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Investigation

Adhesion of dental sealants to enamel with self-etching adhesives in salivary contamination conditions: Influence of the light curing protocol

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ABSTRACT

Purpose: To evaluate the influence of the light curing protocol in the shear bond strength of a sealant to enamel treated with two self-etching adhesive systems, in salivary contamination conditions.
Materials and Methods: The dental sealant (Delton, Dentsply) was applied, after saliva contamination, onto the vestibular enamel of sixty human incisors treated with Xeno III (Dentsply) or Prompt-L-Pop (3M/Espe). These two groups were further divided into two subgroups (n = 15) according to the curing time: 1) the adhesive system was cured with the sealant (co-polymerization), and 2) adhesive and sealant were light cured independently (independent polymerization). After the adhesive procedures, specimens were stored in water (37 °C–24 h) and thermal-cycled. Shear bond strength tests were done in an universal testing machine. Data was analyzed with two-way ANOVA.
Results: There were no statistical differences (p = 0.267) between the adhesive systems tested. The co-polymerization groups (33.3 ± 9.4 MPa) yielded statistically higher shear bond strength values than the independent polymerization groups (28.2 ± 4.7 MPa). Even with saliva contamination, the self-etching adhesive systems used yielded high shear bond strength values.
Conclusions: In the conditions tested, the co-polymerization of the adhesive systems with the sealant led to higher bond strength values to enamel than the independent polymerization.

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Adesão de selantes dentários ao esmalte, com auto-adesivos autocondicionantes em condições de contaminação salivar: Influência do protocolo de fotoativação

Palavras-chave:
Selante de fissuras
Contaminação salivar
Sistemas adesivos auto-condicionantes
Fotopolimerização

R E S U M O

Objectivos: Avaliar a influência do momento da fotopolimerização de dois sistemas adesivos auto-condicionantes, nos valores de resistência adesiva de um selante de fissuras ao esmalte, em condições de contaminação salivar.
Materiais e Métodos: O selante de fissuras (Delton, Dentsply) foi aplicado, após contaminação salivar, sobre o esmalte vestibular de sessenta incisivos humanos condicionados com Xeno III (Dentsply) ou Prompt-L-Pop (3M/Espe). Estes dois grupos foram posteriormente divididos em dois subgrupos (n = 15) de acordo com o momento da fotopolimerização do sistema adesivo: 1) o sistema adesivo foi fotopolimerizado conjuntamente com o selante (co-polymerization), e 2) sistema adesivo e selante foram fotopolimerizados independentemente (independent polymerization). Os espécimes foram armazenados em água (37 °C–24 h) e, posteriormente, submetidos a termociclagem. Os ensaios de resistência adesiva foram realizados numa máquina de testes universal. Os resultados foram submetidos a análise estatística ANOVA duas vias.
Resultados: Não foram registadas diferenças estatisticamente significativas (p = 0,267) entre os sistemas adesivos usados. Os grupos co-polimerizados obtiveram valores de resistência adesiva mais elevados (33,3 ± 9,4 MPa) relativamente aos grupos em que a polimerização foi realizada de forma independente (28,2 ± 4,7 MPa). Mesmo na presença de contaminação salivar, os sistemas adesivos auto-condicionantes testados obtiveram valores de resistência adesiva elevados.
Conclusões: Nas condições testadas, a co-polimerização do sistema adesivo com o selante originou valores de resistência adesiva ao esmalte superiores relativamente à polimerização independente destes dois materiais.

Introduction

The occlusal surfaces of the newly erupted posterior teeth are particularly susceptible to carious lesions due to local conditions such as incomplete maturation of the enamel, infra-occlusion and very complex occlusal anatomy. Removing the bacterial plaque under such conditions is difficult, and those surfaces are, consequently, the most affected by caries.[1,2]

Dental sealants have shown to be a very efficient aid in the prevention of dental caries in susceptible patients.[3,4] However, fissure sealant application must be included in a general prevention program that embraces a correct hygiene instruction and motivation of the child and parents, a controlled diet and a regular professional control.[3]

The classic technique for the application of the sealants includes polishing the tooth surface with pumice and water, conditioning enamel with 35-37 % phosphoric acid gel for 30 s, rinsing and drying, and applying the sealant. However, rinsing the tooth can be unpleasant and may contribute to a disruptive behavior, particularly in young children.[4,5] Thus, mainly when complete isolation is not possible, contamination due to saliva easily occurs and enamel's surface energy decreases, commonly leading to sealant's failure.[6-8]

Some studies have shown that in saliva contamination conditions, the application of an adhesive system before the sealant increases shear bond strength and minimizes microleakage, comparatively to the classical technique.[2,4,6,7,9-14] The application of adhesive systems before the sealant has been describe since 1992. This technique depends on the mechanical retention promoted by the acid conditioning of the enamel and the chemical adhesion between the adhesive and the sealant.[9] Furthermore, with the self-etching adhesives the time and complexity of the treatment is considerably reduced.[15]

However, we found in the literature just one study on the ideal time to light cure adhesive and sealant.[4] In 2005, Torres et al.[4] concluded that in salivary contamination conditions, shear bond strength was not affected by the light curing protocol. Nonetheless, in the same study, the co-polymerization between the adhesive and the sealant, in dry conditions, yielded higher bond strength values than the independent polymerization of the components.

The purpose of the present study was to evaluate the influence of two self-etching adhesive systems and two light curing protocols in the shear bond strength of a filled sealant to enamel, in salivary contamination conditions.

Materials and methods

Specimens used in this study were prepared from randomly selected human non-carious permanent incisors. After

Table 1 - Sealant and adhesive materials used in the study

Material	Composition	Batch n.º	Function
Prompt-L-Pop	Liquid A: HEMA, Purified water, Ethanol, BHT, Highly dispersed silicon dioxide Liquid B: Phosphoric acid modified methacrylate, Mono-fluoro phosphazene modified methacrylate, Urethane dimethacrylate, BHT, Camphoroquinone, Ethyl-4-dimethylaminobenzoate	290226	Adhesive system
Xeno III	Liquid A: Methacrylated phosphoric esters, Bis-GMA, Initiators based on camphorquinone, Stabilizers Liquid B: Water, HEMA, Polyalkenoic acid, Stabilizers	608000376	Adhesive system
Delton	Low viscosity monomers, Triethylene glycol dimethacrylate, Bis-GMA, Titanium dioxide, Sodium fluoride, Barium alumino fluroboro silicate glass (38%), Polymerization initiator, Stabilizer	70125	Sealant

Table 2 - Experimental design

Group 1 Co-polymerization		**Group 2 Independent polymerization**
	1 - Application of the self-etching adhesive system XIII 2 - Salivary contamination	
3 - Sealant application 4 - Co-polymerization 20 s		3 - Adhesive polymerization 10 s 4 - Sealant application 5 - Sealant polymerization 20 s
Group 3 Co-polymerization		**Group 4 Independent polymerization**
	1 - Application of the self-etching adhesive system PLP 2 - Salivary contamination	
3 - Sealant application 4 - Co-polymerization 20 s		3 - Adhesive polymerization 10 s 4 - Sealant application 5 - Sealant polymerization 20 s

extraction, the sixty teeth used in this study were stored in 0.5% chloramine solution for up to 15 days and then stored in distilled water, at 4 °C, until the beginning of the study.[16]

The adhesive systems and sealant used in this study are listed in table 1, along with the composition, batch numbers and codes. All the materials were used according to manufacturer's instructions.

Teeth were fitted on plate A of a Watanabe single-plane lap shear test used to realize the bond strength tests.[17] On each plate, a plastic film (Mylar, Dupont, Wilmington, DE, USA) with a 3 mm diameter's hole was adapted to standardize the adhesion area. The vestibular surface of each tooth was placed on the film with its long axis parallel to the long axis of the plate. The adhesion protocol was then started, applying one of the light curing unfilled self-etching adhesive systems studied, to the enamel of the vestibular surface of the teeth. Xeno III (Dentsply, Konstanz, Germany) was applied on 30 teeth and Prompt-L-Pop (3M/Espe, Seefeld, Germany) was used in other 30 teeth. All specimens were then submitted to contamination with 1 μl of operator's fresh saliva. After this procedure, each group was divided in two other groups according to the light curing protocol used (table 2). Thus, the 60 teeth were randomly divided in four experimental groups (n = 15).

The sealant (Delton, Dentsply, Konstanz, Germany), was applied into the enamel surface with a cylindrical polyvinylsiloxan (Express STD, 3M/Espe) mould with 3.1 mm diameter and 3 mm height. The sealant height inside the mould was about 1 mm and the rest of the mould was filled with Z100 resin (3M/Espe, lot 3021A2) and light cured for 40 s (20 s with the mould and 20 s without it), with a light curing unit (3M/Espe, XL 3000) with 450 mW/cm^2.

Samples were stored at 37 °C, in humidity environment, for 12 h, and then thermal-cycled in 500 cycles (5.5 °C–55 °C).

After these procedures the plate B of the shear test apparatus was set. In this plate, Z100 was applied around the cylinder (sealant + Z100) and cured for 40 s. The space around the dental specimen was filled with type IV dental stone (Gilstone, BK Giulini, Ladenburg, Germany).

Specimens were stored for another 12 h, in the same conditions described above. The shear bond strength tests were done using an universal testing machine (Instron Corp. 4502, Canton, MA, USA) with a cross head speed of 5 mm/min.

Failure mode was analyzed with a stereomicroscope (Meiji Techno, Saitama, Japan) at 20X magnification. The failures were classified in one of three types: type 1–less than 30% of sealant on the adhesion area; type 2–30% to 70% of sealant on the adhesion area; type 3–more than 70% of sealant on the adhesion area.

Data was analyzed with two-way ANOVA, and Tukey Kramer post-hoc test (StatView, version 5.0, SAS Institute, Cary, NC, USA).

Results

The results of this in-vitro study are presented in figure 1.

The highest mean bond strength values were obtained when Prompt-L-Pop was co-polymerized with the sealant (33.751 ± 10.822 MPa) and the lowest mean values were obtained when Xeno III and the sealant were independently polymerized (26.562 ± 5.148 MPa).

As presented in table 3, the curing protocol had a significant influence in the bond strength ($p = 0.0116$). The co-polymerization of the adhesive system and the sealant yielded statistical higher bond strength values, independently of the adhesive used. There were no significant differences ($p > 0.05$) between the bond strength values achieved with the two adhesive systems.

Adhesive failure modes, type 1 (65%) were the most detected, followed by type 2 (23%) and finally type 3 failures (12%).

Discussion

The effectiveness of the correct application of fissure sealants in occlusal caries prevention has been demonstrated by several authors.[3,18-21]

However mainly due to saliva contamination sealant adhesion may fail leading to partial/total loss of sealant and, what is worst, to secondary caries.

In the presence of contamination, studies are unanimous relatively to the advantage of using an adhesive system under the sealant.[2,6,7,10-14] Even in the absence of salivary contamination, some authors concluded that adhesive system application is advantageous, since the bond strength values achieved are higher compared to the classic technique.[11,22] However other authors considered these differences to be insignificant and emphasized the increase in the clinical time of this procedure.[17,23]

With the emergence of 6th generation adhesive systems, the adhesion procedure has become faster.[24] Nonetheless, serious doubts persist on the effectiveness of etching enamel with these systems because of their higher pH compared to orto-phosphoric acid, ally to the hipermineralization of superficial enamel.[15,25] In 2001, Koh et al[26] performed a comparative study with several

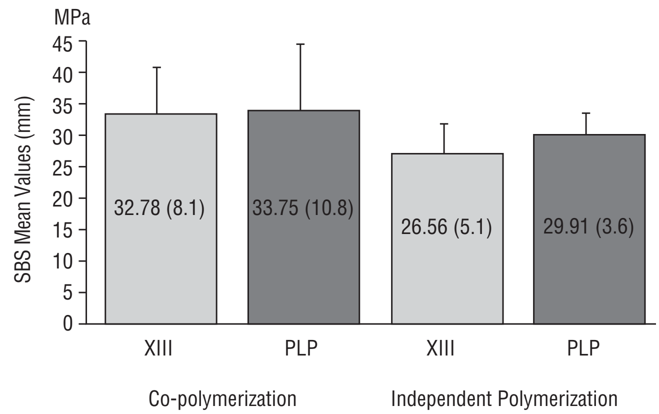

Figure 1 - Mean (standard deviation) shear bond strength values in MPa (XIII –Xeno III; PLP–Prompt-L-Pop).

4th and 6th generation adhesive systems. The results of the shear bond strength to enamel were very similar with the two types of adhesive systems. Prompt-L-Pop (pH = 1) and Xeno III (pH < 1) are considered aggressive systems,[27] and the enamel demineralization pattern produced by them is similar to that promoted by 4th generation adhesives[7,28] with the advantage of simplifying the technique and reducing clinical time.

The self-etching adhesives used in this study allowed satisfactory bond strength values between the sealant and the superficial enamel of human teeth, with no differences between the two adhesive systems tested. However, a relevant result was found. With saliva contamination, the co-polymerization between the adhesive and the sealant yielded higher shear bond strength values compared to the independent polymerization of the two materials. This brings us to the suggestion of co-polymerizing the sealant with a self-etching adhesive in patients where a complete isolation is difficult to achieve. In the study performed by Torres et al,[4] different results were found. They concluded that, in contamination conditions, the light-curing protocol didn't affect the shear bond strength. However, they used a 5th generation adhesive system and performed the salivary contamination on the etched enamel surface. We may, therefore, consider that the authors of that particular study did not found differences in the curing protocol, because the contamination was the main variable that really affected the adhesion, since they have used total-etch adhesive systems. In our study we used self-etching adhesives and although saliva contamination was a constant in all groups, the bond strength values obtained were satisfactory.

Relatively to the failure mode of the specimens in our work, type 1 was the most detected (65%), indicating that the

Table 3 - Two way ANOVA

Dimensions	Degree of freedom	Sum of squares	Mean of squares	F Value	P Value
Adhesive	1	70.006	70.006	1.258	0.2668
Polymerization	1	379.302	379.302	6.818	0.0116*
Adhesive*Polymerization	1	21.189	21.189	0.381	0.5396
Residual	56	3115.6	55.636		

adhesion achieved was lower than the cohesive resistance of the filled sealant used.

In the present study, a filled sealant, widely studied in the literature, was used.[29-31] Since its bond strength is reported to be superior than an unfilled sealant, it should be the first choice in clinical practice. However, a filled sealant is less fluid, and subsequently more resistant to the hybridization of etched enamel. The application of an adhesive system before the sealant could be advantageous to the bond strength of this type of sealants.[32] In the future, it would be important to study the microleakage of a filled fissure sealant applied to enamel treated with a self-etching adhesive system, with the two polymerization protocols followed in this study, mainly to evaluate the filled sealant adaptation to every type of fissures.

Conclusions

Within the limitations of this *in-vitro* study, it was concluded that the self-etching adhesive systems Prompt-L-Pop and Xeno III allowed similar and satisfactory shear bond strength values between a fissure sealant and superficial enamel.

In saliva contamination conditions, the co-polymerization of the adhesive system and the sealant yielded higher bond strength values of the sealant to the enamel than the isolated polymerization of the components.

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