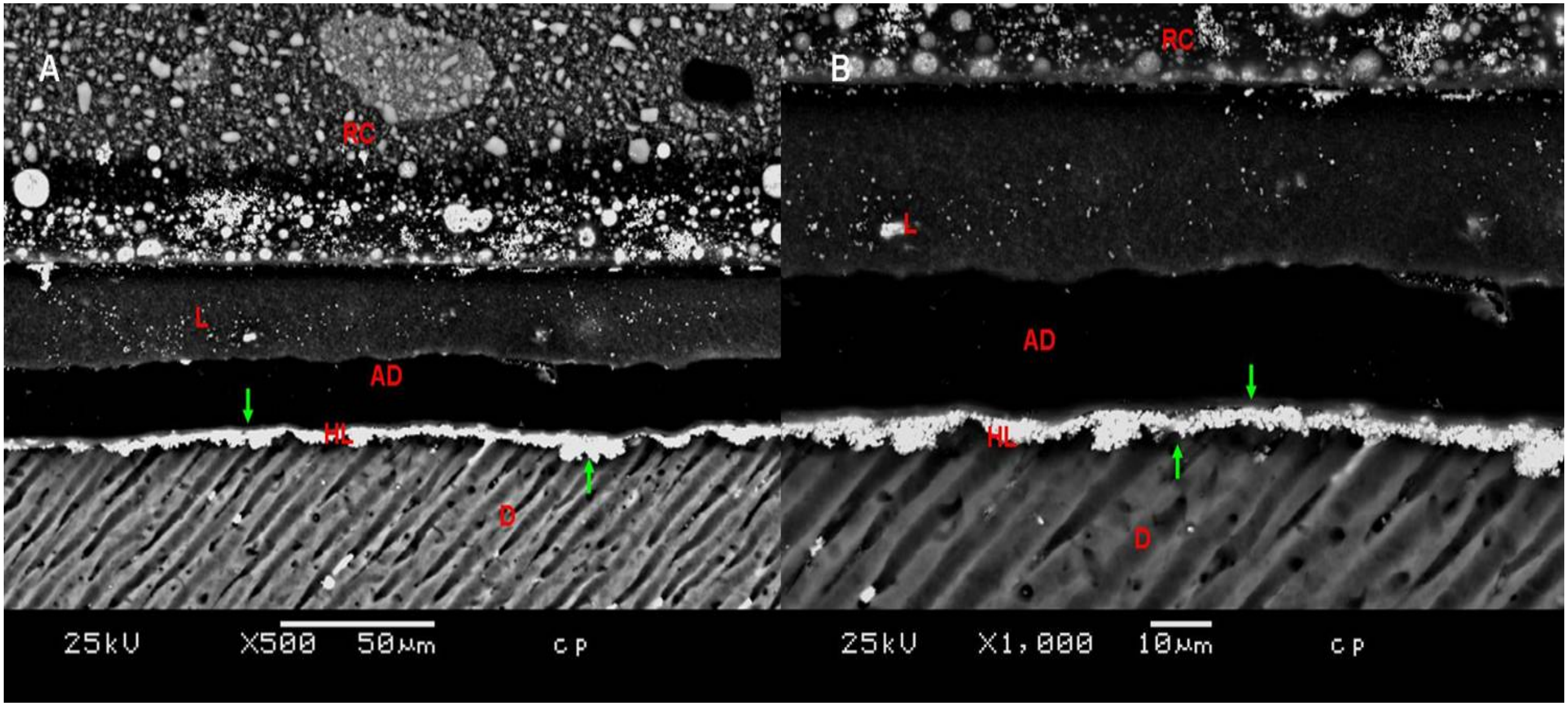

Figure 6. Backscattered images of the group CSEBPL. In (A) (500X) silver deposition (green narrows) was observed in the entire thickness of the Hybrid Layer. In (B) (1000X) some silver depositions is observed in the liner layer. (RC: Resin Cement, D: Dentin, L: Liner-Low viscosity resin).

DISCUSSION

The constant advance of adhesive materials jeoperdize the execution of valid long-term clinical studies, demanding evidence from *in vitro* studies that simulate the oral conditions. The use of thermal and load cycling simulate stresses undertaken by dental restorations and helps to understand the performance of the materials and the mechanisms of degradation.[10,18].

In theory, the fatigue resulting from the masticatory forces generates stresses in the interface tooth/restoration, increasing the damages in this area, [3,13,15,16,22,28,30,31] as well as, the temperature changes induce interfacial stresses due to differences of contraction and expansion in the adhesive interface. These tensions may cause microcracks, which propagates through the interface.[14] Therefore, the thermo-mechanical simulation might provide results closer to the clinical situation.

Several studies that evaluate nanoleakage was performed on a flat surface.[12]In the present study, box-type class II cavities were confectioned, so that the influence of the C-factor was not excluded once the cavity configuration simulates the clinical oral situation.

In etched groups (SB2B and SBFl) the silver penetration in the entire thickness of the hybrid layer is not just the result of adsorption and deposition of silver into the porosities; silver particles can also adheres with collagen fibers indicating that the adhesive system did not completely permeate the demineralized dentin, leaving a hybrid layer with large amounts of porosity which may subsequently allow dentinal or oral fluid to slowly diffuse along the interface and, subsequently, hydrolyze the adhesive resin and collagen.[24,25] However, in group SB2B, more nanoleakage in the hybrid layer was observed, which included the entire thickness of the adhesive layer, different to the SBFl group, where the nanoleakage was limited to the hybrid layer. This can be explained due to the mechanical properties of the material since SBFl used a low viscosity resin as a liner. This material absorbs the stress caused by the thermo-mechanical treatment better than the hydrophobic monomer[7,24]

In "all in one" self-etch groups (CS3 and CS3Pl) a different nanoleakage pattern could be observed. Presumably silver tracer should be absent when no discrepancy exits between the depth of demineralization and the extent of adhesive infiltration. However, silver deposits similar to water tree were observed in both groups and these water channels extend through the adhesive layer, which provides the most direct way for water movement across the polymerized layers.[29] These regions represent areas of sub-optimal conversion within the polymer matrix due to the incompletely removal of solvent.[17,25]On the other hand, since Clearfil S3 adhesive contains HEMA (hydrophilic monomer) and water, it is important to performe strong air drying to evaporate water and solvents. This procedure results in a viscous resin material with entrapped air bubbles remaining on the dentin surface and reduces the thickness of the layer, turning it more susceptible to the polymerization inhibition by oxygen[23]

Additionally, nanoleakage within the liner layer was observed for the CS3Pl group. This can be explained by the composition of this material which presents a lower amount of filler (42%wt) compared to other low viscosity resins (Filtek Flow Z-350: 65%wt). Considering that the filler influences in the mechanical resistance of the material and this, associated to the poorly polymerization of the adhesive, suggests that the liner layer is that support all the stress produced by the thermal-load cycling.

The CSEBPl group, revealed less nanolekage than the others groups and such infiltration was limited to the hybrid layer. This could be explained by the composition of the hybrid layer (resin and collagen fibrils). This adhesive is more stable compared to Clearfil S3 since this system presents primer and bond in separated bottles, while "all in one" adhesives present all the components (primer, bond and solvent) in one bottle, therefore less stable and prompt to phase separation.[11]On the other hand, it has been observed that the combination of the self etch adhesive 2 steps (Clearfil SE Bond) with a low viscosity resin (Protect Liner F) can withstand the thermal-load cycling better, compared to the others groups tested and these results are according to others studies.[19]

CONCLUSIONS

1. All the indirect restorations showed nanoleakage, independent of the material combination used for the resin coating.
2. The combination of self etch 2 steps/low viscosity resin presents a superior behavior showing lower nanoleakage compared to the other groups.

Acknowledgements

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CAPÍTULO 4

Influence of the resin cement in marginal adaptation and bond strength of indirect composite restorations using resin coating.

ABSTRACT

Statement of the problem: Resin Coating Technique (RCT) has been proposed as an attempt to minimize the post-operative sensitivity after cavity preparation for indirect restorations. The effectiveness of this technique was reported by several studies, however, there is a constant advance in adhesive luting cements and these can have a negative or positive influence on this technique. It is still unclear which luting cement is more appropriate to be used with this technique.

Purpose: The aim of this study was to evaluate the marginal adaptation, microtensile bond strength (u-TBS) and failure mode of indirect composite restorations cemented to dentin using different resin cements.

Materials and Methods: Thirty extracted third molars were used in the study; two Class II cavities were prepared in each tooth (mesial and distal surface). After that, was realize the resin coating; for this procedure was used the combination of a self-etch adhesive 2 steps and a low viscosity resin. 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). The cavities were divided in 3 groups for cementation, G1:SE (self-etch resin cement), G2: SA (self-adhesive group) G3: CC (Chemical-Cure group). After 24 hours, the teeth were submitted to thermocycling (2000C/5-55°C) and load cycling (250,000C/30N). Following, Caries Detector (Kuraray) was applied to the restoration margins and washed for 30s. Images from the proximal view of the teeth were captured and evaluated using the software Image Tool 3.0 (University of Texas, USA) for the measurement of the gap formation. Past 24 hours, the restored teeth were sectioned in to serial slabs and u-TBS were measured. The data for both tests, were analyzed with ANOVA and Tukey test ($p<0.05$). In addition failure mode pattern was determined by scanning electron microscopy.

Results: Marginal desadaptation (%) not were significant different between the groups: SA (45.55%), SE(38,84%), CC(37,93%). However, bond strength was significant higher in the group SE (22.27MPa) compared with the other groups SA (15.08MPa) CC (16.47MPa). In relation to the failure mode, reveals the effectiveness of the RCT, independent of the resin cement, due to no exposed dentin tissue.

Conclusion: The group SE showed the highest values of bond strength, revealing the good compatibility with the RCT.

CLINICAL IMPLICATIONS

Within the limits of this study, the more appropriate resin cement to luting indirect composite-restorations with Resin Coating, is the Self-Etch resin cement.

INTRODUCTION

The demand of patients for esthetics has resulted in the use of indirect composite restorations, however, this type of restoration demands a more invasive cavity preparation, may lead to a postoperative sensitivity.[1] Attempting to minimize this hypersensitivity the resin coating technique (RCT) has been proposed.[2] This technique consists of 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 final impression.[3] The advantages of the RCT are to minimize pulp irritation and hypersensitivity caused by mechanical and thermal stimuli and bacterial infiltration, which can occur during impression taking, confection of the provisory restoration and final cementation.

The effectiveness of this technique was reported in several studies [4,5], as well as, improves the union between the restoration and dentin tissue when is used a resin cement[6]. Due to the constant advance in adhesive luting cements, like self-adhesive and self-etching cements, the type of resin cement used to luting the restorations can also influence in the behavior and success of the restorations.

Therefore, the aim of this study was to evaluate the marginal adaptation, microtensile bond strength and failure mode pattern of indirect composite restorations with resin-coating, cemented to dentin using different resin cements.

MATERIALS AND METHODS

Sample preparation

Thirty extracted third molars were used in the study, under the approval of the Committee of Ethics in Research from the Piracicaba School of Dentistry – University of Campinas (113/2005). The teeth were embedded in epoxy resin leaving 2 mm of root exposed. The periodontal ligament was simulated applying a layer of polyether (Impregum, 3M ESPE AG, Seefeld, Germany) over the roots.[7] Two Class II cavities were prepared using diamond burs (#4137 KG Sorensen Barueri SP, Brasil). The cavities had the following dimensions: 4 mm of bucco-lingual width and 3 mm of proximal-axial width.

The gingival margin of the cavity was located 1 mm below the cement-enamel junction. The dimensions and characteristics of the cavities are detailed in Figures 1A and 1B. After that, was realized the resin coating; for this procedure was used the combination of a self-etch adhesive 2 steps (Clearfil SE Bond Kuraray Medical Co.) and a low viscosity composite resin (Protect Liner F Kuraray Medical Co.). For this, was applied over the cavity preparation, the primer for 20s, air-dry and following the bond (light-cure 10s). Subsequent, was applied a thin layer of Protect Liner F and light-cure for 20s. Following the application of the RCT, the cavities were molded with vinyl polysiloxane impression material (Aquasil, Dentsply DeTrey, Konstanz, Germany), and the molds obtained were poured with die gypsum type IV, Velmix (Kerr Dental Laboratory Products, Orange CA, USA). The restorations were confectioned using the Sinfony system (3M ESPE AG, Seefeld, Germany).

The cavities were randomly distributed in 3 groups and cemented according to the manufacturer indications. The resin cements used in each group, composition and the application techniques are described in Table 1.

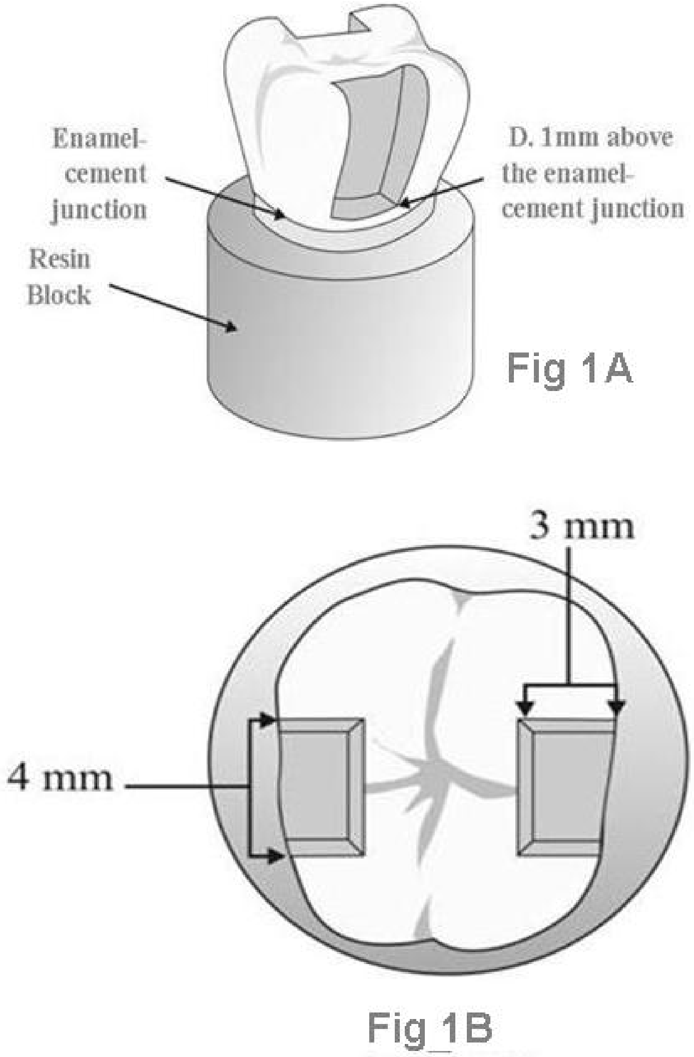

Figure 1 - a. Characteristics and measurements of class II cavity. b. Occlusal view of the cavity preparation.

Table 1. Materials, brand (Lot#), composition, application technique, and manufactures materials used in this study (Bis-GMA:bisphenol-A diglycidil ether dimethacrylate, HEMA: 2-hydroxyethyl metacrylate, MDP: 10 metacryloyloxydecyl dihidrogen phosphate, TEGDMA: triethylene glycol dimethacrylate, 5-NMSA:N-methacryloyl 5-aminosalicylic acid, BPDM: Byphenyl dimethacrylate.

Materials	Brand Lot #	Composition	Application
Group SE **Panavia F2.0 Self Etch cement**	00085B 00205B 51187 51187	EDPrimer 2.0 Primer A: HEMA, MDP, 5-NMSA water, accelerator. Primer B: 5-NMSA, accelerator, water, sodium benzene sulphinate Base paste: hydrophobic aromatic and aliphatic dimethacrylate sodium aromatic sulphinate, N,N-diethanol-p-toluidine,silanized barium glass Catalyst paste: MDP, hydrophobic aromatic and aliphatic photoinitiator,dibenzol peroxide dimetacrylate,hydrophilic dimetacrylate	c,f,d,f,e, f,g,h,i j 20sec each face.
Group SA **RelyX U100 Self Adhesive cement**	308804	Phosphoric acid modified methacrylate monomers, calcium hydroxide,silica,initiators	c, f, d,g,h,i,k for 1min,j20sec each face.
Group CC **One step + C&B Cement Chemical Cure**	0800005537 0800005538 0700004236	Etchant: 32%H_3PO_4 Adhesive:BisGMA,BPDM,HEMA,Photoinitiators,acetona Base paste: Bis-GMA, BIS-EMA, silica Catalyst paste: Bis-GMA, TEGDMA, silica	a, b, c, d, e, f, h, i, k for 7min

Application technique: a: acid technique; b: rinse surface; c:sandblasting d: silane treatment at the restoration; e:apply primer; f: dry with air flow; g:mix the pastes h: apply the mixture; i: removing excess cement; j: light curing under digital pressure; k: digital pressure

Finishing was performed with fine and extra-fine grit diamond burs (2135F and 2135FF, KG Sorensen, Barueri SP, Brasil) and polished with 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 5^{o} to 55^{o}C, 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 a equipment (MSCT-3, Marcelo Nucci ME Instrument, São Carlos SP, Brazil) that consists of five stainless steel pistons with cylindrical tips of 6 mm of diameter and rounded extremities. The equipment applied an intermittent axial force of 50N at a frequency of 2 Hz, totalizing 250,000 cycles. The test was performed under water.

Evaluation of the marginal adaptation

In order to evaluate the marginal adaptation of the restorations, a solution consisting of propylene-glycol and acid red 52 Caries Detector (Kuraray Medical Inc, Okayama, Japan) was applied on the proximal surface of each restoration during 10s. The solution was washed and dried with absorbent paper. Images of the proximal surface of the restorations were captured using a digital camera SLR Canon (Rebel XT, Lake Success, NY, USA) equipped with a Lens 105mm f2.8 Sigma Corporation (EXDG Macro, Kanagawa, Japan) and transferred to a computer equipped with Image Tool 3.0 software (Periodontology Departament, University Texas, Health Science Center, San Antonio, TX, USA). The software was used to measure the gap formed all over the tooth-restoration margin. The gap measurement was converted in percentage, based on the ratio of the dye penetration length and the total length of the restoration margins. The values were submitted to statistical analysis (ANOVA and Tukey's test $p<0.05$).

Microtensile bond strength

After the marginal adaptation evaluation, the teeth were retired of the epoxy resin and was removed the enamel tissue present on the proximal areas 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 in to serial slabs approximately 0.9mm thick using the same slow-speed water-cooled diamond saw. Each slab was then sectioned by the same method 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 device[9] and tested to failure under tension in a universal testing machine Instron (modelo 4411, Corona, Ca, USA) with a 500-N load cell travelling at a crosshead speed of 0.5mm/min. Means and standard deviation were calculed 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 then 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 Electrical Microscopy (JEOL-5600 LV, Japan) operated at 18 kV. Fracture modes were classified according to Table 2

Table 2 – 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

Evaluation of the marginal adaptation

All the groups presented gap formation. Table 3 summarizes the results of marginal desadaptation, presented similar values between groups.

Table 3 - Mean values of gap formation (%) of indirect restorations

Resin Cement	Mean values	Standard deviation
SE	38.84 A	± 17.57
SA	45.55 A	± 26.23
CC	37.93 A	± 20.81

Mean values followed by different letters showed significant difference (Tukey5%).

Microtensile bond strength

Beams with premature failure during sectioning were recorded in the study with the value of “Zero”. Statistically significant differences were observed between groups ($p<0.05$) as described in Table 4. Bond strength was significant higher in the group SE (22,27MPa), groups SA (15,08MPa) and CC (16,47MPa) were not significantly different from each other ($p<0.05$) presented similar values.

Fracture mode analysis

Fracture modes are summarized in the Figure 2, representative SEM photographs of the debonded specimens are represented in the Figures 3-5.

Table 4 – Microtensile bond strength (MPa) of indirect restorations using different resin cements

Resin Cement	Mean values Standard deviation
SE	22.27 ^{A} ± 9.51
SA	15.08 ^{B} ± 9.58
CC	16.47 ^{B} ± 11.61

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

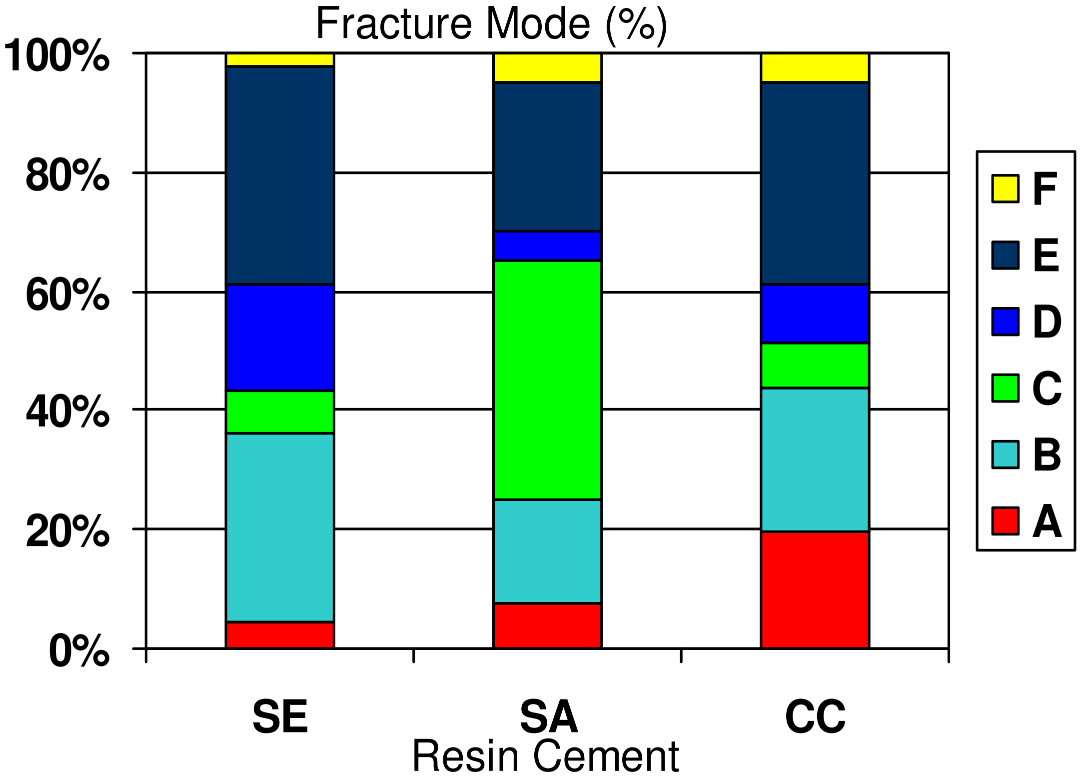

Figure 2. Failure mode after microtensile bond strength test (%).

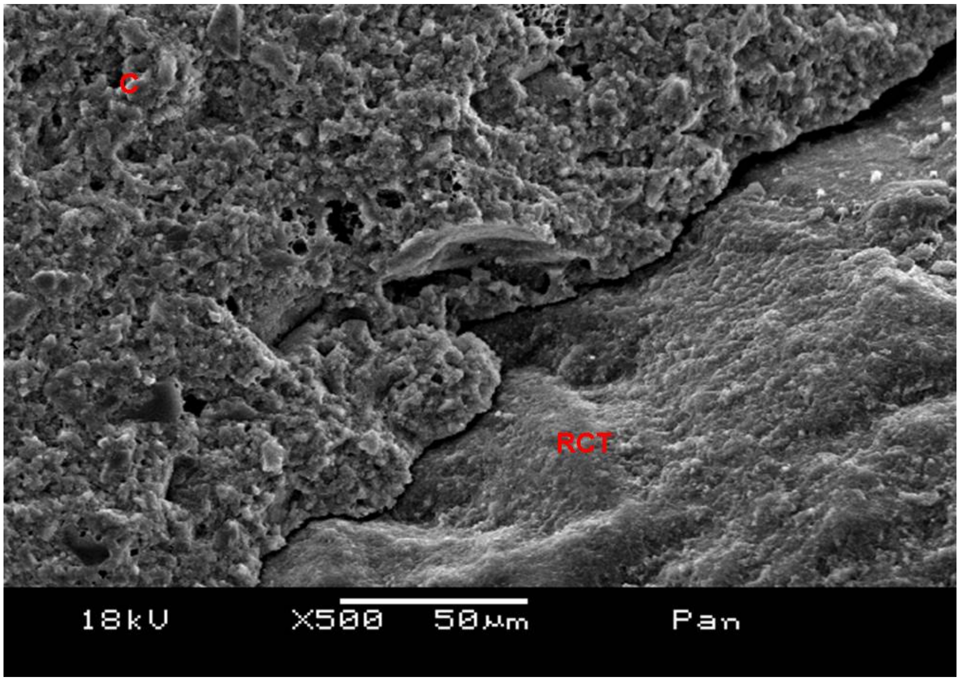

Figure 3. Representative SEM photographs of the debonded specimen that used Self-etch resin cement. Mixed failure at the interface between Resin Coating material and Resin cement. Magnification: 500X. (RCT: Resin coating material, C: Resin cement).

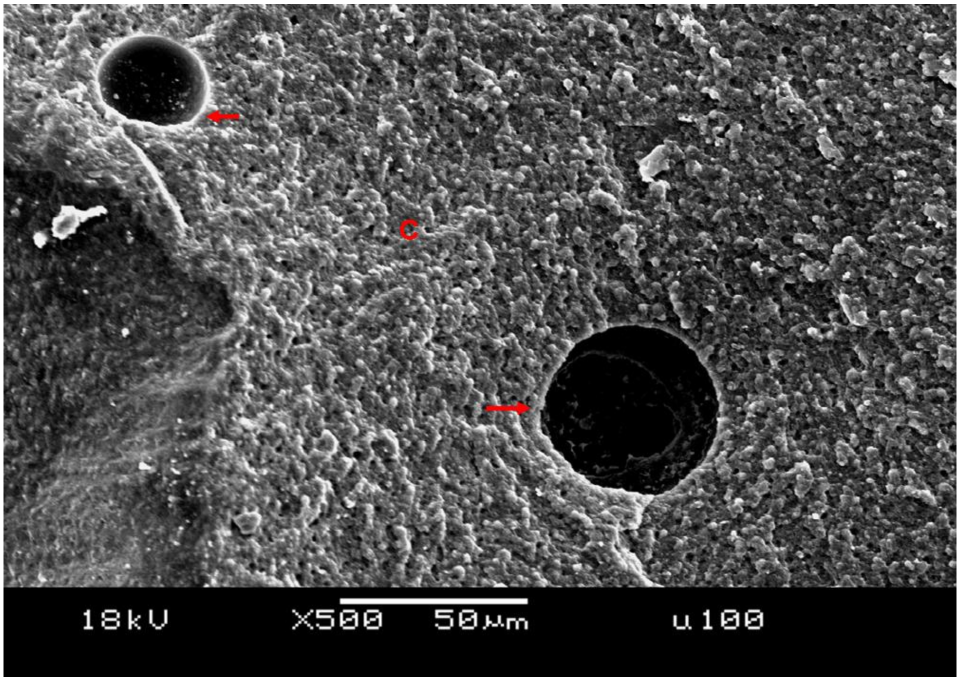

Figure 4. Representative SEM photographs of the debonded specimen that used Self-adhesive resin cement. Cohesive failure at the Resin cement layer. Red narrows showed voids within the resin cement. Magnification: 500X. (RCT: Resin coating material, C: Resin cement).

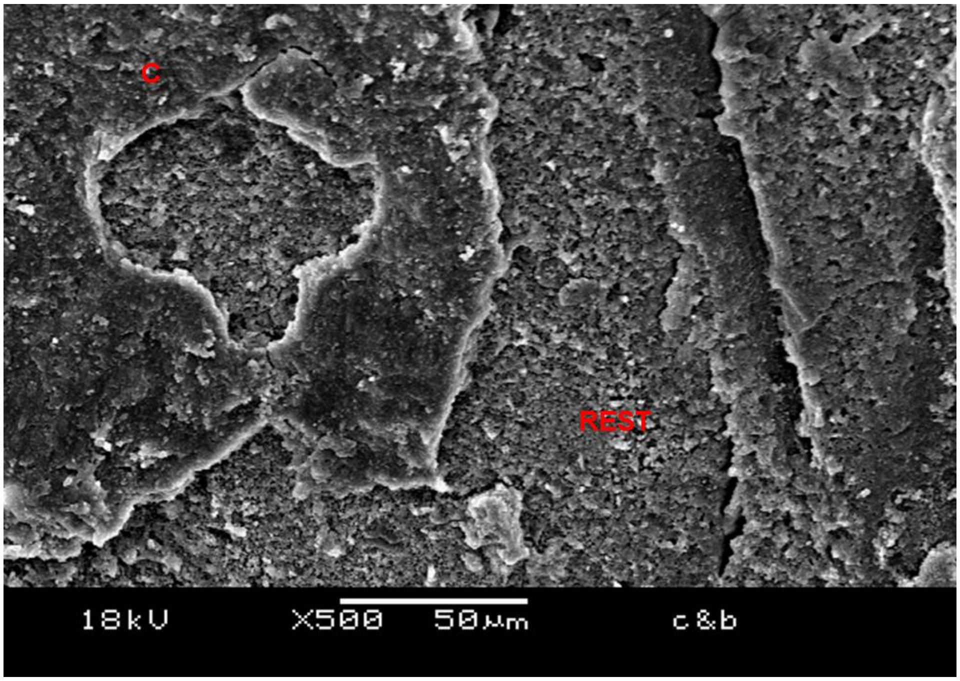

Figure 5. Representative SEM photographs of the debonded specimen that used Chemical-cure resin cement. Mixed failure at the interface between resin cement and restoration. Magnification: 500X. (REST: Restoration, C: Resin cement).

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. The use of thermal and load cycling simulate stresses undertaken by dental restorations in the oral environment and helps to better understand the performance of the materials and the mechanisms of degradation. However, as previously shown, the amount and frequency of cycles, the type of restorative material and the cavity configuration might influence the results.[9,10]

A single application of an adhesive layer to the cavity preparation has been shown to protect the exposed dentin and prevent post-operative sensitivity [1] However, studies have shown that an additional application of a hydrophobic monomer or a low viscosity resin over the adhesive improved the bond strength and marginal adaptation of the restoration.[11, 12]

On the other hand, a material that also influences the success of the restoration is the resin cement used to lute the indirect restorations. For this reason this study investigated which type of cement is more proper to be used with the RCT.

The initial marginal gap formation formed between tooth structure and resin cement is frequently the result of polymerization shrinkage of the resin cement. Therefore a lack of marginal sealing will occur if the adhesion of luting agents to the tooth structure does not compensate for the shrinkage stress of the resin cement. Thus, the use of RCT helps to support this stress and reduce gap formation.

Thermal and load cycling have been frequently used in an attempt to simulate fatigue of restorations in the oral environment. The fatigue resulting from the masticatory forces generates stresses at the tooth/restoration interface, increasing damages in this specific area.[13,14,15,16] Moreover, due to the difference in thermal expansion between the tooth structures and the materials involved will originate microcracks which propagates throughout the adhesive interface and result in fluid percolations through the gaps formed. This, associated to the water sorption of some matrix components, can create deterioration in the material structure. Therefore, all restorations showed marginal desadaptation, but, in different levels.

The self-etching cement (Panavia F) seem to be affected by the water-induced interfacial changes that occurred in relation to the permeability of the acidic ED primer. These interfacial changes were prevented by the use of RCT [6]. The presence of gaps still occurred probably due to the entrapment of voids during paste mixing. Such voids can act as stress raisers during tension or compression, generating crack propagation and, consequently, degradation of the cement interface.[17]

The self-adhesive group (U100) showed gap formation similar than the self-etch group. This can be due to the incorporation of voids during mixing as well as the material's low cohesive resistance,[18] compared with other resin cements. The result will also be the degradation of the interface when the restoration is submitted to thermal and load cycling.

The chemical cure group (C&B) presents an auto-mixing tip that delivers the mixed paste through a syringe type tip directly onto the prepared surface, thus reducing void formation during mixing. However, in this case, gap formation is more related to the poor polymerization of this cement and, consequently, to the physical and mechanical properties of the material. Unpolymerized resin cement absorbs water and this acts like a plasticer. Because of this, weakened areas are created and consequently the resin cement can be degraded, increasing the chances of restoration fractures under masticatory forces.[17,19]

The bond strength results obtained in this study for all groups were lower than observed in previous studies.[20-22] This can be explained by the fact that bond strength studies are usually conducted over a flat tooth surface, and therefore present a very low C-fator (0.2)[23] which does not simulate a clinical situation and can result in an overestimated bond strength value.[24]

Another important factor that can explain lower bond strength results is the incorporation of thermo-mechanical cycling in this investigation. This test can induce microcracks in the adhesive layer located in the gingival wall of the cavity, or even plastic deformations[4] associated to the stress produced by the polymerization shrinkage of the resin cement. Also, it was difficult to obtain beam specimens for the microtensile test since the bonded area evaluated was very reduced.

The self-etch cement group, obtained the highest bond strength values and this is in accordance with others studies. [11,13,25] The use of an additional light-cured resin coating may have provided additional free radicals to enhance the rate and extent of the polymerization of self-etching primers. This probably reduces the permeability of the adhesive layer to water from the substrate and from the environment. The analysis of failure mode in this group revealed a predominance of adhesive and mixed failures between resin coating material and resin cement, confirming the positive results obtained when the RCT was used with this cement.

The self-adhesive group, showed lower values compared with the self-etch group. The main characteristic of self-adhesive cements is that they do not need any previous dentin treatment before cementation procedures, and, consequently the technique is not as critical as others. However, the low bond strength values obtained can probably be explained by the fracture mode which revealed a prevalence of cohesive failures within the resin cement. This suggests that the material has low cohesive resistance and can not resist high stress loads.

The chemical-cure group showed statistically similar bond strength values compared to the self adhesive-group. According to Sanares et al,[26] there is an incompatibility between a etch and rise single bottle system and a chemical resin cement. This occurs because the acidic groups in the uncured layer of simplified adhesive competes with peroxides for the aromatic tertiary amines of the resin, resulting in an acid-base reaction between the adhesive and the resin cement. This reaction minimizes appropriate co-polymerization between both.[27] However, the present study did not find incompatibilities between adhesives and cements when failure mode were evaluated under SEM.

The significant factor that contributed to the failure of the union between adhesive and resin cement is not only the reaction between tertiary amines and acid monomers but also the fact that the adhesive system is applied over a hydrated dentin and this adhesive acts like a permeable layer.[28,29] Since in this study, dentin was covered by the RCT, the permeability factor was excluded and did not influence the bond strength values, isolating the chemical reaction factor such as described by Carvalho et al. [30]In addition, this type of incompatibility is more frequently observed in adhesives with very acidic pHs (1-2) such as those seen for self-etch one step adhesives. In this study, the chemical cure resin cement was used with a etch and rise adhesive (One Step) which has a high pH (4.6), and therefore cannot be associated with chemical incompatibility.[18,31,32]

CONCLUSIONS

1. All the indirect restorations showed marginal desadaptation, independent of the resin cement used for the resin coating.
2. The restorations cemented with the self etch resin cement showed the highest bond strength values.

3. SEM analysis of the fracture modes revealed an efficient performance of the RCT, independent of the resin cement used, since few specimens revealed exposed dentin after fracture.

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CONSIDERAÇÕES GERAIS

Dentre as diversas pesquisas realizadas na Odontologia Restauradora, as pesquisas clínicas são as mais valorizadas. No entanto, devido à constante e rápida evolução, tanto dos materiais como das técnicas restauradoras, é muito difícil realizar uma avaliação clínica em pacientes a longo prazo. Por este motivo, neste estudo *in vitro*, foram simuladas algumas das condições de degradação que aconteceriam *in vivo* na cavidade bucal.

Neste sentido, para todos os estudos foram realizadas cavidades classe II (considerando a influência do fator cavitário) e ensaios de ciclagem térmica e mecânica, na tentativa de simular a fadiga das restaurações em meio úmido e sob constante tensão, já que a maioria dos estudos é realizada em superfície plana e avaliam tanto a adaptação marginal quanto a resistência de união, 24h depois de realizada a restauração (Bouillaguet *et al*., 2001, Nikaido *et al.*, 2002, Shirai *et al*. 2005).

Em relação às faixas de temperatura utilizadas nos banhos de água para ciclagem térmica, existem diversas (0-36ºC, 40-100ºC, 15-45ºC, Peterson *et al*. 1966) 4-60ºC (Kidd *et al*. 1978). Segundo Peterson (1966), sob condições normais, na ingestão de bebidas as temperaturas de 15-45ºC são mais próximas da realidade clinica, comparadas com as outras faixas. No entanto, considerando que a comida refrigerada é mantida a 4ºC, e seguindo as normas ISO, onde recomendam que no caso de ciclagem térmica de materiais restauradores o ideal seria utilizar a faixa de 5-55ºC, se optou por seguir esta faixa de temperatura. Entretanto, alguns estudos reportaram que o teste de ciclagem térmica não influenciaria na resistência de união (Yoshida et al. 1999). Da mesma maneira, o teste de ciclagem mecânica até 100.000 ciclos também não influenciaria na resistência de união de restaurações (Nikaido *et al*. 2002) Não obstante, quando ambos testes são realizados pode-se conseguir uma degradação significativa da interface (Bedran de Castro *et al.* 2004). Por este motivo foram realizados ambos testes.

No experimento do Capitulo 1, pode ser observado que é preciso da utilização de uma camada de *liner* para obter menores valores de formação de fenda, embora nenhuma combinação da Técnica de Selamento Dentinário demonstrou evitar a desadaptação marginal das restaurações, o que leva a pensar que, clinicamente, apesar da evolução dos materiais, a presença de fenda é o primeiro sinal de falha das restaurações adesivas e por conseqüência estas tem um tempo de vida limitado.

Já no Capítulo 2, algumas mudanças aconteceram, pois em um primeiro momento se planejava avaliar a resistência de união das restaurações indiretas com margem em esmalte e em dentina. No entanto, a área da restauração era muito limitada, obtendo-se de 2 a 3 palitos por restauração. O ideal seria não ter perda de palitos; no entanto, não foi o que ocorreu devido à fadiga que foram submetidas, havendo perda de palitos durante o processo de corte e, como conseqüência, a obtenção de poucas amostras. Frente a esse fato, determinou-se avaliar somente as restaurações indiretas com margem em esmalte, já que com margem em dentina, quase todas as amostras falharam durante o processo do corte.

Referente aos resultados do Capítulo 2, observou-se que a melhor combinação para a "Técnica de Selamento Dentinário", foi a utilização do adesivo autocondicionante de 2 passos com a resina composta de baixa viscosidade. Igualmente, foi confirmado que seria insuficiente proteger o preparo cavitário somente com o sistema adesivo sem a aplicação de um "liner" corroborado com os resultados do Capítulo1. Isto ficou demonstrado nos maiores valores de desadaptação, assim como, nos menores valores de resistência de união encontrados, expondo tecido dentinário, como pode ser observado pelas microfotografias em MEV.

Clinicamente, poderia especular que no caso de uma fratura da restauração indireta o tecido dentinário ficaria protegido pela aplicação da "Resin-Coating". Assim, de acordo com os dados desse estudo, a melhor combinação encontrada para a técnica do "Resin Coating" foi o uso de sistema adesivo autocondicionante de 2 passos com resina composta de baixa viscosidade.

Referente à nanoinfiltração (Capítulo 3) houve inicialmente uma dúvida referente ao padrão de infiltração por nitrato de prata, já que durante o procedimento para obtenção das amostras, estas passam por uma série de lixas e feltros para acabamento e polimento, antes de ir para a M.E.V. Assim, partículas poderiam ficar na superfície da amostra e poderiam originar uma aparente infiltração do nitrato de prata. No entanto, está demonstrado na Literatura que não existe diferença entre o padrão de nanoinfiltração de amostras que tenham recebido polimento ou que tenham sido fraturadas, demonstrando que o processo de acabamento e polimento não influencia na visualização da nanoinfiltração em M.E.V. (Li *et al.* 2002).

Pode-se confirmar mais uma vez a eficácia da combinação de sistema adesivo autocondicionante de 2 passos com resina composta de baixa viscosidade, obtendo menor grau de nanoinfiltração. Da mesma maneira, pode ser observado que as combinações da TSD que utilizaram condicionamento ácido, apresentaram altos índices de infiltração e isto pode estar relacionado à discrepância entre grau de desmineralização pelo acido fosfórico e o grau de penetração do adesivo, expondo fibrilas de colágeno e, por conseqüência, sendo mais suscetível à degradação (Sano *et al.* 1994).

Após ter identificado a melhor combinação para a TSD, foi avaliada a interação desta com diferentes tipos de cimento resinosos, por meio do teste de adaptação marginal e resistência de união (Capítulo 4).

Foi demonstrado que a utilização da TSD, associada ao cimento resinoso autocondicionante mostrou ter maior resistência de união comparada aos outros cimentos. Nikaido et al. (2003), já haviam verificado que essa combinação de sistema adesivo e cimento resinoso tinha boa compatibilidade, demonstrando bons resultados em ensaios mecânicos. No entanto, é importante considerar que a técnica indireta consiste em muitos passos, desde o preparo, moldagem, confecção da restauração e cimentação, sendo cada uma destas etapas essenciais no resultado final e no sucesso da restauração.

CONCLUSÕES GERAIS

Com base nos resultados obtidos e dentro dos limites dos presentes estudos, conclui-se que:

1. Nenhuma combinação de TSD foi capaz de prevenir a formação de fenda nas restaurações indiretas. No entanto, observou-se maior desadaptação nos grupos que não utilizaram “liner”.
2. A combinação de adesivo autocondicionante 2 passos/resina de baixa viscosidade apresentou maior resistência de união.
3. Os grupos que não utilizaram “liner” apresentaram menores valores de resistência de união.
4. Referente ao padrão de fratura, nas combinações de TSD que utilizaram “liner”, foi demonstrada a efetividade da técnica, não expondo tecido dentinário; no entanto, nos grupos que não utilizaram “liner, pode-se observar que na maioria das amostras, houve a exposição do tecido dentinário.
5. Independente da combinação utilizada, todas as amostras apresentaram nanoinfiltração por nitrato de prata, embora a combinação de adesivo autocondicionante 2 passos - resina composta de baixa viscosidade apresentou menor grau de nanoinfiltração.
6. O cimento resinoso autocondicionante mostrou ser mais compatível com a TSD, obtendo maior resistência de união, comparado a outros cimentos.

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ANEXOS

COMITÊ DE ÉTICA EM PESQUISA

FACULDADE DE ODONTOLOGIA DE PIRACICABA
UNIVERSIDADE ESTADUAL DE CAMPINAS

CERTIFICADO

O Comitê de Ética em Pesquisa da FOP-UNICAMP certifica que o projeto de pesquisa "**Efeito da técnica de selamento da dentina sobre a resistência adesiva, nanoinfiltração e adaptação marginal de restaurações indiretas após ciclagem térmica e mecânica**", protocolo nº **113/2005**, dos pesquisadores **ALBERTH DAVID CORREA MEDINA e MÁRIO ALEXANDRE COELHO SINHORETI**, satisfaz as exigências do Conselho Nacional de Saúde – Ministério da Saúde para as pesquisas em seres humanos e foi aprovado por este comitê em 19/10/2005.

The Research Ethics Committee of the School of Dentistry of Piracicaba - State University of Campinas, certify that project "**Effects of resin coat technique on tensile strength, nanoleakage and marginal adaptation of indirect restorations after thermal and mechanical load cycling**", register number **113/2005**, of **ALBERTH DAVID CORREA MEDINA and MÁRIO ALEXANDRE COELHO SINHORETI**, comply with the recommendations of the National Health Council – Ministry of Health of Brazil for researching in human subjects and was approved by this committee at 19/10/2005.

Cínthia P Machado Tabchoury
Cínthia Pereira Machado Tabchoury
Secretária
CEP/FOP/UNICAMP

Jacks Jorge Júnior
Coordenador
CEP/FOP/UNICAMP

Nota: O título do protocolo aparece como fornecido pelos pesquisadores, sem qualquer edição.
Notice: The title of the project appears as provided by the authors, without editing.

Dear Dr. Staff,

>

> On August 20, 2008, the manuscript entitled "Evaluation of the marginal adaptation of indirect restorations using different protocols of resin coating" by Alberth Correa-Medina, Andrea de Paula,Regina Puppin-Rontani and Mario Alexandre Sinhoreti, was fully submitted to Operative Dentistry.

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> The manuscript has been assigned the Paper #: 08-031-LR.

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> Sincerely,

>

> Joan Matis
> Editorial Assistant
> Operative Dentistry