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## Estimation of Self-Etch Adhesive System and Resin Cement Shear Bond Strength to Root Dentine with and without Prior Etching – Pull-Out Test and SEM Analysis

Ocena wpływu dodatkowego trawienia na wytrzymałość połączenia zębiny korzeniowej z wkładami koronowo-korzeniowymi wzmocnionymi włóknami szklanymi, cementowanymi za pomocą systemu samotrawiącego – test *pull-out* i analiza SEM

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A – research concept and design; B – collection and/or assembly of data; C – data analysis and interpretation; D – writing the article; E – critical revision of the article; F – final approval of article

### Abstract

**Background.** De-bonding is still considered to be the most common cause of failure concerning fiber posts. The proper application of adhesive is, therefore, crucial to improve the bond strength.

**Objectives.** This study evaluated whether the additional etching before the application of cross-generation self-etch adhesive and resin cement can increase the shear bond strength between the root dentine and fiber post's surface.

**Material and Methods.** Sixteen human one-canal teeth that had been extracted due to periodontal and orthodontic reasons were carefully selected for this study. They were divided into two groups of eight. Preparation of the specimens in both groups was equal except for the cementation procedure. The teeth were sectioned at the cementoenamel junction. The post space was prepared to a depth of 8 mm and a diameter of 1.5 mm with a special machine ensuring the repeatability of post spaces' shape. Posts were cemented using self-etch adhesive; in one group the root dentine was previously etched and the second group was without pretreatment. Posts were cemented with the use of metal rings. Then specimens were embedded in acrylic resin blocks. They were subjected to pull-out test using a Universal Testing Machine specially adapted to maintain the parallelism – regimen. Finally, the posts' surface was evaluated in SEM observation. Obtained results were statistically analyzed.

**Results.** The mean shear bond strength values for group with prior etching of dentine were significantly lower (8.22 MPa) than for the group without prior etching (11.25 MPa). These findings were supported by SEM observations, revealing that in the group with prior etching the resin-dentin-interface failure surpassed the resin-post-interface component.

**Conclusions.** Obtained results indicate that the self – etch adhesives' bond strength to root dentine is decreased in case of prior etching (**Dent. Med. Probl. 2014, 51, 2, 197–204**).

**Key words:** fiber reinforced composite posts, self – etch adhesive, etching, root dentin, pull-out test.

### Streszczenie

**Wprowadzenie.** Jednym z najczęściej spotykanych powikłań dotyczących wkładów wzmocnionych włóknami szklanymi (FRC) jest utrata retencji polegająca na ich odcementowaniu, dlatego właściwe zastosowanie systemów wiążących jest niezwykle ważne.

**Cel pracy.** Ocena wpływu dodatkowego wytrawienia zębiny na wytrzymałość połączenia między wkładami FRC a zębiną korzeniową po użyciu uniwersalnego systemu samotrawiącego typu cross-generation i cementu kompozytowego.

**Materiał i metody.** Badaniu poddano szesnaście jednokanałowych zębów usuniętych ze względów periodontologicznych lub ortodontycznych. Podzielono je na dwie grupy. Próbkę w obu grupach przygotowano w jednakowy sposób, poza procedurą cementowania. Na wysokości połączenia szklwno-cementowego odcięto korony zębów. Łoża pod wkłady wypreparowano na głębokość 8 mm i o średnicy 1,5 mm. W tym celu użyto maszyny zapewniającej powtarzalność kształtu łoża. Wkłady zacementowano z użyciem systemu samotrawiącego. W jednej grupie dodatkowo wytrawiono zębinę. Wkłady cementowano z użyciem metalowych pierścieni. Próbkę zatopiono w akrylowych blockach i poddano je testowi wyciągania w uniwersalnej maszynie testującej, dbając o równoległość wszystkich elementów. Powierzchnię próbek poddano badaniu SEM.

**Wyniki.** Wykazano istotną statystycznie różnicę między wytrzymałością połączenia w grupie, w której dodatkowo wytrawiono zębinę (8,22 MPa) względem grupy, w której zębina nie była wytrawiona (11,25 MPa). Badania SEM wykazały, że w grupie z dodatkowym trawieniem połączenie było zerwane głównie na granicy cement–zębina.

**Wnioski.** Uzyskane wyniki wskazują, że dodatkowe wytrawienie zębiny korzeniowej przed cementowaniem z użyciem systemów samotrawiących pogarsza wytrzymałość połączenia zębina korzeniowa–cement kompozytowy–wkład FRC (*Dent. Med. Probl.* 2014, 51, 2, 197–204).

**Słowa kluczowe:** wkłady wzmocnione włóknami szklanymi, systemy samotrawiące, trawienie, zębina korzeniowa, test wyciągania.

Fiber-reinforced composite (FRC) posts are nowadays commonly used in restorative dentistry. These posts are usually required in endodontically treated teeth with coronal structure loss, especially the anterior teeth. Such teeth are already dehydrated [1] and lack a great deal of their original structure, which makes them weaker and, therefore, more prone to fractures [2]. However, such fractures of teeth with fixed posts are rather encountered with metal or zircon posts, which have a very rigid nature [3–5]. The placement of a fiber post and core reduces the risk of such tooth fractures, even though the root stresses become higher than those reached with metal posts [4]. Because their elastic modulus is similar to that of dentine, the stresses along the fiber post's surfaces decrease, and as a result, the risk of de-bonding is lowered [4]. However, retrospective studies on the clinical performance of fiber posts have previously shown that de-bonding is considered to be the primary cause of FRC posts' failure [6–8].

De-bonding usually occurs at the luting interface between root dentine and the resin cement [9, 10]. The most appropriate and efficient adhesives, and their procedural steps, should therefore be defined as a way to determine a clinically relevant and acceptable bonding strength.

In order to cement FRC posts, either the etch-and-rinse adhesives or self-etch adhesives can be used. Conventional etch-and-rinse adhesives are supposed to provide higher bond strength [11, 12]. However, the self-etch adhesives' application technique is simpler, more repeatable, and less sensitive (the issue of dentine wetness does not exist). Self-etch adhesives do not require a separate etching and rinsing step. Etching and priming are carried out simultaneously because the acidic monomers are combined with the primer into one solution. Currently used self-etch adhesives have the ability to penetrate beyond the smear layer and

modify it, and then ultimately incorporate it into the adhesive resin [13].

In this study, a cross-generation self-etch adhesive was used to cement FRC posts. The manufacturer mentions that, while working with these adhesives, the user has the option of etching the prepared surface prior to the application of the self-etch adhesive. Some researchers can be found incorporating this supposed advantage into their methods with the expectation of improving the performance of this self-etching adhesive [14]. However, there are several researchers who have also proved that this additional etching does not actually improve the bonding strength capacity of self-etch adhesives at all, and may even decrease its bonding strength capacity [15–17]. This phenomenon can be explained by the fact that the mere presence and micro-morphology of the prepared smear layer ultimately determines the micromechanical properties and chemical interactions between the self-etch adhesives' and root dentine's components, as well as the structure of the hybrid layer [15–20]. Following such assumptions, any surface treatment of root dentine can influence the bonding outcomes, whether it will be etching or the application of any chemical agents [14, 18–22].

In this study, the pull-out test was used to obtain a quantitative assessment. As a way to obtain more consistent and reliable results, the steps of preparing specimens were specially formulated, and the pull-out test was eventually modified. Qualitative assessment was performed during SEM analysis of extruded posts.

The aim of this study was to evaluate whether the additional etching before applying the cross-generation self-etch adhesive and resin cement can increase the bond strength between the root dentine and fiber post's surface. The null hypothesis was that there would be no significant differ-

ence between the shear bond strength of self-etch adhesive system to root dentine, with or without prior etching.

## Material and Methods

Sixteen human teeth with one canal (except lower incisors) that were previously extracted due to periodontal and orthodontic reasons were selected for this study. These teeth were stored in a 0.1% thymol solution during the course of the study and were used within a month after their extraction. Any teeth that had irregularly shaped or obliterated canals and had a root length of less than 11 mm long were excluded from this study. Furthermore, any teeth with defects, fractures or previous endodontic treatment, were excluded as well.

The crowns were cut transversally at the cemento-enamel junction with a cylindrical diamond rotary bur (Dentsply Maillefer, Ballaigues, Switzerland) at high speed under water coolant and discarded. The preparation of post space was performed on the specially constructed machine, which enabled us to standardize these spaces, and therefore eliminate any possible variations in the shape of the post space that might occur during typical manual preparation. The post space was enlarged under water coolant with Pessio® Reamers No 2, 3 and 4 (Poldent, Warszawa, Poland). The final shaping of the post space was accomplished with a special drill, matched to the posts (FiberKleer Parallel Post Kit™; Pentron Clinical Technologies LLC., Wallingford, CT) to a depth of 8 mm, with a diameter of 1.5 mm. It was then possible to obtain the same dimensions and proper adaptation of the post, as well as the recommended 0.1–0.3 mm cement thickness [9]. This was followed by the preparation of the transverse micro-cuts on the surface of the root.

In this study, prefabricated FiberKleer® Posts with a diameter of 1.5 mm (FiberKleer Parallel Posts™; Pentron Clinical Technologies LLC.) were used. They are parallel and have a pre-silanized surface. The length of each post is 10.0 mm. These posts were chosen for this study because they are mounted with a retentive head that ensures consistent and repeatable action of grasping each specimen during the pull-out test, and it also prevents any destruction of the post's fibers induced by the holding jaws of the Universal Testing Machine.

These specimens were randomly assigned to two groups of eight teeth, each group being classified according to the procedures that would be used to cement each post. In the first group (A), the additional steps of etching, rinsing and drying of dentine preceded the application of the self-

etch adhesive system. In the second group (B), the same brand of self-etch adhesive was applied, but there was no prior etching of dentine. In the A-group, 37% phosphoric acid was applied into the post space for 15 s, activated, then thoroughly rinsed with a water-filled syringe. In the B-group, the post space was only rinsed with saline. In both groups, the post space was dried with absorbent paper points (VDW GmbH, Munchen, Germany) so that the root dentine stayed moist, but not wet.

The following procedures were then performed equally in both groups. The self-etch adhesive was applied according to the manufacturer's instructions. The universal cross-generation adhesive (Iperbond®; Itena Clinical, Paris, France) was mixed with its chemical bond activator (Bond Activator; Itena Clinical), and was then applied to the inner walls of the prepared post space with a microbrush, and rubbed in for 15 s [23]. Any excess adhesive was then removed with sterile paper points. Following the adhesive application, the dual-polymerizing luting resin cement (Dentocem®; Itena Clinical) was then applied into the post space using a thin, self-mixing tip, and the post was then immediately placed to the full depth of 8 mm. Any excess cement was removed with a microbrush. The polymerizing light rays were transmitted parallel to the long axis of the root and the tip of the polymerizing light was in direct contact with the coronal end of the post for 20 s. Due to the translucency of these posts, and to the dual-polymerizing action of the resin and cement, the most optimal bond strength in all regions of the post space could then be achieved [10, 20].

Cementation was performed with the use of specially fabricated metal rings that were fixed between the retentive head of the post and the root. After the cementation procedures were finished, the specimens were then stored in a (0.9%) saline solution for 48 h to allow for the complete bonding between the adhesive system and resin cement.

The next step was to embed each specimen into a cylindrical block of auto-polymerizing acrylic resin (Duracryl®; Spofa Dental, Jicin, Czech Republic), while concomitantly maintaining the parallelism between all of the elements of the specimen. Previously made transverse cuts on the external surface of the root ensured the complete retention of the specimen within the acrylic block. During the process of acrylic resin polymerization, the specimens were kept in a cold-water bath in order to eliminate the harmful influence of heat. At this point the preparation of the specimens was completed. The parallelism between the post, canal and acrylic resin block was obtained.

This study was performed in cooperation with the Department of Strength of Materials and Structures, Technical University of Lodz. The pull-out test was conducted on the Universal Testing Machine Instron® 4485 (Instron 4485; Norwood, MA, USA) which was specifically adapted for this study. The pull-out test consisted of extruding the post from the canal along its long axis. The specimens were held in place in the machine using two separate holding jaws. The acrylic resin block was secured to the lower jaw. The metal fixation ring was secured to the upper jaw. Both holding jaws were constructed in such a way that, while the specimen was being held firmly in place, the positioning of the jaws could be adjusted in any direction to assume the most optimal parallel position. This enabled the placement of the specimen at the ideal angle of 0 degrees in relation to the long axis of the post.

The final step was performing the pull-out test (now modified) in the Universal Testing Machine. The extrusion load of 1 mm/min was applied to all the specimens under equal conditions.

The results were obtained in N (newtons) and converted to MPa (mega pascals), revealing the magnitude of the achieved shear bond strength.

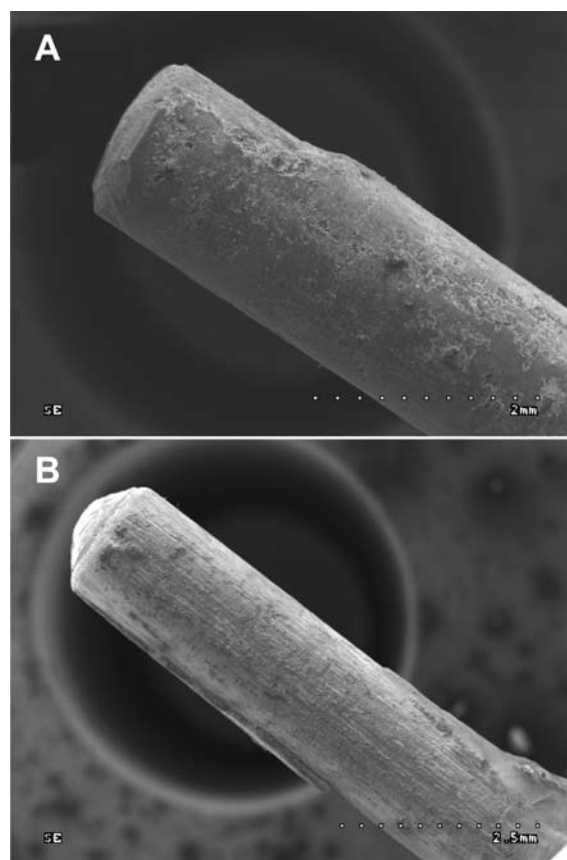
The results were analyzed statistically (Statistica PL 6.0™; StatSoft Polska, Kraków, Poland). The verification of the normal distribution was made using the Kolmogorov-Smirnov's test. The comparative analysis of the strength obtained for specimens with and without previous etching was performed, using the Student's t-distribution test. Statistical significance was set at  $p < 0.05$ .

The last approach was SEM (Hitachi® S-3000 N; Tokyo, Japan) examination of the extruded posts in the pursuit of visually identifying and verifying the surfaces at the de-bonding interface.

Finally, cross-sections of two additional, previously untested specimens representing each group (A and B) were analyzed microscopically to assess the quality of the bonding interface and to identify the presence or absence of any gap that may have been created between the bonded surfaces. They were sliced and polished along their long axis with a metallographic grinder (Metapolan®; Rathenower Optische Werke GmbH, Rathenow, Germany) under water coolant and then were examined microscopically (MA-200; Nikon, Tokyo, Japan). To improve the quality of this microscopic image, the surfaces of both specimens' cross-sections were sprayed with a thermal layer of 99.99% Au (JEE-4X; Jeol, Tokyo, Japan) and were then also subjected to microscopic observation (Hitachi S-3000 N).

## Results

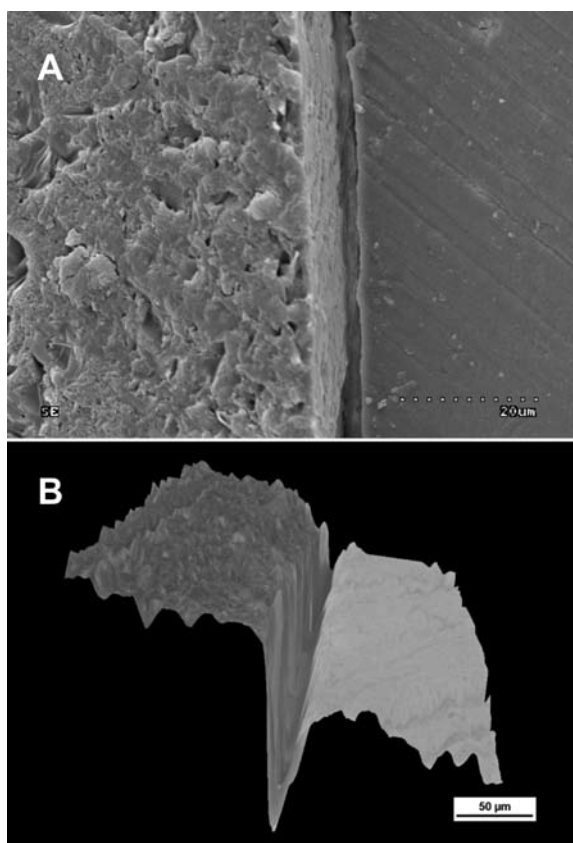
The obtained results indicate the values of the pull-out bond strength in all specimens. In the group with pre-etched dentine, the mean shear



**Fig. 1.** Figure 'A' shows the SEM analysis for the post cemented with previous etching. The de-bonding failure had mixed characteristics with greater dentin resin interface component. The surface of the post is almost entirely covered with the luting cement, as only a small area of the naked post can be seen. Figure 'B' shows the SEM analysis for a post that is cemented without previous etching. In this case the failure of bonding also had mixed characteristics, but the de-bonding occurred mainly at the post-cement interface. In addition, the bonding strength in the apical area appeared to be weaker, as in this region the resin remained on the post. Only small areas of remaining luting cement can be seen on the surface of the posts.

**Ryc. 1.** Przykładowe obrazy uzyskane w skaningowym mikroskopie elektronowym po przeprowadzeniu badawczej ekstruzji wkładów. „A” ukazuje wkład, który był zacementowany z uprzednim trawieniem zębiny. Zerwane połączenie ma charakter mieszany, głównie zerwanie połączenia nastąpiło na granicy cement/zębina korzeniowa. Powierzchnia wkładu jest prawie w całości pokryta resztkami cementu. Rycina „B” ukazuje wkład, który był zacementowany bez dodatkowego trawienia zębiny. W tym przypadku zerwanie połączenia ma również charakter mieszany, ale przebiega głównie na granicy wkład/cement. Dodatkowo są widoczne pozostałości cementu na wkładzie w jego wierzchołkowej części, co świadczy o słabszej wytrzymałości połączenia z zębina korzeniową w części wierzchołkowej

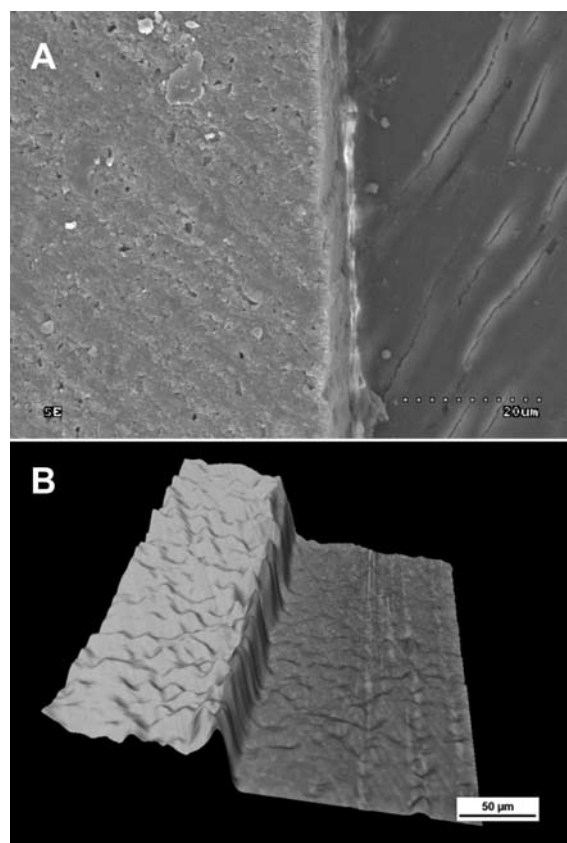




**Fig. 2.** Figure 'A' shows the SEM image of the cross-section of the specimen with etched dentine prior to application of the self-etch adhesive system. The gap between the resin cement and the dentine can be seen. The gap's width amounts to several nanometers. Since this gap is narrower than the diameter of any bacteria, it becomes irrelevant as a contamination risk factor. Figure 'B' shows the image obtained in the optical microscope made to confirm the presence of a gap. It was transformed so that the topography of the surface can be seen. Such a projection shows the gap distinctly

**Ryc. 2.** Rycina „A” przedstawia obraz uzyskany w skaningowym mikroskopie elektronowym – przekrój przez próbkę, uprzednio wytrawioną. Widoczna jest szczelina między cementem a zębina korzeniową, jej szerokość nie przekracza kilku nanometrów. W związku z tym, że jest węższa od średnicy bakterii, nie stanowi wrót zakażenia. Rycina „B” przedstawia obraz uzyskany w mikroskopie optycznym wykonany w celu potwierdzenia obecności szczeliny. Obraz został wykonany w projekcji pokazującej topografię powierzchni przekroju i ukazuje wyraźnie istnienie szczeliny

bond strength value was  $8.22 \pm 0.83$  MPa, with a range from 6.96 MPa to 9.40 MPa. In the group without previously etched dentine, the mean shear bond strength value was  $11.25 \pm 1.84$  MPa, with a range from 8.69 MPa to 13.39 MPa. The distribution of strength values did not deviate from the normal distribution. The Student's t-distribution test revealed that there was a statistically significant difference between groups at  $p < 0.001$ .



**Fig. 3.** Figure 'A' shows the SEM image of the cross-section of the specimen without previous etching of dentine. There is no gap between any of the surfaces. The hybrid layer of the self-etching adhesive can be seen. 'B' shows the topography of the surface. No gap can be seen. The difference in height between the two layers can be seen; this can be caused by a difference in toughness between specific materials during polishing

**Ryc. 3.** Rycina „A” przedstawia obraz uzyskany w skaningowym mikroskopie elektronowym – przekrój przez próbkę zacementowaną bez dodatkowego trawienia. Nie zaobserwowano obecności szczeliny na granicy zębina/cement/wkład. Widoczna jest natomiast warstwa hybrydowa wytworzona przez system wiążący. Rycina „B” przedstawia obraz uzyskany w mikroskopie optycznym przedstawiający topografię powierzchni przekroju, który potwierdza brak szczeliny. Widoczna jest różnica w wysokości dwóch warstw, co może być spowodowane różną twardością materiałów podczas polerowania próbki

The recorded observations of the SEM analysis of the de-bonding-interface of extruded posts are presented in the included figures (Fig. 1). The images of the bonding interface reveal the presence of a gap between the dentine and resin cement interface among the specimens that were treated with previous etching and the second additional microscopic examination supports this finding, and the included figure presents the transformation of the image showing the surface topography (Fig. 2). Finally, the microscopic examination of the speci-

**Table 1.** The table shows the shear bond strength values [MPa] obtained in both groups in the pull-out test in this study.

**Tabela 1.** Tabela ukazuje wartości wytrzymałości połączenia [MPa] uzyskane w badanych grupach w teście pull-out.

	Group-A with previous etching	Group-B without previous etching
Max	9.40	13.39
Min	6.96	8.69
Mean	9.18	11.25
Standard deviation	0.77	1.72
Median	8.19	11.25

men without the additional prior etching found no gap (Fig. 3). The description for the images is included below each figure.

## Discussion

The null hypothesis was that there would be no significant difference between the bond strength of self-etch adhesive systems to root dentine, with or without prior etching, was rejected. The obtained results indicate that the shear bond strength is higher when self-etch adhesive is applied according to the manufacturer's directions without any prior modification of the root dentine. The additional etching deteriorated the bond strength. This phenomenon can be explained by the fact that self-etch adhesives modify the smear layer and incorporate it into the hybrid layer. As the etching step results in complete removal of the smear layer and smear plugs, the micromechanical and chemical interactions with them are interfered with [15, 16, 18]. Also, Van Landuyt et al. [15] reports that although the additional etching preceding the application of self-etch adhesive improves the bond strength to enamel, it significantly impairs bonding effectiveness to dentine. Previous etching results in an inferior quality of hybrid layer that contains submicron gaps, which is therefore prone to nano-leakage [15]. This suggestion is supported by the SEM observation in our study (Fig. 1), because in specimens with prior etching, the failure of bonding occurred at the root dentine – luting cement interface. Additional microscopic examination of the bonding interface in a specially prepared specimen also showed the presence of the gap along the dentine-resin cement interface, which was further supported by the image showing the topography of this surface (Fig. 2). Even though the hybrid layer after additional etching

is thicker than the hybrid layer obtained without etching, its changed structure does not ensure sufficient anchorage for the resin [15]. Moreover, the bond strength is independent of the thickness of the hybrid layer [20, 24]. In addition, the chemical interaction between the functional monomers of the adhesive and the hydroxyapatite crystals that normally have a strong affinity for one another deteriorates, because this extra etching causes the hydroxyapatite crystals to be inaccessible [15]. They also suggest that after etching, the exposed dentine is too rich in collagen fibrils, and this disturbs the infiltration of the resin [25]. Other authors also claim that pre-etching causes deep dentine demineralization leading to incomplete infiltration of the resin. As a result, there is a 'defective zone' at the hybrid layer's base, further decreasing the bond strength [15, 17, 26]. Alternatively, the acidic monomers contained in the self-etch solution condition the dentine in such a way that the collagen network is infiltrated properly and entirely [16, 17]. The other reason for decreased shear bond strength performance in specimens with pre-etching can be attributed to the presence of water while the polymerization process is taking place. Besides the fact that while using the self-etch adhesive systems there is always a risk of leaving too much water before polymerization, as they are water solutions, there is also an additional risk caused by the previous etching. After the removal of the smear layer, the dentinal tubules are opened and can cause dentinal fluid movement. This leads to additional water excess that impairs the bonding capacity [27, 28]. Over-wetting of the dentine combined with the highly water-permeable nature of self-etch adhesives [29] interferes with the polymerization and ultimately has negative effects on the performance of the adhesive resin, which results in a weaker bond strength. All of these hypotheses can together serve to help explain the resulting weaker shear bond strength values obtained for specimens with dentine that was etched prior to the application of a self-etch adhesive.

Analysis of the data obtained from SEM images indicated that there were notable differences found between the bond strength at the dentine/resin-interface and the fiber post/resin-interface, both in specimens with, and without, previous etching. Our SEM observations and analyses were not only consistent with the data, but also revealed the specific contact areas that were responsible for the weaker bonding capacity. The SEM images clearly show the occurrence of de-bonding at either the dentine-resin-interface, the resin-post-interface, or a combination of the two. In both groups the locations of bonding failure had mixed characteristics. However, in both cases there

was adverse advantage of the de-bonding interface combination. The testing until failure of de-bonding manifested better clinical performance in specimens without pre-etching, because it regarded the post-cement interface. Even though in such cases where it can be difficult to remove the cement debris from the inner surface of the post space, it has relevant clinical implications. The dentinal tubules are sealed off by the residual cement and are more resistant to any possible contamination from the oral environment. On the other hand, in specimens with previous etching, the bond strength was better on the post-resin interface. This finding indicates that the adhesion to dentine is impaired. Such observations only stand to further support the results obtained in our pull-out test.

Due to the fact that de-bonding is the main cause of failure of FRC post and core treatments, the optimal adhesive procedural steps should be stated for the clinician to achieve maximum allowable bond strengths. There are many tests aimed at assessing the bond strength value, and the rules of these tests should consist of the measurement of the force that is sufficient to dislodge the post, that ultimately depends on the effectiveness of the luting procedure to the root dentine [30]. In this study the pull-out test was used. The biggest challenge was to maintain the axial alignment of all the elements throughout. In this study this was accomplished by yielding to the importance of

parallelism from the very beginning. Firstly, the specimens were prepared so that all the elements were set in one mutual axis. Then the specimen was fixed in the Universal Testing Machine with the aid of the moveable holding jaws, ensuring the proper axial alignment of the specimen during the pull-out test.

Any chemical root dentine surface treatment before the application of a cross-generation self-etch adhesive should be carefully considered as it may significantly influence their bond strength capacity. This refers not only to the 37% phosphoric acid used in this study, but also may apply to any other chemicals that have a similar effect on the smear layer [21].

Within the limitations of this study, it was concluded that the shear bond strength of self-etch adhesive system to root dentine is decreased when used in cases of additional prior etching. Moreover, in specimens without pre-etching of dentine, the failure of de-bonding was found to occur at the post-cement interface, while in specimens with previous etching of the root dentine, the failure of de-bonding was manifested at the dentine-cement interface. This indicates weaker adhesion to dentine after unnecessary removal of the smear layer. When etching and rinsing precede the application of a self-etch adhesive, this unfortunately results in the presence of a gap width of several nanometers along the dentine-resin cement interface.

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