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ORIGINAL ARTICLE

Influence of cleansing agents on enamel and dentin bond strength to a one-step self-etching adhesive system

Influência de agentes de limpeza em esmalte e dentina na resistência de união de um sistema adesivo auto-condicionante de passo único

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ABSTRACT

Objective: The aim of this study was to detect the influence of application mode of cleansing agents, on enamel and dentin during the adhesion process of a one-step self-etching adhesive system. **Materials & Methods:** 24 human molars were sectioned into halves along the long axis for enamel and dentin tests. The cleansing agents used were 3% hydrogen peroxide (group HP), anionic detergent (group AD), and antiseptic solution (group AS). In the experiment, these agents were applied with (active) and without friction (passive) on enamel and dentin surfaces. A one-step self-etching adhesive system (Clearfil S3 Bond) was applied on dental surface and composite resin cylinders were built using Tygon tubing molds. After 24 h, μSBS (microshear) test (1 mm/ min) and fracture analysis were performed. **Results:** There was no statistically significant difference in bond strength values regarding the application mode of cleansing agents for enamel and dentin ($p \geq 0.05$). The use of antiseptic solution on enamel resulted in bond strength results significantly higher when compared with HP and AD ($p = 0.014$). **Conclusions:** Regarding bond strength to enamel and dentin using one-step self-etching adhesive system, application mode of the cleansing agents used in this study presented the same behavior. Antiseptic solution applied on enamel before the application of a one-step adhesive system increased bond strength performance.

KEYWORDS

Cleansing Agents; Dental Bonding; Dental Adhesives.

RESUMO

Objetivo: O objetivo deste estudo foi detectar a influência do modo de aplicação de agentes de limpeza, em esmalte e dentina durante o processo de adesão de um sistema adesivo auto-condicionante de passo único. **Materiais e Métodos:** 24 molares humanos foram seccionados em duas metades ao longo do eixo para testes de esmalte e dentina. Os agentes de limpeza utilizados foram: peróxido de hidrogênio 3% (grupo HP), detergente aniônico (grupo AD) e solução anti-séptica (grupo AS). No experimento, esses agentes foram aplicados com fricção (ativa) e sem fricção (passiva) em superfícies de esmalte e dentina. O sistema adesivo auto-condicionante de um passo (Clearfil S3 Bond) foi aplicado na superfície dental e cilindros de resina composta foram construídas usando moldes (Tubos Tygon). Após 24 h, o teste de microcisalhamento (1 mm / min) e análise de fratura foram executados. **Resultados:** Não houve diferença estatisticamente significante nos valores de resistência de união em relação ao modo de aplicação de agentes de limpeza para esmalte e dentina ($p \geq 0,05$). O uso de solução anti-séptica no esmalte resultou em resultados de resistência de união significativamente maior quando comparada com HP e AD ($p = 0,014$). **Conclusões :** Em relação à resistência de união ao esmalte e dentina utilizando sistema adesivo auto-condicionante de passo único, o modo de aplicação dos agentes de limpeza utilizados neste estudo apresentaram o mesmo comportamento. A aplicação da solução anti-séptica aplicada sobre o esmalte antes da aplicação de um sistema adesivo de passo único apresentou melhor desempenho de resistência de união.

PALAVRAS-CHAVE

Agentes de limpeza; Cimentos Dentários; Adesivos dentários.

INTRODUCTION

Adhesion to dentin depends on the interaction of adhesive system and the dentin substrate. Their longevity is affected by the preparations coarseness and the dentin-cleaning agent used.[1]

The combination between these cleaning agents and mechanical action are the base of endodontic therapy. Sodium hypochlorite and hydrogen peroxide have been widely used for many years because of their antimicrobial properties[2].

Although sodium hypochlorite alters the mechanical properties of root dentin when used as an endodontic irrigant[3, 4], it is used in order to eliminate both organic and contaminated intracanal contents from dentinal walls by chemomechanical preparation. An antiseptic solution consisting of sodium hypochlorite (0.4% to 0.5%) and boric acid (4%) (Dakin's solution) is frequently used to disinfect canal walls, dissolving remaining pulp tissue.[5]

Hydrogen peroxide has also been used in treating gingival disorders.[6] The concentration level and extent of exposure are the most important features, but the presence of organic and inorganic materials also influences the efficacy of this agent[6].

In dentistry, anionic detergent solution (0.125% sodium sulfate lauryldietyleneglycol ether) is used as a cavity cleanser[7, 8]. It has a low surface tension and high penetration power, adsorption and emulsion properties that assists in surface cleansing.[9]

It is already known that pretreatment with chlorhexidine and ethanol did not affect the bond strength of dentin to a total etch adhesive system[10], but little information is available concerning the effect of the agents aforementioned on adhesive performance on dental substrate. Another point of interest concerns the application mode of these agents and how they influence the performance of adhesive systems.

Once these cleansing agents are used to improve the results of restorative procedures, the aim of this study was to evaluate, by microshear bond strength test, the influence of: (1) application mode of some cleansing agents; (2) the effect of these cleansing agents applied on enamel and dentin during adhesion process of a one-step adhesive system.

The null hypothesis tested were that: (1) bond strength of a one-step self-etching adhesive system is not influenced by the application of cleansing agents; (2) bond strength of a one-step self-etching adhesive system is not influenced by the application mode of cleansing agents.

MATERIAL & METHODS

SAMPLE PREPARATION

Samples of 24 freshly extracted, caries-free human molars, stored in distilled water for no longer than 3 months, were used in this study, which was approved by the Research Ethics Committee of the University of São Paulo (USP) and had the informed consent of the donors (protocol 170/06). The teeth were ground flat for enamel tests and then they were ground to the point of dentin exposition for dentin tests.

Experiment

The different cleansing agents used were all handled (Fórmula & Ação, São Paulo, SP, Brazil) and divided into 3 groups: Group HP: 3% hydrogen peroxide (n = 8); Group AD: anionic detergent solution (0.125% sodium sulfate lauryldietyleneglycol ether) (n = 8), Group AS: antiseptic solution consisting of sodium hypochlorite (0.4% to 0.5%) and boric acid (4%) (Dakin's solution) (n = 8). They were applied on enamel and dentin surfaces in two different ways as described below (two subgroups):

- Subgroup 1 (With friction/active) (n = 4): Cotton pellets soaked with each of the cleansing agents were applied actively for 10 s on the surface.

- Subgroup 2 (Without friction/passive) (n = 4): The cleansing substances were applied passively using a syringe to cover all surfaces, and were left undisturbed for 10 s.

After applying the above-mentioned cleansing agents, the surfaces were rinsed with water spray for 10 s and gently air-dried.

Sample restoration

A one-step sef-etching system (Clearfil S3 Bond/ batch 00026A, Kuraray Co, Osaka, Japan) was applied with a disposable microbrush to enamel and dentin, acoording to manufacturers left for 20 s, dried with high-pressure air flow (more than 5 s) and light-cured for 10 s (J Morita USA Inc., CA, USA) with an average power of 540 mW/cm^2.

Prior to light-curing of the bonding resin, Tygon tubing molds (R-3603, Norton Performance Plastic Co., Cleveland, OH, USA) were mounted on the enamel and dentin surface to limit the bonding area. A micro-hybrid composite resin, shade A3 (Clearfil AP-X/ batch 01042A, Kuraray Co., Osaka, Japan) was placed into the molds with a celluloid sheet matrix placed over the resin, gently flat pressed and photo-cured for 20 s. The manufacturer's recommendations on how to use the adhesive system and resin composite were strictly followed during the restorative procedure.

Because the Tygon molds were bonded tightly to the tooth surface by the simultaneous photo-curing process of the bonding resin, no flash of composite resin extended onto the surface beyond the base of the mold. In this manner, two to four molds of resin, approximately 0.8 mm in diameter and 0.5 mm in height, were bonded to each dental surface. Specimens were stored at 23 °C for one hour prior to removing molds with a scalpel blade. The specimens were then stored in distilled water at 37 °C for 24 h.

Microshear bond strength test (μSBS) (ISO TR 11405)

Before the microshear test was conducted, all samples were checked under an optical microscope (40 x magnification) for defects (Olympus SZ-PT, Tokyo, Japan). Samples that showed interfacial gap formation or bubble inclusion were excluded from the study and replaced by another sample.

Specimens were mounted in a jig so as to place the bonded resin-tooth interface parallel and as close as possible to a wire (diameter 0.20 mm) that was looped around the resin cylinder, in contact with half of the cylinder base for the microshear test, at a cross-head speed of 1mm/ min, using an universal testing machine with 500 N load cell (Mini Instron 4442, Instron Co., Norwood, MA, USA). The microshear bond strength was calculated by dividing the maximum load at failure by the cross-sectional surface area of the bonded surface. If a spontaneous interfacial debonding occurred while the specimens were being mounted or sectioned, the bond strength was recorded as 0 MPa[9, 11].

Fracture analysis

All tested samples were examined under an optical microscope at 40x magnification to identify failure mode. The fractures were categorized as follows: Type 1-adhesive failure between tooth substrate and adhesive resin; Type 2-mixed failure with adhesive failure (Type 1) and cohesive failure in tooth substrate; and Type 3-cohesive failure in composite resin.

Statistical analysis

All data were analyzed using Mann-Whitney test (Minitab 14 Software Minitab Inc., State College, PA, USA) to perform group comparisons ($p < 0.05$).

RESULTS

The application method of the cleansing agents had no influence on the microshear bond strength results of the adhesive system used in enamel (p = 1.0-hydrogen peroxide, p = 0.6625 - anionic detergent, p = 0.6625-antiseptic) or dentin (p = 0.5403-hydrogen peroxide, p= 0.9362-anionic detergent, p = 0.8345-antiseptic) (Table 1). Adhesive failures were observed in enamel groups. Adhesive and mixed failures were observed in dentin groups (Table 1).

Table 1 - Mean microshear bond strengths and standard deviations of enamel and dentin cleansed with hydrogen peroxide, anionic detergent solution, and antiseptic solution (with or without friction). Failure analysis of the specimens.

			MPa	Type 1*	Type 2**	Type 3***
Enamel	Hydrogen peroxide	With friction	18.54 ±4.83a	100%		
		Without friction	17.38 ± 3.34a	100%		
	Anionic detergent solution	With friction	19.73 ± 8.85a	100%		
		Without friction	24.15 ± 1.00a	100%		
	Antiseptic solution	With friction	24.11 ± 1.31b	100%		
		Without friction	23.65 ± 3.36b	100%		
Dentin	Hydrogen peroxide	With friction	20.05 ± 3.64a	92%	8%	
		Without friction	21.51 ± 2.08a	92%	8%	
	Anionic detergent solution	With friction	19.32 ± 8.51a	100%		
		Without friction	19.33 ± 5.30a	93%	7%	
	Antiseptic solution	With friction	20.28 ± 6.87a	92%	8%	
		Without friction	19.70 ± 5.69a	93%	7%	

*Type 1: Adhesive failure between tooth substrate or hybrid-like layer and adhesive resin;
**Type 2: mixed failure with adhesive failure (Type 1) and cohesive failure in tooth substrate;
***Type 3: cohesive failure in resin composite.
Same letters indicate no statistical difference.

When the effect of cleansing agents on the bond strength of enamel was analyzed, it was observed that the antiseptic solution resulted in bond strength significantly higher than the others groups ($p = 0.014$). For dentin, no differences were observed between the groups cleansed with the agents proposed ($p = 0.391$).

DISCUSSION

In this experiment, some cleansing agents were tested on dental surfaces in two different application modes (with / without friction or active / passive application). It plays an important role in the action of some substances[1,12] and friction of the cleansing agent with a cotton pellet to improve cleansing ability can be recommended. [7]

The second null hypothesis tested was accepted in our experiment, since bond strength of a one-step self-etching adhesive system was not influenced by the application mode of the cleansing agents tested.

Additionally, pH of self- etch adhesive systems is directly related to their ability to interact with the underlying tissue. The lower pH value of the adhesive system, the more aggressive it is, promoting complete solubilization of the smear layer[13]. The adhesive system used in this study has a mild aggressiveness potential ($pH > 2.0$) [14-16], which makes adhesive performance more sensitive to the presence of any substance on dental surface [17].

Although some studies have shown a decrease in adhesion values due the presence of residual hydrogen peroxide on enamel even after the application of phosphoric acid [12], this behavior was not verified in our experiment probably due the lower concentration and application time of hydrogen peroxide, in accordance with the first hypothesis.

Rejecting the first hypothesis, the results of this experiment showed that bonding ability to enamel was statistically different according the cleansing agents proposed and the antiseptic solution resulted in higher bond strength values. This behavior express the importance of a cleaned enamel surface to allow the action of one-step system, once literature shows that antiseptic solution used in this study has an action on organic matrix, leaving a mineralized cleaned surface[18]. Dentin is a permeable tissue and cleansing agents might also change other surface properties of dentin such as wet ability [13], which might influence the interaction between the dentin and restorative materials [14]. Sodium hypochlorite present in antiseptic solution increased wet ability[19] and this rough surface could be a clinical benefit as in the case of micromechanical bonding of the adhesive materials that need irregularities on the surface [13]. It is shown that when it is applied prior to the application of self-etch adhesives on dentin, it positively influence the tensile bond strength [15].

On the other hand, other studies show that antiseptic solution produces an oxidizing action that leads to oxidation of some components in the dentin matrix that is critical for the interfacial initiation of polymerization in some adhesive systems, leading to lower bond strengths [20], even after water application[18]. The present study showed that this agent did not affect the bond strength in dentin, possibly due to the lower concentration present in the antiseptic solution applied on the dental substrate.

Application of hydrogen peroxide in the same concentration but for a larger period of time increased wet ability of dentin, which could change the behavior of the adhesive restoration [19]. Another study shows that during bonding procedures, hydrogen peroxide might break down to oxygen and water, generating bubbles or voids that interfere with resin infiltration into etched dentin[20]. This oxygen inhibits the interfacial polymerization of resin bonding materials. Although reduction in bond strength of some adhesive systems applied to dentin may have been caused by the presence of hydrogen peroxide [18,20], this product did not alter the adhesion when applied for 10 s, at 3% of concentration.

Although a study have shown higher bond strength values when anionic detergent solution was applied due its cleansing capacity [7], this behavior was not observed in this study. The results showed that bonding ability was not statistically different when dentin was cleansed with the agents proposed.

There was a predominance of adhesive failures. This behavior was also observed in other studies in which the same adhesive system was used [8,9,21]. This observation must be considered when an adhesive system is chosen because once there is a failure, tooth structure will not be damaged [22]. Additionally, predominance of adhesive failures could be related to the care in the preparation and the rigorous selection of samples tested.

However, this is an *in vitro* study and clinical researches would provide relevant knowledge to professionals. Once some substances can affect the adhesion performance of a total etch adhesive system over time [10], new researches with the agents applied in this study in a long term would be valid.

Many substances are frequently used for the most different purposes in dentistry and they can change physicochemical properties of dental tissue. It is important to know if these substances also change the behavior of adhesive restorations.

CONCLUSION

Regarding bond strength to enamel and dentin using one-step self-etching adhesive system, application mode of the cleansing agents used in this study presented the same behavior. Antiseptic solution applied on enamel before the application of a one-step adhesive system increased bond strength performance.

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