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Does Adhesive Layer Thickness and Tag Length Influence Short/Long-Term Bond Strength of Universal Adhesive Systems? An In-Vitro Study

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Abstract: (1) Background: This study investigated the effect of the adhesive layer thickness and the length of resin tags on dentin bond strength of five universal adhesives applied in self-etch mode. (2) Methods: One hundred and fifty extracted human third molars were used. Five different universal adhesives were applied in self-etch mode on the dentin surface. Half of the specimens were subjected to an aging procedure for six months. A shear bond strength (SBS) test was performed and the results were statistically analyzed with a *t*-test and one-way ANOVA test. Scanning electron microscopy (SEM) was executed to measure the adhesive layer thickness and tag depth. (3) Results: No statistical differences were found between the five adhesive systems after a 24 h storage period, regardless of layer thickness and tag depth ($p < 0.05$). After 6 months of aging in water at 37 °C, Iperbond Max and Scotchbond Universal preserved the bond strength over time ($p < 0.05$), whilst the SBS of Iperbond Ultra, FuturaBond M+, and Ibond Universal decreased significantly after the aging period. No relation was observed between the adhesive thickness or tags' length on SBS. (4) Conclusions: Within the limitation of this study, the stability over time of the bond strength of universal adhesives depends on their compositions regardless of the adhesive layer thickness and/or tags' length.

Keywords: universal adhesives; layer thickness; self-etch; dentin bonding; shear bond strength

1. Introduction

Universal dental adhesives have been developed to minimize the number of bottles and steps of application in order to make them more user-friendly and less time-consuming during their use in dental treatments [1–3]. Such multimode systems have showed a high bonding performance to enamel and dentin surfaces [4,5] both when used in etch-and-rinse, in selective-etch, or self-etch mode [6].

These adhesives could be classified as strong (pH < 1), mild (pH = 2), or ultra-mild (pH > 2.5), based on their acidity [3]. An etching step using orthophosphoric acid was recommended to apply such adhesives onto enamel surfaces [5]. However, bonding on dentinal substrate is more difficult due to its organic composition, dentin permeability, and the tubular structure [7]. Several studies demonstrated that modern universal adhesives applied in self-etch or etch-and-rinse mode can achieve substantial bonding to dentin [8,9].

One of the main advantages of the universal adhesives is that the clinician can select the application mode according to the clinical situation [10]. These adhesives may be used, not only on tooth substrates including resin composite, but also on different substrates, such as silica-based glass ceramics, metal alloys, and zirconia [6,11]. Several modern adhesives contain a functional acid monomer known as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), which can interact and form a chemical bond with calcium ions in hydroxyapatite crystals [4,12]. However, different application procedures are required for those universal systems with different chemical compositions, in particular when containing an acidic functional monomer different from 10-MDP [13]. One of the main requirements of a dental adhesive is to create a resin–dentin bond in the oral cavity with appropriate durability over time due to minimal collagen degradation and monomers hydrolysis [2]. Long-term water storage at 37 °C has been used in several studies to evaluate the durability of bonding performance through shear bond strength (SBS) or micro-tensile bond strength tests [2,4,5]. Some studies believe that the thickness and length of hybrid layer and resin tags, respectively, influence the bonding performance of adhesives at short and long time [14]; however, this aspect has not been yet clarified with modern universal bonding systems, so that it is necessary to study and publish the outcome of such an investigation.

Hence, the aim of the present study was to compare the bond strength of five universal adhesives applied onto the dentin surface at baseline (24 h) and after prolonged water storage (six months in H_2O). Moreover, it was investigated via scanning electron microscopy (SEM) whether the adhesive layer thickness and/or the length of the resin tags could influence the SBS after an aging period of six months. The first null hypothesis was that there would be no differences in bond strength between the five different universal adhesives after long-term water aging. The second null hypothesis was that the adhesive thickness and its resin infiltration into dentinal tubules would play no important role on the bond-strength durability.

2. Materials and Methods

2.1. Sample Selection

One hundred and fifty (150) human caries-free teeth, recently extracted for orthodontic reasons, were used in this study under a protocol approved by the Ethics committee (protocol no. 2018–89). The teeth were immersed in a 1% sodium hypochlorite (NaOCl) solution at 4 °C for 24 h and then stored in saline solution [15].

Two sections were made perpendicular to the longitudinal axis of the tooth crown using a saw microtome (Walter EBNER, Le Locle, Switzerland), to obtain dentin discs of 4 mm in thickness. The coronal surface was then hand-polished using a 320-grit silicon carbide paper (Escil, Chassieu, France) for 60 s continuous underwater irrigation [16].

The teeth were divided into five groups (30 teeth each) based on the universal adhesive systems used in this study. The adhesives were applied in self-etch mode following the manufacturer's protocol (Table 1). A silicone mold (3 mm in diameter) was used to make the resin composite build-ups on the occlusal dentin surface of the specimens using a resin composite Reflectys (ITENA Clinical, Paris, France), which was applied in three layers of 2 mm each. Each layer was light-cured for 40 s using an LED-curing system (Luxite Lampe LED, ITENA Clinical, Paris, France). Fifteen (50%) specimens of each group were stored in distilled water at 37 °C for 24 h, while the other remaining part of the specimens for were stored 6 months.

Table 1. Adhesive system: chemical composition, application process, and manufacturing [3,17–19].

Adhesive	Composition	Application
Iperbond Max—"IPM" (Itena Clinical, Paris, France)	10-MDP, 4-META, methacrylates, photo-initiators, ethanol, water, fumed silica	- Apply (20 s). - Wait until the solvent had completely vapored (20 s) - Dry (5 s) - Light cure (10 s).
Iperbond Ultra—"IPU" (Itena Clinical, Paris, France)	Triethylenglycol dimetracrylate, acrylate polyester urethane aliphatique, bonding acelators, metacrylate hydroxyethil, photo-starters, acetone, ethanol, nanoparticules.	- Apply + rub (20 s). - Dry (5 s) - Light cure (10 s).
Ibond Universal—"IB" (Kulzer, ZA Courtaboeuf, France)	Methacrylate monomers (UDMA), Hydrophilic monomers (4-META), glutaraldehyde, photo-initiators, stabilizers, acetone, and water	- Apply + rub (20 s). - Dry (5 s) - Light cure (10 s).
FuturaBond M+—"FB" (Voco GmbH, Cuxhaven, Germany)	HEMA, BIS-GMA, ethanol, Acidic adhesive monomer (10-MDP), UDMA, catalyst ethanol, water, initiators, and catalysts	- Apply + rub (20 s). - Dry (5 s) - Light cure (10 s).
Scotchbond Universal—"SU" (3M, St. Paul, MN, USA)	10-MDP, HEMA, dimethacrylate resins, Vitrebond copolymer, filler, ethanol, water, initiators, silane	- Apply + rub (20 s). - Dry (5 s) - Light cure (10 s).

2.2. *Shear Bond Strength Test (SBS)*

After each storage period, twelve specimens of each group were mounted to a testing machine (Instron 3345, "ISO/TS 11405 standard"). A constant crosshead speed of 0.5 mm/minute was used to submit the specimens to shear loading until fracture. By dividing the load at failure with the bonded surface area, the SBS (MPa) was calculated. After the SBS test, an optical numeric microscope (Keyence, Osaka, Japan) was used to investigate the failure mode in each specimen. A VHX-5000 software was used to calculate the percentage of each area at 50× magnification to define the type of fracture.

2.3. *Scanning Electron Microscopy (SEM)*

After each storage period, three samples of each group were sectioned along the sagittal plane, at the center of each cylinder using a saw microtome (Walter EBNER, Le Locle, Switzerland). Subsequently, the resin–dentin interfaces of the specimens were etched using 37% phosphoric acid for 10 s, rinsed for 10 s with distilled water, and immersed in a 2.5% NaOCl solution for 3 min [20]. The specimens were finally rinsed with distilled water and dehydrated in a graded series of ethanol solutions. These were then mounted on aluminum SEM stubs, and sputter-coated with gold–palladium alloys (20/80) using a sputtering device (Hummer JR, Technics, CA, USA). The adhesive layer thickness and length of the resin tags (10 measurement for each section) were analyzed using a Quanta 250 FEG scanning electron microscope (FEI Company, Eindhoven, The Netherlands) functioning with a 10 kV acceleration voltage of the electrons.

2.4. Statistical Analysis

The *t*-test was applied using a Sigma plot (release 11.2, Systat Software, Inc., San Jose, CA, USA) to determine whether significant differences existed in the SBS values of each adhesive system between the two storage periods (24 h and 6 months). On the other hand, one-way analysis of variance (ANOVA) was used to determine whether significant differences existed in the SBS values of the five adhesive systems at 24 h and at 6 months. The Shapiro–Wilk test was used to verify the normality of data within all groups. However, normality was never verified. A one-way analysis of variance on ranks test was also used to compare the layer thickness or tag depths. In all tests, a statistical significance level of $\alpha = 0.05$ was adopted.

3. Results

3.1. Shear Bond Strength and Failure Modes

Means and standard deviations, as well as the number of adhesive or mixed failures are depicted in Table 2. In general, higher SBS values were obtained for the tested adhesives after 24 h of a storage period compared to those obtained after 6 months (*t*-test, $p < 0.05$), except for Scotchbond Universal (SU) and Iperbond Max (IPM) ($p > 0.05$) (Figure 1).

Table 2. Shear bond strength means and standard deviations of the five adhesives at the two storage periods and the number of adhesive or mixed failures. FutúraBond M+—"FB", Scotchbond Universal—"SU", Iperbond Max—"IPM", Iperbond Ultra—"IPU", Ibond Universal—"IB". Different superscript letters indicate significant differences in rows, while different superscript numbers indicate significant differences in columns. Significance at 5% significant level ($p < 0.05$).

	FB	SU	IPM	IPU	IB
24 h	$17.6 \pm 3.4^{a,b,1}$	$19.2 \pm 6.4^{a,1}$	$18.6 \pm 4^{b,1}$	$15.9 \pm 3.7^{b,1}$	$15.3 \pm 3.3^{b,1}$
Fracture mode (Adhesive/Mix)	(13/2)	(8/7)	(10/5)	(12/3)	(12/3)
6 months	$14.7 \pm 4.1^{a,2}$	$20 \pm 6.1^{b,1}$	$18.6 \pm 3.9^{a,b,1}$	$12.9 \pm 3.1^{a,2}$	$12.9 \pm 2.3^{a,2}$
Fracture mode (Adhesive/Mix)	(12/3)	(9/6)	(11/4)	(13/2)	(13/2)

After 24 h, no significant difference was found between the SBS values of the five tested adhesives (one-way ANOVA on ranks, $p > 0.05$). Conversely, after six months of storage, no significant difference was found between SU, IPM, and FuturaBond M+ (FB) ($p > 0.05$). SU and IPM had higher SBS values than Iperbond Ultra (IPU) and Ibond Universal (IB) after prolonged storage ($p < 0.05$). Different failure modes were reported (Figure 2 and Table 2).

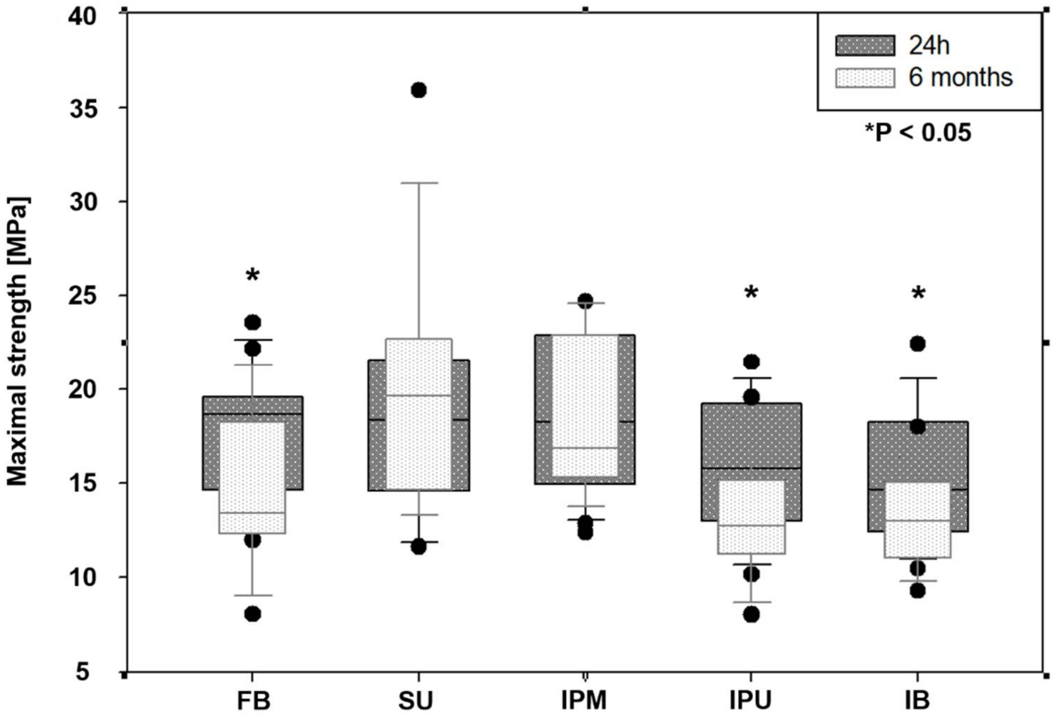


Figure 1. Evolution of the bond strength for the different universal adhesives at 24 h and 6 months. (* $p < 0.05$). FuturaBond M+—"FB", Scotchbond Universal—"SU", Iperbond Max—"IPM", Iperbond Ultra—"IPU", Ibond Universal—"IB".

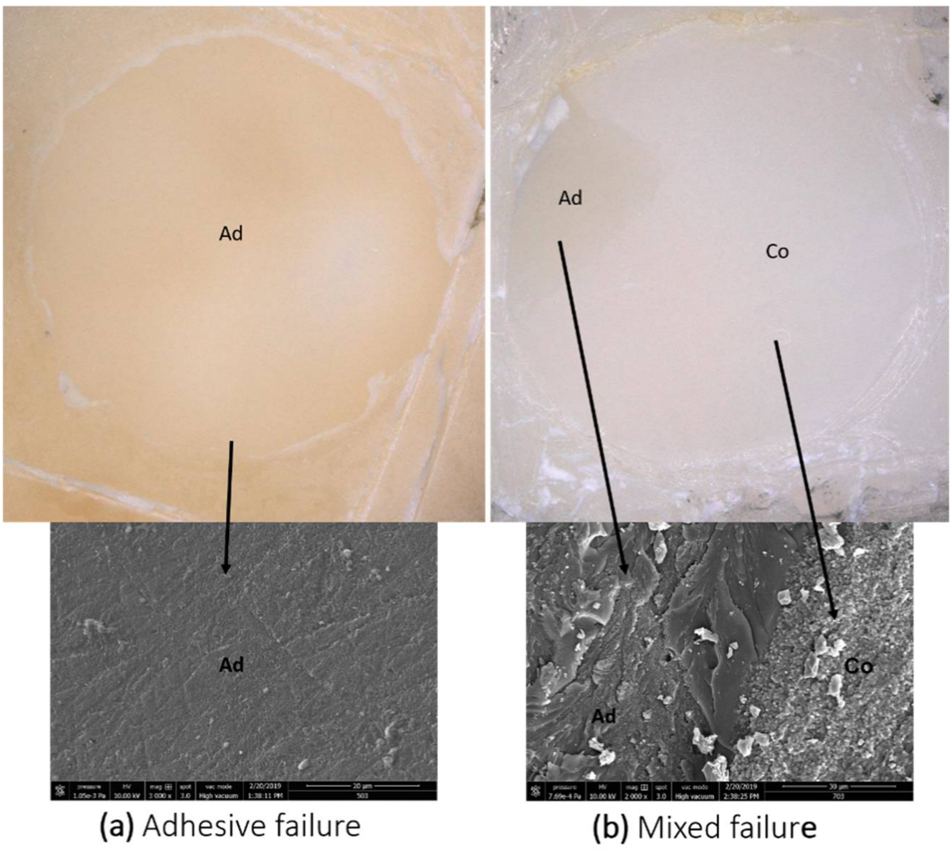


Figure 2. Representative images obtained with optical microscope and scanning electron microscopy (SEM). (**a**) Adhesive failure (×50 magnification); (**b**) mixed failure (×50 magnification). Ad: adhesive; Co: Composite.

3.2. Scanning Electron Microscopy (SEM) Observation of Adhesive–Dentin Interface

The mean thickness of the different adhesive layers ranged between 10 to 17 µm was observed and measured under SEM for all adhesive systems (Figure 3, Table 3). SU and IB mean values were significantly higher than the mean values of IPM and FB ($p < 0.05$). Some specimens were created with IB fractured at the resin–dentin interface, probably due to SEM preparations and working pressure (Figure 3f).

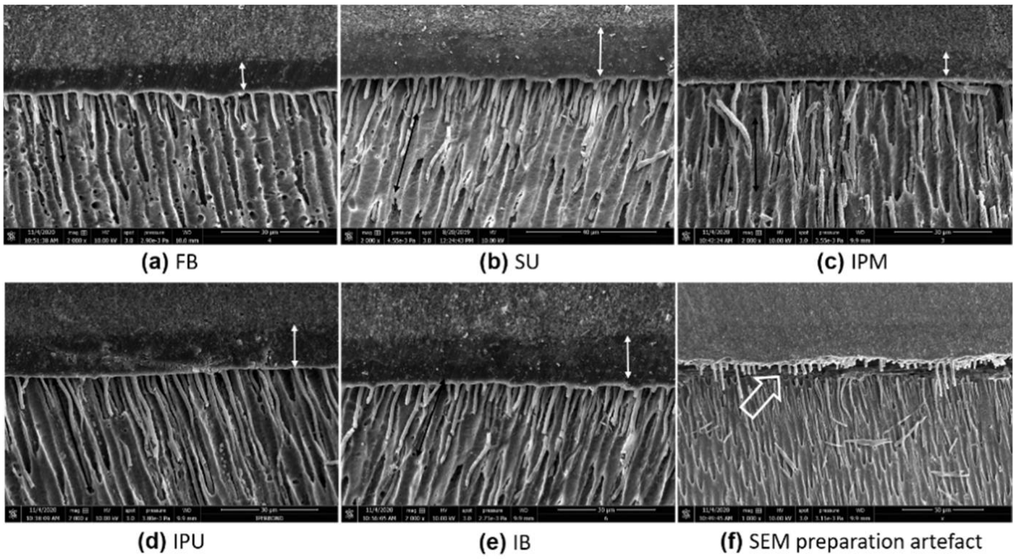
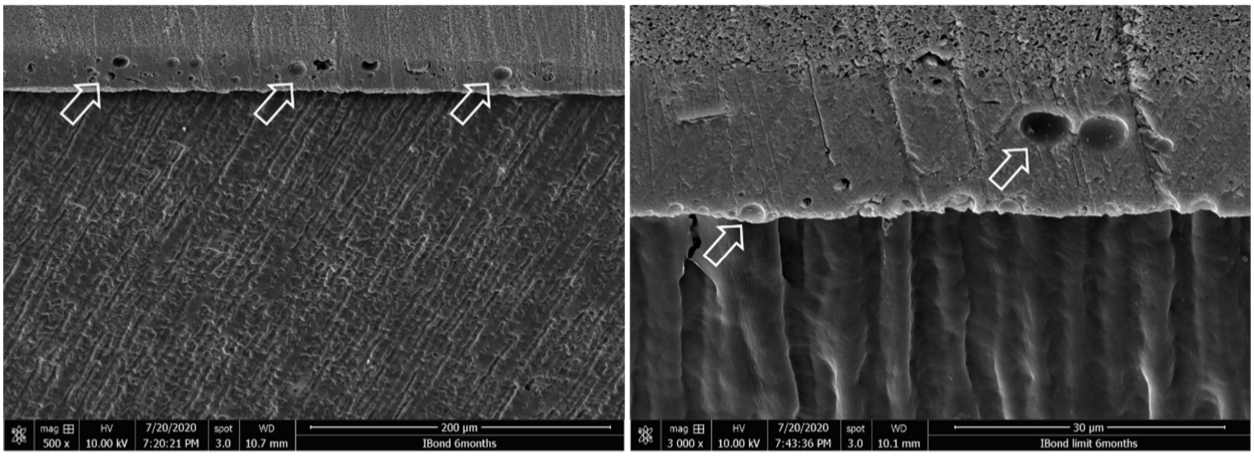


Figure 3. Representative photos of scanning electron microscopy (A-E, x2000 magnification) demonstrating the adhesive layer thickness (white arrows) and tag depth (black arrows). (**a**) FuturaBond M+—"FB"; (**b**) Scotchbond Universal—"SU"; (**c**) Iperbond Max—"IPM"; (**d**) Iperbond Ultra—"IPU", (**e**) Ibond Universal—"IB". (**f**) SEM preparation artefact (x3000 magnification, white arrow).

Table 3. Adhesive layer thickness and tag depth values (means and standard deviations) of the five adhesives. FuturaBond M+—"FB", Scotchbond Universal—"SU", Iperbond Max—"IPM", Iperbond Ultra—"IPU", Ibond Universal—"IB". Superscript letters ($^{a–d}$) indicate significant differences at 5% significant level ($p < 0.05$).

	FB	**SU**	**IPM**	**IPU**	**IB**
Adhesive thickness (µm)	$11 \pm 4^{a,c}$	$15 \pm 6^{a,b}$	$10 \pm 4^{b,d}$	13 ± 7	$17 \pm 9^{c,d}$
Tag depth (µm)	4.6 ± 2.2	4.8 ± 1.8	5.6 ± 2.1	6.1 ± 1.8	5.6 ± 2.3

No relation was observed between the SBS values and the thickness or the depth of resin-infiltration into dentinal tubules (Tables 2 and 3). SU and IB had the same infiltration depth and layer thickness but SU had higher SBS values at 24 h and 6 months. Therefore, thickness and tag length had no impact on the SBS values after the aging periods. One interesting finding was the observation of some voids in two IB six-month-aged samples (Figure 4).



(a) voids in IB sample (x 500) **(b)** voids in IB sample (x 3000)

Figure 4. Representative photos of scanning electron microscopy showed the voids into the IBond Universal adhesive layer (white arrows) after a 6-months water-storage period at 37 °C. (**a**): x500 magnification; (**b**): x3000 magnification.

4. Discussion

Dentin is a tissue characterized by a high organic content, which includes collagen fibers and odontoblasts in dentin tubules; these latter are filled with dentinal fluid [7,21]. It is well-known that the hydrolysis of collagen fibrils during storage in water could decrease the bond strength of the resin–dentin interface [7,18,21]. In this study, the bond strength for all the tested adhesive systems, which were applied following their manufacturers' instructions, was higher after 24 h of storage in water than after long-term storage. Immediately (after 24 h) the SBS test showed no significant differences ($p > 0.05$) between the tested adhesives.

The adhesives SU and IPM preserved the bond strength values after a six-month storage period, whilst the SBS of IPU, IB, and FB decreased after long-term storage ($p < 0.05$). Therefore, the first hypothesis that there would be no difference between the tested adhesives in bond strength after long-term storage must be rejected. A possible explanation might be correlated to the different compositions of the tested materials, which influence the quality of the hybrid layer they create [2,22]. Indeed, the stability and durability of bond strength of IPM and SU could be in part attributed to the quality of 10-MDP used within their formulations. The formation of stable and insoluble nano-layered 10-MDP-calcium salts can significantly improve the bond strength and its durability over time [23]. Initial bond strength and its durability was shown to be important in the functional monomers that have more intense monomer-calcium formation than those creating lower monomer-calcium formation [23,24]. The results of our study are in accordance with those of Zhang et al. [25] and Saikaew et al. [2]; they reported that SU when it is applied in self-etch mode could preserve the bond strength after 12 months of an aging storage period. FB had lower bond strength after long storage, although it contains MDP; this may be due to the quality of the MDP used in the adhesive [26].

In this study, SEM was used to measure the thickness of the resin layer of the five tested adhesives applied onto dentin; the mean thickness ranged between 10 and 17 µm. SU and IB were significantly thicker than the other adhesives ($p < 0.05$). These findings might be explained and related to the adhesive fillers, which could play a crucial role in the flowability of such adhesives. However, no correlation was found between the thicknesses and SBS values of the tested adhesives. It has been reported that the bond strength of resin–dentin interface is independent from the thickness of hybrid and/or adhesive layer [27]. Conversely, the quality of hybrid layer is an important factor that can influence the dentin bond strength, whilst a thick adhesive layer does not offer any increase in bond strength [28].

All the adhesive systems used in this study created resin tags, which infiltrated several microns into dentinal tubules. Therefore, we hypothesized that all such adhesives can sufficiently dissolve the smear layer and allow the resin adhesive to enter into the tubules. However, the results of tag lengths were not significantly different ($p > 0.05$). These findings could be explained by the fact that all the tested adhesives have a mild pH. Thus, the second hypothesis that the adhesive thickness and its resin infiltration into dentinal tubules would play no important role on the bond strength durability must also be rejected.

All the measures for the evaluation of the tag penetration were performed in correspondence of the center of the composite-cylinder in order to prevent the effect of tubules position (density and diameter). Tubules diameters and density increase from the dentin-enamel junction to central dentin area [29].

Regarding the voids within the resin–adhesive layer of IB six-month-aged samples, it could be due probably to an incomplete evaporation of solvents and waters from the dentine interface [30]. Moreover, the gap observed in some specimens (Figure 3f) may be a consequence of specimen preparation, including sectioning procedures and drying steps [7]. The SEM is an effective analytical test to evaluate the resin–dentin interfaces, but with some limitations such as the artefacts (fracture or gap) at the dentin–adhesive interface caused by the different preparation steps [7]. However, one of the limitations of this study is that the bond strength of the tested adhesives was evaluated in vitro by the SBS test on flat dentin surfaces; this neglects an important clinical factor, which is the effect of C-factor [29]. In this study, the samples were tested using the SBS test because it is easier to prepare and no sectioning preparations (stick of 1 mm^2) are required to perform the experiment. Beloica et al. [31] reported that the adhesive system is a significant factor for bond strength, regardless of the testing method used. However, other studies reported that the use of microtensile bond strength in this type of research is better than the SBS test [32]. Therefore, further in vitro and in vivo studies should be performed in the future to confirm the current preliminary results. Moreover, additional studies on different dentin-demineralization depths after adhesive application are recommended in order to analyze the quality of monomer-calcium bond to confirm these latter outcomes.

5. Conclusions

The current results indicate that adhesives with different chemical composition, the type, and the quality of the functional monomer within the materials' composition may influence the bonding performance in terms of degradation during prolonged storage in water, regardless of the adhesive layer thickness and length of the tags' penetration into the dentinal tubules.

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