Adhesion to Intraradicular Dentin: A Review

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Abstract

Luting fiber posts to intraradicular dentin remains an unpredictable goal due to various clinical factors influencing the clinical procedure. Primarily differences can be found between bonding to coronal or to intraradicular dentin due to the different histological characteristics of the substrates, the high C-factor of the endodontic space, the presence of smear layer due to the post preparation technique, incompatibility between some adhesive systems and resin-based cements, and finally the limited access of the post space that may lead the clinician to different mistakes. Thus this article critically evaluates all aspects that may jeopardize the adhesion of luted fiber posts within the endodontic space, suggesting the use of standardized techniques that improve immediate adhesion and stabilize the adhesive interface over time.

The review of the data currently available in peer-reviewed journals suggests that a strict following of a step-by-step clinical standardized technique allows the clinician to minimize bond failure and obtain a clinically reliable durable bond.

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Keywords

Dentin, adhesion, dental bonding systems, fiber posts

1. Introduction

Adhesion is a complex phenomenon involving physical and chemical mechanisms that allows attachment of one material to another. The bonding systems allow resistance to separation between the substrate and the adhesive and stress distribution along the adhesive interface [1]. In addition, and pivotal for all dental restorations, adhesion means the possibility to properly seal the interface between the cavity and the restorative material, thus reducing the risk of post-operative sensitivity, marginal staining and recurrent caries [2].

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The modern concepts of adhesive dentistry started a long time ago with the pioneering studies of Dr. Buonocore that revealed how etching with phosphoric acid could increase bond effectiveness, in terms of either bond strength or sealing ability [3]. As the first adhesives were mainly based on bisphenol glycidyl methacrylate (Bis-GMA) [4], the bonding system was extremely hydrophobic (i.e., no compatibility with water) and, thus, no adhesion was possible with the dentin tissue since it is an intrinsically wet substrate. Later on the development and the incorporation within the adhesive systems of more hydrophilic monomers, such as 2-hydroxyethyl methacrylate (HEMA), allowed adhesives to bond also to dentin [5]. The possibility of the dental adhesives to tolerate water is extremely important, particularly after the introduction of the total etch technique (i.e., simultaneous etching of enamel and dentin that removes smear layers and smear plugs funnelling the tubule orifices) [6] since relatively large amount of water permeates throughout the dentin thickness wetting the cavity surface [7].

Despite major simplifications in dental adhesion, bonding to intraradicular dentin remains an unpredictable goal due to various clinical factors influencing the procedure. In particular, several differences can be found between bonding to coronal and to intraradicular dentin due to the relative limited access of the post space that may lead the clinician to different mistakes, finally jeopardizing the adhesion [8]. Thus there is a need for a standardized step-by-step procedure that should be applicable also within the endodontic space.

2. Dental Bonding Systems and Classification

Dental bonding systems are resin blends possessing both hydrophilic and hydrophobic properties, thus, named amphiphilic. In other words, adhesives are compounds containing both hydrophilic monomers that allow bonding with the tooth structure, as well as hydrophobic monomers contributing to coupling with the restorative materials or resin-based cements.

From a physical perspective, bonding is a relation between the free energy of the tooth surface and the wetting ability of the adhesive solution [9]. If the adhesive cannot wet the substrate, no adhesion can occur. The adhesion process involves enhancing the surface free energy of the tooth structure with an acidic solution (e.g., etching) so as to reduce the contact angle with the bonding solution that should also contain a surfactant agent (e.g., primer) to allow proper wetting of the substrate [10].

Despite their different formulations, all adhesive systems involve three cardinal steps which are considered as pivotal in establishing a durable adhesive interface: (1) etching, (2) priming and (3) bonding [2]. The etching involves an acidic solution that demineralises the enamel/dentin surface thus increasing their surface free energy. The primer is composed of a mixture of hydrophilic monomers and solvents aiming to allow the wettability of the tooth surface and to permit substitution of the water retained within the substrate with the resin monomers. The bonding
agent contains the hydrophobic part of the system that allows coupling with the resin-based restorative materials or the resin cements.

Adhesive systems interact with the dentin tissue following two different strategies: they can remove the smear layer (etch-and-rinse technique) or maintain it as the substrate for bonding procedure (self-etch technique) [2]. The etch-and-rinse strategy is characterized by the application of a preliminary and separate etching step (usually with a gel of 35–37% phosphoric acid) that is later rinsed away. Conversely, the self-etch approach refers to the application of an etchant/primer solution that is only air-dried, thus, also named ‘etch-and-dry’ [11]. As no rinsing occurs after etching, the acidic compound of the self-etch system remains entrapped within the modified smear layer and the acid is buffered by the mineral released from the substrate [12, 13]. The other steps such as priming and bonding can be separate or combined, depending on the adhesive formulation. If the bonding agent is combined with the primer (for the etch-and-rinse technique) or with the self-etching/primer agent (for the self-etch technique) the adhesives are considered as ‘simplified’ [14].

As all adhesives contain etching, primer and bonding agents in their formulations [2], a classification based on the number and combination of the steps constituting the adhesive system is proposed. Thus we can identify four different classes (Table 1):

1. Etch-and-rinse, three-step: adhesive systems characterized by the sequential application of etching, primer and bonding agents as separate and individual solutions (Fig. 1).

2. Etch-and-rinse, two-step: simplified adhesive systems characterized by the use of a combined primer and bonding agents that is applied on the tooth surface after removal of the etching agent.

3. Self-etch, two-step: adhesive systems characterized by a self-etching primer that is dried on the tooth surface, followed by a separate hydrophobic bonding agent (Fig. 2).

4. Self-etch, one-step: simplified adhesive systems characterized by a combination in a single application solution of the etching/primer/bonding agents. These extremely simplified adhesive systems can be formulated as ‘one-bottle’ systems or, alternatively, in ‘multi-bottle’ systems mixed only prior to the clinical application.

When the dentin tissue is instrumented, a smear layer is created on top of the dentin surface [15]. The smear layer is removed by etch-and-rinse adhesive systems during the etching and the following water rinsing and then the sound dentin is surface-demineralized [16]. Since the peritubular dentin is highly mineralized, the use of the etching agent opens the tubules funnelling their orifices and removing all residual smear plugs [17]. The intertubular dentin is demineralized to only a few µm in depth, depending on acid concentration, gel formulation, time and mode of application (e.g., a continuous brushing technique enhances demineralization) [18].
<table>
<thead>
<tr>
<th>Classification of contemporary dental bonding systems and commercial names of some of the most common adhesive systems</th>
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<tr>
<td><strong>Etch-and-rinse</strong></td>
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<tr>
<td>3-step 4th generation</td>
</tr>
<tr>
<td>Etching</td>
</tr>
<tr>
<td>Apply for 15 s, rinse for 15 s, gentle air-dry keeping dentin moist</td>
</tr>
<tr>
<td>Priming</td>
</tr>
<tr>
<td>Apply 1–5 layers, gentle air-dry</td>
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<tr>
<td>Bonding</td>
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<tr>
<td>Apply 1 layer, gentle air-dry, light cure</td>
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<tr>
<td>Adper Scotchbond Multi-Purpose (3M ESPE)</td>
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<tr>
<td>All Bond 3 (Bisco)</td>
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<td>Optibond FL (Kerr)</td>
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<td>Syntac (Ivoclar Vivadent)</td>
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<td>Gluma Solid Bond (Heraeus Kulzer)</td>
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Figure 1. Hybrid layer and resin tag formation created by an etch-and-rinse adhesive system bonded to intraradicular dentin (light microscopy image 100\(\times\)).

Figure 2. Low magnification view of a self-etching adhesive system bonded within the root canal (SEM image 500\(\times\)).

The removal of the mineral phase from dentin surface allows to expose the dentin organic matrix, which is constituted by an intricate network of type I-collagen fibrils (90% of the organic matrix) [19] and other non-collagenous proteins, such as proteoglycans, lipids and enzymes [20]. The resin monomers (primer and bonding agent of the adhesive system) should then fully infiltrate this delicate network of demineralized collagen fibrils, creating the so-called hybrid layer [21]. The bond established by the etch-and-rinse adhesive systems relies only on micromechanical retention between the demineralized dentin matrix and the polymerized adhesive system [22]. The penetration of the adhesive into the funnelled dentin tubules creates the so-called resin tags [23], particularly extended when the adhesive is applied in endodontically treated teeth since no adverse pulpal pressure is present. Although resin tags have been morphologically described by several researchers, their active role in adhesion is controversial as bond strength value is reduced in deep dentin.
where most of the substrate is represented by dentin tubules [24], confirming that the hybridization of the intertubular dentin is critical for adhesion [25].

When self-etch adhesive systems are used, the smear layer is only partially demineralized, depending on the pH and pKa of the etching acidic solution. In relation to the etching ability of the adhesives, self-etch systems are classified as mild, intermediate and strong [2]. Strong self-etch adhesives are able to completely dissolve the smear layer (i.e., similar to the etch-and-rinse strategy) [26], while intermediate to mild systems modify the smear layer and demineralize the dentin matrix leaving residual hydroxyapatite crystals on the collagen fibrils providing additional chemical bond with adhesive monomer [27], that cannot be obtained with etch-and-rinse adhesives since they fully demineralize the collagen.

While self-etch adhesive systems are characterized by a simultaneous demineralization and infiltration (due to the combination of the primer with the etching agent), impregnation of etch-and-rinse monomers occurs after demineralization. For this reason, etch-and-rinse systems (mainly two-step systems where primer and bonding agent are combined) should be applied on wet dentin to maintain large interfibrillar spaces between the demineralized collagen fibrils, thus facilitating monomer impregnation [28]. The solvent of the bonding agent (usually ethanol, water or acetone) acts as a carrier for the resin monomer that enhances the substitution of the residual dentin water. The air-drying that follows the primer/bonding agent application permits solvent evaporation, leaving the resin material within the collagen network, the bonding agent co-polymerizes with the primer creating a hydrophobic surface that can be polymerized and then coupled with the resin-based material or with the resin-based cement.

3. Substrate: Intraradicular Dentin

The analysis of the radicular portion allows to identify pulpal tissue, predentin and mineralized dentin surrounded by cement tissue. Predentin is a layer of un-mineralized organic matrix that lines the innermost pulpal portion and may greatly vary in thickness but still remaining constant during aging since the amount that calcifies is balanced by the addition of newly secreted organic matrix [29]. The synthesis of predentin begins with the production of large-diameter collagen fibrils (called von Korff’s fibres) by the odontoblasts, mainly consisting of type III-collagen fibrils [30, 31]. Predentin and dentin are in direct and dynamic contact as the odontoblasts produce type I-collagen fibrils and proteoglycans by extending their processes into the forming extracellular matrix starting dentinogenesis. These forming structures of un-mineralized organic components will constitute the dentin organic matrix which represents approximately 30% in volume of the mineralized sound dentin. The remnants of the dentin tissue are composed of water (approx. 20%) and minerals such as apatite [19].

As predentin is removed by the instrumentation of the pulpal tissue due to the endodontic treatment (associated with sodium hypochlorite rinses) followed by
the use of calibrated burs to prepare the post space, the substrate for any adhesive luting procedure within the roots is represented by mineralized intraradicular dentin [32]. Several studies investigated composition and structure of the intraradicular dentin, and major differences in bond strengths between intraradicular and coronal dentin were demonstrated and only minor morphological and biochemical differences were reported. Similar to coronal dentin, intraradicular dentin is a non-homogeneous tissue characterized by the presence of tubules extending from the pulp to the tooth periphery [32]. For this reason the intraradicular dentin tissue can be classified in peritubular and intertubular depending on the density and distribution of the tubules. Peritubular dentin is characterized by the presence of a collar of hyper-mineralized tissue and low content of type I-collagen fibrils; conversely, the intertubular dentin is mainly composed of mineralized type I-collagen fibrils [17]. As the number of tubules greatly diminishes toward the apical region of the intraradicular dentin, the ratio between peritubular and intertubular dentin greatly varies from the apical to the coronal third [32, 33]. Moving toward the apex, the substrate modification will induce changes in the impregnation pattern of the adhesive system, thus reducing peritubular dentin infiltration and resin tag formation, while increasing intertubular dentin impregnation [34]. Since it is well known that resin tags contribute only minimally to bond strength [25], the greater amount of intertubular dentin available for hybridization should potentially lead to higher bond strength in the apical rather than in the coronal third of the root canal [32]. However, the overwhelming majority of the studies report that bond strength to intraradicular dentin decreases from the coronal to the apical third of the root canal and it has been shown that the thickness of the hybrid layer is significantly reduced in the apical third. The thinning of the hybrid layer observed by some authors toward the apex was considered as responsible for the lower bond strength due to reduced impregnation of the adhesive system at this level of the root canal [32]. However, this issue is still controversial as some studies reported similar or increased bond strength closer to the apical region showing no differences in terms of thickness of the hybrid layer (Table 2).

As both the thickness of the hybrid layer and the morphology of the resin tags (determined by the substrate) have been recently shown to contribute minimally to the bond strength, we may conclude that the reduced bond strength reported in most of the studies on the intraradicular dentin compared to the coronal dentin might probably be attributed to other factors instead of the substrate morphology, such as the high C-factor of the endodontic space and the difficulty in clinical handling due to the limited endodontic space.

4. Clinical Procedures — Factors Affecting Bonding to Intraradicular Dentin

A fundamental prerequisite for adhesion to intraradicular dentin is represented by the ability of the clinician to obtain a perfectly clean post space as the use of sodium hypochlorite rinses, EDTA, endodontic cements, gutta-percha, or other endodontic
Table 2.
Studies reporting bond strength values to intraradicular dentin

<table>
<thead>
<tr>
<th>Coronal third &gt; apical third</th>
<th>Coronal third ≥ apical third</th>
<th>Coronal third &lt; apical third</th>
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<tbody>
<tr>
<td>Perdigao et al., Am. J. Dent. 2004</td>
<td>Foxton et al., J. Oral Rehabil. 2005</td>
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<td>Bolhuis et al., Quintessence Int. 2004</td>
<td>Ngoh et al., J. Endodont. 2001</td>
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<tr>
<td>Bolhuis et al., Oper. Dent. 2005</td>
<td>Foxton et al., Oper. Dent. 2003</td>
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<td>Kalkan, J. Prosthet. Dent. 2006</td>
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<td>Perez et al., Int. J. Prosthodont. 2006</td>
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<td>Perdigao et al., Dent. Mater. 2007</td>
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<td>Perdigao et al., J. Prosthodont. 2007</td>
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<td>Faria Silva et al., J. Endodont. 2007</td>
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<td>Boff et al., Quintessence Int. 2007</td>
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<td>Wang et al., Dent. Mater. 2007</td>
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<td>Ohlmann et al., J. Dent. 2007</td>
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Figure 3. Fiber-reinforced post luted into a root canal (stereomicroscopic image 10×).

filling materials clearly modify the intraradicular dentin environment [35] (Fig. 3). In addition, the difficulty to control the substrate surface and to properly apply the adhesive and the cement inside the narrow endodontic space is evident.

Several factors may affect intraradicular dentin: the presence and thickness of the endodontic smear layer, the method of post space preparation as well as several adverse clinical factors; in addition, the use of irrigants or medicaments during the endodontic treatment, the use of eugenol-based materials or bleaching agents, the influence of endodontic re-treatment, the possible incompatibility between different
resin-based materials, and geometric factors are some of the most important aspects that need to be clarified.

4.1. Endodontic Smear Layer

The theoretical goals of successful endodontic therapy are the disinfection and the complete obliteration of the root canal space with an inert filling material, creating an optimal seal with the tooth structure. The prerequisite for a tight seal is the closed adaptation of the filling material to the canal walls, which, however, is impaired by the presence of the endodontic smear layer, invariably formed after manual and rotary instrumentation [36]. According to its original definition, the smear layer has been defined as ‘any debris, calcific in nature, produced by reduction or instrumentation of dentin, enamel or cementum’ [37], or as a ‘contaminant’ [38] that precludes interaction with the underlying pure tooth tissue [1]. The burnishing action of the cutting instruments generates frictional heat and shear forces, so that the smear layer becomes attached to the underlying surface in a manner that prevents it from being rinsed off or scrubbed away [1, 39]. The morphological features, composition and thickness of the smear layer are determined by the type of endodontic instrument used, the method of irrigation and tooth substrate at which it is formed [1, 40].

Coronal smear layers reflect the substructure of dentine matrix composition while endodontic smear layer contains inorganic and organic substances that include also fragments of odontoblastic process, microorganisms and necrotic material [15] (Fig. 4). Despite its different composition the thickness of smear layer ranges from 0.5 to 2 µm, in addition to a deeper layer packed into the dentin tubules to a depth of up to 40 µm, obstructing their orifices thus forming the so-called smear plugs (Fig. 5). The smear layer can be detrimental to effective bonding: in early smear layer research, non-acidic adhesives, applied without prior etching, did not penetrate sufficiently enough to establish a bond with intact dentin; such bonds were prone to cohesive failure of the smear layer [24].

Figure 4. SEM micrograph of smear layer created by instrumentation along the wall of the coronal third (1000×).
As previously mentioned in the description of the adhesive systems, two bonding strategies are used to overcome the low attachment strengths of the smear layer. With the etch-and-rinse approach the use of the etching followed by extensive rinsing removes the smear layer prior to the bonding, while in the self-etch approach the smear layer is only modified beforehand and it is incorporated within the hybrid layer complex [2, 26]. The more acidic and aggressive the conditioner, the more completely the smear layer and smear plugs are removed [41].

Within the root canal, dentin surfaces covered with debris and remnants of pulp tissue are not likely to achieve effective bonding [35, 42], because the endodontic smear layer acts as a barrier significantly influencing any adhesive bond formed between the instrumented canal walls and the restorative material [2], as well as the resin cementation of fiber posts. Although there is some controversy regarding the desirability of retaining smear layer in adhesive dentistry, in endodontics, its removal is considered to be advantageous and highly desirable [43]. The endodontic smear layer, in fact, may be infected and may protect the bacteria already present in the dentinal tubules; because of these concerns, one may deem it prudent to remove the initially infected root canals and to allow penetration of intraradicular medications into the dentinal tubules [44]. In addition to the reduced penetration of root canal medicaments, several studies have reported also a better adhesion of obturation materials after removal of the smear layer [45] and the penetration depth within the dentinal tubules of different sealers is also consistently increased (by 10–80 µm) once the smear layer is removed [44, 46].

4.2. Post Space Preparation and ‘Secondary’ Smear Layer

In addition to the ‘traditional’ smear layer produced by manual or rotary instrumentation of the root canal walls, the subsequent preparation of the post space using post drills resulted in an additional and even thicker smear layer composed of debris and sealer/gutta-percha remnants that significantly influenced the adhesion of
Figure 6. SEM micrograph of smear layer along the root canal walls (600×).

fiber posts [43] (Fig. 6). In fact, the action of the drills used to remove the root-filling material to create post space produces a new smear layer rich in sealer and gutta-percha remnants that are plasticized by the frictional heat of the drill and this may diminish the penetration and chemical action of the agents used to bond fiber posts. In addition, only minimal irrigation can be performed inside the endodontic canal. Thus, achieving clean dentinal surfaces after mechanical post space preparation seems to be a critical step for optimal post retention, in particular, when resin cement is used [47]. It has been reported that the use of phosphoric acid after post space preparation resulted in discontinuous areas of deep intertubular demineralization alternating with areas characterized by open tubules and other areas covered by debris, smear layer and gutta-percha and/or sealer remnants as the result of an incomplete chemical dissolution during the etching procedure, due to the penetration into the dentinal tubules of the sealer and the plasticized material during the condensation procedure [48].

To increase retention when resin cement is used, some authors suggested a pretreatment with a chelating agent and sodium hypochlorite before post cementation in order to efficiently remove the large areas that are not available for bonding and resin cementation of fiber posts [49]. Others authors suggested also the use of ultrasonic instrumentation in conjunction with EDTA pre-treatment prior to the bonding procedure, resulting in a decrease of debris and open tubules [48]. Although the removal of this tenacious and thick smear layer is enhanced by the suggested combination of EDTA and ultrasonic instrumentation (Fig. 7), the effectiveness in terms of interfacial post bond strength is still related to the bonding strategy selected, i.e., the etch-and-rinse or the self-etch approach [50].

4.3. Negative Clinical Factors

Besides the success of the root canal treatment, a successful final restoration is mandatory for long-term clinical success. Extensive loss of tooth structure may require post core restoration of a root canal filled tooth. However, in these systems,
lack of adhesion permits apical or coronal microleakage that causes failure of root canal treatment [51]. For this reason, effective adhesion to intraradicular dentin is a fundamental prerequisite to achieve the proper sealing of the endodontic space. However, adhesion to intraradicular dentin may be affected by several factors: the use of certain disinfectant solutions or medications during root canal preparation may have an adverse effect on the bond strength of post to root canal dentin [52] as well as bleaching and re-treatment procedures [42].

4.4. Irrigating Solutions and Medicaments

During irrigation, radicular and coronal dentins are exposed to various solutions used to disinfect the endodontic space. As previously mentioned, this may cause alterations on the dentin surface affecting its interactions with the resin-based materials used either for root canal obturation or for coronal restoration [53].

The effect of some chemicals, such as EDTA and NaOCl, on dentin has been widely investigated [52–55]. Theoretically, NaOCl is an ideal endodontic irrigant, however, despite positive effects, NaOCl causes problems when used in conjunction with resin-based materials due to its strong oxidizing property. NaOCl leaves a dentin surface characterized by an oxygen-rich layer that can significantly reduce bond strength and increase microleakage [52, 54, 55] (Fig. 8). Thus, if resin cements are used to lute endodontic posts, it is important to optimize clinical procedures that result in high bond strength between the adhesive and dentin and between the resin-based material and the post. In this way, adhesion to intraradicular dentin can be favourably influenced with a 10% ascorbic acid and 10% sodium ascorbate after NaOCl irrigation as this additional step has been shown to completely reverse the compromised bond obtained on 5% NaOCl-treated dentin [54]. Interestingly, 10% sodium ascorbate (pH 7) has been reported to be effective and even better than 10% ascorbic acid (pH 4) at restoring high bond strengths to NaOCl-treated dentin, thus, suggesting that ascorbate acts as a reducing agent. It is likely that the oxidizing
action of NaOCl leads to residual free oxygen within the dentin matrix that may critically interfere with the initiation of the interfacial polymerization of resin cements, leading to lower bond strengths [54]. According to Morris et al. [54], by treating dentin with 10% sodium ascorbate, the intraradicular dentin substrate is converted from an oxidized to a reduced surface, speculating that this treatment restores the redox potential of the dentin and facilitates the polymerization of the resins.

Besides NaOCl and EDTA, the use of hydrogen peroxide (H₂O₂) during canal instrumentation effectively removes remnants of pulp tissue and dentin debris, although the use of 3% H₂O₂ solution has been reported to negatively influence bond strength of resin cement to root canal dentin [51, 52, 56]. Hydrogen peroxide breaks down to water and oxygen thus inducing liberation of oxygen by the chemical reaction of hydrogen peroxide with sodium hypochlorite. Oxygen from such chemicals causes strong inhibition of the interfacial polymerization of resin bonding material [56, 57]. After the use of 3% H₂O₂ alone or in combination with NaOCl, residuals of the chemical irrigants and their products are likely to diffuse into dentin along the tubules, contaminating the dentin surface, which may affect the penetration of resin monomers into the dentin structure or the polymerization of the monomers within the demineralized dentin: this is believed to decrease bond strength to dentin [51, 56].

Reduction in resin bond strength to root dentin is reported to be produced also after the use of RC-Prep (Premier Dental Products, Plymouth, PA) as gliding and demineralizing agent [54]. It has been described that this reduction is the result of concurrently contributing factors. Primarily, the presence of hydrogen peroxide in RC-Prep, that breaks down to oxygen and water during bonding procedure, might generate bubbles or voids altering the resin infiltration process as reported also for bleaching agents and 3% H₂O₂ alone or if used in combination with NaOCl [56, 58]. On the other hand, RC-Prep contains in its formulation the poly(ethylene gly-
col) (PG) vehicle (Carbowax®) to provide lubricating properties which might be difficult to be rinsed off, thus interfering with the polymerization process and preventing a complete polymerization of the resin. However, a plausible restoration of the bond strength to control values can be achieved with a post RC-Prep treatment with 10% ascorbic acid [54].

An additional side effect of these intraradicular irrigating solutions is the significant reduction in microhardness of the root canal dentin walls. Sodium hypochlorite, hydrogen peroxide, EDTA all decreased the microhardness value of root dentin [53, 59]. This reduction after irrigation indicates direct effects of the chemical solutions on the components of dentin structure. Although the relative softening effect exerted by chemical irrigants on the dentinal walls could be of clinical benefit as it permits rapid preparation and facilitates the access to small and tight root canals, these alterations affect adhesion and sealing ability of the sealers to the softened chemically treated dentin surfaces [53, 59]. Based on these observations the use of 0.2% chlorhexidine gluconate has been proposed as an irrigating solution to provide optimal obturation due to its harmless effect on the microhardness and roughness of root canal dentin [59]. Additional positive effects are attributed to chlorhexidine supporting its use as a widely effective irrigating solution during endodontic procedures: higher bond strength compared with other irrigants [51], more effectiveness, more residual antibacterial effect and lower toxicity compared to 5.25% NaOCl with similar clinical timing required to eliminate all microorganisms [60, 61]. Moreover, chlorhexidine absorption by dentin and subsequent release from dentin last for 48 to 72 h after instrumentation [62].

Calcium hydroxide paste is sometimes placed in the root canal for its antimicrobial properties and other desirable effects between endodontic appointments. Moreover, as the complete removal of calcium hydroxide before the obturation is almost impossible, residual particles might interfere with bonding in some areas by acting as a physical barrier [63, 64]. In addition, due to its high pH, calcium hydroxide may also neutralize the self-etching/primer solutions of self-etch adhesives significantly reducing its etching effect, resulting in lower bond strength.

4.5. Eugenol

It is well known that eugenol negatively affects the resin polymerization process thus altering the bonding effectiveness. Sealers and temporary restorative materials such as zinc oxide eugenol contain eugenol in its formulation. The eugenol released from these products can permeate dentin [65] and further interact with resin-based restorative materials. As other phenolic compounds, eugenol is a radical scavenger that inhibits the polymerization of resin-based materials [66]. The negative chemical reaction involves the hydroxyl group of the eugenol that tends to protonize the free radicals formed during the polymerization of resin-based materials, thereby blocking their reactivity and reducing the degree of conversion of these materials [67, 68].

In order to avoid the sub-optimal polymerization and the related reduced bond strength due to the use of an eugenol-based sealer (or temporary material), a me-
chanical cleaning of the canal walls involving scrubbing with a detergent or alcohol to remove all visible signs of residual material has been proposed [69, 70]. This procedure may help to remove an oily layer debris before performing the bonding procedure [42].

The use of 37% phosphoric acid as etching agent for most of the etch-and-rinse adhesives has been reported to only incompletely remove remnants of the temporary restoration [71]; however, the phosphoric acid pre-treatment eliminates the contaminated smear layer and results in the demineralization of dentin to a depth of 9–10 µm [72]. This depth of demineralization and the water rinsing after etching reduces the amount of free eugenol and temporary restoration remnants on the dentin surface [66]. Studies have, in fact, demonstrated that the three-step etch-and-rinse adhesive systems allow better and more effective bonding to eugenol contaminated dentin surface compared to the self-etch approach due to the non-removal of the eugenol debris entrapped within the smear layer [66, 73].

Therefore, in daily clinical practice when posts are placed after the use of eugenol-based sealer or eugenol-based temporary materials, the clinicians should prefer the use of an etch-and-rinse adhesive system, as self-etch systems incorporate the eugenol-rich smear layer into the hybrid layer rather than removing it [42].

4.6. Bleaching

Tooth discoloration due to endodontic reasons is a severe problem in aesthetic dentistry [74]. Although aesthetic can be improved using a variety of techniques, tooth bleaching is the most conservative and cost-effective alternative to enhance the appearance of non-vital teeth [75]. Besides the satisfactory results, side effects of this therapy have been reported [76, 77] including decrease of the bond strength of resin-based restorative materials [74]. It has been reported that the lower bond strength after bleaching is due to the oxygen-rich surface left behind hydrogen-based peroxide products, that significantly inhibits the polymerization of the adhesive systems [56, 76, 77]. Thus, the bond strength may be improved by replacing the final restoration several days after bleaching to allow the release of the residual oxygen. If immediate bond is needed, extended time of polymerization may reverse part of the polymerization inhibition [57].

In addition, it has been reported that a high concentration of hydrogen peroxide causes great decrease in dentin microhardness [78, 79]. This is an indirect evidence for mineral loss in the dental hard tissue, which may affect adhesion and sealing ability of sealers to the bleached dentin surfaces [53, 59].

4.7. Re-treatment

During endodontic re-treatment procedures, radicular and coronal dentins are sometimes exposed to gutta-percha solvents deposited within the root canal. Chloroform and, more recently, halothane are the most commonly used gutta-percha solvents employed for this purpose [51]. These agents are strong lipid solvents that may alter the chemical composition of the dentin surface and of the dentin organic matrix.
thus affecting its interaction with resin-based materials used for restorations. The solvents may redeposit on the root canal surface a waxy film interfering with the resin–dentin bonds [51, 80] and causing significant loss of bond strength [51].

4.8. Incompatibility between Simplified Adhesives and Chemical/Dual-Cured Composites

In response to the increasing demand in procedural simplification in the formulation of adhesives, the classical multi-step dentin adhesives have been replaced by simplified systems that are simpler, faster and more user-friendly [14]. However, incompatibility between simplified adhesives (i.e., two-step etch-and-rinse and one-step self-etch adhesives) and chemical/dual-cured composites has been reported [81–84]. The decrease in bond strength was inversely proportional to the acidity of the adhesives [83–85] and adverse chemical interaction between sub-optimally polymerized acidic adhesive resin monomers and the basic tertiary amine catalyst in the composite was thought to be responsible for the observed incompatibility [83]. In the etch-and-rinse strategy, only the two-step process was claimed to show incompatibility, while the conventional three-step total etch involving the use of intermediate resin layer was unaffected [83, 84]. Similarly, within the self-etch bonding strategy, only one-step self-etch adhesives are incompatible with chemical/dual-cured composites [82], due to their more acidic content by virtue of the increased concentration of lower pKa acidic resin monomers, [86] which is responsible for the additional increased permeability shown by one-step self-etch adhesives [82]. These acidic resin monomers can both react with the basic components (aromatic tertiary amine) of the composite, and/or create a hypertonic environment that osmotically draws fluid from the bonded hydrated dentin through the permeable adhesive layer [85, 87, 88]. Tay et al. reported that also the increased permeability that characterized these adhesives, due to the higher concentration of the ionic resin monomers, was responsible for the aforementioned incompatibility [82].

Thus, based on these observations, three-step etch-and-rinse and two-step self-etch adhesives do not show incompatibility with chemical/dual-cured resin composites due to the use of intermediate bonding resin layer: this additional coat of less acidic and less hydrophilic resin layer prevents the negative acid–base reaction since the composite layer does not come into direct contact with the acidic monomer components in the primer layer and, thus, reduces permeability of the resin–tooth interfaces [83, 84].

Most clinicians generally use dual-cured adhesives for bonding to root canal dentin because of their ability to self-polymerize in the absence of light in deeper regions of post cavity. However, clinically, incompatibility of one-step self-etch adhesives with chemical/dual-cured composites precludes their use for indirect bonding procedures in such areas that are not accessible to light-activation [83].

For the above-mentioned reasons the use of self-etch one-step adhesives should be avoided, although the use of a ternary catalyst has been proposed to overcome
Figure 9. A fiber post luted within the root canal with all-in-one adhesive system in conjunction with a dual-cured resin cement. The combination of these materials results in incompatibility of curing.

the acid–base reaction [88]. This solution has been formulated in most etch-and-rinse two-step adhesives with the addition of a separate bottle of activator solution containing ternary catalyst, and in some self-etch one-step adhesives by the incorporation of ternary catalyst as adhesive component [88]. Nevertheless, it has been reported that adhesive permeability still occurs when these activated adhesives are employed for bonding to hydrated dentin [82, 88, 89].

In conclusion, within the root canal the use of one-step self-etch adhesives is not recommended (Fig. 9) while the 2-step etch-and-rinse adhesives should be employed only in association with the chemical activator in order to circumvent the adverse acid–base reaction although light curing still remains mandatory to obtain a complete adhesive polymerization, overcoming the chemical interaction with cements and finally avoiding low bond strength and resin leaching.

4.9. Cavity Geometric Factors

The employment of methacrylate-based resin materials in endodontic focuses the attention also on the shrinkage stresses associated with their polymerization. During polymerization process, the intermolecular spaces between the monomers are reduced generating sufficient shrinkage stresses to debond the material from dentin, thereby decreasing retention and increasing leakage [90]. In clinical practice, the polymerization phenomenon leads, in fact, to stress development, gap formation and potential bacterial presence at the tooth/resin material interface; however, in addition to polymerization shrinkage, several other factors may influence shrinkage stress and gap formation. Feilzer et al. reported that shrinkage stress is related to the cavity configuration factor (C-factor), defined as the ratio of bonded to unbonded surface areas of the restoration [91], and when the so-called ‘C-factor’ of a restoration is above a certain limit, the stress development exceeds the bond strength of the bonding agents present [92]. The extent of shrinkage stress is, however, dependent also on the viscoelastic properties of the resin material: at a given polymerization shrinkage, the most rigid resin material will produce the highest shrinkage stress,
and, consequently, increase gap formation at the tooth–resin interface [93, 94]. Only if the shrinkage stress caused by the wall-to-wall contraction of the resin material can be relieved by sufficient elastic yielding of the surrounding materials the bond may survive [95], revealing that a major problem associated with endodontic bonding is the lack of relief of shrinkage stresses created in deep root canals [96, 97] which is strictly dependent on the cavity geometry and the resin film thickness [92]. Within the root canal the cavity geometry is unfavourable, since the unbounded surface area becomes small as a consequence of insufficient stress relief in association with high probability that one or more bonded areas will pull off or debond [90]. Braga et al. [98], analyzing the influence of cavity dimensions on shrinkage stress in composite restorations, showed that the cavity depth had a stronger influence than diameter; thus, if we reflect these data to the endodontic cavity configuration, the influence on shrinkage stress is even worse.

Similarly to bonding to root canals, the cementation of endodontic posts to prepared root post spaces is critical for the negative geometric factors, and was described by Feltzer et al. as the worst scenario in achieving gap-free interfaces [92]. According to Bouillaguet et al. [99], the estimated C-factor in post spaces may, in fact, even exceed 200, compared to the coronal restoration values that range between 1 and 5. More recently, Tay et al. [90], using a modelling approach, investigated the geometric variables influencing root-filling bonding adhesive materials to canal walls revealing a negative correlation between C-factor in bonded root canals and sealer thickness: in addition to the C-factor, in fact, the contribution of the other geometric attribute, i.e., the ‘S-factor’, was taken into consideration. As the thickness of the adhesive layer is reduced, the volumetric shrinkage is reduced, which results in the final reduction in shrinkage stress (S-factor). However, it was concluded that the interaction of these two factors (C- and S-factor) predicts that bonding of adhesive root-filling material is highly unfavourable when compared to indirect intracoronal restoration with similar resin film thickness [90].

All the above-mentioned problems limit bond strength to intraradicular dentin. Recently, some authors hypothesized that the retention of fiber posts into root canals was mainly due to friction of the post itself along the cavity walls through the interposition of luting material [100, 101]. However, as friction occurs only after bonding failure, i.e., until the adhesive interface is effective, friction should not play a significant role in terms of post retention. Friction will improve post luting in case no bonding occurs at the coronal part of the abutment, otherwise, even in the presence of low bond strength, the post will continue to be luted to the dental structure due to the bonding interface with the coronal dentin until debonding of the radicular part of the post occurs. Friction is additionally influenced by the granulometry of the luting material [102]; however, it is still unclear if a macrofilled cement can develop a higher ‘bond strength due to friction’ or not.

Goracci et al. [103] investigating the effect of post luting with and without the employment of adhesive systems reported no statistical differences between the groups, and the results were mainly attributed to the limited number of samples
and, in particular, to the low value recorded in the study. In addition, the standard deviation of the two tested groups was very high, thus, the authors do not believe that it can be assumed that ‘retention of fiber posts is mainly due to friction’. More recently, Carvalho et al. [104] showed that the application of the adhesive system was mandatory for obtaining high bond strength to root dentin and that the use of unfilled bonding resins used as luting material consistently increased the bond strength to intraradicular dentin. The important role of unfilled resin for luting fiber posts was further supported by recent findings [105].

Considering the main types of failures reported in clinical practice (i.e., debonding), another important issue related to the geometric factors can be related to the circular shape of the posts which does not correspond to the root shape, at least in the oval root canals. In addition, also the post diameter should be taken into account, fitting properly only the most apical part of the root canals while remaining usually too thin in the most coronal compartment. These two aspects determine a wide thickness of luting cement, which becomes the weakest part of the system under occlusal loading; thus, for minimizing the cement’s thickness the employment of ‘anatomic posts’ [106], ‘oval posts’, indirect luting procedure [107, 108], and/or additional smaller sizes of posts may be useful in clinical practice [109].

4.10. Ethanol-Wet Bonding Technique

An ethanol-wet bonding technique has been recently proposed to improve bonding to dentin [110]. The original wet-bonding technique of etch-and-rinse adhesives requires that water originally present within the interfibrillar spaces of the collagen network be displaced by the polar solvents contained in these adhesives, and ultimately be replaced by pure resins [110]. It has been reported that by replacing water in the demineralized collagen matrix with ethanol, phase separation of the hydrophobic dimethacrylates can be prevented as they are applied to ethanol-saturated instead of water-saturated dentin [110, 111]. Using a macromodel of the hybrid layer to predict how well adhesives can bond to dentin, Pashley et al. [110] indicated that the ethanol-wet bonding might be superior to water-wet bonding and several studies evaluated the effectiveness of this technique [111–113].

Few investigations evaluated the effects of the ‘ethanol-wet bonding technique’ on luting fiber posts revealing that this procedure did not improve bond strength with the fiber post surface [114]; however, it can improve bond strength to intraradicular dentin (Fig. 10).

The main problem to employ this technique is due to the complex clinical procedure based on several steps, to the instability of its component (shelf-life), in addition to the fact that the system is not available on the market yet. In vital teeth, possible pulp damages due to application of pure ethanol on vital dentin for fixing the collagen fibers are theoretically reasonable (thus, several in vivo studies are currently ongoing to challenge the pulp effects of this technique). Although the concept of ethanol-wet bonding may sound radical to a clinician who may have doubts on the potential effects on vital pulp, Pashley et al. remarked that such a procedure was
not so different from the application of acetone-based or ethanol-based primers, as most of these primers may contain up to 85% ethanol or acetone [110]. Based on these observations, further laboratory and clinical studies are needed to test and further validate this procedure.

5. Failure of the Adhesive Interface

As clinical investigation revealed that failure of the bond occurs after some time, researchers recently have focused their attention on the chemical phenomena that occur during aging. Since the hybrid layer is a complex mixture of collagen and adhesive monomers, both components may be affected by aging.

Two degradation patterns were morphologically described within the hybrid layer thickness after storage in water for 1 year: (1) hydrolysis of the resin from interfibrillar spaces and (2) disorganization of collagen fibrils [115]. These phenomena clearly weaken the strength of resin–dentin bond allowing leakage, marginal staining, and finally failure of the adhesive interface with clinical debonding (Fig. 11).

Since resin degradation is related to water sorption within the hybrid layer [116], resin blends characterized by low water sorption result in the formation of bonds that are more stable than hydrophilic adhesive systems which are prone to water sorption. In addition, as the hydrolytic degradation of the resin monomers occurs only in presence of water, adhesive hydrophilicity, water sorption and subsequent hydrolytic degradation are correlated [116–118]. This allowed the researchers to hypothesize that simplified adhesives (i.e., etch-and-rinse two-step, and self-etch one-step that are characterized by the presence of both hydrophilic and ionic resin monomers into the bonding agent) are less stable than un-simplified adhesives (etch-and-rinse three-step and self-etch two-step) which are characterized by the presence of a non-solvated hydrophobic resin coating [11, 119]. The lack of hy-
drophobic characteristics exhibited by simplified adhesives has also been revealed by morphological evidences that their hybrid layer behaves as a semi-permeable membrane after polymerization, allowing movement of water throughout the interface [82]. Recent studies also correlated adhesive permeability with its polymerization kinetics [120]. Interestingly, all simplified adhesives exhibited sub-optimal polymerization which was correlated with their high permeability to fluid movement due to the presence of high concentrations of hydrophilic monomers while un-simplified adhesives showed higher extents of polymerization that were correlated with less permeability to water [121]. As the extent of polymerization within the endodontic space may be reduced due to limited access of the tip of the curing light unit, the extension of curing time could be an important clinical tip suggested during luting of the post, in conjunction with the employment of translucent fiber posts: the use of posts made with translucent components permits the curing light to pass through.

Similarly to resin monomers the collagen fibrils constituting the hybrid layer can also degrade after time contributing to the weakening of the hybrid layer structure [122]. In fact, these extrinsic degradation mechanisms of the resin–dentin interface that originate in the adhesive above the hybrid layers are accompanied by intrinsic degradation mechanisms that originate from beneath dentin hybrid layers [7]. The recent reports of collagenolytic and gelatinolytic activities in partially demineralised dentin collagen matrices [122–124] are indirect proofs of the existence of matrix metalloproteinases (MMPs) in human dentin. MMPs are a class of zinc- and calcium-dependent endopeptidases [125] that are trapped within the mineralized dentin matrix during tooth development [125–128]. The release and activation of these endogenous enzymes during dentin bonding [122] are thought to be responsible for the in vitro manifestation of thinning and disappearance of collagen fibrils from incompletely infiltrated hybrid layers in aged, bonded dentin [119, 123, 124, 129]. Collagen degradation at the bottom of hybrid layers has been subsequently
confirmed *in vivo* in both primate [130] and human studies [131]. The involvement of host-derived MMPs in this degradation process has also been indirectly confirmed, since the application of chlorhexidine, an inhibitor of MMPs [132], to acid-etched human primary dentin resulted in the preservation of collagen integrity within the hybrid layers after the application of a simplified etch-and-rinse adhesive [131]. Unfortunately, a definitive cause and effect relationship between the different procedures employed in the etch-and-rinse technique and the degradation of the dentin hybrid layers has not been yet established. Presumably, phosphoric acid demineralization could have activated the MMPs, trapped within the mineralized dentin [122], resulting in collagenolytic and gelatinolytic activities identified within the hybridized dentin. However, using fluorescein-labeled collagen enzymatic assay, it was found that treatment of mineralized dentin powder with 37% phosphoric acid gel for 15 s actually reduced the inherent collagenolytic activity of mineralized dentin, probably due to its high acidity (pH 0.7), that partially denatures the MMPs [122] causing confusion as to how dentin hybrid layers could be degraded over time. In a recent study Mazzoni *et al.* [133] revealed the potential roles of the adhesives in dentin proteolytic activities using a modeling approach in which the relative proteolytic activities derived from dentin were quantified before and after the sequential applications of the phosphoric acid etchant and an etch-and-rinse adhesive. Within the limits of the study, it was concluded that simplified etch-and-rinse adhesives can activate new endogenous enzymes present in dentin that counteract the MMPs previously inactivated by phosphoric acid-etching, providing a plausible explanation for both *in vitro* and *in vivo* observations of the degradation of dentin hybrid layers [133]. Similarly, also the less aggressive (i.e., less acidic) versions of self-etch adhesives were tested, highlighting the same effect of activation of endogenous MMPs present in crown dentin [134].

With the increasing popularity in bonding to root canals, it was not known if intraradicular dentin possessed similar intrinsic degradation mechanisms that might adversely affect the longevity of resin–dentin bonds. Thus, Tay *et al.* [135] demonstrated, with the same modelling approach, that intraradicular dentin possesses latent collagenolytic activity which can be activated by mild self-etching adhesives. In fact, in this study the decrease in fluorescence associated with the calcium chelator, EDTA, and the classic MMP inhibitor, chlorhexidine, indirectly showed that latent MMPs were present in the instrumented intraradicular dentin and could be further activated by mild self-etch adhesives, resulting in an increase of the collagenolytic activity up to 15-fold [135]. This is an important finding with potential clinical implications since MMPs are zinc-activated, calcium-dependent enzymes; thus, the use of 17% EDTA as an endodontic irrigant may result in chelation of the calcium ions that are required for functioning of these enzymes. Similarly, the use of chlorhexidine as an endodontic irrigant has potential merits, apart from its well-documented antimicrobial benefits, in inactivating MMP activities. However, as reported by Tay *et al.*, 2% chlorhexidine and 17% EDTA only partially inhibited the MMPs activated by the self-etch adhesives, concluding that time, concentration...
and method of delivery to be employed for maximal MMP inactivation have to be optimized in future studies [135].

In conclusion, as several studies stressed the effectiveness of aging process on bonding interfaces and some of them considered the possible detrimental effect of water on bonding interface of luted fiber posts, the role of water within the root canal should be considered. The main hypothesis of water contact along the interface of root dentin/luted post is based on the study by Chersoni et al. [136], demonstrating that permeability of simplified adhesives results in water movement, even in root-treated dentin. However, more recently, a similar protocol [137] was repeated demonstrating that under in vivo conditions, water was not in direct contact with the bonding/luting/post system (Figs 12 and 13), findings that were further confirmed by several other clinical and laboratory studies [138] (Figs 14 and 15). Thus, we can say that the presence of water blisters detected by Chersoni et al.

Figure 12. SEM micrograph of root canal dentin after being treated with absolute ethanol for 1 min in accordance with the substrate conditioning of the ethanol-wet bonding technique (500×).

Figure 13. Water droplets imaged with SEM following the replica technique within the endodontic space (2000×).
Figure 14. A fiber post luted into a root canal after storage in water for 6 months. No degradation of the post is noticeable (SEM image 60×).

Figure 15. A fiber post after storage in water for 6 months with evident signs of degradation.

[136] may be attributed to the experimental procedure or to incomplete solvent evaporation of the tested adhesives instead of water presence [138, 139].

It should be considered that the aging process occurring at the adhesive interface has been predominantly studied in coronal dentin under simulated laboratory conditions whilst almost no data are available on the aging process affecting bonding to root dentin. As the vitality of the tooth determines fluid movement through the dentinal tubules due to the presence of the pulpal fluid, aging of the adhesive interface in intraradicular dentin cannot be related to water mediated phenomena, while other reasons such as degradation caused via the activity of endogenous enzymes such as matrix metalloproteinases can be hypothesized [122, 124]. In fact, a restoration placed on coronal dentin has a bonding interface directly exposed to the oral environment and consequently subjected to ‘water aging process’, whilst the bonding interface created within the root canal is not in direct contact with water unless a very small amount of intrinsic dentin is formed by water [32]. Moreover,
it was recently reported that under in vivo conditions (after 5 years of clinical function) post surfaces that remained exposed to oral environment showed only a small amount of wear because of occlusion while no degradation occurred due to water uptake [140] (Figs 16 and 17).

6. Conclusions

Although in the last 15 years fiber posts have been widely investigated with different techniques and, in many aspects, there has been rapid progress in materials and techniques. Many new developments will take place in the near future before
Figure 17. (a) A post exposed on occlusal surface of a second molar after 5 years of clinical function. (b) Higher magnification view of Fig. 17a under SEM (500×).

reaching the most ideal procedure for luting posts into root canals of endodontically treated teeth.

References


