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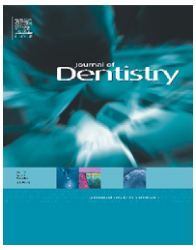

Bond strengths, degree of conversion of the cement and molecular structure of the adhesive–dentine joint in fibre post restorations

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ABSTRACT

Objectives: Because fibre post restorations are influenced by multiple factors such as the types of bonding materials, the dentine region and the time under moist exposure, this study sought to determine the bond strength of endodontic restorations and its relation to the degree of conversion of the cement layer and the molecular structure of the dentine-bonded joints.

Methods: The performance of 2 etch-and-rinse (All-Bond 2 and One-Step Plus) and 2 self-etch (Clearfil SE Bond and Xeno III) adhesives at post spaces regions, after 7 d or 4 m, was evaluated. FRC Postec Plus posts were cemented to the root canal with a dual-cure resin cement (Duo-Link). Transverse sections of the tooth were subjected to push-out testing, to degree-of-conversion measurements and to hybrid layer evaluation through μ-Raman spectroscopy.

Results: Coronal bonding was higher than cervical and middle bonding. The hybrid layer was thicker for the etch-and-rinse systems, with thicknesses decreasing towards the middle region. The degree of conversion measured for the 3-step etch-and-rinse group after 4 m was significantly higher than that for the self-etching groups.

Conclusions: Although not totally stable at the adhesive–dentine interface, the 3-step etch-and-rinse adhesive in the coronal dentine provided the best bond strength, degree of conversion of the cement and hybrid layer thickness in post restorations, in both short- and long-term analyses.

1. Introduction

Whilst the use of posts of varying design are of demonstrated success in restoring endodontically treated teeth, the introduction of polymer-like post materials, that can bond to dentine, has special application for restoring functionality when the tooth has been severely damaged by caries, trauma, a congenital disorder or internal resorption.[1–3] Prefabricated fibre posts made of a transparent epoxy-resin matrix can be used with chemical-, light- and dual-cure luting agents, providing a low-modulus restoration that is analogous to the soft dentine layer and may help to prevent cracks propagating into the dentine.[4,5]

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The low light irradiance[6–8] and the difficulty of moisture control[9] at the deepest levels of the root preparation are amongst the issues that complicate the cement cure in post restorations. Moreover, ultrastructural observations have confirmed the presence of a superficial interaction between the acidic resin monomers of the oxygen inhibition layer of certain adhesives and the resin cement initiators, decreasing the cement curing.[8] The acidic monomers are hydrophilic and can also create water channels across the adhesive layer, disturbing cement polymerisation and hydrolysing the adhesive–dentine interface.[9–11]

More recently, Raman spectroscopy has been used to assess the degree of conversion of methacrylate resins in the initial stages of cure.[12] Hardness tests are indirect forms of measuring the depth of cure of cements in post spaces,[6,13] but they are ineffective for measurements beyond 10 mm,[6] where Raman spectroscopy seems to indicate that a sharp contact indenter will not be needed. If the factors contributing to failure at the adhesive/dentine interface are to be effectively isolated and understood, an understanding of the chemical structure of these surfaces is required. Raman spectroscopy with lateral spatial resolution less than 2 μm has also been used to study the adhesive dentine interface at a molecular level,[14] and to the current authors' knowledge, there has not been an investigation into the chemical differences of the interface in adhesive endodontic restorations.

As for bond strength tests, they do not allow for direct identification of the hybrid layer components, as well as the degrading components that will appear over time. Nevertheless, strength tests, namely, the push-out test, along with morphologic evaluations have been largely used for the study of bonded endodontic restorations.[15–19]

Since the primary function of the post is to provide retention, it is important to address separately the events that affect the adhesion of fibre post restorations and to know how they act together for overall performance. Accordingly, the purpose of this current work was to study the mechanical properties of bonded endodontic restorations, considering the degree of conversion of the cement layer and the molecular structure of the adhesive/hybrid layer dentine interface. Our hypothesis was that the bond strength, the cement cure and the hybrid layer formation would be superior for the etch-and-rinse systems in the crown region. To test this hypothesis, we used push-out bond strength tests and μ-Raman spectroscopy, as a function of some of the most important variables that can compromise bonding according to previous literature: the type of bonding agent,[18] the tooth region[19] and the time in aqueous storage.[20,21]

2. Materials and methods

The current study was conducted on 80 human maxillary incisors and canines obtained from the Human Tooth Bank of the Department of Odontology at Taubaté University at Taubaté, Brazil. The tooth bank procedure includes cleaning the teeth with periodontal curettes and storing them in distilled water (−4 °C). Selection of specimens was based on teeth with straight root canals and without caries or root resorption. Teeth of similar length were also given preference.

The teeth were randomly divided into 8 groups (n = 10): (a) Group 1—treated with the 3-step total dentine etching adhesive system, All-Bond 2 (BISCO, Schaumburg, IL, USA); (b) Group 2—treated with the 2-step total etch dentine adhesive system, One-Step Plus (BISCO, Schaumburg, IL, USA); (c) Group 3—treated with the 2-step self-etching adhesive system, Clearfil SE Bond (Kuraray Medical Inc., Kurashiki, Okayama, Japan); and (d) Group 4—treated with the 1-step self-etch adhesive system, Xeno III (DeTrey Dentsply, Konstanz, Germany). Groups 1–4 were treated and evaluated after 7 d of storage in artificial saliva at 37 °C. Groups 5, 6, 7 and 8 were respectively treated with the same adhesives and analysed after 4 months of storage in artificial saliva. The chemical composition of the adhesives is presented in Table 1.

The crown of each tooth was removed 4 mm coronal to the cement–enamel junction (CEJ), perpendicular to the long axis of the tooth on the buccal aspect, by means of a water-cooled diamond saw at low speed (Saw, South Bay Technology, San Clemente, CA, USA). The pulp was then removed with a no. 15 K file (Dentsply Maillefer; Ballaigues, Switzerland). The root canal was widened up to 4 mm short of the apex with no. 15, 20, 25, and 30 files (Dentsply Maillefer; Ballaigues, Switzerland) followed by a no. 2 Largo bur (Dentsply Maillefer; Ballaigues, Switzerland). At each change of instrument, the root canal was thoroughly irrigated with 0.5% NaOCl, and suction was performed. To receive the posts, the roots were prepared with a no. 3 bur of the FRC Postec post system (Ivoclar Vivadent, Schaan, Liechtenstein). Each root was positioned in the centre of a silicone mould (3 mm × 3 mm ×3 mm), and the surrounding space was filled with clear, chemically cured acrylic resin (Jet, Artigos Odontológicos Clássico, São Paulo, SP, Brazil). To allow the tooth long axis to be as perpendicular as possible to the ground, embedding was performed with the no. 3 bur of the post system inside the root canal, with its upper part connected to a surveyor (Bio Art Equipamentos Odontológicos; São Carlos, SP, Brazil). After the storage period, all of these procedures allowed the specimens to be cut into transverse segments where the adhesive interface format was approximately that of the frustum of a cone.

Before cementation, the external lateral walls of the teeth received a coat of black nail varnish to allow for passage of light only through the most coronal portion, since the root is clinically covered by periodontal tissues. The materials were applied following the manufacturers' instructions (Table 1). Two layers of the bonding resin of each adhesive were applied to dentine and light-cured for 10 s. The posts were luted with Duo-Link cement, prepared by mixing equal parts of base and catalyst for 10 s until a homogeneous colour was achieved. The cement was then inserted into the root canal using a no. 40 Lentulo bur (Dentsply Maillefer; Ballaigues, Switzerland), and the post was placed into position. With the intent to seal the coronal entrance, until the storage period passed the excess cement was left on top. Each tooth was light-cured for 40 s (Optilux 501-SDS Kerr, Danbury, CT, USA) at a light intensity of 650 mW/cm^2. The embedded teeth were attached to a metallic base. The metallic base was connected to a sectioning machine, and the teeth were sectioned perpendicular to their long axis with a diamond saw (650 Diamond Saw, South Bay Technology, San Clemente, CA, USA) under water irrigation. The first 0.5-mm section was discarded because the

Table 1 – Chemical compositions, batch numbers and bonding procedures of the adhesive systems tested.

Adhesive	Component	Batch #	Composition	Application protocol
All-Bond 2 (3-step etch and rinse)	Etchant (UniEtch)		32% Phosphoric acid	Etch[a] dentine for 15 s. Rinse[a] and dry[b].
	Primer A	0600004826	Acetone, ethanol, Na-N-tolylglycine glycidyl methacrylate	Dispense and mix equal amounts of Primer A and B. Apply[c] and air dry thoroughly.
	Primer B	0600003705	Acetone, ethanol, Biphenyl dimethacrylate	
	Pre-Bond Resin	0600004127	Bisphenol A diglycidylmethacrylate Triethyleneglycol dimethacrylate Benzoyl peroxide	Mix equal numbers of drops of Pre-bond™ and Dentine/ Enamel Bonding Resin. Apply[c] and light-cure for 20 s.
	Dentine/Enamel Bonding Resin	0600004127	Bisphenol A diglycidylmethacrylate Urethane dimethacrylate, HEMA	
One-step Plus (2-step etch and rinse)	Etchant (Uni-Etch)	–	32% Phosphoric acid	Etch[a] dentine for 15 s. Rinse[a] and dry[b].
	Adhesive	0500005247	Biphenyl dimethacrylate, HEMA, acetone, glass	Apply[c] and light-cure.
Clearfil SE Bond (2-step self-etch)	Self-etching primer	00727A	HEMA, hydrophilic dimethacrylate, 10-MDP, toluidine, camphorquinone, water	Apply[c] Primer. Leave undisturbed for 20 s.
	Adhesive	01044A	BisGMA, Silanated silica, HEMA, hydrophilic dimethacrylate 10-MDP, toluidine, camphorquinone	Apply[c] Bond. Light-cure.
Xeno III (1-step self-etch; two-component system)	Liquid A	0605000261	HEMA, purified water, ethanol, 2,6-Di-tert-butyl-p hydroxytoluene, nanofiller	Mix liquids A and B. Apply[c] and leave undisturbed for 20 s. Airthin. Light-cure.
	Liquid B	0605000261	Pyro-EMA, PEM-F, UDMA, BHT, Camphorquinone, EPD	

[a] Etching and water-rinsing were performed with long-tipped syringes.
[b] Drying was performed with three absorbent paper points (# 60).
[c] All adhesive applications were made with a root canal microbrush. Excess was always removed with a new microbrush.

excess cement could lead to overestimation of the bond strength values. Overall, 6 sections, measuring nearly 1.5 mm in thickness, were prepared, with 2 from each study region (coronal; cervical/middle regions of the root).

Three segments (1 per region) of each tooth were randomly selected for the push-out test. The segment was positioned on a metallic device with a central opening larger than the root canal diameter. The most coronal portion was always placed facing downward in relation to the load tip (apical-coronal load). The tip, a metallic cylinder with a diameter of 0.85 mm at the end, was pressed onto the post centre in an attempt to avoid touching the dentine. The test was performed in a servo-hydraulic machine (Instron 8872, Instron, Canton, MA, USA) at a crosshead speed of 1 mm/min with a load cell of 50 kgf. It should be noted that the calculation of the interface area was performed with the formula for calculating the lateral area of a right cone frustum. The radius (r) was obtained by measurement of the internal diameters of the bases corresponding to the internal diameter of the root canal walls in the segment. The load for fracture was attained in kgf, and the bond strength was calculated in MPa.

The 3 remaining segments were used to determine the degree of conversion of the cement as well as for the analysis of the adhesive penetration. Before the spectra were taken, the segments were smoothed for 1 min with 4000-grit SiC paper and swabbed with 5% NaOCl to remove the slurry. An argon ion laser ~514.5 nm served as the excitation source. To prevent chemical reactions or dehydration during the experiments, the laser output power was controlled to less than 5 mW. The μ-Raman system consisted of holographic optics, a single ~1800 groove/mm grating, 0.5-m spectrometer and a liquid nitrogen cooled CCD detector (11 003 330 pixels). The spectrometer was regularly calibrated according to the neon emission spectrum. The precision of the measured frequency was better than 2 cm^{-1}. The spectrum was obtained from the frequency range of 1200–1900 cm^{-1} with 3 scans. The rate of unreacted carbon–carbon double bonds (% C=C) was determined from the ratio of absorbance intensities of aliphatic C=C (peak height at 1637 cm^{-1}) against an internal standard before and after the specimen was cured. The aromatic carbon–carbon bond (peak height at 1608 cm^{-1}) absorbance was used as an internal standard. The degree of conversion was determined by subtracting the % C=C from 100%. The analyses were carried out in 5 sections of every region, values averaged and mean values fitted to a combination of Lorentzian and Gaussian modes using Origin software (OriginLab, Northampton, MA, USA).

Linear spectra were taken at 1–1.5 μm intervals starting at the cement layer and going along lines across the dentine–adhesive interface at random sites within the intertubular dentine of each section region. The scanning was done once at each section, corresponding to 1 region from a total of 3 teeth per group.

The Raman spectra of the bonding agents occurred at 1720 cm^{-1} (carbonyl), 1609 cm^{-1} (phenyl C···C), 1454 cm^{-1} (C–H deformation), 1185 (dimetil-gem), and 1111–1118 cm^{-1} (C–O–C).

The major features related to dentine collagen were 1238–1245 cm^{-1} (Amide III, NH_2 deformation, random coil), 1273–1280 cm^{-1} (Amide III, NH_2 deformation, alpha-helix), 1453 cm^{-1} (C–H deformation), and 1660–1667 cm^{-1} (Amide I, carbonyl stretching). The mineral content was identified by the features 961 cm^{-1} (phosphate) and 1072 cm^{-1} (carbonate).

The treatments of sloping and curved backgrounds were performed using the wavelet transform technique, mainly due to the low signal-to-noise ratio in the sample spectra. For this purpose, the spectra were decomposed using the db4 Daubechies wavelet with 8 decomposition levels.[22] Baseline correction was achieved by replacing the approximation coefficients at the last decomposition level with zero. In addition, 'denoising' was carried out by means of the universal method for threshold selection.[23]

Initial raw spectra for representative samples of each bonding agent and region were also collected from an integrated fully automated confocal Raman imaging system (LabRam ARAMIS, Horiba Jobin Yvon Inc., Edison, NJ, USA). These spectra served as a reference against which all subsequent spectra were compared because the former were less influenced by background noise, receiving no further treatment.

Non-tested specimens were demineralised with phosphoric acid for 5 min and deproteinised with NaOCl 5% and examined for tag formation. The fracture modes of the tested specimens were also examined. All specimens were desiccated for 24 h and gold-sputtered under vacuum (10^{-6} Torr). The analyses were done in a Philips XL 30 SEM (Eindhoven, The Netherlands) under vacuum (2×10^{-5} mbar).

Minitab 15.1 (Minitab Inc., State College, PA, USA), Statistica (StatSoft, Tulsa, OK, USA) and Statistix 8.0 (Analytical Software, Tallahassee, FL, USA) were used for statistical analysis. Statistical assumptions were considered in the conducting of a normality test, and the models followed a normal distribution. In addition to the descriptive statistics, parametric inferential statistics were conducted with repeated-measures ANOVA (RM ANOVA), Tukey's adjustment test and paired Student's t test. Pearson's correlation was used to study the degree of linear relationship between bond strength and degree of conversion. p values less than 0.05 were considered to be statistically significant in all tests.

3. Results

The bond strength means and standard deviations are displayed in Fig. 1. Only the ''region'' had a significant influence on the bond strength (3-way RM ANOVA, $p < 0.05$). Statistically significant differences were found between the coronal region (4.2 ± 1.9 MPa) and the cervical (3.3 ± 2.2 MPa) and middle (3.4 ± 1.8 MPa) regions, which were not statistically different from one another.

The main effects, as well as the interaction between ''adhesive'' and ''time'', had a significant influence on the degree of conversion of the cements. Homogeneous groups were established with Tukey's test, showing that the degree of conversion of All-Bond 2 after 4 m was significantly higher than that of Clearfil SE Bond after 7 d and Xeno III after both times (Table 2).

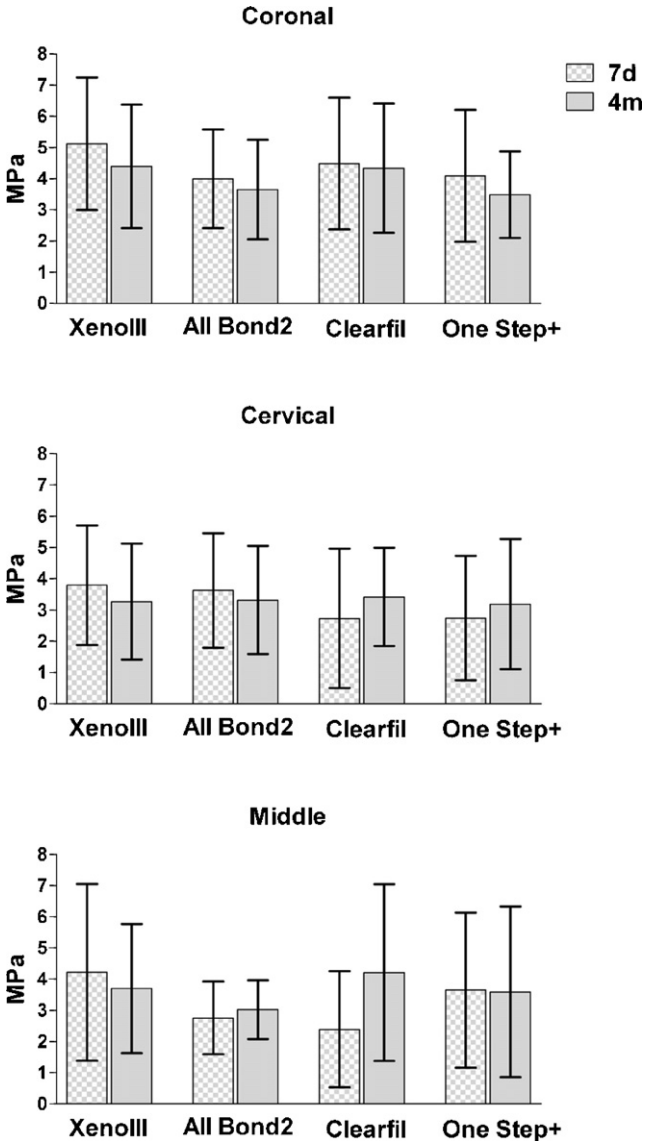

Fig. 1 – Bond strength obtained with the adhesive systems as a function of tooth region and storage time.

The separate effect of the region was studied with the paired Student t test followed by Bonferroni's correction ($p < 0.05$). The data for all regions were significantly different ($p = 0.017$), being coronal > cervical > middle.

Table 2 – Means[a] (%) of the degree of conversion as a function of the adhesive system and time. (Numbers in parentheses are standard deviations.).

Adhesive	Time	
	7 d	4 m
All-Bond 2	52 (13.9^{AB})	72 (12.1^{A})
Xeno III	26 (23.0^{C})	46 (19.0^{BC})
Clearfil SE Bond	38 (20.0^{BC})	51 (7.6^{AB})
One-step Plus	53 (10.0^{AB})	59 (2.5^{AB})

[a] Different letters mean statistically significant differences.

Weak correlation coefficients (Pearson, $r < 0.5$) with no statistically significant differences ($p < 0.05$) resulted in no linear dependence between the bond strength and the degree of conversion data.

The All-Bond 2 specimens showed a pattern of demineralisation and adhesive penetration that decreased towards the middle region. At times, the bonding agent did not penetrate the whole extension of the demineralised dentine of the deepest level (Fig. 2A and B).

Generally, the adhesive penetration decreased along the root canal for all systems. Clearfil SE Bond presented only a superficial interaction with dentine (ca. 1–2 μm), which remained roughly the same throughout the regions. The mildest interaction with dentine resulted in increased amounts of bonding agent coming into contact with the cement layer, forming a combined layer of increased packing of C–O–C density at 1111 cm^{-1}.

After 4 m, the All-Bond 2 bands appeared less intense, and an enlargement of the features between 1600 and 1700 cm^{-1} at about 5 μm of coronal and cervical regions was observed. Similarly, One-Step Plus presented weaker signals after 4 m, with evidence for collagen exposition.

Clearfil SE Bond was the most stable adhesive in terms of the intensity of its bands as well as the extension of its penetration into the dentine. As for Xeno III, the elution of the bonding agent led to collagen exposition after 4 m.

The tag formation characteristics according to bonding agent are displayed in Fig. 3A, B, C and D.

At 4 m, some sections of One-Step Plus presented signs suggestive of degradation (not shown).

The fracture analysis was based on the main features observed under SEM of representative specimens. Overall, after 7 d of storage, all the bonded interfaces were extruded (cement debonded from tooth and post debonded from cement), including the post fibres (Fig. 3E and F). At 4 m, the hybrid layer/cement interface was disrupted with hardly any harm to the post-cement interface and the post fibres (Fig. 3G and H).

4. Discussion

The literature about fibre post restorations is vast, mainly due to the various issues surrounding them. In this study, we aimed to investigate how some of those issues are related and which are preponderant for retention. The findings suggest that the dentine–adhesive joint is definitely the weakest link of such restorations.[20]

The bond strength differences in the crown and cervical/middle parts of the post space suggest that the middle hybridisation of the post space is negligible for overall retention. The bond strength superiority of the coronal region in the present study is in accordance with reports from other studies concerning adhesive endodontic restorations.[13,19,24–26] In that regard, several factors contribute to improved retention in the crown, from the efficiency of root canal brushes for spreading the adhesive, to moisture control and structural differences such as tubuli numbers.[9,27]

The reason we did not use a root filling material was that removal of the filling is critical. Thus, canal obturation was not performed because it could lead to greater variability in the bond strength results and not show the adhesion between the adhesive and the dentine, which was the objective of the present study. Plus, a filling like gutta-percha complicates the Raman readings by increasing the sample fluorescence.

The degradation processes depend mostly on the diffusion rate that can be accelerated by working with tiny specimens. Fewer than 90 d of storage were necessary to degrade microtensile specimens.[28] The current findings showed no differences in the bond strengths of the 7-day and 4-month specimens, which may have occurred due to the storage of the intact roots as opposed to thin sections. This method was preferred because the deepest parts of the post space are not in direct contact with saliva.

Although no statistical differences regarding the type of bonding agent and the time for analysis were observed, the SEM micrographs showed differences in tag formation, hybrid layer appearance and fracture characteristics. The tag formation looked more intricate, with the presence of lateral branches,[27] for the etch-and-rinse systems than for the self-etching systems. Previous investigators found that, under push-out testing, failures occurred predominantly at the cement/dentine interface, which was corroborated by the present results.[9,24,25] However, an important observation was that the fracture analysis showed that the hybrid layer became more vulnerable after 4-month storage in water, as the 7-day specimens generally showed disruption of all bonded interfaces and extrusion of the post fibres.

In addition to the differences in hybrid layer appearances showed by SEM, the hybrid layer quality through μ-Raman spectroscopy varied depending on the bonding agent. These findings did not seem to have influenced the bond strengths, reinforcing the idea that the relationship amongst the bonding agent wettability, the hybrid layer and the bond strength is not obvious.[29] After 7 d, the hybrid layer seemed intact, with thicknesses that decreased towards the middle region of the post space. The amount of bonding agent that infiltrated dentine was higher for the etch-and-rinse systems. After the 4-month storage, a decrease of BisGMA bands in all groups, followed by an increase of collagen bands in the first microns, suggested collagen exposition as a result of polymer hydrolysis/monomer elution. The collagen features were mainly Amide I disordered collagen with broader small peaks at 1626, 1650 and 1680 cm^{-1} (normal sharp collagen bands occur at 1663–1667 cm^{-1}).

The extrinsic and intrinsic water[30] in unprotected collagen zones causes fibril loosening due to the loss of collagen helicity.[31] This usually happens with the use of simplified bonding agents because of their hydrophilic nature.[9] Water is also responsible for activating MMPs, which are enzymes present in the collagen matrix that may degrade collagen in the long term.[32] Therefore, in the present study, the micrographs and spectra of 4-month One-step Plus and Xeno III presented signs of degradation, which was plausibly triggered by hydrophilic components such as HEMA.[33,34] The degradation was also extended to the cement close to dentine, where cracks were formed probably due to water sorption.[35]

The spectra showed that the mineral removal caused by Clearfil SE Bond was mild and its penetration into dentine was limited when compared to that of the etch-and-rinse bonding

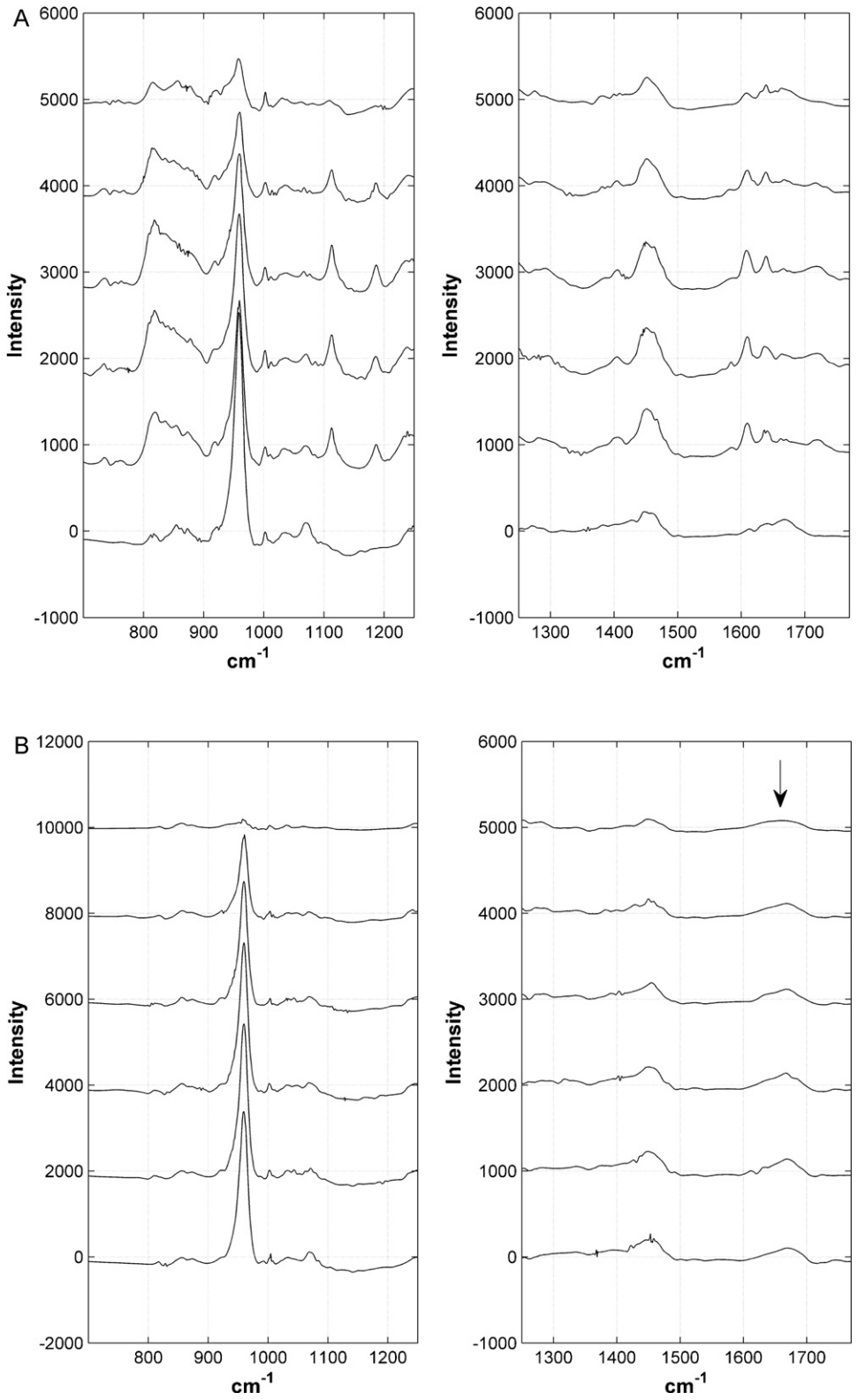

Fig. 2 – The differences in the penetration of All-Bond 2 after 7 d are discernible between the coronal region (A) and the middle part of the root canal preparation (B). The heights of the bands at 1610 (aromatic ring) and 1640 cm^{-1} (vinyl) are similar (A). In contrast with the sharp bands at 1663 cm^{-1} (Amide I) at the coronal level, broad bands of disordered collagen in the middle section (arrow) probably resulted from the lack of adhesive penetration in the demineralised root dentine.

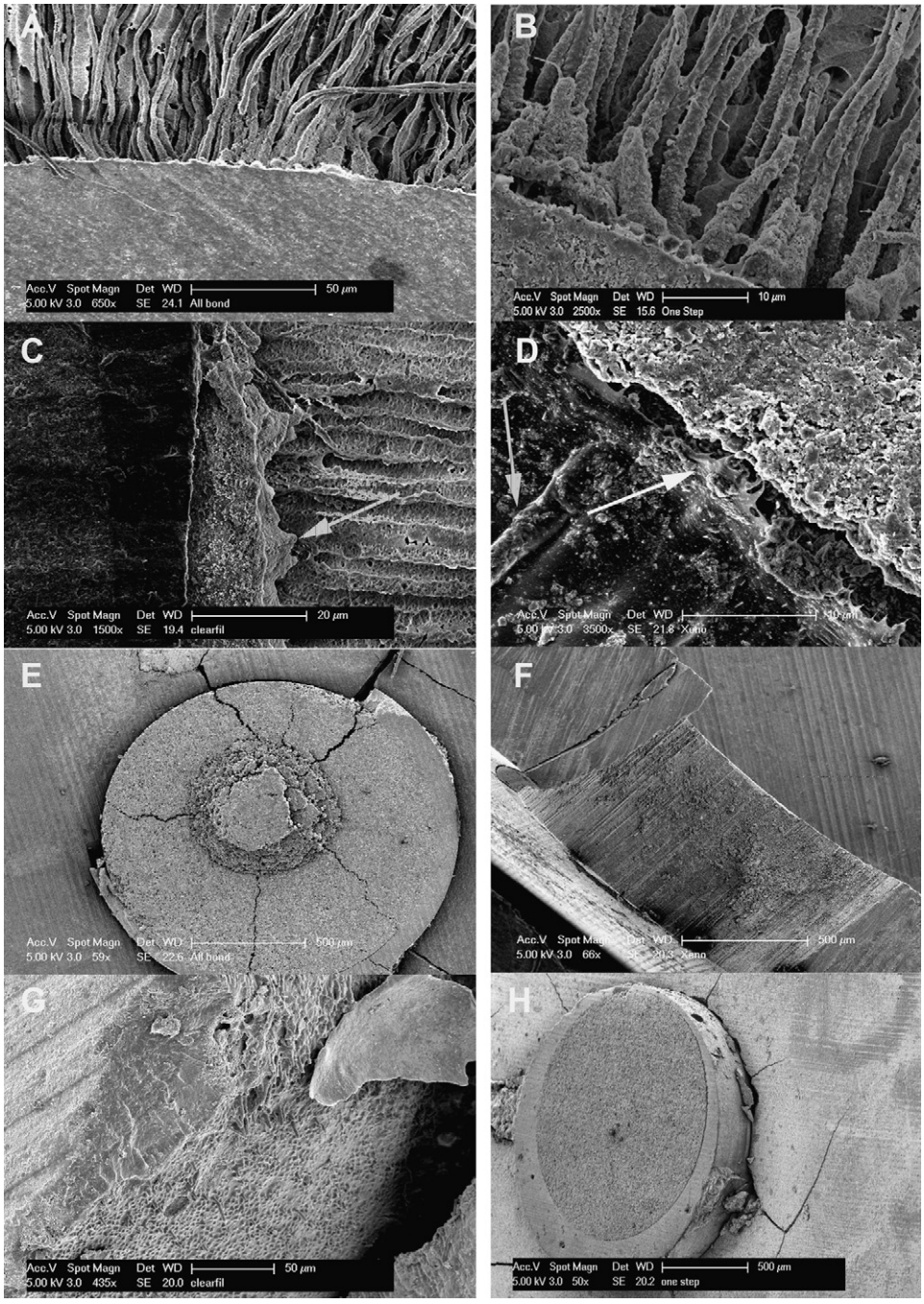

Fig. 3 – Interfaces formed by All-Bond 2 (A), One-Step Plus (B), Clearfil SE Bond (C) and Xeno III (D), at the cervical level. The grey arrows (C and D) show the tags, and the white arrow in D depicts the fragile appearance of the hybrid layer formed with Xeno III. Fractured cervical sections of All-Bond 2 (E) and Xeno III (F) after 7 days' storage. Extrusion of the bonded restoration and/or extrusion of the post fibres is depicted. The 4-month fractured coronal sections of Clearfil SE Bond (G) and One-Step Plus (H) show that the fractures occurred mainly within the hybrid layer, with fracture of the tags.

agents. Over the months, however, the intensity of the bands and the extension of the adhesive penetration did not change as in the other materials, which may have been influenced by the presence of 10-MDP, which bonds chemically to dentine through ionic binding to calcium.[36,37] Although not statistically significant, in corroboration with the Raman findings regarding the stability of Clearfil SE Bond there was an increase in the bond strength means for the cervical and middle regions of the 4-month groups. The 1-step self-etching agent was slightly more aggressive in mineral removal, but its 4-month spectra were quite difficult to read due to the heterogeneity of the degraded hybrid layer.

The lack of penetration in the whole extension of demineralised dentine was detected in both etch-and-rinse and self-etch modalities. Clinically, this might be translated into a poor sealing that threatens the prognosis of endodontically treated

teeth.[26] Moreover, non-encapsulated collagen is susceptible to degradation.[38]

Cement curing was influenced by all 3 factors: bonding agent, root canal region and time. The current results agree with most studies that measured the degree of conversion indirectly through hardness testing, showing a decrease of values in the depths of the post space.[5,13,25,39] The unique aspect of this study was using μ-Raman spectroscopy, which does not require contact, making the measurement possible even if the cement is not hard enough.

In the current study, in which only the bonding agent varied, the impact of the adverse acid–base reaction on the degree of conversion of the cement was observed. Xeno III led to the lowest degree of conversion, mainly in the region where the curing light was less intense. After 4 m, despite the lower conversion tendency in the deepest regions, the degree of conversion seemed to have increased. Nevertheless, the differences caused by the bonding agent in the degree of conversion of the cement did not correlate to the bond strength, and the same was seen in a study of post restorations where the hardness and the bond strength of the cement were measured.[40] On the one hand, greater degree of conversion in the polymer matrix correlated with higher contraction stresses.[41] As a result, the stress that appears as tensile forces at the interface may form stress-relieving gaps, diminishing the bond strength. On the other hand, the residual monomers present at 7 d may well have been leached to the medium, decreasing the number of carbon double bonds and not increasing stiffness of the restoration. That means the increase in the degree of conversion was possibly apparent with the residual monomer eluted after 4 m.[42] In this regard, one could say that at 7 d the reaction is still occurring, thus explaining the low initial values of the self-etch groups (Table 2). However, the monomer elution rationale is more likely, because the degree of conversion increase was not accompanied by bond strength improvements. The high standard deviations are probably a result of the discrepancies of conversion in the three regions of the self-etch groups.

5. Conclusions

Hitherto, it seems that conventional 3-step etch-and-rinse bonded to coronal dentine provides superior bond strength, degree of conversion of the cement and hybrid layer quality, although the sealing ability has still to be questioned in future studies. This conforms to our anticipated hypothesis. But these results are valid only for the materials and procedures presented herein. The great variety of marketed bonding agents, fibre post and cements, plus the necessity to build up a core and to reproduce mechanical fatigue for better similarity to that achieved in the clinics are some of the issues that future work will have to address.

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