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RESISTÊNCIA DE UNIÃO À DENTINA DE UM SISTEMA ADESIVO DE CONDICIONAMENTO ÁCIDO ELETIVO

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RESISTÊNCIA DE UNIÃO À DENTINA DE UM SISTEMA ADESIVO DE CONDICIONAMENTO ÁCIDO ELETIVO

Dissertação apresentada à Universidade Guarulhos para obtenção do título de Mestre em Odontologia. Área de Concentração em Dentística.

Orientador: Prof. Dr. José Augusto Rodrigues
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Dedico esta dissertação,

À minha família,

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RESUMO

O objetivo deste estudo foi avaliar a resistência de união por microtração (MTBS) na interface restauração/dentina de um sistema adesivo de condicionamento eletivo com as técnicas convencional com condicionamento ácido prévio ou autocondicionante. Trinta e seis terceiros molares humanos livres de cárie, foram coletados e armazenados em solução de timol a 0,1%. A dentina média foi exposta por uma lixa 600, os dentes foram divididos em seis grupos (n = 6) e os sistemas adesivos foram aplicados de acordo com os grupos: Clearfil SE Bond (CSE - Kuraray), Scotchbond Universal (SBU-SE - 3M ESPE) aplicado como um adesivo autocondicionante de passo único; Scotchbond Universal (SBU-ER - 3M ESPE) aplicado como adesivo convencional com condicionamento ácido prévio de 2 passos, Adper Single Bond Plus (SBP - 3M ESPE), Optibond Solo Plus (OSP - Kerr), e Adper Prompt L-Pop (LPOP - 3M ESPE). Restaurações foram construídas com a resina composta TPH 3 (Dentsply Caulk) e fotoativadas em três incrementos de 2 mm cada. Os espécimes foram seccionados com um disco de diamante de baixa velocidade com refrigeração de água nas direções X e Y para se obter os corpos de prova que foram testados por tensão, a uma velocidade de 1,0 mm/min. As análises estatísticas foram realizadas utilizando-se ANOVA um critério e testes de Fisher DMS (α=0,05). Os grupos SBU-ER, SBU-SE e SBP apresentaram os maiores valores de MTBS e diferiram estatisticamente dos grupos CSE, e LPOP (DMS Fisher). O grupo OSP mostrou valores de MTBS intermediários e estatisticamente diferentes apenas de LPOP que apresentou os menores valores MTBS. O uso do sistema adesivo com condicionamento eletivo na dentina com as técnicas convencional com condicionamento ácido prévio ou autocondicionante não comprometeram a resistência de união.

Palavras-Chave: Resistência a microtração; Métodos de união; Agentes de união a dentina; Adesivo universal

ABSTRACT

The aim of this study was to evaluate the microtensile bond strength (MTBS) in the adhesion of restoration/dentin of an elective etching adhesive system with the etch-and-rinse or self-etching approach. Thirty-six caries-free, human third molars were collected and stored in a 0.1% thymol solution. The middle dentin were exposed by 600-grit silicon carbide paper, teeth were divided into six groups (n=6), and adhesive systems were applied according to groups: Clearfil SE Bond (CSE - Kuraray), Scotchbond Universal (SBU-SE - 3M ESPE) applied as a 1-step self-etch adhesive; Scotchbond Universal (SBU-ER - 3M ESPE) applied as a 2-step etch-and-rinse adhesive, Adper Single Bond Plus (SBP - 3M ESPE), Optibond Solo Plus (OSP - Kerr), and Adper Prompt L-Pop (LPOP - 3M ESPE). Build-ups were constructed with TPH 3 (Dentsply Caulk) and cured in three increments of 2 mm each. Specimens were sectioned with a slow-speed diamond saw under water in X and Y directions to obtain bonded slabs that were tested to failure in tension at a crosshead speed of 1.0 mm/min. Statistical analyses were computed using one-way ANOVA and Fisher´s LSD Tests (α=0.05). The SBU-ER, SBUSE, and SBP showed the highest MTBS values and statistically differed from CSE, and LPOP (Fisher's LSD). The OSP group showed intermediary MTBS and statisticaly differed only from LPOP that presented the lowest MTBS values. The use of elective etching adhesive system in the dentin with the etch-and-rinse or self-etching approach does not compromise the bond strength.

Keywords: Tensile Strength; Dental Bonding/methods; Dentin-Bonding Agents; Multi-purpose Adhesive

SUMÁRIO

1. INTRODUÇÃO

Sistemas adesivos dentários são agentes utilizados para promover a adesão entre a estrutura dental e a resina composta por meio da hibridização do esmalte dental e da dentina. Essa hibridização, ou seja criação da camada hibrida que promove a retenção à estrutura dental e selamento, apresenta diferentes mecanismos de formação entre esmalte dental e dentina (Chaves et al., 2002).

No esmalte dental, tecido composto por aproximadamente 96% de minerais, a hibridização ocorre após a aplicação do ácido fosfórico. Esse ácido condiciona a superficie do esmalte dental, promovendo uma desmineralização que a torna áspera, aumentando a área de superfície e criando microretenções que serão preenchidas pelo monômero resinoso (Hara et al., 1999).

Já em dentina, um substrato com uma composição diferente do esmalte dental - maior quantidade de material orgânico - a camada híbrida se forma após a desmineralização da hidroxiapatita pela ação do ácido fosfórico e consequente exposição das fibras colágenas. Essas fibras colagenas devem ser mantidas úmidas após a remoção do ácido fosforico para que monômeros hidrófilos carreados por solventes penetrem entre elas. Nesse contexto, pode-se utilizar tanto sistemas adesivos de três passos, nos quais aplica-se o monômero hidrófilo contido em frasco separado, seguido pela aplicação de um monômero hidrófobo ou pode-se utilizar um sistema de dois passos no qual os monômeros hidrófilos e hidrófobos estão associados em um único frasco (Miyazaki et al., 1998; Pashley et al., 2011).

Assim, a técnica de união de resina/esmalte é criada pela infiltração de monomeros hidrófobos e a união da resina/dentina depende da infiltração de monômeros hidrofílicos resinosos em um substrato dentinário úmido previamente desmineralizado seguido pela infiltração de monômeros hidrofobos (Breschi et al., 2008;. Vaidyanathan, Vaidyanathan, 2009; Pashley et al., 2011).

Embora a união ao esmalte dentário seja considerada eficaz e geralmente apresenta valores elevados de resistência de união quando se emprega a técnica do condicionamento ácido total seguido pela aplicação do sistema adesivo (Shono, 1995) a união na interface resina/dentina com a técnica do condicionamento ácido total é mais difícil de se alcançar, porque a união à dentina depende da adequada hibridização de componentes orgânicos (Loguercio et al., 2008). Uma condição essencial para a formação da camada híbrida é a manutenção da matriz orgânica da dentina hidratada após a desmineralização com o ácido fosfórico, que suporta a expansão das fibras de colágeno e preserva a integridade dos espaços interfibrilares. Esta disposição é compatível com o processo de união, uma vez que permite uma infiltração adequada dos monômeros resinosos e pode ser alcançada por meio da técnica de condicionamento total úmida associada aos monômeros resinosos dissolvidos em solventes orgânicos não aquosos ou de uma solução aquosa de *primers* hidrofílicos (Pashley et al., 2011). No entanto, a situação de completa penetração dos monômeros resinosos pelas fibras colágenas é difícil de se alcançar.

Uma das razões para a incompleta penetração dos monômeros resinosos dentro da camada híbrida é o movimento do fluido no interior do complexo de túbulos dentinários durante a infiltração da resina (Spencer et al., 2010). Alternativamente, a técnica de união de autocondicionamento utiliza monômeros acídicos que combinam o condicionamento ácido da superfície do dente e o uso do *primer* em um único procedimento, reduzindo o risco de falhas técnicas como os observados nos sistemas convencionais (Van Meerbeek et al., 2011).

A vantagem da técnica autocondicionante é a desmineralização da dentina simultâneamente à penetração de resina, o que conduz a formação de uma camada híbrida fina, favorecendo seu uso em áreas próximas a polpa e otimizando a camada híbrida que torna-se totalmente preenchida pelo adesivo (Tay et al., 2000; Van Meerbeek et al., 2003).

Alguns estudos têm demonstrado que este condicionamento mais brando poderia comprometer a união ao esmalte quando ele não foi

desgastado durante o preparo cavitário, pois a superfície exposta ao meio oral apresenta-se mais resistente à desmineralização, e consequentemente mais resistente à ação dos monômeros auto-condicionantes mais brandos (Di Hipólito et al., 2012). No entanto, o sucesso da união ao esmalte, com os sistemas autocondicionantes, com pH brando ou agressivo, ou seja, sem uma etapa de condicionamento ácido prévio, ainda representa um grande desafio para odontologia adesiva (Bouillaguet et al., 2001). Além disso, recentes estudos de nanoinfiltração da camada híbrida levantaram algumas suspeitas na teoria de que os adesivos autocondicionantes nem sempre garantem a infiltração completa de resina (Torii et al., 2002; Carvalho et al., 2005; Van Landuyt et al. ., 2006; Liu et al., 2011). Assim, na união com o esmalte dental os adesivos autocondicionantes não são tão eficazes como em dentina, e geralmente não é indicado sua utilização em superfícies de esmalte dental, sem uma prévia utilização do ácido fosfórico, tanto para procedimentos restauradores diretos como indiretos, especialmente na superfície de esmalte dental não tratada (Kanemura et al., 1999).

Algumas pesquisas indicam que a técnica convencional de três passos continua a ser a preferida para o esmalte dental (Van Meerbeek et al., 2003), mas o condicionamento seletivo do esmalte dental combinado ao adesivo autocondicionante com um pH “brando” pode, portanto, ser hoje recomendada para alcançar uma eficaz e duradoura união ao esmalte dental e dentina (Van Meerbeek et al., 2011; Yoshida et al., 2003). Dessa forma, associa-se as vantagens da técnica de condicionamento ácido do esmalte com as vantagens da técnica de adesivos autocondicionantes na dentina sem aumentar o número de passos clínicos.

No entanto, o clínico deve ter prudência com o condicionamento seletivo, pois esse deve ser aplicado nas superfícies de esmalte dental porém se pode condicionar inadvertidamente as paredes dentinárias durante o processo. Se isso ocorrer, o adesivo autocondicionante pode não ter a capacidade de penetrar corretamente na dentina previamente condicionada para produzir uma adequada camada híbrida e garantir uma resistência de união de alta qualidade. Além disso, alguns adesivos autocondicionantes

podem ter o desempenho reduzido quando unidos às superfícies dentinárias condicionadas (Liu et al., 2011).

Por outro lado, alguns fabricantes estão desenvolvendo adesivos autocondicionantes em que o esmalte dental e a dentina podem ser tratados com o método de preferência do clínico, ou seja, eles são eletivos e podem tanto ser utilizados com condicionamento ácido total, como autocondicionantes ou pela técnica do condicionamento seletivo do esmalte dental, para todas as restaurações, diretas e indiretas. Além disso, esses novos sistemas podem diminuir os erros técnicos durante o condicionamento seletivo do esmalte dental, quando ocorre o condicionamento inadvertido da dentina. No entanto, ainda são poucos os estudos que descrevem a utilização e o desempenho de sistemas adesivos eletivos em dentina.

2. PROPOSIÇÃO

O objetivo deste estudo foi avaliar a resistência de união entre dentina/restauração por meio de ensaio de microtração, a performance de um sistema adesivo de condicionamento eletivo utilizado no modo autocondicionante ou com condicionamento ácido prévio.

3. DESENVOLVIMENTO

Capítulo 1- Effect of six month storage on tensile bond strength of new elective etching adhesive system on dentin in self-etching or etch-and-rinse approach

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Effect of six month storage on tensile bond strength of new elective etching adhesive system on dentin in self-etching or etch-and-rinse approach

ABSTRACT

The aim of this study was to evaluate the microtensile bond strength (MTBS) in the adhesion of restoration/dentin of an elective etching adhesive system with the etch-and-rinse or self-etching approach after six months of water storage. Thirty-six caries-free, human third molars were collected and stored in a 0.1% thymol solution. The middle dentin were exposed by 600-grit silicon carbide paper, teeth were divided into six groups (n=6), and adhesive systems were applied according to groups: Clearfil SE Bond (CSE - Kuraray), Scotchbond Universal (SBU-SE - 3M ESPE) applied as a 1-step self-etch adhesive; Scotchbond Universal (SBU-ER - 3M ESPE) applied as a 2-step etch-and-rinse adhesive, Adper Single Bond Plus (SBP - 3M ESPE), Optibond Solo Plus (OSP - Kerr), and Adper Prompt L-Pop (LPOP - 3M ESPE). Build-ups were constructed with TPH 3 (Dentsply Caulk) and cured in three increments of 2 mm each. Specimens were sectioned with a slow-speed diamond saw under water in X and Y directions to obtain bonded slabs that were tested to failure in tension at a crosshead speed of 1.0 mm/min seven days or 6 months after adhesion procedures. Statistical analyses were computed using Repeated-Measures ANOVA and Fisher´s LSD Tests (α=0.05). There was no statistical significant differences between seven days or six months. SBU-ER, SBUSE, and SBP showed the highest MTBS values and statistically differed from CSE, and LPOP (Fisher's LSD). The OSP group showed intermediary MTBS and statistically differed only from LPOP that presented the lowest MTBS values. The use of elective etching adhesive system in the dentin with the etch-and-rinse or self-etching approach does not compromise the bond strength and showed stable bonds after six months of water storage.

Keywords: Tensile Strength; Dental Bonding/methods; Dentin-Bonding Agents; Universal Adhesive.

INTRODUCTION

Dental adhesive systems are agents used to promote adhesion between dental structure and composite resin. Bonding to dental enamel is considered effective and usually presents high bond strength values (Shono, 1995). Resin-dentin bonds are more difficult to achieve than resin-enamel bonds, because dentin bonding relies on organic components (Loguercio *et al*, 2008). However, adhesives systems should present similar performance on enamel and dentin to increase the clinical performance.

Usually resin-dentin bonds are created by infiltration of hydrophilic resin monomers into a previously demineralized dentin matrix (Breschi *et al*., 2008; Vaidyanathan, Vaidyanathan, 2009). An essential condition for the formation of the hybrid layer is the maintenance of the dentin organic matrix moist after demineralization, which supports the expansion of the collagen fibrils and preserves the integrity of the interfibrillar spaces. This disposition allows an appropriate infiltration of the resin monomers dissolved in non-aqueous organic solvents or an aqueous solution of hydrophilic primers (Pashley et al., 2011).

Alternatively, the self-etch bonding technique uses acidic monomers that combine tooth surface etching and priming in a single procedure, reducing the risk of technical deficiencies from etch-and-rinse systems (Van Meerbeek et al., 2011).

The advantage of the self-etching adhesives (SEA) technique is the simultaneous dentin demineralization and resin penetration, which should lead to a thin and an optimally infiltrated hybrid layer, even to dentin close to the pulp (Tay et al., 2000; Van Meerbeek et al., 2003). Also, SEAs are less aggressive than the phosphoric acid used in etch-and-rinse technique (Shinohara et al. 2006), and the bonding to enamel is not as effective as it is in dentin, and it is generally not indicated for use on enamel surfaces without a prior phosphoric acid etching step for direct or indirect restorative procedures, especially in the unground enamel (Kanemura et al., 1999; Hanabusa et al., 2012).

Some researches indicate the three step etch-and-rinse approach as preferred for enamel (Van Meerbeek et al., 2003; Van Meerbeek et al., 2011; Yoshida et al., 2003), but selective enamel etching combined with a ‘mild’ pH SEA can therefore today be recommended to achieve effective and durable bonding to enamel and dentin (Van Meerbeek et al., 2011; Yoshida et al., 2003).

Nevertheless, clinicians may have concerns with the selective enamel etching is whether etchant can be retained into the enamel surfaces and not inadvertently overflow dentin walls in the selective etching process and after rinse stet the dentin could be air dried as well. If it occurs, the adhesive may not have the capacity to properly penetrate the etched dentin to produce a suitable hybrid layer to ensure high bonding strength and sensitivity prevention. Also, some SEAs may have reduced performance when bonding to etched dentin surfaces (Van Landuyt et al., 2006; Liu Y et al., 2011).

On the other hand, some manufactures have developed SEAs that treat enamel and dentin, as clinicians preferred etching method, such as etch and rinse, self-etch or selective-etch approach to all direct and indirect restorations. However, few studies describe the use and the performance of such adhesive systems.

The aim of this study was to evaluate the microtensile bond strength in the adhesion of restoration/dentin of an elective etching adhesive system performance in the etch-and-rinse and in the self-etching techniques and the stability after six months of water storage. The null hypothesis of this study is that elective etching adhesive system does not differ from bond strength to dentin using the etch-and-rinse or the self-etching modes after seven days or six months of water storage.

METHODS AND MATERIALS

Experimental design

The factor under study was “adhesive” system in six levels (n=6), studied by repeated measurements at seven days and six months in water staorage after adhesion procures. The experimental adhesive groups were: SBU-SE- Scotchbond Universal (3M ESPE, St. Paul, MN, USA) applied as a

1-step self-etch adhesive; SBU-ER- Scotchbond Universal (3M ESPE, St. Paul, MN, USA) applied as a 2-step etch-and-rinse adhesive, the etch-and-rinse control groups were SBP- Adper Single Bond Plus (3M ESPE, St. Paul, MN, USA), OSP- Optibond Solo Plus (Kerr, Orange, CA, USA), and the control self-etching groups were CSE- Clearfil SE Bond (Kuraray, Kurashiki, Japan), and LPOP- Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) (Table 1). The dependent variable was microtensile bond strengh (MTBS) in MPa.

Table 1 – Materials used in this study.

Material	***Components****	***Batch Number***	***Manufacturer´s Recommended Protocols*** **
CSE- Clearfil SE Bond (Kuraray, Kurashiki, Japan)	Self-etching primer: MDP, HEMA, DMA , catalyst, water Bonding: MDP, HEMA, DMA, Bis-GMA, Filler, catalyst	Bond: 01657A Primer: 01107A	e; f; g (10s)
SBU- Scotchbond Universal (3M ESPE, St. Paul, MN, USA)	MDP Phosphate Monomer, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Water, Initiators, Silane.	457855	Self-etch: e; f; g (10s) Etch-and-rinse: a (15s); b(15s); c; e; h; i (10s)
SBP- Adper Single Bond Plus (3M ESPE, St. Paul, MN, USA)	Bis-GMA, HEMA, dimethacrylates, methacrylated polyalkenoic acid, copolymer, initiators, water, ethanol and silane-treated silica nanofillers.	N318844	a (15s); b (10s); c; d; f; g (10s)
OSP - Optibond Solo Plus (Kerr, Orange, CA, USA)	Bis-GMA, HEMA, GPDM, initiator, ethanol, fumed silica, barium aluminoborosilicate and sodium hexafluorosilicate	4312056	a (15s); b (10s); c; e (15s); f; g (20s)
LPOP - Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA)	Liquid 1 (red blister): Methacrylated phosphoric esters, Bis-GMA, Initiators based on camphorquinone, Stabilizers Liquid 2 (yellow blister): Water, HEMA, Polyalkenoic acid, Stabilizers	465223	d; f; g (10s)
TPH3 (Dentsply Caulk, Milford, DE, USA)	Bis-GMA, Bis-EMA, TEG-DMA, TiO_2, silica, Ba-B-F-Al-Si Glass	L697363E Shade: A3	g (20s)
ESPE Scotch Bond Etchant (3M ESPE, St. Paul, MN, USA)	35% phosphoric acid, silica	7523	-

* Abbreviations: Bis-GMA: bisphenol-glycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; DMA: dimethacrylate; GPDM: glycerol phosphate dimethacrylate; UDMA: Urethane dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; PENTA: phosphonated penta-acrylate ester; EDMAB: Ethyl 4-dimethyl amino benzoate.

** Application techniques – a: acid etching; b: rinsing; c: excess moisture removed from the preparation using a cotton pellet; d: application of two consecutive coats of adhesive; e: application of one coat of the adhesive; f: gently air-drying; g: light-cure.

Specimens' preparation

Thirty-six caries-free, human third molars were collected according to the local Institutional Review Board (# 19/2009), with the informed consent of the donors. They were stored in a 0.1% thymol solution at 4°C and used within one month following extraction.

Prior to bonding procedures, the roots and occlusal enamel were removed and the exposed middle dentin surfaces were wet-polished with 600-grit silicon carbide paper under running water, until enamel were removed to create a standard smear layer only on dentin (Di Hipolito et al., 2012).

Bonding Procedures

In the groups SBU-ER, SBP and OSP the 35% phosphoric acid with silica thickener (ScotchBond™ Etchant, 3M ESPE, St. Paul, MN, USA) was used to etch the dentin surfaces.

The dentin was thoroughly washed as recommended by the manufacturer and the substrate remained wet for adhesive application. After that, the adhesives were applied following the respective manufactures instructions (Table 1) and light-cured using a photo-activating device (LED, Radii Plus - SDI, Victoria, Australia) with a power output of 2,000mW/cm^2.

Subsequently, three 2-mm-thick increments of a resin composite (TPH3 Dentsply Caulk, Milford, DE, USA) were built up on each bonded surface and individually light-cured for 20s, constituting a crown of 6.0 mm in height.

Microtensile Bond Strength Test

The restored teeth were stored in distilled water at 37ºC for seven days. After this period of storage, they were serially sectioned in a longitudinal direction into 1.0-mm-thick slabs, using a diamond saw (IsoMet 1000; Buehler Ltd, Lake Bluff, IL, USA) under water cooling. Each slab was further transversally sectioned to produce bonded specimens or sticks of approximately 1.0 mm^2 in cross-sectional area. Five sticks for each tooth were tested at this time (seven days) or after six months. The sticks were individually fixed to a custom-made testing with a cyanoacrylate adhesive (Loctite Super Bond Gel; Henkel, Düsseldorf, Germany) and subjected to tensile load at a crosshead speed of 1.0 mm/min until failure (EZ Test, Shimadzu, Kyoto, Japan). Dimensions of each sides of the bonded specimen were measured using a digital micrometer (Mitutoyo Co., Tokyo, Japan) and the MTBS values were expressed in MPa.

Failure mode was observed in a stereomicroscope at a 100x magnification (PanTec, Panambra Ind. e Técnica SA, São Paulo, Brazil) and scored according to one of three failure modes: cohesive failure in dentin, adhesive failure between dentin and composite and cohesive failure in composite resin. Instead of classifying failure as mixed, the area percentage of each type of failure in each fractured specimen was recorded.

Hybrid layer

Two sticks of each group were sequentially polished, coated with carbon (MED 010) and hybrid layer was observed under Scanning Electron Microscopy (LEO 440 VP). Representative areas of the interfaces were photographed.

Statistical analysis

Five slabs were tested for each tooth, and the mean bond strength value was considered (n=6). Bond strength values were statistically analyzed by Repeated-Measures ANOVA in SPSS, followed by Fisher´s LSD Tests at 95% confidence level.

RESULTS

No pre-test failures occurred. The power observed were 0.87 at one week and 0.86 at six months. There was no statistical significance between MTBS at one week and six months. The results of the MTBS are given in table 2. Statistical significant differences were observed among the tested adhesive groups ($p < 0.05$). The SBU-ER, SBU-SE, and SBP showed the highest MTBS means and statistically differed from CSE and LPOP according to Fisher's LSD tests. The OSP group showed intermediary MTBS values and did not differ from the other groups except from LPOP that showed the lowest MTBS values (Table 2).

Table 2- Means and standard deviations (SD) in MPa of microtensile bond strengths, and Fisher's LSD test results for each adhesive system.

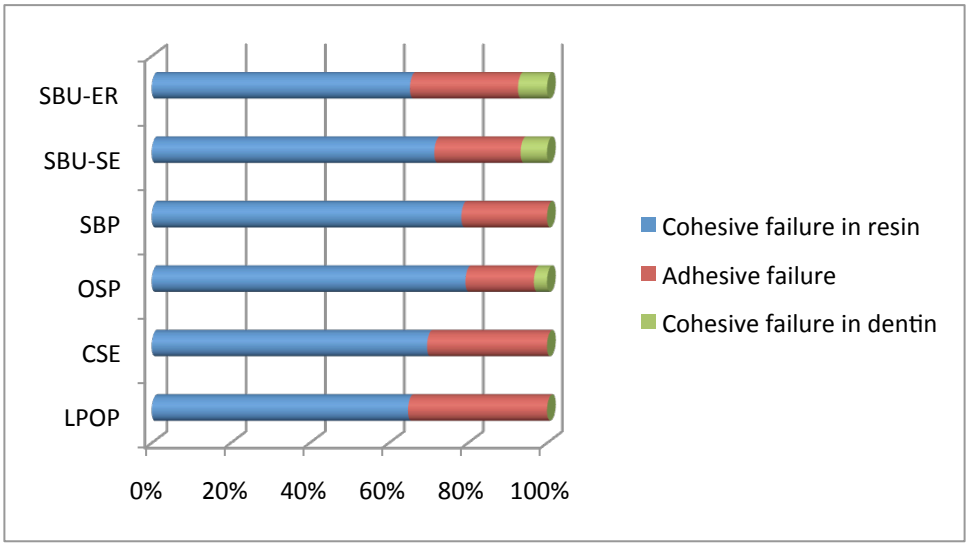
Groups	*Mean at one week (sd)*	*Mean at six months (sd)*	*General Mean (sd) Tukey HSD**

SBU-ER	61.0 (14.1)	63.8 (10.3)	63.8 (10.2) A
SBU-SE	61.6 (23.3)	60.3 (16.5)	60.2 (16.5) A
SBP	61.7 (17.0)	60.7 (15.7)	60.7 (15.7) A
OSP	58.6 (10.2)	59.8 (9.6)	60.0 (9.7) AB
CSE	41.4 (16.2)	47.8 (13.5)	47.8 (13.4) BC
LPOP	35.5 (15.5)	36.9 (17.7)	36.9 (17,8) C

*Different letters indicate statistically significant differences ($p < 0.05$).

The most frequent failures in all groups were cohesive in resin, followed by adhesive failure. The groups SBP, CSE, and LPOP showed no cohesive failure in dentin (Graph 1).

Graph 1 – Percentage of failure types in the experimental groups



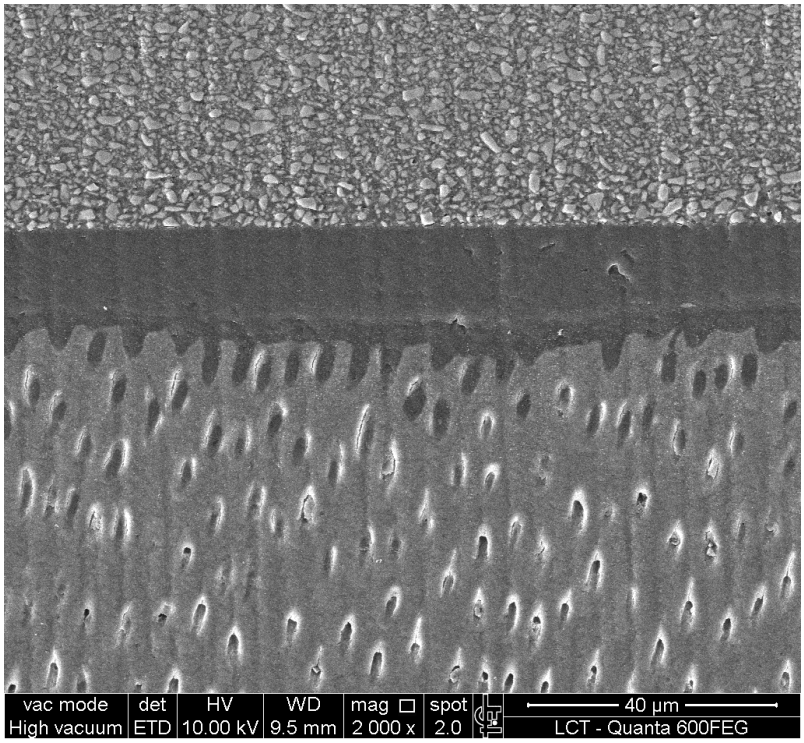


Figure 1 - Representative images of the hybrid layer in the group treated with Scotchbond Universal on etching and rinse mode.

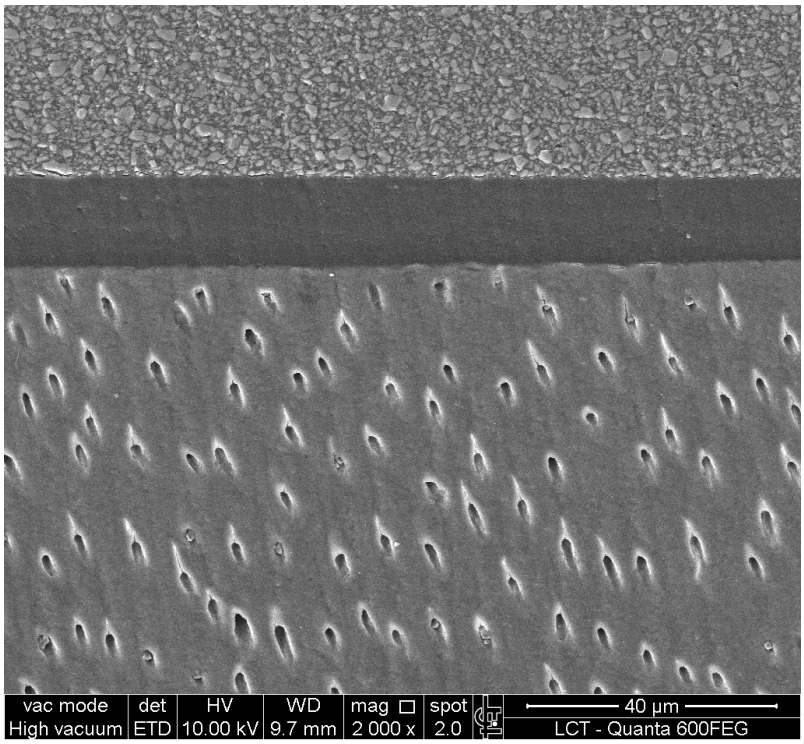


Figure 2 - Representative images of the hybrid layer in the group treated with Scotchbond Universal on self-etching mode.

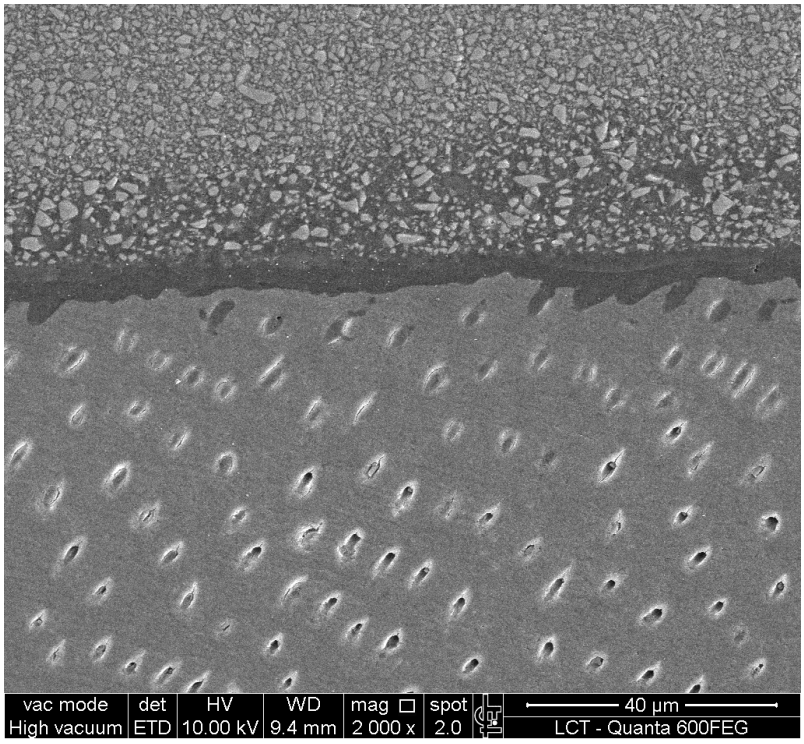


Figure 3 - Representative images of the hybrid layer in the group treated with Adper Single Bond Plus.

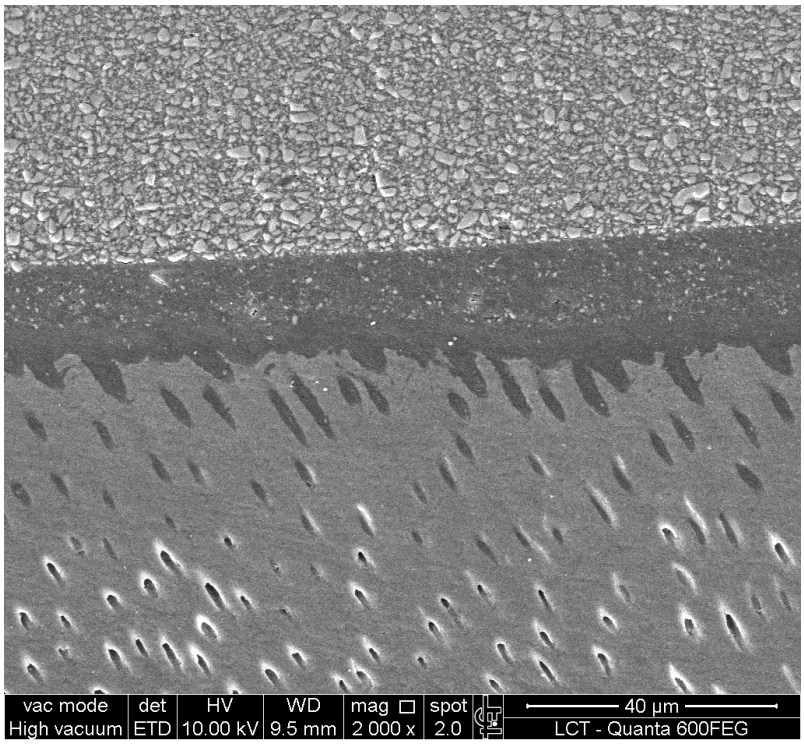


Figure 4 - Representative images of the hybrid layer in the group treated with Optibond Solo Plus.

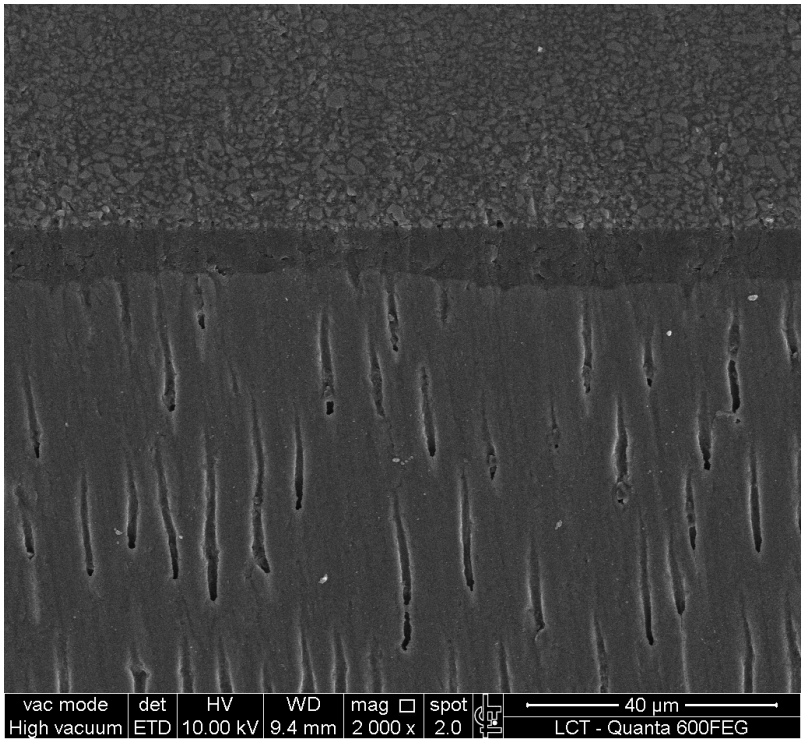


Figure 5 - Representative images of the hybrid layer in the group treated with Clearfil SE Bond.

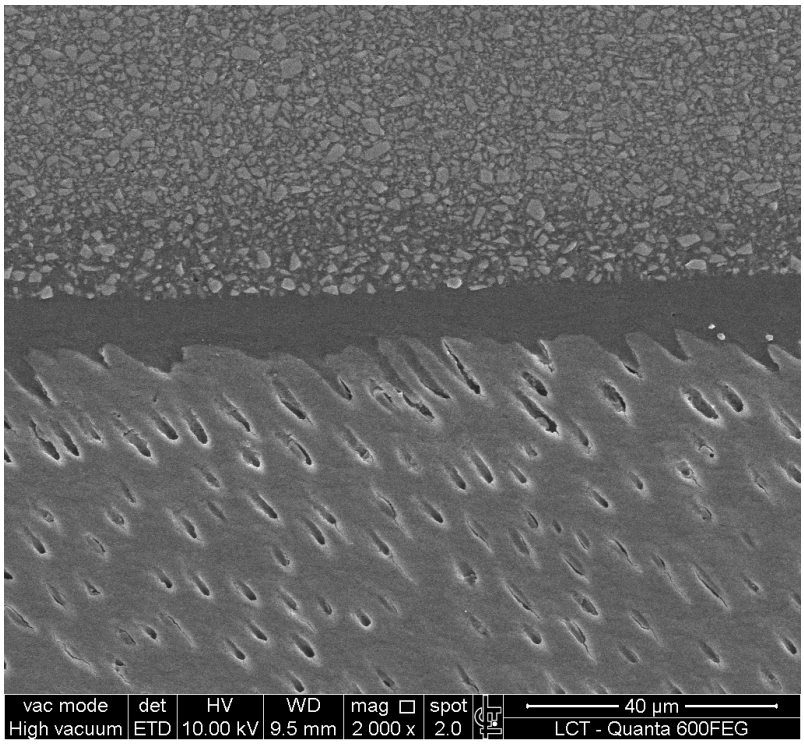


Figure 6 - Representative images of the hybrid layer in the group treated with Adper Prompt L-Pop.

DISCUSSION

The bond of composite resins to dentin has been the main subject of numerous studies. The water “wet bonding” technique solve the major problem of collagen collapse after acid-etching and resulted in better resin penetration into acid etched dentin (Liu et al., 2011). However, the adhesive resins are still unable to infiltrate in all etched dentin and some voids may remain under the hybrid layer causing increase in the sensitivity and hydrolytic degradation (Hara et al., 1999).

As a promise the SEAs were developed. Theoretically, they are able to demineralize and infiltrate dentin simultaneously (Carvalho et al., 2005). However, they are not as efficient in enamel as in dentin and results in low bond strength values to enamel. Consequently, the bond between enamel and resin fails after some time of function in the mouth (Kiremitçi et al., 2004). Clinically, the selective enamel etching technique may reliable to create microscopic roughness in enamel to provide the micromechanical retention and avoid the over etch of dentin. Then, both substrates enamel and dentin may be well conditioned to promote a good interaction with a self-etching adhesive.

However, some studies with SEAs have been showed lower bond strengths when accidentally dentin is acid etched. It happens due to a resin failure, in the reason of the adhesive resin could not penetrate deeply enough between the demineralized dentin (Opdam, 1998; Cardoso, 1999). Since the balance between dentin demineralization depth and extent of monomer penetration of the self-etching adhesives is the key to a good quality bond between enamel and resin (Sano, 1994), elective etching adhesives has been developed.

This study evaluated the null hypothesis that an elective etching adhesive system has the same bond strength to dentin as etch-and-rinse adhesives systems, and also using the etch-and-rinse or the self-etching approaches and the results lead to the acceptance of the null hypothesis in short or long-term laboratorial research.

Although, phosphoric acid can demineralize dentine more deeply than a mild SEA, the tested elective etching adhesive system (SBU) showed bond strength values compatible to the etch-and-rinse adhesive systems (SBP and

OSP) in both modes of use, self-etching and etch-and-rinse, and are in agreement with Perdigão et al., (2012) which found that SBU groups ranked in the same statistical subset regardless of the dentin treatment. Hanabusa et al. (2012) also found similar results to another elective adhesive (G-Bond Plus) in the immediate bond strength to dentin with self-etch or etch-and-rinse approach. Also the experimental groups, SBU-ER and SBU-SE, showed more incidence of cohesive and adhesive failures.

Mena-Serrano et al. (2013), found in a dentin class V clinical trial that behavior of the multimode adhesive does not depend on the bonding strategy at 6 months. In the present study the storage in water also do not impairs the bond strength showing the stability of self-etching (SE) and etch-and-rinse (ER) approaches.

This elective etching adhesive system (SBU) has a mild pH (2.7), and a composition similar to others etch-and-rinse systems available which may explain the behavior similar to the etch-and-rinse systems. However, it has the functional MDP (10-methacryloyloxydecyl dihydrogen phosphate) monomer, which provides suitable self-etching properties to be used in this mode. Although the MDP monomer is present in the CSE group, it showed lower bond strength than SBU in the self-etching mode (SE). MDP can chemically interact with hydroxyapatite imparting better resistance towards degradation by prevention of micro and nanoleakage (Yoshida et al., 2004).

Recently, some researcher have described the presence of a self-assembled nano-layering as a result from interaction of the functional MDP, first with synthetic hydroxyapatite (Perdigão et al., 2012; Shinohara et al., 2006), followed with enamel and dentin (Di Hipólito et al., 2012). This layer provides multi-functional properties to the interface with, in particular, direct benefit to bond durability (Koshiro et al., 2006). It seems that the strong hydrophobic nature of the nano-layered structure may help to protect the hybrid layer against biodegradation (Breschi et al., 2008), improving the protection of collagen against degradation, it turns the residual hydroxyapatite more resistant to acidic dissolution (Pashley et al., 2011).

Both SBU and CSE adhesives may present such self-assembled nano-layering of two 10-MDP molecules, joined by stable MDP-Ca salt formation, and development of a stronger phase at the adhesive interface, which must

also increase the mechanical strength of the adhesive and may explain the documented favorable bond strength of this adhesives and clinical longevity of bonds (Yoshida et al., 2003)

However, the CSE has been demonstrated lower enamel bond strength values than the conventional adhesive systems, both when applied as self-etching or with previous acid-etching (Carvalho, Turbino, 2009). Therefore, the previous enamel etching did not improve enamel bonding and also could not improve dentin bonding. Probably the higher bond strength observed to SBU-SE may be due to interaction with other compounds as the Vitrebond™ copolymer, which is related to improve adhesion to moist and dry dentin and result in some chemical bond to mineral phase of dentin and enamel or due to the type and percentage of solvent which could allow more penetration in conditioned dentin (Mitra et al., 2009).

Though, the control self-etching adhesives CSE and LPOP showed more incidence of cohesive failures in dentin, they presented the higher percentage of adhesive failures in this study which are in agreement with the lower bond strength verified in the microtensile test.

SBU performed well in the self-etching (SBU-SE) mode and etch-and-rinse (SBU-ER) mode and provides the flexibility for the clinician to choose one adhesive to use independent of their preference of technique to achieve bond strength to dentin compatible with usual etch-and-rinse adhesives systems.

However, this is one the firsts studies evaluating the bond strength of an elective adhesive system to dentin and more information are needed in respect to its interaction with dentin in etch-and-rinse mode to form the hybrid layer and also the interaction with dentin and smear layer in self-etching mode followed by the clinical trials

CONCLUSION

The in vitro use of electing etching adhesive system in the dentin with the etch-and-rinse or self-etching modes do not compromise the bond strength and showed stable bonds after six months of water storage.

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4- CONCLUSÃO

De acordo com os resultados observados, no artigo redigido, pode-se concluir que o uso dos adesivos de condicionamento ácido eletivo podem ser utilizados como autocondicionantes ou com condicionamento ácido prévio na dentina sem causar prejuízos a resistência de união.

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Anexo 1 – Saídas da análise estatística

Within-Subjects Factors

Measure: MEASURE_1

MTBS	Dependent Variable
1	MTBSa
2	MTBSb

Between-Subjects Factors

		Value Label	N
Adhesives	1,00E+000	CSE	6
	2,00E+000	SBY-SE	6
	3,00E+000	SBU-ER	6
	4,00E+000	SBP	6
	5,00E+000	OSP	6
	8,00E+000	LPOP	6

Box's Test of Equality of Covariance Matrices[a]

Box's M	8,024
F	,443
df1	15
df2	4922,741
Sig.	,967

Tests the null hypothesis that the observed covariance matrices of the dependent variables are equal across groups. a. Design: Intercept + Adhesives Within Subjects Design: MTBS

Multivariate Tests[a]

Effect		Value	F	Hypothesis df	Error df	Sig.	Noncent. Parameter	Observed Power[c]
MTBS	Pillai's Trace	,011	,340[b]	1,000	30,000	,564	,340	,087
	Wilks' Lambda	,989	,340[b]	1,000	30,000	,564	,340	,087
	Hotelling's Trace	,011	,340[b]	1,000	30,000	,564	,340	,087
	Roy's Largest Root	,011	,340[b]	1,000	30,000	,564	,340	,087
MTBS * Adhesives	Pillai's Trace	,029	,179[b]	5,000	30,000	,968	,897	,086
	Wilks' Lambda	,971	,179[b]	5,000	30,000	,968	,897	,086
	Hotelling's Trace	,030	,179[b]	5,000	30,000	,968	,897	,086

	Roy's Largest Root	,030	,179[b]	5,000	30,000	,968	,897	,086

a. Design: Intercept + Adhesives Within Subjects Design: MTBS

b. Exact statistic

Post Hoc Tests Adhesives

Multiple Comparisons

Measure: MEASURE_1

	(I) Adhesives	(J) Adhesives	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
						Lower Bound	Upper Bound
LSD	CSE	SBY-SE	-16,3048*	7,48364	,037	-31,5885	-1,0212
		SBU-ER	-17,7927*	7,48364	,024	-33,0763	-2,5090
		SBP	-16,5662*	7,48364	,035	-31,8499	-1,2826
		OSP	-14,6777	7,48364	,059	-29,9613	,6060
		LPOP	8,4150	7,48364	,270	-6,8686	23,6987
	SBY-SE	CSE	16,3048*	7,48364	,037	1,0212	31,5885
		SBU-ER	-1,4878	7,48364	,844	-16,7715	13,7958
		SBP	-,2614	7,48364	,972	-15,5450	15,0223
		OSP	1,6272	7,48364	,829	-13,6565	16,9108
		LPOP	24,7199*	7,48364	,002	9,4362	40,0035
	SBU-ER	CSE	17,7927*	7,48364	,024	2,5090	33,0763
		SBY-SE	1,4878	7,48364	,844	-13,7958	16,7715
		SBP	1,2265	7,48364	,871	-14,0572	16,5101
		OSP	3,1150	7,48364	,680	-12,1686	18,3986
		LPOP	26,2077*	7,48364	,001	10,9241	41,4914
	SBP	CSE	16,5662*	7,48364	,035	1,2826	31,8499
		SBY-SE	,2614	7,48364	,972	-15,0223	15,5450
		SBU-ER	-1,2265	7,48364	,871	-16,5101	14,0572
		OSP	1,8885	7,48364	,802	-13,3951	17,1722
		LPOP	24,9813*	7,48364	,002	9,6976	40,2649
	OSP	CSE	14,6777	7,48364	,059	-,6060	29,9613
		SBY-SE	-1,6272	7,48364	,829	-16,9108	13,6565
		SBU-ER	-3,1150	7,48364	,680	-18,3986	12,1686
		SBP	-1,8885	7,48364	,802	-17,1722	13,3951
		LPOP	23,0927*	7,48364	,004	7,8091	38,3764
	LPOP	CSE	-8,4150	7,48364	,270	-23,6987	6,8686
		SBY-SE	-24,7199*	7,48364	,002	-40,0035	-9,4362
		SBU-ER	-26,2077*	7,48364	,001	-41,4914	-10,9241
		SBP	-24,9813*	7,48364	,002	-40,2649	-9,6976
		OSP	-23,0927*	7,48364	,004	-38,3764	-7,8091