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Posted Date: 22 February 2024

doi: 10.20944/preprints202402.1282.v1

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Effect of Dentin Irrigants on Push-Out Bond Strength in Resin Cementation Protocols for Fiber Post in Endodontically Treated Teeth: An *In Vitro* Study

Sandra García Varela [1,*,†], João Carlos Ramos [2,†], María José Ginzo Villamayor [1,†], Pablo Castelo Baz [1,†], Ramón Méndez Díaz [1], Marcos Aníbal Anache D`Abate [1], Tania Gancedo Gancedo [1], Manuel Ruíz Piñón [1], Soledad Mareque Bueno [1] and Benjamín José Martín Biedma [1,†]

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Abstract: A The aim of this study is to analyze the effect of different endodontic irrigants and adhesive systems on resin bond strength of fiber post cementation. 144 single-rooted, unrestored human teeth were endodontically treated and randomly divided into 12 groups according to 4 endodontic irrigants (distilled water as control; EDTA 17%; NaOCl 5%; clorhexidine 2%) and 3 different adhesive/resin cement systems (etch-and-rinse: ortofosforic acid, Parabond® A+B/Paracore®; self-etch: ParaBond® Non-Rinse Conditioner, Parabond® A+B /Paracore®; Universal: Clearfil™ Universal Bond/ Clearfil™ DC Core Plus). 48 h after post cementation ten teeth from each group were cross sectioned into 3 discs (cervical, middle and apical regions). 30 specimens of each group (n=30) were submitted to push-out test at a crosshead speed of 1 mm/min. The remaining 2 teeth of each group were sectioned in the same manner and the resin-dentin interface was evaluated using scanning electron microscopy (SEM). The results were statistically analyzed with the ANOVA and Tukey's test ($p<0,01$). The adhesive protocols and post space region showed no significant effect on bond strength ($p>0.01$). The combination of NaOCl 5% and ClearfilTM Universal Bond reduced the adhesive strength ($p<0.01$). The NaOCl 5%, in relation with other irrigants, significantly decreased the push-out bond strength.

Keywords: Bond strength; push-out; fiber post; hybrid layer; dental adhesive; dentin; chlorhexidine; EDTA; Sodium hypochlorite

1. Introduction

Annually, millions of prefabricated fiberglass posts are sold worldwide to restore teeth that have lost a significant portion of their coronal structure in order to regain uniform, functional, and aesthetic tooth restoration. Intraradicular fiber posts must be cemented to prevent additional stress on the dentin [1–4].

The bond strength between the cement and dentin surface can be altered based on dentin adhesive etching strategies (etch-rinse, self-etch and universal adhesives)[5–8].

The action of acid etching lead to collagen demineralization and hydrolytic degradation; the demineralized collagen layer may result in incomplete resin monomer infiltration [9] and may degrade due to the collagenolytic action of endogenous enzymes MMPs and Cysteine Cathepsins released during acid etching [10–12]. The use of endodontic irrigants may variably improve the bond strength between the cement and dentin collagen [13–18].

 doi:10.20944/preprints202402.1282.v1

Ethylenediaminetetraacetic acid (EDTA) can demineralize dentin without altering the collagen layer and chelate Calcium and Zinc ions necessary for MMPs and hydrolases [19]. NaOCl possesses oxidative and proteolytic capabilities [20] and enhances dentin surface tension and monomer wettability [21]. Chlorhexidine (CHX) is a biguanide agent that exhibits substantivity by binding to demineralized dentin [6,22]. CHX is a nonspecific inhibitor of both MMPs and cysteine cathepsins in a dose-dependent manner [11,23] and mimics tissue inhibitor of metalloproteinases (TIMPs) that preserve the collagen layer from degradation [24,25]

The aim of this study was to analyze the effect of different endodontic irrigants and adhesives systems with different acid etching strategies on the resin-dentin interface using Push-Out test and scanning electron microscopy (SEM) for fiber post cementation. Null hypotheses tested: (1) the type of irrigant, (2) the type of adhesive/cement, and (3) the region within the post space do not impact resin bond strength.

2. Materials and Methods

One hundred and forty four single-rooted, unrestored and caries-free human teeth with straight root canals were extracted, due to periodontal reasons, with prior informed consent from the patient and stored in 0.5% chloramine T at 4 °C for one week. The research protocol was reviewed and approved by the Autonomous Committee of Research Ethics of Galicia (CAEIG 2015/443). The surface of the teeth were cleaned with a periodontal curette and all of them were stored in distilled water at 5ºC until use (4 weeks) [8,26].

Each tooth was decoronated at the cement-enamel junction level using a diamond disc (PM 943.104.100 Ø 10mm 0.15mm L., Komet, Lemgo, Germany) under water cooling and treated consistently by the same operator [27]. Endodontic treatment involved the Proglider and Protaper Next System (X1, X2, X3) (Dentsply Maillefer, Ballaigues, Switzerland), 5% NaOCl (Panreac Química SLU, Barcelona, Spain), Guttacore #30, and AH Plus Jet (Dentsply DeTrey GmbH, Konstanz, Germany). The roots were provisionally sealed with Cavit G (3M Deutschland GmbH, Dental Products, Neuss, Germany). The apical foramen was sealed with dentin adhesive [26], ClearfilTM Universal Bond (Kuraray Noritake Dental Inc, Okayama, Japan), following the manufacturer's instructions. Subsequently, the teeth were stored in distilled water at 37°C for 24 hours to allow complete cement setting (Cultura, Vivacare, diagnostic line, VIVADENT Switzerland) [26,28]. In all teeth, an 11 mm segment of the 1.5 mm diameter Tenax® Fiber White (TFW) glass post (Coltène/Whaledent GmbH, Langenau, Germany) was cemented. The post section is cylindrical in its cervical and middle portions, smooth, and free from grooves or striations. The post space has a cylindrical shape, and the posts were cemented inverted using only their cylindrical portion. The post exhibited the same cross-sectional shape along the entire length of the post space ensuring uniformity. This approach allows for a consistent cross-sectional area along the entire post space, enabling regional comparison of values and minimizing potential biases arising from taper, as investigated in the push-out test [29]. Post space was prepared with water spray coolant at low speed 5000 r.p.m. [30]. Each drill was used to prepare 12 teeth and then discarded. The post space preparation began with Peeso drills 1, 2, 3 (Maillefer Instruments Holding Sàrl, Ballaigues, Switzerland). Subsequently, cylindrical burs of 1.14 mm, 1.25 mm, 1.4 mm (Parapost® Fiber LuxTM (PFL), Coltène/Whaledent AG, Altstätten, Switzerland) were utilized, followed by the use of a conical bur of 1.5 mm (TFW) and finally, a cylindrical bur of 1.5 mm diameter (PFL) (Figure 1).

Figure 1. Burs and drills used to prepare the post space.

The post space was rinsed with water and dried using an intraradicular aspiration system with Roeko Surgitip Endo 0.35 mm cannulas (Coltène/Whaledent AG, Altstätten, Switzerland). Finally, the adaptation of the post was verified using its cylindrical portion up to 11 mm.

The post surfaces were acid-etched using 9.6% hydrofluoric acid (Ultradent Products, Inc., Utah, USA) for 60 seconds. Subsequently, they were rinsed with distilled water to remove any residual hydrofluoric acid and immersed in 96% ethyl alcohol (Laboratorios e Industrias Noriega, S.L., Oviedo, Spain) for 60 seconds to eliminate water remnants on the post surface. Posts were then dried using an oil-free dry air syringe. Prior to cementation, they were silanized (Ultradent Products, Inc., Utah, USA) for 1 minute, followed by application of oil-free dry air using a syringe [31,32].

Cementation and polymerization protocols

The teeth were randomly divided into 12 groups (Table 1).

Table 1. Different groups of study according to irrigation and cementation protocols. 12 teeth in each group (n=12).

	Ortophosforic acid 37% PARABOND A+B PARACORE	Acondicionador Non Rinse PARABOND A+B PARACORE	CLEARFIL UNIVERSAL CLEARFIL DC CORE PLUS
DISTILLED WATER (control)	G1	G2	G3
EDTA 17%	G4	G5	G6
NaOCl5%	G7	G8	G9
CHX 2%	G10	G11	G12

In all groups, 2 ml of irrigant was applied for 1 minute, it was removed using a suction cannula (Roeko Surgitip-endo, Coltene Group, Altsttäten, Sweden), and standardized paper points #60 (Coltène/Whaledent GmbH, Langenau, Germany). The irrigants used were distilled water (Dw) (Groups: 1, 2, 3) as control, CanalPro™ EDTA 17% (EDTA) (Coltène/Whaledent AG, Altstätten, Switzerland) (Groups: 4, 5, 6), NaOCl 5% (NaOCl) (Panreac Química SLU, Barcelona, Spain) (Groups: 7, 8, 9), and chlorhexidine digluconate 2% in distilled water (CHX) (Groups: 10, 11, 12). Adhesives were actively applied [33] following the manufacturer's instructions. The cementation sequence for the groups was randomized until all groups were completed (Table 1). The adhesives techniques employed were as follows:

- Etch-rinse adhesive (Groups: 1, 4, 7, 10): 37% orthophosphoric acid (Oa) (Ivoclar Vivadent AG, Schaan, Liechtenstein) for 15 seconds, rinsing with distilled water (15 s) using a Canal Pro syringe with Slotted-End Tips, 27 ga. (Coltène/Whaledent AG, Altstätten, Switzerland), aspiration of the water in post space with a suction tip (Roeko surgitip-endo 0.35 mm, Coltène/Whaledent AG, Altstätten, Switzerland), and drying using standardized absorbent paper points #60 (Coltène/Whaledent GmbH, Langenau, Germany). The corresponding irrigant was applied and removed as previously explained. Subsequently, the adhesive system was applied: Parabond® Primer A and Primer B (PAB) (Coltène/Whaledent AG, Altstätten, Switzerland) was actively applied to the dentin surface of the post space for 30 seconds. A disposable mixing well (Dentsply DeTrey GmbH, Konstanz, Germany) and a microapplicator (Root Canal Applicator Tip, Dentsply DeTrey GmbH, Konstanz, Germany) were used. After the application time, excess adhesive was removed with standardized absorbent paper points #60 (Coltène/Whaledent GmbH, Langenau, Germany) and air-dried using the syringe for 2 seconds. The combination of paper points and air is effective in removing residual water or solvent from the post space [34]. Cementation: The resin cement ParaCore® (PC) (Coltène/Whaledent AG, Altstätten, Switzerland) was directly applied to the post space using its own dispensing and mixing syringe. The applicator tip was placed at the base to fill the entire receiving space of the post. As the cement was extruded, the tip was withdrawn until it exited the post space.

-Two-step self-etch adhesive (Groups: 2, 5, 8, 11): Application of the irrigant as describe in groups (1, 4, 7,10) followed by the etchant conditioning. It was performed by actively applying ParaBond® Non-Rinse Conditioner (Coltène/Whaledent AG, Altstätten, Switzerland) (PNR) for 30 seconds using a microapplicator (Root Canal Applicator Tip, Dentsply DeTrey GmbH, Konstanz, Germany). After the application time, excess was removed with standardized absorbent paper points #60 (Coltène/Whaledent GmbH, Langenau, Germany) and air-dried using the syringe for 2 seconds. Subsequently, the PAB adhesive and PC cement was applied as describe in groups (1, 4, 7, 10).

-Universal adhesive (Groups: 3, 6, 9, 12): Application of the corresponding irrigant as describe in groups (1, 4, 7, 10) followed by the self-etch adhesive ClearfilTM Universal Bond (CUB) (Kuraray Noritake Dental Inc, Okayama, Japan). It is actively applied to the dentin surface of the post space for 10 seconds using a microapplicator (Root Canal Applicator Tip, Dentsply DeTrey GmbH, Konstanz, Germany). Excess adhesive and solvent were removed with standardized absorbent paper points #60 (Coltène/Whaledent GmbH, Langenau, Germany), and air-dried using the syringe for 5 seconds. Photopolymerization was carried out for 5 seconds at 1600 mW/cm² using the LED unit S.P.E.C. 3 (Coltène/Whaledent GmbH, Langenau, Germany). Cementation: The resin cement ClearfilTM DC Core Plus (CCP) (Kuraray Noritake Dental Inc, Okayama, Japan) is directly applied to the post space using its own dispensing and mixing syringe. The applicator tip was placed at the base to fill the entire receiving space of the post. As the cement was extruded, the tip was withdrawn until it exited the post space.

The chemical polymerization was allowed to proceed for 4 minutes, followed by light activation starting from the coronal portion of the post [15]. This was carried out using the LED curing unit S.P.E.C.3 (Coltène/Whaledent GmbH, Langenau, Germany) with an output power of 1600 mW/cm² for 20 seconds. The curing unit's tip was placed on the remaining part of the Tenax® Fiber White post (Coltène/Whaledent AG, Altstätten, Switzerland) protruding from the root, which remained of consistent length across all teeth. To prevent light from curing the lateral root areas and simulate the oral cavity conditions, a black cardboard shielded the root during photopolymerization was used (Figure 2).

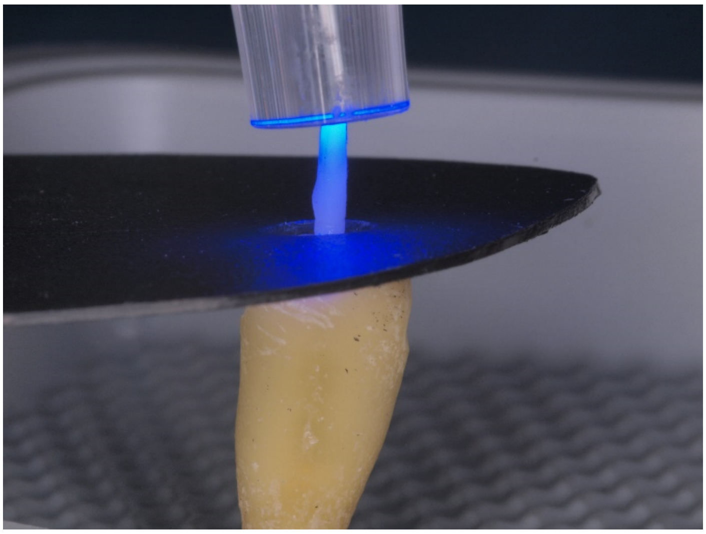


Figure 2. Photopolymerization of the resin cement.

Each tooth was submerged in distilled water and kept in a dark environment for 48 hours at 37 °C to ensure complete cement setting [35].

Push-out test

Teeth were fixed in acrylic resin cylindric blocks (10 mm diameter and 25 length) (Probase Cold Monómero, Ivoclar Vivadent AG, Schaan, Liechtenstein) keeping 0.5 mm of the cervical part of the tooth out of the resin. All teeth were vertically centered with post parallel to the walls checked with a parallelometer (050312, Mestra, Bizkaia, Spain). Ten teeth were randomly selected from each group. Each tooth was sectioned perpendicular to its axis using a precision cutting machine at 1000 rpm (Accutom 5, Struers, Ballerup, Denmark) with water cooling (Figure 3).

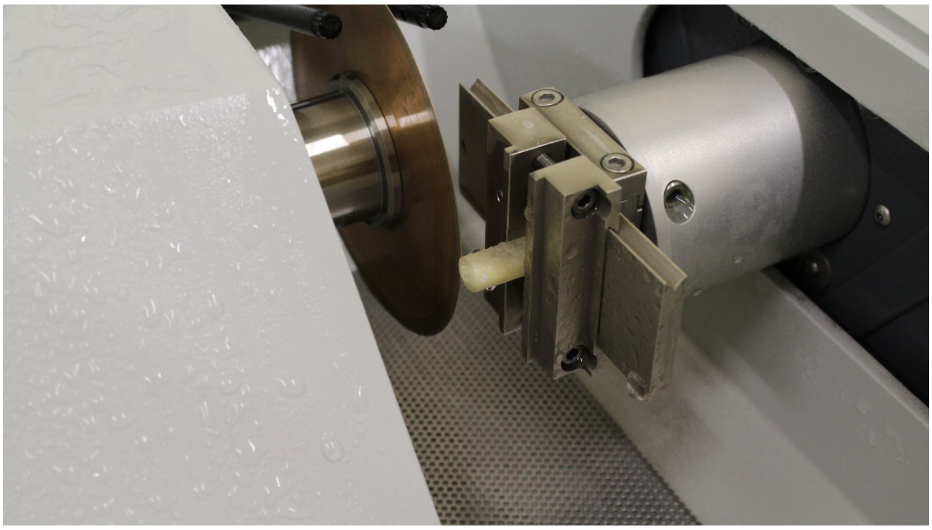


Figure 3. Sectioned perpendicular to the axial axis of the specimen with a precision cutting machine.

Firstly, an initial cut was made to discard the coronal portion of the post at 1000 µm from the cement-enamel junction. Subsequently, three sections of 1100 µm were obtained from each tooth at distances of 1500 µm (coronal), 4500 µm (middle), and 7500 µm (apical) from the coronal portion. Discs that displayed any loss of integrity were excluded from the statistical analysis.

The discs obtained from each group (n=30) were individually placed in the universal testing machine (Autograph®, Model AG-IS, Shimadzu Corporation, Kyoto, Japan). The attachment applying pressure onto the post has a diameter of 0.9 mm. and was positioned centrally on the surface of the post (Figure 4)

 doi:10.20944/preprints202402.1282.v1

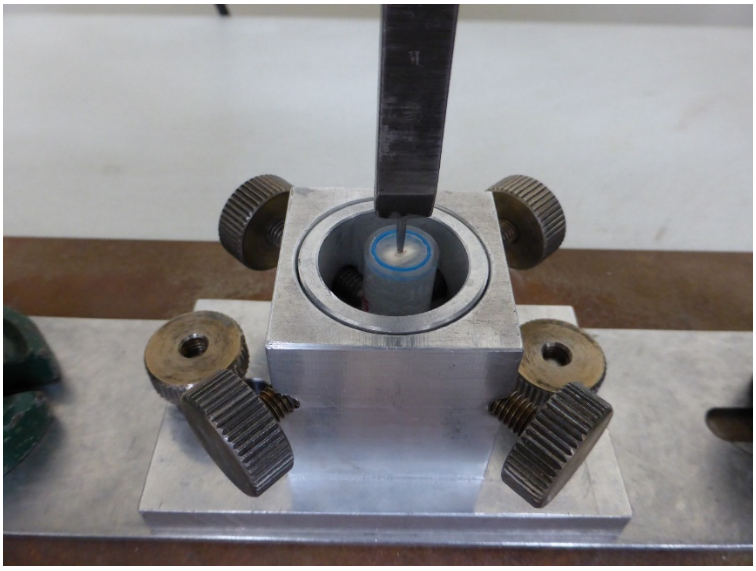


Figure 4. Test Push-Out.

The loads were perpendicular to the post's surface and applied at a crosshead speed of 1 mm/min. The adhesive strength was calculated in MPa by dividing the maximum force required to cause specimen fracture, expressed in Newtons (N), by the adhesive surface area, expressed in square millimetres (mm^2). The lateral bonding surface area of the cylinder was determined using the formula: $Al = 2\pi rh$, where π is a constant (3.1416), "r" is the radius of the post, and "h" is the thickness of the disc [16,36].

3. Results

The mean bond strength values and standard deviations are summarized in Table 2.

Table 2. Mean results with standard deviation for push-out test values in MPa. Mean values with the same letters are not significantly different at P< .01. Superscripts compare from left to right (rows) and subscripts compare from top to bottom (columns). Every Group of Study (n=30 discs).

	Etch&Rinse adhesive Orthofosforic acid 37% Parabond A+B Paracore	*Two steps self-etch adhesive* ParaBond® Non-Rinse Conditioner Parabond A+B Paracore	*Universal adhesive* Clearfil Universal Clearfil DC Core Plus	Total
Distilled Water (control)	G1= $16{,}760^{a}_{a}$±4,43	G2= $16{,}261^{a}_{a}$±5,88	G3= $17{,}067^{a}_{bc}$±4,33	$16{,}696_{ac}$
EDTA 17%	G4= $17{,}069^{a}_{a}$±5,03	G5= $14{,}951^{a}_{a}$±5,14	G6= $16{,}663^{a}_{ac}$±4,84	$16{,}228_{ab}$
NaOCl 5%	G7= $14{,}606^{a}_{a}$±3,81	G8= $14{,}095^{a}_{a}$±4,64	G9= $12{,}919^{a}$±3,79	13,873
CHX 2%	G10= $17{,}304^{a}_{a}$±5,95	G11= $16{,}479^{a}_{a}$±3,04	G12=$16{,}604^{a}_{ab}$±4,58	$16{,}796_{bc}$
Total	**$16{,}435^{a}$**	**$15{,}447^{a}$**	**$15{,}813^{a}$**	**$15{,}898^{a}$**

Regarding adhesive protocols (etch-and-rinse, two-step self-etch, and universal adhesives), no significant differences in bond strength were observed (ANOVA test, $p > 0.01$). However, significant differences were found concerning the irrigants used (ANOVA test, $p < 0.01$). Two-by-two comparisons using the post hoc Tukey test revealed that NaOCl 5% displayed significantly lower adhesion values ($p < 0.01$).

Individual analysis of each irrigant showed no significant differences in adhesion strength (ANOVA, $p > 0.01$). However, individual analysis of each adhesive protocol indicated that the ClearfilTM Universal Bond and ClearfilTM DC Core Plus universal systems, when combined with NaOCl 5%, significantly decreased bond strength (Tukey's test, $p < 0.01$).

No regional differences in bond strength were observed based on root zones or groups (ANOVA, $p > 0.01$) (Table 3).

Table 3. Mean resistance (MPa) in the push-out test for the coronal, middle, and apical regions of the post space.

	Coronal	Media	Apical
(MPa)	16,198	16,333	15,163

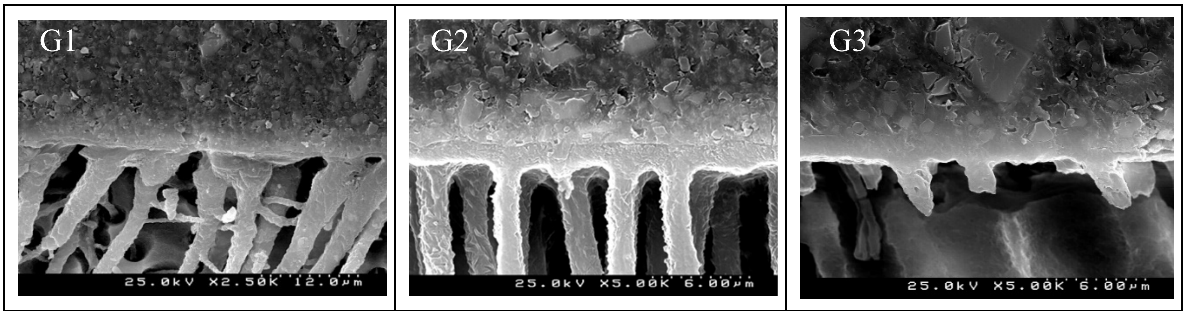
SEM Images

The SEM images of each group are depicted in Figure 5. In the groups with etch-and-rinse adhesive (Oa/irrigant/PAB/PC) (Figure 5. G: 1, 4, 7, 10) or two-step self-etch adhesive (irrigant/PNR/PAB/PC) (Figure 5. G: 2, 5, 8, 11) were observed numerous resin tags with a thickness of 2.5 μm, featuring microporosities and lateral branches (Ø 1.2 μm), and a hybrid layer thickness between 1.5 and 4.5 μm. The hybrid layer is greater in groups treated with EDTA (Figure 5. G: 4, 5) and CHX, especially when combining Oa/CHX 2% (Figure 5. G: 10).

Groups using etch-and-rinse adhesive show a mode II of peri and intertubular demineralization. The resin tags are conical with a diameter at the tubular entry of 4-6 μm (Figure 5. G: 1, 4, 7, 10).

In groups treated with Universal adhesive (irrigant/CUB/CCP), the hybrid layer thickness is 1 μm and 1.5 μm, and the density and length of tags are lower (Figure 5. G: 3, 6, 9). The tags are short, cylindrical, and have few lateral branches except in the group using CHX (Figure 5. G: 12), where they are longer and exhibit greater irregularities on their surface. In the group treated with Universal adhesive and NaOCl, there is a reduction in the number and length of tags, and they do not exhibit lateral branches (Figure 5. G: 9).

In all groups, a basally dense zone appears, and a gradual union between dentin, adhesive, and resin cement is observed without fissures or gaps.



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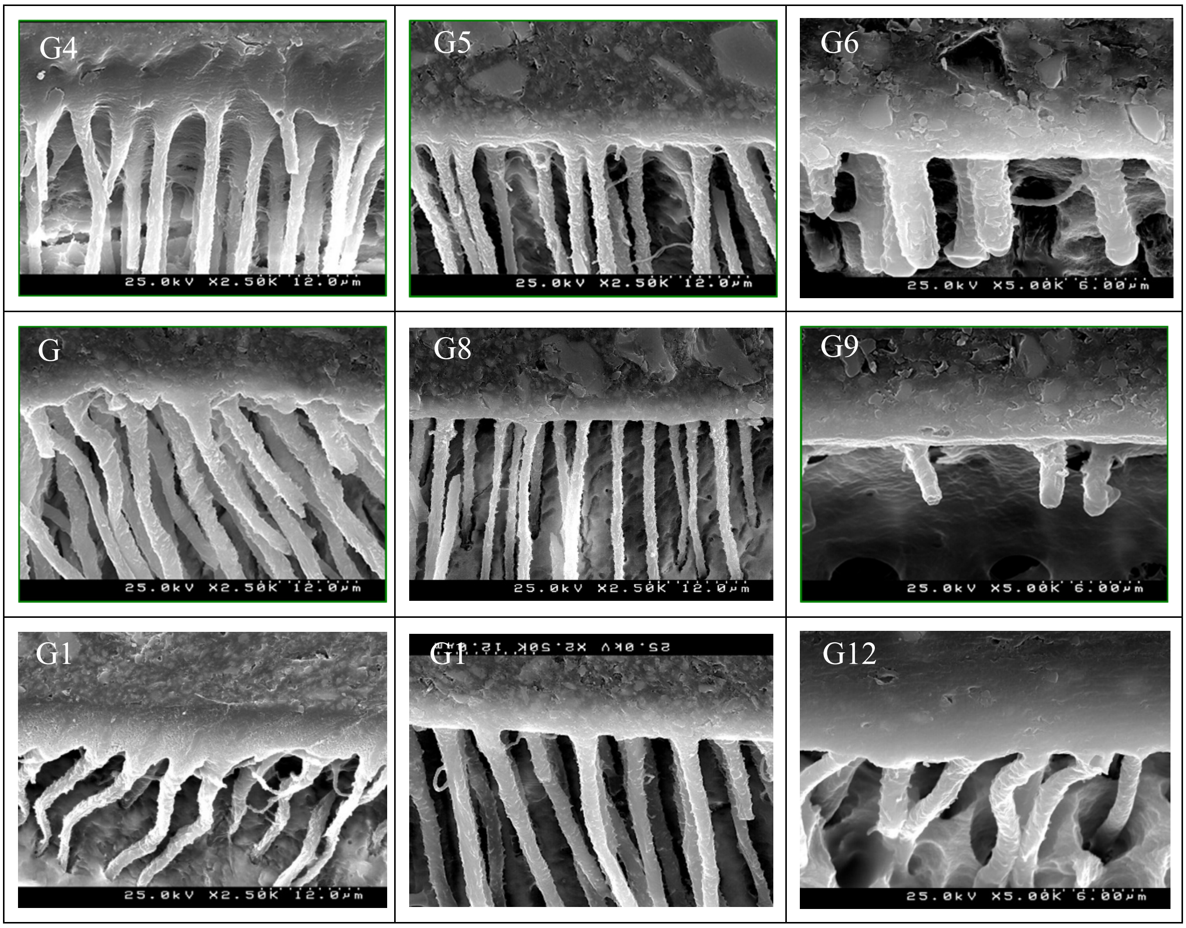

Figure 5. Scanning electron microscope (SEM) photomicrographs showing the cement-adhesive-dentin interface of the different groups: G1: (37% orthophosphoric acid (Oa)/ Distilled Water (Dw)/ Parabond® Primer A and Primer B (PAB)/ resin cement ParaCore® (PC))(Oa/Dw/PAB/PC); G2: (Dw/ ParaBond® Non-Rinse Conditioner (PNR)/PAB/PC); G3: ((Dw/ ClearfilTM Universal Bond (CUB)/ ClearfilTM DC Core Plus (CCP)); G4: (EDTA 17%/Dw/PAB/PC); G5: (EDTA/PNR/PAB/PC); G6: (EDTA/CUB/CCP); G7: (Oa/NaOCl/PAB/PC); G8: (NaOCl/PNR/PAB/PC); G9: (NaOCl/CUB/CCP); G10: (Oa/CHX/PAB/PC); G11: (CHX/PNR/PAB/PC); G12: (CHX/CUB/CCP).

4. Discussion

About *in vitro* studies, there is controversy surrounding the conduct of such studies to analyse the bond strength at the interface between materials and the dental surface. *In vivo* studies replicate the normal state of teeth but there are serious challenges in including enough patients and establishing prolonged follow-up. Variations exist in the type of teeth used, as well as in the directions and speeds of applied forces, making the results not directly comparable [37]. The experimental model we have employed allows us to assess the adhesion resistance in different samples in a standardized, repeatable, and objective manner. Such *in vitro* studies are crucial for evaluating and comparing different variables, as they objectively reveal the behaviour of materials.

To simplify and standardize the determination of adhesive forces the cylindrical portion of Tenax® Fiber White was cemented. The consistent cross-sectional area simplifies and standardizes the determination of adhesive forces allowing for the comparison of adhesion values at a regional level and reducing variations due to friction [29]. Among the various mechanical tests available for assessing adherence, the push-out test is considered a viable alternative to evaluate the post-cement-dentin complex adherence [38]. In comparison to the tensile test, the push-out test conducted in this study is more reliable in measuring adhesive strength since it presents fewer premature failures, specimen preparation is simpler, and it shows an acceptable data distribution variability [39,40]. The force application should be centralized on the post; otherwise, friction with parts of the dentinal walls

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might increase and alter the results. For this reason, the diameter of the device applying force in the push-out test was smaller than the post diameter (1.5 mm) and sufficient to avoid post deformation during load application[36].

To study the morphological characteristics of the resin-dentin interface in three dimensions, a scanning electron microscope (SEM) was employed. SEM allows for the examination of the three-dimensional morphology of the resin-dentin interface. In this study, magnifications of 5,000x were obtained. The main drawbacks of SEM lie in its limitation to observing only the surface of the sample, and the processing for its visualization may introduce artifacts [41].

An advantage for the clinical application of fiber posts would be to combine the cementation of the fiber post inside the root canal with the reconstruction of the core in a single-stage procedure. This would provide a time-saving benefit [26]. Consequently, several manufacturers offer post-core systems. This combination has been described as secondary monoblock [42]. However, a previous study pointed out the potential negative effects of core materials in the adhesive cementation of fiber posts due to their higher filler content [43]. Post & Core systems are available with different dentin conditioning protocols, such as self-etch or etch-and-rinse. Evaluation of both adhesive systems revealed contradictory results. Some studies found no differences [44] while others reported higher adhesive strength in self-etch systems [45]. Other authors observed greater bond strength in the etch-and-rinse adhesive system compared to the self-etch system [46].The failures in homogeneity within the cement layer during the adhesive cementation of fiber posts has been described in the literature [47,48]. The potential beneficial effects of voids within the cement layer have been extensively discussed in the literature [47]. Voids can compensate for the harmful effect of a high C-factor within the post space by reducing tension in the cement. However, air bubbles can substantially weaken the composite [49]. The use of self-mixing tips can reduce the occurrence of these voids [50]. Post-endodontic restorations not performed immediately after completing endodontic treatment allow simultaneous cementation and core build-up. This may result in higher polymerization stress and a reduction in interface strength due to the higher filler percentage required for core reconstruction materials [43]. In this study, the two types of core cements (Paracore® and ClearfilTM DC Core Plus) used have a similar volume and weight of inorganic filler, although it is slightly higher in ClearfilTM DC Core Plus. However, no significant differences were found in adhesive strength regarding the adhesive systems and cements used.

The tags observed in the groups where etch-and-rinse adhesive was used exhibit increased conicity due to mode II demineralization caused by orthophosphoric acid. This acid removes the inorganic tissue inside and around the tubules, especially in the tubules perpendicular to the dentin surface[51]. The length of these tags ranges from 10 to 25 μm, except in those treated with Universal adhesive, which show shorter length, lower density, and a thinner hybrid layer [52]. However, the resistance levels are similar, possibly due to the chemical binding of the MDP monomer to the residual hydroxyapatite linked to collagen fibers and the low solubility of the formed calcium salt [7,9,52–62].

In this study, a greater thickness of hybrid dentin does not provide increased retention [63]. This might be because a thicker layer of demineralized collagen could reduce resin infiltration, decreasing tensile strength and its elastic modulus [64–67].

SEM images reveal more pronounced microporosities and irregularities, mainly in the groups using the etch-and-rinse adhesive system (Figure 5: G1, 4, 7, 10) and two-step self-etch adhesive (Figure 5: 2, 5, 8, 11). These irregularities might be due to increased dentin demineralization and more irregular priming by Parabond® [22]. In all groups, a basally dense area appears. Some studies suggest it might correspond to hydroxyapatite crystals around partially demineralized collagen fibers, resin-infiltrated, providing greater bond strength and durability [68–70]. It might correspond to hybrid dentin formation, associated with the presence of glycosaminoglycans on the surface of tags, due to their combination with the peritubular lamina limitans [68,71].

In this study, no significant differences were found in the bond strength between the etch-and-rinse, two-step self-etch, or universal adhesive groups, confirming the second null hypothesis. This is likely because the evaluation took place 48 hours after cementation, and adhesive surface degradation had not occurred yet.

No significant differences were found concerning the zones of the post space (Table 3), in line with other studies [72–74], thus confirming the third null hypothesis. This could be attributed to proper debris removal [75,76], control of intra-canal moisture using suction tips [77] and active application of the adhesive that facilitates the diffusion of acid monomers and reduces adhesive layer degradation [33,76,78–81]. It depends on careful execution of adhesive protocol [63]. Other factors favoring apical adhesion in this study include the use of self-mixing tips to minimize bubble formation [26] and allowing sufficient working time to prevent premature setting of adhesives and cements.

In this study, 5% NaOCl significantly affects adhesive bonding, particularly ClearfilTM Universal Bond, thus not confirming the first null hypothesis. 5% NaOCl alters the micromechanical interaction between adhesives and dentin by reducing calcium and phosphorus content, elastic modulus, microhardness, and flexural strength [82]. When collagen layer is dissolved by NaOCl, the oxygen released interfere with resisn polimeration [83]. The use of antioxidant/reducing agents could reverse the oxidative effects of NaOCl and facilitate adhesive monomer polymerization [84,85]. Several studies indicate that dentin type, application time and NaOCl concentration influence self-etch adhesive bonding [16,86]. Active application of self-etch adhesives after NaOCl irrigation [33] and delayed dental restoration might be indicated to enhance adhesive strength [87].

In this study, no significant differences in bond strength were observed between 17% EDTA, 2% CHX, and distilled water, consistent with findings in other studies [19,27,88,89]. This variability could stem from the pH effects, application form, duration, and concentration of EDTA, which might explain the different outcomes in several studies [21]. EDTA solution has relatively low surface tension, which could enhance dentin wetting and improve adhesion [90]. Its mechanism involves demineralization by carboxylic groups, removing the mineral part of dentin without denaturing the collagen layer, allowing residual apatite crystals to chemically bond with adhesive functional monomers, particularly self-etch adhesives [91]. This leads to a thinner smear layer and hybrid layer without extensive collagen denaturation, potentially increasing adhesive interface strength [27]. In this study, SEM images reveal an increased hybrid layer thickness that doesn’t correspond to higher adhesion resistance. With prolonged exposure time, there’s an increase in dentinal tubule opening and the depth of demineralization within [21]. A shorter EDTA exposure time might create a thinner hybrid layer and greater adhesion resistance. Although EDTA is not a specific quelator of calcium, it sufficiently acts as a metalloenzyme or metalloproteinase inhibitor, abolishing their catalytic activity, inactivating them, and inhibiting reactions catalyzed by these enzymes [21].

In this study, CHX does not exhibit significantly higher adhesion values compared to distilled water (Dw) or EDTA. However, it does show high absolute levels of resistance [25], particularly in the group using 37% orthophosphoric acid, consistent with other studies [23,92–95], while also displaying a thicker hybrid layer. This might be attributed to CHX's significant substantivity, enabling it to bind electrostatically and reversibly to mineralized and, notably, demineralized dentin [22]. CHX increases dentin surface energy [92,96,97], thereby enhancing resin tubular infiltration [98,99] and reducing collagen degradation within the first 24 hours [100,101]. In comparison to NaOCl, it achieves higher adhesive forces, possibly due to its non-oxidizing nature [82,102]. Its action is dose dependent [11] and diminishes in the presence of calcium chloride, suggesting that its chelating properties may be crucial in improving adhesion by inactivating MMPs [95,103].

5. Conclusions

Within the limitations of the present study, it is concluded that 5% NaOCl for 1 minute significantly reduces adhesive strength compared to the other irrigants and Clearfil™ Universal Bond. There are no significant differences in adhesion among the three adhesive procedures studied or among the coronal, middle, and apical regions of the prepared canal.

Author Contributions: Conceptualization, Benjamín Martín Biedma, João Carlos Ramos, Sandra García Varela, Pablo Castelo Baz; methodology, Sandra García Varela, Benjamín Martín Biedma, João Carlos Ramos; software, María José Ginzo Villamayor, Sandra García Varela, Pablo Castelo Baz; validation, Benjamín Martín Biedma, João Carlos Ramos, Sandra García Varela, Pablo Castelo Baz; formal analysis, María José Ginzo Villamayor, Sandra García Varela; investigation, Sandra García Varela, João Carlos Ramos, Benjamín Martín Biedma; resources, Sandra García Varela, Tania Gancedo Gancedo, Ramón Mendez, Marcos Anibal Anache D´abate; data curation, Sandra García Varela, María José Ginzo Villamayor; writing—original draft preparation, Sandra García Varela, María José Ginzo Villamayor, Pablo Castelo Baz; writing—review and editing, Sandra García Varela, María José Ginzo Villamayor, Pablo Castelo Baz, Manuel Ruíz Piñón, Soledad Mareque Bueno; visualization, Sandra García Varela, Pablo Castelo Baz; supervision, Sandra García Varela, Benjamín Martín Biedma, João Carlos Ramos; project administration, Sandra García Varela, João Carlos Ramos, Benjamín Martín Biedma; funding acquisition, Sandra García Varela, João Carlos Ramos, Benjamín Martín Biedma. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: The study was conducted according to the guidelines of the Declaration of Helsinki and approved by the Autonomous Committee of Research Ethics of Galicia (CAEIG 2015/443).

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Acknowledgments: The authors express gratitude to Dr. Jose M. Ferreira (Aveiro) and Amparo Otero (UICEC, A Coruña) for their technical and administrative support.

Conflicts of Interest: The authors declare no conflict of interest.

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