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FACULDADE DE FARMÁCIA, ODONTOLOGIA E ENFERMAGEM
PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA

LÍVIA DE OLIVEIRA BARROS

**AVALIAÇÃO DO MÉTODO DE PREPARO CAVITÁRIO NA INTEGRIDADE
MARGINAL E RESISTÊNCIA DE UNIÃO DE SISTEMAS ADESIVOS À DENTINA
HUMANA**

FORTALEZA

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Área de Concentração: Clínica Odontológica

Orientador: Prof. Dr. Vicente de Paulo Aragão Saboia

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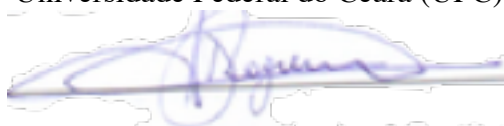
BANCA EXAMINADORA



Prof. Dr. Vicente de Paulo Aragão Saboia (Orientador)
Universidade Federal do Ceará (UFC)



Prof. Dr. Victor Pinheiro Feitosa
Universidade Federal do Ceará (UFC)



Prof. Dr. Alessandro Dourado Loguécio
Universidade Estadual de Ponta Grossa (UEPG)



Profª. Drª. Profa. Dra. Denise Sá Maia Casselli
Universidade Federal do Ceará – (UFC)



Profª. Drª Vanara Florêncio Passos
Universidade de Fortaleza – (UNIFOR)

Dedicatória

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"Veni, Vidi, Vici."

(Júlio César, 47 a.c.)

RESUMO

Smear layer (SL) é uma camada de detritos orgânicos e inorgânicos que recobre a superfície dentária após a confecção de um preparo cavitário. O método de preparo cavitário e, conseqüentemente, as características micromorfológicas da SL podem afetar o desempenho dos sistemas adesivos. Este estudo foi dividido em dois capítulos, cujos objetivos foram: 1) Avaliar o efeito do método de preparo cavitário nas características micromorfológicas da superfície dentinária e na resistência de união (RU) de sistemas adesivos à dentina (Capítulo 1); 2) Avaliar o efeito do método de preparo cavitário na adaptação e selamento marginal da interface esmalte/restauração produzida por dois sistemas adesivos (Capítulo 2). Foram utilizados oitenta molares humanos hígidos extraídos nos quais foram confeccionadas cavidades padronizadas classe I (5 X 4 x 3 mm), com margens em esmalte, de acordo com o método de preparo cavitário utilizado: (1) Ponta diamantada de média granulação; (2) Ponta diamantada de média granulação seguida por ponta diamantada de granulação extra-fina; (3) Broca carbide de 12 lâminas e (4) Broca carbide de 12 lâminas seguida por broca carbide de 30 lâminas. Incrementos de resina composta foram inseridas nas cavidades após a aplicação dos sistemas adesivos Adper[®] Scotchbond[®] Multipurpose (SBMP) e Clearfil[®] SE Bond (CSE). Réplicas de resina epóxica da superfície oclusal das restaurações foram obtidas após serem submetidas 20.000 ciclos termomecânicos. Os espécimes foram cortados para obtenção de palitos da interface resina-dentina que foram testados em microtração e classificados conforme o padrão de fratura (Capítulo 1). As réplicas foram analisadas em MEV para avaliação de microfendas marginais (Capítulo 2). Dentes adicionais (n=16) foram preparados para avaliação qualitativa em MEV das características micromorfológicas da SL e da superfície dentinária (Capítulo 1). Outros dentes (n=48) foram submetidos aos mesmos procedimentos restauradores descritos acima e imersos em solução de azul de metileno a 2% para avaliação da microinfiltração marginal (Capítulo 2). Para cada teste foi aplicada a análise estatística apropriada. Avaliação qualitativa em MEV revelou SL mais espessa e superfície dentinária mais irregular para as pontas diamantadas quando comparadas as brocas carbide. O método de preparo cavitário afetou a RU de ambos sistemas adesivos. A ponta diamantada de média granulação apresentou os maiores valores de RU para SBMP e a broca carbide de 30 lâminas para CSE (p<0,05) (Capítulo 1). Os

métodos de preparo cavitário não afetaram a porcentagem de fendas marginais e a quantidade de microinfiltração marginal para o SBMP ($p>0,05$). CSE apresentou a maior porcentagem de margens livres de fendas e menor microinfiltração marginal ($p<0,05$) quando utilizado com ponta dimantada de média granulação (Capítulo 2). O método de preparo cavitário afeta as características da SL, da micromorfologia dentinária e, conseqüentemente, a RU. Superfícies dentinárias mais irregulares promoveram melhores valores de RU com o SBMP. Por outro lado, a irregularidade e a espessura da SL parecem comprometer o desempenho do sistema autocondicionante. O efeito do método de preparo cavitário no selamento e na adaptação marginal dependeu do sistema adesivo utilizado, uma vez que afetou mais o sistema adesivo autocondicionante.

Palavras-chave: Preparo da cavidade dentária. Lama dentinária. Adesivos dentinários. Resistência à microtração. Adaptação marginal dentária.

ABSTRACT

Smear layer (SL) is an amorphous layer of organic and inorganic debris formed on the surface of dentin after mechanical preparation of a cavity with rotating or manual instruments. The cavity preparation method and micromorphological characteristics of SL and underlying dentin surface, might affect the adhesive performance. The current study is divided in two chapters, whose aims: 1) To evaluate the effect of dentin surface preparation method on micromorphological characteristics of dentin surface and on the microtensile bond strength (μ TBS) of two adhesive systems to dentin. (Chapter 1); 2) To evaluate the effect of cavity preparation method on marginal integrity and sealing ability of resin-tooth interface produced by two adhesive systems (Chapter 2). Standardized class I cavities (5 x 4 x 3 mm), with margins in enamel, were prepared in sound extracted human molars, according to the preparation method (n=20): (1) Medium grit diamond bur; (2) Medium grit diamond bur followed by extra-fine grit diamond bur; (3) 12 blades Tungsten carbide bur and (4) 12 blades Tungsten carbide bur followed by 30 blades Tungsten carbide bur. Composite restorations were inserted in cavities after Adper™ Scotchbond™ Multipurpose (SBMP) or Clearfil™ SE Bond (CSE) application. Epoxy replicas of restorations were obtained after 20,000 thermo-mechanical cycles. Resin-dentin bonded sticks were prepared for μ TBS testing, pulled until failure at crosshead speed of 0.5 mm/min and failure pattern was evaluated (Chapter 1). For evaluated of restorations marginal integrity, epoxy replicas were observed under scanning electron microscope (SEM) to analysis of the percentage of gap-free margins (Chapter 2). Additional teeth (n=16) were prepared using same groups tested and investigated by SEM to micromorphological qualitative analysis of dentin surface and SL characteristics (Chapter 1). Extra teeth (n=48) were prepared, restored and then immersed in 2% methylene blue for dye penetration analysis (Chapter 2). Under SEM analysis, medium grit diamond bur produced thicker SL and more irregular dentin surface than Tungsten carbide burs. SBMP adhesive yielded the highest μ TBS when medium grit diamond bur was used and for CSE 30 blades Tungsten carbide bur group showed the highest values ($p < 0.05$) (Chapter 1). Cavity preparation methods did not affect the percentage of gap-free margins and dye penetration for SBMP ($p > 0.05$). CSE exhibited the highest percentage of gap-free margins and lowest dye penetration when medium grit diamond bur was used ($p < 0.05$) (Chapter 2). Dentin surface preparation

method affected SL characteristics, dentin surface topography, subsequently, resin-dentin bond strengths. Dentin surface irregularity was able to improve resin-dentin bond performance for SBMP. SL thickness and irregularity may compromise bonding efficacy of the self-etching system. The influence of the cavity preparation method on the marginal integrity and sealing ability were dependent of the adhesive used, once it affected more the performance of self-etching adhesive.

Keywords: Dental cavity preparation. Smear layer. Dentin-bonding agents. Microtensile bond strength. Dental marginal adaptation.

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1 INTRODUÇÃO GERAL

O mecanismo de adesão à dentina está fundamentado na difusão de monômeros resinosos no interior do substrato dentinário, parcialmente desmineralizado, através do processo conhecido como hibridização (NAKABAYASHI; KOJIMA; MASUHARA, 1982). Contudo, durante a remoção de tecido cariado e realização do preparo cavitário são utilizados instrumentos cortantes rotatórios ou manuais que produzem uma camada composta de detritos orgânicos e inorgânicos que recobre a superfície dentária e oblitera os túbulos dentinários, chamada de lama dentinária ou *smear layer* (SL) (KOIBUCHI; YASUDA; NAKABAYASHI, 2001). A SL pode dificultar ou impedir uma hibridização adequada e, conseqüentemente, a sua remoção ou modificação durante o processo de adesão é essencial para formação da camada híbrida e união satisfatória à dentina (PASHLEY; CARVALHO, 1997; VAN MEERBEEK et al., 2003).

Os sistemas adesivos sofreram muitas modificações e podem ser classificados, atualmente, em convencionais e autocondicionantes. Os adesivos convencionais necessitam do passo do condicionamento ácido separadamente, normalmente feito com ácido fosfórico em diferentes concentrações, sendo 37% a mais comum. O condicionamento tem a função de remover a SL ou lama dentinária proveniente dos restos do preparo cavitário, abrindo os túbulos dentinários para a penetração dos monômeros resinosos. Os adesivos autocondicionantes não necessitam da aplicação do ácido isoladamente, pois possuem em sua formulação, monômeros resinosos acídicos que desmineralizam e infiltram os tecidos dentais simultaneamente (VAN MEERBEEK et al. 2003; CARVALHO et al. 2004). Enquanto o condicionamento com ácido fosfórico remove a SL, os sistemas adesivos autocondicionantes são capazes de modificá-la ou dissolvê-la e condicionar o substrato dentário subjacente, incorporando a SL à camada híbrida (WATANABE; NAKABAYASHI; PASHLEY, 1994; TAY et al., 2000; MIGUEZ et al., 2003).

Alguns estudos têm demonstrado que a espessura da SL e a topografia da superfície dentinária, após a remoção desta camada, afeta a resistência de união (RU) dos sistemas adesivos à dentina (OGATA et al., 2001; KOIBUCHI; YASUDA; NAKABAYASHI, 2001; VAN MEERBEEK et al., 2003). A espessura, rugosidade e densidade da SL podem ser determinadas pelo tipo, velocidade e granulação do

instrumento rotatório utilizado na confecção do preparo cavitário (DIAS; PEREIRA; SWIFT JR, 2004; BARROS et al., 2005).

Pontas diamantadas de média granulação criam uma superfície dentinária mais irregular e SL mais espessa quando comparada a preparos cavitários nos quais pontas diamantadas de fina granulação são utilizadas (OLIVEIRA et al., 2003; HOSOYA et al., 2004). Por outro lado, brocas carbide promovem superfícies dentinárias mais regulares e SL com menor espessura (OLIVEIRA et al., 2003; DIAS; PEREIRA; SWIFT JR, 2004; BARROS et al., 2005) em relação às pontas diamantadas, independente de sua granulação. Isto pode ocorrer devido ao fato de as brocas carbide utilizarem lâminas de corte ao invés de partículas abrasivas como as encontradas nas pontas diamantadas.

Diferentes autores (TAGAMI et al., 1991; INOUE et al., 2001; OGATA et al., 2001; OLIVEIRA et al., 2003; HOSOYA et al., 2004) reportam que a espessura da SL, gerada por instrumentos rotatórios cortantes, afeta diretamente os valores de RU de sistemas adesivos à dentina. Por outro lado, outros estudos (TAY et al., 2000; TANI e FINGER, 2002; REIS et al. 2005; KESHIMA et al., 2005) mostram que o método de preparo cavitário tem pouca ou nenhuma influência na RU do adesivo ao substrato dentinário. Dentro desse contexto, o efeito do tipo de instrumento rotatório na RU de sistemas adesivos à dentina é controverso e necessita de maiores esclarecimentos.

Além disso, tem sido proposto que a rugosidade das paredes do preparo cavitário pode influenciar no molhamento e na qualidade de união de sistemas adesivos/dente (EICK et al., 1972). O desenvolvimento de uma adesão satisfatória requer o contato íntimo entre agente adesivo e substrato dentário (AL-OMARI; MITCHELL; CUNNINGHAM, 2001). Dessa forma, o acabamento das paredes do preparo cavitário pode afetar a RU, uma vez que, promove a formação de um substrato mais uniforme e margens da cavidade mais regulares. Superfícies mais lisas permitem a melhor infiltração do agente adesivo, reduzindo o risco de incorporações de bolhas que pode inibir a polimerização da agente resinoso (STANGEL; ELLIS; SACHER, 2007).

Apesar da contínua evolução no desempenho dos sistemas adesivos e resinas compostas, a integridade marginal das restaurações estéticas ainda é considerada um desafio (DAVIDSON e FEILZER, 1997; BENETTI et al., 2014). Restaurações adesivas com qualidade marginal satisfatória são menos susceptíveis a pigmentação marginal, presença de microfendas, irritação pulpar, fraturas, perda de retenção, ou ainda, microinfiltração de bactérias e fluídos orais, com consequente desmineralização dos tecidos dentais e reincidência de cáries (KIDD e BEIGHTON, 1996; OPDAM et al., 2010). O uso de materiais e técnicas restauradoras adequadas são essenciais para minimizar o estresse na interface adesiva e seus efeitos deletérios (KNOW; FERRACANE; LEE, 2012).

Diante do exposto, o objetivo deste estudo *in vitro* foi avaliar o efeito de diferentes métodos de preparo cavitário na RU de sistemas adesivos à dentina, nas características micromorfológicas da superfície dentinária e na adaptação e selamento marginal de restaurações adesivas diretas.

2 PROPOSIÇÃO

Essa tese de doutorado será apresentada em dois capítulos, tendo como objetivos:

Capítulo 1: Avaliar o efeito do método de preparo cavitário nas características micromorfológicas da superfície dentinária e na resistência de união de sistemas adesivos à dentina humana.

“Effect of dentinal surface preparation method on the bonding strength of dental adhesives”

Capítulo 2:

Avaliar o efeito do método de preparo cavitário na adaptação e no selamento marginal da interface dente/restauração produzida por dois sistemas adesivos.

“Effect of cavity preparation methods on marginal integrity and sealing ability of composite restorations”

3 CAPÍTULOS

REGIMENTO INTERNO

Esta tese está baseada no Artigo 46 do Regimento Interno do Programa de Pós-graduação em Odontologia da Universidade Federal do Ceará, que regulamenta o formato alternativo para dissertações de Mestrado e teses de Doutorado e permite a inserção de artigos científicos de autoria ou co-autoria do candidato. Desta forma, esta tese é composta de dois capítulos contendo artigos a serem submetidos para publicação no periódico *The Journal of Adhesive Dentistry*.

Capítulo 1

“Effect of dentinal surface preparation method on the bonding strength of dental adhesives”

Barros LO, Paulillo LAMS, Saboia VPA

Capítulo 2

“Effect cavity preparation methods on marginal integrity and sealing ability of composite restorations”

Barros LO, Paulillo LAMS, Saboia VPA

3.1 CAPÍTULO 1

Effect of dentinal surface preparation method on the bonding strength of dental adhesives

L.O. Barros¹, L. A. M. S. Paulillo², V.P.A. Saboia¹

¹ Department of Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil;

² Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, Brazil.

Correspondence: Prof. Vicente Saboia, Department of Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil - Gilberto Studart street, 770 Aptº 901 – Cocó, CEP: 60.190-750, Fortaleza, Ceará, Brazil, Tel: +558588074623 Fax: +558533668232; e-mail: vpsaboia@yahoo.com

Keywords: Dental cavity preparation, smear layer, microtensile bond strength, dentin adhesive

ABSTRACT

Purpose: To evaluate the effect of different surface preparation methods on micromorphological characteristics of dentin surface and on the microtensile bond strength (μ TBS) of two adhesives to human dentin. **Materials and Methods:** Eighty standardized class I cavities (5 x 4 x 3 mm), with margins in enamel, were prepared in sound extracted human molars, according to the surface preparation method (n=20): (1) Medium grit diamond bur; (2) Medium grit diamond bur followed by extra-fine grit diamond bur; (3) 12 blades Tungsten carbide bur and (4) 12 blades Tungsten carbide bur followed by 30 blades Tungsten carbide bur. Composite restorations were inserted in the cavities after Adper™ Scotchbond™ Multipurpose (SBMP) or Clearfil™ SE Bond (CSE) application. Specimens were submitted to 20,000 thermo-mechanical cycles. Resin-dentin bonded sticks were prepared for μ TBS test, pulled until failure at crosshead speed of 0.5 mm/min and failure pattern was evaluated. Data were statistically analyzed with two-way ANOVA and Tukey test ($\alpha=5\%$). Additional teeth (n=16) were prepared using the same groups tested and investigated by SEM to micromorphological qualitative analysis of dentin surfaces and smear layer (SL). **Results:** SBMP yielded the highest μ TBS (31.6 ± 4.6 MPa) when medium grit diamond bur was used ($p<0.05$). For CSE, the μ TBS was highest when bonded to dentin prepared with carbide bur followed by 30 blades carbide bur (26.6 ± 1.5 MPa) ($p<0.05$). Lower bond strengths were associated with higher percentages of adhesive failures. **Conclusion:** Dentin preparation method affected SL characteristics, dentin surface topography and, subsequently, resin-dentin bond strengths. Higher dentin surface roughness attained with medium grit diamond bur was able to improve resin-

dentin bond performance for SBMP. Higher SL thickness and roughness may compromise bonding efficacy of the self-etching system. Proper bur and adhesive selection are essential to optimize resin-dentin bonding.

Keywords: Dental cavity preparation, smear layer, microtensile bond strength, dentin adhesive

INTRODUCTION

Dentin smear layer (SL) is produced whenever a tooth is altered with rotary or manual instruments during cavity preparation.^{7,30} It varies in thickness, roughness, density and degree of attachment to the underlying tooth structure according to surface preparation method.^{4,19} Some studies^{2,17,25} reported that diamond burs produced thicker SL and rougher surfaces, whereas dentin prepared with carbide burs was smoother and covered with a thinner SL.

The literature has shown the importance of removal or modification of the SL for the development of a hybrid layer to obtain optimal adhesion^{21,29} once the presence of this layer affects the outcome of bonded restorations.⁷

Based on the current adhesion strategy, there are two major approaches to produce an effective bond between resin-dentin. The etch-and-rinse adhesives employ phosphoric acid to remove the SL, followed by primer/adhesive applications. On the other hand, self-etching adhesives utilize nonrising acidic resin monomers to modify the SL, etch and prime dentin, simultaneously.³³ The subsequent bonding process incorporates this modified SL within the resin-dentin bond interface.³²

It has been reported that bond strength (μ TBS) of adhesive to dentin depends on characteristics of the SL created by cutting instruments.^{18,26,27} While several studies have demonstrated low μ TBS over thick SL,^{13,31} especially when self-etching adhesives are used, there has been lots of published work showing little or no

influence of SL thickness on the bond strength.^{12,20,24,30} Thereby, the effect of dentin surface preparation method on bond strength of adhesives to dentin still remains unclear and worths further investigation.

Moreover, cavity walls roughness may influence the wettability and bonding quality of adhesive agents.⁸ The development of an adhesive bonding requires establishing intimate contact between the adhesive and tooth surface.¹ Thus, the use of instruments for finishing of cavity walls may influence the adhesion. Finishing procedure brings to a uniformity and regularity of the margins that eliminates the asperities and the unsubstained prisms. A smooth surface makes easier the flowing of the adhesive resin, reducing the risk to hold air bubbles that could inhibit the resin polymerization.²⁸

The aim of the present study was to evaluate the micromorphological characteristics of dentin and μ TBS of two adhesives, a three-step etch-and-rinse system and a two-step self-etching system, bonded to dentin surface prepared with different preparation methods: (1) diamond bur or (2) carbide bur. The tested null hypothesis was that, for each adhesive approach, there are no difference in resin-dentin bond strengths and micromorphological features among different dentin preparation methods.

MATERIALS AND METHODS

Tooth Preparation

Eighty recently extracted human non-carious third molars were used under a protocol approved by Institutional Review Board (COMEPE/UFC 983.135). The teeth were stored in 0.01% thymol solution at 4° C and used within 3-month after extraction. After being copiously rinsed in running water, cleaned and pumiced,

cuspal enamel of the tooth crowns was ground using 320-grit wet silicon carbide paper mounted in a polisher machine (Aropol 2V – Arotec, São Paulo, Brazil), under running water, without exposing coronal dentin.

Specimens were equally and randomly assigned to four groups (n=20) according to method of dentin surface preparation:

Group 1: Teeth cavities were prepared using a cylindrical medium grit (100 μm grain size) diamond bur (#3098, KG Sorensen, Barueri, Brazil);

Group 2: The cavities were prepared as in Group 1, followed by a cylindrical extra-fine grit (15 μm grain size) diamond bur (#3098 FF, KG Sorensen, Barueri, Brazil);

Group 3: Teeth cavities were prepared using a cylindrical 12 blades Tungsten carbide bur (#56, Beavers Dental, ON, Canada);

Group 4: The cavities were treated as in Group 3, followed by a cylindrical 30 blades Tungsten carbide bur (#9572 FF, Beavers Dental, ON, Canada).

Standard class I cavities measuring 5 mm (mesiodistal) \times 4 mm (buccolingual) \times 3 mm (depth) were prepared according to the methods described above. Within these dimensions, the C-factor (ratio between the bonded area and the free surface of the cavity) was approximately 3.7. The cavities were prepared with a water-cooled highspeed turbine using a standard cavity preparation device. The turbine is attached to this device that permits the controlled movement of the bur on the x, y and z axes. Each preparation was designed so that the occlusal margin was in the enamel. A new bur was used for each of the five preparations.

Bonding procedures

Each group was further subdivided into two subgroups (n=10) and treated with one of the two adhesives: Adper™ Scotchbond™ Multipurpose (SBMP, 3M ESPE, St.

Paul, USA) and Clearfil™ SE Bond (CSE, Kuraray Medical Inc, Tokyo, Japan), a three-step etch-and-rinse and a two-step self-etching adhesive system, respectively. Adhesives were applied according to the manufacturer's instructions (Table 1), except for cavosuperficial angle conditioning, which was etching using 37% phosphoric acid (Condac, FGM, Joinville, Brazil) for 30 s, for CSE. The cavities were restored using a light-cured resin composite (Charisma Opal, Shade A2, Heraeus Kulzer, Hanau, Germany) inserted in 2 mm-thick oblique increments and individually polymerized for 20s each, using a LED light-curing unit (Radium-cal, SDI Limited, Victoria, Australia) with an output intensity of 1200 mW/cm². A single operator performed all procedures in order to exclude inter-operator variation. After immersion in distilled water for 24 h at 37°C, finishing and polishing of restorations surface was carried out using sequential aluminium oxide paper discs (Sof-Lex PopOn, 3M ESPE, St. Paul, USA) under low speed.

Thermo-mechanical fatigue loading

The resin-bonded specimens were submitted to thermo-mechanical fatigue loading using a thermo-mechanical cycling machine (MSFT, Elquip, São Carlos, Brazil) programmed to perform simultaneously 20,000 mechanical cycles under a load of 80 N, at a rate of 2 Hz, and thermal-cycles at temperatures between 5°C and 55°C, with a dwell time of 30 s at each bath temperature.

Microtensile bond strength testing

Bonded specimens were serially sectioned, parallel and perpendicular to the long axis of the tooth, using a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, USA) under water cooling to obtain 1mm-thick beams, in accordance with microtensile test *non-trimming* technique (Fig. 1).

Each beam was individually measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) and attached to a testing jig with cyanoacrylate

glue (SuperBonder flex gel, Henkel Ltda, Düsseldorf, Germany). Beams were stressed to failure under tension using a microtensile testing machine (Emic, DL2000, São José dos Pinhais, Brazil) at a crosshead speed of 0.5 mm/min. The number of prematurely debonded sticks per group during specimen preparation was also recorded.

Dentin side of the failed bonds was analyzed using a stereoscopic light microscopy (Stemi 2000-C, Carl Zeiss Jena, Jena, Germany) with 50 x magnification and classified according to the failure mode as adhesive (A), cohesive in dentin (CD), cohesive in composite (CC) or mixed (M).

Bond strength data were collected and analyzed using SigmaStat 3.5 (Systat, Chicago, USA). Premature failures from μ TBS were included in the statistical analysis as 0 MPa. As the normality (Kolmogorov-Smirnov) test passed ($p>0.05$), a two-way ANOVA and Tukey's test were employed to examine the effects of adhesive and dentin surface preparation methods ($\alpha=5\%$).

Scanning electron microscopy (SEM) analysis

Sixteen additional third molars ($n=2$ for each subgroup) were used to describe micromorphological aspects of prepared dentin surface, SL thickness and dentin characteristics after conditioning, under SEM (Quanta FEG 450, FEI, Amsterdam, Netherlands). The preparation of the cavities was carried out as described for bond strength test. After cavity preparation, each tooth was cut into halves, and two half-tooth was used for each group to observe SL thickness and micromorphology of dentin surface. For observation of dentin surface after etching, 37% phosphoric acid or primer of CSE was applied on the other half-tooth surface, as recommended by manufacturer. After application time, phosphoric acid gel was flushed with water, whereas primer components were removed by rising the specimens with 100% ethanol

for 5 min and placed in distilled water for further 5 min.²⁰ The specimens were fixed in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 for 12 h at 4°C. After fixation, samples were rinsed with 20 ml of 0.2 M sodium cacodylate buffer at pH 7.4 for 1 h, with three changes, followed by deionized water for 1 min. All specimens were dehydrated in an ascending series of ethanol to 100% followed by immersion in hexamethyl disilazane (HMDS) for 10 min and allowed to air-dry.²² The specimens were gold sputter-coated and observed using SEM.

RESULTS

Microtensile bond strength

All information regarding microtensile bond strength are shown in table 2. The number of prematurely debonded sticks per test group affected all groups in a similar extent (Table 2).

Two-way ANOVA revealed that significant differences were observed for the main factors “dentin surface preparation method“ ($p < 0.001$) and “adhesive systems” ($p < 0.001$). The interaction between these two factors was also significant ($p < 0.001$), indicating that the effect of adhesive on bond strength results was dependent upon the dentin surface preparation method.

For SMBP, the highest μ TBS ($p < 0.05$) was observed when cavity was prepared with medium grit diamond bur (31.6 ± 4.6 MPa). On the other hand, no statistical difference was observed among the groups ($p > 0.05$), which used medium grit diamond bur followed by extra-fine grit diamond bur and 12 blades carbide bur followed or not followed by 30 blades carbide bur (21.4 ± 4.7 MPa, 22.1 ± 5.1 MPa and 17.9 ± 4.3 MPa, respectively).

For the CSE, the highest bond strength was found using 12 blades carbide bur followed by 30 blades carbide bur group (26.6 ± 1.5 MPa), but no difference was shown among the remaining groups. However, CSE exhibited significantly lower mean bond strength (18.6 ± 3.1 MPa) than SBMP (31.6 ± 4.6 MPa) in medium grit diamond bur group.

Examination of failure mode by stereoscopic light microscopy indicated that the most of surfaces failed at adhesive interface (adhesive failure) and lower bond strengths were associated with higher percentages of adhesive failures (Table 3).

Scanning electron microscopy (SEM) evaluation

Evaluation of prepared dentin surface

Under SEM analysis, medium grit diamond bur (100 μm grain size), produced the thickest SL (2.5 μm) with irregular and more corrugated surface, traversed by deep grooves (Fig. 2a and 2b). Extra-fine grit diamond bur (15 μm grain size) created a moderately thick SL (2.0 μm) with less irregular surface as compared to medium grit diamond bur (Fig. 2 c and 2d).

The group prepared with 12 blades Tungsten carbide bur produced a thin (1.5 μm) and relatively regular SL, showing uniform scratches on the cut surface (Fig. 3a and 3b). Specimens prepared with 30 blades Tungsten carbide bur showed the smoothest surface with narrow grooves on the dentin (Fig. 3c and 3d).

Evaluation of treated dentin surface

SEM observations of the conditioned dentin surfaces with 37% phosphoric acid and CSE primer revealed differences among the various surface preparation methods. Dentin prepared with medium grit diamond bur treated with CSE showed partial removal of SL and peritubular dentin appeared to be slightly etched (Fig 5b). Dentin prepared with extra-fine grit bur displayed partial demineralization of SL with dentinal tubules remaining occluded with smear plugs (Fig 5d). The peritubular dentin

was also etched and the porosity of the intertubular dentin was similar than medium grit diamond bur prepared dentin. Dentin prepared with Tungsten carbide burs after CSE primer application exhibited most removal of SL and partial dissolution of smear plugs. Slight etching of peritubular dentin was observed (5f and 5h).

For phosphoric acid groups, there were no differences in terms of removal of the SL between diamond and carbide groups. The SL and smear plugs were completely removed (Fig. 5a, 5c, 5e, 5g). The only distinct differences were dissolution of smear plugs which seemed to be more resistant to the acid remained in some tubules (Fig. 5c) when extra-fine grit diamond bur was used.

DISCUSSION

The presence of the SL on ground dentin has been considered as a barrier for resin infiltration during bonding. This zone of debris is a mixture of partly denatured collagen fibrils, other organic materials and several compounds, according to the underlying dentin surface.⁷ Moreover, differences in surface preparation methods can be produce a variety of SL characteristics that have been reported to affect the bond strengths of resin to dentin.^{5,9,18,20,31} Furthermore, it has been related that the effect of dentin preparation on bond strength also depends on the adhesive used.^{9,18,19}

Results of the current study showed that μ TBS of SBMP or CSE were significantly different when bonded to dentin with distincts dentin surface preparation methods. Thus, since there was a difference, for each adhesive, when distincts dentin preparation methods were used, the null hypothesis must be rejected.

The etch-and-rinse adhesive yielded the highest bond strength value when only medium grit diamond bur was used (31.6 ± 4.6 MPa). Medium grit diamond bur created a thicker and more uneven surface^{4,5,19,20} (Fig. 2a and 2b) while a more

compact SL was observed with extra-fine diamond bur, created by tiny abrasive particles present on this cutting instrument (Fig. 2 c and 2d). It is speculated, that this variation in SL density may have contributed to partial removal of SL and smear plugs in some areas by phosphoric acid⁴ (Fig. 5c).

However, this observation is in contrast with previous studies that affirm phosphoric acid completely removes the SL and it is not sensitive to the method used to create the SL.²⁰ Although, once the 37% phosphoric acid removes the whole SL, the striated topography created by dentin surface preparation remained intact beneath. It is reasonable to assume that by increasing the roughness and surface area, the bond strength would be higher, since it would increase the true area of surface bonded by resin.^{3,11} Thus, the lower roughness surface resulting from the extra-fine diamond bur would probably give lower bond strengths for SBMP. Indeed, one may suppose that the dentin surface roughness has a more prominent role than SL thickness on the bond strength of the etch-and-rinse adhesive to dentin.

Carbide burs generated the smoothest surface with apparently homogeneously distributed and thin SL and smear plug³⁴ (Fig. 3). This might be due to the fact that carbide burs use bladed cutting rather than the abrasive cutting of diamond burs.²⁶ Although, extra-fine diamond bur produces a rougher surface and thicker SL than carbide burs,⁵ in this study, bond strength of these dentin surface preparation methods were similar, when SBMP was used. This outcome was expected once phosphoric acid entirely removes the SL²³ and partially dissolves the surrounding peritubular dentin, allowing more resin to infiltrate into the dentin tubule.¹⁴

On the other hand, 12 blades carbide bur followed by 30 blades carbide bur, promoted the highest μ TBS for CSE (26.6 ± 1.5 MPa). Thirty blades carbide bur produced apparently a relatively flat and groove-free substrate (Fig. 3c). The thin SL,

produced by 30 blades carbide bur had a higher permeability and allowed infiltration of the primer acidic monomers through the SL towards the underlying dentin to create a hybrid layer, resulting in higher resin-dentin bond strengths. Moreover, it was hypothesized that smooth dentin surface may improve adhesive wettability by reduction of the contact angle.³ Based on these findings, this might be the cutting instrument of choice for obtaining higher bond strengths when self-etching systems are used in agreement with a previous investigation.⁵

Clearfil SE bond is a “mild” self-etching adhesive with pH 1.9 and incorporate the SL and smear plugs into the hybridized complex. As a result of its weak acidity, it may not be able to penetrate completely through the thick SL produced by diamond burs to demineralize the underlying dentin. Bonding of CSE to dentin surface prepared by Tungsten carbide burs was improved 33%, when compared to diamond burs.

The use of diamond bur resulted an undulated dentin, with higher contact angle, and a dense and thick SL frequently accumulated at the deepest part of the undulations.⁴ The higher the roughness of a substrate, the more difficult is for an adhesive solution to spread over it.¹² Several authors have shown a lower percentage of opened tubules when the roughness of SL was high, which demonstrates that the adhesive was not able to wet the substrate.^{18,19,20} This fact may interfere with wettability of the self-etching adhesive and its ability to penetrate such SL, thus, reducing μ TBS to dentin. Moreover, it is also reported lower resin bond strength of self-etching adhesives bonded to enamel prepared with diamond burs.^{6,15} This compromised enamel bonding should, to a certain extent, be attributed to interference of bur debris smeared across enamel during cavity preparation.¹⁵

For CSE groups, no difference in bond strength was observed when single medium grit diamond bur combined or not with a extra-fine grit was used. This finding is in agreement with results reported by others studies.^{20,24,30} Conversely, previous reports^{5,9,13,19} show that the thick of SL affected dentin bond strengths of self-etching systems. This results may be attributed to the similar removal pattern of the SL by the acidic primer in dentin surfaces prepared with diamond burs combined or not with extra-fine ones. Inou et al (2001)¹⁰ compared the bond strengths of two self-etching adhesives using a medium grit and a extra-fine grit diamond bur prepared dentin. Lower bond strengths were assumed to be related to remaining SL (after primer application) produced by a medium-grit diamond bur. This finding is not in agreement with our results, however, in that study, authors used all-in-one adhesives, which did not seem to remove the SL well enough due to their higher pH. Reported values of bond strength of single-step adhesives have been lower than those of the two-step self-etching adhesive systems¹⁶, like CSE.

Dentin surface preparation method and the cutting instrument affect the surface topography and SL characteristics and, subsequently, resin–dentin bond strengths. In general, dentin surface roughness was able to improve resin-dentin bond performance for SBMP; contrariwise, SL thickness and roughness seemed to compromise bonding efficacy of CSE. The results of the present work suggests that CSE should be used in vivo with a surface preparation method that produces a thin and smooth SL likewise finishing carbide burs, while SBMP does not need cavity finishing procedures. Furthermore, other additional factors (speed of dental bur, amount of pressure applied, substrate and coolant used during preparation) could also affect the SL formation and these variables should be focused on further investigations.

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Clinical relevance: Dentin surface preparation method affects surface topography and SL characteristics and it could influence the interaction between adhesive and substrate.

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Table 1 - Materials compositions, pH and mode of application.

MATERIAL	COMPOSITION (*)	pH PRIMER/ PHOSPHORIC ACID	MODE OF APPLICATION		
			Acid etching	Primer application	Adhesive application
Adper™ Scotchbond™ Multipurpose (3M ESPE, St. Paul, USA) Batch # N295761	Primer: HEMA, solvents Adhesive: HEMA, bis-GMA	<0.5	Apply 37% phosphoric acid to enamel for 30 s and to dentin for 15 s. Rinse for 30 s. Excess of water was removed with absorbing paper.	Apply the primer for 20 s with agitation. Gently air blow for 5 s.	Apply the adhesive and light cure for 10 s.
Clearfil™ SE Bond (Kuraray Medical, Tokyo, Japan) Batch # 01147A	Primer: 10-MDP, HEMA, tertiary amine, hydrophilic DMA, water Adhesive: 10-MDP, HEMA, bis-GMA, tertiary amine, hydrophilic DMA, silanized colloidal silica, photoinitiator.	1.5	Apply 37% phosphoric acid to enamel for 30 s. Rinse for 30 s. Excess of water was removed with air blow.		Apply the adhesive, gently air blow and light cure for 10 s.
Charisma™ Opal (Heraeus Kulzer, Hanau, Germany) Batch # 010024	Bis-GMA, barium aluminium glass, silica.	—	Apply to the cavity in thin layers (max. 2 mm) and adapt carefully to the cavity walls. Light cure for 20 s.		

Abbreviations: HEMA= 2-hydroxyethylmethacrylate; BisGMA= bisphenol glycidyl methacrylate; 10-MDP= 10-methacryloyloxydecyl dihydrogen phosphate; DMA= dimethacrylate.

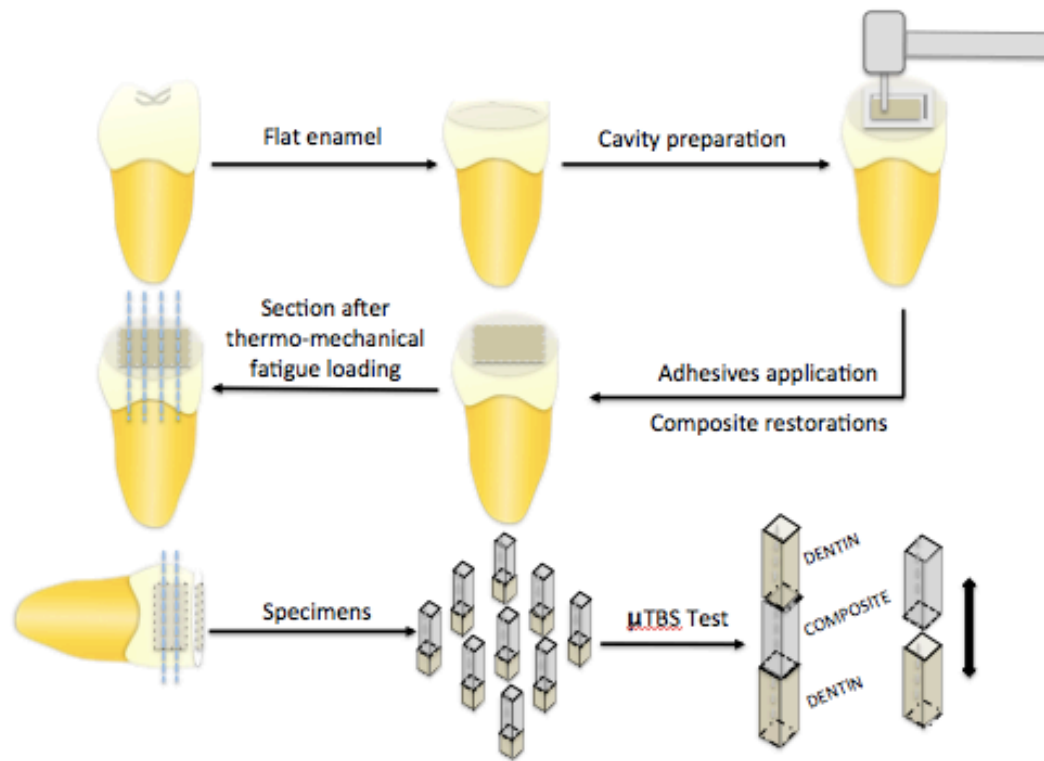


Figure 1 – Schematic illustrating the study design.

Table 2 – Mean and standard deviation (MPa) for microtensile bond strength values according to each experimental group, as well as, statistical analysis (*)

<i>Groups</i>	SBMP	CSE
Medium grit diamond bur	31.6±4.6 ^{A,a}	18.6±3.1 ^{B,b}
Medium grit + extra-fine grit diamond bur	21.4±4.7 ^{A,b}	17.8 ±5.3 ^{A,b}
12 blades Tungsten carbide bur	17.9±4.3 ^{A,bc}	17.9 ±2.6 ^{A,b}
12 blades Tungsten carbide bur + 30 blades Tungsten carbide bur	22.1±5.1 ^{A,b}	26.6 ±1.5 ^{A,a}

Mean ± standard deviation (number of premature failed sticks/number of cohesive failures/number of intact sticks tested) of μ TBS results (expressed in MPa). (*) Different lower case letters in column and capital letters in row indicate statistical difference ($p < 0.05$).

Table 3 – Number of specimens (%) and failure mode according to each experimental group

ADHESIVE SYSTEM	GROUPS	FAILURE MODE (%)			
		A	M	CD	CR
Adper™ Scotchbond™ Multipurpose	Medium grit diamond bur	85.1	5.2	9.7	0.0
	Medium grit + extra-fine grit diamond bur	90.0	4.0	3.3	3.7
	12 blades Tungsten carbide bur	96.0	4.0	0.0	0.0
	12 blades Tungsten carbide bur + 30 blades Tungsten carbide bur	87.3	9.7	0.0	3.0
Clearfil™SE Bond	Medium grit diamond bur	93.0	3.5	3.5	0.0
	Medium grit + extra-fine grit diamond bur	93.7	3.3	3.0	0.0
	12 blades Tungsten carbide bur	95.0	3.0	2.0	0.0
	12 blades Tungsten carbide bur + 30 blades Tungsten carbide bur	87.1	9.7	3.2	0.0

A = adhesive failure; M = mixed failure; CD = cohesive failure in dentin and; CR = cohesive failure in resin composite.

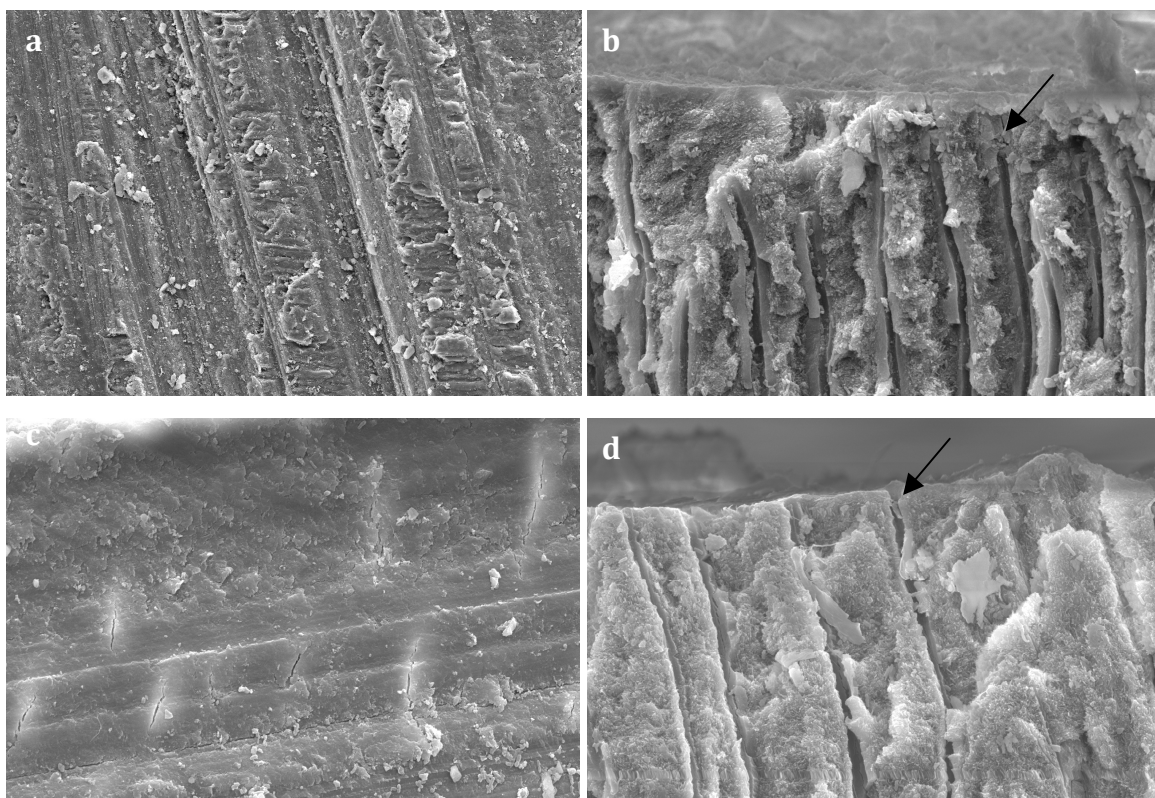


Fig. 2 – (a) and (b) SEM micrograph of dentin surface prepared with medium grit diamond bur. (a) An irregular smear layer with deep grooves was observed (500 X); (b) SEM longitudinal image of dentin tubules show a thick smear layer and dentinal tubules occluded by smear plugs (arrow) (2500 X); (c) and (d) SEM micrograph of dentin surface prepared with a medium grit followed by extra-fine grit diamond bur. (c) An irregular smear layer with more uniform scratches was observed (500 X); (d) SEM longitudinal image of dentin tubules show a thick and compact smear layer, and dentinal tubules closed by smear plugs (arrow) (2500 X).

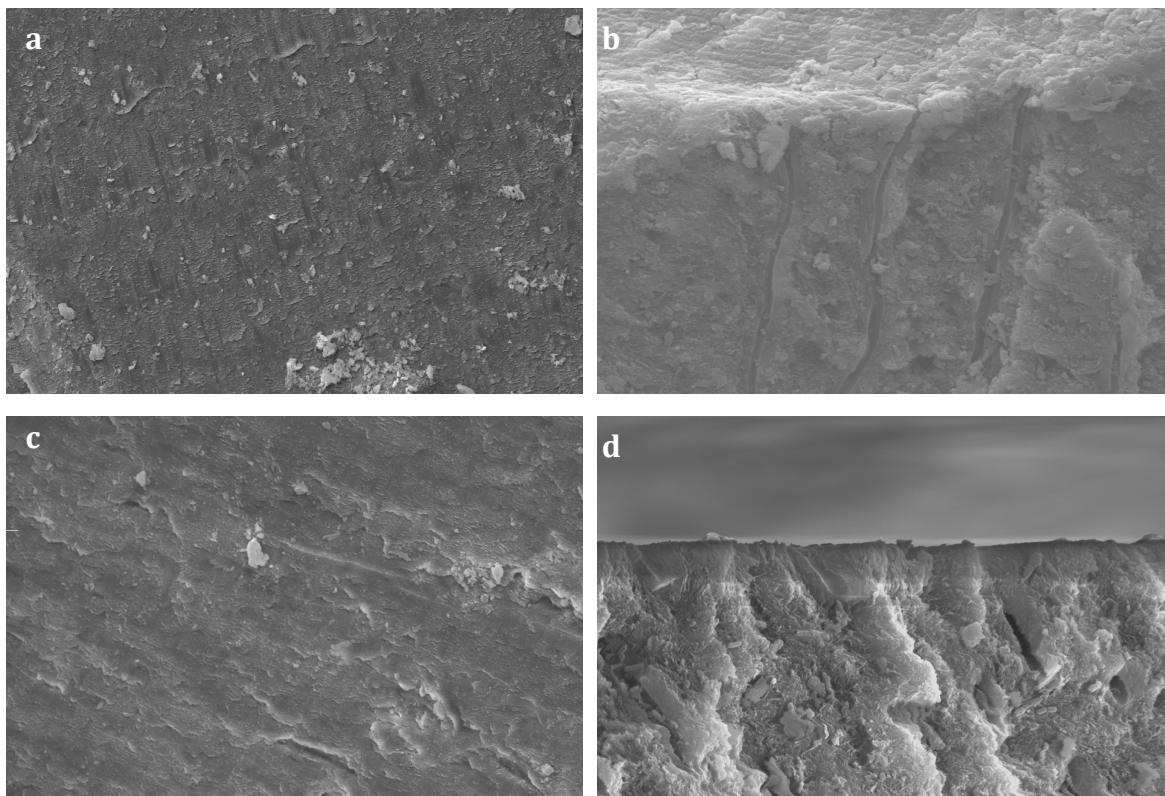


Fig. 3 – (a) and (b) SEM micrograph of dentin surface prepared with a Tungsten carbide bur. **(a)** A smoother smear layer with regular scratches was observed (500 X); **(b)** SEM longitudinal image of dentin tubules show a thin smear layer covered dentinal tubules (2500 X). **(c) and (d)** SEM micrograph of dentin surface prepared with a Tungsten carbide bur followed by a 30 blades carbide bur. **(c)** A relatively flat and groove-free smear layer was observed (500 X). **(d)** SEM longitudinal image of dentin tubules show a thin smear layer covered dentinal tubules (2500 X).

PHOSPHORIC ACID

CSE PRIMER

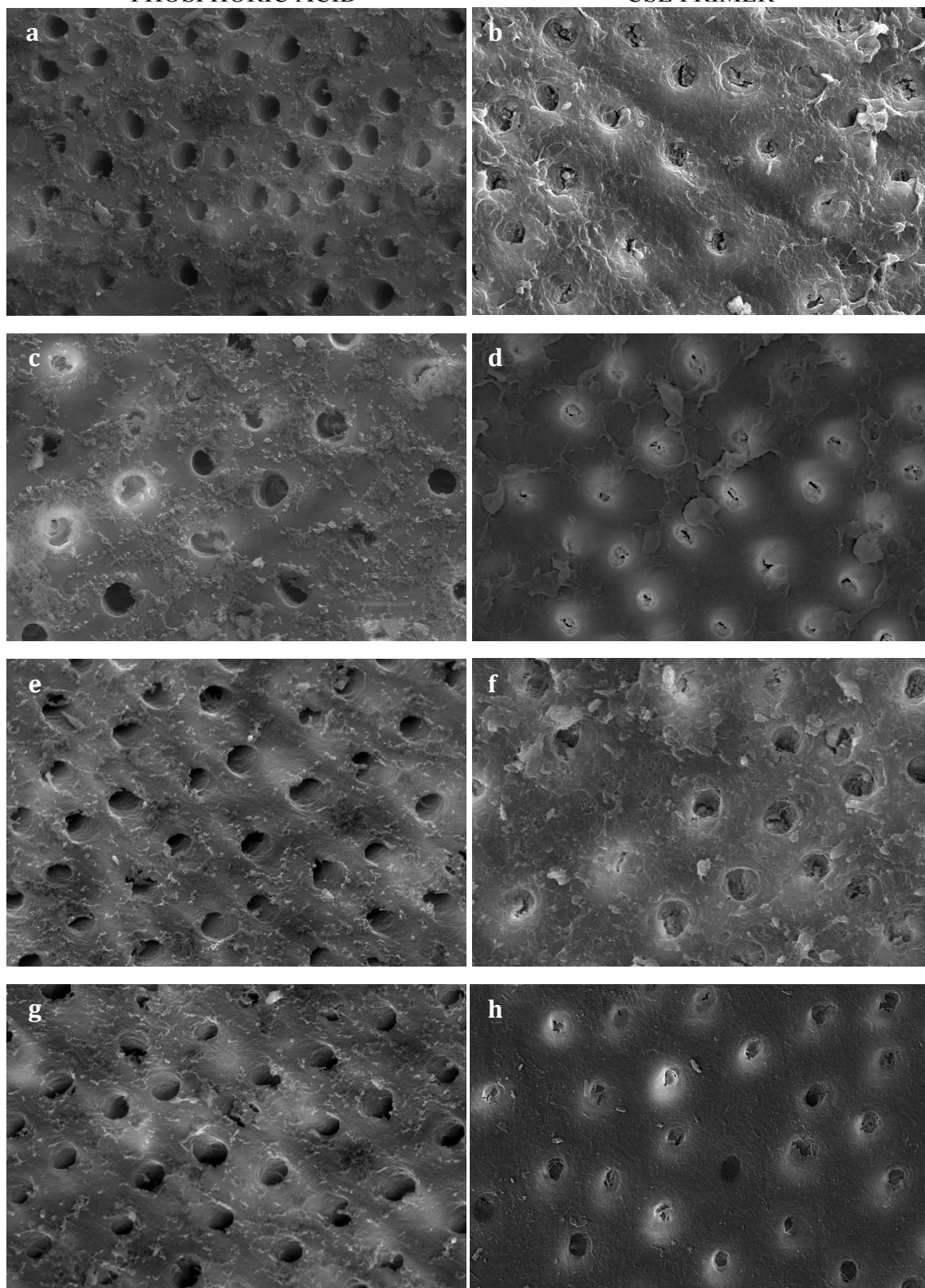


Fig. 4 – SEM micrograph of cut dentin surface after treatment with 37% phosphoric acid or CSE primer. Dentin surface cut with medium grit diamond bur after treatment with (a) phosphoric acid and (b) CSE primer. Dentin surface cut with medium grit diamond bur followed by extra-fine diamond bur after treatment with (c) phosphoric acid and (d) CSE primer. Dentin surface prepared with Tungsten carbide bur after treatment with (e) phosphoric acid and (f) CSE primer. Dentin surface prepared with Tungsten carbide bur followed by 30 blades carbide bur after treatment with (g) phosphoric acid and (h) CSE primer (2000 X).

3.2 CAPÍTULO 2

Effect of cavity preparation methods on marginal integrity and sealing ability of composite restorations

L.O. Barros¹, L.A.M.S. Paulillo², V.P.A. Saboia¹

¹ Department of Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil;

² Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, Brazil.

Correspondence: Prof. Vicente Saboia, Department of Restorative Dentistry, Federal University of Ceará, Fortaleza, Brazil - Gilberto Studart street, 770 Aptº 901 – Cocó, CEP: 60.190-750, Fortaleza, Ceará, Brazil, Tel: +558588074623 Fax: +558533668232; e-mail: vpsaboia@yahoo.com

Keywords: Dental cavity preparation, smear layer, gap formation, dentin adhesive

ABSTRACT

Purpose: To evaluate the effect of cavity preparation method on marginal integrity and sealing ability of composite restorations bonded to human dentin using two adhesive systems. **Materials and Methods:** Standardized class I cavities (5 x 4 x 3 mm), with margins in enamel, were prepared in sound extracted human molars, according to the cavity preparation method (n=20): (1) Medium grit diamond bur; (2) Medium grit diamond bur followed by extra-fine grit diamond bur; (3) 12 blades Tungsten carbide bur and (4) 12 blades Tungsten carbide bur followed by 30 blades Tungsten carbide bur. Composite restorations were inserted in the cavities after Adper™ Scotchbond™ Multipurpose (SBMP) or Clearfil™ SE Bond (CSE) application. Specimens were submitted to 20,000 thermo-mechanical cycles. To evaluate the marginal integrity of the restorations after thermo-mechanical cycling, epoxy replicas were observed under scanning electron microscope (SEM) and the percentage of gap-free margins was analyzed. Data were statistically analyzed with non-parametric Kruskal-Wallis analysis ($\alpha=5\%$). Extra teeth, for each experimental group, were prepared, restored and immersed in 2% methylene blue solution for dye penetration analysis. **Results:** Preparation methods did not affect the marginal integrity and dye penetration of restorations bonded with SBMP ($p>0.05$). CSE exhibited the highest percentage of gap-free margins and lowest dye penetration when medium grit diamond bur was used ($p<0.05$). **Conclusion:** The cavity preparation method affected more significantly the marginal integrity and sealing ability of the self-etching adhesive. Diamond burs seemed to be the instrument of choice for obtaining better marginal quality and sealing ability for CSE.

Keywords: Dental cavity preparation, smear layer, gap formation, dentin adhesive

INTRODUCTION

Microleakage around resin composite restorations results from the formation of gaps at the joint of restoration and cavosurface margin.¹⁴ Gap formation may be related to shrinkage of resin during polymerization¹ and/or poor adhesion of bonding agents between tooth structure and composite material.²⁹ Adhesive restorations with improved marginal integrity are less inclined to marginal staining, pulpal irritation, post operative sensitivity, tooth fracture and recurrent caries which may affect the longevity of restoration and ultimately the vitality of the dental pulp.²⁹

One of the factors that can influence marginal integrity is the removal of carious dentin and cavity preparation method.²¹ To obtain an adequate bond to underlying dental substrate, smear layer (SL) must be treated prior to the application of adhesive resin^{8,26} using an acidic conditioner.

Quality and thickness of SL severely depends on types and coarseness of the cutting instrument used²⁷ and in different conditions this layer has various properties.¹⁷ Many variations in SL of prepared teeth with different dental instruments have been reported that can affect bond strength of adhesives to dentin¹⁷ and, consequently, sealing ability of resin restorations.¹⁴ Coarse diamond bur produces an irregular and a rough surface with a thicker SL when compared with other grits and Tungsten carbide bur^{3,28}, which produces a thin and smooth layer.^{5,17} Thick SL results in an increase number of closed tubules²⁰ which can interfere with bonding of adhesives once it is not easy for some resin monomers to permeate dentin smears and impregnate the underlying dentin.^{15,17}

Moreover, cavity walls roughness may influence the wettability and bonding quality of adhesive agents.⁷ The development of an adhesive bonding requires establishing intimate contact between the adhesive and tooth surface.² Thus, the use of instruments for finishing of cavity walls may influence the adhesion. Finishing procedure brings to a uniformity and regularity of the margins that eliminates the asperities and the unsubstained prisms. A smooth surface makes easier the flowing of the adhesive resin, reducing the risk to hold air bubbles that could inhibit the resin polymerization.²⁵

This *in vitro* study was designed to evaluate the effect of cavity preparation method on marginal integrity and sealing ability of composite restorations bonded to human dentin using an etch-and-rinse and a self-etching adhesives. The tested null hypothesis was that there are no differences in marginal gap formation and dye penetration at enamel/composite resin interface after different cavity preparation methods.

MATERIALS AND METHODS

Tooth Preparation

Eighty non-carious human third molars were collected after the patients' informed consent had been obtained under a protocol reviewed and approved by Institutional Review Board (COMEPE/UFC 983.135). Teeth were stored at 4°C in an aqueous solution of 0.01% thymol and used within three months after extraction. After being scaled and cleaned with slurry of pumice and water, occlusal enamel were ground flat using 320-grit wet silicon carbide paper mounted in a polisher machine (Aropol 2V – Arotec, São Paulo, SP, Brazil) under running water, without exposing of dentin surface.

Specimens were equally and randomly assigned to four groups (n= 20) and Class I standard cavities (4 mm buccolingually, 5 mm mesiodistally and 3 mm in depth) were prepared, using cavity preparation machine, according to the method of cavity preparation:

Group 1: Cavities were prepared using a cylindrical medium grit (100 µm grain size) diamond bur (#3098, KG Sorensen, Barueri, Brazil);

Group 2: The teeth were prepared as in Group 1, followed by a cylindrical extra-fine grit (15 µm grain size) diamond bur (#3098 FF, KG Sorensen, Barueri, Brazil);

Group 3: Cavities were prepared using a cylindrical 12 blades Tungsten carbide bur (#56, Beavers Dental, ON, Canada);

Group 4: The teeth were prepared as in Group 3, followed by a cylindrical 30 blades Tungsten carbide bur (#9572 FF, Beavers Dental, ON, Canada).

The occlusal margins of the cavities were located in the enamel. Each bur was mounted in air turbine using air and water spray coolant and used to cut five teeth.

Bonding procedures

The samples of each group were further divided into two subgroups (n=10) on the basis of bonding agents used: Adper™ Scotchbond™ Multipurpose (SBMP, 3M ESPE, St. Paul, USA) and Clearfil™ SE Bond (CSE, Kuraray Medical Inc, Tokyo, Japan), a three-step etch-and-rinse and a two-step self-etching adhesive system, respectively. The composition and mode of application of adhesives is presented in Table 1. Adhesives were applied according to the manufacturer's instructions, except for cavosuperficial angle conditioning, which was etching using 37% phosphoric acid (Condac, FGM, Joinville, Brazil) for 30 s, for CSE (Table 1).

After application of adhesive on the cavity, the samples were restored with a resin composite (Charisma Opal, Shade A2, Heraeus Kulzer, Hanau, Germany) using the incremental technique (2 mm-thick oblique increments). Each layer was individually polymerized for 20 s at 1200 mW/cm² (Radii-cal, SDI Limited, Victoria, Australia).

Specimens were stored in water at 37°C for 24 h and restorations were sequentially finished using a series of graded flexible discs (SofLex PopOn, 3M ESPE, St. Paul, MN, USA) under low speed. Restorations were then polished with 1200 and 2000-grit abrasive paper mounted in a polisher machine under running water (1 min, light pressure for each cycle) and fine polished using sequential wad discs and diamond paste (3, 0.5 and 0.25 µm; Buehler Ltd., Lake Bluff, USA) in order to generate a highly polished surface. The samples were submerged in an ultrasonic bath for 10 min to remove the debris over the margins.

Thermo-mechanical fatigue loading

Resin-bonded samples were then subjected to thermo-mechanical cycling (MSFT, Elquip, São Carlos, SP, Brazil) for 20,000 mechanical cycles under a load of 80 N, at a rate of 2 Hz, and thermocycling at a temperature of 5°C and 50°C with dwell time of 30 s.

After thermo-mechanical fatigue loading, impressions of the occlusal surface of the restorations were taken using individual moulds fabricated with vinyl polysiloxane putty (XT Express, 3M ESPE, St. Paul, MN, USA) and then filled with light body material (XT Express, 3M ESPE, St. Paul, MN, US). Epoxy replicas were obtained by pouring epoxy resin (Epoxy Cure Resin, Buehler Ltd., Lake Bluff, USA) into each impression of restorations.

Analysis of marginal integrity

Epoxy replicas were mounted on aluminium stubs with carbon tape (Koch Instrum Cient, São Paulo, Brazil) and were sputter-coated with gold. The enamel-composite margins of the replicas were examined under SEM (Quanta FEG 450, FEI, Amsterdam, Netherlands) at 180 x magnification. Marginal integrity was assessed according to the criteria “gap-free margin” versus “margin with gap”.⁶ The gap measurements for each specimen was recorded and percentage of “gap-free margin” after thermo-mechanical fatigue loading was calculated in relation to the total marginal interface.

Dye penetration test

Additional teeth (n= 48) were prepared and restored using same procedures for each subgroups tested according describes previously. Restored teeth were divided equally in two subgroups: immediately tested and after 20,000 thermo-mechanical cycles.

Each tooth was covered with nail varnish (Colorama, São Paulo, Brazil) except within 1 mm around margins of the restoration. The root apices sealed using sticky wax. Specimens were immersed in 2% methylene blue solution for 4 hours²⁴, removed and rinsed under running water until all the dye was removed from the surface.

The teeth were sectioned longitudinally in a buccolingual direction through of restoration using a slow-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, USA). Each section was examined under a stereomicroscope at 20× magnification to determine dye penetration along preparation walls. The most severe degree of dye penetration for each section was recorded. All measurements were made by one examiner. The following scoring criteria are used: 0- No dye penetration; 1- Dye

penetration less than half the length of lateral wall; 2- Dye penetration up to the full length of the lateral wall; and 3- Dye penetration reaching the pulpal wall.

Statistical analysis

Considering the marginal integrity assessment, the non-parametric data were analyzed using the Kruskal-Wallis one-way analysis of variance test by ranks at a significance level of $p < 0.05$. The Dunn's test was used to determine significant differences among the groups.

The scores of dye penetration were tabulated, interpreted and the results were statistically analyzed using a nonparametric Kruskal-Wallis test and Tukey's test for comparison ($\alpha = 5\%$). Regarding the dye penetration, there was a significant effect of thermomechanical loading for all groups ($p < 0.05$). The statistical analysis was performed using SigmaStat 3.5 software (Systat, Chicago, IL, USA).

RESULT

The percentages and standard deviations of gap-free margins for each group observed under SEM are shown in Table 2. These results shows that, for SBMP, there was no significant difference in all groups ($p > 0.05$), regardless the cavity preparation method used. For CSE, the highest percentage of gap-free margins was observed in the diamond burs groups (gap-free margins = 100%). There were no statistical differences ($p > 0.05$) between carbide burs. Representative SEM images are shown in Figure 1.

The spreading of dye penetration scores (%) and statistical analysis at the occlusal margins for SBMP and CSE are presented in Table 3 and 4, respectively. There were no statistical differences in dye penetration among the groups ($p < 0.05$) for the etch-and-rinse adhesive. Self-etching adhesive showed the best result (lowest

dye penetration) when preparation was performed using medium grit diamond bur, which resulted in a statistically significant difference ($p < 0.05$) when compared with carbide burs groups. All the groups showed more dye penetration after thermo-mechanical loading.

DISCUSSION

The present results demonstrated that cavity preparation method and SL characteristics had no influence on the percentage of gap-free enamel margins, after thermo-mechanical loading, for SBMP, agreeing with reported literature.^{11,13} In etch-and-rinse adhesive, phosphoric acid completely removes the SL and is also responsible for enamel and dentin demineralization,¹⁶ allowing resin infiltration through the nanometer spaces created on these substrates. This permits a strong mechanical interlocking between adhesives and tooth substrates, leading to higher bond strength.²³ For the enamel, etching might have produced similar bonding substrate in spite of distinct preparation methods. This fact may explain the similar bonding performance, regardless of preparation method, when SBMP was used.

Regarding sealing ability of SBMP, no significant differences were observed between rotary preparation groups. However, a numeric reduction in dye penetration can be observed when medium grit diamond bur and carbide bur were used. It has been reported that the roughness of dentin and enamel surfaces is increased by coarseness of the rotary instrument^{2,10} and the surface topography caused by the cutting instrument continued onto the etched surface.¹⁰ An increase in roughness results in a greater area available for bonding^{4,9} and could, therefore, increase sealing ability, thus, justifying the results obtained in the current study.

Marginal integrity and sealing ability of the self-etching adhesive were affected by method of cavity preparation. When diamond burs were used no gaps

were seen after thermo-mechanical loading (Fig. 1A) and less dye penetration was observed, although no statistical significant difference was observed in comparison to Tungsten carbide burs.

Diamond bur produces a thick, dense and rough SL^{20,27} and some researchers have reported reduced μ TBS and higher mean gap widths for self-etching adhesives when applied on thick SL substrates.^{11,12,17,18} The higher the roughness of a substrate, the more difficult it is for an self-etching adhesive solution to spread over it.¹¹

However, other studies^{11,15,19,22} related no correlation between μ TBS and marginal integrity. It has been suggested that the lack of correlation between the bond strength and the gap measurements is due to the fact that these two properties are taken from different specimens, where other variables such as C-factor and polymerization shrinkage may alter the results.⁹ Besides, in the present work, all the restorations had margins in enamel which are less affected by the method of preparation,¹⁴ at least in terms of SL production. Therefore, a reduction in μ TBS to dentin does not necessarily correlates with worse marginal adaptation. This may explain no influence of the SL thickness on gap formation¹³ or dye penetration observed in this study for CSE.

Retention, marginal and clinical dye penetration are usually the key parameters recorded to assess clinical effectiveness of adhesives.²² The self-etching adhesive evaluated in this study seemed to show a promising clinical prognosis compared to the etch-and-rinse adhesive, once it exhibited a better marginal integrity when diamond bur was used (Fig 1A and 1B), agreeing with reported literature.²²

Different from SBMP, CSE contains the functional monomer 10-MDP, which can interact chemically with the hydroxyapatite, forming a very stable nanolayered interface producing micromechanical and chemical bonding to the dental substrates.³¹

This chemical bonding promoted by 10-MDP is more effective and also more stable in water than that provided by other functional monomers like 4-MET and phenyl-P.³⁰

The influence of cavity preparation method on the marginal integrity and sealing ability were dependent of the adhesive used once it affected more significantly the self-etching adhesive. Within the limits imposed in the experimental design, diamond burs seemed to be the instrument of choice for obtaining higher marginal integrity and lower dye penetration of resin-tooth interface produced by resin composite and CSE adhesive.

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Clinical relevance: Care should be taken in selecting proper instrument for each adhesive, as the best marginal integrity and sealing ability was observed for self-etching adhesive bonded to cavity prepared by diamond bur.

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Table 1 - Materials compositions, pH and mode of application

MATERIAL	COMPOSITION (*)	pH PRIMER/ PHOSPHORIC ACID	MODE OF APPLICATION		
			Acid etching	Primer application	Adhesive application
Adper™ Scotchbond™ Multipurpose (3M ESPE, St. Paul, USA) Batch # N295761	Primer: HEMA, solvents Adhesive: HEMA, bis- GMA	<0.5	Apply 37% phosphoric acid to enamel for 30 s and to dentin for 15 s. Rinse for 30 s. Excess of water was removed with absorbing paper.	Apply the primer for 20 s with agitation. Gently air blow for 5 s.	Apply the adhesive and light cure for 10 s.
Clearfil™ SE Bond (Kuraray Medical, Tokyo, Japan) Batch # 01147A	Primer: 10-MDP, HEMA, tertiary amine, hydrophilic DMA, water Adhesive: 10-MDP, HEMA, bis-GMA, tertiary amine, hydrophilic DMA, silanized colloidal silica, photoinitiator.	1.5	Apply 37% phosphoric acid to enamel for 30 s. Rinse for 30 s. Excess of water was removed with air blow.		Apply the adhesive, gently air blow and light cure for 10 s.
Charisma™ Opal (Heraeus Kulzer, Hanau, Germany) Batch # 010024	Bis-GMA, barium aluminium glass, silica.	—	Apply to the cavity in thin layers (max. 2 mm) and adapt carefully to the cavity walls. Light cure for 20 s.		

Abbreviations: HEMA= 2-hydroxyethylmethacrylate; BisGMA= bisphenol glycidyl methacrylate; 10-MDP= 10-methacryloyloxydecyl dihydrogen phosphate; DMA= Dimethacrylate.

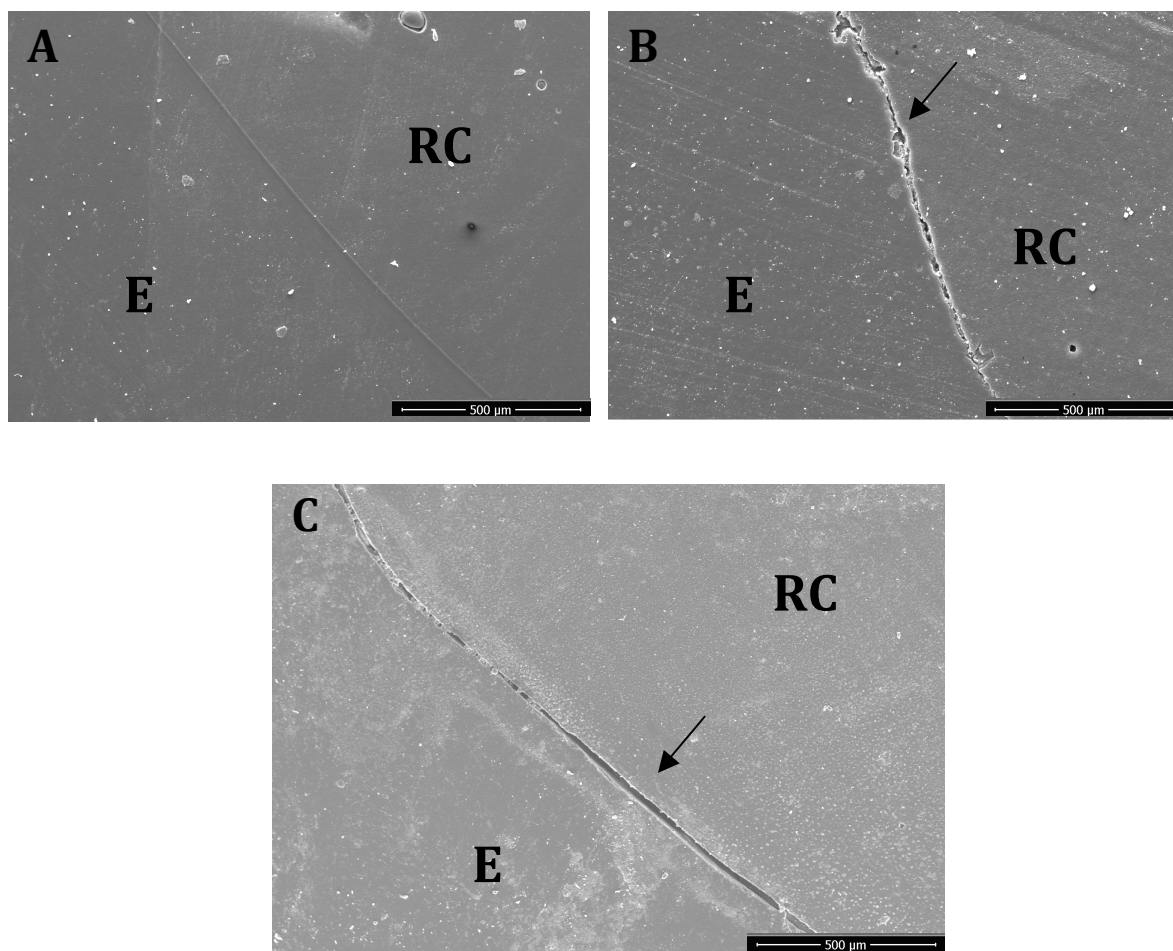


Figure 1 - SEM micrographs (180 X) of epoxy resin replicas reproduced from impressions of Class I cavities prepared with different cavity preparation methods. **A.** Replica of CSE with 'gap-free margin' of diamond bur group, after thermo-mechanical loading. **B.** Gap formation (arrow) between SBMP and enamel of diamond bur group, observed after thermo-mechanical loading. **C.** Gap formation (arrow) between resin composite and enamel, observed in carbide bur/CSE group. (RC: resin composite; E: enamel).

Table 2 – Percentage of ‘gap-free margin’ and statistical analysis for each experimental group (*).

<i>Groups</i>	SBMP	CSE
Medium grit diamond bur	92.6 (9.7) B	100 (0.0) A
Medium grit + extra-fine grit diamond bur	93.5 (6.5) B	100 (0.0) A
12 blades Tungsten carbide bur	97.2 (3.8) B	98.9 (1.1) B
12 blades Tungsten carbide bur + 30 blades Tungsten carbide bur	98.4 (2.2) B	93.4 (7.1) B

*Different letters indicate statistical difference (p<0.05).

Table 3 – Spreading on the dye penetration scores (%) for SBMP, as well as, statistical results (SR).

GROUPS	DYE PENETRATION SCORES (%)								SR
	Immediate*				Aged**				
	0	1	2	3	0	1	2	3	
Medium grit diamond bur	53	37	5	5	45	18	9	28	B
Medium grit + extra-fine grit diamond bur	50	30	10	10	10	57	19	14	AB
12 blades Tungsten carbide bur	55	33	12	0	41	43	8	8	B
12 blades Tungsten carbide bur + 30 blades Tungsten carbide bur	35	31	30	4	30	30	15	25	AB

Same letters are statistically similar ($p > 0.05$). For all aged (**) groups was observed more dye penetration than immediate (*) groups ($p < 0.05$).

Table 4 – Spreading on the dye penetration scores for CSE, as well as, statistical results (SR).

GROUPS	DYE PENETRATION SCORES (%)								SR
	Immediate*				Aged**				
	0	1	2	3	0	1	2	3	
Medium grit diamond bur	65	15	20	0	58	25	17	0	C
Medium grit + extra-fine grit diamond bur	36	45	19	0	29	33	12	26	BC
12 blades Tungsten carbide bur	18	32	41	9	18	14	32	36	AB
12 blades Tungsten carbide bur + 30 blades Tungsten carbide bur	30	45	5	20	4	26	26	44	AB

Same letters are statistically similar ($p > 0.05$). For all aged (**) groups was observed more dye penetration than immediate (*) groups ($p < 0.05$).

4 DISCUSSÃO GERAL

De forma geral, os resultados desse estudo reforçam os achados relatados na literatura de que a qualidade e quantidade da *smear layer* (SL) dependem do método de preparo cavitário (OGATA et al., 2001; DIAS et al., 2004; KOASE et al., 2004), uma vez que foi possível detectar características distintas em relação a espessura e rugosidade de SL nos diferentes métodos utilizados. Pontas diamantadas produziram uma superfície dentinária mais irregular e SL mais espessa quando comparada a preparos cavitários nos quais brocas carbides foram utilizadas, corroborando com os achados reportados na literatura (OLIVEIRA et al., 2003; HOSOYA et al., 2004; BARROS et al., 2005).

Vários autores reportam (TAGAMI et al., 1991; INOUE et al., 2001; OGATA et al., 2001; KOIBUCHI; YASUDA; NAKABAYASHI, 2001; OLIVEIRA et al., 2003; HOSOYA et al., 2004) que a espessura e a rugosidade da SL afetam os valores de RU de sistemas adesivos à dentina. Os resultados do presente estudo confirmam os achados prévios (YIU, et al., 2008) para o sistema autocondicionante, uma vez que CSE apresentou melhor RU quando utilizado sobre SL mais fina e regular, produzida pela broca carbide de 30 lâminas.

Por outro lado, SBMP alcançou maiores valores de RU em cavidades preparadas com ponta diamantada de média granulação, confirmando relatos de que a espessura da SL tem pouca influência na RU de sistemas adesivos de condicionamento total (TAY et al., 2000; TANI e FINGER, 2002; REIS et al. 2005; KESHIMA et al., 2005), visto que estes sistemas utilizam ácido fosfórico para condicionamento do substrato dentinário e remoção total da SL (PRATI et al., 1998; PERDIGÃO e LOPES, 1999; OLIVEIRA et al., 2003). No entanto, no presente estudo, a rugosidade do substrato dentinário pareceu influenciar os valores de RU para o SBMP. O aumento da rugosidade da dentina promoveu maior área de superfície para adesão da resina ao substrato dentinário e, conseqüentemente, maiores valores de RU foram obtidos (JUNG; WEHLEN; KLIMEK, 1999; AYAD; JOHNSTON; ROSENSTIEL, 2009).

Embora, no presente estudo, o método de preparo cavitário tenha afetado a RU à dentina dos dois sistemas adesivos, este teve influência mais evidente no selamento e adaptação marginal da interface esmalte/restauração produzida pelo sistema

autocondicionante. A adaptação e selamento marginal da interface de união SBMP/esmalte não foram influenciados pelo instrumento rotatório utilizado, reforçando resultados descritos em estudos anteriores (KESHIMA et al., 2005; MALEKIPOUR; SHIRANI; TAHMOURESPOUR, 2010). Por outro lado, o CSE apresentou melhor performance quando associado a pontas diamantadas, demonstrando a não correlação entre espessura de SL *versus* formação de fendas/microinfiltração marginal (LOGUERCIO et al., 2006).

Comparando os sistemas adesivos, o CSE apresentou melhor qualidade marginal em relação ao SBMP, quando utilizado com pontas diamantadas. Isto pode ser explicado pelo fato de o CSE possuir o monômero funcional 10-MDP que interage quimicamente com a hidroxiapatita do substrato dentário, promovendo interfaces de união mais estáveis do que as produzidas por outros monômeros resinosos, como o 4-META, por exemplo (YOSHIDA et al., 2000; YOSHIDA et al., 2004).

Os achados deste estudo fornecem evidências de que diferentes métodos de preparo cavitário produzem SL com padrões micromorfológicos distintos, bem como substrato dentinário com diferentes características que podem afetar o desempenho da interface de união dos sistemas adesivos. De forma geral, o preparo da cavidade com broca carbide em dentina e com ponta diamantada para acabamento das paredes em esmalte parece ser o método mais indicado para obtenção de interfaces de união com integridade marginal e resistência adesiva satisfatórias para os sistemas autocondicionantes. Para os sistemas de convencionais, a ponta diamanta de média granulação é o instrumento rotatório cortante de eleição. Diante disso, os resultados obtidos neste estudo podem colaborar para a seleção do método de preparo cavitário mais adequado para cada sistema adesivo, a fim de otimizar a obtenção de interfaces de união dente/restauração mais resistentes e duradouras.

5 CONCLUSÃO GERAL

Com base nos resultados desta tese, pode-se concluir que:

- Diferentes métodos de preparo cavitário produzem *smear layer* com padrões micromorfológicos distintos, em relação a espessura e rugosidade, bem como diferentes características do substrato dentinário que afetam a RU de sistemas adesivos à dentina;
- O método de preparo cavitário afetou, de forma mais evidente, a adaptação e o selamento marginal de restaurações aderidas com adesivo autocondicionante, comparando-se ao sistema adesivo convencional.

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