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**PAULA MARIA MENDES ALVES**

# AVALIAÇÃO DA RESISTÊNCIA DE UNIÃO À DENTINA DE SISTEMAS ADESIVOS UNIVERSAIS APLICADOS NO MODO CONVENCIONAL E AUTOCONDICIONANTE

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Dissertação apresentada à Universidade Guarulhos como parte dos requisitos para obtenção do título de Mestre em Odontologia. Área de concentração: Dentística

Orientador: Prof. Dr. André Figueiredo Reis

Co-orientador: Prof. Dr. José Augusto Rodrigues

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A Comissão Julgadora dos trabalhos de Defesa de Dissertação de MESTRADO, intitulada "AVALIAÇÃO DA RESISTÊNCIA DE UNIÃO À DENTINA DE SISTEMAS ADESIVOS UNIVERSAIS APLICADOS NO MODO CONVENCIONAL E AUTOCONDICIONANTE" em sessão pública realizada em 31 de maio de 2016, considerou a candidata PAULA MARIA MENDES ALVES aprovada.

**COMISSÃO EXAMINADORA:**

**1.** Prof. Dr. André Figueiredo Reis (UNG)____________________

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**3.** Profa. Dra. Caroline Ely (UNG) Caroline Ely____________________

Guarulhos, 31 de maio de 2016.

# DEDICATÓRIA

*A você, meu filho, que ainda não chegou, mas já mudou tudo aqui dentro e aqui fora.*

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“Eu quero ser tudo que sou capaz de me tornar.”
**(Katherine Mansfield)**

# RESUMO

O objetivo deste estudo foi analisar a resistência de união (RU) à dentina produzida por novos sistemas adesivos universais aplicados no modo convencional ou auto-condicionante. Sessenta dentes humanos foram divididos em 6 grupos de acordo com os diferentes adesivos universais: Scotchbond Universal (SBU - 3M ESPE), Clearfil Universal (CFU - Kuraray), Futurabond U (FBU - VOCO), Xeno Select (XS - Dentsply De Trey), Prime&Bond Elect (PBE - Dentsply) e All Bond Universal (ABU - BISCO). Em seguida, foram subdividos em 2 subgrupos, de acordo com o modo de condicionamento: convencional ou autocondicionante (n=5). Após a aplicação dos adesivos, coroas de compósito foram construídas e os dentes restaurados foram seccionados em palitos com área de secção transversal de $1mm^2$ para o ensaio de microtração. O ensaio de microtração foi realizado em uma máquina de ensaio universal na velocidade de 1mm/min. Os dados de RU foram analisados pela ANOVA a 2 critérios e teste de Tukey. Resultados: SBU, XS e ABU apresentaram valores de resistência de união significativamente maiores quando utilizados no modo convencional ($p<0,05$). CFU, FBU e PBE não apresentaram diferença significativa na resistência de união quando utilizados tanto no modo convencional quanto no modo autocondicionante ($p>0,05$). Conclusão: A performance adesiva dos adesivos universais foi similar ou maior quando foram utilizados no modo convencional em comparação ao modo autocondicionante.



**Palavras-chave:** Adesivos Dentinários, Restauração Dentária Permanente, Resistência à Tração

## ABSTRACT

Purpose: The aim of this study was to analyze the bond strength to dentin produced by new universal adhesive systems used in self-etch and etch-and rinse application modes. Materials and Methods: Sixty human teeth were divided in 6 groups according to the different universal adhesive systems: Scotchbond Universal (SBU - 3M ESPE), Clearfil Universal (CFU - Kuraray), Futurabond U (FBU - VOCO) Xeno Select (XS - Dentsply De Trey), Prime&Bond Elect (PBE – Dentsply Caulk) and All Bond Universal (ABU, Bisco). Then, the teeth were subdivided into 2 subgroups, according to the application mode: etch-and-rinse or self-etch. Composite crowns were built after application of the adhesive systems and the restored teeth were sectioned in both “X” and “Y” directions into sticks with a cross-sectional bonded area of approximately $1mm^2$. The microtensile test was carried on a universal testing machine operated at a crosshead speed of 1mm/min. Bond strength values were statistically evaluated using two-way ANOVA and the Tukey post-hoc test. Results: SBU, XS and ABU presented significantly higher bond strength values when applied on etch-and-rinse mode ($p<0.05$). CFU, FBU and PBE presented no significant difference in bond strength values between etch-and-rinse and self-etching groups ($p>0.05$). Conclusion: Adhesive performance of Universal Adhesives was similar or higher when they were used in etch-and-rinse mode in comparison with the self-etching mode.



**Keywords:** Universal adhesives, microtensile bond strength, self-etching, etch-and-rinse.

# SUMÁRIO

# 1. INTRODUÇÃO

Os procedimentos restauradores da Odontologia Moderna se baseiam na efetividade de materiais com característica adesiva à estrutura dentária. As resinas compostas e os sistemas adesivos possibilitam tratamentos mais conservadores, estéticos e com maiores indicações e opções de tratamento. No entanto, a longevidade clínica destas restaurações ainda é uma preocupação devido à tendência de degradação progressiva da interface adesiva a longo prazo (De Munck et al., 2005).

A principal causa de falhas em restaurações adesivas é a presença de infiltrações marginais que leva consequentemente à redução da resistência de união. (Gaengler et al., 2004; Opdam et al., 2004). Frente a este problema, o profissional tende a substituir as restaurações de resina composta com mais frequência. Assim, diversos aspectos quanto à resistência de união e durabilidade destes materiais à estrutura dental devem ser considerados, tais como a característica hidrófila da superfície dentinária, as propriedades físico-químicas dos adesivos e sua interação com o esmalte a dentina. (Van Meerbeek et al., 2003; Cardoso et al., 2008).

A dentina é um tecido heterogêneo em sua composição e morfologia, e dinâmico em sua fisiologia quando comparado ao esmalte, e constitui-se por uma rede de túbulos que se estendem desde a câmara pulpar até à junção amelodentinária. No interior dos túbulos alojam-se os prolongamentos dos odontoblastos que se localizam na polpa, além de fibras nervosas e fluído pulpar. A dentina é um composto biológico de uma matriz de colágeno preenchida com cristalinos de apatita com tamanhos submicrométricos-nanométricos, cálcio deficientes e ricos em carbonatos dispersos entre cilindros ocos paralelos hipermineralizados de tamanho micrométrico e pobres em colágeno (túbulos dentinários contendo dentina peritubular). (Marshall et al., 1997). A composição química da destina é de aproximadamente 50% volume mineral, 20% água e 30% matriz orgânica (LeGeros, 1991; Marshall et al., 1997).

Alguns fatores fazem com que a dentina seja um tecido de difícil adesão, como a quantidade de túbulos dentinários, a presença da pressão pulpar com consequente aumento da umidade, presença do esfregaço ou *smear layer* produzido pelos instrumentos rotatórios, manter obrigatoriamente a dentina úmida para evitar o colabamento das fibrilas colágenas, cárie na dentina que diminui a resistência coesiva e a presença de minerais nos túmulos e a sensibilidade da técnica. (Perdigão, 2010).

Kramer e McLean em 1952 começaram os estudos mais importantes dos adesivos para esmalte e dentina, utilizando os monômeros resinosos desenvolvidos por Oskar Hagger. Em 1955, Buonocore sugeriu uma forma de tratamento do esmalte com ácido fosfórico, que resultava em maiores fatores de resistência de união. John Gwinnett, em 1967, observou em microscopia eletrônica e relatou que os monômeros adesivos podiam penetrar nos prismas de esmalte condicionados e envolver os cristais de apatita, tornando-os ácido resistentes. Esta foi a primeira descrição da camada híbrida, embora o termo não tenha sido introduzido.

Fusayama et al., em 1979, preconizaram o condicionamento total do esmalte e da dentina e Nakabayashi et al., em 1982, detectaram a formação de uma camada híbrida pela penetração e polimerazação da resina entre o colágeno da dentina desmineralizada pelo condicionamento ácido. Isso caracterizou a hibridização da dentina tornando os sistemas adesivos e as resinas compostas materiais de uso rotineiro para os clínicos.

Novos sistemas adesivos são constantemente desenvolvidos e comercializados visando a simplificação e otimização no tempo despedido para a execução dos procedimentos restauradores e a minimização das falhas na complexa aplicação dos sistemas adesivos. Atualmente existem os adesivos convencionais com condicionamento ácido prévio, os adesivos autocondicionantes, e os adesivos universais. Os adesivos convencionais com condicionamento prévio podem ser classificados em dois ou três passos, e os adesivos autocondicionantes podem ser de um ou dois passos, dispensando o condicionamento com ácido fosfórico a 37%. Os sistemas adesivos universais podem ser utilizados tanto com condicionamento ácido prévio quanto na forma autocondicionante.

## 1.1 Adesivos Convencionais

Nos adesivos convencionais de 3 passos, o condicionamento ácido inicial da dentina e do esmalte é necessário, removendo-se por completo todo o esfregado ou *smear layer* existente. Esse condicionamento promove uma desmineralização dentinária com profundidade entre 3 a 5 µm e expõe um emaranhado de fibras colágenas completamente desprovidas de hidroxiapatita (Van Meerbeek et al., 1992; Perdigão et al., 1996). O próximo passo consiste na aplicação de monômeros hidrófilos específicos como o HEMA (2-Hidroxietilmetacrilato) dissolvidos em solventes orgânicos como a acetona, etanol ou água. Enquanto o HEMA é responsável pela manutenção do molhamento e reexpansão da rede de colágeno, os solventes deslocam a água da superfície dentinária (Nakabayashi et al., 1992; Carvalho et al., 2003). Em um terceiro passo, um adesivo livre de solventes é aplicado, levando à penetração de monômeros hidrófobos não somente nos espaços interfibrilares da rede de colágeno, mas também dentro dos túbulos dentinários. Após esta infiltração, estes monômeros são polimerizados formando uma interface adesiva denominada camada híbrida, a qual em combinações com extensões resinosas no interior dos túbulos promove uma retenção micromecânica da restauração adesiva (Van Meerbeek et al., *1993).*

A partir da evolução destes sistemas adesivos de 3 passos, uma versão mais simplificada foi desenvolvida unindo diferentes composições (*primer* e agente hidrófobo) em uma única solução. Apesar do protocolo de uso mais simplificado, estes adesivos apresentam performance inferior quando comparados aos adesivos de condicionamento prévio com ácido fosfórico de 3 passos. Devido a união em uma única solução dos monômeros hidrófilos, solventes, ativadores, e monômeros hidrófobos, a natureza hidrófila destes adesivos os torna mais susceptíveis a degradação hidrolítica, onde muitas vezes os solventes utilizados não são completamente evaporados, permanecendo presentes na interfase adesiva após sua polimerização (Finger et al., 1999; Van Meerbeek et al., 2005)

## 1.2 Adesivos Autocondicionantes

Os adesivos autocondicionantes surgiram com o objetivo de simplificar os passos clínicos de aplicação do adesivo, ocorrendo a desmineralização e infiltração dos monômeros de modo simultâneo. A aplicação direta do adesivo exclui a etapa do controle da umidade dentinária pós-condicionamento, evitando o colabamento das fibrilas colágenas, quando a dentina desmineralizada apresenta ressecada. A aplicação do adesivo em dentina desmineralizada com excessiva quantidade de água dissolve o adesivo e produz separação de fase do mesmo. Isso foi descrito como “overwet phenomenon”, e compromete a adesão à dentina excessivamente úmida (Reis et al., 2007).

Os sistemas adesivos autocondicionantes não exigem um condicionamento ácido prévio da estrutura dental, pois contém monômeros que simultaneamente condicionam e agem como um *primer* sobre os tecidos dentários. Devido à natureza ácida destes adesivos, a *smear layer* pode ser parcialmente dissolvida e as subestruturas de destina e esmalte desmineralizados (Tay et al., 2000). Este protocolo é considerado menos complexo e pouco sensível à técnica, resultando em uma performance clínica confiável. No entanto certas preocupações são levantadas quanto à sua durabilidade e efetiva resistência de união, tal apreensão tende a ser material-dependente e muitos adesivos autocondicionantes demonstram performance clínica e laboratorial satisfatória (Peumans et al., 2005).

Os monômeros funcionais presentes nestes adesivos e seu potencial de interagir com o esmalte e a dentina definem o sucesso de uma camada híbrida íntegra e efetiva. Os adesivos autocondicionantes são classificados de acordo com o seu potencial ácido, sendo considerados como agressivos aqueles com pH $\geq 2$ (De Munck et al., 2005). Adesivos autocondicionantes suaves ou leves desmineralizam parcialmente a dentina, deixando um montante considerável de cristais de hidroxiapatita ao redor e no interior das fibrilas colágenas. Esta hidroxiapatita residual é fundamental para uma interação química dos monômeros funcionais e um processo efetivo de adesão química concomitante à adesão micromecânica (Yoshida *et al.,* 2004). A camada híbrida destes adesivos não é mais profunda do que 1 μm e extensões resinosas raramente são observadas, porém tais critérios não são fundamentais para uma adesão efetiva e estável (Manhart et al., 2010).

O fator primordial para o sucesso destes adesivos é sua composição com a presença do monômero funcional 10-MDP e sua grande interação química com o conteúdo parcialmente mineralizado existente. Outros monômeros como o 4-META e o fenil-P também possibilitam esta interação química adesiva. Estes monômeros contém grupos carboxila e fosfato que são capazes de promover uma adesão iônica com o cálcio existente na hidroxiapatita (Yoshida et al., 2000). No entanto, a adesão química promovida pelo monômero 10-MDP não somente expressou-se mais efetiva como também mais estável em ambiente aquoso quando comparado a performance do 4-META e fenil-P (Sano et al., 1999).

A efetividade da resistência de união dos sistemas adesivos autocondicionantes é atribuída à sua habilidade em desmineralizar e infiltrar a superfície dentinária simultaneamente em uma mesma profundidade, teoricamente impedindo falhas adesivas (Tanumiharja et al., 2000). Um fator que pode interferir a efetividade destes materiais é o tipo de esfregado produzido pelo substrato dentário (Koibuchi et al., 2001). A técnica e instrumentos utilizados para a adequação cavitária produzem diferentes tipos de *smear layer* com espessura, densidade e grau variável de ligação com a estrutura dentária subjacente (Kenshima et al., 2006). Como os adesivos autocondicionantes interagem com a *smear layer*, seu potencial ácido pode ser tamponado pelo conteúdo mineral de uma *smear layer* densa e espessa, resultando em uma interação pobre com o substrato dentinário subjacente (Camps & Pashley, 2000). Sendo assim, a utilização de técnicas e instrumentais que auxiliem a produzir uma camada mais delgada e menos compacta de debris durante as adequações cavitárias, como o uso de brocas diamantadas ultra-finas, aumentam a efetividade dos adesivos autocondicionantes.

## 1.3 Adesivos Universais

A busca por adesivos cada vez mais práticos quanto a sua aplicação clínica levou ao desenvolvimento de um novo grupo de sistemas adesivos que tem como característica principal uma abordagem flexível quanto ao condicionamento da estrutura dental. Estes adesivos permitem ao profissional escolher entre o condicionamento total do substrato, o condicionamento seletivo do esmalte, ou utilizar o modo autocondicionante destes materiais.

Considerando as diferenças na decisão profissional para a seleção do tipo de sistema adesivo e número de passos a ser empregado, alguns fabricantes tem investido em sistemas adesivos mais versáteis para passar ao profissional a decisão de qual estratégia adesiva utilizar: modo convencional ou autocondicionante. Essa nova família de adesivos é conhecida por adesivos universais e representa a última geração de adesivos que estão no mercado (Rosa et al, 2015).

Os adesivos universais foram formulados a partir do conceito “all-in-one” que já existe nos adesivos autocondicionantes de passo único, mas neles foram incorporados a versasatilidade de poderem se adaptar à cada situação clínica. (Wagner et al, 2014).

No modo autocondicionante, estes adesivos comportam-se de modo similar aos adesivos autocondicionantes clássicos, onde os monômeros funcionais agem promovendo adesão à destina e esmalte cortados. Alguns adesivos universais podem ser considerados adesivos autocondicionantes suaves com pH ≈ 2.7, logo, frente a este pH mais elevado muitas vezes o condicionamento seletivo do esmalte pode ser realizado. Consequentemente, estes adesivos podem ser aplicados em esmalte e dentina previamente condicionado por ácido fosfórico, ondeiam remoção plena do esfregaço é realizada (Chen et al, 2015).

Estudos mostram que adesivos universais usados tanto no modo convencional, como no modo autocondicionante produzem excelentes valores de resistência de união ao substrato (Hanabusa et al, 2012), porém temos poucas informações sobre novos adesivos universais presentes no mercado.

## 2. PROPOSIÇÃO

O objetivo deste estudo foi analisar a resistência de união à dentina produzida por sistemas adesivos universais aplicados no modo convencional ou auto-condicionante.

# 3. DESENVOLVIMENTO

## Effects of etching mode on bond strength of universal adhesives

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**Keywords:** Universal adhesives, microtensile bond strength, self-etching, etch-and-rinse.

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**Abstract**

Purpose: The aim of this study was to analyze the bond strength to dentin produced by new universal adhesive systems used in self-etch and etch-and rinse application modes. Materials and Methods: Sixty human teeth were divided in 6 groups according to the different universal adhesive systems: Scotchbond Universal (SBU - 3M ESPE), Clearfil Universal (CFU - Kuraray), Futurabond U (FBU - VOCO) Xeno Select (XS - Dentsply De Trey), Prime&Bond Elect (PBE – Dentsply Caulk) and All Bond Universal (ABU, Bisco). Then, the teeth were subdivided into 2 subgroups, according to the application mode: etch-and-rinse or self-etch. Composite crowns were built after application of the adhesive systems and the restored teeth were sectioned in both “X” and “Y” directions into sticks with a cross-sectional bonded area of approximately 1mm$^2$. The microtensile test was carried on a universal testing machine operated at a crosshead speed of 1mm/min. Bond strength values were statistically evaluated using two-way ANOVA and the Tukey post-hoc test. Results: SBU, XS and ABU presented significantly higher bond strength values when applied on etch-and-rinse mode ($p<0.05$). CFU, FBU and PBE presented no significant difference in bond strength values between etch-and-rinse and self-etching groups ($p>0.05$). Conclusion: Adhesive performance of Universal Adhesives was similar or higher when they were used in etch-and-rinse mode in comparison with the self-etching mode.

## Introduction

Bonding to enamel and dentin is mainly accomplished by micromechanical interlocking between synthetic, naturally degradable polymers, and enamel or dentin collagen fibrils (Nakabayashi et al., 1982). Effective, long-lasting bonding to dentin has been a challenge to dental clinicians, because in order to promote adhesion to dentin, the mineral phase needs to be totally or partially removed, and substituted by an adhesive solution, that will permeate this collagen-rich layer, and polymerize in situ, forming what has been called the hybrid layer (Nakabayashi et al., 1982; Hashimoto et al., 2000; Sano et al., 1999).

Different approaches, with different numbers of steps and degrees of sensitivity have been used to bond resin-based materials to enamel and dentin (Kanca, 1992; De Munck 2003; Tay & Pashley 2001; De Munck et al., 2005; Reis et al., 2007). Efforts have been directed to reduce the number of steps and technique sensitivity. One-bottle self-priming etch-and-rinse systems, as well as single-step self-etching adhesives are simplified versions of their multiple-step precursors (De Munck et al., 2005), and have been recently combined and marketed as Universal adhesives (Perdigão et al., 2012; Makishi et al., 2016). These multimodal adhesives may be used in etch-and-rinse mode, self-etch mode or selective-etch mode, depending on the clinician's preference (Rosa et al, 2015; Zhang et al., 2016).

Although recent studies reported that universal adhesives applied using either the etch-and-rinse or the self-etch mode produce excellent immediate bond strength to bonding substrates (Hanabusa et al, 2012), limited information is available on the newest universal adhesives recently introduced by different manufacturers. Thus, the aim of this study was to evaluate the bond strength to dentin produced by six universal adhesives applied either on etch-and-rinse or self-etching mode. The tested null hypothesis was that there is no difference in bond strength produced by universal adhesives applied on etch-and-rinse or self-etching mode.

**Materials and Methods**

Sixty freshly extracted human third molars were used. Teeth were obtained following an approved protocol by the review board of the University of Guarulhos. After disinfection and removal of soft tissues, flat coronal dentin surfaces were exposed using 600-grit SiC paper under running water to create a standardized smear layer.

Teeth were randomly assigned to six experimental groups, which were restored using six commercially available universal adhesive systems: Clearfil Universal (Kuraray), Scotchbond Universal (3M Espe), Futurabond U (Voco), Prime&Bond Elect (Dentsply Caulk), All Bond Universal (Bisco) and Xeno Select (Dentsply De Trey). Composition, batch number and application instructions are listed in Table 1.

Teeth were randomly assigned to 6 test groups, according to the universal adhesives used, and then subdivided into 2 subgroups according to the application mode: etch-and-rinse or self-etching (n=5). For the etch-and-rinse groups, 35% phosphoric acid was applied for 15 s, thoroughly rinsed with water, and excess water was removed with cotton pellets. Care was taken not to dehydrate dentin surfaces prior to adhesive application. For self-etching groups, dentin surface was dried with an air stream prior to adhesive application.

After application of the adhesive resins according to manufacturers instructions, composite crowns of 5 mm in height were built up incrementally with composite resin (TPH3, Shade A3, Dentsply Caulk, Milford, DE, USA). A LED light-curing unit (Radii Plus

- SDI, Victoria, Australia) with a power output of 1,500mW/cm$^2$ was used to polymerize all specimens. The restored teeth were stored in distilled water at 37$^o$C for 24 hours.

Afterwards, restored teeth were serially sectioned perpendicular to the adhesive-tooth interface into slabs, and the slabs into beams with a cross-sectional bonded area of approximately 1 mm$^2$ using a diamond saw (Isomet 1000; Buehler, Lake Bluff, IL, USA). Beams were fixed to the grips of a universal testing machine (EZ Test; Shimadzu Corp, Kyoto, Japan) using a cyanoacrylate adhesive (Loctite Super Bonder Gel; Henkel, Düsseldorf, Germany) and tested in tension at a crosshead speed of 1 mm/min until fracture occurred. Maximum tensile load was divided by specimen cross-sectional area, measured with a digital caliper (Mitutoyo Co., Tokyo, Japan), to express results in units of stress (MPa). Five beams were selected from each restored tooth, and the average value for each tooth was used in the calculations.

Bond strength values were statistically evaluated using a two-way ANOVA and the Tukey post-hoc test at a preset significance level of 0.05. Statistical analyses were performed using a personal computer program (SAS V9, SAS Institute, Cary, NC).

Failure modes were determined by visual examination of fractured specimens in a stereomicroscope at a magnification of 50X (PanTec, Panambra Ind. e Tecnica SA, Sao Paulo, Brazil). Failure was classified according to one of four types: cohesive failure in dentin, adhesive failure at the adhesive-dentine interface, cohesive failure in resin composite or mixed failure.

**Scanning Electron Microscopy**

For electron microscopy analysis of resin-dentin interfaces, two additional teeth were prepared for each group. Adhesive systems were applied according to manufacturers' recommendations and teeth were restored with a thin layer of SDR composite resin (Dentsply Caulk). After light curing with an LED with a power output of 1500mW/$cm^2$, specimens were stored in water for 24 hours prior to sectioning. Specimens were serially sectioned into 2-mm thick slabs and embedded in epoxy resin (Epoxycure, Buehler Ltd, Lake Bluff, IL, USA). Afterwards, they were polished with 400, 600, 800 and 1200-grit SiC paper (Buehler) and 6, 3, 1 and 0.25 µm diamond paste (Arotec, Sao Paulo, SP, Brazil). Then, specimens were dehydrated in ascending ethanol series, and coated with gold. Resin-dentin interfaces were observed with a field emission scanning electron microscope (Quanta 600 FEG).

Table 1. Universal adhesives, short name, manufacturer, pH, composition, and application instructions.

| Adhesive, (Batch Number) | pH | Composition | Self-Etch | Etch-and-Rinse |
|---|---|---|---|---|
| Scotchbond Universal (SBU) 3M Espe, St Paul, MN, USA (554836) | 2.7 | 2-HEMA, 10-MDP, dimethacrylate resins, VitrebondTM copolymer, silane, filler, ethanol, water, iniciators | 1. Apply the adhesive to the prepared tooth and rub in for 20s<br>2. Gently air-dry the adhesive for 5 s for the solvent to evaporate<br>3. Light cure for 10 s | 1. Apply etchant for 15 s<br>2. Rinse for 10 s<br>3. Air dry 2 s<br>4. Apply adhesive as for the self-etch mode |
| Clearfil Universal Bond (CFU) Kuraray, Tokyo, Japan (C40001) | 2.3 | HEMA, MDP, Bis-GMA, ethanol, camphorquinone, hydrophilic aliphatic dimethacrylate, silane coupling agent, colloidal silica, water, and accelerators | 1. Apply bond and rub for 20 s or 40 s<br>2. Dry by blowing mild air for 5 s<br>3. Light cure for 10 s | 1. Apply etchant for 10 s<br>2. Rinse thoroughly<br>3. Dry<br>4. Apply adhesive as for the self-etch mode |
| Futurabond U (FBU) VOCO, Cuxhaven, Germany (1333352) | 2.3 | 2-HEMA, Bis-GMA, HEMA, acidic adhesive monomer, urethane dimethacrylate, catalyst, silica nanoparticles, ethanol | 1. Activate single dose adhesive package<br>2. Apply adhesive to the cavity surface using the Voco Single Tim brush and rub adhesive in for 20 s<br>3. Dry adhesive with dry, oil-free air for at least 5 s<br>4. Light cure for 10 s | 1. Apply etchant for 15 s<br>2. Rinse for 10 s<br>3. Air dry 2 s<br>4. Apply adhesive as for the self-etch mode |
| Xeno Select (XS) Dentsply De Trey, Konstanz, Germany (1402000636) | 1.3 | Bifunctional acryl resin with amide functions, Acryloylamino alkylsulfonic acid, "inverse" functionalized phosphoric acid ester, Camphorquinone, Coinitiator, Butylated benzenediol, Water, tert-Butanol | 1. Apply the adhesive to the prepared tooth and rub in for 20s<br>2. Gently air-dry the adhesive for 5 s for the solvent to evaporate<br>3. Light cure for 10 s | 1. Apply etchant for 15 s<br>2. Rinse for 10 s<br>3. Air dry 2 s<br>4. Apply adhesive as for the self-etch mode |
| Prime&Bond Elect (PBE) Dentsply Caulk, Milford, DE, USA (141008) | 2.5 | Mono-, di- and trimethacrylate resins, PENTA, diketone; organic phosphine oxide, cetylamine hydrofluoride, acetone, water, self-cure activator | 1. Apply generous amounts of adhesives to thoroughly wet all tooth surfaces<br>2. Agitate applied adhesive for 20 s. Re-apply to coat preparation for the entire 20 s period<br>3. Remove excess solvent by gentle drying with clean, dry air for at least 5 s<br>4. Light cure for 10 s | 1. Condition enamel for at least 15 seconds and dentin for 15 seconds or less.<br>2. Rinse for 15 s<br>3. Dry<br>4. Apply adhesive as for the self-etch mode |
| All Bond Universal (1300006652) | 3.2 | 2-HEMA, 10-MDP, Bis-GMA, ethanol, water, initiators | 1. Apply two separate coats of adhesive with agitation for 10-15 s per coat<br>2. Evaporate solvent by thorough air-drying for least 10 s. No visible movement of adhesive<br>3. Surface should have a uniform glossy appearance. If not, repeat steps 1 and 2<br>4. Light cure for 10 s | 2. Rinse thoroughly<br>3. Remove excess water by blotting surface with an absorbent pellet or high volume evacuation for 1-2 s, leaving the preparation visibly moist<br>4. Apply adhesive as for the self-etch mode |

Abbreviations: 2-HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol A glycidyl methacrylate; PENTA, dipentaerythritol penta acrylate monophosphate.

**Results**

Mean bond strength values and standard deviation for the different groups are are shown in Table 2. Two-way ANOVA revealed that there were statistically significant differences for the factor “universal adhesive” ($p<0.00021$) and for the factor “etching mode” ($p=0.00001$). In addition, it identified a significant interaction between the two factors ($p=0.00157$). Tukey post-hoc test showed significant differences among adhesive systems for the different etching modes ($p<0.05$).

Scotchbond Universal, Xeno Select and All Bond Universal presented significantly higher bond strength values when applied on etch-and-rinse mode ($p<0.05$). Clearfil Universal, Futurabond U and Prime&Bond Elect presented no significant difference in bond strength values between etch-and-rinse and self-etchnig groups ($p>0.05$).

When the etch-and-rinse mode was used, Scotchbond Universal and Xeno Select presented the highest μTBS values, with no significant difference between them ($p>0.05$). However, Xeno Select was not significantly different from the other groups ($p>0.05$). For the self-ecthing mode groups, the highest μTBS values were presented by Futurabond U and Scotchbond Universal, with no significant difference between them ($p>0.05$). However, Scotchbond Universal was not significantly different from the other Universal Adhesives when used in self-etching mode ($p>0.05$).

Figure 1 shows the distribution of fracture patterns for the different groups. Failure mode analysis revealed that the majority of failures were adhesive at the adhesive-dentin interface for most groups, except for Scotchbond Universal and All Bond Universal applied on etch-and-rinse mode, which presente a high number of cohesive failures in resin composite.

Representative images of resin-dentin interfaces are presented in Figures 2 to 5. A very thin hybrid layer and short resin tags were observed when the universal adhesives were applied in the self-etching mode (Figs. 2 to 4). When applied in etch-and-rinse mode, a thicker hybrid layer (approximately 5 μm) and long resin-tags were observed.

**Table 2.** Mean bond strength values in MPa (SD) produced by the universal adhesives applied in self-etching and etch-and-rinse modes.

| Universal Adhesives | Etch-and-Rinse | | Self-etching | |
|---|---|---|---|---|
| Scotchbond Universal | 96.8 (14.9) | Aa | 47.5 (17.6) | ABb |
| Clearfil Universal | 52.2 (11.1) | Ba | 36.6 (13.0) | Ba |
| Futurabond U | 63.7 (14.4) | Ba | 67.5 (5.3) | Aa |
| Xeno Select | 76.1 (31.5) | ABa | 40.4 (10.7) | Bb |
| Prime&Bond Elect | 56.0 (8.4) | Ba | 40.7 (7.2) | Ba |
| All Bond Universal | 65.0 (7.1) | Ba | 27.6 (4.2) | Bb |

Means followed by different letters (lower case – row, upper case – column) differ among them by Tukey test at 95% confidence level.

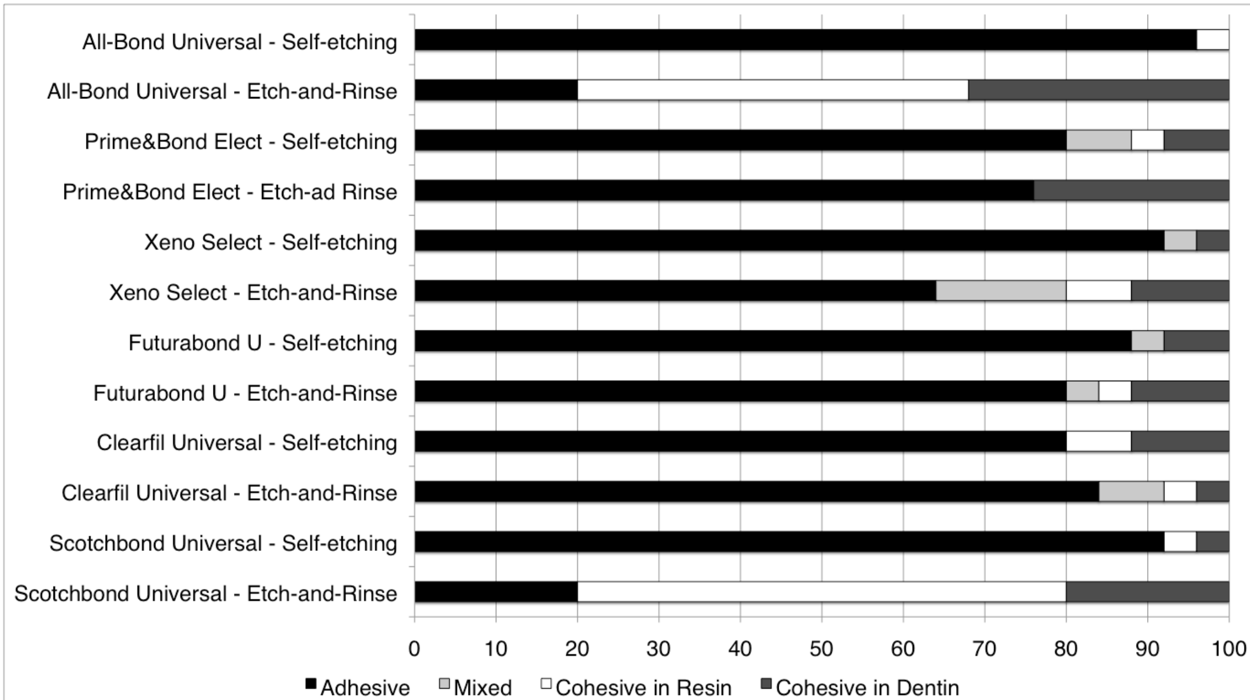


**Figure 1.** Distribution of failure modes for the different groups.

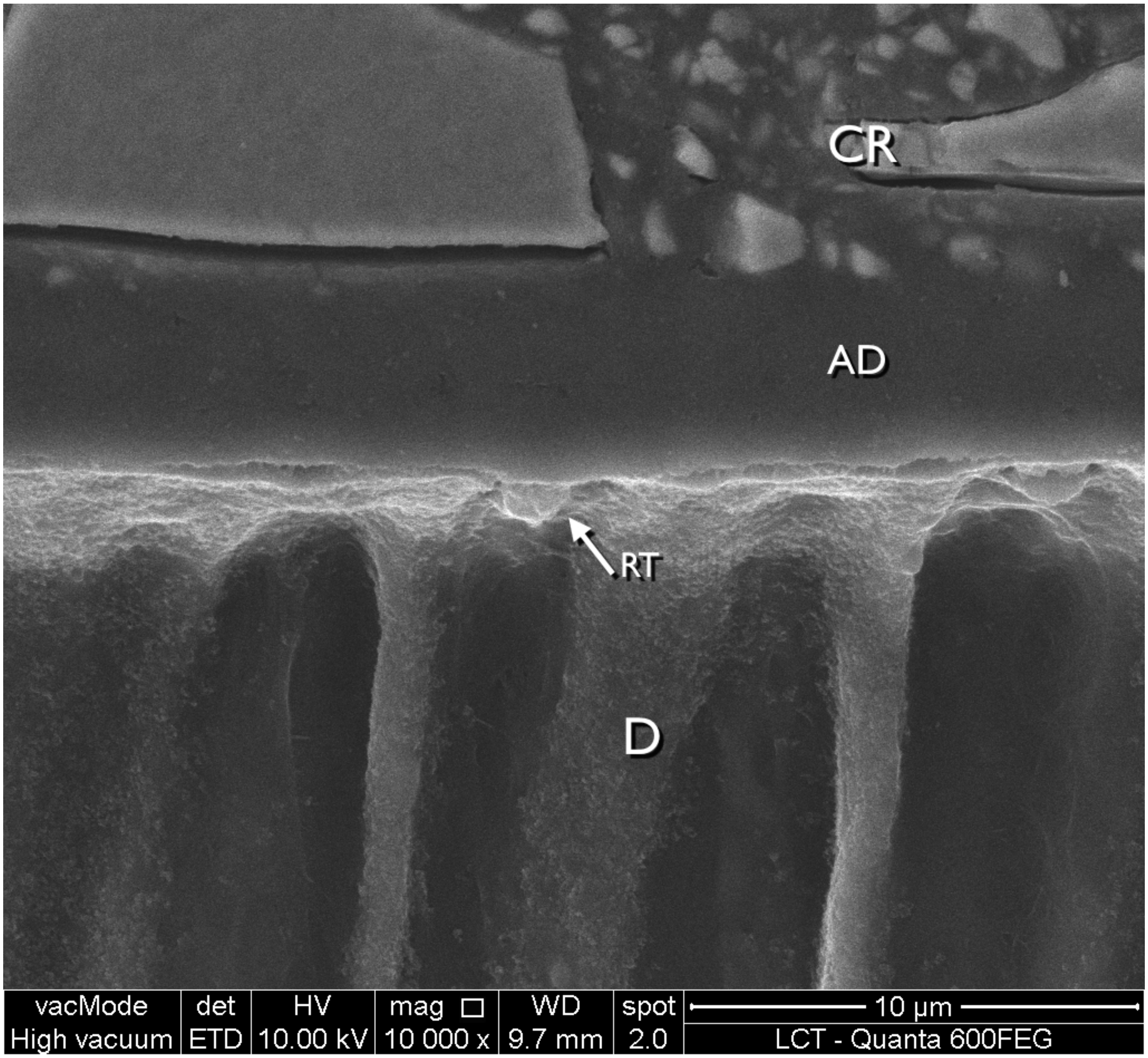


**Figure 2.** Representative scanning electron micrograph of a resin-dentin interface produced with All Bond Universal applied on self-etching mode. D – dentin; AD – adhesive layer; RT – resin tag.

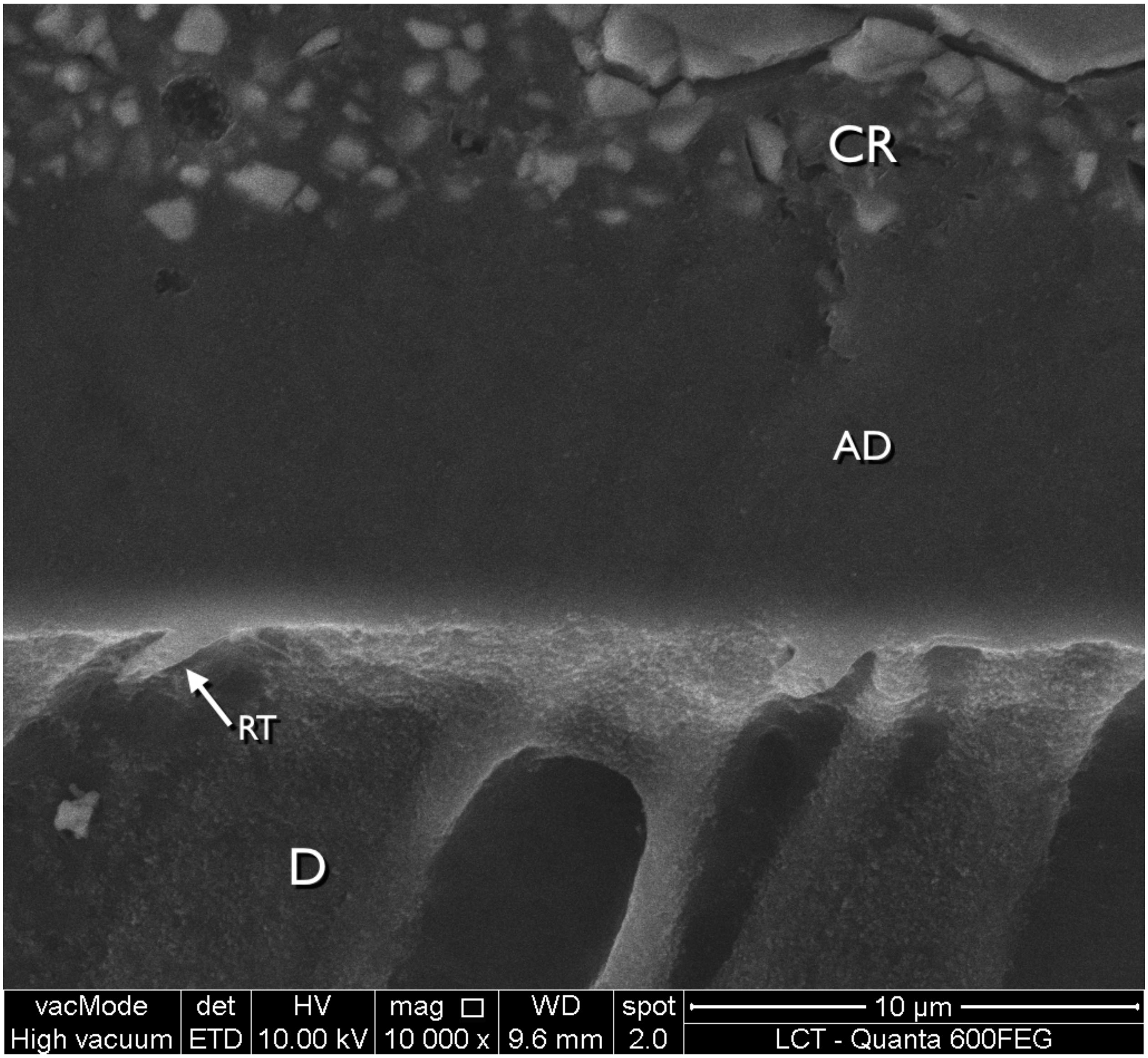


**Figure 3.** Representative scanning electron micrograph of a resin-dentin interface produced with Clearfil Universal Bond applied on self-etching mode. D – dentin; AD – adhesive layer; RT – resin tag; CR – composite resin.

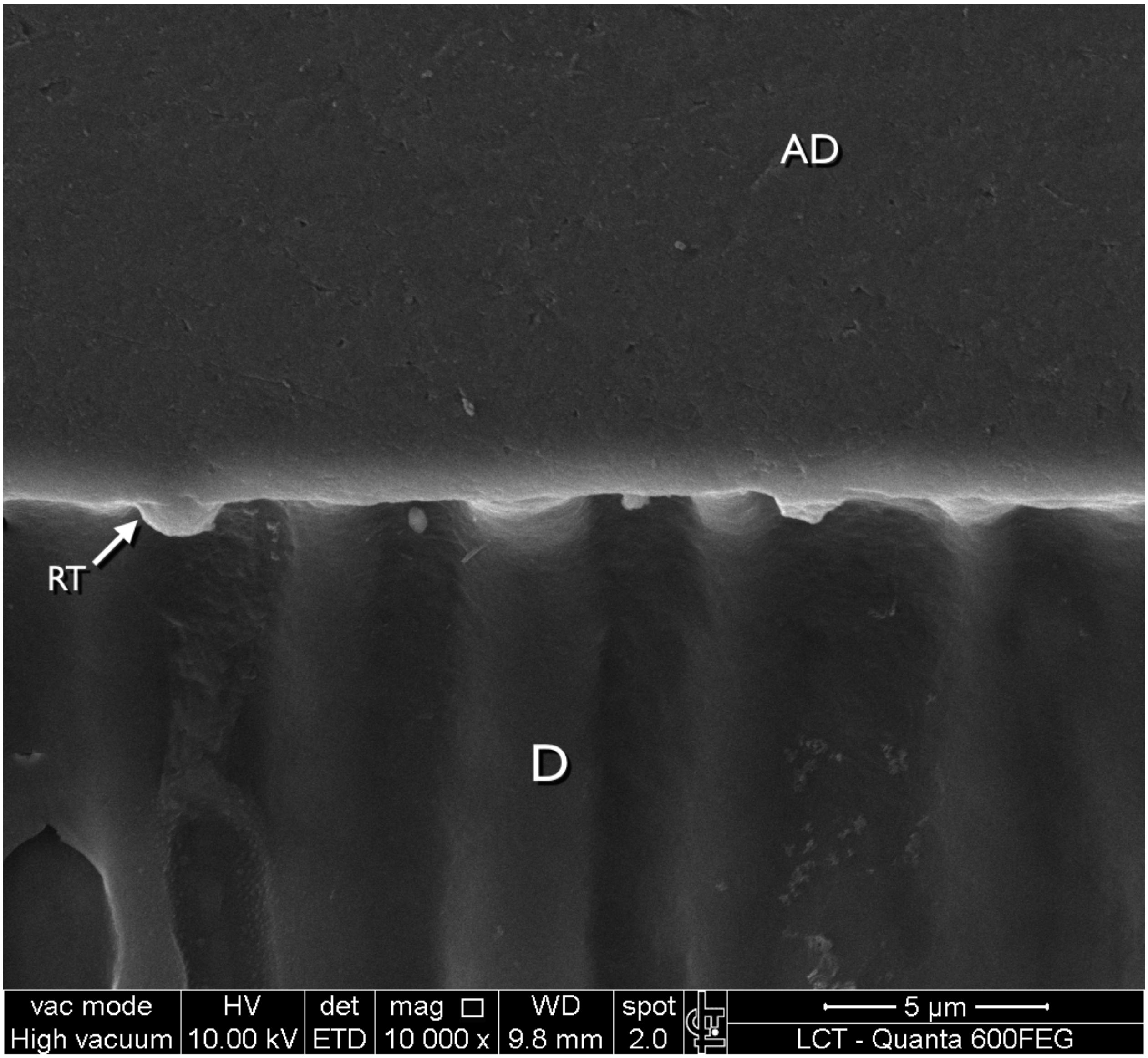


**Figure 4.** Representative scanning electron micrograph of a resin-dentin interface produced with Scotchbond Universal applied on self-etching mode. D – dentin; AD – adhesive layer; RT – resin tag.

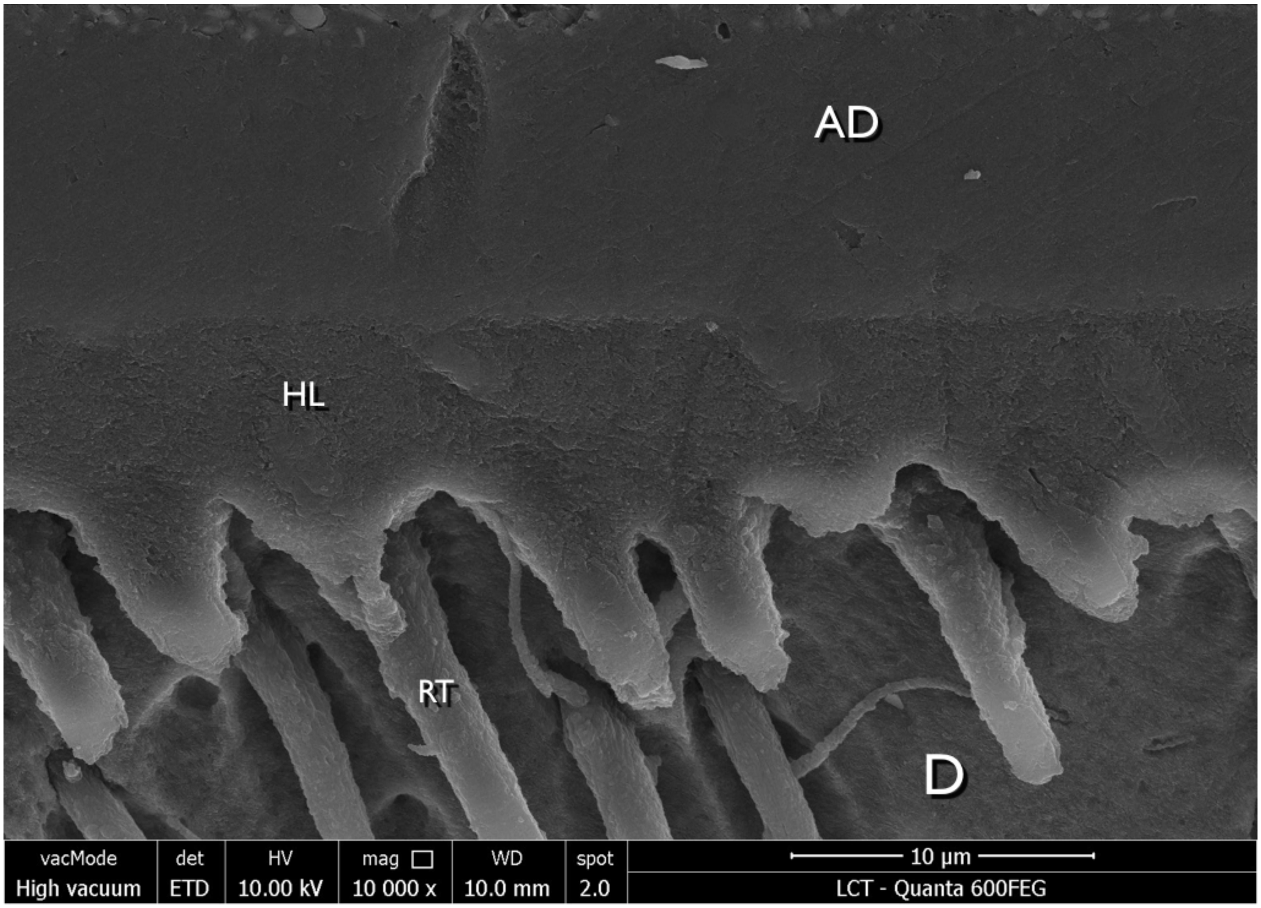


**Figure 5.** Representative scanning electron micrograph of a resin-dentin interface produced with Scotchbond Universal applied on etch-and-rinse mode. D – dentin; AD – adhesive layer; RT – resin tag; HL – hybrid layer.

**Discussion**

Recently, a new type of single-step self-etching adhesive has been introduced. This type of self-etching adhesive is categorized as “universal” or “multi-mode” as it can be used either with the etch-and-rinse or the self-etching approaches. (Munoz et al., 2013; Wagner et al., 2014; Munoz et al., 2015; McLean et al., 2015). Therefore, universal adhesives are used with phosphoric acid pre-etching in the etch-and-rinse or selective-etch approaches, which enhances bond strength to enamel. In addition, it also provides a simplified self-etching approach for dentin substrate (Takamizaua et al., 2016). However, this type of adhesive was only recently introduced to the market, and there is little information as to whether the different etching modes achieve equivalent bonding performance to dentin. Our null hypothesis was rejected, because for three of the tested universal adhesives, bond strength was significantly higher when the etch-and-rinse approach was used.

The resin composition as well as the presence and type of fillers might play important roles in bonding effectiveness (Tay et al., 2004). Each self-etch adhesive contains its specific functional monomer that, to a large extent, determines its actual adhesive performance (Yoshida et al., 2004). The specific molecular formula of the functional monomer and the dissolution rate of its calcium salt are thought to influence bonding efficacy and stability. The potential to chemically interact with interfacial hydroxyapatite might be helpful to achieve bond durability. This interaction occurs with mild self-etching adhesives that partially demineralize the dentin surface. It has been shown that MDP (10-methacryloxydecyl dihydrogen phosphate) is effective in bonding to hydroxyapatite, and seems to be very stable.

Three of the tested adhesives (Clearfil Universal, Scotchbond Universal and All

Bond Universal) present MDP as functional monomer. While the components in these materials are similar, there may be differences in the quantities and proportions of water, solvent, MDP, and dimethacrylate resins among the adhesives. There is a possibility that such differences may influence viscosity and wettability of each bonding agent, affecting the ability of resin monomers to penetrate into decalcified dentin (Takamizawa et al., 2016). When used in self-etching mode, these three MDP-containing universal adhesives presented bond strength values that were not significantly different from each other. However, when used on etch-and-rinse mode, Scotchbond Universal presented significantly higher bond strengths.

More than a decade ago, when single step self-etching adhesives were first introduced to the market, they were not recommended for use in the etch-and-rinse mode, because lower bonding performance to dentin was observed when phosphoric acid was used prior to adhesive application (Tay & Pashley, 2001; Torii et al., 2002; Van Landuyt et al., 2006). In the present investigation the immediate adhesive performance of the recently introduced universal adhesives was always significantly higher or not significantly different when used in the etch-and-rinse mode. Demonstrating that adjustments in the chemical formulation of single-step self-etching adhesives were made so they can also be used in etch-and-rinse mode. It seems that the problem of bonding mode incompatibility has been solved by manufacturers through blending less acidic resin monomers in the appropriately reduced concentrations with other resin monomers (Chen et al., 2015).

Three of the tested adhesives (Scotchbond Universal, Xeno Select and All Bond Universal) presented significantly lower bond strength values when used in the self-etching mode, in comparison with the etch-and-rinse groups. This reduction probably occurs due to the higher pH of these adhesives, classified as ultra-mild systems, in comparison with the other products. The interaction depth with dentin depends on the pH of the adhesives (Chen et al., 2015). Depending on the pH, self-etch adhesives may be classified into ultra-mild (pH > 2.5, 0.2–0.5 mm interaction depth), mild (pH ≈ 2; 0.5–1 mm interaction depth), intermediate (pH, 1–2; 1–2 mm interaction depth), and strong (pH ≤ 1, ≥ 5 mm interaction depth, similar to etching with phosphoric acid) (Van Meerbeek et al., 2011; Chen et al., 2015). More aggressive self-etching systems present higher contents of acidic monomers and water, resulting in increased hydrophilicity, which will result in increased water sorption, and consequently, decreased hydrolytic stability (Tay and Pashley, 2001; Tay and Pashley, 2003). In addition, continued etching along the base of hybrid layers after polymerization of those adhesives can occur (Wang & Spencer, 2005). Among the products tested, All Bond Universal presents the highest pH, 3.2. Even though not significantly different, it also presented the lowest bond strength values when used in the self-etching mode. On a recent study by Chen et al. (2015), TEM observations revealed that it presented the shallowest interaction with dentin, approximately 0.2 μm.

On the other hand, when used in the self-etching mode, Futurabond U presented the highest bond strength values. However, in recent reports on the long-term performance of universal adhesives, Zhang et al. 2016 and Chen et al. 2015 reported remarkable decrease in bond strengths and nanoleakage with signs of water-treeing on resin dentin interfaces produced with Futurabond U (Zhang et al. 2016 and Chen et al. 2015). In fact, all universal adhesives tested in the above-mentioned study of Zhang et al., with the exception or Prime&Bond Elect and Scotchbond Universal (applied in self-etching mode), presented significant reduction in bond strengths after 12 months of storage.

Previous investigations are in accordance with the present study, which demonstrated similar or higher performance, clinically or in vitro, when universal adhesives are applied in the etch-and-rinse mode (Wagner et al., 2014; Munoz et al., 2013; Perdigão et al., 2014). Even though hybrid layer thickness is approximately 10 times thicker when used in etch-and-rinse mode (≈5 μm) in comparison with the self-etching approach (≈0.5 μm), thicker hybrid layers formed in dentin substrates have been shown not to necessarily produce higher bond strengths (Harada et al., 1998).

## Conclusion

According to the results of the present investigation, the immediate bonding performance of Universal Adhesives was similar or higher when they were used in etch-and-rinse mode in comparison with the self-etching mode.

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# 4. CONCLUSÃO

De acordo com os resultados do presente estudo, pode-se concluir que a resistência de união imediata dos sistemas adesivos universais testados foi similar ou maior quando estes foram utilizados no modo convencional em comparação ao modo autocondicionante.

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