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Bonding of adhesive resin to intraradicular dentine: a review of the literature

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Abstract

Bonding to root canal dentine is widely practiced by modern dentists, both in general as well as specialist practices. Materials, such as resin-based root canal sealers and resin cements, are routinely used in endodontic treatment, like root canal obturation and luting fiber post for post and core restorations. Though bonding to root canal dentine in principle is the same as bonding to coronal dentine, there are several structural differences between coronal and radicular dentine substrates. Also, the consequences of pulpal necrosis and subsequent endodontic therapy could alter the root dentine substrate significantly, which would have an impact on root dentine bonding. Therefore, this comprehensive review was performed from

the published literature retrieved from “Pubmed” database using specific keywords “root, “dentine”, “resin”, “bonding”. The relevant articles were selected after screening the title and abstracts. Further relevant articles included in this review were identified from the reference lists of the originally retrieved articles. In this review, we have narrated the various aspects of bonding to intraradicular root dentine. Successful bonding to root dentine depends on proper understanding of this unique bonding substrate.

1. Introduction

Root dentine lies on the external and internal surfaces of the root. External root dentine is exposed when there gingiva recession followed by abrasion/caries of exposed cementum at the cervical region (Figure 1). Whilst the internal root dentine lines the root canal spaces (Figure 2). In clinical circumstances, the potential of bonding occurs to both types of root dentine. There are several circumstances in which adhesion to radicular dentine may be employed. These include: (a) bonding to external root dentine at the cervical area or on coronal third of a tooth’s root that was exposed from gingival recession and subsequent caries or due to non-carious lesions (Figure 3). This could be achieved with the use of glass ionomer cements (GIC), resin-modified glass ionomer cements (RMGIC) or resin composites with dentine adhesives, (b) bonding to internal root dentine for obturation of root canal with gutta percha or resin-based cones (Figure 4). This could also be achieved either with the use of glass ionomer (GI) sealer or resin-based sealer, (c) bonding to internal root dentine for cementation of fibre post (Figure 5). This could also be achieved using GIC, RMGIC or resin cements (with or without dentine adhesives).

The root canal is a confined space and bonding to root dentine in such a space is very challenging. In addition, there are several other factors, such as necrotic pulp debris, bacterial toxins, irrigants, smear layer and sealer, which could interfere with resin bonding to root

dentine, either directly or indirectly. Recently, the activities of MMPs have been identified in radicular dentine [1]. These MMPs would degrade the exposed, uninfiltated collagen fibrils at the base of the hybrid layer when activated. Hence, clinicians should have a clear understanding of these challenges and perform adequate measures to overcome them to achieve optimal and durable resin-root dentine bonding.

Resin bonding to root dentine had consistently shown lower bond strength values, when compared to coronal dentine [2,3,4]. Ferrari et al. [5] reported that like coronal dentine bonding, root dentine bonding also depends on the quality of hybrid layer at the bonded interface. The key to achieving an adequate hybrid layer depends on optimum infiltration of adhesive resin to the entire depth of demineralized dentine.

The aim of this article is to provide a comprehensive review of the published literature on resin bonding to intraradicular dentine /root canal dentine/internal root dentine. Chemical bonding to root dentine (both internal and external) with polyacrylic acid (PAA)-based glass ionomer (GI) materials deemed to be beyond the scope of this paper and is therefore not discussed.

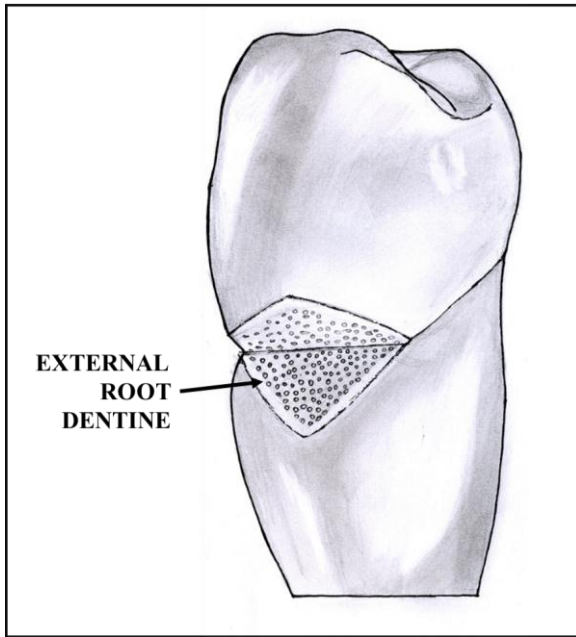


Figure 1. External root dentine exposed at the cervical root region from gingival recession and subsequent abrasion/root caries.

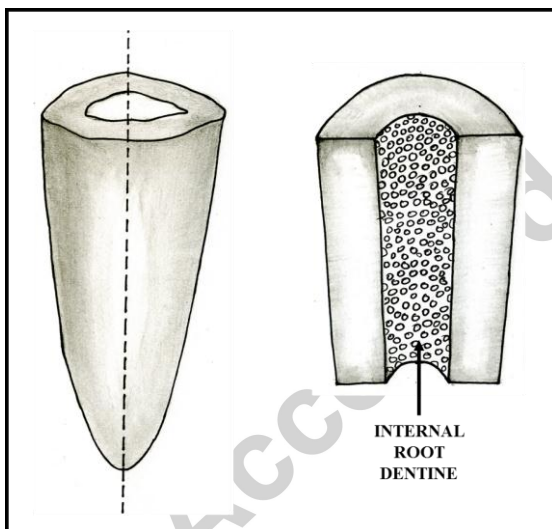


Figure 2. Internal root dentine lies within the root and lines root canal space.

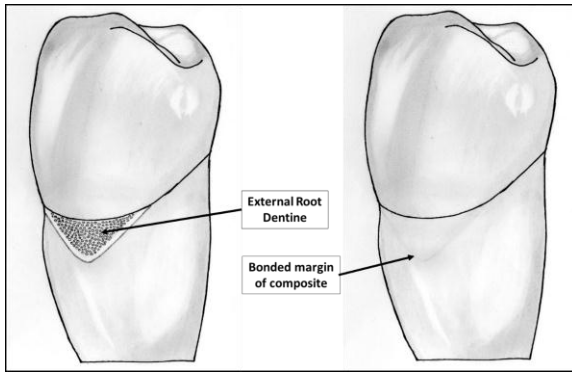


Figure 3. Bonding to external root dentine with composite resin for restoration of root caries or non-carious cervical lesions.

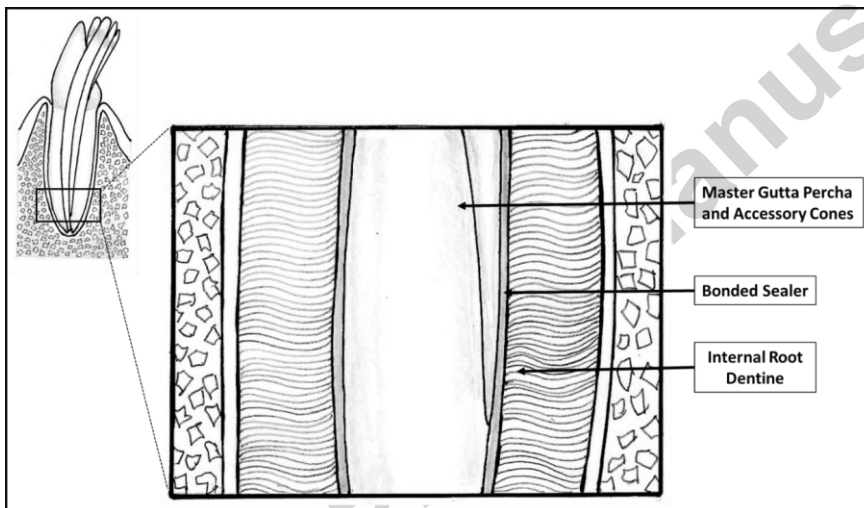


Figure 4. Bonding to internal root dentine with resin sealer for root canal obturation.

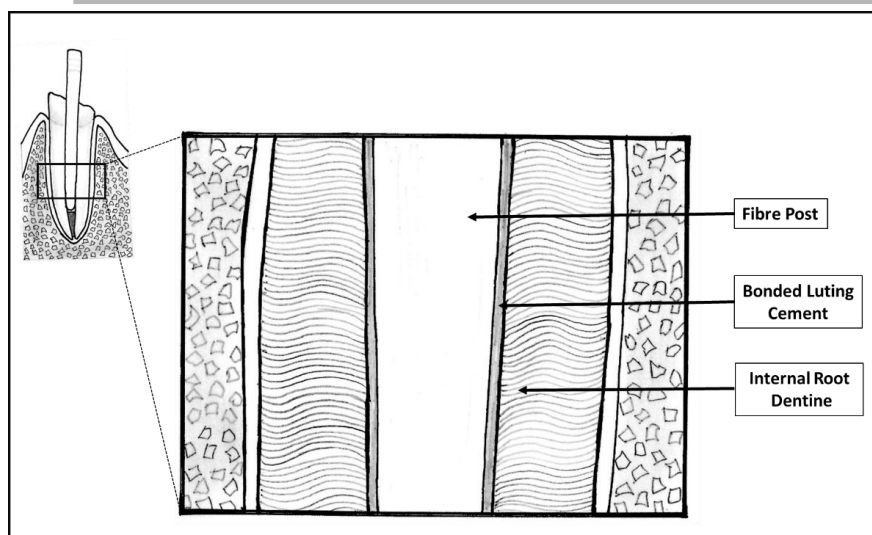


Figure 5. Bonding to internal root dentine with resin cement for luting fibre post.

2. Article selection process:

Specific key words “root”, “dentine”, “bonding”, “resin” were used and the “Pubmed” database was investigated in May 2014. Relevant articles were chosen based on screening through the titles and the abstracts. Further relevant articles were identified from the reference list of the initially retrieved articles and were retrieved using further electronic or manual search. All the relevant articles were individually reviewed by all three authors (ME, CY and JM) and the findings and/or conclusions derived were summarized and narrated in this review.

3. Root dentine

3.1 Regional differences of root dentine

There are differences in the histology of dentine at different levels of the root. Accordingly, the dentine tubular diameter and its density decrease from the coronal to the apical root region [6]. This could be of importance, from resin bonding point of view, as the increase in density of dentine tubuli would lead to a decrease in the availability of intertubular dentine

for micromechanical retention. Resin tags, however, are understood to contribute less to resin-dentine bond strength [7].

Mjør et al. [8] have reported some other challenges in achieving adequate resin bonding to apical root dentine. Such challenges are uneven secondary dentine, presence of tissue on the root canal wall similar to cementum, many accessory root canals as well as the presence of attached and free pulp stones. Only a few studies have discussed the thickness of hybrid layer, which at the apical root region is very thin, in comparison to that at the cervical or middle root region. However, like resin tags, even the hybrid layer thickness does not correlate with resin-dentine bond strength [9].

3.2 Root dentine as a bonding substrate – the challenges

Root dentine is a unique substrate to which to bond. Firstly, it lacks vitality, which directly affects its hydration level and therefore its biomechanical properties [10]. Secondly, it is modified by various chemical agents used in endodontic therapy that could have an influence on the bond strength achieved by an adhesive resin [11]. In this part of the review, the major challenges in achieving a good resin bond to root dentine are discussed.

3.2.1 Sclerosis

Sclerotic dentine is an altered dentine substrate created by repeated cycles of demineralization and remineralization. The surface of such altered dentine is hypermineralized and the dentinal tubuli are filled with mineral casts that make this unique substrate more resistant to demineralization by acid or acidic resin monomers prior to resin bonding. Accordingly, the cervical sclerotic root dentine yielded significantly lower bond strength values in comparison to normal cervical root dentine [12].

3.2.2 Cavity configuration or 'c' factor

The 'c factor' is the ratio of bonded to unbonded dentine surface area in a cavity. In bonding to coronal dentine, this value is very low and only becomes critical, when the value is beyond 5 [13]. In bonding to intraradicular dentine, the 'c' factor sometimes exceeds 200, based on the surface area of bonding. This is challenging as materials used in resin bonding do not achieve adequate bond strength to root dentine and the bonded interface would debond easily due to this relatively high 'c' factor [1].

3.2.3 Visibility and access

Unlike coronal dentine, visibility and access to root dentine is usually inadequate, which lead to great difficulties in bonding to root canal/post space. Optimum bonding to intraradicular dentine substrate is not an easy clinical procedure, even for an experienced endodontist.

3.2.4 Difficulty in moisture control

Moisture control is essential for optimum resin-dentine bonding. However, it is very difficult to achieve adequate moisture control in the root canal/post space, because it retain water by surface tension [10]. Also, the presence of accessory or lateral root canal(s) in some teeth could make adequate moisture control of the root canal/post space even more difficult. Problems may arise when bonding to such a substrate with etch-and-rinse adhesives, as the solvent(s) incorporated in these adhesives may not be able to eliminate the excess water within the clinically relevant air-drying time.

4. Bonding to root canal dentine

4.1 *Effect of endodontic irrigants on bonding to root canal dentine*

The root canal dentine is subjected to treatment by various chemical irrigants during endodontic therapy such as, root canal instrumentation prior to canal obturation; post space preparation as well as bonding with adhesives and resin cements for luting fibre-reinforced composite posts. The commonly used endodontic irrigants are sodium hypochlorite (NaOCl), ethylenediamine tetraacetic acid (EDTA) and chlorhexidine gluconate.

Sodium hypochlorite dissolves dentine collagen and helps in the removal of organic debris resulting from root canal instrumentation and post space preparation. As NaOCl does not have an effect on the inorganic content of dentine, EDTA is often used with NaOCl for removal of inorganic debris during endodontic instrumentation. Chlorhexidine gluconate is another commonly used endodontic irrigant as it possesses antimicrobial properties. However, the use of these irrigants can alter the characteristics of root dentine, which in turn may affect resin-root dentine bonding.

Ishizuka et al. [14] and Inoue et al. [15] have shown that the use of NaOCl as an irrigant affected the bond strength to root dentine and this effect was adhesive-dependent. Mayhew et al. [16] compared the effect of four irrigants on their bonding efficacy of resin cements to root canal dentine. They found that 50% citric acid ($C_6H_8O_7$) and 37% orthophosphoric acid (H_3PO_4) improved bond strength of resin cement to root canal dentine; whilst 5.25% NaOCl and 0.9% saline (NaCl) had no effect.

Several studies have shown that pretreatment of root canal dentine with 5% NaOCl affected bond strength of resin adhesives and cements [11,17,18], as a result of the entrapment of oxygen from NaOCl in demineralized dentine, which could have adversely affected resin

polymerization. It has been suggested that the use of an irrigant with reduction potential could reverse the oxidizing effect of NaOCl. Accordingly, Vongphan et al. [19] and Weston et al. [20] employed a sodium ascorbate irrigant to reverse the negative oxidizing effect of NaOCl on resin-dentine bonding. Li et al. [21] tested the manufacturer's recommendation of using sodium toluene sulfinic acid (SA) on the NaOCl-treated dentine and confirmed the bond strength regaining effect of SA treatment.

EDTA diluted in water is another commonly used root canal irrigant in endodontics. It is usually used as a final rinse before bonding the resin sealer to root dentine. Garcia-Godoy et al. [22] showed that 17% EDTA could effectively remove the smear layer from dentine of the root canal-treated teeth. They also tested another rinse, Biopure MTAD and concluded that though both rinses effectively removed the smear layer, they could cause a collapse of the dentine matrix structure. This in turn would lead to poor resin infiltration and subsequent poor hybrid layer formation. In contrary, Vilanova et al. [23] showed that 17% EDTA, when used as a final rinse for 5 min, produced high resin-dentine bond strength with the resin sealer. They also rated different irrigants in achieving good bond strength with root dentine as:

1% NaOCl + 17% EDTA > 17% EDTA = 24% EDTA gel = 2% Chlorhexidine (CHX) gel > 1% NaOCl.

Bitter et al. [24] studied the different final irrigation protocols of post space and their effects on bonding of fibre post with adhesive systems. They concluded that self-etch adhesive resin cements produced significantly higher bond strength to dentine, when irrigation of the post space was carried out with 18% EDTA/5.25% NaOCl; while for etch-and-rinse adhesives, optimum bonding could be achieved with the use of 1% NaOCl, along with passive ultrasonic irrigation to clean the post space. The oxidizing effect of NaOCl and its negative effect on

root dentine bonding with etch-and-rinse adhesives could be nullified by rinsing it off with water and subsequent use of phosphoric acid etching.

Lambrianidis et al. [25] reported that both EDTA and CHX, when used as an irrigant in the form of a gel, may leave some remnants on the surface of root canal dentine. This could prevent the diffusion of adhesive resin into demineralized dentine and compromised bonding. Interestingly, some studies [26,27] found that when NaOCl and CHX were mixed together, they produced a precipitate and subsequent discoloration of dentine. This could be attributed to the acid-base reaction between NaOCl and CHX could be the reason for such behavior. This is an important observation because precipitation and subsequent discoloration could adversely affect resin bonding and aesthetics of the final restoration.

Demiryürek et al. [28] compared various surface treatments and concluded that the acetone-based cleansing agent, Sikko Tim, along with ethanol and ethyl acetate produced the highest bond strength of fibre post to root canal dentine, when compared with EDTA, orthophosphoric acid, citric acid and control groups. However, they also observed that Sikko Tim along with some other agents in the study group did not effectively remove the smear layer and remnants of the sealers from the post space. In summary, the use of NaOCl during root canal treatment with 17% EDTA as a final rinse could be a suitable strategy to achieve optimum bonding with resin-based materials to root canal dentine.

4.2 *Effect of root canal sealers on bonding to root canal dentine*

Root canal sealers are used in combination with gutta percha or resin cones, such as Resilon for an endodontic obturation. They are either bondable or non-bondable. In either case, it is very difficult to completely remove sealers from the surface of root canal dentine, while

preparing a post space. Hence, the sealers can directly or indirectly influence resin-dentine bonding.

Demiryürek et al. [29] and Teixeira et al. [30] showed that the root canal sealers, based on their chemical composition, can have an effect on bond strength of fibre posts luted to root canal dentine. Eugenol-based sealers have been shown to have a negative effect on resin and root canal dentine bond strength [31,32]. Schwartz et al. [33] concluded that eugenol, which remained on the surface of root canal dentine, affected polymerization of resin adhesives and interfered with resin penetration into the dentine matrix. In contrast, Mannocci et al. [34] and Davis and Connell [35] demonstrated no difference in bond strength of fibre posts, which have been luted to root dentine and treated with eugenol- and non-eugenol-based sealers.

Unlike zinc oxide (ZnO)-based sealers, there are no contradictory results for calcium hydroxide (CaOH₂)-based sealers on bond strength of fiber post luted to root canal dentine. Menezes et al. [32] and Hagge et al. [36] have shown that there was no difference in resin to root canal dentine bond strength between the groups with calcium hydroxide-based sealers and the control group, in which no obturation of root canal was done and therefore no sealer was used. In summary, the use of zinc-oxide eugenol-based root canal sealers could have a negative effect on bonding of resin-based materials to root canal dentine; whilst calcium hydroxide-based sealers may not have such an effect.

4.3 Effect of pulpal remnants on bonding to root canal dentine

Root canals have a complex anatomy and complete debridement of such a structure is very challenging and cumbersome. Due to this anatomical factor, root canal dentine, unlike coronal dentine poses another challenge as a substrate for resin bonding. Peters et al. [37] showed that a significant part of the root canal walls were untouched by endodontic

instrumentation. Nair et al. [38] reported that routine root canal irrigants were not completely effective in such undebrided root canal walls. Thus, bonding of adhesive resins to internal root dentine with residual pulpal remnants on its surface can be severely compromised.

4.4 Effect of smear layer on bonding to root canal dentine

Eick et al. [39] demonstrated that the smear layer was composed of fragments within the size range of 0.5-15 μm . McComb and Smith [40] described the smear layer on the instrumented root canal surfaces and proposed that it contained dentine debris, remains of pulp tissue, odontoblastic processes and bacteria. Later in 1977, Lester and Boyde [41] described the smear layer as being composed predominantly of inorganic particles, for the reason that sodium hypochlorite was not able to remove it. Czonstkowsky et al. [42] observed that the motor-driven instruments, such as Gates-Glidden or post drills, produced quantitatively more smear layer than hand files.

Several studies have been performed to examine the effect of smear layer produced on coronal dentine to adhesion with self-etch adhesives. Oliveira et al. [43] stated that a thick smear layer on dentine surface affected the bond performance of self-etch adhesives. By contrast, Reis et al. [44] and Tay et al. [45] reported that the bonding efficacy of self-etch adhesives to dentine was not affected by the thickness of smear layer. Yang et al. [46] reported that the smear layer incorporated in the bonded interfaces of self-etch adhesives consisted of disorganized collagen fibrils and was prone to degradation.

Serafino et al. [47] stated that post space preparation led to a thick smear layer that routinely contained remnants of dentine, sealer and gutta-percha. The thick smear layer could affect the bonding of fibre post to root canal dentine. Coniglio et al. [48] opined that post space preparation produced a thick smear layer and that just following the manufacturer's

recommendation to rinse the post space with water alone before bonding might not be sufficient.

With contemporary etch-and-rinse adhesives, the smear layer is completely removed by the acid etching procedure; whilst with self-etch adhesives, the smear layer is incorporated within the hybrid layer. Hence, in bonding fibre posts to root canal dentine, the smear layer and its effect on the bonding are a substantial concern.

Mayhew et al. [16] stressed the importance of complete removal of smear layer for effective bonding of fibre posts to root canal dentine. Gu et al. [49] emphasized that the effect of smear layer thickness on root canal dentine bond strength self-etch adhesives was unpredictable. They also demonstrated that EDTA was the most effective irrigant to remove the smear layer from the post space and hence it should be used, when self-etch adhesives were used for bonding fibre posts. It has been stated by Habelitz et al. [50] that rinsing with EDTA did not affect the unaltered collagen fibrils and the intrafibrillar minerals. This property of EDTA may help in the stability of collagen fibrils and facilitate interfibrillar resin penetration. This action could subsequently produce more stable resin-dentine bonds.

Different irrigation protocols for removal of smear layer from the post space before bonding have been suggested. Accordingly, Zhang et al. [51] suggested an ultrasonic agitation of EDTA/NaOCl irrigant to improve bond strength of fibre posts to root canal dentine with self-etch adhesives. Conversely, Scotti et al. [52] suggested etching of the post space with 32% phosphoric acid before bonding the fibre post to root canal dentine with self-etch adhesives. In summary, the use of EDTA as a post space irrigant could enhance the bonding performance when self-etch adhesives are used to bond fibre post to the root canal dentine.

5. Resin-based dental materials used in bonding to root canal dentine

Adhesive resin materials have been in use for more than two decades in dentistry. With the progressive development of resin-based dental materials, the concepts of resin bonding were applied to root canal dentine substrate as well. Accordingly, resin-based bonding materials have several applications in endodontics, such as obturation materials, dentine adhesives and cements. Each of these materials has greatly improved over time due to the advancements in their chemical compositions. In this part of the review, each resin-based bonding material will be discussed in detail.

5.1 Resin root canal obturation materials

Resin root canal obturation materials include resin-based sealers and resin-based cones. The resin-based sealers could either be bondable or non-bondable to root canal dentine. There are four generations of methacrylate resin-based root canal sealers. The first generation sealer material was Hydron™ (Hydron Technologies, Pompano Beach, FL, USA), which appeared in the mid-1970s [53]. The major component of this sealer was poly(2-hydroxyethylmethacrylate), also known as poly(HEMA). It was very hydrophilic and therefore exhibited increased water absorption, swelling and severe leakage [54]. In addition, the material induced severe inflammatory reactions [55] and was soon withdrawn from the market. Two decades after Hydron™ was off the market, the second generation resin-based endodontic sealer named EndoREZ™ (Ultradent, South Jordan, UT) was introduced [56]. This endodontic sealer employed acidic and hydrophilic resin monomers. The composition of the sealer enabled it to penetrate through the dentinal tubuli after removal of the smear layer from the root canal wall [57].

In the last five years, the advancements in resin-based root canal sealers have been rapid and therefore, the third and fourth generations root canal sealers have been released in the market. The third generation resin-based sealers [58] (*e.g.* RealSeal™; SybronEndo, Orange, CA, USA) used a separate self-etching primer and a flowable resin composite. Thus, the root canal dentine was etched and primed in the first step and thereafter the flowable composite was used as a sealer [59].

The fourth generation resin sealers exhibited a self-etching potential; while the fifth generation *i.e.* the most recent, consists of the self-adhesive luting resin composites. The fourth generation resin-based sealers (*e.g.* MetaSEAL™, Parkell, Farmington, NY, USA; and RealSeal SE™, SybronEndo Corporation, Orange, CA, USA) [60,61] were in principle, similar to the latest self-adhesive resin composite cements. These sealers combined the self-etch primer with a minimally filled flowable composite. This fourth generation resin-based sealer is considered to be more user friendly as it does not require a separate etching and priming procedure. These sealers, when used in combination with resin-based cones (*e.g.* Resilon™) for obturation, are claimed to behave like the so-called “monoblock”, due to chemical bonding between these materials [62].

With resin-based sealers, they may be non-etching or self-etching. EDTA has been used as the final rinse after root canal instrumentation and before application of resin-based sealer to root canal dentine, for the removal of smear layer and partial demineralization of root canal dentine [63,64,65]. Skidmore et al. [66] and Mamootil and Messer [67] showed that resin-based sealers produced significantly higher bond strength to root canal dentine than zinc oxide-based sealers.

Several studies compared the sealing efficacy of conventional gutta-percha/non-bondable sealers with that of bondable sealer/resin cones and concluded that the bondable sealer/resin cones yielded lower bond strength to root canal dentine [68,69]. In addition, more voids [68,69] and gaps [70] were shown to be present with the bondable sealer/resin cones. Subsequently, Santos et al. [71] compared the sealing ability of a bondable resin-based sealer (Epiphany/Resilon) with a non-bondable resin sealer (AH plus/gutta-percha) as control. It was concluded that root canal obturation with a non-bondable sealer provided a superior seal than the bondable resin-based sealer, even after a 6-month storage of the specimens.

Researchers have tried to answer the question “Do resin-based root canal obturation materials reinforce the roots?” Grande et al. [72] concluded that endodontic obturation materials and their respective adhesive procedures failed to have an impact on the mechanical properties of root canal dentine. William et al. [73] demonstrated that the cohesive strength and modulus of elasticity of resin-based obturation materials were very low and therefore would not be able to reinforce the roots. Conversely, Hiraishi et al. [74] stated that Resilon (resin based cones for obturation) lacked free radicals on the surface, which are necessary for polymerization with the resin sealer. Hence, the authors doubted the bondability of Resilon with resin sealer. In addition, Stuart et al. [75] found no difference between Resilon and gutta-percha for strengthening and reinforcement of immature roots. Nevertheless, Tay and Pashley [76] explained that due to the unfavourable geometric factors that prevailed in the root canal space, obturation with resin-based materials could produce very high stresses at the resin-dentine interface. This could lead to gap formation in the sealer-dentine interface. In summary, the existing *in vitro* studies do not support the presented hypothesis that resin-based root canal obturation materials reinforce roots.

Zmener et al. [77] studied the effect of root canal dentine moistness on the sealing ability of resin sealers and concluded that hydrophilicity of resin sealers was unable to absorb all of the moisture in dentine completely. Therefore, residual water entrapped between root canal dentine and the sealer affects the resultant bond strength. Nagas et al. [78] studied the degree of moistness on root canal dentine and its effect on resin sealer bond and suggested that keeping the canal lightly moist before obturation with resin sealers would promote bonding.

5.2 *Resin adhesives for bonding to root canal dentine*

Dental adhesives are broadly classified into “etch-and-rinse” and “self-etch” types. The etch-and-rinse adhesive applies a separate etching procedure to demineralize the superficial dentine surface. This is followed by priming and adhesive applications that are done either separately or in a combined form. Accordingly, they are classified into 3-step and 2-step etch-and-rinse adhesives. A self-etch adhesive does not involve the etching procedure, instead it employs an acidic resin monomer to demineralize and prime dentine simultaneously. This is followed by a separate adhesive application in the 2-step self-etch adhesive. The most simplified version of this dental adhesive combines the two steps of self-etch adhesive into one, so that just one application of adhesive will perform etching, priming and adhesive coating.

The dental adhesives used in coronal dentine bonding are also applicable for bonding to root canal dentine. Furthermore, due to the potential difficulty in light-curing of adhesive, some of the commercial adhesives are available as self-cure/chemical-cure or dual-cure for use in root canal/post-space, especially in the deeper areas of the root canal/post space region. Aksornmuang et al. [79] demonstrated that the light-curable dentine adhesives are suitable for bonding to root canal dentine. It has been suggested that based on the type of adhesive, the

increase in photo-irradiation time could improve the bonding efficacy of such adhesives to root canal dentine. Furthermore, the same authors also tested the use of Clearfil SE Bond™ for bonding of fibre posts to root canal dentine and showed that by increasing the photo-irradiation time, failures of bonded specimens at the resin-dentine interface could be prevented [80].

Dental adhesives used in bonding of root canal dentine are predominantly used for fibre-reinforced composite posts [81]. For this clinical application, resin-based luting cements are used along with resin adhesives. Fibre posts are being increasingly used in contemporary clinical practice, because they are less stiff compared to conventional metallic posts and should therefore reduce the chances of vertical root fracture [82,83]. Moreover, fibre posts can be luted to root canal dentine with resin adhesives and resin cements [81]. Core build-ups with resin composites on a fibre post, in particular a glass fibre post, produce good aesthetic restorations when covered with a ceramic crown.

Since fibre posts can be immediately luted into the post space, they eliminate the need for a temporary post. This is in contrast to a tooth that requires a cast metal post, in which a temporary post is usually required during the period of laboratory fabrication of the cast post. This is very important because evidence has shown that teeth restored with temporary posts had similar levels of contamination, when compared with control teeth that did not receive any restorations [84].

Success of fibre posts depends upon the fibres and their proper luting to root canal dentine, which in turn depends upon proper application of resin adhesives and luting cements. It has been shown that the most common cause for failure of fibre post restorations is post debonding [85]. In fact, several studies have compared the effectiveness of different types of

resin adhesives on bonding of fibre posts to root canal dentine. Goracci et al. [86] demonstrated that etch-and-rinse adhesives produced significantly higher bond strength, when compared to self-etch adhesives in bonding fibre post to root canal dentine with a resin cement. Among the etch-and-rinse adhesives, 3-step etch-and-rinse adhesives showed a better sealing ability to the root canal dentine than 2-step etch-and-rinse adhesives [34,87]. Similar results have also been reported by Ferrari et al. [88]. In contrary to this, some studies [89,90] have shown that self-etch adhesives produced better bonding to root canal dentine than etch-and-rinse adhesives. Among the self-etch adhesives, there was no difference in bonding efficacy between the 2-step and 1-step adhesives [91].

From the point of technique sensitivity and ease of use of resin adhesives in bonding to root canal dentine, it has been suggested [9] that self-etch adhesives would be better than etch-and-rinse adhesives, as the latter require careful removal of the excess water after rinsing the acid etchant from the confined root canal/post space, before carrying out the bonding procedure. However, limitations of acidic resin monomers in self-etch adhesives to demineralize root canal dentine covered with a very thick smear layer from post space preparation in particular, should not be overlooked. Regarding the different curing modes, no significant difference in bond strength to root canal dentine was found between the light-cured and self-cured adhesives [91,92]. In bonding of fibre posts to root canal dentine, Hayashi et al. [93] showed that self-cure etch-and-rinse adhesives formed stable bonds in all of the tested root regions.

Cheong et al. [94] and Suh et al. [95] reported the incompatibility between acidic resin monomers in self-etch adhesives and chemically-activated composite resin. Suh et al. [95] further explained that the acidity of resin monomers in self-etch adhesives deactivated the

tertiary amine activators in the self-cured composites and this was the reason for their incompatibility. Some commercial adhesives contain aromatic sulfonates to counteract the incompatibility between the acidic resin monomers and the self/dual-cure resin composites. Suh et al. [95] and Say et al. [96] found that such attempts did not solve the problem as the quality of the resultant polymerization of the chemical/dual-cure resin composite was still inferior.

Considering the various contemporary adhesive systems and luting cements available in the market for bonding fibre post to root canal dentine, Monticelli et al. [97] emphasized that it is very important to follow the manufacturer's recommendation to achieve the best results. In summary, literature shows mixed results on the bonding performances to root canal dentine by various types of commercial adhesives. Manufacturer's recommendation must be followed to achieve an optimum bonding to root canal dentine with any type of dentine adhesives.

5.3 Resin luting cements for bonding to root canal dentine

Resin-based cements have been shown to be superior to conventional cements for retaining post in the root canal post space [98] and are believed to reinforce the root [99]. Resin cements are classified according to their modes of curing as: chemically-cured, light-cured and dual-cure. Chemically-cured luting cements are not easy to use as they have a very limited working time. Light-cured cements are difficult to cure at the apical part of the post space, even with the use of a light transmitting fibre post [100]. Hence, the dual-cure resin cements are commonly used for luting fibre post to root canal dentine. Dual-cure luting cements produce higher bond strengths to root canal dentine, when compared to chemically-cured cements [1,17]. However, dual-cure resin cements do not achieve an optimum degree of monomer conversion in the absence of light [101]; hence, light-curing is recommended

[102]. Foxton et al. [91] studied the bonding performance of dual-cure resin core materials to root canal dentine and concluded that there was no difference in bond strength in either the coronal or apical post-space regions, regardless of whether the polymerization was initiated by chemicals or light.

Foxton et al. [91] reported on the limitations of luting a fibre post in a flared canal, because there is an excessively thick layer of resin cements in such a canal, particularly at the cervical region. Such thick cement would not be able to combat the occlusal loading after restoration of the root with a crown. However, back in 1994, Assif and Gorfil [103] suggested to use a stiffer composite resin at the post-dentine interface, where the cement thickness could exceed 500 μm . Torbjörner et al. [104] demonstrated from a finite element analysis that luting cement was less stiff than the post and dentine and therefore could concentrate a high level of stress. Bouillaguet et al. [1] and Ari et al. [17] reported that resin luting cement in a root canal post space should be able to dissipate the polymerization stress by flow, which would prevent the stress accumulation at the cement-dentine interface. Otherwise, the stress would lead to post debonding.

Panavia™, a gold standard, resin-based dual-cure cement, has been studied extensively and proven to be good for clinical luting applications. Panavia™ is composed of a phosphate monomer, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP). This monomer is capable of forming a chemical bond with the residual calcium ions that remain after the dentine conditioning step. The resultant MDP-calcium salt is stable in the aqueous environment and hence the dentine bond formed with this cement is expected to be durable. Mannocci et al. [34] showed that Panavia™ resulted in a significant reduction of micro-leakage, when compared to other luting cements used for bonding fibre posts to root canal dentine.

In recent years, a new type of resin luting cement known as “self-adhesive resin cements” have gained popularity. This group of resin cements does not require etching and priming steps prior to application for cementation, as it combines the acidic and hydrophilic monomers in the cement composition. This unique composition performs demineralization and priming of dentine together with the luting function. This group of luting cements is probably easier to use as it eliminates numerous steps in application that were required with the previous generation of luting cements. Moreover, since the number of clinical steps is reduced, this type of luting cements is claimed to be less technique sensitive [105]. The manufacturers of this type of resin cement claims that these cements exhibit both micro-mechanical and chemical bonding to root canal dentine.

Bitter et al. [3] compared several resin luting cements for their bonding effectiveness to root dentine and concluded that a self-adhesive resin cement, RelyX Unicem™ (3M 3M ESPE, Seefeld, Germany), showed the highest bond strength among all tested cements. Sadek et al. [106] tested the bonding efficacy of RelyX Unicem™ luting cement to root canal dentine using the push-out test method. They demonstrated that the cement gained its maximum bond strength to dentine after 24 h of the luting procedure. By contrast, Calixto et al. [107] showed that the self-adhesive resin cements produced very low bond strengths to root canal dentine and among the different root regions, bond strength was the lowest in the apical third.

Some researchers have blamed the weaker etching potential of self-adhesive resin cements for the low bond strength and concluded that such cements are less effective than etch-and-rinse and self-etch adhesives [86,108]. Methacrylated phosphoric esters present in the self-adhesive resin cements are not acidic enough to demineralize the root canal dentine that lie beneath the thick smear layer produced from the post space preparation [86]. Furthermore,

Kumbuloglu et al. [101] reported that this group of resin cements displayed reduced monomer to polymer conversion after light polymerization. Zicari et al. [109] demonstrated increased micro-leakage in the root canals with fibre post luted with self-adhesive resin cements, when compared to those luted with resin cements after bonding with etch-and-rinse or self-etch adhesives.

Attempts have been made to improve root dentine bonding performance of self-adhesive resin cements. Zorba et al. [110] tested the bonding efficacy of self-adhesive resin cement to root dentine after removal of the smear layer that was produced from the post-space preparation. Accordingly, they concluded that self-adhesive resin luting cement bonded better to root dentine after removal of the smear layer using EDTA/NaOCl. However, the authors used water as a final rinse to counteract the negative effect of NaOCl on resin polymerization. In the same study, the authors demonstrated that if such a prior irrigation protocol is followed, the self-adhesive resin cement could bond better to the root dentine than cement with prior self-etch step for bonding. Erdemir et al. [111] studied the effect of prior bonding of post space with a 1-step self-etch adhesive and concluded that such procedures did not improve the bonding efficacy of self-adhesive luting cement.

6. *In vitro* bond strength test methods for bonding to root canal dentine

The laboratory tests that are used to evaluate the bond strength of resin-based materials to root dentine include the pull-out test or tensile strength test, micro-tensile strength and push-out test.

6.1 *Tensile test method*

The pull-out test is a conventional test method and is unreliable because when testing the bonding of a fibre post to root dentine, the entire post is pulled out of the root canal, exactly

in an opposite direction to its path of insertion. Anatomic retentive factors and non-uniform stress distribution along the adhesive interface are likely to influence the test results [112].

6.2 *Micro-tensile test method*

Micro-tensile bond strength test method is an improved version of the tensile test method, as the bonded specimens are sectioned into smaller sized sub-specimens in this method and are then individually subjected to the pull-out test. Hence, unlike the tensile test method, the stresses are more evenly distributed along the bonded interfaces [113]. The bond strength of the specimen is calculated from the mean bond strength data of the tested specimens. This is one of the most commonly used methods for bond strength testing of a fibre post to root dentine. The bonded roots are sectioned horizontally as the first step and then the slices are trimmed for pull out testing. Goracci et al. [2] demonstrated that the trimming procedure of the bonded roots luted with fibre posts led to an increased percentage of premature failure of the specimens. Hence, this test method is not an optimal method for testing bond strength of a fibre post luted to root dentine.

6.3 *Push-out test method*

Also referred as “micro push-out test” or “thin slice micro push-out test”, it is the most reliable and commonly used laboratory method to test the bond strength of a fibre post luted to root dentine [2]. It produces shear stress at both interfaces (post-cement and cement-dentine) of the luted fibre post to root dentine with resin cements [114]. This simulates the stress accumulation during clinical functioning of the restored tooth [115]. Drummond et al. [116] reported that fracture of the specimens tested by this method occurs parallel to the bonded interface and hence, the test has been referred as a “true shear test”.

The push-out test leads to less premature failures of the specimens and reduced variation in the distribution of bond strength data [2]. Soares et al. [117] from his finite element analysis confirmed that the push-out test method is a relevant bond strength test for fibre post luting to root dentine. Unlike the “pull-out” or “tensile test”, root canal/post space regional bond strength measurement is possible with the push-out test method.

The above-mentioned advantages of this test would only be applicable, when used to test thin slices of root sections. When applied to thick root sections, or when attempts are made to push the entire post out of the root, it leads to accumulation of uneven stresses at the adhesive interfaces [31,115].

7. Root canal regional bond strength

The root canal anatomy is complex; additionally, there are structural variations within each segment of the root. Adequate moisture control is challenging to achieve in the deeper parts of the root canal space and complete removal of medicaments and sealers from the apical part is extremely difficult. Rueggeberg and Caughman [118] concluded that the intensity of the light diminishes as the distance of the light source increases. Hence, dentine in the deeper part of the root canal might be a challenging substrate for adequate resin bonding through light-polymerization of the resin.

In vitro studies on resin bonding to root dentine mostly examined and compared bond strength data between different root regions. Accordingly, the studies compared the bond strength between cervical, middle and apical root regions. Results from published studies on regional bond strength in the root canal/post-space are quite variable. Several studies demonstrated higher bond strength to the cervical root canal dentine than to deeper root regions [90,119,120]; while other studies have reported a lower bond strength at the cervical

root region [1,92,121]. Several other studies did not report any variations in bond strengths in different root regions [12,31,85,91,122,123,124]. Numerous factors could have led to such variation among the published studies, these include differences in the teeth used in the studies, slightly different materials, variations in the operator(s) skills, and different test methods that were adopted.

Studies which achieved higher bond strength with cervical root dentine, when compared to apical region, attributed the results to the differences in dentinal tubular density between the cervical and apical root regions. By contrast, those studies with the opposite results explained that bond strength to root dentine is related more to the area of solid intertubular dentine than to tubular density [90,91].

In general, several clinical procedures have been suggested to prevent potential low bond strength in the deeper parts of the root dentine: use of a microbrush to carry the resin adhesives to deeper parts of the root canal post-space [125], use of light curing units with high light intensity [126], extended photo-irradiation time [79] and use of dual-cure resin luting cements [1,17].

7.1 *In vitro* aging methods of the bonded specimens

In vitro research studies have been conducted to simulate the clinical conditions. *In vitro* resin-dentine bond strength studies should not only assess the immediate bonding behavior, but also study the durability of the bonded interfaces. There are different methods of aging the bonded specimens *in vitro*.

The slow aging method is to store the bonded specimens immersed in de-ionized water [127], or artificial saliva [128] for months to years. Some *in vitro* research studies had performed

aging using an accelerated method, such as thermo-cycling [3], cyclic loading [129] or storage of the sectioned specimens in 10% NaOCl solution for 5 h [130].

Bitter et al. [3] followed a protocol of thermo-cycling (for 5000 cycles) of the bonded specimens in deionized water from 5 to 55 °C with an immersion time of 30 s in each bath and a transfer time of 2 s. It has been reported that this aging method could replicate the thermal stresses that occurs in the oral cavity. Albaladejo et al. [129] showed that cyclic loading method of artificial aging could cause micro-cracks at the resin-dentine interfaces. Rathke et al. [131] concluded that longer storage of the bonded specimens in combination with thermo-cycling and/or mechanical loading promoted degradation of the bonded interface, which in turn decreased the longevity of the bond.

7.2 *In vivo* studies on bonding fibre post to root canal dentine

There are very few studies in the literature that have evaluated the clinical performances of fibre post bonded to root dentine. Fredriksson et al. [132] from his retrospective analysis for 2-3 years concluded that teeth restored with carbon fibre posts only had a 2% failure rate. While Glazer [133], from his 28 months prospective study, showed that the teeth restored with carbon fibre posts had a failure rate of 7.7%. Ferrari et al. [134] reported an overall low failure rate of 3.2% for teeth restored with fibre posts. However, the failures due to final restorations were not accounted in the study.

Conversely, King et al. [135] in their prospective study of 87 months' duration demonstrated an inferior performance of teeth restored with fibre posts (71% survival rate), when compared to those restored with cast posts (89% survival rate). However, as with the study by Ferrari et al. [134], the authors failed to account for the failures due to the final restorations. It is

impossible to draw valid conclusions from the limited number of clinical studies because of the low level of evidence. Hence, well-designed controlled clinical studies with long term follow-up are required to truly assess the clinical performances of teeth restored with fibre posts bonded to root dentine with resin adhesives and luting cements.

8. Degradation of resin-root dentine bonded interface

In the last decade, *in vitro* studies have identified that resin-dentine interface bonded with contemporary resin adhesive systems degraded with time. Degradation occurs from hydrolysis of simplified resin adhesives due to continuous absorption of water even after polymerization [136,137] and collagenolysis of uninfiltated demineralized dentinal collagen fibrils [138].

Hydrolytic degradation of contemporary simplified resin adhesives is due to the presence of high concentration of hydrophilic resin monomers. The endogenous collagenolytic activities of demineralized collagen are from matrix metalloproteinases (MMPs) and cysteine cathepsins (CCs) [5,139]. Tay et al. [140] demonstrated that self-etch adhesives increased the collagenolytic actions of proteases in root dentine. This is an important finding as most of the contemporary resin adhesives used for root dentine bonding belong to the self-etch category.

9. Experimental strategies to improve resin bond durability to root dentine

Various experimental strategies have been developed to improve the longevity of resin bonding to dentine. One of the strategies used was to infiltrate hydrophobic resin adhesive monomers into demineralized dentine as the hydrophobic resin monomers are hydrolytically stable and more durable dentine bonds are formed [141]. After acid etching, the demineralized dentine is made suitable for infiltration of hydrophobic resin monomers. It has

been suggested that saturation of demineralized dentine with ethanol [142] removed the water and thereby facilitated infiltration of hydrophobic resin adhesives into demineralized dentine. Ethanol treatment of demineralized collagen is known to reduce the diameter of the collagen fibrils [143] as well as the water-retaining interfibrillar glycosaminoglycans [144]. Thus, ethanol treatment can be expected not only to help in the dehydration of the demineralized dentine, but also to improve resin infiltration. Sadek et al. [145] also suggested the use of tubular occluding agents along with ethanol treatment of demineralized dentine for successful bonding with a hydrophobic adhesive resin. Subsequent study has demonstrated preservation of bond strength when ethanol wet bonding was used with hydrophobic adhesive resin [146].

Other risk factors in the degradation of resin-dentine hybrid layer are the effects of endogenous proteolytic enzymes. Reynolds and Meikle [147] explained that in a healthy state, the tissue inhibitors of MMPs (TIMPS) would check the degradation activity of the MMPs and therefore the tissues would be protected from collagenolytic action. Tay et al. [140] suggested that after pulp extirpation from the root canal and obturation with synthetic materials, the TIMPS were lost and therefore the activities of MMPs were not controlled in a physiological manner. Hence, it becomes important to treat the demineralized dentine with synthetic MMP inhibitors, such as CHX, to prevent degradation of resin-dentine bonded interface [148]. Chlorhexidine has a broad spectrum MMP inhibition potential and it also inhibits dentine matrix-bound cysteine cathepsins [148,149]. Similar to coronal dentine, CHX treatment has been shown to preserve durability of resin bonded to root dentine [150,151,152].

Another experimental strategy to arrest degradation of resin-dentine bond has been to increase the resistance of collagen to degradation by enzymes. Several collagen cross-linkers

have been tested and shown to improve the mechanical properties of demineralized collagen and preserve the bond with adhesive resin. Grape seed extract was shown to improve the resin-dentine bond strength and the mechanical properties of the demineralized dentine collagen matrix because it is rich in proanthocyanidins (PAs) [153,154]. Green et al. [155] have since shown preservation of resin-dentine bond strength by incorporating PA into the adhesive.

Kalra et al. [156] studied the effect of PAs on root dentine and their bonding to resin-based sealer. When demineralized root dentine was treated with PAs, there was an enhanced resistance of the cross-linked collagen fibrils to enzymatic challenge with collagenase. The PA-treated root dentine also showed greater bond strength and bond durability with a resin-based sealer. Recent studies have shown that carbodiimide, a synthetic collagen cross-linker, can also effectively cross-link dentine collagen and inhibited matrix-bound proteases, which in turn leads to preservation of the resin-dentine bond [157,158].

10. Future research recommendations:

There are only few studies in the literature that have evaluated the clinical performances of fibre post bonded to root dentine. It is impossible to draw valid conclusions from these studies because of low level of evidence. Hence, well-designed controlled clinical studies with long term follow-up are required to truly assess the clinical performances of teeth restored with fibre posts bonded to root dentine with resin adhesives and luting cements.

11. Clinical implications:

From the published literature, clinicians need to be aware and understand that root dentine is very different from coronal dentine for bonding of resin-based materials. Hence, the

strategies used for bonding to coronal dentine should not be simply extrapolated to root dentine. If the differences are respected when bonding to root dentine, then an optimum resin bond can be achieved.

12. Conclusions

In conclusion we may say that,

- (1) Several factors such as, presence of sclerotic dentine, very high cavity configuration or 'c' factor, inadequate visibility and access, difficulty in moisture control can pose great challenges in achieving optimum resin bonding to internal root dentine.
- (2) The use of sodium hypochlorite during root canal treatment with 17% EDTA as a final rinse could be a suitable strategy in order to achieve optimum bonding with resin-based materials to internal root dentine.
- (3) The use of zinc-oxide eugenol-based root canal sealers could have a negative effect, whilst calcium hydroxide-based sealers may not have such an effect on bonding of resin-based materials to internal root dentine.
- (4) Bonding of adhesive resins to internal root dentine with residual pulpal remnants on its surface can be severely compromised.
- (5) The use of EDTA as a post space irrigant could enhance the bonding performance when self-etch adhesives are used to bond fibre post to internal root dentine.
- (6) The existing *in vitro* research studies do not support the presented hypothesis that resin-based root canal obturation materials reinforce roots.
- (7) Literature shows mixed results on the bonding performances of various types of commercial adhesives to root dentine. Manufacturer's recommendation must be followed to achieve an optimum bond to root dentin with any type of dentine adhesives.

(8) Resin-root dentine bonded interfaces degrade with time. Several experimental strategies have been recommended to arrest the degradation of resin-root dentine bonded interfaces.

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