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PROGRAMA DE PÓS-GRADUAÇÃO EM ODONTOLOGIA
MESTRADO EM ODONTOLOGIA**

LÍVIA DE OLIVEIRA BARROS

**ESTABILIDADE DA INTERFACE DE UNIÃO DE SISTEMAS ADESIVOS
CONVENCIONAIS APLICADOS À DENTINA SATURADA COM ALCOÓIS**

**Fortaleza
2011**

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Dissertação de Mestrado a ser apresentada ao Programa de Pós-Graduação em Odontologia da Faculdade de Farmácia, Odontologia e Enfermagem da Universidade Federal do Ceará como requisito parcial para a obtenção do título de Mestre em Odontologia.

Área de Concentração: Clínica Odontológica

Orientador: Prof. Dr. Vicente de Paulo Aragão Saboia

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DEDICATÓRIA

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“Nós devemos ser a mudança que queremos ver no mundo.”

Mohandas K. Gandhi

RESUMO

Existe um consenso de que a instabilidade da interface de união resina-dentina está relacionada com a quantidade de monômeros hidrofílicos presentes no sistema adesivo. Dessa forma, interfaces adesivas mais duradouras seriam alcançadas com a utilização de sistemas adesivos mais hidrofóbicos. Uma maneira de unir monômeros hidrofóbicos à dentina é através da saturação da matriz dentinária desmineralizada por etanol ao invés de água (ethanol-wet bonding technique). O objetivo deste trabalho *in vitro* foi avaliar o efeito do protocolo de saturação dentinária, utilizando alcoóis, na estabilidade da interface de união produzida por sistemas adesivos convencionais de dois passos. Para isso, foram utilizados 20 terceiros molares humanos, que tiveram a dentina coronária exposta e foram distribuídos aleatoriamente em quatro grupos experimentais (n=5): Adper Single Bond 2 (SB) aplicado sobre dentina saturada com água ou etanol; e XP Bond (XP) aplicado sobre dentina saturada com água ou tert-butanol. O protocolo de saturação da dentina foi realizado através da aplicação de 2 µl de etanol 100% ou tert-butanol 99,5% diretamente sobre a dentina por um período de 60 s. Um platô de resina composta foi confeccionado sobre superfície dentinária após a aplicação do sistema adesivo e os espécimes foram cortados pela técnica *non-trimming* de obtenção de palitos para o teste de microtração. Os palitos de cada dente foram igualmente divididos em dois subgrupos: imediatamente testado ou envelhecido em solução de NaOCl a 10% por 1 hora. Os espécimes foram tracionados até a ruptura da união a uma velocidade de 1 mm/min e sua força de união mensurada. Os palitos fraturados foram analisados e classificados de acordo com o modo de fratura em mista, adesiva, coesiva em dentina e coesiva em compósito, e os valores expressos em porcentagem. Os valores de resistência de união foram estatisticamente analisados usando os testes ANOVA a três critérios e Tukey ($\alpha=0.05$). Discos extras de dentina foram submetidos aos procedimentos adesivos de cada grupo testado e utilizados para investigar as características morfológicas da interface de união através de microscopia óptica após imersão em solução amoniacal de nitrato de prata. Os resultados do teste de microtração mostraram que a imersão em solução de NaOCl reduziu significativamente a força de união em comparação aos grupos controle ($p<0.05$) e aumentou a nanoinfiltração das interfaces adesivas de todos os sistemas adesivos testados. A saturação da dentina com etanol 100% reduziu os valores de resistência adesiva para SB quando comparado ao grupo controle, e esta mesma tendência foi observada na infiltração de prata. O uso do tert-butanol 99,5% não afetou os valores de resistência adesiva para o XP, assim como a deposição de nitrato de prata na interface adesiva.

Conclusão: Os protocolos simplificados de saturação da dentina com alcoóis utilizados no presente estudo não foram capazes de melhorar a estabilidade da interface de união para sistemas adesivos convencionais de dois passos.

Palavras-chave: adesivos dentinários, resistência adesiva, etanol, solvente.

ABSTRACT

There is a consensus that the resin-dentin bond instability is correlated with increased hydrophilic resin monomer content in dentin adhesive. Thus, more durable bonds could be created when more hydrophobic resins were used. One way to bond hydrophobic monomers to acid-etched dentin is saturating the demineralized dentin with ethanol instead of water (ethanol-wet bonding technique). The objective of this *in vitro* study was to evaluate the effect of “alcohol wet bonding” technique on the stability of adhesive interface produced by two-step etch-and-rinse adhesives systems. For this, it was used 20 human third molars that had superficial dentin exposed and were randomly divided into four experimental groups (n=5): Adper Single Bond 2 (SB) bonded to acid-etched dentin saturated with water or ethanol; and XP Bond (XP) bonded acid-etched dentin saturated with water or 99.5% tert-butanol. The simplified dentin dehydration protocol was performed using 2 μ l 100% ethanol or 99.5% tert-butanol directly applied in dentin for 60 s. Composite build-ups were built on dentin surface and specimens were cut into non-trimming dentin-composite beams to microtensile testing. Beams from each tooth were divided equally in two subgroups: immediately tested and aged by immersion in 10% NaOCl solution for 1 h. Specimens were pulled until failure at crosshead speed of 1 mm/min and bond strength was calculated. Fractured sticks were analyzed and classified according to the failure mode as mixed, adhesive, cohesive in dentin and cohesive in composite; and expressed in percentage. Data from μ TBS test were statistically analyzed using three-way ANOVA and Tukey tests ($\alpha=0.05$). Additional dentin disks were bonded using the same groups tested and the morphological characteristics of adhesive interface were investigated by light microscopy after immersion in ammoniacal silver nitrate solution. μ TBS results showed that NaOCl solution reduced significantly bond strength comparing with the control groups ($p<0.05$) and increased the silver nitrate interfacial deposit for all adhesives tested. SB used in ethanol saturated-dentin showed significant lower bond strength values in comparison with SB control group, and this same tendency was observed in the silver nitrate deposition. The use of tert-butanol did not influence XP bond strengths values and silver nitrate deposits.

Conclusion: The “alcohol wet bonding” simplified technique used in the present study was not able to improve resin-dentin bond stability for simplified etch-and-rinse adhesive systems.

Keywords: dentine-bonding agents, bond strength, ethanol, solvent.

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Introdução Geral

1. INTRODUÇÃO GERAL

A odontologia adesiva é caracterizada pelo uso de materiais resinosos que tiveram seu aperfeiçoamento com a técnica de condicionamento ácido proposto por Buonocore em 1955. Desde então vários estudos têm sido desenvolvidos com a finalidade de aperfeiçoar a adesão do material restaurador ao substrato dentário (VAN MEERBEEK *et al.*, 2003). Embora a adesão ao esmalte dentário seja descrita com alto índice de sucesso clínico, a adesão à dentina ainda é considerada deficiente, principalmente a longo prazo, devido às complexas características morfofuncionais desse tecido (PASHLEY *et al.*, 2004).

A durabilidade da interface dentina/resina é de extrema importância para a longevidade das restaurações adesivas, já que a degradação desta interface pode enfraquecer a resistência de união (CARRILHO *et al.*, 2004) e ocasionar problemas clínicos indesejáveis como sensibilidade pós-operatória, infiltração marginal, perda de retenção da restauração e o aparecimento de cáries secundárias (BRESCHI *et al.*, 2008; GOING, 1972). A estabilidade da interface de união é possível a partir da criação de uma camada híbrida compacta e homogênea. Dessa forma, idealmente, o substrato dentinário, previamente desmineralizado, deve ser completamente infiltrado pelo adesivo, evitando áreas de incompleta impregnação da matriz dentinária (BRESCHI *et al.*, 2008).

No conceito atual de adesão à dentina, a água desenvolve função essencial na prevenção do colapso da rede de fibrilas colágenas, expostas pelo condicionamento ácido prévio, permitindo a infiltração dos monômeros resinosos na intimidade do tecido dentinário e a formação da camada híbrida (SHIN *et al.*, 2009). A água é o solvente com melhores características para manter a trama de colágeno expandida em função da sua capacidade de romper pontes de hidrogênio formadas entre os peptídeos das fibrilas quando a dentina é desidratada e manter as fibrilas em seu grau máximo de expansão (PASHLEY *et al.*, 2007).

Embora a água desenvolva um papel crítico na formação da camada híbrida, a sua presença no interior da interface de união pode dificultar a interdifusão de

monômeros hidrófobos presentes no sistema adesivo, insolúveis em água, resultando na formação de uma interface adesiva deficiente. Há ainda que se considerar que a água presente durante a hibridização da dentina é impossível de ser eliminada por completo pelos procedimentos clínicos usuais (YIU *et al.*, 2005), podendo se tornar um fator de comprometimento da polimerização do adesivo. Esse conjunto de fatores favorece a degradação precoce da camada híbrida através de um efeito combinado de hidrólise de seus componentes resinosos e por um processo hidrolítico/enzimático das fibras colágenas expostas pelo condicionamento ácido e não totalmente protegidas pela resina (HASHIMOTO *et al.*, 2000; PASHLEY *et al.*, 2004; CARRILHO *et al.*, 2005; LOGUERCIO *et al.*, 2005).

A fim de promover uma melhor infiltração dos monômeros resinosos na dentina, reduzir a sensibilidade do sistema adesivo à intrínseca umidade dentinária (TAY, PASHLEY, 2003) e minimizar a separação de fase na interface adesiva, monômeros hidrofílicos são adicionados a composição dos sistemas adesivos atuais (SPENCER, WANG, 2002). Entretanto, estudos têm demonstrado que quanto maior a hidrofiliabilidade do sistema adesivo, maior a sorção de água pela interface adesiva (UNEMORI *et al.*, 2003; YIU *et al.*, 2006), mais rápida a deterioração das suas propriedades mecânicas (YIU *et al.*, 2004; ITO *et al.*, 2005) e, conseqüentemente, menor a longevidade clínica da restauração (YE *et al.*, 2008; YE *et al.*, 2009). Em estudo realizado por Tay *et al.* (2002), observou-se que sistemas adesivos simplificados, que não possuem a aplicação de uma camada de adesivo hidrofóbico puro, comportaram-se como membranas semipermeáveis após polimerização, permitindo a passagem de água pela interface adesiva. Diante disso, esforços (TAY *et al.*, 2007; SAURO *et al.*, 2010) têm sido concentrados no sentido de desenvolver uma técnica capaz de otimizar a união de adesivos mais hidrófobos à dentina, promovendo assim, a obtenção de restaurações mais estáveis.

O uso de solventes anidros, como o etanol, tem sido proposto como alternativa para saturar a dentina desmineralizada em substituição à água (ethanol-wet bonding technique) (PASHLEY *et al.*, 2007; TAY *et al.*, 2007; SADEK *et al.*, 2008). O etanol apresenta a capacidade de re-expandir ou manter a matriz de colágeno expandida, ainda que de modo mais lento e em menor escala quando comparado à água (PASHLEY *et al.*, 2001; AGEE *et al.*, 2006). Além disso, na presença de etanol,

monômeros mais hidrófobos se dissolvem com maior facilidade e a difusão é favorecida por toda a extensão da zona desmineralizada (BECKER *et al.*, 2007), minimizando a existência de fibrilas de colágeno expostas e não protegidas na base da camada híbrida.

Essa estratégia envolve a lenta substituição da água, presente no interior da matriz colágena desmineralizada, através de uma desidratação química, utilizando concentrações ascendentes de etanol (SADEK *et al.*, 2010). Teoricamente, com essa técnica, a matriz colágena desmineralizada torna-se menos hidrofílica e mais receptiva aos monômeros hidrofóbos, reduzindo a separação de fase (WANG, SPENCER, 2003) na interface de união. Além disso, a técnica de saturação do substrato dentinário com o etanol permite uma melhor infiltração do agente de união na trama de colágeno, visto que, a maioria dos monômeros hidrófobos são solúveis em etanol (SHIN *et al.*, 2009), tornando o substrato dentinário mais compatível com os monômeros resinosos contidos nos sistemas adesivos.

Outro fator relevante e que participa em certo grau do processo de adesão, é o enrijecimento das fibrilas quando da aplicação de etanol puro ou misturas de HEMA/ etanol, resultando em menor contração da mesma quando o solvente é evaporado (GARCIA *et al.*, 2005; BECKER *et al.*, 2007). Dessa forma, os espaços interfibrilares são mantidos mesmo após a evaporação do solvente, favorecendo a infiltração posterior da resina. Além disso, a dentina submetida à saturação com etanol apresenta um aumento do espaço interfibrilar quando comparada à dentina saturada com água (HOSAKA *et al.*, 2009). Esse fato poderia promover uma maior quantidade de resina ao redor das fibrilas colágenas (HOSAKA *et al.*, 2009) e, conseqüentemente, maiores valores de resistência adesiva quando comparada a técnica convencional (NISHITANI *et al.*, 2006).

Embora a técnica de saturação com etanol pareça promissora (SADEK *et al.*, 2008; PASHLEY *et al.*, 2007; TAY *et al.*, 2007), esta envolve um passo extra na substituição da água por etanol 100%. Além disso, a técnica descrita por alguns estudos *in vitro* (SADEK *et al.*, 2008; SADEK *et al.*, 2010) requer a utilização de um protocolo com aplicações de etanol em concentrações ascendentes na superfície dentinária, consumindo um tempo clínico de aproximadamente 3-4 min (SADEK *et*

al., 2010), para a obtenção de resultados satisfatórios, o que dificulta a viabilidade clínica dessa técnica. Por esta razão, protocolos de desidratação mais simplificados têm sido utilizados (HOSAKA *et al.*, 2009; SADEK *et al.*, 2010). Entretanto, estudos recentes têm observado, que curtos períodos de saturação podem não ser suficientes para a completa substituição da água, presente no interior da matriz dentinária desmineralizada, pelo etanol. Dessa forma, a presença de água residual na dentina (OSORIO *et al.*, 2010; SADEK *et al.*, 2010) pode resultar na pobre infiltração dos monômeros hidrófobos (SADEK *et al.*, 2010) presentes nos sistemas adesivos. Nesse caso, o uso de um adesivo contendo monômeros hidrofílicos poderia auxiliar na melhor infiltração do adesivo na dentina desmineralizada, diminuir a permeabilidade em comparação a um adesivo hidrofóbico (SAURO *et al.*, 2009; CADENARO *et al.*, 2009) e aumentar a resistência de união imediata à dentina (NISHITANI *et al.*, 2006).

Os monômeros resinosos dos sistemas adesivos são misturados a solventes, tais como: água, etanol, acetona ou por uma combinação destes (VAN MEERBEEK *et al.*, 2003). XP Bond (Dentsply, Konstanz, Germany) é um sistema adesivo convencional de dois passos, desenvolvido recentemente, que possui o álcool tert-butanol como solvente. Tem sido relatado que esse adesivo tem a capacidade de penetrar a matriz colágena colapsada, resultando em menor sensibilidade técnica e melhor desempenho clínico (BLUNCK, KNITTER, JANH, 2007). Contudo, não existem estudos que relatem o uso desse sistema adesivo associado à técnica de saturação da matriz dentinária com o seu próprio solvente (tert-butanol).

Ainda que os benefícios do emprego do etanol para a qualidade de união tenham sido relatados na literatura, a combinação dessa técnica com adesivos simplificados disponíveis comercialmente, ainda não foi amplamente investigada, principalmente, com relação à estabilidade da interface adesiva. Dessa forma, maiores evidências em relação a essa estratégia, no que diz respeito ao protocolo de desidratação, tipo de solvente utilizado, o tempo de aplicação e a durabilidade da interface adesiva obtida através dessa técnica, são necessárias para justificar a indicação desse passo clínico extra.

Proposição

2. PROPOSIÇÃO

O presente trabalho teve como objetivos:

2.1 Objetivo Geral

- Avaliar *in vitro* o efeito do protocolo de saturação dentinária, através do uso de alcoóis (etanol ou tert-butanol), na estabilidade da interface de união de sistemas adesivos convencionais de dois passos.

2.2 Objetivos Específicos

- Avaliar a resistência de união à microtração da interface de união de sistemas adesivos convencionais de dois passos quando aplicados na dentina saturada com etanol ou tert-butanol e submetida a envelhecimento artificial;
- Avaliar, através de microscopia óptica, a infiltração de prata nas interfaces adesivas.

Capítulo

3. CAPÍTULO

Esta dissertação 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. Por se tratar de pesquisas envolvendo seres humanos, ou parte deles, o projeto de pesquisa foi submetido à apreciação do Comitê de Ética em Pesquisa, tendo sido aprovado (Anexo 1). Assim sendo, esta dissertação é composta de um artigo científico que será submetido ao periódico *Operative Dentistry*, conforme descrito abaixo:

Stability of interface created by simplified etch-and-rinse adhesives to alcohol-saturated acid-etched dentin

BARROS LO, APOLONIO FM, LOGUERCIO AD & SABOIA VPA

Stability of interface created by simplified etch-and-rinse adhesives to alcohol-saturated acid-etched dentin

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SHORT TITLE: Stability of adhesive interfaces under alcohol-wet bonding technique

CLINICAL RELEVANCE: The “alcohol wet bonding” simplified technique used in the present study was not able to improve resin-dentin bond durability for simplified etch-and-rinse adhesive systems.

ABSTRACT

Purpose: To evaluate the effect of “alcohol wet bonding” technique on durability of the adhesive interface produced by two-step etch-and-rinse adhesives systems.

Materials and Methods: Twenty human third molars had superficial dentin exposed and were randomly divided into four experimental groups: Adper Single Bond 2 (SB) bonded to acid-etched dentin saturated with: water (control) or ethanol; and XP Bond (XP) bonded acid-etched dentin saturated with water (control) or tert-butanol. A simplified dentin dehydration protocol was performed using 100% ethanol or 99.5% tert-butanol directly applied in dentin for 60 s. Composite build-ups were built on dentin surface and specimens were cut into *non-trimming* dentin-composite beams. Beams from each tooth were divided equally in two subgroups: immediately tested and aged by immersion in 10% NaOCl solution for 1 h. Specimens were pulled until failure at crosshead speed of 1 mm/min and pattern failure was evaluated. Data were statistically analyzed with three-way ANOVA and Tukey tests ($\alpha=0.05$). Additional dentin disks were bonded using the same groups tested and investigated by light microscopy after immersion in ammoniacal silver nitrate solution.

Results: SB control group showed significant higher bond strength values in comparison with SB used in ethanol saturated-dentin ($p<0.05$), and this same tendency was observed in the silver nitrate deposition. The use of tert-butanol did not influence XP bond strengths values ($p>0.05$) and silver nitrate deposits. NaOCl solution significantly reduced bonding of all groups tested ($p<0.05$) and also increasing the silver nitrate interfacial deposit.

Conclusion: The “alcohol wet bonding” simplified technique used in the present study was not able to improve resin-dentin bond durability for simplified etch-and-rinse adhesive systems.

Keywords: bond strength, ethanol-wet bonding, solvent, dentine.

INTRODUCTION

In the water wet bonding technique, after acid etching, the presence of water in the demineralized dentin is essential to prevent the collapse of the dentin matrix. The space that is occupied by the water surrounding the non-collapsed collagen fibrils can then be infiltrated by the resin monomers to form the hybrid layer.¹

However, the presence of residual water in the current wet bonding technique² is a potential factor for phase separation of the hydrophobic monomers which has limited water solubility.¹ Traditionally, hydrophilic resin monomers are used in the contemporary dentin adhesives to enhance their wetting properties and to avoid phase separation when hydrophobic dimethacrylates are added to water.³ Nevertheless, it has been reported that the current generation of dentin adhesives has been criticized as being too hydrophilic⁴ and absorbing too much water following polymerization,⁵ which lowers their stiffness when compared with that of more hydrophobic resins.⁶ This increased water sorption can result in the accelerated degradation and decreased mechanical properties that affect the long-term survival of the interface and associated restoration.^{7,8} Presumably, if dentin is bonded with more hydrophobic resins, the interface would absorb less water and produce more durable bonds.⁵

Recent studies have shown that it is possible to bond hydrophobic resin monomers to acid-etched dentine^{9,10} with a new technique called “ethanol wet bonding”.¹⁰ This technique involves slowly replacing water within the demineralized collagen matrix with ascending concentrations of ethanol, allowing the latter to penetrate the collagen matrix without causing additional shrinkage of the interfibrillar spaces.¹¹ The ethanol saturated dentine is less hydrophilic and prevents phase separation of hydrophobic resin monomers.¹² Moreover, because most hydrophobic monomers are also soluble in ethanol, it is possible to use an adhesive with a higher ratio of hydrophobic to hydrophilic monomers.¹ When acid etched dentin was saturated with 100% ethanol instead of water, the bond strengths of both hydrophilic and hydrophobic resin increased significantly.^{5,13}

Although “ethanol wet bonding” looks promising,^{9,10,14} the protocol is time consuming and technique sensitive.¹¹ The ethanol saturation protocol is performed by use of ascending concentration of ethanol over a period of 3 to 4 minutes¹¹ which is clinically unrealistic.¹⁵ For this reason, simplified ethanol dehydration protocols were used.^{11,13} On the other side, short periods of ethanol saturation could not be

enough for complete replacement of water within the acid-etched intertubular dentine and the dentinal tubules by ethanol. Most likely, residual water present in within the acid-etched dentin^{11,15} can result in poor wetting by the hydrophobic adhesive.¹¹ In this case, the use of a hydrophilic monomer could help the infiltration of the adhesive on dentin and decreasing the permeability in comparison with hydrophobic adhesive,^{16,17} as well as, increasing the immediate bond strength to dentin.⁵ To extent of our knowledgement, there is no studies that evaluated this issue regarding immediately and aging bond strength on commercial simplified adhesive system.

The monomers of adhesive systems are carried by a solvent which is usually water, ethanol, acetone or a combination of those.¹⁸ Recently, it was formulated a two-step etch-and-rinse adhesive (XP Bond, Dentsply) which uses the alcohol tert-butanol as the solvent. It is claimed to be less technique sensitive due to an improved ability to diffuse through partially collapsed demineralized dentin.¹⁹ Nevertheless, there is no studies using this adhesive system on dentin saturated with its solvent. Thus, more evidence in relation to ethanol wet bonding regarding to water replacement protocol, time application and bonds durability are needed to justify this extra step.

The present study was performed to evaluate the effect of “alcohol wet bonding” technique on the durability of adhesive interface produced by two-step etch-and-rinse adhesives systems on alcohol (ethanol or tert-butanol) saturated dentin, immediately after bonding and after artificial ageing in 10% NaOCl, by microtensile bond strength evaluation and silver nitrate uptake. The null hypothesis tested was there is no difference in adhesive performance and stability between the use of “alcohol wet bonding” technique and water wet bonding technique.

METHODS & MATERIALS

Tooth Preparation

Twenty recently extracted human non-carious third molars were used under a protocol approved by Institutional Review Board. The teeth were stored in 0.01% thymol solution at 4° C and used within 2-month after extraction. After being cleaned and pumiced, tooth crowns were flattened using a low-speed diamond saw under water irrigation (Isomet, Buehler Ltd., Lake Bluff, USA), and a standardized smear layer was produced on the exposed coronal dentin using 600-grit wet silicon carbide

paper mounted in a polisher machine (Aropol 2V – Arotec, São Paulo, SP, Brazil). Each tooth was etched with 35% phosphoric acid gel (3M ESPE, St Paul, MN, USA) for 15 s and rinsed thoroughly with distilled water for 15 s. Excess water was removed from the surface with absorbing paper (Melitta, São Paulo, SP, Brazil) and the dentin was kept visibly moist.

Bonding procedures

Specimens were equally and randomly assigned to 4 groups (n=5) and treated with one of the two adhesives: Adper Single Bond 2 (SB; 3M ESPE, St Paul, MN USA) and XP Bond (XP; Dentsply DeTrey, Konstanz, Germany), containing ethanol or tert-butanol solvents, respectively. In the control groups, SB and XP were applied according to the manufacturer's instructions to visibly moist water-saturated demineralized dentin in which was applied 2 µl of distilled water for 60 s (Table 1). In the specimens of the experimental groups, the water-moist dentin surfaces were saturated with 2 µl of 100% ethanol (Vetec, Rio de Janeiro, RJ, Brazil) for SB and 99,5% tert-butanol (Vetec, Rio de Janeiro, RJ, Brazil) for XP. The solution was directed applied in dentin for 60 s. Composite build-ups were constructed with a light-cured resin composite (Filtek Z250, 3M ESPE, St Paul, MN, USA) in four 1mm-thick increments and individually polymerized for 20 s each, using a halogen light-curing unit (Eliopar Freelight 2, 3M ESPE, St Paul, MN, USA) with an output intensity of 650 mW/cm².

Testing procedures

The bonded specimens were serially sectioned in both x and y directions across the adhesive interface with a diamond blade in an Isomet saw (Isomet, Buehler Ltd., Lake Bluff, USA) under water cooling to obtain 1mm-thick beams, in accordance with the microtensile test *non-trimming* technique.

After being stored for 24 h in distilled water at 37°C, beams from each tooth were divided equally into two subgroups. One subgroup was tested immediately, while the other was stored in 10% NaOCl for 1 h at room temperature.

Microtensile testing

All specimens were extensively rinsed under tapping water, individually measured with a digital caliper (Absolute Digimatic, Mitutoyo, Tokyo, Japan) and stressed until failure under tension using a simplified microtensile testing machine (Microtensile Tester, Bisco, Schaumburg, IL, USA) at a crosshead speed of 1

mm/min. The number of prematurely debonded sticks per group during specimen preparation was also recorded.

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

Light microscopy

Eight additional teeth (n=1 for subgroup) were processed for interfacial silver nitrate evaluation. Specimens were prepared as previously described in the item bonding procedure and, after that, specimens were then cut into 4 transverse sections, 1mm-thick, to expose the hybrid layer. Bonded specimens were stored for 24 h in distilled water or 1 h in 10% NaOCl solution.^{21,22} The slices were covered with nail varnish, leaving 1 mm exposed at the interface and immersed in 50 wt% ammoniacal silver nitrate (AgNO₃) solution (pH 9.5) according to the protocol described by Tay *et al.* (2002).²⁰ After immersion in the trace solution, specimens were then thoroughly rinsed in distilled water and were immersed in photodeveloping solution.

The silver nitrate-impregnated specimens were fixed on glass slides using cyanoacrylate glue (Super Bonder flex gel, Henkel Ltd, Düsseldorf, Germany) and flattened with SiC papers on increasing fine grits (600, 800, 1200 and 2400) in a polisher under running water. Images of all interfaces at 400x magnification under light microscopy (Nikon Eclipse E 800, Nikon, Tokyo, Japan) were obtained and silver nitrate representative images were chosen for each tested group.

Statistical analysis

Bond strength data were collected and analyzed using SPSS 17.0 (SPSS, Chicago, IL, USA). As the normality (Kolmogorov-Smirnov test) assumption of the data appeared to be valid, a three-way ANOVA was employed for statistical analysis to examine the effects of adhesive system, storage period and dentin treatment, and interaction of these three factors on tensile bond strength. Tukey's test was employed for multiple comparisons ($\alpha=0.05$). Silver nitrate evaluation was only performed qualitatively.

RESULTS

Microtensile bond strength evaluation

Number of specimens and failure mode, as well as, means and standard deviations of microtensile bond strength, are shown in table 2 and 3, respectively. The number of prematurely debonded sticks per test group affected all groups to a similar extent and all premature failures occurred during the cutting procedure. For this reason, this report was not included in the statistical analysis.

Examination of the fractures surfaces by light microscopy indicated that although the majority of surfaces failed at the adhesive interface (adhesive failure), lower bond strengths were associated with higher percentages of adhesive failures. When the specimens were stored in 10% NaOCl for 1 h, the number of adhesive failures increased for all the groups (Table 2).

After 24 h, SB showed higher bond strength values on water saturated-dentin (44.9 ± 7.6 MPa) in comparison with 100% ethanol saturated-dentin (36.8 ± 8.3 MPa). On the other side XP did not shown statistical significance among saturated dentin with tert-butanol or with water saturated-dentin (35.5 ± 8.8 and 32.7 ± 9.7 MPa, respectively).

Storage in 10% NaOCl reduced the bond strength in all groups tested. After ageing in 10% NaOCl for 1 h, XP exhibited significantly higher bond strengths values (23.8 ± 7.3 MPa and 22.3 ± 7.2 MPa, respectively for water- and alcohol-based), when compared with the SB groups regardless of the water replacement protocol (14.2 ± 3.3 MPa and 11.5 ± 2.9 MPa, respectively for water- and alcohol-based).

Interfacial silver nitrate evaluation

Representative light microscopy images of the adhesive interface produced by SB on water saturated dentin 24 h after bonding, showed no nanoleakage expression (Fig. 1A). However, when SB was bonded to ethanol saturated dentin, minimal silver deposition along the adhesive interface was observed (Fig. 1C). The results of interfacial nanoleakage analysis of the adhesive interface created by XP on water or tert-butanol saturated dentin, 24 h after bonding, showed low levels of silver deposition along the interfaces (Fig. 2A and Fig. 2C). After 1 h of 10% NaOCl ageing, all adhesive interfaces showed extensive and homogeneous deposition of silver throughout the adhesive interface (Fig. 1B, Fig. 1D, Fig. 2B and Fig. 2D).

DISCUSSION

The results of the present study showed that the use of SB on 100% alcohol-saturated dentin achieved significantly lower bond strength values than water-wet

bonding control group. Conversely, the use of 99.5% tert-butanol did not affect bond strength values when compared to the water-wet bonding control group for XP adhesive system. Thus, since there was difference when the alcohol-water replacement protocol was applied, the null hypothesis was partially rejected.

The ethanol-wet bonding protocol is one try to coax hydrophobic monomers to infiltrate in demineralized wet dentin.^{9,10} The ethanol-wet bonding technique proposed for dentin dehydration¹⁴ is analogous to the technique employed by electron microscopists for tissue embedding.²³ This chemical dehydration procedure is unlikely to be clinically attractive due its extended time demand, justifying the search of a most simplified dehydration protocols.

Since hydrophobic resins are considerably difficult to apply to dentin, an alternative version of the ethanol wet-bonding technique is to apply hydrophilic adhesives to ethanol-saturated demineralized dentin.²⁴ However, to the simplified ethanol-wet bonding technique, a decreased in the bond strength values for simplified etch-and-rinse adhesive system, like SB, was found, confirming that this technique is extremely technique-sensitive,^{11,25} even with three absolute ethanol applications.¹¹

When simplified protocol was used, evaporation of water from the water-saturated collagen matrix prior to the rising with absolute ethanol could have resulted in the collapse and shrinkage of collagen fibrils.¹⁵ The collapse of demineralized dentin matrices is an active process, involving the rapid, spontaneous development of new hydrogen bonds between adjacent collagen peptides that results in stiffening of the matrix in a collapsed state.¹⁰

According to the theory of solubility parameters (δh)^{26,27} different solvents like ethanol ($\delta h= 20.0$), methanol ($\delta h= 24$) and water ($\delta h= 40.4$), were successful in breaking those interpeptide hydrogen bonds, allowing the matrix to soften to the point that it can expand rapidly with water or methanol, or more slowly with ethanol.¹⁰

In the present study we suppose that the amount of water in the SB adhesive system was not able to re-expand the extremely collapsed matrix after the chemical dehydration of dentin by 100% ethanol, resulting in poor monomer infiltration and in a deficient hybrid layer, like observed in the silver nitrate uptake microscopy, as well as observed in the Sadek *et al.* (2010)¹¹ study.

Also, ethanol has a vapor pressure of 52.50 mmHg at 23 °C, while water has 21.05 mmHg, thus ethanol evaporates much more quickly than water.²⁸ In addition

this, short periods of ethanol saturation could not be enough for complete replacement of water within the acid-etched intertubular dentin and the dentinal tubules by ethanol.¹¹

On the other hand, XP BOND is a recently formulated adhesive based on tert-butanol (2-methyl-2-propanol) as solvent. This solvent consists of a C4-body with an alcohol group surrounded by three methyl groups, making it completely miscible with water and polymerizable resins both.²⁹ Vapor pressure of the different kinds of solvents at 20 °C is given 2,330 Pa for water, 4,133 Pa for tert-butanol and 5,900 Pa for ethanol.³⁰ Once tert-butanol evaporates more slowly than ethanol we suppose that it would result in less shrinkage of the demineralized dentin matrix. It could increase the bond strength as was found in this work.

It has been described that XP adhesive system is capable to penetrate dry and collapsed demineralized dentin to improve the bonding procedure and result in a less technique-sensitive application²⁹ and this was recently confirmed by Orellana *et al.* (2009).³¹ In this study, authors compared the microtensile bond strength of three different total etch adhesives (XP Bond/Dentsply, Excite/Ivoclar/Vivadent and Prime & Bond NT/Dentsply) applied on wet and dry dentin. The authors observed that XP Bond adhesive system with tert-butyl alcohol as a solvent, had higher values of microtensile bond strength than the Prime & Bond NT and Excite, which use acetone and ethanol as solvents, respectively, on both moist and dry dentin.³¹ Authors correlated the results obtained in this study a chemical reaction between collagen and XP Bond adhesive components.

A rapid method of simulating the bond interface degradation *in vitro* is to expose the resin-dentin interface to a strong, non-specific oxidizing and deproteinizing agent such as sodium hypochlorite (NaOCl).²² The rationale for the use of NaOCl is the expedited NaOCl-induced degradation of suboptimally impregnated collagen fibrils (unprotected by the resin) caused by incomplete impregnation of the demineralized dentin layer, that typically occurs in etch-and-rinse adhesive systems. This is considered to be one of the main reasons for hybrid-layer instability, as shown by fractographic analysis of *in vivo*-degraded resin-dentin bonds.³²

In the present study the ageing by storage in 10% NaOCl decreased the bond strength values for SB and XP, as well as, similarly to previous investigations,^{21,22,33} regardless of water replacement protocol used (alcohol or water). Also, it was

observed increasing of silver nitrate uptake in all light microscopy evaluated after immersion in 10% NaOCl for 1 h.

Although the wet-bonding with ethanol achieved higher bond strengths even with hydrophilic resins than were possible with water-saturated matrices,⁵ the major problem is the use of hydrophilic monomers. When more hydrophilic adhesive monomers are used, the resultant hybrid layer tends to be more prone to hydrolytic degradation.^{34,35}

CONCLUSION

Within the limitations of this study, it may be concluded that the “alcohol wet bonding” simplified technique used in the present study, it was not able to improve resin-dentin bond durability for simplified etch-and-rinse adhesive systems.

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Table 1 - Adhesive composition and mode of application

MATERIAL	COMPOSITION (*)	MODE OF APPLICATION		
		Acid etched	Saturated demineralized dentin	Adhesive application
Adper Single Bond 2 (3MESPE, St. Paul, MN, USA) Batch # 9XJ	HEMA, BisGMA, dimethacrylate functional, copolymer of polyacrylic and polyalkenoic acids, water, ethanol, nanofiller, photo-initiator	Apply phosphoric acid to dentin for 15 s. Rinse for 15 s. Dry with absorbent paper.	WATER-BASED TECHNIQUE: rewetted with 2 µl of water for 60 s.	Apply two consecutive coats of adhesive for 15 s with gently agitation. Gently air for 5 s to evaporate the solvent. Light cure for 10 s.
			ALCOHOL-BASED TECHNIQUE: rewetted with 2 µl of ethanol 100% for 60 s.	
XP Bond (Dentsply DeTrey, Konstanz, Germany) Batch # 0804002271	TCB-resin, PENTA,UDMA, TEGDMA, HEMA, butylated benzenediol, ethyl-4-dimethylaminobenzoate, camphorquinone, nanofiller, t-butanol	Apply phosphoric acid to dentin for 15 s. Rinse for 15 s. Dry with absorbent paper.	WATER-BASED TECHNIQUE: rewetted with 2 µl of water for 60 s.	Apply the adhesive for 20 s undisturbed. Gently air for 5 s to evaporate the solvent. Light cure for 20 s.
			ALCOHOL-BASED TECHNIQUE: rewetted with 2 µl of tert-butanol 99.5% for 60 s.	

Abbreviations: HEMA = 2-hydroxyethylmethacrylate; BisGMA= 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]-phenyl propane; TCB-resin = carboxylic acid-modified dimethacrilate; PENTA = phosphoric acid-modified acrylate resin; UDMA = urethane dimethacrylate; TEGDMA – triethyleneglycol dimethacrylate.

* Information as received from manufacturer.

Table 2 – Number of specimens (%) and failure mode according to each experimental group

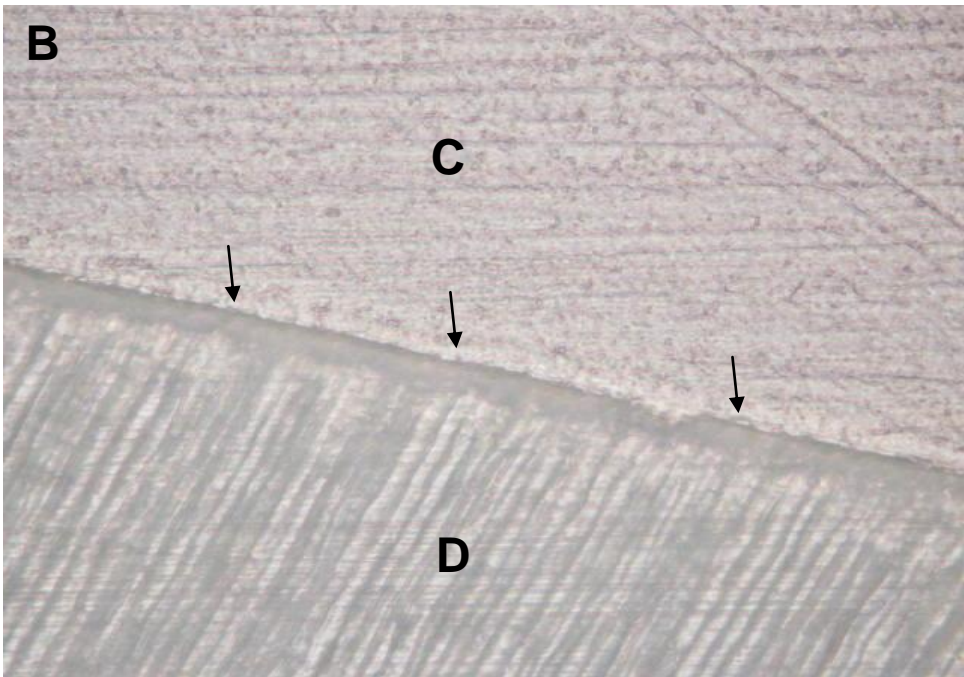
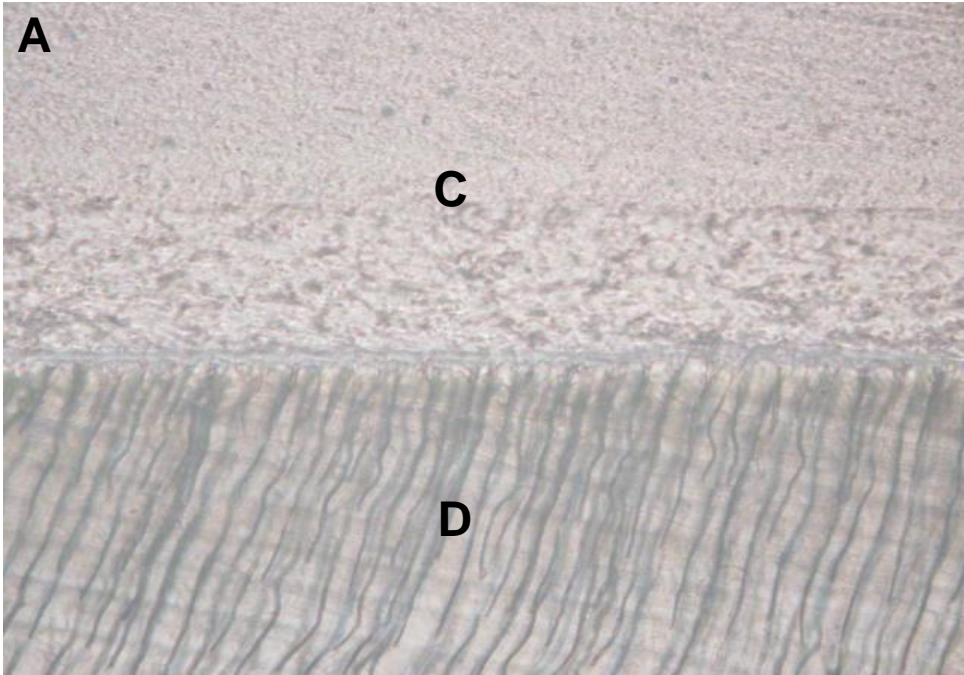
GROUPS		FAILURE MODE (%)							
Adhesive system	Saturated demineralized dentin	Immediate				Aged in NaOCl			
		A	M	CD	CR	A	M	CD	CR
Adper Single Bond 2	Water	87.1	3.2	9.7	0.0	94.4	2.8	2.8	0.0
	Alcohol	90.0	0.0	3.3	6.7	100	0.0	0.0	0.0
XP Bond	Water	90.9	6.0	0	3.1	94.2	0.0	5.8	0.0
	Alcohol	94.4	0.0	2.8	2.8	100	0.0	0.0	0.0

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

Table 3 – Mean and standard deviation (MPa) for microtensile bond strength values according to each experimental group, as well as, statistical analysis (*)

GROUPS			
Adhesive system	Saturated demineralized dentin	Immediate	Aged in NaOCl
Adper Single Bond 2	Water	44.9 ^{Aa} ± 7.6 (1/30)	14.2 ^{Bd} ± 3.3 (3/32)
	Alcohol	36.8 ^{Ab} ± 8.3 (0/30)	11.5 ^{Bd} ± 2.9 (1/31)
XP Bond	Water	35.5 ^{Ab} ± 8.8 (3/30)	23.8 ^{Bc} ± 7.3 (4/30)
	Alcohol	32.7 ^{Ab} ± 9.7 (6/30)	22.3 ^{Bc} ± 7.2 (3/42)

Mean ± standard deviation (number of premature failed sticks/number of intact sticks tested) of μ TBS results (expressed in MPa). (*) Groups identified by different subscript letters are significantly different ($p < 0.05$). Capital letters compare horizontal groups and the lowercase letters the vertical ones.



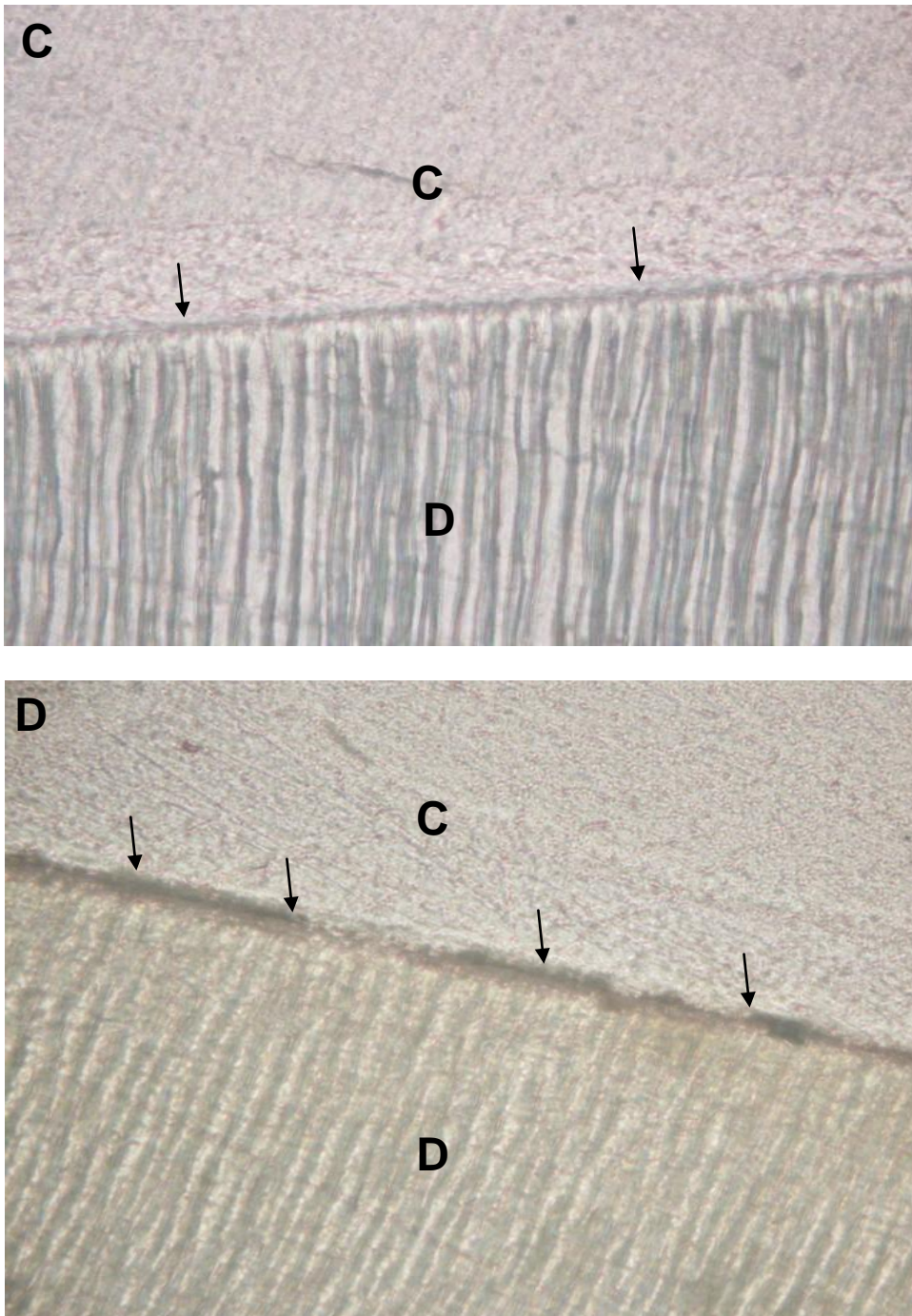
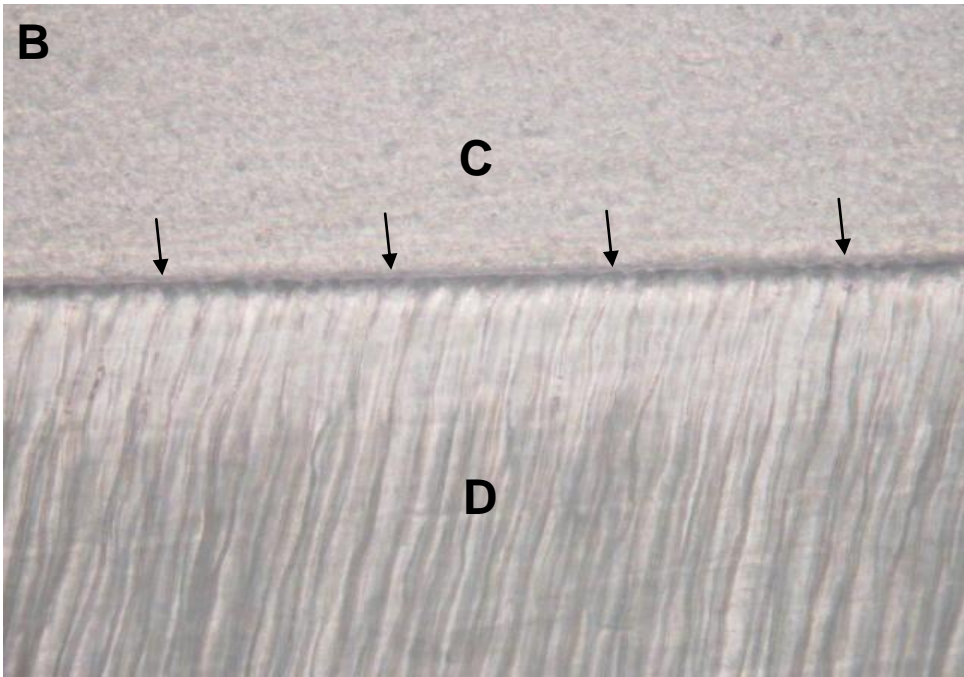
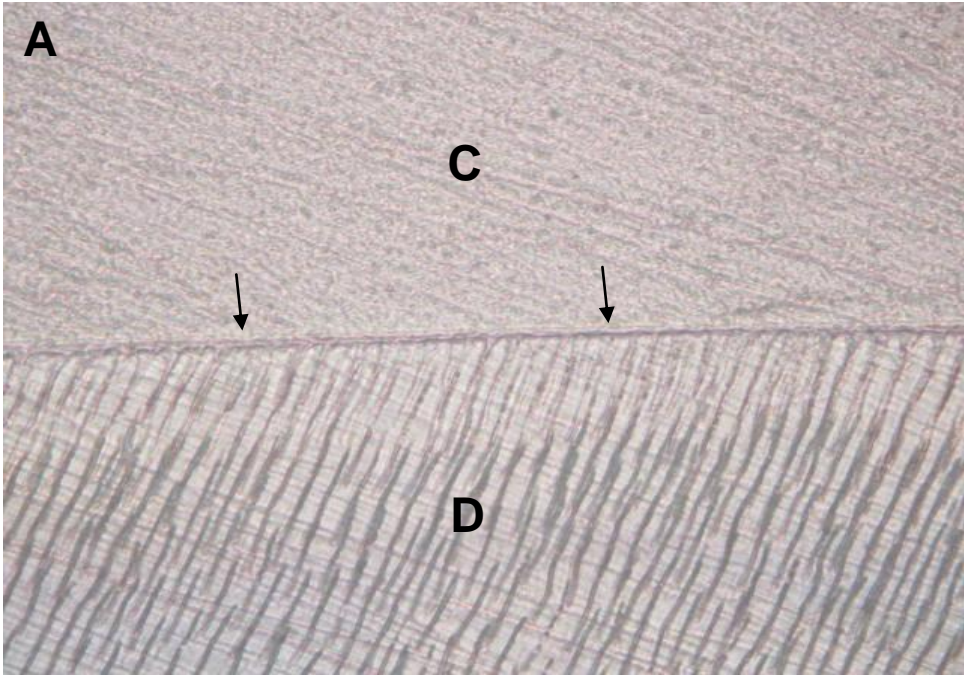


Fig. 1 – Light microscopy images showing representative nanoleakage interfacial expressions. Pointers: silver deposits; c: composite resin; d: dentin. (A) Adhesive interface produced by SB bonded on water saturated dentin (control group), showing no silver uptake. (B) After 10% NaOCl storage, the adhesive interface showed silver deposits. (C) Adhesive interface created by SB on ethanol saturated dentin, showing minimal silver deposition along the adhesive interface. (D) After aged in 10% NaOCl, the adhesive interface showing extensive and homogeneous silver deposits throughout the adhesive interface (400 x).



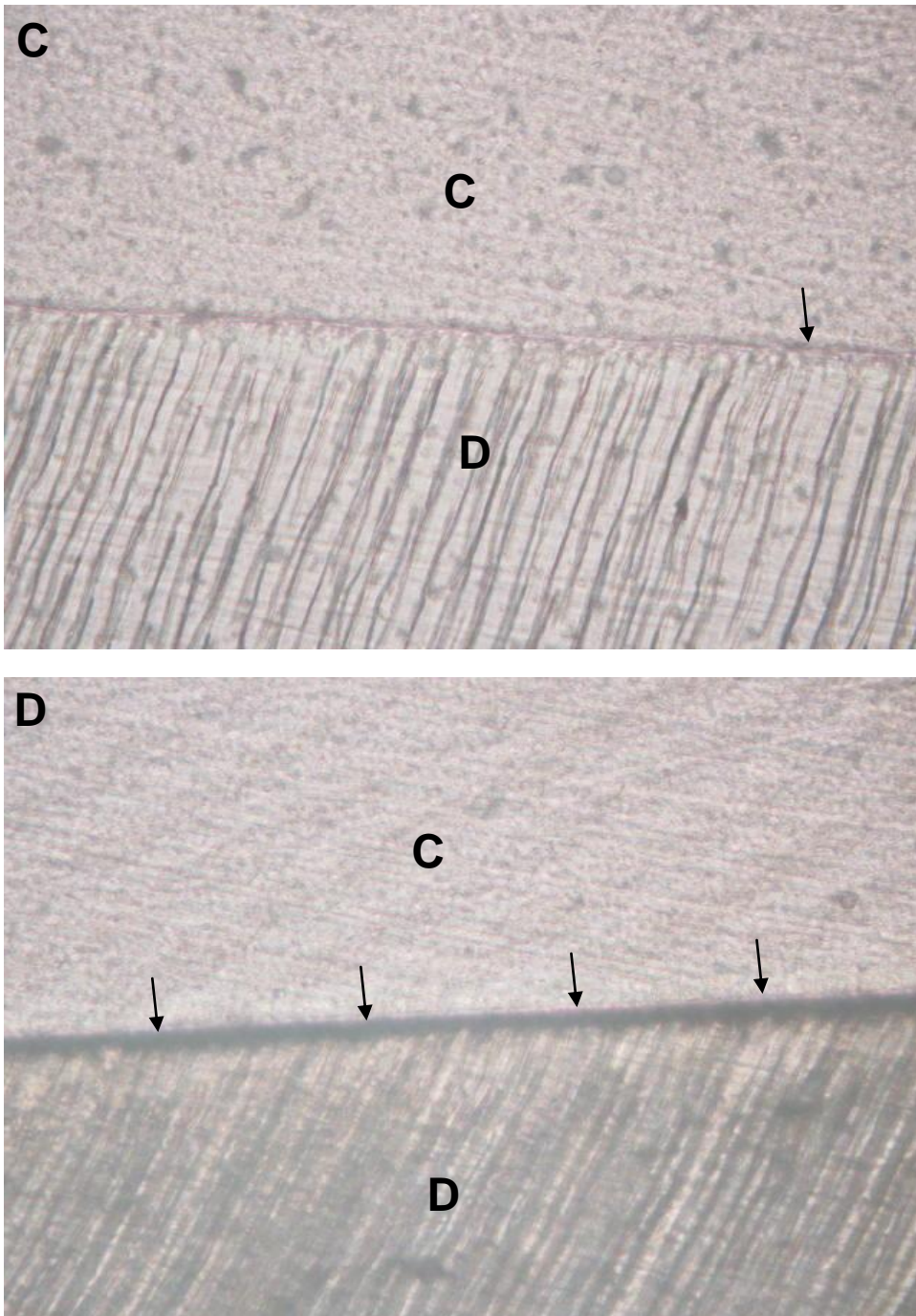


Fig. 2 – Light microscopy images showing representative nanoleakage interfacial expressions. Pointers: silver deposits; c: composite resin; d: dentin. (A) Adhesive interface produced by XP bonded on water saturated dentin (control group), showing minimal silver uptake. (B) After 10% NaOCl storage, the adhesive interface showed an increase in size of these silver deposits. (C) Adhesive interface created by XP on tert-butanol saturated dentin, showing some silver deposits points. (D) After aged in 10% NaOCl, the adhesive interface showing significant increases in silver deposits throughout the adhesive interface (400 x).

Conclusão Geral

4. CONCLUSÃO GERAL

Da avaliação dos resultados obtidos neste trabalho, pode-se concluir que:

- Os protocolos de saturação da dentina com etanol ou tert-butanol, utilizados no presente estudo, não foram capazes de melhorar a estabilidade da interface de união para os sistemas adesivos convencionais de dois passos.

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Anexo



MINISTÉRIO DA SAÚDE - Conselho Nacional de Saúde -
Comissão Nacional de Ética em Pesquisa - CONEP



Academia Cearense de Odontologia
Centro de Educação Continuada
A Excelência do Cuidado Por Sempre na Odontologia Cearense



COMITÊ DE ÉTICA EM PESQUISA – ACO/CEC

PARECER FINAL

Processo Nº 191

Projeto de Pesquisa: Estabilidade da interface de união de sistemas adesivos de condicionamento total aplicados à dentina saturada com etanol ou tert-butanol.

Pesquisador(a) Responsável: Livia de Oliveira Barros

PARECER

O projeto tem como objetivos avaliar a estabilidade da interface de união de sistemas adesivos convencionais de dois passos aplicados à dentina com etanol ou tert-butanol em diferentes tempos de aplicação..

Este projeto foi submetido ao Comitê de Ética em Pesquisa, sendo **APROVADO**, por estar de acordo com o que determina a Resolução CNS 196/96..

Fortaleza, 20 de dezembro de 2010.

Léa Maria Bezerra de Menezes

Léa Maria Bezerra de Menezes
Coordenadora do Comitê de Ética em Pesquisa da
ACO/CEC