FACULDADE DE ODONTOLOGIA DE PIRACICABA

UNIVERSIDADE ESTADUAL DE CAMPINAS

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Cirurgião-Dentista

EFEITO DE FONTES DE LUZ LED E CONDICIONAMENTO ÁCIDO DO ESMALTE NA RESISTÊNCIA À MICROTRAÇÃO, DUREZA KNOOP E FORMAÇÃO DE FENDAS DE RESTAURAÇÕES CLASSE I EM COMPÓSITO UTILIZANDO ADESIVOS AUTOCONDICIONANTES

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Orientador: Prof. Dr. Luís Alexandre Maffei Sartini Paulillo

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“...Põe quanto és no mínimo que fazes...”

Fernando Pessoa

Resumo

Os objetivos deste estudo foram: a) avaliar o efeito do condicionamento seletivo do esmalte cavosurperficial e da fonte de luz LED na resistência à microtração em dentina de restaurações Classe I em compósito utilizando adesivos autocondicionantes; b) avaliar a dureza Knoop (KHN) de restaurações de cavidades Classe I em compósito utilizando adesivos autocondicionantes fotoativados com diferentes fontes de luz LED; c) Avaliar a formação de fendas antes e após ciclagem termomecânica de restaurações em compósito fotoativadas por LED e utilizando adesivos autocondicionantes em diferentes protocolos de aplicação. No experimento 1, noventa e seis cavidades Classe I foram preparadas em molares humanos, e restauradas com compósito micro-híbrido. Os espécimes foram distribuídos em 12 grupos (n=8), de acordo com a fonte de luz LED (Radii-Cal - RD, Flash Lite 1401 - FL e Ultra-Lume 5 - UL), sistema adesivo autocondicionante (Clearfil SE Bond – CSE e Clearfil S^3 Bond – S3) e condicionamento seletivo do esmalte com ácido fosfórico (com e sem). Após o acabamento e polimento das restaurações as mesmas foram submetidas à fadiga termo-mecânica e os palitos para o ensaio de microtração foram obtidos da interface de união entre a parede pulpar dentinária e a restauração. Após o teste mecânico os dados foram submetidos à análise estatística (ANOVA três fatores e teste de Tukey com $\alpha=0.05$). O condicionamento seletivo do esmalte para o S3 diminuiu a resistência de união em dentina e não afetou o desempenho do CSE. Além disso, o LED de terceira geração promoveu maiores valores de resistência à microtração para o adesivo de dois passos. Para o S3 as fontes de luz promoveram resistência adesiva semelhante em dentina. No estudo 2, utilizou-se o mesmo método de preparo das amostras do primeiro estudo, formando-se 6 grupos (n=16) pela interação entre os fatores em estudo, LED (RD, FL e UL), sistema adesivo autocondicionante (CSE e S3). As

parcelas subdivididas foram diferentes profundidades (superficial, média e profunda). Após a confecção das restaurações, as mesmas também foram submetidas à fadiga termo-mecânica e em seguida, seccionadas no sentido longitudinal para avaliação da KHN nas diferentes profundidades. Após análise estatística (ANOVA dois fatores com parcela subdividida e teste de Tukey com α=0.05), observou-se que o LED de terceira geração promoveu valores de KHN semelhante aos aparelhos de pico único e que a região superficial, em geral, apresentou melhor qualidade de polimerização. No estudo 3, utilizou-se 192 terceiros molares, onde cavidades Classe I foram preparadas e restauradas com o mesmo método utilizado no primeiro estudo, 12 grupos (n=16) pela interação entre os fatores em estudo, LED (RD, FL e UL), sistema adesivo autocondicionante (CSE e S3) e condicionamento seletivo do esmalte com ácido fosfórico (com e sem), antes e após a termociclagem. Após a restauração, réplicas em resina epóxica foram confeccionadas e as margens foram analisadas em microscópio eletrônico de Varredura (MEV). Após submeter os espécimes da restauração à fadiga termo-mecânica, novamente réplicas em resina epóxica foram feitas e analisadas em MEV. Após análise estatística (Kruskal Wallis e teste de Wilcoxon pareado, α=0.05), observou-se que o condicionamento seletivo do esmalte promoveu maior integridade marginal para o S3, mostrando ser um passo adicional eficiente na técnica de aplicação desse adesivo em restaurações Classe I em compósito. Além disso, a formação de fendas para adesivo autocondicionante de dois passos não foi influenciado pelo condicionamento ácido prévio do esmalte, nem pelo LED utilizado para fotoativação.

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Palavras-chave: Adesivo autocondicionante, LED, esmalte dental, ácido fosfórico, resistência da união, dureza Knoop,adaptação marginal

Abstract

The aim of this work was to: a) evaluate the effect of enamel selective enamel etching and LED curing units on dentin microtensile bond strength of Class I composite restorations; b) evaluate the effect of different LED light units and self-etch adhesives on the Knoop hardness profile (KHN) of Class I composite restorations; c) evaluate the gap formation before and after thermomechanical loading of Class I composite restorations photocured by LED and using different application protocols of self-etch adhesives systems. In experiment 1, ninety-six Class I cavities were made in human molars and restored with a microhybrid composite. For the restorative procedure, teeth were assigned in 12 groups (n=8) formed by the studied factors interactions, LED curing unit (Radii-Cal - RD, Flash Lite 1401 -FL and Ultra-Lume 5 - UL), self-etch adhesives (Clearfil SE Bond - CSE and Clearfil S^3 Bond – S3) and selective enamel acid etching (with and without). After finishing and polishing approach, specimens were submitted to thermo-mechanical ageing and sticks were obtained from the bonding interface between adhesive and pulpal wall which were submitted to a microtensile load in a universal testing machine. After statistical analysis, (three-way ANOVA and Tukey test with α=0.05) it was shown that the pre-etched enamel approach for Clearfil S^3 decreased dentin bond strength while it did not affect the bond performance of Clearfil SE. Thus, the polywave LED promoted higher microtensile bond strength values for the two-step adhesive. When Clearfil S^3 was used, the light curing units yielded similar dentin bond strength. In the experiment 2, 96 Class I cavities were prepared in the same way as the previous experiment. Teeth were assigned to 18 groups (n=8) by the interaction among the studied factors, LED curing source (RD, FL and UL), self-etch bond system (CSE and S3), depth (superficial, medium and deep). After the restorative procedure, specimens were submitted to thermo-mechanical ageing

and then sectioned in the longitudinal direction for the Knoop hardness measurement in the different depths. Data were submitted to three-way ANOVA and Tukey test with a pre-set alpha of 0.05). The results showed that the third generation LED promoted similar KHN values to the single-peak ones and the superficial surface, in general, presented greater cure quality compared to the others surface depths. In the experiment 3, 192 third molars were used and Class I preparations were restored in the same way as the first study. Specimens were assigned to 12 groups (n=16) formed by the studied factors interaction, LED (RD, FL and UL), self-etch adhesives (CSE and S3), selective enamel acid etching (with and without) and before and after thermomechanical loading. After the restoration procedure, epoxy resin replicas were made and the marginal adaptation was analyzed in a scanning electron microscope (SEM). Then specimes were submitted to thermalmechanical loading and again, epoxy resin replicas were made and analyzed by SEM. After statistical analysis (Kruskal Wallis and Wilcoxon matched-pairs signed-ranks test, with a pre-set alpha of 0.05), it was shown that selective enamel etching yielded higher marginal integrity for S3, showing to be an efficient additional step for the bonding technique of this adhesive in Class I composite restorations. Also, the two-step self-etch adhesive gap formation was not influenced by the selective enamel etching, neither by the LED curing light.

Key-words: Self-etch adhesive, LED, dental enamel, phosphoric acid, bond strength, Knoop hardness, gap formation.

Sumário

Introdução

Para se obter união adequada aos substratos dentais podem ser utilizadas duas técnicas para a formação da camada híbrida, condicionamento total do esmalte e dentina e adesivos autocondicionantes (BRACKETT *et al.,* 2008; PERDIGÃO, LOPES e GOMES, 2008). Os adesivos autocondicionantes possuem técnica mais simplificada, requerem menor tempo clínico para aplicação, não necessitam de condicionamento ácido prévio do esmalte e dentina, além de não promover a completa remoção da *smear layer* (PERDIGÃO *et al.,* 2006; PERDIGÃO, LOPES e GOMES, 2008).

Os primeiros sistemas autocondicionantes foram desenvolvidos através do aumento da concentração dos monômeros resinosos ácidos no primer (WATANABE *et al*., 1994). Esses sistemas apresentam dois passos de aplicação: primer autocondicionante seguido da aplicação de resina hidrófoba (KNOBLOCH *et al*. 2007; UEKUSA *et al.,* 2007; BAGIS *et al.,* 2008). Por outro lado, os adesivos autocondicionantes de passo único podem ser apresentados em dois frascos, em que os líquidos são misturados antes da aplicação, e em frasco único. Estes sistemas possuem simplicidade da técnica e têm mostrado inadequada penetração dos monômeros resinosos na estrutura dental, podendo resultar em degradação acelerada da interface de união compósito/esmalte (PERDIGÃO *et al.,* 2006; KNOBLOCH *et al*. 2007; UEKUSA *et al.,* 2007; BAGIS *et al.,* 2008).

Com o intuito de aumentar a longevidade e qualidade da união dente/restauração, tem-se sugerido o condicionamento seletivo do esmalte cavosuperficial antes da aplicação de adesivos autocondicionantes. (WATANABE *et al.,* 2008; ERICKSON *et al*., 2009; ERMIS *et al*., 2010; PEUMANS *et al*., 2010). A baixa capacidade de desmineralização da estrutura do esmalte pelos adesivos autocondicionantes de acidez

média torna-se insuficiente para promover um aumento da energia livre de superfície do esmalte e permitir uma maior penetração dos monômeros das resinas adesivas. Dessa maneira, a aplicação prévia do ácido fosfórico como passo adicional na utilização de adesivos autocondicionantes permite uma mais profunda desmineralização dos prismas e regiões interprismáticas do esmalte dental, promovendo maior resistência da união e, conseqüentemente, integridade marginal (WATANABE *et al.,* 2008; ERICKSON *et al*., 2009; ERMIS *et al*., 2010; PEUMANS *et al*., 2010).

No entanto, em restaurações Classe I, em compósito, a dentina também pode ser afetada por este passo técnico adicional, já que o aumento da resistência da união no esmalte pode gerar maior tensão na interface de união em compósito/dentina (BORTOLOTTO *et al*., 2007; TASCHNER *et al*., 2010). Com isso, torna-se importante avaliar a influência do condicionamento ácido seletivo do esmalte cavosuperficial em restaurações Classe I em compósito na interface de união em dentina.

Por outro lado, a qualidade da união entre compósito e dente depende de muitos fatores, dentre os quais, a tensão gerada durante a polimerização do compósito (CALHEIROS *et al.,* 2007; CUNHA *et al.,* 2008). Se a tensão de contração for maior que a resistência de união entre sistema adesivo e substrato dental, poderá haver falha precoce da mesma, ocasionando formação de fendas e infiltração marginal de produtos do metabolismo bacteriano, além de sensibilidade pós-operatória (SILVA *et al.,* 2006; CALHEIROS *et al.,* 2007; BRANDT *et al.,* 2008).

Assim, para o compósito, a tensão gerada durante a polimerização depende do fotoiniciador, geometria da cavidade, composição do monômero, concentração de catalisador e ainda do tipo e quantidade de partículas de carga, além da técnica restauradora e da fonte de luz (ALONSO *et al.,* 2007; CUNHA *et al.,* 2008).

Existem diferentes fontes de luz utilizadas para a polimerização de compósitos fotoativados, como a luz de lâmpada halógena, LED, arco de plasma e Laser de argônio (CUNHA *et al.,* 2008). O fotoiniciador mais utilizado nos materiais ativados pela luz é a canforoquinona, que possui seu pico de absorção luminosa em 468nm (ANTONSON, ANTONSON E HARDIGAN, 2008). Por isso foram desenvolvidas as fontes de luz LED que emitem luz com comprimento de onda próximo a essa faixa de absorção de luz pela canforoquinona (455-480 nm), além de possuírem algumas vantagens clínicas como durabilidade, leveza, ergonomia, menor aquecimento e não requererem filtros (AVARAMUDHAN *et al.,* 2006; OWENS e RODRIGUEZ, 2007; ANTONSON, ANTONSON E HARDIGAN, 2008; BRANDT *et al.,* 2008).

As primeiras unidades emissoras de luz LED, chamadas de primeira geração, possuíam várias lâmpadas LED sem intensidade luminosa satisfatória, em torno de 150 mW/cm^2 (CAMILOTTI et *al.,* 2008). Por isso, foram desenvolvidos LED de segunda geração, com intensidade luminosa mais alta resultando em maior qualidade de polimerização dos materiais resinosos à base de canforoquinona (PRICE, FELIX e ANDREOU, 2003; AVARAMUDHAN *et al.,* 2006; OWENS e RODRIGUEZ, 2007; ANTONSON, ANTONSON E HARDIGAN, 2008; CAMILOTTI et *al.,* 2008).

Por outro lado, as diferentes cores das resinas compostas podem influenciar a qualidade da fotoativação, devido à quantidade de canforoquinona presente em cada material (GOMES, *et al*., 2006). Como a canforoquinona possui cor amarelada ela é substituída e/ou reduzida pela adição de outros fotoiniciadores, como a 1-fenil-1,2-propanodiona (PPD) que são excitados por luz com comprimentos de onda menor, como a luz violeta, com 405 nm (PRICE, FELIX e ANDREOU, 2005; GOMES, *et al*., 2006). Assim foi desenvolvido o LED de terceira geração que possui maior efetividade de fotoativação para os compósitos com cores claras indicados para restauração de dentes

clareados. Este aparelho usa dois diferentes tipos de LED e possuem comprimentos de onda que variam de 350 a 500 nm (comprimentos de onda da luz azul e violeta), com o intuito de excitar todos os fotoiniciadores, e não apenas a canforoquinona (ANTONSON, ANTONSON E HARDIGAN, 2008; CAMILOTTI *et al*., 2008).

Em virtude da complexa interação entre estabilidade marginal, força de união e efetividade de polimerização em cavidades classe I em compósitos, se faz necessária a avaliação desses parâmetros para se obter uma técnica clínica com prognóstico mais previsível. Dessa forma, o condicionamento ácido seletivo do esmalte cavosuperficial pode influenciar na integridade da união dos adesivos autocondicionantes com o substrato dental em restaurações Classe I que, se inadequada, pode ocasionar sensibilidade pós-operatória e subseqüente falha na união entre adesivo e dente.

Capítulo 1

Influence of selective enamel etching and LED curing source on dentin bond strength of self-etch adhesives in Class I composite restorations

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Summary

The aim of this study was to evaluate the influence of selective enamel etch and LED curing unit on dentin microtensile bond strength (μTBS) for self-etch adhesive systems in Class I composite restorations. Thus, on 96 human molars, a box-shaped Class I cavity was made maintaining enamel margins. Self-etch adhesives (Clearfil SE – CSE and Clearfil S^3 – S3) were used to bond a microhybrid composite. Before the application of the adhesives, half of the teeth were enamel etched for 15s with 37% phosphoric acid; the other half was not etched. The adhesives and composite curing was achieved with three light curing units (LCUs): one polywave (UltraLume 5 - UL) and two single-peak (FlashLite 1401 - FL and Radii Cal - RD) LEDs. After, specimens were submitted to thermo-mechanical aging and longitudinally sectioned in the "x" and "y" directions to obtain bonded sticks (0.9 mm^2) to be tested in tension at 0.5 mm/min. The failure mode then was recorded. The μTBS data were submitted to a three-way ANOVA and Tukey's (α=0.05). The double interactions enamel etching/adhesive system and LCU/adhesive system were statistically significant ($p \leq 0.05$). For S3, the selective enamel etching provided lower μTBS values (20.7 ± 2.7) compared to the non-etched specimens (26.7 ± 2.2). UL yielded higher μTBS values (24.1 ± 3.2) in comparison to the photoactivation approach with FL (18.8 ±3.9) and RD (19.9 ±1.8) for CSE. The two-step CSE was not influenced by the enamel etching ($p \geq 0.05$). In conclusion, enamel acid etching in Class-I composite restorations affects the dentin μTBS of the ultra-mild one-step self-etch adhesive Clearfil S^3; however, they do not alter the two-step self-etch bond system Clearfil SE retention. The polywave LED promoted better bond strength for the two-step adhesive compared to the single-peak ones.

Keywords: Acid etching, Enamel, Self-etch adhesives, Light curing units, bond strength

Introduction

Resin composites have been widely used in direct aesthetic restorative procedures. Self-etching bond systems were introduced in order to reduce the sensitivity of techniques that affect the bonding ability of adhesive systems; they also decrease the number of steps in the bonding approach because they include acidic monomers that simultaneously condition and prime both enamel and dentin.[1,2] The absence of the rinsing approach and partial removal of the smear layer and smear plugs with these adhesives leads to less technique-sensitive, time-consuming procedures, and consequently, a possible reduction of postoperative sensitivity results.[2,3,4,5]

Moreover, a recent study has shown that the enamel smear layer compromises bonding by mild self-etch adhesives.[6] Separate preliminary etching is known to remove the smear layer created by instrumentation and to demineralize the dental substrate. The prior enamel phosphoric acid etching as an additional step for self-etch adhesives has been proposed to improve the durability of the enamel bond.[7,8,9,10] Hence, the selective enamel increases marginal adaptation of self-etch adhesives as evidenced by numerous studies.[4,5,11,12,13,14]

While it is clear that selective enamel etching may have some beneficial effects on marginal integrity in Class-V cervical lesions,[5,11,13] the literature provides no evidence for the bonding performance of self-etching adhesives in Class I cavities for direct composite restorations *in vitro*. Since Class-V cervical lesions restorations present smaller amounts of dental enamel, it may not be the best model for understanding the real effects of enamel etching on dentin substrate and the stress development on the tooth/adhesive interface, like Class I restorations.[13,15] In this sense, the literature gives no evidence regarding the role of prior enamel acid etching on the bond strength of self-etch adhesives to dentin in Class I cavities in vitro.

Another important fact to consider in restoring class I cavities with self-etch bond systems is the selection of an efficient light curing unit (LCU). The LCU should guarantee a satisfactory monomer conversion of the adhesive and composite. Consequently, it may improve physical and mechanical properties, such as ultimate tensile strength and bond strength to dental substrate. [16,17,18,19,20] Light emitting diodes (LEDs) have been shown to effectively cure composites and dental adhesives, emitting unfiltered blues light.[17,18,21] These LCUs generate a narrow spectral range that targets the absorption wavelength of camphorquinone, yielding low amounts of wasted energy and minimum heat generation.[22,23]

Previous studies have shown that current LED curing units can replace, in most situations, the conventional QTH light units.[24,25,26] Some adhesive systems are not well cured with single-peak LEDs[21] due to alterative photoinitiators content; to overcome this problem, polywave third generation LEDs were introduced in the market.[24,25,26] These LEDs emit light wavelengths targeting the absorption peak of camphorquinone. However, they deliver additional light output at the UV-Vis region of the electromagnetic spectrum (400 - 415 nm). The second peak in the UV-VIS region may promote a satisfactory cure of these adhesives, containing alternative photoinitiators systems, such as phenyl propanedione (PPD), bis-alquyl phosphinic oxide (BAPO) and Trimethylbenzoyl-diphenyl-phosphine Oxide (TPO).[26] This point is crucial because some manufacturers do not state all of the photoinitiators systems used in their proprietary products,[25,27] and the selection of an adequate LCU is primordial to yielding an adequate adhesive polymerization and resin/dentin bond strength.

Thus, this study aimed to investigate the influence of selective enamel acid etching and different LED curing units on dentin microtensile bond strength (μTBS) of self-etching bond systems in class I composite restorations. The first tested hypothesis was that the

previous enamel etching would not affect microtensile bond strength values of the tested adhesive systems. The second hypothesis was that the third generation polywave LED would present higher dentin bond strength values for the tested bond systems.

Materials and Methods

LED Curing Units

Three light-emitting diodes (LEDs) were tested: FlashLite 1401 (FL) (Discus Dental, Culver City, CA, USA), Radii-Cal (RD) (SDI Limited, Victoria, Australia) and UltraLume LED 5 (UL) (Ultradent, South Jourdan, Utah, USA). Characteristics of the LCUs used in this work are presented in Table 1. Irradiance (mW/cm^2) was measured dividing the output power (measured by a calibrated power meter - Ophir Optronics, Har – Hotzvim, Jerusalem, Israel) by the tip end area of the LCUs. The Spectra of the LED units was measured with a calibrated spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA), as shown in Figure 1. The energy dose was standardized to approximately 11 J/cm^2 for the bonding systems curing approach and 22 J/cm^2 for the cure of each composite increment. Because the RD light source mandatorily operates in ramp mode for 5s, these initial seconds were discarded and further application was realized in order to maintain the standardized irradiance, consequently equaling the energy density obtained for each of the LCUs.

Specimen's Preparation

Ninety-six freshly-extracted, sound human molars were selected. This study had the approval of the Ethical Committee of the State University of Campinas, Piracicaba Dental School. The teeth were cleaned, their roots inserted in polystyrene resin and the occlusal surfaces wet polished with 320-grit SiC paper under running water (Politriz, AROTEC – São Paulo, SP) in order to expose a flat enamel surface without exposing dentin. Box-

shaped class I cavities were prepared using #56L carbide burs (KG Sorensen, Barueri, SP, Brazil) at high-speed, under air–water cooling. A custom-made preparation device allowed the standardization of the cavity dimensions to 5 mm mesio-distal length, 5 mm bucco-lingual width and 3 mm depth, all cavity margins on enamel substrate. The used bur was replaced after four cavity preparation.

After cavity preparation, teeth were assigned to 12 groups (n=8) according to the three studied factors: selective enamel etching, bond system and curing light (2 conditioning protocols x 2 adhesives x 3 LEDs). Two self-etch adhesive systems (two-step and one-step) were used for the bonding procedure: Clearfil Tri-S Bond (pH= 2.7; Kuraray Medical Inc., Tokyo Japan) or Clearfil SE Bond (pH = 2.0; Kuraray Medical Inc., Tokyo Japan). The composition, application mode and batch number of the adhesive systems are presented in Table 1. All groups were restored with B1-shade Charisma microhybrid composite (Heraeus Kulzer, Hanau, Germany) using an incremental oblique technique with increments of approximately 2mm thick. Then, the finishing and polishing procedures were performed with medium-, fine-, and extra fine- grit aluminum oxide disks (SoftLex – 3M/ESPE), respectively. After polishing, the specimens were submitted to 200.000 mechanical loading (2Hz) and 500 thermal cycles (ranging from 5 to 55°C with a dwell time of 60s in each bath with an interval of 5s) at a thermo-mechanical device ER-11000 (ERIOS, São Paulo, Brazil) in order to simulate similar ageing ofto the composite restorations in oral environment conditions.

Microtensile Bond Strength

After storage in distilled water at 37ºC for 24h, specimens assigned to the MTBS test were longitudinally sectioned in both “x” and “y” directions across the pulpal wall bonded interface. This sectioning was done with a diamond saw (Isomet 1000, Buehler Ltd, Lake Bluff, IL, USA) at 300 rpm to obtain sticks with a cross-sectional area of

approximately 0.9 mm^2. The cross-sectional area of each stick thus was measured with the digital caliper to the nearest 0.01 mm and recorded for the bond strength calculation. Sticks were individually attached to a jig for microtensile testing with cyanoacrylate resin (Super Bond gel, Loctite, Henkel, Brazil) and subjected to a tensile force in a universal testing machine (EMIC DL500, São José dos Pinhais, Brazil) at a crosshead speed of 0.5 mm/min until failure.

Bond failure modes were evaluated with a scanning electron microscope (JEOL, JSM-5600LV, Scanning Electron Microscope, Japan) and classified as: 1) cohesive (failure exclusive within dentin or resin composite, 2) adhesive (failure at resin/dentin interface or cohesive in adhesive resin) or 3) mixed (mixed with cohesive failure of the neighboring substrates). The obtained data were subjected to a three-way ANOVA and Tukey´s test at a pre-set alpha of 0.05.

Results

Microtensile Bond Strength

Approximately 16 sticks could be obtained per tooth, including those with premature debonding. Pre-test failures were included in the statistical analysis as half of the minimum bond strength measured for each tooth.[27] The results of μTBS are shown in tables 3, 4 and 5. The double interactions between the factors adhesive x selective etching ($p \leq 0.05$) and adhesive x LED light ($p \leq 0.05$) were statistically significant.

Phosphoric acid etching pre-treatment of enamel prior to application of S3 led to lower dentin μTBS values ($p \leq 0.05$). However, for CSE, the pre-etched enamel yielded a similar dentin μTBS mean compared to the non-etched specimens. Dentin bond strength of CSE did not statistically differ from S3 when prior etching was used before the adhesive application. Moreover, when these adhesives were applied following the conventional

approach, S3 presented higher bond strength mean in comparison to the two-step self-etch adhesive.

All LED curing units promoted similar dentin μTBS for the one-step self-etch bond system S3 ($p \geq 0.05$). In contrast, the two-step CSE showed higher dentin μTBS (24.1 ± 3.2) when UL was used in comparison to the single-peak LEDs FL (18.8 ± 3.9) and RD (19.9 ± 1.8).

SEM evaluation of the fractured surfaces showed predominant adhesive failures for CSE when the enamel prior acid etching was used for the bonding approach (Figure 3). A similar failure mode was found for S3 in the same condition. However, when the self-etch adhesives were conventionally used, mixed failures were present (Figure 4), and one cohesive failure was shown on composite for CSE when photocured with FL.

Discussion

The selective enamel etching is related to increased bond performance of self-etch adhesives to ground enamel.[8,9] However, the influence of this additional procedure on the bond strength to pulpal dentin in Class-I composite restoration has not been clarified. In this sense, the pre-etching increases the surface energy of the enamel substrate and favors mechanical interlocking with the bonding resin.[28] In this study, the bonding ability of self-etch adhesives to pulpal dentin with different application approaches was evaluated in Class I restorations. These Class I preparations frequently present enamel in the following manner: on the cavossurface, some internal margins and dentin on the pulpal wall, and on the angle formed by the lateral wall and pulpal floor, with different bonding substrates, compared to a Class V or a Class III cavity. In contrast, there are many studies evaluating the bonding performance of self-etch adhesives and prior acid etching on flat enamel surfaces.[29,30,31] These circumstances do not simulate clinical situations for direct

composite application because the bond performance of dental adhesives occurs with the shrinkage stress influenced by the cavity configuration and presented dental substrates.[32,33]

Based on the results, the first hypothesis was partially validated, since for CSE, the previous acid etching did not influence the μTBS results. In contrast, S3 adhesive showed lower dentin μTBS when this additional etching step was performed. The conventional application of the self-etch adhesive implies a good retention performance for dentin and a poor bonding approach for enamel.[5,28,31] Thus, the slightly inferior enamel bonding performance may yield minor stress at the margins, resulting in fewer gaps and paramarginal enamel fractures.[15] However, when enamel is pre-etched before the self-etch adhesive application, higher bond strength between enamel/adhesive and resulting stress may occur and affect dentin bond performance. The selective etching for the S3 application may have caused higher stress while the adhesive layer bonded to dentin might have disrupted and decreased the dentin μTBS.

In addition, the stress might have been enhanced due to the thermal and mechanical ageing promoted to the class I restorations, favoring the decrease in the retention values. The failure mode was mainly adhesive when the previous acid etching was performed for S3; therefore, mixed failures were more present when this adhesive was used in the conventional way. For the CSE, the application of the hydrophobic resin layer may have supported the stress promoted by enamel selective etching, but did not affect the dentin μTBS when compared to the unique layer applied for the S3. It is suggested that previous enamel etching be considered for S3 in restorations whose retention primarily depends on a strong bond to the enamel surface, such as large Class III and IV or veneer restorations.

In recent studies,[5,11,14,31] selective enamel etching was shown to improve marginal integrity of composite restorations using either a two-step or a one-step self-etching adhesive. The decrease in marginal gaps therefore is an important bonding behavior because gap formation represents the first sign of restoration failure, which can be clinically evidenced by marginal staining.[5,8,11,34,35] However, the S3 can promote a decrease in the bonding ability of this single-step adhesive to the retention on the pulpal dentin. Therefore, for a gold-standard CSE, it is assumed that prior acid etching can promote better marginal sealing without compromising the dentin μTBS.

The impact of the adhesive bonds to the dental substrate of composite restorations is strongly related to LCU efficacy, which will promote a satisfactory materials polymerization.[21] LED technology has been used for resinous materials polymerization as a substitute for conventional QTH curing lights.[36,37] However, the conventional single-peak second generation LEDs still harm the cure of dental resins due to the narrower spectra emitted to the camphorquinone initiator.[38,39,40] They do not allow an optimal cure for other photosensitizes, like phenil propanedione and TPO, present in some dental materials.[26] Polywave LEDs were introduced in order to overcome these concerns, promoting the conversion of this UV-Vis photoinitiator content.[24,25,26]

In the present work, the second hypothesis was partially validated, because for CSE, the polywave third generation LED UL promoted higher dentin μTBS values compared to the single-peak second generation ones. However, S3 presented similar dentin bond strength regardless of the LCU used. For CSE, the results are in accordance with authors [21,41] who observed that single-peak LEDs promoted a lower degree of cure for CSE compared to a LCU with a large spectrum range distribution, comprising the UV-Vis wavelength. This finding may be attributed to the photoinitiator content of CSE; the manufacturer may omit the presence of other photosensitizers. Another possible

explanation is that the absorption peak of camphorquinone can be moved to lower wavelengths. The LED unit continues to deliver shining light because the structural part of this initiator, responsible for the short-wavelength absorption, remains unchanged while the photoreaction at the initial maximum absorption peak (468 nm) ends.[42] It may contribute to better bond strength values with the polywave LED UL, which have broader emission spectra, and it may have promoted a better monomer conversion compared to the single-peak ones. However, it is questionable whether 10 to 20 seconds of light activation is sufficient to enable this effect.

The dentin bond strength of S3 was not affected by the LCU used. A thin adhesive layer was applied on the enamel and dentin surface of the Class I preparation, and the curing light may have excited the material's photoinitiator content. Nevertheless, the energy dose for all LEDs was standardized at approximately 11 J/cm^2 for the bonding system, which may have delivered the same quantity of photons to allow the satisfactory cure of S3. Consequently, similar dentin bond strength values were achieved, regardless of the spectrum range and irradiance of the LCUs.

Although the etching approach for self-etch adhesives is recommended by many authors,[4,5,11,12,13,14] the inclusion of an additional step for these bond systems may be controversial, since these adhesives intend to represent a clear simplification of the clinical application of bond systems. In this sense, the adverse points of this selective approach include the increase of the bonding steps and the technique sensitivity.[13]

Although some in vitro studies exist, final conclusions regarding the role of the enamel selective acid etching for self-etch adhesives in Class I composite restorations will depend on the outcomes of clinical trials. Clinical long-term studies and investigations of this approach's retention ability for bond systems in the oral environment can best evaluate the quality and durability of these restorations.

Conclusion

The additional enamel acid etching in Class-I composite restorations affects the dentin bond strength of the ultra-mild one-step self-etch adhesive Clearfil S^3. However, this selective step should be indicated for two-step self-etch bond systems, since it did not alter Clearfil SE retention. The polywave LED promoted better bond strength for the two-step adhesive compared to the single-peak ones. Moreover, the LCUs presented similar cure potential for the one-step self-etch adhesive.

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LEGENDS

Figure 1. The spectrum range distribution of each LCU used in this study.

Figure 2. Specimen preparation for the microtensile bond strength test. (A) Sound human molar. (B) Box-shaped class I cavity on the flat enamel tooth surface. (C) Sections in both "x" and "y" directions across the composite restoration to obtain rectangular sticks. (D) Rectangular stick with a cross-sectional area of approximately 0.9 mm^2. (E) Stick attached to a jig for microtensile testing.

Figure 3. SEM of the fractured specimen showing mixed failure within cohesive in adhesive layer and adhesive failure.

Figure 4. SEM of the fractured specimen showing a pure adhesive failure within the bonding interface.

Figure 5. SEM observation of Clearfil S^3 - osmosis induced droplets due to the adhesive hydrophilicity, especially because of the presence of HEMA. It is not a phase separation droplets, because this adhesive do not present phase separation – Clearfil S3

Figure 6. SEM observation of the interface between the bond system and dentin at 350X magnification in the mixed failure mode.

Figure 6. SEM observation of the adhesive failure mode in 950X of the dentin surface in the adhesive failure mode.

Table 1. Characteristics of the LCUs used in this study.

LCU	Manufacturer	Type	Tip Diameter (mm)	Irradiance (mW/cm^2)	Radiant exposure (J/cm^2)
FlashLite 1401	Discus Dental, Culver City, CA, USA	Single-peak	7	1077	22
Radii Cal	SDI Limited, Victoria, Australia	Single-peak	7	731	22
UltraLume LED 5	Ultradent products Inc, South Jourdan, Utah, USA	Polywave	11 x 7	800	22

Table 2. Composition, application mode and manufacturer's information for the adhesive systems tested.

Adhesive systems	Composition	Application mode
Clearfil SE Bond (Kuraray Medical Inc., Tokyo Japan)	Primer (Batch #00896A): water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate. Adhesive (Batch #01320A): MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimathacrylate, N,N-diethanol p-toluidine bond, colloidal silica.	Apply Primer for 20s. Mild air stream. Apply Bond. Gentle air stream. Light cure at a energy density of 11J.
Clearfil S3 Bond (Kuraray Medical Inc., Tokyo, Japan)	Adhesive (Batch #00116A): MDP, Bis-GMA, HEMA hydrophobic dimethacrylate, dl-camphorquinone, silanated colloidal silica, ethyl alcohol and water.	Apply the bond system for 20s. Gentle air stream for 10s. Light cure at a energy density of 11J.

Table 3. Overall microtensile bond strength means and standard deviations (MPa)

Adhesive system	**Light curing unit**					
	FlashLite 1401		Radii Cal		UltraLume 5	
	Etching	No etching	Etching	No etching	Etching	No etching
Clearfil SE Bond	19.0 (3.6)	18.7 (3.9)	19.0 (4.1)	20.8 (3.0)	23.0 (3.7)	25.3 (2.4)
Clearfil S^3	21.8 (2.0)	26.9 (2.9)	20.2 (2.6)	26.2 (3.2)	20.2 (3.8)	26.9 (3.6)

obtained in each experimental condition.

Table 4. Means and standard deviation of the microtensile bond strength for the interaction between adhesive system and selective etching.

Adhesive System	**Selective acid etching**	
	Etching	No etching
Clearfil SE Bond	20.3 (3.3)Bb	21.6 (3.2)Bb
Clearfil S^3	20.7 (2.7)Bb	26.7 (2.2)Aa

Mean values followed by different small letters in the column and capital letters in the row differ statistically among themselves for the Tukey test at the level of 5%.

Table 5. Means and standard deviation of the microtensile bond strength for the interaction between adhesive system and light curing unit.

Adhesive system	**Light curing unit**		
	FlashLite 1401	Radii Cal	UltraLume 5
Clearfil SE Bond	18.8 (3.9) Bb	19.9 (1.8) Bb	24.1 (3.2) Aa
Clearfil S^3	24.1 (2.8) Aa	23.2 (3.4) Aa	23.5 (3.8) Aa

Mean values followed by different small letters in the column and capital letters in the row differ statistically among themselves for the Tukey test at the level of 5%.

Table 6. Percentage of specimens (%) from Clearfil SE Bond adhesive distributed according to the failure mode.

LCU	Prior phosphoric acid etching			Without prior phosphoric acid etching		
	A	M	C	A	M	C
FlashLite 1401	75	25	0	64	24	1
Radii Cal	100	0	0	100	0	0
UltraLume LED 5	60	40	0	70	30	0

A: adhesive failure and cohesive failure within the adhesive; M: mixed failure; C: cohesive failure within dentin or composite resin.

Table 7. Percentage of specimens (%) from Clearfil S^3 adhesive distributed according to the failure mode.

LCU	Prior phosphoric acid etching			Without prior phosphoric acid etching		
	A	M	C	A	M	C
FlashLite 1401	80	20	0	50	50	0
Radii Cal	70	30	0	66	34	0
UltraLume LED 5	85	15	0	60	40	0

A: adhesive failure and cohesive failure within the adhesive; M: mixed failure; C: cohesive failure within dentin or composite resin.

Figure 1

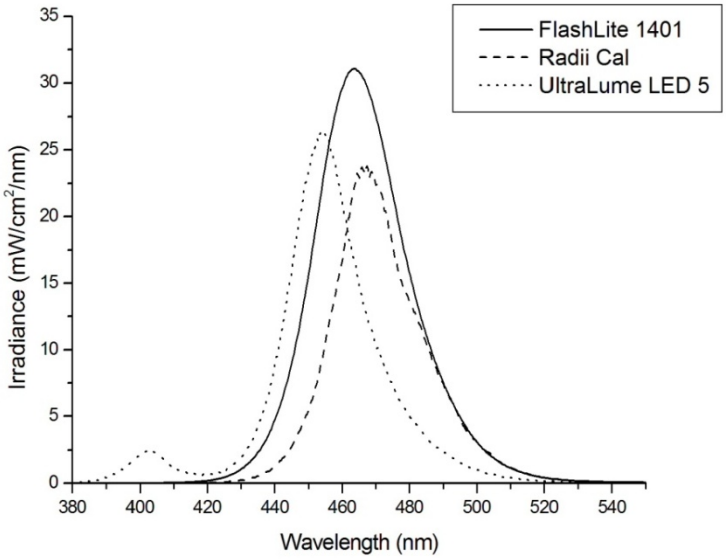


Figure 2

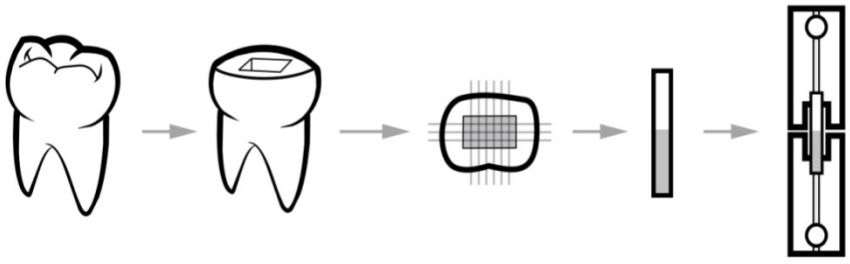


Figure 3

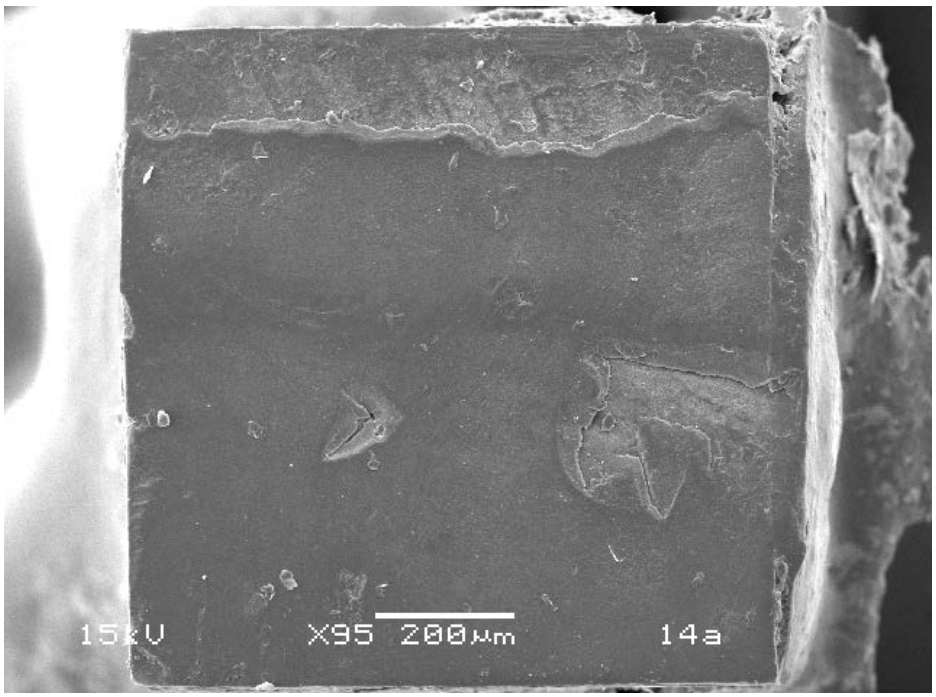


Figure 4

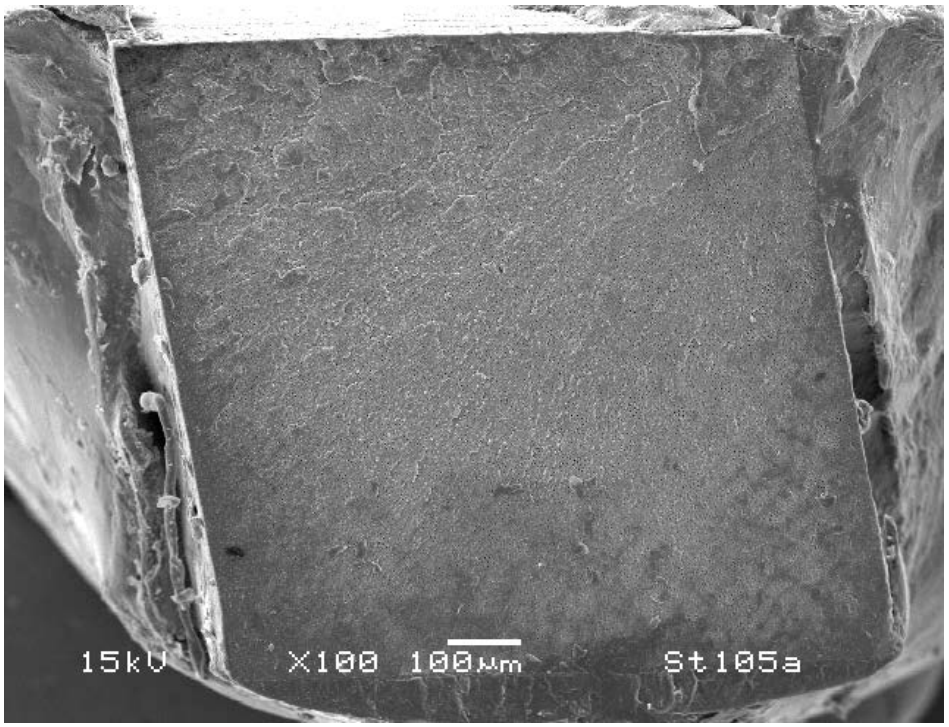


Figure 5

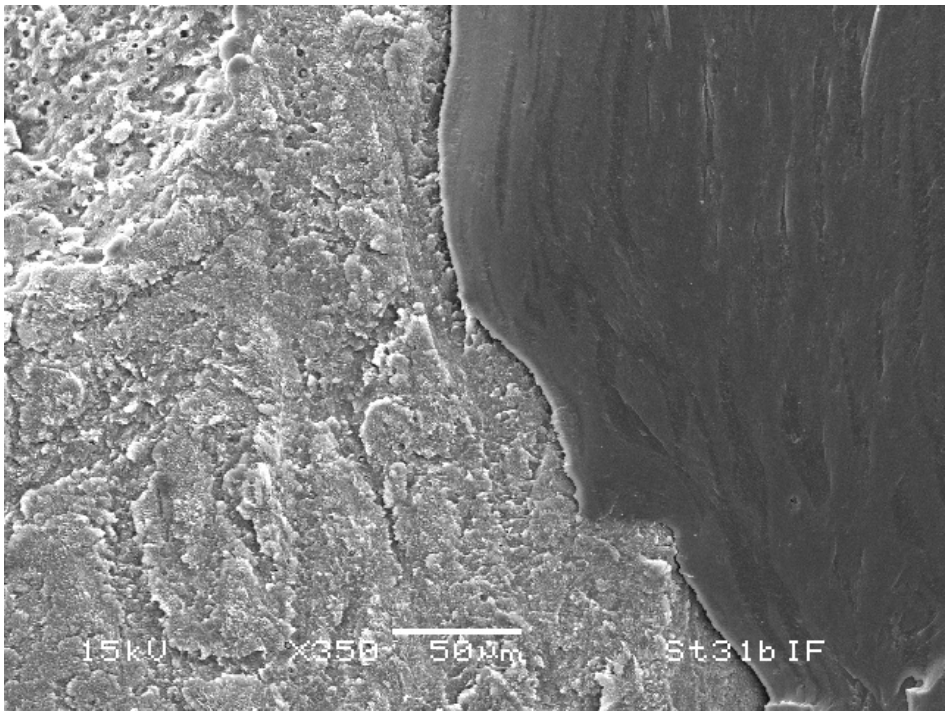


Figure 6

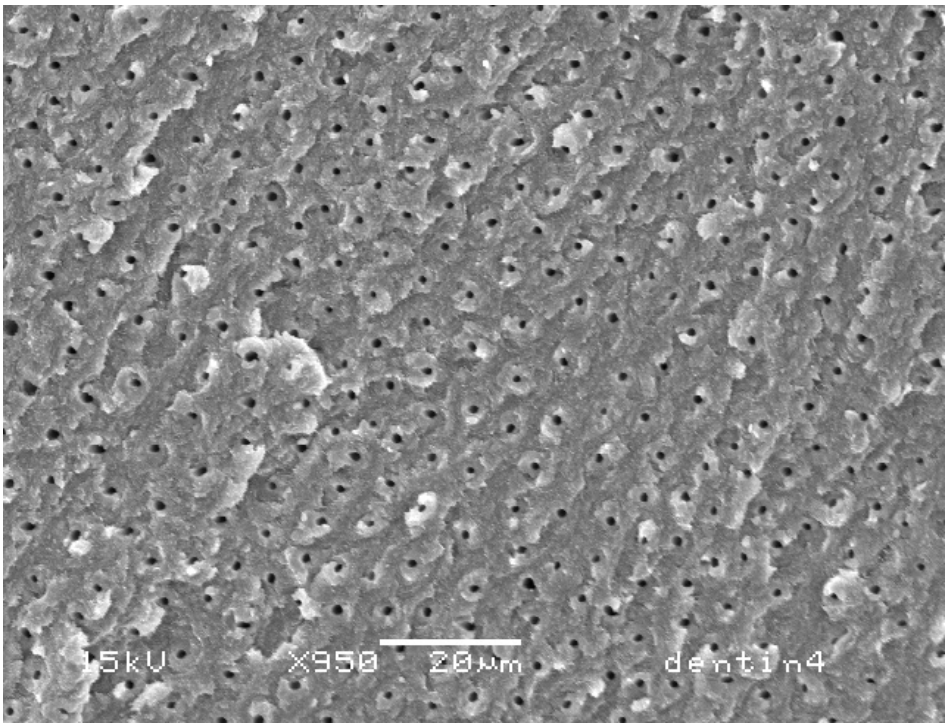
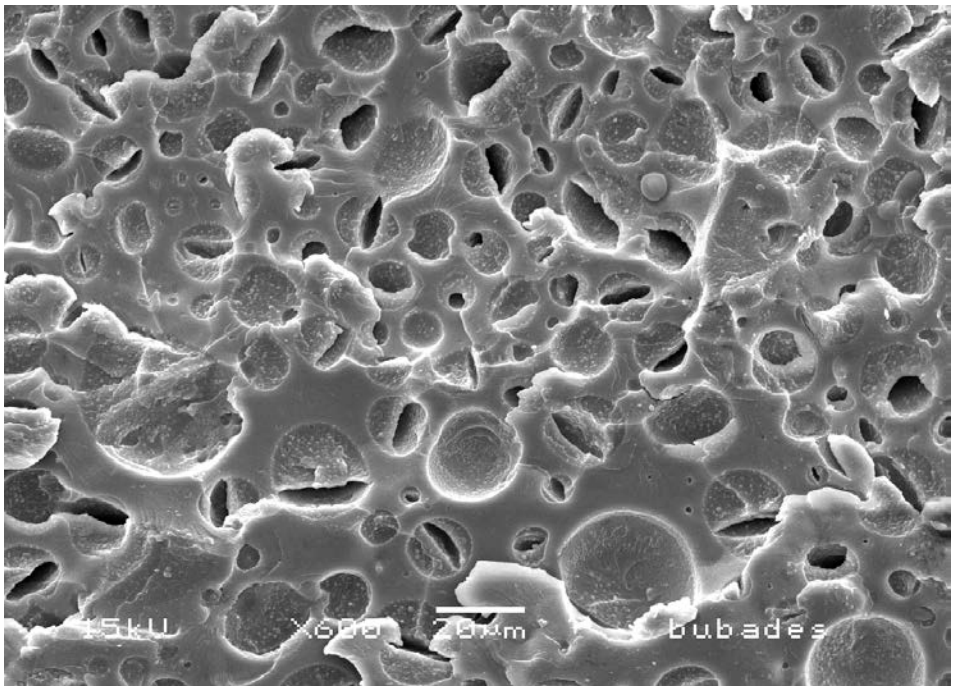


Figure 7



Capítulo 2

Effect of polywave and single-peak LED curing lights and self-etch bond system on the Knoop hardness depth profile of Class I composite restorations

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Summary

Aim: to evaluate the effect of polywave and single-peak LEDs and self-etch adhesive systems on the Knoop hardness of resin composites in Class I restorations. **Methods:** One polywave (UltraLume 5 - UL) and two single-peak (FlashLite 1401 - FL and Radii Cal - RD) light-emitting diodes (LEDs) were evaluated in association with two self-etch bond systems (Clearfil SE Bond - CSE and Clearfil S^3 Bond – S3) in Class I composite restorations. The energy dose of cure was standardized at 22J/cm^2 for composites and 11J/cm^2 for the adhesive. Ninety-six Class I cavities were prepared in human molars (n=16) and restored incrementally using a microhybrid resin composite (Charisma). After finishing and polishing, specimens were bisected in the buccal-lingual direction and submitted to the Knoop microhardness test. The indentations were made on the internal composite surface of each half-crown at different depths (superficial, medium and deep) and the Knoop hardness number of each tooth was taken in each depth. Data were submitted to a two-way ANOVA with subdivided parcels and Tukey test (α=0.05). **Results:** The single-peak LED FL presented similar KHN values compared to UL and RD. There was no difference between the two bond systems evaluated ($p \geq 0.05$). Composite hardness decreases when the distance between the LCU tip end and the material's surface increases, except for FL when the Clearfil SE Bond was used. **Conclusions:** Knoop hardness of the tested composite is dependent of the curing distance. Regardless of the LCU, similar composite hardness was obtained when S3 was applied. When Clearfil SE Bond was used, the single-peak LED FL yielded similar hardness means for all composite depths. The polywave LED promoted higher KHN values in the superficial compared to the other surface depths. The single-peak LED FL yielded higher hardness means for all depths measurements compared to the polywave device.

Keywords: Photoactivation, light curing units, Knoop microhardness, light-emitting diode, self- etch adhesives

Introduction

Resin composites have been widely used in direct aesthetic restorative procedures. However, many factors limit the performance of resin-based materials, especially depth of cure and monomer conversion.[1,2] Due to the fact that most current resin composites are light-activated materials, the curing light needs to pass through the composite mass and yield an adequate degree of conversion.[3,4,5] The light curing unit (LCU) should permit a satisfactory polymerization even in deeper areas rather than only the superficial composite irradiated increments.[2] Consequently, the top composite surface generally is better polymerized than the surfaces distant to the curing light tip end due to light attenuation, dispersion and absorbance when it passes through the composite mass.[6] Therefore, poor polymerized composites can compromise the final mechanical properties of the restorations, decreasing the color stability.[6] and increasing the residual monomers content, which can cause pulpal damage.[7]

In this sense, choose a correct light curing source is important to achieve a satisfactory degree of conversion and consequently, improved mechanical properties, such as hardness.[8,9] Light emitting diodes (LEDs) units has been shown to effectively cure resin-based materials using gallium nitride semiconductors, emitting unfiltered blues light.[10,11,12,13] These LCUs generates a narrow spectral range that targets the absorption wavelength of camphorquinone, with a peak value of 468 nm allowing low amounts of wasted energy and minimum heat generation.[14,15] It was claimed that this could provide higher monomer conversion efficiency and reduce the required exposure time.[16] Studies have shown that current LED curing units can replace, in most situations, the conventional QTH light units.[17,18,19]

Because some esthetic resinous materials do not have camphorquinone as the photoinitiator due to yellowing, polywave LEDs were introduced in the market to overcome

this problem (Camilloti et al., 2008, Brandt et al., 2010; Price et al., 2010). These third generation LEDs emits light wavelength in the visible region, targeting the absorption peak of camphorquinone, however they deliver additional light output at the UV-Vis region of the electromagnetic spectrum (400 - 415 nm).[20,21,22] This secondary short wave violet light may yield the adequate polymerization of resins that contains an alternative photoinitiators found in some specific types of resin-based dental materials, such as phenyl propanedione.[22] It is especially important in the curing procedure of resin composite for bleaching teeth or for value characterization because other photosensitizers are present on material's composition, however, manufacturers do not state all of the photoinitiators systems used in their proprietary products.[5,8]

The interaction with the adhesive system also has a significant influence on the hardness values.[23] Faria e Silva et al. [23] showed that the conventional one-bottle bond system provided higher composite Knoop hardness values compared to a two-step self-etch one, especially when modulated curing methods are used. Hence, adhesive system composition and acidity may influence the degree of conversion of the composite restorative assessed by Knoop microhardness. Most of the previous studies measured composite Knoop hardness outside a tooth cavity, with no adhesive influence of hardness values.[2,21] This may be a limitation of these works because in dental practice, the resin composite would be surrounded by an adhesive layer. So, evaluating whether the composite hardness is affected by the subjacent adhesive layer inside tooth cavities need further investigation.

Despite hardness is considered a measure of material's resistance to bending, scratching and abrasion, it is a reliable method to determine how well a resin composite is cured, and a good correlation between the DC of the resin and Knoop hardness has been

reported.[3,4,24,25,26] A composite resin may be considered well cured when the hardness of the opposing surface is equivalent to at a least 80-90% of the top surface hardness.[27]

Thus, the aim of this study was to investigate the influence of different LED curing units and the self-etching bond systems on the Knoop microhardness depth profile of a microhybrid composite in class I restorations. The first tested hypothesis was that the polywave LED would present higher Knoop hardness values regarding the composite depth. The second hypothesis was that the one-step self-etch bond system would affect the Knoop hardness of the tested composite.

Materials and methods

Light Curing Units (LCUs)

Three light-emmiting diodes (LEDs) were tested: FlashLite 1401 (FL) (Discus Dental, Culver City, CA, USA), Radii-Cal (RD) (SDI Limited, Victoria, Australia) and UltraLume LED 5 (UL) (Ultradent, South Jourdan, Utah, USA). Details of the LCUs used in this study are shown on Table 1. The output power (mW) of each light curing unit (LCU) was measured with a calibrated power meter (Ophir Optronics, Har – Hotzvim, Jerusalem, Israel). Then, irradiance (mW/cm^2) was determined by dividing the output power by the tip end area. Spectral distributions were measured with a calibrated spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA). So, beam distribution and irradiance data were integrated using the Origin 6.0 software (OriginLab Northampton, MA, USA). The LCUs spectrum range distribution is shown on Figure 1. Energy density was standardized to approximately 11J for the bonding system curing and 22J for each composite increment photoactivation. Since the Radii-Cal light source mandatorily operates in ramp mode during 5s, these initial seconds were discarded and further application was realized in

order to maintain the standardized irradiance and consequently equaling the energy dose obtained for each of the units.

Specimen's preparation

Ninety six sound human third molars were selected. This study had the approval of the Ethical Committee of the State University of Campinas, Piracicaba Dental School. The teeth were cleaned, root inserted in polystyrene resin and the occlusal surfaces were wet polished with 320-grit SiC paper under running water (Politriz, AROTEC – São Paulo, SP) to expose a flat enamel surface without exposing dentin. Box-shaped class I cavities were prepared using #56L diamond burs (KG Sorensen, Barueri, SP, Brazil) at high-speed, under air–water cooling. A custom-made preparation device allowed the standardization of the cavity dimensions to 5 mm mesio-distal length, 5 mm bucco-lingual width and 3 mm depth, remaining all cavity margins on enamel substrate. The bur was always replaced after five cavity preparation.

After cavity preparation, teeth were assigned to 6 groups according to the curing light and bond system (3 LEDs x 2 Adhesives). Two self-etch adhesive systems (two-step and one-step) were used for the bonding procedure: Clearfil Tri-S Bond (pH= 2.7; Kuraray Medical Inc., Tokyo Japan) or Clearfil SE Bond (pH = 2.0; Kuraray Medical Inc., Tokyo Japan). The composition, application mode and batch number of the adhesive systems presented in Table 2. All groups were restored with B1-shade Charisma microhybrid composite (Heraeus Kulzer, Hanau, Germany) using incremental oblique technique with increments with approximately 2mm thick. After, the finishing and polishing procedures were performed with medium-, fine-, and extra fine- grit aluminum oxide disks (SoftLex – 3M/ESPE), respectively. After polishing, the specimens were submitted to 200.000 mechanical loading (2Hz) and 500 thermal cycles (ranging from 5 to 55°C with a dwell time

of 60s in each bath with an interval of 5s) at a thermo-mechanical device ER-11000 (ERIOS, São Paulo, Brazil) in order to simulate similar ageing of the composite restorations in the oral environment conditions.

Knoop hardness measurement

Each restored tooth after thermo-mechanical ageing was half-sectioned in buccal-lingual direction using a slow-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA), parallel to the long axis. Each half was embedded in polystyrene resin to facilitate handling and Knoop microhardness measurements. The included halves were wet polished with 600, 1200 and 2000 grit abrasive SiC papers and then polished with diamond pastes (Arotec Ind. Com., São Paulo, Brazil) in a polishing machine under water cooling.

For the Knoop hardness measurement, three indentations were made under 50 g load at 15 seconds in a microhardness tester (HMV-2000, Shimadzu, Japan), at three different depths (superficial, medium and deep). Indentations were made at twelve positions on each half specimen (Figure 2). Lateral indentations were performed at 30 µm from the adhesive layer. For curing depth profile, the readings obtained in the microhardness test were transformed into Knoop Hardness Number (KHN). Data were submitted to a two-way ANOVA with subparcels and Tukey´s test at a pre-set alpha of 0.05.

Results

Statistical significant differences of the KHN values were found for the independent factors curing unit ($p \leq 0.05$) and surface depth ($p \leq 0.05$) and for the triple interaction of

the factors (curing unit x bond system x surface depth) ($p \leq 0.05$). Means and standard deviations of KHN for the studied factors and the interaction are shown on table 3.

The polywave and single-peak LEDs presented similar curing performance, presenting no statistically different in the KHN values. There was no difference between the two bond systems evaluated ($p \geq 0.05$). Composite hardness decreases when the distance between the LCU tip end and the material's surface increases, except for FL when the Clearfil SE Bond was used, presenting similar KHN values (67.7 ± 3.4, 63.8 ± 3.3 and 62.1 ± 4.6 for superficial, medium and deep surfaces respectively).

Discussion

Microhardness measurements have been shown a well correlation with the degree of conversion of resinous materials [4,24,25,28,29] provided that these composites do not have different formulations.[3] In this sense, microhardness was used by some authors [24,25,28,30,31] to study composite cure as a function of depth. LCUs can have the performance evaluated by hardness depth profile.[30,31] The present study used the Knoop hardness depth profile to investigate the curing efficacy of different LEDs in dental confinement at class I restorations, bonded with self-etch bonding systems.

The first hypothesis tested was rejected, since the polywave LCU did not presented higher KHN values in comparison to the single-peak LEDs units for the curing of the microhybrid B1 shade composite. This can be explained by the energy dose that was standardized at 22J/cm^2, despite of the irradiance differences among the LCUs tested. FL presented higher irradiance (1077 mW/cm^2), followed by UL (800 mW/cm^2) and RD (731 mW/cm^2). Due to the fact that RD and UL presented almost similar irradiance values they presented similar KHN values. For FL, the similar KHN values observed in all depths when CSE was used may be explained by the higher irradiance, even if the energy dose was standardized. Thus, higher the emitted irradiance, higher the KHN values for the LCUs

studied. This is in accordance to some studies [32,33,34] that evaluated the effect of light curing irradiance on composite curing, in which higher irradiance values promoted higher composite cure and increased mechanical properties, such as cross-link density, DC, KHN.[32,33,34] In contrast, higher irradiance yields higher composite shrinkage stress and consequently increases the marginal gap formation on the bonding interface.[14,34] Thereby, the secondary beam of the third generation LED did not provide better hardness performance of the B1 shade composite, which should have less yellowing characteristic and had allowed higher light energy transmission through the composite mass.[34] It may be explained due to the UV-Vis LEDs location at the UL light tip. These accessory LEDs are located in each corner of the tip with 11 mm of length. The Class I cavity prepared on the human molar had 5 x 5 mm of area and maybe these accessory LEDs might not have satisfactory irradiated the composite surfaces and promoted the lower KHN values. These fact is not in agreement with a previous study, which found that polywave LEDs promote better composite Knoop hardness values compared to single-peak ones, even when the light curing tip end is located at long distance.[8]

Many studies did not standardize the energy dose, [8,23] which can have influenced the final results of composite properties and did not tested the emitted light, but only the LCUs brands.[8,23] Some LCUs works in ramp and soft-start curing modes, yielding different light output and energy density, compared to LCUs that work in the continuous light.[32,35,36] Modulated light emission influences composite's selected properties, like hardness, cross-linking of the polymer network and wear resistance.[23,32,35,36] In this study, only LCUs irradiance and light characteristics were tested, since the energy dose was the same for all conditions.

In the present work, the self-etch bond systems did not influence the resin composite KHN in the tested conditions, rejecting the second hypothesis. The similar

adhesives pH values may explain the similar hardness values for both self-etch bond system. Acidic pH can decrease resin microhardness, due to the light polymerization inhibition, affecting the composite final cure.[23] However, the results of the present study showed that both adhesive systems (one-step and two-step) promoted similar composite Knoop hardness. Hence, it would be expected that the most hydrophilic adhesive Clearfil S^3 Bond would decrease the Knoop hardness of the tested composite. The aqueous content of the adhesive systems may have not have influenced the composite cure under confinement, at the box-sapped class I cavity, despite some studies state that an oxygen-inhibited layer can decrease composite cure.[37,38] However, the adhesive layer is distant from the majority of the composite mass and its pH did not influence the final hardness evaluation.

Curing depth profile of the composite resin is greatly affected by the light attenuation caused by the distance of the LCU tip end and the material's surface.[21,39] Despite the energy dose emitted at the 0 mm distance from the tip end and the composite surface is considered the corrected one, in the initial composite increments inside a class I cavity do not absorb the maximum energy density due to irradiance reduction.[8,21] The single-peak with high irradiance FL, when the class I was restored with a two-step self-etch bond system could provide an uniform resin composite cure at al depths. It may be explained since high irradiance promotes higher photons penetration through the composite mass, polymerizing the initial increments, even under long distance between the restorative material and the tip end. It also was accomplished by the RD, for superficial and medium depth, compared to the deep composite portion. Therefore, the polywave LED promoted similar deep and medium KHN values, lower than the superficial ones. So, the polywave light spectra did not yielded increased KHN values, showing that for camphorquinone-based composites, the narrower spectrum peaks at approximately 467

nm, could provide better composite cure, compared to the polywave LED studied, which has an additional light peak ranging from 454 nm to 403 nm and can contribute to the final irradiance emission of this LCU.

The composite used in the present study, Charisma (Heraeus Kulzer, Germany), has high inorganic filler volume content (64%) and monomeric system based on a component with high molecular weight (BisGMA), that leads to a low volumetric shrinkage (about 2%) and a satisfactory conversion, according to manufacturer´s information. However, the B1 shade still may present only camphorquinone/amine as the unique photoinitiator system. This might be the reason that the polywave LED did not promoted higher KHN values for this composite. In addition, FL showed higher KHN performance, compared to third generation polywave LCU, due to the narrow spectral range that targets the absorption wavelength of camphorquinone and the higher emitted irradiance.

Clinical trials evaluating the LED efficacy for resin composite polymerization should be encouraged, since the *in vitro* laboratorial conditions are not the same as the presented on clinical practice. Some important cautions should be taken, such as LED periodic light irradiance measurement, in order to guarantee a correct energy dose delivered and some technical attention are needed to guarantee a correct composite polymerization in Class I restorations.

Conclusion

All LCUs promoted similar composite Knoop hardness when the one-step and two-step self-etch adhesives were used. When Clearfil SE Bond was used, the single-peak LED FlashLite 1401 yielded similar hardness means for all composite depths. The polywave LED promoted higher KHN values in the superficial compared to the other surface depths.

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Figure 1. The spectrum range distribution of each LCU used in this study.

Figure 2. Specimen preparation for the Knoop hardness test. (A) Sound human molar. (B) Box-shaped class I cavity on the flat enamel tooth surface. (C) Slice made in the buccal-lingual direction of the restored specimen. (D) Two halves of the restored specimen with the Knoop depth profile indentations designs.

Table 1. Characteristics of the LCUs used in this study.

LCU	Manufacturer	Type	Tip Diameter (mm)	Irradiance (mW/cm^2)	Radiant exposure (J/cm^2)
FlashLite 1401	Discus Dental, Culver City, CA, USA	Single-peak	7	1077	22
Radii Cal	SDI Limited, Victoria, Australia	Single-peak	7	731	22
UltraLume LED 5	Ultradent products Inc, South Jourdan, Utah, USA	Polywave	11 x 7	800	22

Table 2. Composition, application mode and manufacturer's information for the adhesive systems tested.

Adhesive systems	Composition	Application mode
Clearfil SE Bond (Kuraray Medical Inc., Tokyo Japan)	*Primer* (Batch #00896A): water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate. *Adhesive* (Batch #01320A): MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimathacrylate, N,N-diethanol p-toluidine bond, colloidal silica.	Apply Primer for 20s. Mild air stream. Apply Bond. Gentle air stream. Light cure at a energy density of 11J.
Clearfil S^3 Bond (Kuraray Medical Inc., Tokyo, Japan)	*Adhesive* (Batch #00116A): MDP, Bis-GMA, HEMA hydrophobic dimethacrylate, dl-camphorquinone, silanated colloidal silica, ethyl alcohol and water.	Apply the bond system for 20s. Gentle air stream for 10s. Light cure at a energy density of 11J.

Table 3. Means and standard deviation of Knoop hardness number (KHN) for the tested conditions.

Surface depth	FlashLite 1401		Radii Cal		UltraLume 5	
	CSE	CS^3	CSE	CS^3	CSE	CS^3
Superficial	67.7 (3.4) Aa	68.5 (3.9) Aa	65.4 (2.5) Aa	66.9 (3.7) Aa	69.9 (4.9) Aa	67.4 (3.3) Aa
Medium	63.8 (3.3) Aa	59.5 (3.4) Ab	59.5 (4.8) Aab	59.6 (5.0) Ab	58.2 (4.3) Ab	59.6 (3.8) Ab
Deep	62.1 (4.6) Aa	57.9 (3.5) Ab	57.9 (3.5) Ab	55.8 (4.1) Ab	56.0 (5.2) Ab	57.3 (5.8) Ab

Mean values followed by different small letters in the column and capital letters in the row differ statistically among themselves for the Tukey test at the level of 5%. ( ) – Standard Deviation.

Figure 1

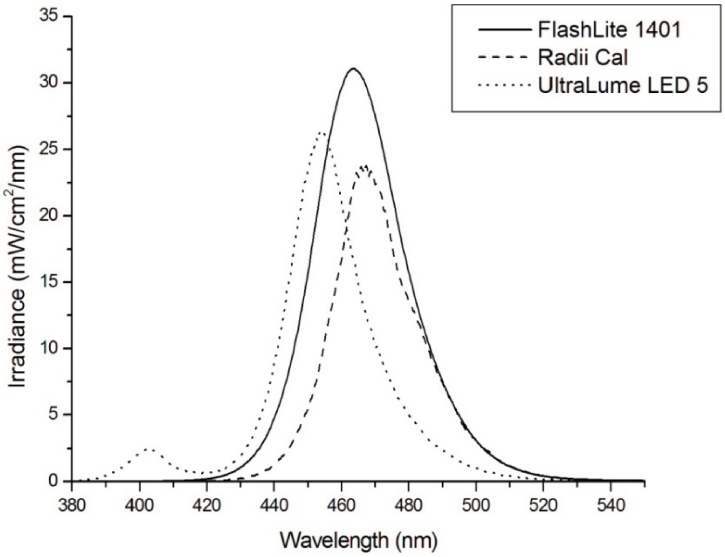
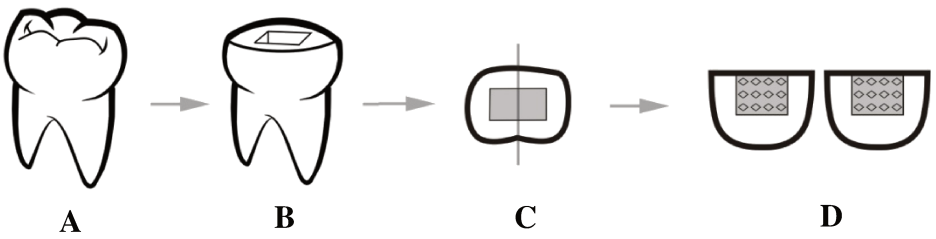


Figure 2



Selective enamel etching: effect on marginal adaptation of self-etch LED-cured bond systems in aged class I composite restorations

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Summary

The aim of this study was to evaluate the influence of previous enamel etch and LED curing unit (LCU) on gap formation of self-etch adhesive systems in Class I composite restorations after thermomechanical ageing (TMA). Thus, on 192 human molars, a box-shaped Class I cavity was prepared maintaining enamel margins. Self-etch adhesives (Clearfil SE – CSE and Clearfil S^3 – S3) were used to restore the preparation with a microhybrid composite. Before adhesives application, half of the teeth were enamel etched for 15s with 37% phosphoric acid; the other half was not etched. For the photoactivation of the adhesives and composite, three light curing units (LCUs) were used: one polywave (UltraLume 5 - UL) and two single-peak (FlashLite 1401 - FL and Radii Cal - RD) LEDs. After this, epoxy resin replicas of the occlusal surface were made and the specimens were submitted to thermomechanical aging. Then, replicas were made again from the aged specimens for marginal adaptation analysis by scanning electron microscopy (SEM). Data were submitted to Kruskal-Wallis and Wilcoxon test (α=0.05). Before TMA, when enamel was etched before the application of S3, no gap formation was observed; however, there were gaps at the interface for the other tested conditions, with statistical difference ($p \leq 0.05$). After TMA, the selective enamel etching previous to S3 application, regardless of the LCU, promoted higher marginal adaptation compared to the other tested groups ($p \leq 0.05$). Prior to TMA, higher marginal integrity was observed, in comparison with specimens after TMA ($p \leq 0.05$). SE and S3 cured with FL, no differences of gap formation were found between before and after ageing (5.3 ± 3.8 and 7.4 ± 7.5 respectively, especially when the S3 was used in the conventional protocol. When cured with RD or UL and was not etched, Clearfil Tri-S presented the higher gap formation. In conclusion, the previous enamel etching promote better marginal integrity for Clearfil Tri-S, showing to be an efficient additional step for class I composite restorations. The two-step

self-etch adhesive was not influenced by the selective enamel etching neither by the LED curing unit.

Keywords: Acid etching, Enamel, Self-etch adhesives, Light curing units, gap formation

Introduction

Composite restorations have been widely used in clinical practice, due esthetic and some biomechanical properties similar to dental hard tissues.[1] For the bonding procedure of these adhesive restorations, self-etching bond systems were introduced in order to decrease the number of bonding technical steps, since the presence of acidic monomers in their composition yields etching and priming simultaneously of the dental hard tissues. [2,3] It promotes a partial removal of smear layer and consequently, the formation of smear plugs, decreasing tooth postoperative sensitivity and leading the adhesive protocol less time-consuming. [3,4,5,6]

Self-etching adhesives are known to exhibit a good bonding performance to dentin and a poor bonding behavior to enamel. [3,4] In order to remove the smear layer created by instrumentation, demineralize the enamel substrate and increase bond quality and durability, a selective enamel phosphoric acid etching before the application of a self-etch adhesive has been proposed. [7,8,9,10,11] Some studies evidenced that this additional enamel etching decreases gap formation when self-etch adhesives are used. [5,6,12,13,14,15,16] However, this additive step for direct composite restoration procedure was only evaluated in Class II, III and V restorations. [5,6,13,14,15,16] Since the Class I preparation exhibits a high C factor configuration and enamel surrounding the superficial margins, it is considered the best model for understanding the real effects of enamel etching and the stress development on the tooth/adhesive interface.[14,17]

An adequate bond system cure is another important fact to consider in restoring class I cavities to ensure a good bond performance and marginal integrity. An efficient light curing unit (LCU) should guarantee a satisfactory adhesive and composite degree of conversion, which may improve their physical and mechanical properties, yielding a good marginal sealing.[18,19,20,21,22] Quartz-tungsten halogen (QTH) lamps have been largely used in restorative procedures; however it presents some drawbacks like bulb, filter and reflector degradation over time and lifetime approximately 40 to 100 hours.[18,19,20] In this sense, light emitting diodes (LEDs) have been shown to overcome these problems, promoting an adequate cure of resin composites and dental adhesives, emitting unfiltered blues light.[18,19,20] These LCUs generate a narrow spectral range that targets the absorption wavelength of camphorquinone, yielding low amounts of wasted energy and minimum heat generation, with higher lifetime and less decrease of light intensity.[18,19,20] Thus, the curing potential of current LED curing units has been shown to be similar of the presented by conventional QTH light units.[23,24,25]

It is known that some resinous materials, like resin cements and some adhesive systems are not well cured with conventional single-peak LEDs due to alterative photoinitiators content.[26] In this sense, LEDs with an additional wavelengths (polywave third generation LEDs) were developed, emitting light wavelengths at the spectra that targets the absorption peak of camphorquinone and another one at the UV-VIS region (400 – 415 nm).[23,24,25] This polywave behavior is expected to yield an adequate cure of adhesives and composites that contain alternative photoinitiators systems, such as phenyl propanedione (PPD), bis-alquyl phosphinic oxide (BAPO) and Trimethylbenzoyl-diphenyl-phosphine Oxide (TPO).[25] Since manufacturers did not state all the photoinitiators content is it important to choose an adequate LCU that would polymerize all resinous

materials,[25,26,27] yielding a great marginal seal, improving the composite restorations' durability.

Thus, this study aimed to investigate the influence of prior enamel etching and LED curing lights on gap formation of self-etch bond systems in class I composite restorations. The first tested hypothesis was that the selective enamel etching would improve the marginal adaptation of the adhesive systems in the Class I composite restorations. The second hypothesis was that the third generation polywave LED would present lower gap formation for the tested bond systems. Moreover, the third hypothesis was that the thermomechanical fatigue would increase interfacial debonding, promoting higher gaps at the superficial margins.

Materials and methods

One hundred and ninety-two healthy human third molars were selected. The teeth were collected after obtaining the patient's informed consent under a protocol approved by the State University of Campinas ethical review board (#057/2009). The teeth were cleaned, included in polystyrene resin and their occlusal surfaces were wet polished with 320-grit SiC paper under running water (Politriz, AROTEC – São Paulo, SP) to expose a flat enamel surface area without exposing dentin. Then, typical Class I cavities were prepared using #56L carbide burs (KG Sorensen, Barueri, SP, Brazil) at high-speed, under air–water cooling. After five cavity preparation, the bur was always replaced. Preparations had a standard size, with cavity dimensions of 5 mm mesio-distally length, 5 mm buccal-orally width and 3 mm deep, remaining all cavity margins on enamel substrate. Two self-etch adhesive systems were used for the bonding protocols: Clearfil Tri-S Bond (One-step self-etch, pH= 2.7; Kuraray Medical Inc., Okayama Jp), Clearfil SE Bond (Two-step self-etch, pH = 2; Kuraray Medical Inc.). The composition, application mode and batch number of the adhesive systems presented in Table 1. After cavity preparation, teeth were

assigned to 12 groups (n=16) according to the three studied factors: selective enamel etching, bond system and curing light (2 conditioning protocols x 2 adhesives x 3 LEDs).

For the photoactivation procedure three light-emmiting diodes (LEDs) were tested: FlashLite 1401 (FL) (Discus Dental, Culver City, CA, USA), Radii-Cal (RD) (SDI Limited, Victoria, Australia) and UltraLume LED 5 (UL) (Ultradent, South Jourdan, Utah, USA). Prior to the restorative procedure, the output power (mW) of each light curing unit (LCU) was measured with a calibrated power meter (Ophir Optronics, Har – Hotzvim, Jerusalem, Israel). Then, irradiance (mW/cm^2) was determined by dividing the output power by the tip end area. Spectral distributions were measured with a calibrated spectrometer (USB2000, Ocean Optics, Dunedin, FL, USA). So, beam distribution and irradiance data were integrated using the Origin 6.0 software (OriginLab Northampton, MA, USA). The spectrum range distribution of each LCU is shown on figure 1 and the characteristics of the LCUs are presented in Table 2. Also, Energy density was standardized to approximately 11J for the bonding system curing and 22J for each composite increment photoactivation. For RD, due to the fact that it mandatorily operates in ramp mode for 5s, these initial seconds were discarded and a continuous light was delivered in order to keep the irradiance standardized, consequently equaling the energy density obtained for each of the LCUs.

All groups were restored with B1-shade Charisma microhybrid composite (Heraeus Kulzer, Hanau, Germany) using an incremental oblique technique with 6 increments, of approximately 2mm thick. The first layer was applied horizontally and light cured, followed by two oblique layers. Next, another 3 layers were placed in the same way as described before, until the cavity was completely filled. Then, the finishing and polishing procedures were performed with medium-, fine-, and extra fine- grit aluminum oxide disks (SoftLex – 3M/ESPE), respectively. After polishing, impressions with a low viscosity vinyl polysiloxane material (Express XT, 3M ESPE, St Paul, MN, USA) of the teeth were taken and a first set

of epoxy resin replicas (Epoxicure Resin, Buehler, Lake Bluff, IL, USA) was made for SEM evaluation. In sequence, specimens were submitted to 200.000 mechanical loading (2Hz) and 500 thermal cycles (ranging from 5 to 55°C with a dwell time of 60s in each bath with an interval of 5s) at a thermo-mechanical device ER-11000 (ERIOS, São Paulo, Brazil) in order to simulate similar ageing ofto the composite restorations in oral environment conditions.

Further, new impressions of the teeth were made and another set of replicas was made for each restoration. All replicas were mounted on aluminum stubs, sputter coated with gold and were evaluated with a scanning electron microscope (JEOL, JSM-5600LV, Scanning Electron Microscope, Japan) as before at 200X magnification. SEM analysis of the composite/enamel marginal adaptation was performed by one operator having experience with quantitative margin examination and was blinded to the restorative procedures. The marginal integrity between resin composite and enamel was expressed as a percentage of the entire superficial margin length.

Enamel Etching patterns

Eighteen half-teeth (n = 3 for the two self-etch adhesives with and without prior enamel etch and negative and positive controls) were grounded and randomly assigned into 12 groups. For the positive control a 37% phosphoric acid treatment was realized and for the negative, the enamel surface did not receive any treatment. The experimental groups were treated with the two tested bond systems using manufacturer's information with or without the prior enamel etching. Then, the treated surfaces with the bond systems were thoroughly rinsed with alternate baths of acetone (20s) and ethanol (20s), in an attempt to remove the self-etch primers and the monomer components. All specimens were dehydrated in ascending grades of ethanol (25%, 50%, 75%, 90%) for 10 min each

and immersion in 100% ethanol for 30 min. After storage at 37°C for 24 hours, specimens were sputter coated with gold and analyzed by SEM.

Statistical analysis

For the statistical analysis, as the data did not exhibit normal data distribution (Kolmogorov-Smirnov test), nonparametric tests were used (Kruskal-Wallis for groups' comparison and Wilcoxon matched-pairs signed-ranks test for pairwise comparisons before and after thermomechanical ageing) with a pre-set alpha of 0.05.

Results

Marginal adaptation analysis

The results of the marginal adaptation analysis are shown on table 3. Before the termomechanical loading, when enamel was etched before the application of Clearfil Tri-S, no gap formation was observed; however, there were gaps at the interface of the Clearfil SE Bond regardless the energy source, with statistical difference (Kruskal Wallis test; $p \leq 0.05$). After ageing, also the selective etching previous to the Clearfil Tri-S application, when RD or UL were used, promoted higher marginal adaptation compared to the other tested groups (Kruskal Wallis test; $p \leq 0.05$).

Prior to thermomechanical loading, higher marginal integrity was observed, in comparison with after ageing (Wilcoxon test; $p \leq 0.05$). Also, when the bonding systems were cured with FL, only when Clearfil Tri-S was used in the conventional way there were no differences between the restoration before and after ageing (5.3 ± 3.8 and 7.4 ± 7.5 respectively). When cured with RD or UL and was not etched, Clearfil Tri-S presented the higher gap formation.

Enamel etching patterns

The SEM enamel etching patterns are show in Figures 3 (a, b) and 4 (a, b) and 5 (a, b, c).

Discussion

An important factor to promote clinical success of Class I resin composite restorations is a satisfactory enamel marginal adaptation. The presence of gaps is considered as the first sign of restoration failure, clinically evidenced by marginal staining.[28] Also, when detectable marginal disrupting is presented, these interface defects could lead to interfacial leakage.[28,29] In the oral environment, many pulpal sensitivities and responses are related when bacterial leakage occurs along the tooth/composite bonding interface.

In the present work, the first hypothesis was partially validated, since only the gap formation for the one bottle all-in-one self-etch adhesive Clearfil Tri-S was affected by the selective enamel etching additional procedure, regardless of the LCU. It may be explained by some characteristics of this mild one-step adhesive, like its pH and etching potential.[3,4] As showed by the enamel etching patterns evaluation by SEM, Clearfil Tri-S alone promotes a smooth enamel demineralization, not increasing the free energy surface and bond penetration. It's pH of approximately 2.6 could not promote an adequate demineralization of enamel surface, and consequently, a poor bond strength. This weak bond interface is easily affected by composite shrinkage stress during the photocuring procedure.[7,8,28] In this sense, the selective enamel etch procedure could have promoted a deeper dissolution of prisms cores and boundaries in a etching patter type III, increasing the free energy surface of this substrate and consequently, increasing the percentual of gap free margins.

Therefore, for Clearfil SE Bond, the previous acid etching did not affect the gap formation of the composite restorations. It may be occurred by the lower pH of the adhesive system, approximately to 2.0, which could have promoted higher enamel demineralization compared to Clearfil Tri-S alone, regarding the curling light. Also, the

studied two-step bond system contains a separate hydrophobic resin that is applied after the acidic primer. This hydrophobic resin coat can improve bond durability, especially due to the structural polymer network that is not hydrophilic and can maintain optimal bonding behaviour after fatigue stress.[8,30]

The second hypothesis of this study was rejected because the polywave third-generation LED did not improve the marginal adaptation of the resin composite restorations. The photoinitiator's content present on composite and adhesive resin formulations seems to be camphorquinone, as informed by manufacturer's. So, the polywave LED for camphorquinone based resin exhibit a similar behaviour as like the single-peak second generation LEDs. This may be explained due to the fact that both adhesive systems and the microhybrid composite present camphorquinone and did not have the interfacial integrity affected. Also, they may have presented a similar degree of cure and consequently, less marginal shrinkage stress, preserving the superficial marginal adaptation between composite and enamel.

Another fact to discuss is the morphology of the UltraLume 5 tip, in which there is a central LED that emits visible light at the peak spectra of camphorquinone and four accessory LEDs in the corner of the tip that emits UV-VIS wavelengths. In a class I preparation, with 5 mm^2, as the condition presented in this work, light emitted by the accessory LEDs would not reach the adhesive resin since the tip dimensions are higher than the tooth preparation one.[24] Consequently, only the central LED with absorption peak of camphorquinone from UltraLume may have irradiated the adhesive resin, promoting similar gap formation as FlashLite and Radii Cal. These fact is not in agreement with a previous study, which found that polywave LEDs promote better resin mechanical properties compared to single-peak ones, even when the light curing tip end is located at long distance. [22]

For half of the tested groups the thermomechanical loading promoted higher gap formation, partially validating the third hypothesis. When specimens were restored using Clearfil Tri-S without selective enamel etching, the thermomechanical ageing promoted less marginal integrity, except when FL was used for photopolymerization. The previous enamel etch may have guaranteed a higher bond performance due to the increase in the enamel free energy surface caused by a deeper enamel demineralization.[4,5,14] The etching pattern of Clearfil Tri-S with previous acid treatment shows a deeper demineralization of enamel cores and boundaries, favoring the penetration of the adhesive resin. Consequently, the thermomechanical effect was not capable to induce higher gaps at the interface, preserving the enamel/composite bonding. For FL, the ageing may not have affected bonding to enamel because this LCU emitted higher light irradiance compared to the other curing devices, even if the energy dose was standardized. This higher irradiance may have cause a bond disrupting at the interface both before and after thermomechanical fatigue. [32,33,34]

Therefore, for Clearfil SE, Ultralume curing light promoted the maintenance of the marginal integrity even after the thermomechanical ageing, regardless of the additional enamel etching. It may be explained by the hydrophobic layer of this bond system, which may have not been influenced by the composite shrinkage stress and consequently maintained the bond interface with no alterations. This additional layer can promote a higher monomer conversion and allow a marginal integrity after thermomechanical loading. Also, when RD was used with enamel etching, the ageing loading did not influence the percentual of marginal gaps.

Although some in vitro studies exist, final conclusions regarding the role of the enamel selective acid etching for self-etch adhesives in Class I composite restorations will depend on the outcomes of clinical trials. Clinical long-term studies and investigations of

this approach's retention ability for bond systems in the oral environment can best evaluate the quality and durability of these restorations.

Conclusion

The previous enamel etching promotes better marginal integrity for Clearfil Tri-S, showing to be an efficient additional step for class I composite restorations. The two-step self-etch adhesive was not influenced by the selective enamel etching neither by the LED curing unit. In general, the mild one-step self-etch bond system preserved the marginal adaptation integrity when enamel was previous etched, except when the single-peak LED FlashLite, with higher irradiance, was used.

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LEGENDS

Figure 1. The spectrum range distribution of each LCU used in this study.

Figure 2. A – The white arrows point interfacial gap formed between resin composite and enamel. B – The white arrows indicate a perfect marginal seal between enamel and composite. C - Note the composite/resin interface presenting some interfacial gaps. The white arrows point the correct location of the marginal gaps. D – A high magnification, showing the interfacial gaps between composite and dental enamel.

Figure 3. A – SEM photomicrography showing the dental enamel without any acid treatment. B – 37% phosphoric acid etched enamel, showing an etching pattern type II, with the dissolution of the prisms cores.

Figure 4. A – SEM photomicrography showing the smooth enamel etching promoted by Clearfil Tri-S, with no dissolution of prisms cores and boundaries. B – SEM photomicrography showing the smooth primer etching of Clearfil SE, with a etching pattern type I, with only dissolution of prisms boundaries.

Figure 5. A - SEM photomicrography of Clearfil Tri-S with previous phosphoric acid etching, showing a deep dissolution of prisms cores and boundaries, with an etching pattern type III (characteristics of the type I and II). The white arrows point the characteristics of etching pattern type I, with intact prism core, while the black arrows show characteristics of the etching pattern type II, with higher dissolution of prisms peripheries. B – SEM photomicrography of Clearfil Tri-S with higher magnification 5000x. C – SEM photomicrography of Clearfil SE with previous acid etching, with an etching pattern type III, pointed by the white arrows.

Table 1. Composition, application mode and manufacturer's information for the adhesive systems tested.

Adhesive systems	Composition	Application mode
Clearfil SE Bond (Kuraray Medical Inc., Tokyo Japan)	*Primer* (Batch #00896A): water, MDP, HEMA, camphorquinone, hydrophilic dimethacrylate. *Adhesive* (Batch #01320A): MDP, bis-GMA, HEMA, camphorquinone, hydrophobic dimathacrylate, N,N-diethanol p-toluidine bond, colloidal silica.	Apply Primer for 20s. Mild air stream. Apply Bond. Gentle air stream. Light cure at a energy density of 11J.
Clearfil S^3 Bond (Kuraray Medical Inc., Tokyo, Japan)	*Adhesive* (Batch #00116A): MDP, Bis-GMA, HEMA hydrophobic dimethacrylate, dl-camphorquinone, silanated colloidal silica, ethyl alcohol and water.	Apply the bond system for 20s. Gentle air stream for 10s. Light cure at a energy density of 11J.

Table 2. Characteristics of the LCUs used in this study.

LCU	Manufacturer	Type	Tip Diameter (mm)	Irradiance (mW/cm^2)	Composite Radiant exposure (J/cm^2)	Adhesive Radiant exposure (J/cm^2)
FlashLite 1401	Discus Dental, Culver City, CA, USA	Single-peak	7	1077	22	11
Radii Cal	SDI Limited, Victoria, Australia	Single-peak	7	731	22	11
UltraLume LED 5	Ultradent products Inc, South Jourdan, Utah, USA	Polywave	11 x 7	800	22	11

Table 3. Results of the gap formation analysis (% - SD) of enamel margins before and after thermomechanical ageing (TMA)

Tested conditions	Before ageing (%)	After ageing (%)
Flashlite/no etch/Clearfil SE	2.6 (4.7)B	5.6 (4.5)AB*
Flashlite/etch/Clearfil SE	0.6 (1.3)B	3.0 (2.8)AB*
Flashlite/no etch/Tri-S	5.3 (3.8)B	7.4 (7.5)AB
Flashlite/etch/Tri-S	0A	2.4 (4.9)A*
Radii Cal/no etch/Clearfil SE	3.2 (3.9)B	13.1 (14.7)AB*
Radii Cal/etch/Clearfil SE	3.6 (8.2)B	6.2 (8.6)AB
Radii Cal/no etch/Tri-S	3.0 (3.1)B	12.4 (10.7)B*
Radii Cal/etch/Tri-S	0A	2.9 (5.7)A
UltraLume/no etch/Clearfil SE	3.5 (4.0)B	3.9 (4.4)AB
UltraLume/etch/ClearfilSE	3.7 (3.7)B	4.8 (4.9)AB
UltraLume/no etch/Tri-S	3.1 (4.3)B	8.6 (7.5)B*
UltraLume/etch/Tri-S	0A	0.9 (2.61)A

Same letters within column indicate no statistically significant difference ($p \leq 0.05$, Kruskal Wallis and Dunn comparison). Asterisks stand for $p \leq 0.05$; Wilcoxon matched-pairs signed-rank test.

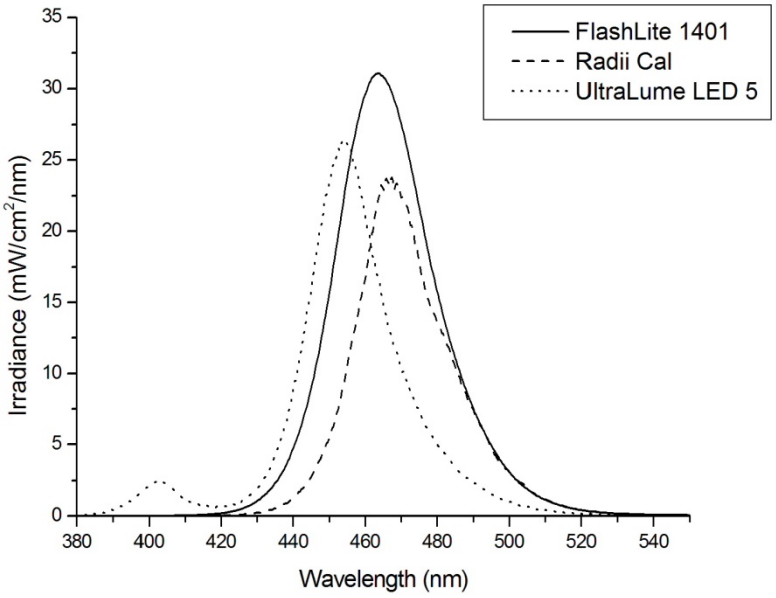
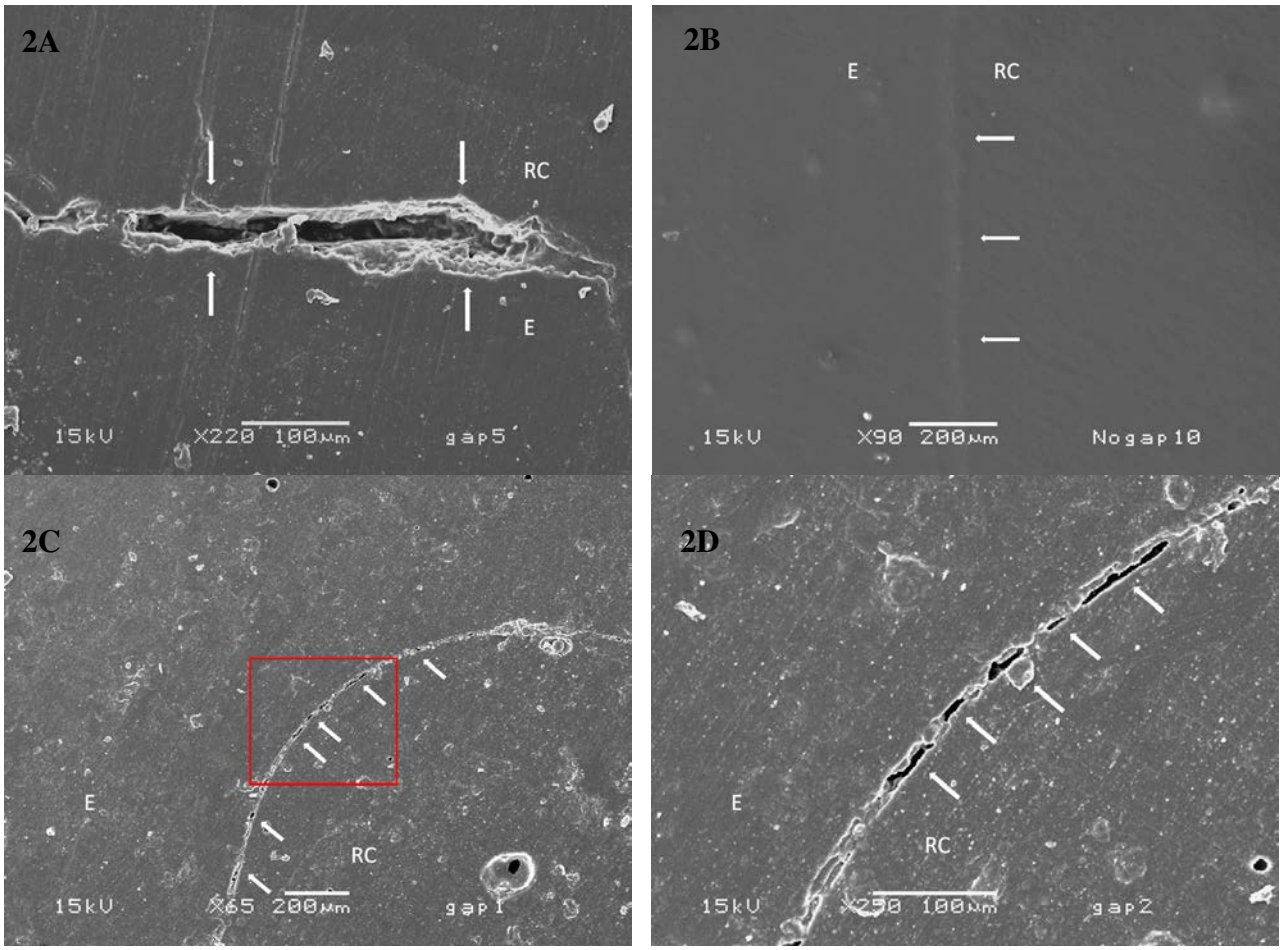
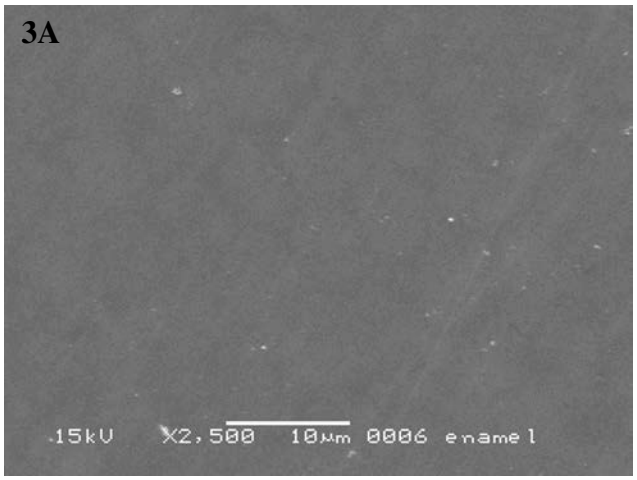


Figure 1. Spectra of the LED light curing units used.

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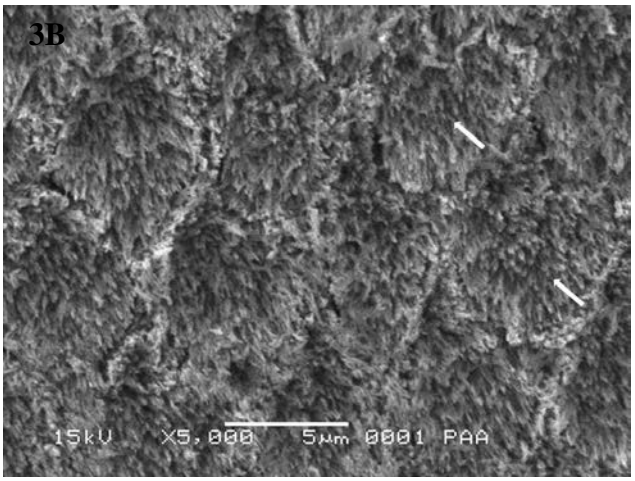
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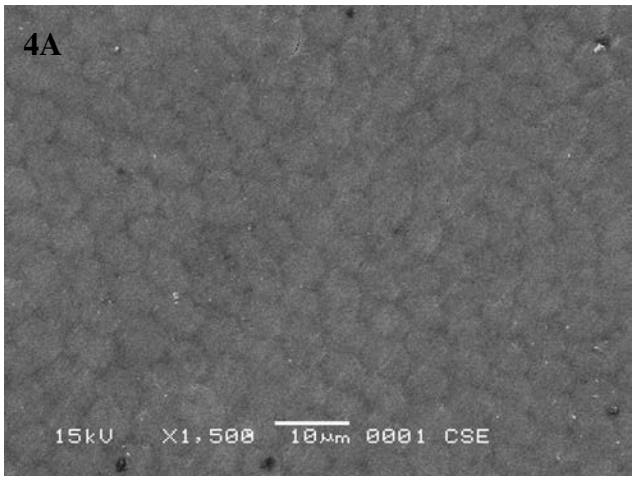
4A
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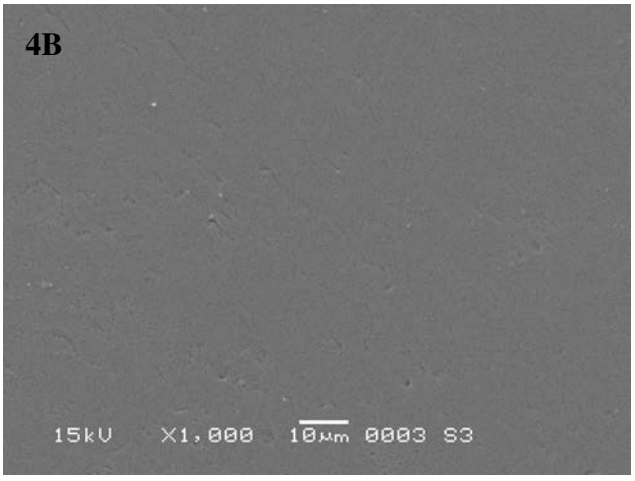
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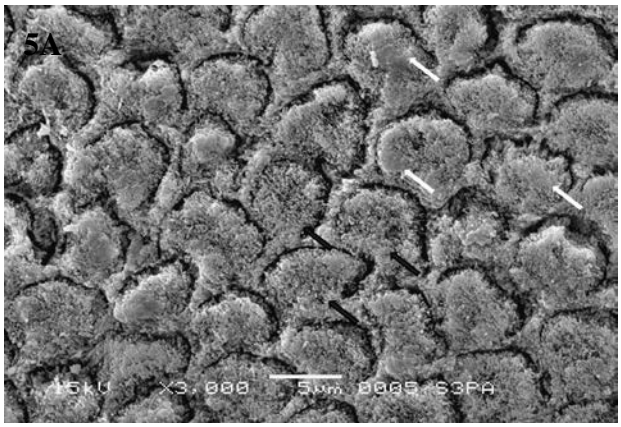
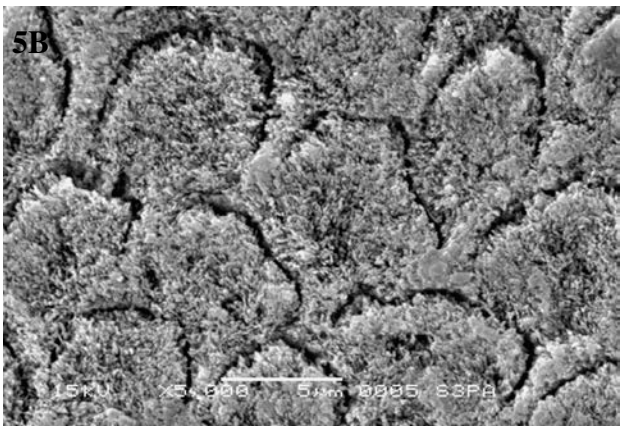
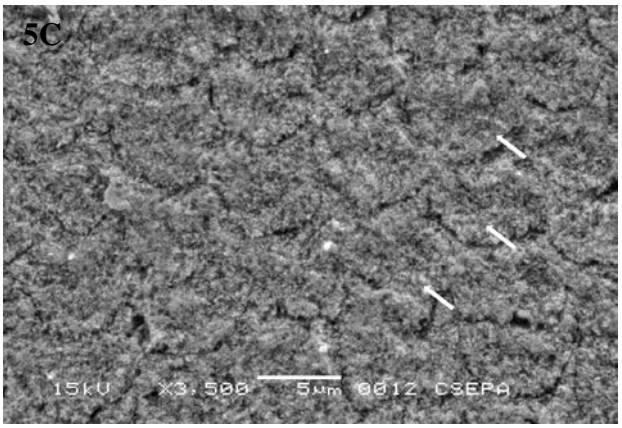

5A
15kV X3,000 5µm 0009 S3PA


5B
15kV X5,000 5µm 0005 S3PA


5C
15kV X3,500 5µm 0012 CSEPA

Conclusão Geral

De acordo com as condições experimentais estudadas e baseado nos resultados encontrados foi possível concluir que:

- O condicionamento ácido seletivo do esmalte, em restaurações Classe I em compósito, influenciou negativamente a resistência à união em dentina para o adesivo autocondicionante Clearfil S^3 Bond, porém, para o Clearfil SE Bond, o condicionamento prévio do esmalte não alterou a performance de união em dentina.
- A fotoativação com LED de terceira geração UltraLume LED 5 acarretou maiores valores de resistência à união para o Clearfil SE Bond, comparado ao LED de pico único. Adicionalmente, o adesivo autocondicionante Clearfil S^3 Bond não mostrou diferença estatisticamente significante dos valores de adesão, independente da fonte de luz LED utilizada.
- Em geral as fontes LED testadas proporcionaram ao compósito maior dureza na região superficial, não havendo diferença entre as profundidades média e profunda. Com exceção do FlashLite 1401 quando usado para ativar o adesivo Clearfil SE Bond, que apresentou dureza uniforme para o compósito em todas as profundidades estudadas.
- A fotoativação com as fontes LED testadas promoveu valores de dureza Knoop semelhante para a resina composta quando ambos os adesivos foram utilizados.
- O condicionamento seletivo do esmalte promoveu menor formação de fendas após envelhecimento da restauração por ciclagem termomecânica

para o adesivo Clearfil S3 bond, exceto quando foi utilizado o LED de pico único FlashLite.

- A adaptação marginal da restauração classe I quando utilizado o adesivo autocondicionante de dois passos Clearfil SE bond não foi influenciada nem pelo condicionamento prévio do esmalte, nem pela fonte de luz LED utilizada.

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Apêndice

Materiais e métodos referentes ao capítulo 1

1.Materiais

Foram utilizados 2 sistemas adesivos autocondicionantes (Clearfil S^3 Bond, Clearfil SE Bond) (Figuras 1a e 1b), 1 resina composta microhíbrida (Charisma) (Figura 2), um ácido fosfórico a 37% (Condac 37 – FGM – Figura 3) e 3 fontes de luz LED (Ultra-Lume 5, Radii-Cal e Flash Lite 1401) (Figuras 4a, 4b e 4c).

Tabela 1. Nome Comercial, composição e fabricante dos sistemas adesivos e compósito utilizados nas restaurações de cavidades classe I.

Nome Comercial	Composição	Fabricante
Clearfil S^3 Bond	2-Hidroxietil Metacrilato (20-30%), Etanol (<20%), Bisfenol A Diglicidilmetacrilato, 10-Metacriloiloxidecil dihidrogenado fosfatase,Sílica Coloidal, D1-Canforoquinona, Água, Iniciadores, Aceleradores, Outros, pH: 2,7	Kuraray Medical Inc. Okayama, JP
Clearfil SE Bond	Primer: 10-Metacriloiloxidecil dihidrogenado fosfatase (MDP), HEMA, Dimetacrilato Hidrofílico, Canforoquinona, Amina terciária, Água. Bond: HEMA, 10-Metacriloiloxidecil dihidrogenado fosfatase (MDP), Bis-GMA, Dimetacrilato Hidrofílico, Amina terciária, Sílica Coloidal silanizada, Canforoquinona. pH:1.8	Kuraray Medical Inc. Okayama, JP
Charisma		Heraeaus Kulzer

	Vidro Silicato de Flúor-Alumina-Boro-Bário, sílica coloidal, Bis-GMA, TEGDMA	

Tabela 2. Nome Comercial, características e fabricante das fontes de luz LED que serão utilizados nas restaurações de cavidades classe I.

Material Comercial	Descrição	Densidade de Potência	Fabricante
Ultra-Lume 5	Aparelho fotoativador LED, com fio, opera na eletricidade. Apresenta abertura de saída de 11x7mm. Possui lâmpadas com onda infravermelho. Não utiliza ponteira condutora. LED de terceira geração.	800 mW/cm^2	Ultradent
Radii-Cal	Aparelho fotoativador LED, sem fio, opera com bateria de lítio. Não utiliza ponteira condutora. Apresenta protetor descartável com abertura de saida de 7mm. LED de segunda geração.	731 mW/cm^2	SDI Limited
Flash Lite 1401	Aparelho fotoativador LED, sem fio, opera com bateria de lítio. Não utiliza ponteira condutora. Radiômetro imbutido Apresenta protetor descartável com abertura de saida de 7mm. LED de segunda geração.	1.077 mW/cm^2	Discus Dental

Figura 1. Sistemas adesivos autocondicionantes.

Figura 2. Compósito Charisma

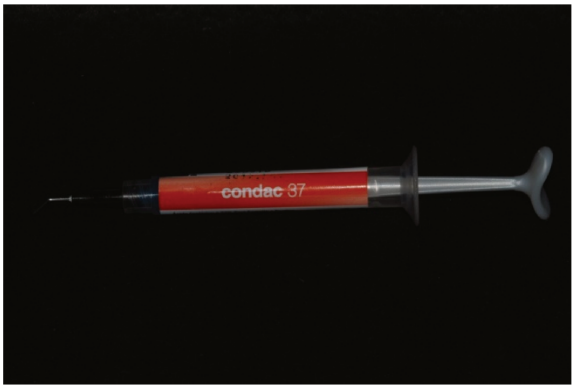
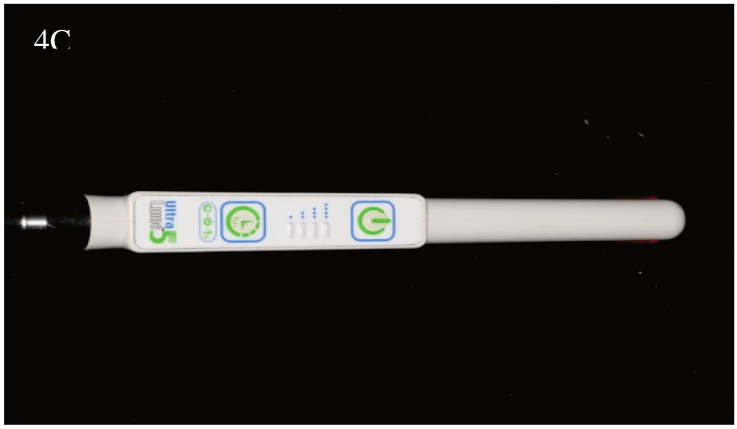


Figura 3. Ácido fosfórico

Figura 4. Fotopolimerizadores LEDs utilizados no estudo.



2. Método

2.1 Seleção e inclusão dos dentes

Foram utilizados 96 terceiros molares humanos hígidos (Figura 5) após aprovação no Comitê de Ética em Pesquisa da Faculdade de Odontologia de Piracicaba, Universidade Estadual de Campinas, que após a coleta foram armazenados em solução aquosa de timol a 0,1%, tamponado. Os debris foram manualmente removidos com lâmina de bisturi e polidos com taças de borracha e pasta de pedra-pomes e água. Após este procedimento, os dentes foram armazenados em água destilada até o momento de sua utilização.

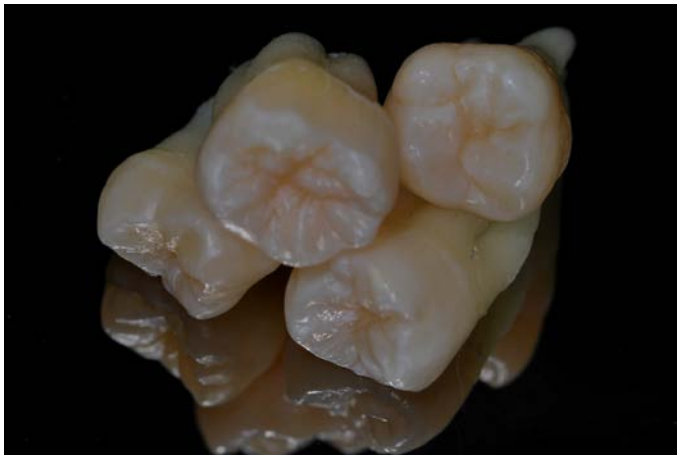
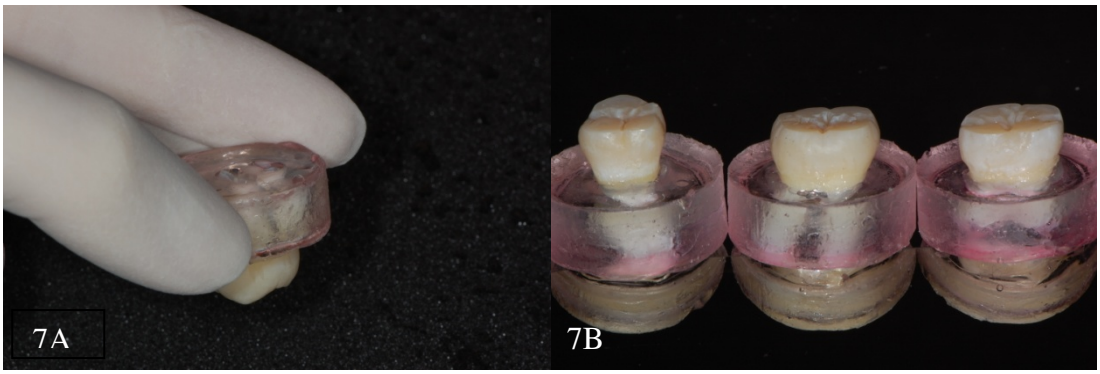


Figura 5. Molares humanos hígidos.

Em seguida, os dentes foram posicionados pela raiz em cera utilidade dentro de matrizes feitas de tubo de PVC medindo 2,5 cm de diâmetro interno e 1 cm de altura, para inclusão em resina de poliestileno, facilitando o preparo cavitário (Figura 6).

Figura 6. Dente sendo incluído em resina de poliestireno.

Previamente à confecção das restaurações, os dentes foram levados em lixadeira (Politriz, AROTEC – São Paulo, SP) para redução das cúspides tomando-se o cuidado para não expor a dentina subjacente (Figura 7 A, B).



Ao final do desgaste das cúspides, foram confeccionadas cavidades Classe I, com broca 56L (KG Sorensen) (Figura 8), sob constante refrigeração e em alta rotação, em máquina padronizadora de preparos. As cavidades possuíam as medidas: 5 mm mésio-distal, 4 mm vestíbulo-lingual e 3 mm de profundidade com o ângulo cavosuperficial em esmalte.

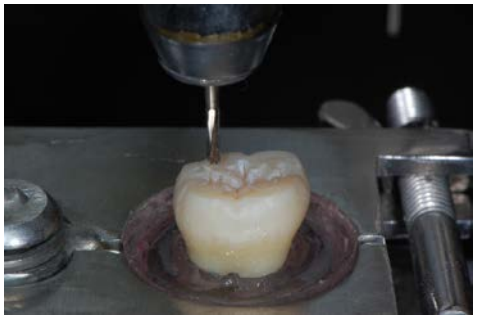


Figura 8. Broca em posição para o início da confecção da cavidade.

Após o preparo cavitário, os dentes foram divididos nos devidos grupos experimentais por sorteio:

- Grupo 1 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Ultra-Lume 5;

- Grupo 2 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Radii-Cal;

- Grupo 3 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Flash Lite 1401;

- Grupo 4 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Ultra-Lume 5;

- Grupo 5 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Radii-Cal;

- Grupo 6 - Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Flash Lite 1401;

- Grupo 7 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%;

Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Ultra-Lume 5.

- Grupo 8 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Radii-Cal;

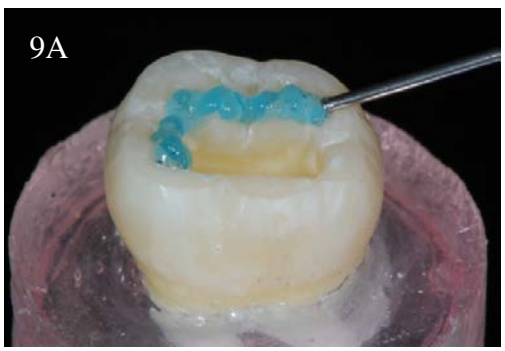
- Grupo 9 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Flash Lite 1401

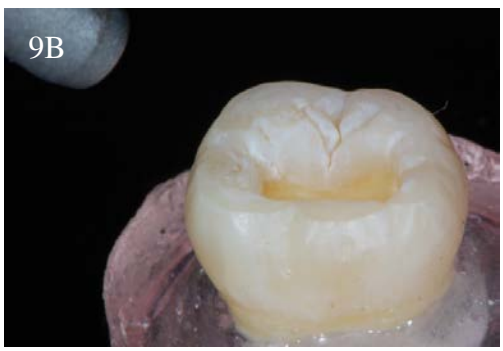
- Grupo 10 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Ultra-Lume 5.

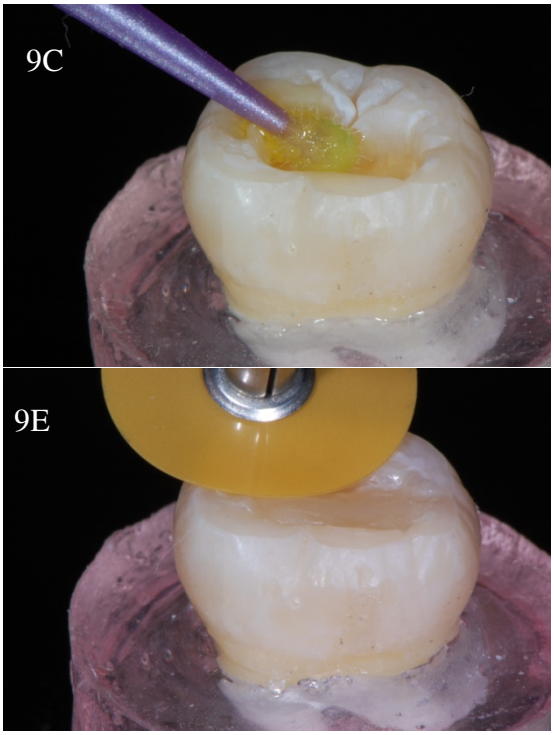
- Grupo 11 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Radii-Cal.

- Grupo 12 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Flash Lite 1401.

2.2 Confecção das restaurações







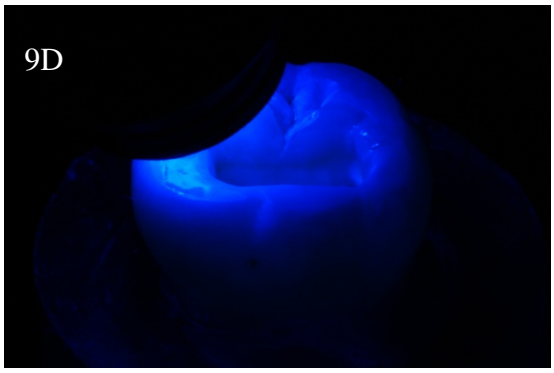


Figura 9. Processo restaurador com o condicionamento seletivo do esmalte (A), secagem (B) aplicação e fotoativação do adesivo (C e D) e polimento da restauração (E).

Para se restaurar essas cavidades foram utilizados o compósito Charisma cor B1 e 2 sistemas adesivos autocondicionantes: Clearfil S^3 Bond e Clearfil SE Bond (Figura 9).

2.2.1 – Condicionamento ácido

Para os grupos que receberam o condicionamento do esmalte cavosuperficial, foi aplicado ácido fosfórico a 37% por 30 segundos, e posterior lavagem por 30 segundos com água. Já para os grupos sem o condicionamento do esmalte, o condicionamento foi realizado pelos próprios constituintes do sistema adesivo aplicado durante o tempo determinado por cada fabricante.

2.2.2 – Aplicação dos adesivos

1) Clearfil S^3 Bond

Secou-se a dentina com suave jato de ar e posterior aplicação ativa do adesivo no esmalte e dentina por 20 segundos. Após, realizou-se secagem com suave jato de ar e fotoativação.

2) Clearfil SE Bond

Secou-se a dentina com suave jato de ar e posterior aplicação de 2 camadas do primer por 20 segundos em esmalte e dentina. Após secagem com jato de ar, 1 camada do adesivo foi aplicada ativamente por 20 segundos e polimerizado por 10 segundos.

2.2.3 – Fotoativação

A intesidade luminosa foi aferida com um radiômetro previamente à confecção das restaurações. Foi utilizado o método de fotopolimerização com luz contínua em todos os aparelhos de luz LED. Para o Radii obter luz contínua, foram dispensados os 5 primeiros segundos de ativação luminosa, já que este aparelho inicialmente opera no modo rampante de emissão luminosa.

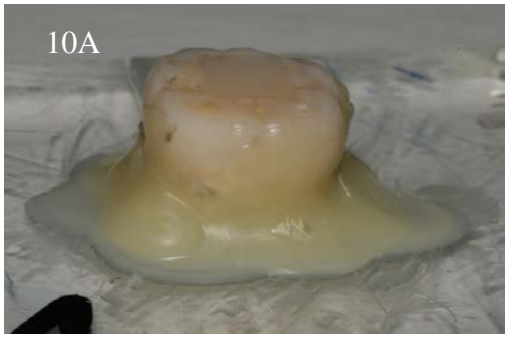
2.2.4 – Inserção e fotopolimerização do compósito

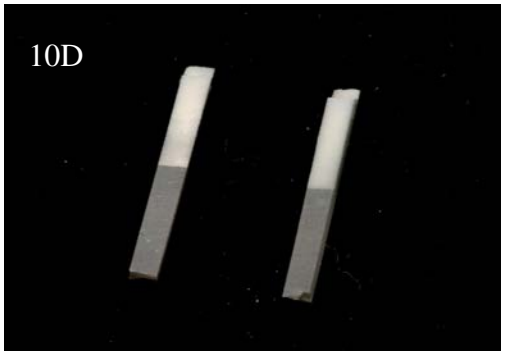
O compósito Charisma (Heraeaus Kulzer) foi inserido em seis incrementos de no máximo 2 mm na cavidade confeccionada. A resina foi fotoativada com densidade energética padronizada de 22 J/cm^2. Após isso, os corpos-de-prova foram armazenados em água destilada a 37°C por 24 horas para a realização do polimento com discos Sof-Lex (3M Espe) nas granulações fina e extra-fina.

Após, as amostras foram submetidas à fadiga termo-mecânica, com 200.000 ciclos mecânicos e 500 ciclos térmicos com banhos entre 5 e 55°C ±1°C, com 60 segundos em cada banho em máquina ER-11000 (ERIOS, São Paulo, Brazil).

Ensaio de microtração

Cada dente foi posicionado em uma cortadera metalográfica de precisão, utilizando disco de alta concentração de diamante (Buehler, São Paulo, BR) realizando, cinco cortes no sentido mésio-distal, e após o reposicionamento do dente, foram realizados mais quatro cortes no sentido vestíbulo-ligual paralelos ao longo eixo do mesmo, para se obter paraleleptidos (palitos) de aproximadamente 0,9 x 0,9 mm^2, os quais foram fixados com cola de cianoacrilato (Super Bond gel, loctite, Henkel, Brasil) ao aparelho de microtração fixo à Máquina de ensaio universal (Emic Equipamentos e Sistemas de Ensaio LTDA, São José dos Pinhais. PR - Brasil). O ensaio foi conduzido com uma célula de carga de 5 Kg, a uma velocidade de 0,5 mm/min, até a ruptura da amostra. Foram anotados os valores de resistência máxima fornecidos pela máquina em kgf. Depois de calculada a área da interface de união em cada palito com auxílio de um paquímetro digital (Mitutoyo, Japão), os valores de resistência de união foram convertidos em MPa através da fórmula: MPa = kgF * 9,8 (constante)/ área (expressa em mm^2). (Figura 10)





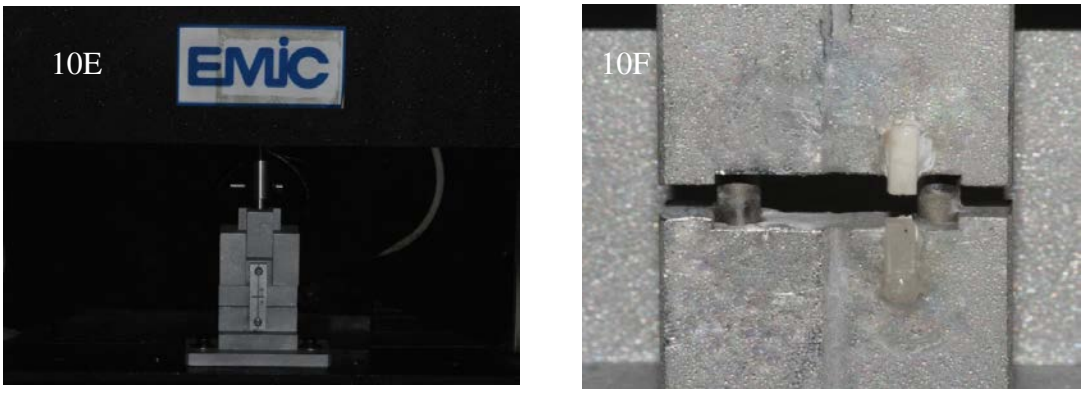


Figura 10. A) Amostra fixada na placa de acrílico. B) Espécime sendo seccionado em cortadeira metalográfica. C) Primeiros cortes para a confecção dos palitos. D) Palitos da interface resina/dentina obtidos para o ensaio de microtração. E) Dispositivo de microtração em máquina de ensaio universal. F) Ensaio de microtração finalizado com a separação da interface.

Materiais e métodos referentes ao capítulo 2

1. Materiais

Os mesmo utilizados para o capítulo 1, exceto pelo gel de ácido fosfórico a 37%.

2. Método

Da mesma maneira que no capítulo 1, foram utilizados 96 terceiros molares humanos hígidos após aprovação no Comitê de Ética em Pesquisa da Faculdade de Odontologia de Piracicaba, Universidade Estadual de Campinas, e incluídos em resina de poliestireno. A superfície oclusal dos dentes foi previamente desgastada até promover uma superfície plana antes da realização das restaurações, os dentes foram levados a lixadeira (Politriz, AROTEC – São Paulo, SP) para redução das cúspides tomando-se o cuidado para não expor a dentina subjacente.

Após o preparo cavitário seguindo o mesmo protocolo já exposto para o capítulo 1,

os dentes foram divididos nos devidos grupos experimentais por sorteio (adesivo x fotoativação):

- Grupo 1 – Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Ultra-Lume 5;

- Grupo 2 – Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Ultra-Lume 5;

- Grupo 3 – Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Radii-Cal;

- Grupo 4 – Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Radii-Cal;

- Grupo 5 – Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Flash Lite 1401;

- Grupo 6 – Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Flash Lite 1401;

Ensaio de microdureza Knoop

As amostras após o polimento da restauração foram levadas à cortadeira metalográfica de precisão e foi realizado apenas um corte no centro da restauração, no sentido mésio-distal paralelo ao longo eixo do dente. As fatias foram inclusas em cilindros de resina de poliestireno para facilitar a leitura no microdurômetro. Estas amostras receberam acabamento com lixas de carbeto de silício números 600, 1000 e 2000 e polimento com pasta diamantada de granulações de 6, 3, 1, e 0,5 mm (Arotec Ind. Com.,

São Paulo, Brasil) com discos de feltro em politriz giratória com refrigeração à água (Figura 11 A e B).

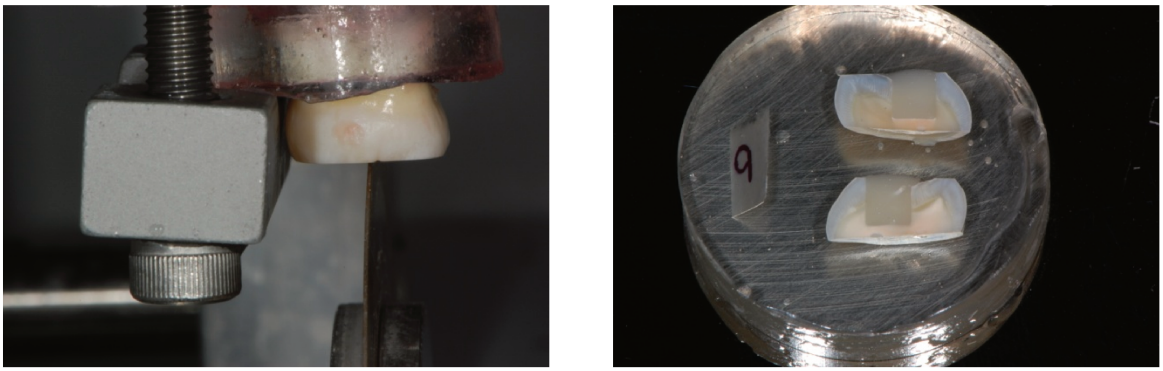


Figura 11. A) Dente sendo seccionado em duas metades. B) Amostra incluída e polida, pronta para a realização do ensaio de microdureza Knoop.

Após, as leituras foram realizadas em três profundidades: superficial, média e profunda. A superficial foi realizada a 100 µm do ângulo cavosuperficial, a profunda a 20 µm da camada de adesivo e a terceira na metade eqüidistante entre essas duas medidas, como mostrado na figura 12:

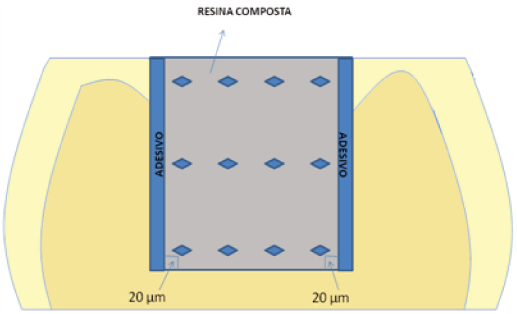


Figura 12. Desenho esquemático das indentações do teste de microdureza Knoop a serem realizadas no corpo-de-prova.

A microdureza foi mensurada através de identações sob carga de 50g em 15 segundos de penetração (HMV-2000, Shimadzu, Japan) (Figura 13 A e B).

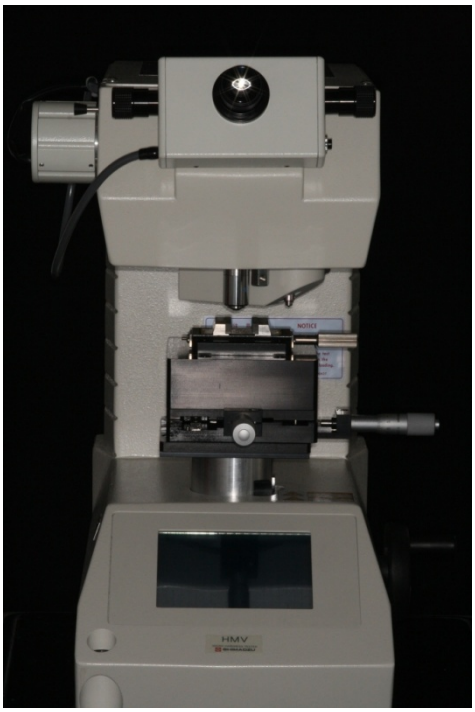


Figura 13. MIcrodurômetro HMV-2000 (Shimadzu)

Materiais e métodos referentes ao capítulo 3

1. Materiais

Os mesmo utilizados para o capítulo 1, exceto pelo gel de ácido fosfórico a 37%.

2. Método

Foram utilizados 192 terceiros molares humanos hígidos após aprovação no Comitê de Ética em Pesquisa da Faculdade de Odontologia de Piracicaba, Universidade Estadual de Campinas, e incluídos em resina de poliestireno.

A superfície oclusal dos dentes foi previamente desgastada até promover uma superfície plana antes da realização das restaurações, os dentes foram levados a lixadeira (Politriz, AROTEC – São Paulo, SP) para redução das cúspides tomando-se o cuidado para não expor a dentina subjacente.

Após o preparo cavitário seguindo o mesmo protocolo já exposto para o capítulo 1, os dentes foram divididos nos devidos grupos experimentais por sorteio (adesivo x fotoativação x condicionamento do esmalte):

- Grupo 1 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Ultra-Lume 5;

- Grupo 2 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Radii-Cal;

- Grupo 3 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com o sistema Flash Lite 1401;

- Grupo 4 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Ultra-Lume 5;

- Grupo 5 – Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Radii-Cal;

- Grupo 6 - Sem condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com o sistema Flash Lite 1401;

- Grupo 7 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%;

Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Ultra-Lume 5.

- Grupo 8 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Radii-Cal;

- Grupo 9 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil S^3 Bond; Fotopolimerização com sistema Flash Lite 1401

- Grupo 10 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Ultra-Lume 5.

- Grupo 11 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Radii-Cal.

- Grupo 12 - Condicionamento do esmalte cavosuperficial com ácido fosfórico a 37%; Sistema adesivo autocondicionante Clearfil SE Bond; Fotopolimerização com sistema Flash Lite 1401.

Análise da adaptação marginal

Após o polimento das amostras, estas foram armazenadas em água destilada a 37°C por 24 horas. Moldes da superfície oclusal de cada dente foram realizados, utilizando para isso o material de impressão por adição Express XT (3M ESPE, St Paul, MN, USA). Os moldes foram vazados com resina epóxica (Resina Epóxica Buehler, IL 60044-1699, USA) e as réplicas obtidas foram preparadas para análise em Microscopia

Eletrônica de Varredura (JEOL, JSM-5600LV, Scanning Electron Microscope, Japão), através da cobertura com fina camada de ouro (Balzers-SCD 050 Sputter Coater, Liechtenstein) para avaliação da adaptação nas margens em esmalte (Figura 14). As fotomicrografias foram feitas da superfície oclusal em magnitudes de 200X. Logo após, as amostras foram submetidas ao envelhecimento por fadiga termomecânica, constituída de 500 ciclos com banhos entre 5 e 55°C ±1°C, com 60 segundos em cada banho e tempo de transferência de 7 segundos aliada a 200.000 ciclos mecânicos (2Hz) em máquina de desgaste termomecânico ER – 37000 (Erios, São Paulo, Brasil) (Figura 15). Após o envelhecimento, novos modelos da face oclusal em resina epóxica foram confeccionados como descrito anteriormente, e nova análise em Microscopia Eletrônica de Varredura da adaptação da margem em esmalte das restaurações pós-ciclagem foram realizadas (Figura 16). As amostras foram inicialmente visualizadas com 25X de aumento, em seguida as margens foram observadas com aumentos de até 200X para elucidar possíveis dúvidas na qualidade da margem. Em seguida, a mensuração das fendas foi feita diretamente no monitor do microscópio, utilizando a ferramenta Multi point measuring device, com aumento de 25X, observando-se o perímetro total das cavidades. Regiões da margem cavitária em que se observava uma transição contínua e sem fendas entre a restauração e a estrutura dentária foram classificadas coma margem perfeita, enquanto regiões que apresentavam perda de adesão interfacial, com ausência de continuidade entre a restauração e a estrutura dentária foram classificadas como fenda marginal e mensuradas. Adicionalmente, o comprimento total da margem cavitária foi mesurado para determinação da porcentagem de fendas.

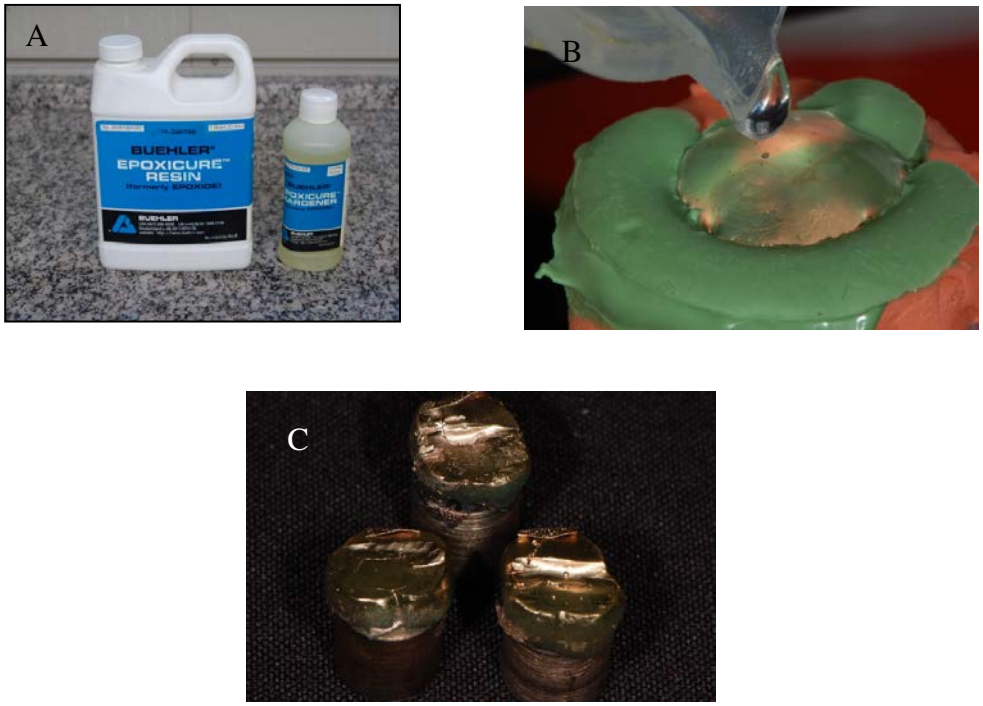


Figura 14. A) Resina epóxica e catalisador. B) Molde em silicone de adição vazado com resina epóxica. C) Réplicas metalizadas.

Figura 15. Máquina de fadiga termomecânica ER – 37000.

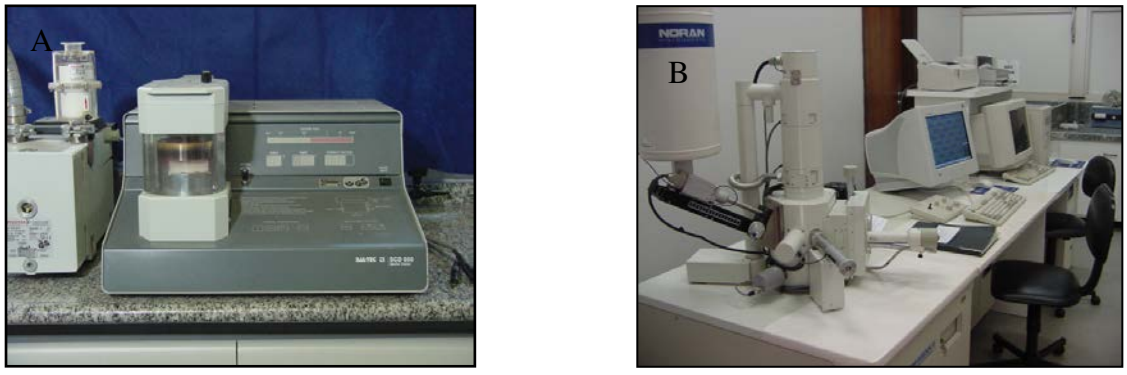


Figura 16. A) Metalizador. B) Microscópio Eletrônico de Varredura.

Análise do padrão de condicionamento

Para analisar o padrão de condicionamento dos sistemas autocondicionantes foram utilizados 2 fragmentos dentais em esmalte, para cada condição de condicionamento estudado. Cada fragmento mediu 5 mm X 6 mm e foram extraídos da região central da face vestibular da coroa dos dentes. Os fragmentos foram planificados através da lixadeira (Politriz, AROTEC – São Paulo, SP) com lixas de carbeto de silício 600 e 1200, e posteriormente armazenados em água destilada. Foram analisados dois tipos de condicionamento:

Ácido fosfórico a 37%

A superfície do esmalte foi condicionada por 30 segundos. Em seguida lavada com água por 30 segundos e secada com ar até a visualização do aspecto opaco.

Adesivos autocondicionantes

Para os adesivos autocondicionantes, após a aplicação sobre a superfície do esmalte, pelo tempo determinado pelo fabricante, o primer foi removido através de lavagens alternadas em álcool absoluto (Etanol PA – 99,8% F. Maia Ind e Co. Ltda, São Paulo – SP – Brasil) e acetona (Acetona PA – 99,8% F. Maia Ind e Co. Ltda, São Paulo – SP – Brasil) por 10 segundos cada banho, durante 1 minuto, para a dissolução dos resíduos remanescentes de monômero na superfície.

Ácido fosfórico a 37% + adesivo autocondicionante

A superfície do esmalte foi condicionada por 30 segundos. Em seguida, lavada com água por 30 segundos e secada com ar. Posteriormente, aplicaram-se os primers como descrito anteriormente e removido da mesma maneira.

Após o condicionamento, todos os espécimes foram submetidos à desidratação (imersão em solução de álcool a 25%, 50% e 75% por 20 minutos cada, 95% por 30

minutos e 100% por 60 minutos e visualizados em Microscopia Eletrônica de Varredura (JEOL, JSM-5600LV, Scanning Electron Microscope, Japão), através da cobertura com fina camada de ouro (Balzers-SCD 050 Sputter Coater, Liechtenstein) para avaliação do padrão de condicionamento do esmalte. As fotomicrografias foram feitas da superfície vestibular em magnitudes de 1500 a 5000X.

Anexo 1

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 **"Avaliação da fonte de luz e do tratamento superficial do esmalte cavosuperficial na resistência à microtração, microdureza Knoop e formação de fendas de sistemas adesivos autocondicionantes"**, protocolo nº 057/2009, dos pesquisadores Luis Alexandre Maffei Sartini Paulillo e Eduardo José Carvalho de Souza Junior, satisfaz as exigências do Conselho Nacional de Saúde - Ministério da Saúde para as pesquisas em seres humanos e foi aprovado por este comitê em 10/06/2009.

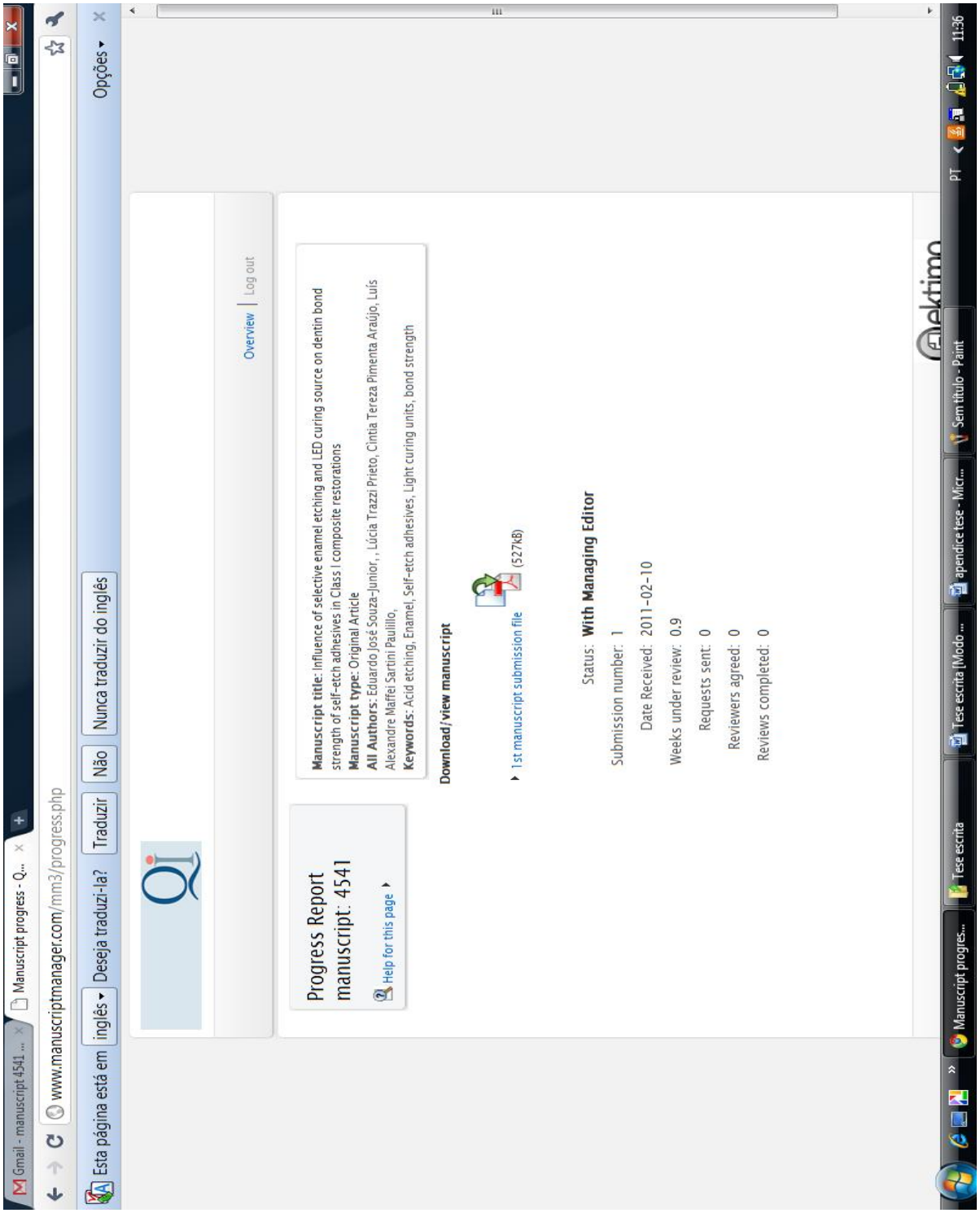
The Ethics Committee in Research of the School of Dentistry of Piracicaba - State University of Campinas, certify that the project **"Evaluation of light source and superficial treatment of cavosuperficial enamel on microtensile bond strength, Knoop microhardness and gap formation of self-etch adhesive systems"**, register number 057/2009, of Luis Alexandre Maffei Sartini Paulillo and Eduardo José Carvalho de Souza Junior, comply with the recommendations of the National Health Council - Ministry of Health of Brazil for research in human subjects and therefore was approved by this committee at .

Prof. Dr. Pablo Agustin Vargas
Secretário
CEP/FOP/UNICAMP

Prof. Dr. Jacks Jorge Junior
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.

Anexo 2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Manuscript type: Original Article
All Authors: Eduardo José Souza-Junior, , Lúcia Trazzi Prieto, Cíntia Tereza Pimenta Araújo, Luís Alexandre Maffei Sartini Paulillo,
Keywords: Acid etching, Enamel, Self-etch adhesives, Light curing units, bond strength
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Status: With Managing Editor
Submission number: 1
Date Received: 2011-02-10
Weeks under review: 0.9
Requests sent: 0
Reviewers agreed: 0
Reviews completed: 0
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