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ORIGINAL ARTICLE

Influence of dentin pretreatment on bond strength of universal adhesives

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ABSTRACT

Objective: The purpose of the present study was to compare bond strength of different universal adhesives under three different testing conditions: when no pretreatment was applied, after 37% phosphoric acid etching and after glycine application.

Materials and methods: One hundred and fifty bovine permanent mandibular incisors were used as a substitute for human teeth. Five different universal adhesives were tested: Futurabond M+, Scotchbond Universal, Clearfil Universal Bond, G-Premio BOND, Peak Universal Bond. The adhesive systems were applied following each manufacturer's instructions. The teeth were randomly assigned to three different dentin surface pretreatments: no pretreatment agent (control), 37% phosphoric acid etching, glycine pretreatment. The specimens were placed in a universal testing machine in order to measure and compare bond strength values.

Results: The Kruskal–Wallis analysis of variance and the Mann–Whitney test were applied to assess significant differences among the groups. Dentin pretreatments provided different bond strength values for the adhesives tested, while similar values were registered in groups without dentin pretreatment.

Conclusions: In the present report, dentin surface pretreatment did not provide significant differences in shear bond strength values of almost all groups. Acid pretreatment lowered bond strength values of Futurabond and Peak Universal Adhesives, whereas glycine pretreatment increased bond strength values of G Praemio Bond adhesive system.

Introduction

The functional-based classification of adhesive systems includes two main groups: total-etch (or etch and rinse) and self-etch systems [1]. This classification is based on the adhesion strategy and provides clinicians information on the adhesion mechanism and on the characteristics of the adhesive systems. Adhesion of resin-based materials to dentin requires three steps: etching (conditioning), priming and bonding [1]. Even if for many years, three-step etch-and-rinse systems have been considered to be the gold standard for bonding, developments in adhesive dentistry have led to introduction in clinical practice of new adhesive systems. In order to minimize working time, one-step self-etching adhesive systems have been developed thus reducing the three stages into a single procedure [2].

Different dentin pretreatments have been introduced in order to maximize bond strength performances of adhesive systems such as acid etching and air abrasion with glycine application [3].

Acid etching of dentin allows removal of the smear layer and demineralizes the subsurface. This is a predictable clinical procedure, but some factors inherent to conditioning of dentinal tissue can influence the bonding characteristics of adhesives [4,5]. Dentinal collagen exposed by an etch-and-rinse adhesive has been demonstrated to be highly vulnerable to hydrolytic and enzymatic degradation processes [6,7].

Air-power polishers might be used as pretreatment in order to avoid dentinal acid etching. Air abrasion is a technique for cavity treatment, which involves the use of glycine powder, in a fine stream of compressed air. As the particles collide with dentin, the kinetic energy of the particles is released, resulting in fracture of microscopic fragments. It has been suggested that air abrasion at high pressure can roughen the tooth surface, making it receptive to composite resin without the need for acid-etching, even if this effect is still controversial [8,9].

Enamel and dentin bonding has progressed from multi-step systems to simplification of the application

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procedure in order to eliminate technique-sensitivity and reduce working time [10]. The simplified options include three different alternatives: the two-step etch-and-rinse systems (combining priming and bonding functions in a single solution), the two-step self-etch (that combine the conditioning and priming), and the all-in-one adhesives (that combine the three essential components in a single solution) [11,12]. When using self-etch adhesives, a hybrid layer is formed with the smear layer incorporated [4].

Recently, some manufacturers introduced all-in-one self-etch universal adhesives that can be used as self-etch or as etch-and-rinse adhesives [13,14]. These adhesives thereby offer to the clinicians versatility during restoration placement by the inclusion of monomeric constituents capable of producing chemical adhesion to natural dentition. Concerning these materials, immediate ultramorphological and bond strength studies have been published [13,14]. Moreover, also aging influence on bond strength has been tested showing a decrease in bonding effectiveness with nanoleakage at the adhesive/dentin interface [14]. Thus, the effects of dentin pretreatment, both with acid etching and with air-power polishers, are still unknown.

Therefore, the purpose of the present study was to compare bond strength of different universal adhesives under three different pretreatment conditions: 1 – no pretreatment applied, 2–37% phosphoric acid etching and 3 – glycine application. The null hypothesis of the study was that dentin pretreatment had no significant effect on the shear bond strength of the various universal adhesives tested.

Materials and methods

Preparation of specimens

One hundred and fifty bovine permanent mandibular incisors have been extracted and collected as a substitute for human teeth [15]. The criteria for tooth selection were: freshly extracted teeth, intact buccal enamel with no cracks caused by extraction. The teeth were stored in a solution of 0.1% (wt/vol) thymol, cleansed of soft tissue and subsequently embedded in self-curing acrylic resin (Rapid Repair, DeguDent GmbH, Hanau, Germany). Cuboidal Teflon mold was filled with the acrylic resin encasing each specimen and allowing the buccal surface of enamel to be exposed. Each tooth was oriented so that its labial surface was parallel to the shearing force. The teeth were sectioned parallel to the occlusal surface to expose mid-coronal dentin that was wet abraded with a polishing

machine (APL-4; Arotec S.A. Ind Com, Cotia, Brazil) with a 600-grit silicon carbide abrasive paper (SiC) disks for five seconds.

Before application of the adhesive systems, the labial surface of each incisor was cleaned for 10 seconds with a mixture of water and fluoride-free pumice using a rubber-polishing cup and a low-speed hand-piece. The dentin surface of each specimen was rinsed with water to remove pumice or debris and then dried with an oil-free air stream.

The materials tested in this study included five universal adhesives: Futurabond M+, Scotchbond Universal, Clearfil Universal Bond, G-Premio BOND, Peak Universal Bond. The specifications of adhesive systems tested are listed in Table 1.

The universal adhesives were applied to teeth surfaces following each manufacturer's instructions. The bonding interface was limited to $2\,mm^2$ in order to have a better stress distribution during loading [16]. The teeth were randomly assigned into three groups (each made of 50 specimens) according to different dentin surface pretreatments. In each group, five subgroups were created with 10 specimens for each adhesive studied.

In the first group ($n = 50$), no pretreatment agent was applied. In the second group ($n = 50$), etching was performed using 37% phosphoric acid for 15 s (total etch; Ivoclar Vivadent AG, Schaan, Liechtenstein) while in the third group ($n = 50$), glycine pretreatment was applied. Sub-groups were created as follow: sub-group 1 – Futurabond M+, sub-group 2 – Scotchbond Universal, sub-group 3 – Clearfil Universal Bond, sub-group 4 – G-Premio BOND, sub-group 5 – Peak Universal Bond.

All universal adhesives were cured using a LED curing light (Celalux 2 High-Power LED curing light, Voco GmbH, Cuxhaven, Germany) for 10 seconds at a light intensity of $1000\,mW/cm^2$. After adhesive application, a nanohybrid composite resin (Grandio-Voco GmbH, Cuxhaven, Germany) was inserted into the dentinal surface by packing the material into cylindrical-shaped plastic matrices with an internal diameter of 2 mm and a height of 2 mm. Excess composite was carefully removed from the matrix with an explorer. The composite was cured with a LED curing light for 20 s at a light intensity of $1000\,mW/cm^2$ according to manufacturer instructions. All samples were stored in distilled water at 37 °C for 24 h.

Shear bond strength testing

After storage, the specimens were secured in the lower jaw of an Instron universal testing machine

Table 1. Adhesive systems used in the study.

Adhesive	Manufacturer	Batch number	Composition	pH
Futurabond M+	VOCO, Cuxhaven, Germany	1442146	2-hydroxyethyl methacrylate (10–25%), BIS-GMA (10–25%), ethanol (10–25%), acidic adhesive monomer (2.5–5%), Urethanedimethacrylate (2.5–5%), catalyst, pyrogenic silicic acids, catalyst.	2
Scotchbond Universal	3M ESPE, St Paul, MN, USA	547836	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond™ Copolymer, filler, ethanol, water, initiators, silane.	2.7
Clearfil Universal Bond	Kuraray, Medical, Sakazu, Okayama, Japan	3.E0007	Bisphenol A diglycidylmethacrylate (15–35%), 2-hydroxyethyl methacrylate (10–35%), ethanol ($<$20%), methacryloyloxydecyl dihydrogen phosphate, hydrophilic aliphatic dimethacrylate, colloidal silica, camphorquinone, zirconium oxide, accelerators, initiators, water.	2.3
G-Premio BOND	GC Corporation, Tokyo, Japan	1409041G	Acetone (25–50%), 2-hydroxy-1,3-dimethacrylaxypropane (10–20%), methacryloyloxydecyl dihydrogen phosphate (5–10%), 2,2-ethylenedioxydiethyl dimethacrylate (1–5%), diphenyl(2,4,6-trimethylbenzoyl)-phosphine oxide (1–5%), 2,6-di-tert-butyl-p-cresol ($<$0.5%).	1.5
Peak Universal Bond	Ultradent Products Inc., South Jordan, Utah, USA	B9528	Ethyl alcohol ($<$20%), 2-hydroxyethyl methacrylate ($<$16%), methacrylic acid ($<$6%), chlorhexidine di(acetate)($<$0.3%).	1.9

Table 2. Descriptive statistics (MPa) of the different groups.

Adhesive	Code	Pretreatment	Mean	SD	Min	Mdn	Max	Tukey[a]
Futurabond M+	FB	NO	10.71	1.937	7.66	10.87	12.96	A,C
		AC	7.202	0.537	6.673	7.041	8.057	B
		GLY	8.586	1.825	5.699	8.724	10.63	A,B
Scotchbond Universal	SB	NO	8.841	2.716	6.636	8.673	13.28	A,B
		AC	7.743	0.822	6.466	7.908	8.713	A,B
		GLY	6.695	0.492	5.948	6.837	7.264	A,B
Clearfil Universal Bond	CUB	NO	6.223	1.471	4.482	6.071	8.517	A,B
		AC	6.944	3.678	2.498	6.786	12.66	A,B
		GLY	7.139	0.837	5.85	7.296	8.149	A,B
G-Premio BOND	GBU	NO	7.705	1.588	5.431	7.775	9.911	A,B
		AC	8.717	0.881	7.352	8.878	9.767	A,B
		GLY	12.95	2.05	10.28	12.81	16.03	C
Peak Universal Bond	PEAK	NO	6.67	3.342	0.906	8.159	9.223	A,B
		AC	2.508	1.26	0.317	3.06	3.368	D
		GLY	7.39	0.314	7.135	7.245	7.866	A,B

[a]Post hoc significance.

(Model 3343, Instron Corp., Canton, MA). Specimens were placed so that the bonded cylinder base was parallel to the shear force direction. Specimens were loaded at a crosshead speed of 1 mm/min [1,17,18] in an occluso-gingival direction. The maximum load necessary to debond the specimens was recorded in Newton (N) and then converted into MPa as a ratio of Newton to surface area of the cylinder.

Statistical analysis

Statistical analysis was performed with R 3.2.0 software (The R Foundation for Statistical Computing, Wien, Austria). Descriptive statistics, including the mean, standard deviation, median and minimum and maximum values, were calculated for all groups.

The normality of the data was calculated using the Shapiro–Wilk test. The Kruskal–Wallis test was applied to determine whether significant differences in debond strength values existed among the various groups. The Mann–Whitney test was used as post hoc. Significance for all statistical tests was set at $p < .05$.

Results

Descriptive statistics are presented in Table 2. The Kruskal–Wallis test showed the presence of significant differences among the various groups ($p < .0001$). As shown in Figure 1, the post hoc Mann–Whitney testing showed no significant differences in shear bond strength values among the five different adhesives tested when no dentin pretreatment was performed ($p > .05$). Acid etching pretreatment significantly
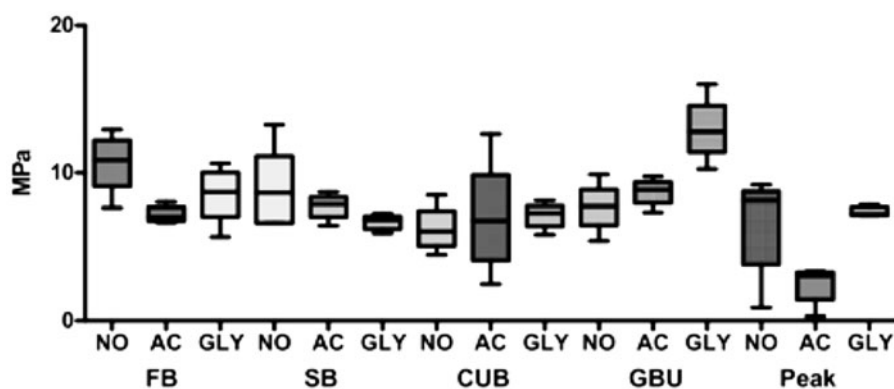
Figure 1. Shear bond strength box plot (MPa) of the different groups.

reduced the shear bond strength for Futurabond M + and Peak Universal Bond ($p < .05$) when compared with no pretreatment group. When orthophosphoric acid was applied, the lowest shear bond strength values ($p < .01$) were reported when using peak adhesive system, whereas no significant differences were reported among the other adhesives ($p > .05$).

When glycine was applied, the highest strength values ($p < .05$) were reported with G-Premio BOND adhesive system, and no significant differences were reported among the other adhesives ($p > .05$). For each adhesive tested, glycine pretreatment did not influence bond strength values except for Scotchbond Universal and G-Premio BOND ($p < .05$). Scotchbond Universal showed significantly lower bond strengths when glycine was applied while G-Premio BOND showed inverse tendency.

Discussion

Based on our results, the null hypothesis was partially rejected.

No significant differences were reported among the five adhesives tested at baseline, with no pretreatment ($p > .05$). Therefore, in this case, the bond behaviour is the same for all adhesives tested.

When acid pretreatment was applied no significant differences were reported in shear bond strength values of the various adhesives, except for Futurabond and Peak systems, that significantly decreased their bond strength values. These results are important for daily practice, when the clinician approaches a cavity with enamel and dentin exposed. In this case, these Universal adhesives require different modes of application and acid pretreatment should be localized only to enamel. This is in agreement with a study on bond strength on enamel after acid pretreatment of universal adhesives [19].

On the other hand, when glycine pretreatment was performed no significant differences were reported in shear bond strength values of the various adhesives, except for G Premio Bond system, that significantly increased its bond strength values. This adhesive is the adhesive with lower pH value (Table 1) but the role of pH on glycine pretreatment effects on shear bond strength values is still unknown.

A limitation of this study is the *in vitro* procedure that, even if essential to quickly evaluate the bonding effectiveness to dentin of newly developed adhesives, is not completely reliable and comparable with clinical trials even for the choice of bovine teeth. As long-term clinical follow-up is time-consuming and difficult to perform with standard methods, *in vitro* bond strength test are frequently used to assess dental material and techniques. For this purpose, shear, micro-shear, tensile and micro-tensile tests have been developed [1].

In the present investigation, bovine teeth were used as a substitute of human enamel. Bovine lower incisors are used for many reasons. In fact, it is easier to obtain a sufficient number of bovine teeth than human teeth. Moreover, the bigger surface area of bovine lower incisors allows correct preparation of a standardized bonding area. Finally bovine teeth, derived from animals of similar genetic lineage and dietary environment, might show higher homogeneity of mineral composition than different human teeth, which are collected from various donators with diverse dietary or fluoride supplementation [13]. On the other hand, results obtained from *in vitro* tests have to be confirmed with *in vivo* studies.

The main concern of the present report is that in literature only few studies have been reported about the effect of dentin pretreatment on the bond strength values of the single-step self-etch adhesive [20,21]. Moreover, in order to improve the reliability of the analyses, the bonded interface was limited to $2\,mm^2$, thus increasing precision of the testing procedures. Using this method, it resulted in higher apparent bond strengths at failure than found when using specimens with higher surface area [22].

In the present investigation, air polishing with glycine has been tested. To our knowledge in literature, dentine glycine pretreatment has been tested only with CAD/CAM nanoceramic material [23], showing a significant increase of shear bond strength values in pretreated groups. No studies tested glycine pretreatment on dentine tissue testing different adhesive systems. Air-power polishers are available with a broad range of powders. When selecting a powder, two aspects have to be weighed: the effectiveness desired from the cleaning, and surface abrasion [24]. A powder characterized by greater abrasiveness removes both undesirable and desirable tissues. Thus powders that are claimed to treat gently the polished surfaces have been introduced. Conversely, the consequence of this atraumatic surface polishing, may be the reduced cleaning effectiveness. So, when selecting a powder, effectiveness must be weighed against abrasiveness. In this study, we selected a glycine-based powder for negligible abrasion. Other aspects to consider when using air-powder polishers can also be influential, such as the ratio of powder to water, the amount of pressure, the fullness level of the air-powder polisher, and type of polisher [25]. In the present study, however, those factors remained constant and their influence thus marginalized.

Conclusions

Although adhesive systems have become simpler, careful management is still required, especially regarding the influence of substrate pretreatment on bonding performance. Acceptable bond strength values could be obtained with no dentin pretreatment regardless the adhesive system used in reduced sensitivity technique conditions. In the present report, acid pretreatment lowers bond strength values of Futurabond and Peak Universal Adhesives, whereas glycine pretreatment increases bond strength values of G Praemio Bond adhesive system.

Disclosure statement

The authors declare that they have no conflict of interest.

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