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Influence of Prior Phosphoric-Acid Etching On Enamel Bond Durability of Multimode Adhesives Systems Apllied in Etch-And-Rinse Mode

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Abstract **Original Research Article**

Objective: Verify if the use of MAS in ER mode could compromise bonding effectiveness compared to its previous version, the etch-and-rinse adhesive system (EAS). ***Materials and Methods:*** Twenty human molars were divided into 2 groups (N=10) to the adhesive system used, the MAS - Scotchbond Universal Adhesive (3M ESPE) and the 2-step EAS - Adper Single Bond 2 (3M ESPE). The crown of each tooth was sectioned into halvesand the mesial/distal surfaces were used. The investigated adhesive agents were applied according to manufacturer's instructions. Resin composite cylinders were built after adhesive application. After stored for 24 hours and six months in 37°C distilled water, the specimens were subjected to microshear test in universal testing a machine at a crosshead speed of 0.5 mm/minute. The results were analyzed with one-way ANOVA and the Tukey test. ***Results:*** The two adhesive systems investigatedproduced produced similar values of bond strength to enamel regardless of the storage conditions. After 6 months of storage, both adhesive systems did not show significant reduction of bond strength when compared to the initial bond strength ($p < 0.05$). ***Conclusions:*** The application of the multimode adhesive system in the etch-and-rinse technique, on acid-demineralized enamel surface did not influence the behavior of the adhesive. ***Clinical Significance:*** *This study shows the importance to present another viable option adhesive system, with fewer steps, optimizing the dentist's work.*

Keywords: *adhesives systems; etch-and-rinse mode; etching-and-enxaguar; clinical study; laboratory.*

INTRODUCTION

Since the introduction of the acid etching technique in 1955, the creation of irregularities and microporosities in enamel was considered a key role in adhesive restorations. Enamel is a uniform substrate, composed essentially of inorganic crystals, well organized in prisms [1]. Adhesive protocols can be achieved through acid etching of enamel which creates an irregular surface, increasing its surface energy. When the adhesive is applied to the surface of the previously conditioned enamel, the monomers infiltrate the irregularities by capillary attraction and copolymerize, resulting in improved enamel retention [2].

A wide range of adhesive systems have been developed for adhesive restorations purposes and there is a clear trend to simplify the clinical use of these systems in dentin and enamel substrates.

With adhesive systems evolution, systems were developed dismissing previous acid conditioning of dental structures, instead, dissolution through a self-etching primer is generally used. Thus, the washing phase is eliminated in the adhesion process [3]. The self-etching adhesives consist of polymerizable acid monomers generally phosphoric acid esters, with relatively higher pH than the conditioning acid [4]. These monomers are able to demineralize and infiltrate dental tissues simultaneously [5]. The advantages of self-etching adhesive systems are the possibility to achieve adhesion with a simple solution that performs as conditioner and primer[3] and also easy to use, reducing time and number of clinical procedures [6]. It is reported as a less sensitive technique [7] and by dismissing washing procedure; a decrease of clinical time is expected as decrease of manipulation bias [7, 8].

The simplification of dhesive procedures with decrease of operative steps were quickly accepted by clinicians, however such simplification does not necessarily reduce the technique sensitivity and could not implicate in a better bond with dental tissues. Bond effectiveness is a persist clinical interest issue in a short or long term [9]. Thus, current adhesives are often labeled as technically sensitive application materials and a simple failure in the clinical procedure is penalized as a rapid degradation of the adhesive interface with early arising of marginal infiltrations in adhesive restorations. Hence, there is a high demand for simple and technically less sensitive adhesive systems, prompting manufacturers to develop new products in a short time span [10].

Therefore, an adhesive system that able the professional to use it with different protocols without presenting great variations in the final result would be interesting. The possibility of the clinician to decide which specific adhesion protocol is most appropriate for the prepared cavity using a versatile and less sensitive to technical variations material would be highly desirable[9]. Universal adhesives offer clinicians the choice of using the conventional technique (etch-and-rinse-ER) or the self-etch (SE) technique to bond to dental substrates [11]. They are called multimodal (SAMM) or universal adhesives (SAU), due to their versatile instructions for use [12].

Single-bottle universal adhesive systems can be applied in both modes, as they include the conventional two-step (ER) mode and self-etching (SE) options applied in one or two steps. Some of these adhesives are an evolution of the prior conventional category with the same composition basis, however, through the inclusion of hydrophilic acid monomers in their formulations, this adhesive now has a self-etching feature. Considering this characteristic of the universal adhesive systems provided by the acidic components incorporated in its formulation and also its versatility of being able to be used in the conventional or self-etching technique, it must be considered that the use of this adhesive in conventional mode (ER) can be unsuitable for use on the enamel substrate. This is due to fact that an overconditioning of enamel could be occurring during the application process of the acidic adhesive on enamel previously demineralized by the phosphoric acid.

Thus, this study aimed to verify if the use of a new multi-modal adhesive (Single Bond Universal Adhesive, SU, 3M ESPE, St Paul, MN, USA) in conventional mode could compromise adhesion strength compared to its previous version, the traditional two-step Etch-and-rinse, (Adper Single Bond 2, SB, 3M ESPE, St. Paul, MN, USA). The null hypotheses tested were that (1) the bond strength of the multimodal adhesive to the enamel was not different from the traditional adhesive, and (2) the preconditioning with phosphoric acid did not influence the initial bond strength and after 6 months storage in water 37°C, of the universal adhesive system.

METHODOLOGY

Human third molars extracted less than 6 months ago and stored under refrigeration after being extracted were used to perform this study. Previously to the extraction, an informed consent of the patients was obtained. This study protocol was approved by the Federal University of the Jequitinhonha and Mucuri Valleys ethical review board (CAAE: 19229513.5.00005108).

Tested Materials

Two adhesive systems were used for bonding composite resin to human dental enamel: the conventional Single Bond 2 adhesive system and the Single Bond/3M-ESPE universal adhesive system. All materials were used according to manufacturer's guidelines and polymerized with an Optilight LD MAX (Gnatus) LED light set with 600mW/cm^2 power, at the time recommended by the manufacturer.

Sample preparation

Teeth root portion were removed at the cement-enamel junction with double-sided diamond disk (KG Sorensen). To obtain an enamel surface, dental crowns were sectioned buccolingually, producing 8x2 mm enamel fragments from the mesial and distal sections of each dental crown (Fig. 1a). The mesial and distal crown fragments were then incorporated into the self-curing polystyrene resin, and after resin polymerization, the tooth-resin set were slightly polished with 1200 silicon carbide abrasives (Carburundum, Saint-Gobain Abrasives LTD, Guarulhos, SP, Brazil) in a metallographic precision saw (PLFDV, Fortel) under constant cooling to expose a smooth and flat area of enamel. (Fig. 1b). Twenty teeth were randomly distributed in 2 experimental groups (n = 10) according to the adhesive system used, Adper Single Bond 2 (3M ESPE) Etch-and-rinse

adhesive system and universal adhesive system, Scotchbond Universal Adhesive (3M ESPE). Previously to the bond strength test, specimens from each group were stored in distilled water for 24 hours and for 6 months of aging period. The distilled water was changed weekly.

Restorative procedures

Prior to the restorative procedures, a standard adhesive tape with a circular perforation in the center of 1 mm diameter was placed on the flat enamel surface in order to delimit adhesion areas. Then, the investigated adhesive agents were applied according to manufacturer's instructions and photoactivated for 10 seconds with Optilight LD MAX (Gnatus) photopolymerizer with 600mW/cm^2 power (Fig. 1c). After application and photoactivation of the adhesives, Tygon matrices (TYG -030, Sainto-Gobain Performance Plastic Maime Lakes, FL, USA) of approximately 1 mm high and 0.75 mm diameter, were positioned on the surface of enamel and filled with Filtek Z250 microhybrid composite resin (3M ESPE). Next, the set was photoactivated for 40 seconds.

Microshear test

The restored enamel fragments were stored for 24 hours in 37°C distilled water. Subsequently, the Tygon matrices were removed with a scalpel blade, and samples for the 24 hours group were tested. For this purpose, samples were individually coupled in a microshear device. Each composite resin cylinder was wrapped by a steel wire (0.2 mm diameter) which was attached to the top of the universal test machine EZ Test-L (Shimadzu Corporation, Tokyo, Japan). Areas closer to the base of the restoration were involved and restauration was pulled until rupture (Fig. 1d). Samples were loaded at 0.5 mm/min rate until fracture and esults were expressed in Mega Pascal (MPa). Same procedures were performed for experimental groups that were tested after the 6-month storage period.

Statistical analysis was used to determine which of the adhesives were most effective. The data were analyzed using the SAS statistical program (SAS Institute Inc., Cary, NC - v.9.1.3). The ANOVA test and the Tukey post hoc test at 0,5 confidence level were used.

RESULTS

Mean values of microshear bond strength, including the standard deviation of the two bonding systems investigated and the two storage conditions (24 hours (I) and after 6 months of storage in water - (F) are shown in Table 1.

The type of adhesive did not influence the results, statistical similarities were verified with the application of the Tukey test at 5% significance level in both storage conditions. After 6 months of storage, the two adhesive systems did not show significant reduction of bond strength when compared to the initial bond strength ($p < 0.05$).

Table 1 shows the mean values of enamel bond strength (± standard deviation) of the adhesive systems after 24 h and 6 months of storage in water.

Table-1: Results of microshear bond strength (Mpa) (standard deviation), one way ANOVA, Tukey *post hoc* (α<0.05)

Adhesive system	Bond strength (Mpa)
SBUI	48.56(28.6)[a]
SBUF	46.08(23.5)[a]
SB2I	36.97(20.1)[a]
SB2F	34.31(14.2)[a]

* SBU - Scotchbond Universal, I –Initial; F- Final; SB2 –Single Bond 2

** No statistical differences were found between groups.

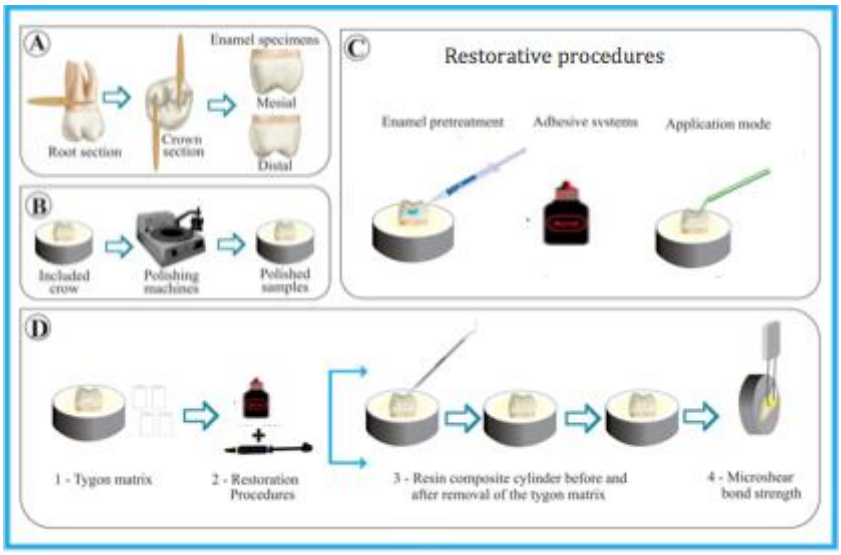

Fig-1

DISCUSSION

The first null hypothesis tested, which it stated that the bond strength of the multimodal adhesive would not be different from the traditional adhesive, was accepted. The two adhesives investigated Scotchbond Universal and the traditional Single Bond 2 adhesive systems present in their formulations the polyalkanoic acid copolymer known as the Vitrebond or VCP copolymer. This copolymer chemically binds to calcium in the hydroxyapatite through the carboxylic groups that replace the phosphate ions in the substrate and perform ionic bonds with calcium. Previous studies demonstrated favorable results for dental tissues bonding and attributed this good performance partially to the chemical union promoted by the VCP copolymer present in the formulations of the investigated adhesives [13]. Therefore, this could explain the similar result for bond strength values of both adhesives, probably provided by the chemical bonding with the hydroxyapatite of the dental substrate.

However, the universal adhesive differs from the Adper Single Bond 2 adhesive in their formulation mainly in the partial substitution of dimethacrylate monomers by the 10 MDP dimethacrylate (10-Methacryloiloxydecyl dihydrogenphosphate) monomer[14] 10MDP is a functional monomer with potential for chemical interaction with hydroxyapatite. The interaction occurs between calcium phosphate group and hydroxyapatite, forming a stable salt. The stability of this calcium salt was correlated with a high binding force of 10-MDP with enamel and dentin immediately and also after storage in water. Therefore, the universal adhesive system could be benefited to the additional chemical bond with enamel, although it is more acidic, due to its self-etching feature. Several other studies related a good performance of adhesive systems with the presence of acid functional monomer – MDP [15].

Although MDP monomer is able to perform strong ionic bonds with calcium of the tooth structure, these bonding results were not significantly superior to the universal adhesive system when compared to its predecessor Single Bond 2. This outcome could be related to the presence of VCP in the universal adhesive system, which may have competed with 10-MDP by hydroxyapatite, neutralizing the union of this monomer, which may have compromised the superior performance of the universal adhesive system [9, 16].

The second null hypothesis stating that the preconditioning with phosphoric acid will not influence the initial bond strength and after 6 months of storage in water, of the universal adhesive system can be confirmed. The application of the universal adhesive system in the etch-and-rinse technique, on an already demineralized enamel surface, by the preconditioning with phosphoric acid could promote over-conditioning and compromise the bond strength, since this adhesive contains acid monomer in its composition. However, this aspect did not influence the behavior of the adhesive, corroborating the authors' findings that the previous acid etching in the application of self-etching adhesive systems is a potential technique, presenting favorable enamel bonding results [17-21].

Despite the concern with over-conditioning using an acid primer on already conditioned enamel, the results of this study showed no differences between the two adhesives. The preconditioning with acid removes the entire smear layer that could compromise adhesion of the adhesive and also enables a more intimate contact of the adhesive components (MDP and VCP) with the dental structure, that is, it increased the reactivity of these components with the the dental structure[22].

The storage in distilled water for 6 months (F) did not present significant decrease of bond strength values when compared to the results of 6 months ($p > 0.05$), regardless of the adhesive system used. Some authors reported that the additional chemical bond promoted by the components present in the formulations of the current adhesive systems can contribute to greater stability and longevity of the bond [10,13].

Although literature indicates several *in vitro* studies on adhesives, especially one step adhesive systems, most of these studies used only short-term results [23-25]. The 6-month storage time span could be a short period to detect any significant differences and to predict the long-term clinical behavior of investigated dental adhesives that may be highlighted as a limitation of this study. However, the fact that the universal adhesive is considered part of a new category of simplified multimodal dental adhesives that lack clinical data, this short-term evaluation is justified. Faster analysis that estimate the clinical performance of new materials and restorative techniques are essencial for the investigation of adhesive restorative techniques, providing in the literature, in a short time span, data on the quality of adhesive systems while the materials are still present in the dental market.

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