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Micro Push-Out Bond Strength of Resin Composite to Dentin in Primary Dentition Using Three Universal Adhesives with Different pH: An In Vitro Study

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Abstract: To evaluate the bond strength of different universal adhesives on deciduous tooth dentine and their relationship with the composition and potential of hydrogen (pH). **Methods:** An in vitro micro push-out test on 150 samples ($n = 50$) per group per adhesive, namely, Adhese Universal (ADH; Ivoclar Vivadent), Futurabond U (FUT; Voco GmbH) (Test), and Scotchbond Universal with pre-conditioning (SCO; 3M) (Control), to record bond strength (BS) and type of adhesive failure. **Results:** The results of the different adhesives (megapascals (MPa)) varied, showing no statistical significance. The corresponding averages are in MPa: ADH, 13.66 ± 2.81 ; FUT, 14.48 ± 2.88 ; SCO, 14.98 ± 3.96 . Additionally, the frequency of type of failure was as follows: mixed (60.7%), adhesive (27.3%), and cohesive (12%). **Conclusions:** SCO, with a pH of 2.7, showed greater resistance to fracture, while FUT, with a pH of 2.3 and no pre-conditioning, approached the same values, being a one-step adhesive. No relationship was found between failure and type of adhesive.

Keywords: universal adhesives; micro push-out; deciduous dentition; dentine

1. Introduction

Current adhesive systems allow for the improvement of clinical procedures due to their component chemicals and mechanism of action, reducing the operating time of application, handling of use, and side effects on the patient [1]. Universal or multi-mode adhesives are essentially one-step adhesives, combining an acidic primer and adhesive in a single solution and allowing a self-etching, total, or selective etching technique application. These systems can be applied to different dental surfaces, namely, wet and dry demineralized dentine [2,3], enamel, and even ceramic materials, composites, and metals [4].

Universal adhesives can be considered dependent materials [5] as their components modify their mechanical properties [6,7]. The adhesive composition is mainly based on a mixture of resin monomers HEMA (facilitates resin diffusion within the collagen network) and Bis-GMA (decreases shrinkage by polymerization and is fast curing to improve the mechanical properties of the polymer), inhibitors or stabilizers (antioxidants capable of eliminating free radicals, preventing spontaneous initiation and propagation of polymerization), solvents (which decrease viscosity and promote resin filtration), photoinitiators

(which contribute to the formation of a stable adhesive interface), and filler particles [8,9]. Universal adhesive systems have a composition similar to self-etching adhesives [3]. They contain phosphorus monomers, mainly 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate), that generate ionic bonds with hydroxyapatite by means of nano-layers (nano-layering) [10]. This monomer presents a chemical bond with the hydroxyapatite present in dentine and enamel.

To facilitate the use of universal adhesives in clinical situations, whether etching is required or not, the manufacturers mix resin monomers with lower acidity in appropriate concentrations [11], increasing the potential for hydrogenation. The depth of demineralization in the dentine is linked to this potential of the acidic monomer [12]. Depending on the degree of acidity, we classify them as: strong pH = 1, intermediate pH ~ 1.5, slight pH ~ 2, and ultra-low pH = 2.5. Those with a low pH or pH close to 1.5 contain functional monomers that mainly demineralize hard dental tissues; those with a pH greater than 1.5 partially demineralize the substrate, interacting chemically with the rest of the hydroxyapatite. On the other hand, ultra-low acidic monomers expose dentin collagen in a very superficial way [13–15].

This adhesive action in permanent dentition is not the same as in deciduous dentition for reasons such as dentin and enamel composition and structure, interfering with clinical bonding [16]. Dental substrates in deciduous dentition have a lower mineral content and less thickness than those in permanent dentition, along with a more accentuated prismatic layer in the enamel and a greater tubular density in the dentine, therefore reducing the intratubular area available for adhesion [10,16,17]. There is controversy in the results of different studies as to whether permanent substrates present more or less adhesion strength than deciduous ones. In this line, Pires et al. conducted a systematic review where they concluded that deciduous dentin had different fracture resistance and independent studies were needed [17]. A study published by Ghajari et al. reported significantly greater resistance to fracture in dentin in permanent teeth, confirming the need not to analyze both dentitions in the same group [18].

Based on the hypothesis that the different composition and pH of the universal adhesives used may interfere with the mechanical properties of the different universal adhesive systems, with the rise of adhesive systems in dental medicine, these materials need to be analyzed to identify the best strategy for clinical use in order to choose the most suitable adhesive system in daily clinical practice. Therefore, the aim of the study is to evaluate the bond strength of three universal adhesives on the deciduous tooth structure and their relationship with composition and pH by means of a mechanical micro push-out study and by studying the corresponding fracture type.

2. Materials and Methods

2.1. Study Design

A total of 150 deciduous extracted molars were divided into 3 restorative groups ($n = 50$) to evaluate the bond strength of three universal adhesives. The sample size was determined using Epidat 2.0 software (The EpiData Association, Odense M, Denmark). A minimum of 50 specimens was required in each group for a power of 80%, assuming a common standard deviation of 2.10 and a significance level of 0.05 based on a previous pilot study.

The study was performed in accordance with national regulatory requirements, the ethical principles of the Declaration of Helsinki for medical research involving human subjects. It was evaluated and approved by the Ethics Committee for Research at the Clínico San Carlos Hospital in Madrid, Spain (registration code of the study: CEIC 20/398-E). Written informed consent was obtained from all the participants' parents.

2.2. Study Groups

The specimens were randomly divided into 3 groups ($n = 50$); two adhesives were used as self-etchers (experimental groups)—Adhese[®] Universal (Ivoclar Vivadent AG, Schaan,

Liechtenstein) (ADH) and Futurabond[®] U (Voco GmbH, Cuxhaven, Germany) (FUT)—and the third group (control group) had a total-etch adhesive prior to the application of Scotchbond[™] Universal (3M ESPE, St. Paul., MN, USA) (SCO) (Table 1).

Table 1. Composition and pH of adhesives used.

Adhesive	pH Maker	Commercial Brand	Composition	pH Study
ADH	2.5–3	Adhese [®] Universal (Ivoclar Vivadent AG, Schaan, Liechtenstein)	Methacrylates (10-MDP and MCAP), HEMA, Bis-GMA, D3MA, ethanol, water, highly dispersed silicon dioxide, initiators, and stabilizers	3.7
FUT	2.3	Futurabond [®] U (Voco GmbH, Cuxhaven, Germany)	10-MDP, 2-HEMA, Bis-GMA, acid monomer adhesive, dimethacrylate urethane, catalyst, silica nanoparticles, ethanol	2.4
SCO	2.7	Scotchbond [™] Universal (3M ESPE, St. Paul., MN, USA)	10-MDP, Vitrebond copolymer, HEMA, Bis-GMA, dimethacrylate resin filler, silane, initiators, ethanol, water	3.2

In order to confirm the data provided by the manufacturer on the pH of the adhesives used in the study, we carried out our own measurements using a pH meter in the laboratories of the Department of Inorganic Chemistry, Faculty of Chemistry, Complutense University of Madrid (Crison Instruments, SA, Barcelona, Spain) (Table 1).

2.3. Specimen Preparation

The occlusal dentine surface of each specimen was exposed using a polishing machine (Struers Dap-7 polishing machine) with coarse sandpaper and with the help of a tungsten bur at fast speed and abundant irrigation (Figure 1a).

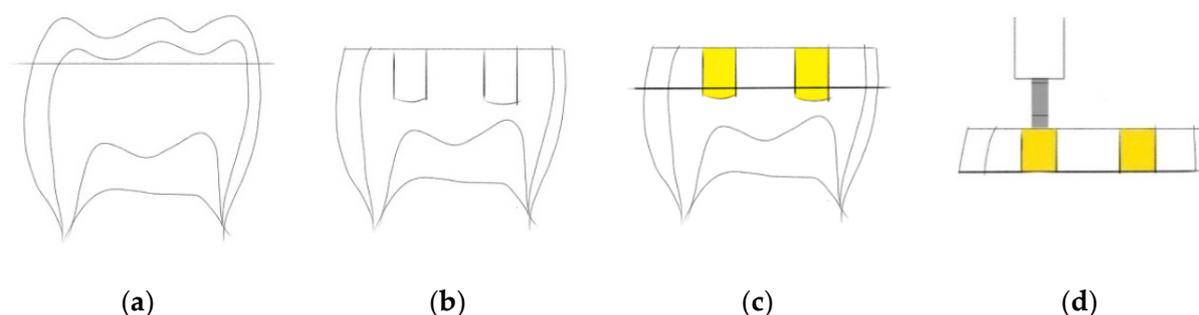


Figure 1. Illustration of specimen preparation and mechanical test of the study. (a) Enamel elimination; (b) cavity preparation; (c) cavity filing and sample cutting; (d) micro push-out test.

A type I black cavity was prepared with a round tungsten turbine bur (H1.314.016) with a 1.6 mm diameter and a 2 mm depth (Figure 1b). The corresponding adhesive system was applied, and the cavity was filled with composite (Voco GmbH A2 Grandioso, Cuxhaven, Germany) according to the experimental group and the instructions of its manufacturer. The teeth were sectioned off transversely to their longitudinal axis using a cutting machine (Exact[®] Cutting Unit 400C, Exact Tools Oy, Helsinki, Finland) and

abundant irrigation (Figure 1c). The samples that could not be fixed due to their small size were placed in an acrylic block (self-curing acrylic material, Dentsply, Germany).

2.4. Micro Push-Out Technique

Each specimen was mechanically tested with Hounsfield Universal Test Equipment® (Croydon, England) (Figure 2a), exerting the load by means of its cylindrical steel punch, centered on the restoration, with no contact with the adjacent dentine until failure was achieved (Figures 1d and 2b). For each specimen, the bond strength was calculated in MPa in the following equation: Failure load (N)/Bonding interface area (mm²). The bonding interface area was calculated using the formula for the side surface of a cylinder [19]: $A = 2\pi rh$. During the mechanical tests, a standard loading speed of 0.5 mm/min was applied. The values obtained were recorded by Metrotest's advanced testing software.

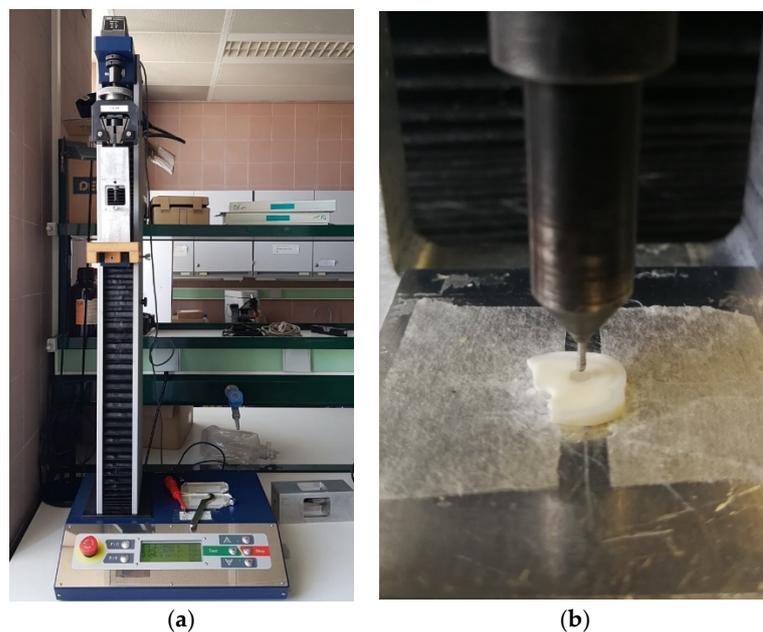


Figure 2. (a) Hounsfield Universal Test Equipment® (Croydon, England); (b) cylindrical steel punch centered on the specimen in the Universal Test Machine.

The failure type was examined using a Leica® MZ 12 stereomicroscope (Leica, Bensheim, Germany) (9.2× magnification) and classified into three categories: adhesive fracture between composite and dentin, cohesive, and mixed.

2.5. Statistical Analysis

A statistical and descriptive analysis of the adhesion strength variable was performed. The exploration was carried out using a box plot, a Q-Q plot of adjustment to normality and the histogram, and the Kolmogorov–Smirnov goodness test.

Descriptive analysis was performed with the usual tools: the range of observed values, mean, and standard deviation. For a significance contrast of the means, the 1-factor ANOVA test was chosen. Frequencies and percentages were calculated for the analysis of the type of fracture. The chi-squared test was used to examine the relationship between the variable type of adhesive and the form of failure. A 1-factor ANOVA test was used to verify the relationship between bond strength values and the type of fracture (IBM Corp, released 2017, IBM SPSS Statistics v 25.0 for Windows; Armonk, NY, USA).

3. Results

The samples were evaluated per adhesive group ($n = 50$) and in total ($n = 150$) according to their fracture resistance variable (MPa). The average value of the ADH group was 13.66 ± 2.81 ; for FUT, it was 14.48 ± 2.88 ; for SCO, it was 14.98 ± 3.96 (Table 2).

Table 2. Bond strength descriptive analysis. Variable adhesion strength depending on the type of adhesive ($n = 50$) and in the total sample ($n = 150$).

Bond Strength Subgroup	Exploration Form			Centrality		Variability		
	Asymmetry	Kurtosis	KS Test: p -value	Mean	Median	Range (Min/Max)	SD	Interquartile range
ADH	0.76	0.03	0.209 ^{NS}	13.66	13.07	9.84/21.3	2.81	4.07
FUT	0.92	0.89	0.114 ^{NS}	14.48	13.57	10.34/23.66	2.88	4.36
SCO	0.12	0.65	0.504 ^{NS}	14.98	14.07	8.74/28.75	3.96	5.31
AF	Asymmetry	Kurtosis	KS Test: p -value	Mean	Median	Range (Min/Max)	SD	Interquartile range
Group Total	1.16	1.88	0.015 [*]	14.37	13.4	8.74/28.75	3.28	4.36

^{NS}: Not significant deviation ($p > 0.05$); the variable is normally distributed; ^{*}: slight significant deviation ($p < 0.05$), the variable tends towards the normal model; AF: adhesion failure.

The normality of the distribution of the variables was accepted, and the one-way ANOVA test showed the absence of statistical significance between the groups, except for the means of ADH and SCO, which presented a tendency towards statistical significance $p = 0.057$ (Table 3). As a result, the ADH group had lower adhesion strength than the SCO group, and the FUT group values were very close to the control SCO with pre-conditioning.

Table 3. Comparative inferential analysis: 1-factor ANOVA. Adhesion strength for resistance to fracture, depending on the type of adhesive ($n = 50$).

Type of Adhesive	Descriptive Statistics		Paired Test Signification		
	Mean (SD)	95% CI	ADH	FUH	SCO
ADH	13.66 (2.81)	12.87–14.46	-	0.418 ^{NS}	0.057 ^{NS}
FUT	14.48 (2.89)	13.67–15.31	-	-	0.728 ^{NS}
SCO	14.98 (3.96)	13.86–16.11	-	-	-

SD: standard deviation; CI: confidence interval; ^{NS}: not significant deviation ($p > 0.05$).

The most frequent type of failure was mixed (60.7%), followed by adhesive (27.3%) and cohesive (12%). Using the chi-square test, the variable type of failure and the type of adhesive used were crossed without finding statistical evidence that relates them. Using the ANOVA test, the relationship between the AF values and the type of fracture was checked; the results (Table 4) showed that the values were higher when it was an adhesive fracture (14.66 MPa) or mixed (14.34 MPa) and lower when it was cohesive (13.92 MPa), without any statistical significance.

Table 4. Comparative inferential analysis: 1-factor ANOVA. Bond strength for resistance to fracture, depending on the type of fracture.

Type of Fracture	Descriptive Analysis			Paired Test Signification		
	N	Mean SD	95% CI	Adhesive	Cohesive	Mixed
Adhesive	41	14.66 (3.63)	13.52–15.81	-	0.46 ^{NS}	0.608 ^{NS}
Cohesive	18	13.92 (3.38)	12.24–15.60	-	-	0.606 ^{NS}
Mixed	91	14.34 (3.13)	13.69–15.00	-	-	-

SD: standard deviation; CI: confidence interval; ^{NS}: not significant deviation ($p > 0.05$).

4. Discussion

Universal adhesive systems have very similar chemical compositions; the difference in the proportion and quantity of the elements influence the penetration of the adhesive into demineralized dentine [20]. The adhesives used in this study share some components: a hydrophilic monomer (HEMA, hydroxyethyl methacrylate) that facilitates the diffusion of resin in wet collagen and increases susceptibility to hydrolysis [21], and a hydrophobic monomer (Bis-GMA, bisphenol A-glycidyl methacrylate). They include solvents such as water and ethanol (which are essential for the composition of adhesives that need to be bonded to dentine), silane, dimethacrylate resins, initiators, and stabilizers. The main compound is the 10-MDP monomer, which interacts with the residual hydroxyapatite surrounding the collagen fibers, thus improving bond strength. Furthermore, the bond of 10-MDP to calcium creates a salt (MDP-Ca) that protects against hydrolysis as it is a hydrolytically stable salt [12]. Regarding the differences in chemical composition, MCAP, urethane dimethacrylate, and the Vitrebond copolymer stand out. Using a pH meter (Crison Instruments, SA, Barcelona, Spain), we measured the pH of the adhesives and found that there were differences with the data provided by the manufacturers (Table 1). Due to the rapid growth of adhesive systems and their diverse characteristics, each of them should be analyzed to identify the best mechanical properties and strategy of use.

The *in vitro* mechanical micro push-out test results showed similar adhesion strength between the self-etching FUT and SCO with prior acid etching and a greater, although not significant, difference between SCO and ADH. ADH, with an ultra-high pH of 2.5–3, would have a lower capacity to dissolve the smear layer and demineralize the underlying dentine surfaces when applied as a self-etch [16,17]. Therefore, this statement could explain the lower fracture resistance compared with the other adhesive systems used in the study. The control SCO has a lower acidic pH, similar to ADH, but its pre-conditioning with orthophosphoric acid would have improved the penetration of the resin into the dentine. Such a pre-etching strategy is more sensitive to the operator, but the presence of MDP, the polyalkenoic acid copolymer (Vitrebond), and silane in its composition allows the hydration of collagen, creating the hybrid layer even in dry demineralized dentine [3]. Vitrebond is the exclusive component in this system, causing a chemical and spontaneous bond to the hydroxyapatite, showing greater adhesion strength than if it were not present [6].

In this study, the two adhesives used as self-etchers present different results: ADH 13.66 < FUT 14.48 MPa, in accordance with their results with the ultra-high pH of the ADH group (2.7–3), and more acidic pH of the FUT group (2.3). The FUT adhesive, with a mild pH degree, obtained similar mechanical results when compared to the control. That result could be due to traces of hydroxyapatite remaining for chemical interaction with a functional monomer [3]. A higher pH does not guarantee a higher bond strength, and the use of different solvents will not influence the results [9]. There could be a lack of knowledge by manufacturers in the application of self-etching agents, which improve properties by increasing the number of coats when applied [22]. The FUT adhesive, in turn, presented in a single dose, would avoid the deterioration of the compound's properties and the evaporation of solvents, besides being a faster and easier application. Regarding the hydrogen ion's potential and bond strength, we conclude that a more basic pH adhesive could mean a lower resistance to fracture. It could improve mechanical properties by acidifying the adhesive by lowering the pH or by applying previous conditioning, taking against the latter option the need for greater dexterity by the clinician and the possible appearance of post-operative tooth sensitivity. If acidifying the adhesive is chosen, the one-step self-etching strategy should be used, which is especially useful for pediatric patients due to its speed.

Regarding failure type, mixed fractures were more frequently reported, in agreement with Cunha and Dos Santos [23,24]. In the present study, it was associated with higher adhesion strength to adhesive and mixed fractures, although without significance, similar to the findings of Egilmez et al. [25]. However, Alonso and Brandt et al. found minimal cohesive failure, in agreement with the present study, yet associated with the group with

higher adhesion strength, perhaps due to the diverse methodology employed, namely, preparation in bovine dentition [26,27].

There is a lack of homogeneity among the used criteria regarding the pre-conditioning of dentin in primary dentition. Some authors have concluded that this is a result of higher bond strength if the substrate is pre-conditioned [2,3,28,29], similar to the results of the SCO group in this study (14.98 ± 3.96 MPa). However, Thanaratikul et al. reported no difference, according to the strategy used on dentin in pediatric patients [30]. On the other hand, Kensche et al. obtained different results depending on the adhesive and its composition, regardless of the strategy used [31]. Overall, many authors have concluded that the differences are due to the different composition of the universal adhesives rather than the methodology used [5]. The lack of significance, especially between the two-step control SCO and the universal adhesive as a self-etching FUT, could indicate an approach to the objectives of finding simpler and faster techniques for the treatment of pediatric patients in the clinic. Further randomized clinical trials are recommended in order to assess the longevity of different universal adhesives on the deciduous tooth structure to demonstrate their effectiveness in a real clinical scenario.

5. Conclusions

Within the limitations of the present study, the following conclusions can be drawn:

- FUT, with a lower pH of 2.3, presents higher penetration capacity at the dentin tubule, providing greater fracture resistance with a self-etching strategy.
- Despite the fact that SCO has an ultra-low pH of 2.7, its adhesion strength is improved when combined with a previous acid etching.
- There was no statistical significance when relating failure to the type of adhesive.

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Informed Consent Statement: Written informed consent was obtained from the parents of all participants.

Data Availability Statement: The data that support the findings of this study are available from the corresponding author, P.M.-M., upon reasonable request.

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