Adhesive Dentistry and Endodontics. Part 2: Bonding in the Root Canal System—The Promise and the Problems: A Review

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Abstract

One of the recent trends in endodontics has been the development of bonded obturating materials, in an effort to provide a more effective seal coronally and apically. Materials utilizing dentin adhesive technology have been borrowed from restorative dentistry and adapted to obturating materials. This review discusses the obstacles to effective bonding in the root canal system, the progress that has been made, and possible strategies for improved materials in the future. Much of the literature reviewed and many of the principles discussed are taken from the restorative dentistry literature and applied to the unique environment of the root canal system. *(J Endod 2006;32:1125–1134)*

Key Words

Adhesive dentistry, dentin bonding, endodontic sealers, obturation, radicular dentin

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Microorganisms are the cause of pulpitis and apical periodontitis (1–3) as well as failure in endodontic treatment (3, 4). One goal of endodontic treatment is the reduction or elimination of microorganisms from the root canal system. Complete elimination of microorganisms can not be achieved consistently with current treatment methods, however (5, 6). Therefore, an additional goal of treatment is to seal the root canal system from the outside environment with an obturating material and entomb any residual microorganisms. None of the current dental materials provide a hermetic, leak proof seal, however (7, 8).

Gutta-percha has been the traditional endodontic obturating material, used in combination with a sealer containing zinc oxide and eugenol, calcium hydroxide, or epoxy resin. In recent years obturating materials and sealers have been developed based on dentin adhesion technologies borrowed from restorative dentistry, in an attempt to seal the root canal system more effectively. Also, posts are often bonded in place. Effective bonding in the environment of the root canal system is a challenge, however, because of anatomy and limitations in the physical and mechanical properties of the adhesive materials. A previous article provided an overview of adhesive dentistry as it relates to endodontics and the restoration of access cavities (9). This review will discuss how the unique environment of the root canal system presents a particular challenge for bonded materials, but also has promise for more effective obturating materials. It will discuss the limitations of current materials, the progress that has been made, and possible strategies for the future.

Bonding Resin to Dentin

Current theory of dentin bonding was first described by Nakabayashi et al. in 1982 (10). They described a process that is still used with some of today's adhesive materials. It is a three-step process that allows hydrophobic (water hating) restorative materials to adhere to the wet dentin surface. An acid is applied to the dentin surface and rinsed off, removing the smear layer, demineralizing the superficial dentin, and exposing the collagen matrix. A resinous material, incorporated in a volatile liquid carrier, such as acetone or alcohol, is then applied to the demineralized dentin. The carrier penetrates the moist dentin surface and carries the resinous material into the collagen matrix and dentinal tubules. The dentin is then air dried to evaporate the carrier, leaving the resinous material behind. The volatile liquid/resinous material is known as the primer. An unfilled or lightly filled resin is then applied to the dentin surface and light cured. This material, known as the adhesive, co-polymerizes with the resin already in the collagen matrix, locking it onto the dentin surface (10–13), and providing a hydrophobic surface for co-polymerization with hydrophobic restorative resin materials. The resin infiltrated collagen matrix is commonly referred to as the hybrid layer. With most products, the hybrid layer is between 2 and 5 μm in thickness (14).

Hybridization is the primary process used today to bond hydrophobic restorative resin materials to dentin. Contrary to common belief, the dentinal tubules make only a minor contribution to dentin adhesion. The majority of the retention is provided by micromechanical retention from the collagen matrix in the intertubular dentin (15–17). One study quantified the contribution of the dentinal tubules at 15% (18). Whereas micromechanical retention is believed to be the primary source of retention, there is also a small amount of chemical interaction with dentin with some adhesive systems (19).

Successful dentin adhesives have been available since the late 1980s. At that time most of them utilized the three-step system similar to the one described by Nakabayashi et al.: etch and rinse, primer, adhesive. These are currently described in the dental literature as three-step etch and rinse and are marketed as fourth generation adhesives. Subsequent simplified adhesives were developed that combined some of the steps and are described as two-step etch and rinse or fifth generation; two-step self-etch or sixth generation; and one-step self-etch or seventh generation. These materials all depend on micro-mechanical interlocking from the collagen matrix for retention. The three-step products are still the most effective and simplifying the process by combining steps has generally proven to result in an inferior bond (14, 20–27). Most of the current adhesive research is with the simplified adhesives and they will probably continue to improve with time. The most promising data so far for simplified adhesives has been with the two-step self-etching adhesives (28–29).

The only current resin obturating sealer that utilizes dentin adhesive technology is Epiphany (Pentron Clinical Technologies, Wallingford, CT). It is used like a two-step self-etching (sixth generation) adhesive system. An acidic primer is applied to the dentin surface. It penetrates through the smear layer and demineralizes the superficial dentin. The acid and primer have been combined, eliminating one step in the process. The acidic primer is air dried to remove the volatile carrier and then Epiphany sealer, a dual-cured resin cement is applied and polymerized. The smear layer is incorporated into the hybrid layer.

Adhesive materials are frequently compared using bond strength and microleakage tests. Bond strength refers to the force per unit area required to break the bond between the adhesive material and dentin. It is usually described in megapascals (MPa) that is Newtons per square millimeter. Typical dentin bond strengths reported for adhesive resins are 20 to 30 MPa, although they can be much higher depending on the testing methods. Microleakage is perhaps more important for endodontic applications than bond strength. Even if a material has relatively low bond strength to dentin it may be a good obturating material if it is effective in preventing microleakage. None of the current adhesive materials provide a leak proof seal, however (8, 22, 30–33). A more complete discussion of dentin and enamel bonding can be found in part 1 of this review (9).

The Limitations of Dentin Bonding

From the restorative literature, we know that dentin bonding materials are widely used, but have limitations. Many of the limitations are related to polymerization shrinkage. When resin based materials polymerize, individual monomer molecules join to form chains that contract as the chains grow and intertwine, and the mass undergoes volumetric shrinkage (34). Resin based restorative materials shrink from 2 to 7%, depending on the volume occupied by filler particles and the test method (34–37). The force of polymerization contraction often exceeds the bond strength of dentin adhesives to dentin, resulting in gap formation along the surfaces with the weakest bonds (26, 34, 35, 38–40). Separation often occurs within the hybrid layer (41), but can occur in other areas. Resins in thin layers generate very high forces from polymerization contraction (34, 38, 41, 42).

The root canal system has an unfavorable geometry for resin bonding (43). Configuration factor or C-Factor, the ratio of bonded to unbonded resin surfaces (35), is often used as a quantitative measure of the geometry of the cavity preparation for bonding. The greater the percentage of unbonded surfaces, the less stress is placed on the bonded surfaces from polymerization contraction. The unbonded surfaces allow plastic deformation or flow within the resin mass during polymerization (35, 44). A class 5 cavity preparation, for example, has a favorable geometry with a ratio of approximately 1:1. There are few if any walls that directly oppose each other and approximately one-half of the resin surface is not bonded. In the root canal system, the ratio might be 100:1 (35). Virtually every dentin wall has an opposing wall and there are minimal unbonded surfaces. Any ratio greater than 3:1 is considered unfavorable for bonding (45). Because of this unfavorable geometry, it is not possible to achieve the gap free monoblock suggested in the advertisements of some products. Interfacial gaps are virtually always present in bonded restorations in restorative dentistry (46), obturating materials (47), and bonded posts (48, 49), and gap formation increases with time (50).

Another limitation of dentin bonding is deterioration of the resin bond with time. This is a process that is well documented in vitro (23–26, 51–58) and in vivo (59, 60). Loss of bond strength is first detectable in the laboratory at 3 months (26). Interfacial leakage increases as the bond degrades (61, 62). Functional forces have been shown to contribute to the degradation of the resin bond in restorative applications (26, 63–65). This is also undoubtedly true in the root canal system where torsional and flexural forces stress the dentin/resin interface repeatedly during function and parafunction. Repeated stress causes microfractures and cracks within the resin (26). Unpolymerized resin also contributes to the breakdown of the bond (26). The three-step etch and rinse adhesives exhibit less degradation than the other adhesives (26, 57, 58, 66). To be clinically relevant, published bonding studies should report results with no less than 3 months of aging. For in vitro studies, some method of simulating functional forces should be used.

One of the most important factors in the strength and stability of the resin/dentin bond is the completeness of resin infiltration into the demineralized dentin. If the resin doesn't completely infiltrate, fluid movement between the hybrid layer and unaffected dentin speeds the degradation of the bond (26, 67–70). Water ingress can cause hydrolysis and plasticizing of the resin components. Plasticization is a process in which fluids are absorbed by the resins, causing them to swell, resulting in degradation of their mechanical properties (26). Hydrolysis can break the covalent bonds within collagen fibrils and the resin polymers (51). This process is enhanced by enzymes released by bacteria (71) and from the dentin itself (67). The breakdown products diffuse out of the interfacial area, which weakens the bond, and allows more fluid to ingress. Collagen degradation is thought to occur via host-derived matrix metalloproteinases (MMPs) that are present in dentin and released slowly over time (67). MMPs are also released by bacteria, along with other enzymes (67, 71), but bacteria are not necessary for collagen degradation to occur (67). It is interesting to note that chlorhexidine is an MMP inhibitor that can arrest degradation of the hybrid layer in vivo (72). It may be possible to incorporate MMP inhibitors in future adhesive resin systems.

The adhesive systems that are most effective demineralize to the proper depth and then infiltrate to the full depth of demineralization (51). Prolonged etching times may create a demineralized zone that is too deep for effective resin infiltration (51, 73, 74), resulting in a weaker bond and accelerated degradation. A demineralized zone of about 10 μm was found when a 5-minute soak of MTAD was used before bonding procedures (75), resulting in incomplete resin infiltration. The effectiveness of demineralization/infiltration varies with every dentin adhesive system, which helps to explain the great variability reported in the literature.

Glass-Ionomer Cements

Traditional glass-ionomer cements consist primarily of alumina, silica, and polyalkenoic acid and are self-curing materials. They are the only restorative materials in which the primary bonding mechanism is chemical (76). They form an ionic bond to the hydroxyapatite at the dentin surface (77) and obtain micromechanical retention to the etched surface of the hydroxyapatite crystals (78, 79). Like adhesive resins, glass-ionomer cement loses bond strength over time (79). Some glass-ionomer materials possess antimicrobial properties (80–82).

When placing glass-ionomer cements, the surface is cleaned and then treated with a weak acid (76, 79). The acid removes debris from the dentin surface, removes the smear layer, and exposes hydroxyapatite crystals. It etches the hydroxyapatite, but there is minimal dissolution (76, 78). Because glass-ionomer cements rely on ionic bonding to the hydroxyapatite, strong acids should be avoided because they cause almost total elimination of mineral from the dentin surface (83). Removal of the smear has generally been shown to improve the bond of glass-ionomer cement to dentin (79, 84, 85).

Most of the current glass-ionomer restorative materials contain resin and are referred to as resin modified glass-ionomer materials. They contain a light-cure resin that provides for rapid polymerization on the surface. Most resin modified glass ionomers utilize the same bonding mechanisms as traditional glass ionomers.

Several glass ionomer-obturating materials are available and more are in development. Ketac Endo (3M ESPE, St. Paul, MN), a traditional glass-ionomer material has been around the longest and has a small following.

Is Radicular Dentin Different Than Coronal Dentin?

Several investigators have studied the composition and structure of radicular dentin and found minor differences from coronal dentin. In the apical one-third of the root, there are fewer dentinal tubules (86–88) and consequently, less resin tag formation during bonding procedures (87, 89). This is potentially a positive feature if the adhesive materials can be applied effectively, because more intertubular dentin is available for hybridization (87). As previously stated, resin tags make only a minor contribution to bond strength (15–18). In some apical areas the dentin is irregular and devoid of tubules (86). After bonding procedures, the hybrid layer was found to be thinner in the apical areas by some authors (87, 90, 91) and no difference was found by others (89, 92, 93). Results varied depending on the products used (89). These differences appear to be of little importance because thickness of the hybrid layer has not been shown to influence adhesive capacity (21, 94, 95). Some authors have reported higher bond strengths to dentin in the apical one-third (88, 96, 97), some have reported lower bond strengths (91, 98, 99), and some have reported little difference (90, 93, 100–104). The results vary depending on the adhesive system used (90, 91, 93, 101, 103). Two studies reported higher bond strengths in the pulp chamber than the cervical dentin (105, 106). High initial bond strengths (23.5 MPa) are achievable with radicular dentin (90), and are comparable to those reported for coronal dentin. A recent article reported that radicular dentin in the apical third is often sclerotic and the tubules are filled with minerals that resemble those from peritubular dentin (107). This process starts in the third decade of life and progresses in an apical-coronal direction. It is a potential impediment to effective dentin adhesion and will require further investigation, but as long as there is adequate intertubular dentin available, it may not prove to be a significant finding. At this point in time, viewing the literature as a whole, there appear to be no compositional or structural impediments to bonding to radicular dentin.

How is the Endodontic Environment Different for Bonding Than the Restorative Environment?

In addition to the unfavorable geometry, as previously discussed, there are several other factors that make bonding in the root canal system a challenge.

The Problems of Using Adhesive Materials Deep in the Root Canal System

Performing the bonding steps is problematic deep in the root canal system. Uniform application of a primer or adhesive in the apical one-third is difficult at best and the primer must be applied properly for effective bonding. Once the primer is applied, the volatile carrier must be evaporated. This can also be problematic in the apical one-third. It is difficult, and probably a bad idea, to blow air into the apical one-third. Application and drying of the primer with paper points, as recommended by at least one manufacturer, is probably not very effective for either task. If the acetone or alcohol carrier is not completely removed, the bond is adversely affected (108). An in vitro post study by Bouillaguet, et al. (98) reported lower bond strengths were achieved bonding in the root canal system than to flat prepared samples of radicular dentin. These results are not surprising for the reasons previously discussed. More effective methods must be developed to deliver the acid and primer deep in the root canal system and to remove the volatile carrier. In teeth with small, complex anatomy, this may not be possible.

Contact between components in adhesive materials and the apical tissues is a concern. The affects are unknown from extrusion of solvents such as acetone, unpolymerized resin or HEMA (hydroxyethyl methacrylate, which can be hyperallergenic and is contained in many primers).

The Problems With Dual-Cured and Self-Cured Resins

Because penetration with a curing light is limited in the root canal system, dual-cured or self-cured resin adhesives must be used. Dual-cured resins contain components that provide rapid light polymerization in those areas where the curing light penetrates effectively and a slower chemical polymerization in those areas where the light is not effective. Adhesives and sealers that contain a self-cure component are a mixed blessing, however.

On the plus side, the slower polymerization process allows the material to flow in the pregel stage, which provides some stress relief from polymerization contraction at the resin/dentin interface (40, 109). Self-cured resins have less conversion of monomer to polymer than light-cured resins, which lessens the forces from polymerization contraction (40) and air bubbles, incorporated into the resin during the mixing process, provide a stress relief mechanism (110) by increasing the surface area of resin that is not bonded to dentin (41). Of course, unpolymerized resin and air bubbles have negative effects on the mechanical properties and chemical stability of the resin.

From the restorative literature, we know that the self-etching adhesive systems generally have low bond strengths when used with self-cured composites or dual-cured composites that have not been light activated (104, 111–114). This varies somewhat by product, however (103), and was not a universal finding (101). Reduced bond strengths with self-etching materials have also been reported with bonded posts (115). There are two aspects to this problem.

Self-cured resins contain tertiary amines in the catalyst, which initiate the polymerization reaction and have a high pH. Loss of bond strength may result when an acidic primer is used. Because the acid is not rinsed off after application, residual acid can partially neutralize the high pH amines in the self-cured component of the adhesive or sealer, making them less effective in the chemical polymerization process

(112, 113, 116, 117). The buffering properties of dentin help to lessen this effect, especially with the weak self-etching primers. Dual-cured composites exhibit bond strengths comparable to light-cured composites in the areas that are effectively light-cured (104), because they are not dependent on the basic amines for polymerization.

The second problem with the self-etching adhesive systems when used with self- or dual-cured resins is that they are highly hydrophilic and act as permeable membranes. The chemical polymerization process is slow. Epiphany sealer, for example, takes 30 minutes to polymerize in the deep, self-cured areas (118). Extended setting time for self-cured resins is beneficial for stress relief, but the prolonged time allows diffusion of moisture from the dentin through the hydrophilic primer, which creates water blisters along the interface with the slow polymerizing resins. This moisture contamination reduces bond strength and facilitates leaching of water-soluble components from the resin, which may further contribute to the breakdown of the bond (20, 113, 116, 119–121). This phenomenon occurs in vitro and in vivo (119), and in vital as well as endodontically treated teeth (121). It is not a problem in the areas that are light polymerized (104) or with the three-step etch and rinse adhesives (121). Polymerization of an unfilled resin layer over the acidic primer reduces the problems of permeability (122).

The Problems With Irrigating Solutions and Medicaments

Sodium hypochlorite is commonly used as an endodontic irrigant because of its antimicrobial and tissue dissolving properties. It causes alterations in cellular metabolism in microorganisms and destruction of phospholipids and degradation of lipids and fatty acids. Its oxidative actions cause deactivation of bacterial enzymes (123). It is an ideal endodontic irrigant in many ways, but causes problems when used with adhesive resins. Because it is a strong oxidizing agent, it leaves behind an oxygen rich layer on the dentin surface that results in reduced bond strengths (124–130), and increased microleakage (131). Oxygen is one of the many substances that inhibit the polymerization of resins (132). The oxygen rich dentin surface is probably an important reason for the low bond strengths reported for adhesive resin sealers. It is possible to achieve normal, high bond strengths (23.5 MPa) to radicular dentin under ideal conditions (90), as opposed to the low bond strengths reported for adhesive endodontic sealers ($<$6 MPa). One possible solution to this problem is the application of a reducing agent to the dentin after sodium hypochlorite irrigation. Reducing agents such as ascorbic acid and sodium ascorbate are reported to reverse the negative affects of sodium hypochlorite (124, 128, 131).

Other materials that are applied to dentin during endodontic procedures have been tested for their effects on bonding. Not surprisingly, hydrogen peroxide leaves behind an oxygen rich surface that inhibits bonding (126, 127). Reduced bond strengths were also reported after the use of RC prep (Premier Dental Products, Plymouth Meeting, PA) (125). Electrochemically activated water has gained a following as an irrigating solution. One of the active ingredients is hypochlorous acid, a strong oxidizing agent also found in sodium hypochlorite (133). No loss of bond strength is reported from chlorhexidine use with resins (126, 134, 135) or resin-modified glass-ionomer materials (136). Caries detector did not affect resin bond strengths (137, 138), but chloroform and halothane cause significant loss of bond strength (139). Sodium hypochlorite and ethylenediaminetetracetic acid (EDTA) have also been shown to degrade the mechanical properties of dentin (140–142). Problems posed by certain irrigating solutions and medicaments must be overcome if resin bonding is to be effective in the root canal system.

Eugenol

Eugenol is one of many substances that inhibits the polymerization reaction of resins (143) and can interfere with bonding (97, 102). Eugenol containing endodontic sealers can be a problem with bonded posts. The effects of the eugenol can be minimized if the proper procedures are followed, however. The canal walls should be cleaned mechanically and then scrubbed with alcohol or a detergent to remove all visible signs of sealer. Sealers and temporary cements leave behind an oily layer of debris that must be removed before bonding procedures (144, 145). Once the dentin surface is clean, an etch and rinse adhesive system should be used. The strong acid demineralizes the dentin surface to a depth of about 5 μm and removes the eugenol rich layer. Studies have shown that the three-step etch and rinse procedure allows effective bonding to eugenol contaminated dentin surfaces (146, 147). An etch and rinse adhesive system should be used, because the self-etching systems incorporate the eugenol rich smear layer into the hybrid layer, rather than removing it. Eugenol has no effect on glass-ionomer cements (148).

Other Barriers to Effective Bonding

Effective dentin bonding requires a surface that is free of debris and remnants of the pulp. Studies have shown that significant portions of the canal walls are not touched by endodontic instruments in the shaping process (149, 150) and our irrigants are not totally effective in those unprepared areas either (6). Dentin surfaces that are covered with debris and remnants of pulp tissue are not likely to achieve effective bonding.

Calcium hydroxide paste is sometimes placed in the root canal system between appointments for its antimicrobial properties and other desirable effects. It is not possible to remove all the calcium hydroxide from the root canal system, however, before obturation (151–153). Concerns have been expressed that residual calcium hydroxide paste could prevent effective bonding in some areas; that it can act as a physical barrier, and that the high pH may act to neutralize the acid primer in self-etching adhesives. A recent article by Wang et al. (154) reported no difference in microleakage with Resilon between teeth with and without the use of calcium hydroxide as an intermediate dressing. Further research is needed, however, to confirm their findings.

Some clinicians use alcohol as a final rinse to aid in drying the canals. Most dentin adhesive systems need moisture present in superficial dentin to be effective (14), so a final alcohol rinse is not recommended with an adhesive resin sealer.

Retreatment

Retreatment is always a concern with a new material. Resilon is soluble in chloroform and other solvents, and several studies show it is easily removed by a variety of methods (155–157). Epiphany, on the other hand, like other resins, is not soluble in the solvents commonly used in dentistry. Removal of resin sealers from fins and accessory canals or deep bifurcated canals is difficult. Removing bonded resin is likely to be that much more difficult.

Does Removal of the Smear Layer Matter?

For a relatively small issue, this question has been studied extensively and remains somewhat controversial. Removal of the smear layer has generally been shown to increase bond strength to dentin for glass-ionomer materials (79, 84, 85) and unbonded resin materials (158), although the bond strengths are still quite low (85, 158). Removal of the smear layer is reported to reduce microleakage for most sealers (159–163). EDTA has been used for many years in endodontics for this function (164). Acids work equally well (142). Care must be used not to over treat the dentin surface, however (142). Removal of the smear layer has additional benefit in infected teeth because bacteria are one of its components.

Current Resin and Glass-Ionomer Obturating Materials

AH 26 and AH Plus

AH 26, which was later modified to AH Plus, have been available as root canal sealers for many years. Both are described as epoxy-resin sealers. They are generally placed in the canal without any dentin preparation or dentin adhesive and can be used with any obturating technique. Their popularity has been due, in part, to the fact that they contain no eugenol, which inhibits the polymerization of resins (143) and can interfere with bonding (97, 102). Low bond strengths are reported for the epoxy resin sealers to gutta-percha (165, 166) and to dentin (6 MPa or less) (158, 165–168). Use of a dentin bonding agent improved bond strength of AH-26 (167, 169) but no improvement was shown in a study with AH Plus and Thermafil (170). In leakage studies, AH 26 and AH Plus generally performed equal to or better than other sealers (47, 162, 171–174), although this finding was not universal (175). Removal of the smear layer was generally found to be advantageous (158, 162, 163, 176). The use of chlorhexidine did not affect the apical seal of AH 26 (177).

EndoRez

EndoRez is an endodontic sealer that is based on the urethane dimethacrylate (UDMA) molecule, similar to many restorative resins. It has additives to make it hydrophilic so it can be used in the wet environment of the root canal system. It is very effective in penetrating the dentin tubules and exhibits initial close adaptation to the dentin. However, gap formation results from polymerization shrinkage (178). EndoRez does not utilize a dentin bonding system. It has been recommended for a single gutta-percha cone technique but can be used with other obturating methods.

Several investigators have evaluated EndoRez. Two studies showed it to be biocompatible (179, 180), whereas another, utilizing a different test, reported it to be somewhat cytotoxic (181). Very low bond strengths to dentin are reported for EndoRez (158), and it performed poorly in leakage studies compared to other sealers (151, 181, 182). It was not found to have antimicrobial properties (183). Coating gutta-percha with resin did not prevent gap formation or leakage (184). A clinical study with EndoRez reported a 91.3% success rate at 14–24 months (185). So far published studies have not shown a particular benefit to using EndoRez over other sealers, but as with most new materials, research is underway to try to improve its performance.

Resilon/Epiphany

Resilon/Epiphany (Pentron) is the only current resin-based obturating system that utilizes a dentin adhesive. Product advertising states that it is more effective than existing obturating materials because it utilizes a resinous obturating material and an adhesive resin sealer, creating a monoblock of dentin/adhesive/obturating material. The Resilon cones consist of is a vinyl polyester material with methacrylate polymer, glass filler particles, and opacifiers added. Its appearance and manipulation are similar to gutta-percha. Epiphany sealer consists of a self-etching primer that is used with a lightly filled dual-cured UDMA sealer/adhesive. Product advertising states that this system can be used with any obturating method.

The three initial published studies were sponsored by the manufacturer. In the first study, obturation with the Resilon system was shown to strengthen the teeth slightly (186). These results are countered by a recent independent study in which Resilon was not found to reinforce immature teeth (187). Williams, et al., reported that neither Resilon nor gutta-percha has adequate stiffness to reinforce teeth (188). Similar minor strengthening effects have also been reported for AH-26 and Ketac Endo (189). It is doubtful that any of these findings are clinically significant. The second company sponsored study reported less leakage in vitro with the Resilon system than with AH-26 after 3 weeks (175). Three weeks is not adequate aging of the specimens, however, and the bonds were not stressed during the storage period. These results are supported by a recent Canadian study (190) but countered by a study by Tay et al. (47) who found no difference in microleakage with the same materials. The third study utilized dogs and compared Resilon/Epiphany with gutta-percha/AH26 that was intentionally contaminated with microorganisms. Teeth obturated with the Resilon system had less periradicular inflammation after 3 months (191).

Recent independent research has been somewhat unfavorable toward Resilon/Epiphany. Tay et al. reported that Resilon was susceptible to alkaline (192) and enzymatic (193) hydrolysis. Surface erosion was evident in Resilon samples in as little as 20 minutes of immersion in an alkaline hydrolyzing agent (192). Similarly, surface erosion and more than 50% weight loss was reported when Resilon was exposed to lipase and esterase for 96 hours (193). Biodegradation by bacterial/salivary enzymes and oral/endodontic bacteria is a concern for the current formulation of Resilon. Because obturating materials are placed in the protected environment of the root canal system, however, it is questionable whether these results have great clinical significance. Versiani et al. reported that Epiphany was outside the acceptable range for solubility and dimensional stability, as described by the ANSI/ADA standards (194), but was within the acceptable range for setting time, flow, and thickness. Melker et al. reported that Resilon was found to exhibit no antimicrobial activity (195), despite the fact that bioactive glass is one of the components and is considered to have antimicrobial properties.

To achieve a monoblock, as advertised by the manufacturer, high bond strengths are necessary between the dentin and sealer, as well as between the sealer and obturating material. Bond strengths of only 4 to 6 MPa have been achieved between Epiphany and dentin (Dr. Martin Trope, personal communication). This is similar to the bond strength reported for fiber posts and resin luting agents (135). Bond strengths of less than 2 MPa are reported between Epiphany and Resilon (168, 196, 197) and one study reported lower bond strengths than with gutta-percha and AH 26 (168). This is not surprising, because unpolymerized resin must be available in both materials to achieve co-polymerization (198). There is no unpolymerized resin in Resilon. A recent study found gaps present in teeth obturated with Resilon/Epiphany as well as gutta-percha/AH 26, and there was no difference in microleakage (47). The gaps in the Resilon/Epiphany group were between Epiphany and the dentin wall. In the other group the gaps were between AH 26 and gutta-percha (47). None of the specimens exhibited a monoblock. These findings challenge the concept of a monoblock and the results of a previous study (186) that Resilon/Epiphany strengthens the tooth.

Ketac Endo

Ketac Endo (ESPE) is a traditional glass-ionomer cement that was developed as an endodontic sealer, but never gained wide acceptance. It offered little benefit in leakage studies (171, 189, 199, 200) and was generally considered difficult to retreat. Several new obturating systems that utilize glass-ionomer materials are currently entering the marketplace, but there is little information and no research available at this time.

Efforts to Overcome the Problems with Resin Adhesives

For adhesive materials to be effective deep in the root canal system, new, innovative delivery methods are needed. Experiments are underway with microbrushes and micropipettes for delivering acidic primers into the apical one-third. Similarly, microsuction may be the answer for removing the volatile primer components, such as acetone, from the apical one-third.

The problems associated with sodium hypochlorite must be overcome for resin bonding to be effective in the root canal system. The use of reducing agents, as previously discussed, is one possible solution. Alternative, nonoxidizing irrigants would also eliminate this problem.

The problems posed by dual-cured and self-cured materials must be addressed. Use of the three-step, etch and rinse adhesive systems eliminates most of the problems if it can be delivered effectively. If a self-etching primer is used, the ones that utilize weak acids offer the most promise because residual acid is more effectively neutralized by the buffering effects in dentin than the strong self-etching primers. The problem with moisture permeability can be overcome with adhesive systems that are less hydrophilic and allow polymerization of the sealer without the presence of moisture. With current materials, these are the variables that need to be manipulated to optimize bonding in the root canal system.

Because polymerization shrinkage is a big part of the problem, development of shrink free obturating materials would go a long way toward a more effective seal. Research has been underway since the 1980s to develop shrink free restorative composites (201–205). Nonshrinking resins would allow the adhesive bond to mature without stress and obviate the need for high bond strengths. Even after years of research into nonshrinking resins, none have made it to the market. However, as the physical demands on obturating materials is less than on restorative materials, development of non-shrinking resin obturating materials may be more achievable. A small amount of setting expansion, in fact, would enhance the sealing properties of the material.

The ultimate obturating material would be self adhesive, eliminating the need for a separate adhesive system and its associated problems. The glass ionomers are the only self-adhesive materials currently available in dentistry but so far, they lack desirable properties for an obturating material.

Discussion

A limited amount of research evidence has been published about bonding in the root canal system. Most of the knowledge about adhesion to dentin has been published in the restorative literature and relates to coronal dentin. An effort has been made in this review to extrapolate that knowledge to highlight the problems that can be expected in endodontic applications.

In the age of adhesive endodontic products, much ado has been made about gutta-percha substitutes. However, like gutta-percha, their primary function is to occupy space. The more important issue is the sealer and its properties. The ideal obturating material would provide a monoblock, in which the root becomes a perfectly sealed, stable, solid mass with no gaps. If the properties of the sealer could be optimized, this might be possible. The ideal sealer might even eliminate the need for a gutta-percha substitute. The ideal obturating material would possess the following properties:

1. Easy to manipulate.
2. Amenable to different obturating methods.
3. Stable in the oral environment.
4. Radiopaque.
5. Biocompatible.
6. Antimicrobial.
7. Nonshrinking or expands 0.5% during polymerization.
8. Self-adhesive.
9. Forms a stable bond to dentin that does not degrade with time and function.
10. Forms a bond that is not affected by oxidizing agents like sodium hypochlorite.
11. Strengthens the tooth.
12. Easily removed for post placement or retreatment.

Unfortunately, we are not close to development of an obturating material that meets all these criteria. Some current research is focused on adding ingredients that expand to the current methacrylate based materials like UDMA, to offset polymerization shrinkage. Other researchers are working on obturating materials that are entirely new.

Recent emphasis in endodontics has been on developing adhesive resin sealers, but glass-ionomer materials have several advantages over resins. They possess 9 of 12 of the above characteristics of the ideal sealer. They are more dimensionally stable during the setting reaction than resins and do not generate high forces from polymerization contraction, so C-factor is not a big issue. From a biohazard standpoint, glass-ionomer materials do not contain components such as acetone or HEMA. The biggest drawback to current glass-ionomer materials is their hardness, making retreatment difficult.

When observing the development of new adhesive products, several things should be considered. With most dental products, initial company sponsored research tends to be favorable, but independent research tends to provide a more accurate picture of a product's strengths and weaknesses. Scanning electron microscope (SEM) depictions of the bonded interface can be misleading. Although it is possible to find areas that depict a perfect interface from virtually any specimen with the SEM, interfacial gaps are always present somewhere in the specimen with current materials (46–48). To be clinically relevant, published bonding studies should report results with at least 3 months of aging. For in vitro studies, some method should be used to simulate functional forces. If these minimal criteria are not met, the results should be viewed with skepticism.

Adhesive obturating materials are in the early stages of development. Although none of the current materials appear to offer a big advantage over traditional obturating materials, none are likely to come to a disastrous end like Hydron (Hydron Technologies, Inc., Pampano Beach, FL) in the 1980s (206). Current adhesive resins used in endodontics are based on restorative resins that have been used clinically for almost 20 years. Furthermore, resin sealers such as AH-26 have produced clinical success for almost 30 years.

There are a number of reasons to consider using the new obturating materials. Resilon, for example, handles like gutta-percha and manipulates easily. It is highly radiopaque and provides a radiographic look that some clinicians find desirable. Some endodontists use adhesive materials to market their practices to restorative dentists who consider themselves to be bondodontists. These potential benefits must be weighed against the additional clinical steps necessary to use adhesive materials, and the additional cost.

Although adhesive obturating materials have greater potential than traditional materials, at this point in their development there is no clear benefit to their use. However, continued research and development is likely to result in improvements and in new, more effective materials. The principles discussed in this review may be used to evaluate their progress. (100)

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References

1. Kakehashi S, Stanley HR, Fitzgerald RJ. The effects of surgical exposures of dental pulps in germ-free and conventional laboratory rats. Oral Surg Oral Med Oral Path 1965;20:340–9.
2. Siqueira JF Jr, Rocas IN, Favieri A, Abad EC, Castro AJ, Gahyva SM. Bacterial leakage in coronally unsealed root canals obturated with 3 different techniques. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2000;90:647–50.
3. Siqueira JF Jr. Aetiology of root canal treatment failure and why well-treated teeth can fail. Int Endod J 2001;34:1–10.
4. Lin LM, Skribner JE, Gaengler P. Factors associated with endodontic treatment failures. J Endod 1992;18:625–7.
5. Sjogren U, Figdor D, Persson S, Sundqvist G. Influence of infection at the time of root filling on the outcome of endodontic treatment of teeth with apical periodontitis. Int Endod J 1997;30:297–306.
6. Nair PN, Henry S, Cano V, Vera J. Microbial status of apical root canal system of human mandibular first molars with primary apical periodontitis after "one-visit" endodontic treatment. Oral Surg Oral Med Oral Pathol Oral Radiol Endod 2005;99:231–52.
7. Hilton TJ. Can modern restorative procedures and materials reliably seal cavities? In vitro investigations: part 1. Am J Dent 2002;15:198–210.
8. Hilton TJ. Can modern restorative procedures and materials reliably seal cavities? In vitro investigations: part 2. Am J Dent 2002;15:279–89.
9. Schwartz RS, Fransman R. Adhesive dentistry and endodontics: materials, clinical strategies and procedures for restoration of access cavities: a review. J Endod 2005;31:151–65.
10. Nakabayashi N, Kojima K, Masuhara E. The promotion of adhesion by the infiltration of monomers into tooth substrates. J Biomed Mater Res 1982;16:265–9.
11. Hansen SE, Swift EJ. Microleakage with Gluma: effects of unfilled resin polymerization and storage time. Am J Dent 1989;2:266–8.
12. Erickson RL. Surface interactions of dentin adhesive materials. Oper Dent 1992;(Suppl 5):81–3.
13. Crim GA. Prepolymerization of Gluma 4 sealer: effect on bonding. Am J Dent 1990;3:25–7.
14. Van Meerbeek B, De Munck J, Yoshida Y, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent 2003;28:215–35.
15. Tagami J, Tao L, Pashley DH. Correlation among dentin depth, permeability, and bond strength of adhesive resin. Dent Mater 1990;6:45–50.
16. Tao L, Pashley DH. Shear bond strengths to dentin: effects of surface treatments, depth and position. Dent Mater 1988;4:371–8.
17. Pereira PN, Okuda M, Sano H, Yoshikawa T, Burrow MF, Tagami J. Effect of intrinsic wetness and regional difference on dentin bond strength. Dent Mater 1999;15:46–53.
18. Gwinnett AJ. Quantitative contribution of resin infiltration/hybridization to dentin bonding. Am J Dent 1993;6:7–9.
19. Yoshida Y, Nagakane K, Fukuda R, et al. Comparative study on adhesive performance of functional monomers. J Dent Res 2004;83:454–8.
20. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic? J Can Dent Assoc 2003;69:726–31.
21. Inoue S, Vargas MA, Abe Y, et al. Microtensile bond strength of eleven contemporary adhesives to dentin. J Adhes Dent 2001;3:237–45.
22. Fabianelli A, Goracci C, Ferrari M. Sealing ability of packable resin composites in class II restorations. J Adhes Dent 2003;5:217–23.
23. De Munck J, Van Meerbeek B, Yoshida Y, et al. Four-year water degradation of total-etch adhesives bonded to dentin. J Dent Res 2003;82:136–40.
24. Armstrong SR, Vargas MA, Fang Q, Laffoon JE. Microtensile bond strength of a total-etch 3-step, total-etch 2-step, self-etch 2-step, and a self-etch 1-step dentin bonding system through 15-month water storage. J Adhes Dent 2003;5:47–56.
25. Shirai K, De Munck J, Yoshida Y, et al. Effect of cavity configuration and aging on the bonding effectiveness of six adhesives to dentin. Dent Mater 2005;21:110–24.
26. De Munck J, Van Landuyt K, Peumans M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. J Dent Res 2005;84:118–32.
27. Peumans M, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B. Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent Mater 2005;21:864–81.
28. Van Meerbeek B, Kanumilli P, De Munck J, Van Landuyt K, Lambrechts P, Peumans M. A randomized controlled study evaluating the effectiveness of a two-step self-etch adhesive with and without selective phosphoric-acid etching of enamel. Dent Mater 2005;21:375–83.
29. Inoue S, Van Meerbeek B, Vargas M. Adhesion mechanism of self-etching adhesives. In: Tagami J, Toledano M, Prati C, eds. Third International Kuraray Symposium of Advanced Dentistry, 3–4 December 1999, Granada, Spain. Como, Italy: Graphice Erredue, 2000.
30. Pilo R, Ben-Amar A. Comparison of microleakage for three one-bottle and three multiple-step dentin bonding agents. J Prosthet Dent 1999;82:209–13.
31. Bouillaguet S, Duroux B, Ciucchi B, Sano H. Ability of adhesive systems to seal dentin surfaces: an in vitro study. J Adhes Dent 2000;2:201–8.
32. Ceballos L, Osorio R, Toledano M, Marshall GW. Microleakage of composite restorations after acid or Er-YAG laser cavity treatments. Dent Mater 2001;17:340–6.
33. Ozturk B, Özer F, Belli S. An in vitro comparison of adhesive systems to seal pulp chamber walls. Int Endod J 2004;37:297–306.
34. Feilzer AJ, de Gee AJ, Davidson CL. Curing contraction of composites and glass ionomer cements. J Prosthet Dent 1988;59:297–300.
35. Carvalho RM, Pereira JC, Yoshiyama M, Pashley DH. A review of polymerization contraction: the influence of stress development versus stress relief. Oper Dent 1996;21:17–24.
36. Bayne SC, Thompson JY, Swift EJ Jr, Stamatiades P, Wilkerson M. A characterization of first-generation flowable composites. J Am Dent Assoc 1998;129:567–77.
37. Labella R, Lambrechts P, Van Meerbeek B, Vanherle G. Polymerization shrinkage and elasticity of flowable composites and filled adhesives. Dent Mater 1999;15:128–37.
38. Feilzer AJ, de Gee AJ, Davidson CL. Setting stress in composite resin in relation to configuration of the restoratives. J Dent Res 1987;66:1636–9.
39. Perdigao J, Lambrechts P, Van Meerbeek B, et al. The interaction of adhesive systems with human dentin. Am J Dent 1996;9:167–73.
40. Braga RR, Ferracane JL, Condon JR. Polymerization contraction stress in dual-cure cements and its effect on interfacial integrity of bonded inlays. J Dent 2002;30:333–40.
41. Feilzer AJ, de Gee AJ, Davidson CL. Increased wall-to-wall curing contraction in thin bonded resin layers. J Dent Res 1989;68:48–50.
42. Alster D, Feilzer AJ, de Gee AJ, Davidson CL. Polymerization contraction stress in thin resin composite layers as a function of layer thickness. Dent Mater 1997;13:146–50.
43. Tay FR, Loushine RJ, Lambrechts P, Weller RN, Pashley DH. Geometric factors affecting dentin bonding in root canals: a theoretical modeling approach. J Endod 2005;31:584–9.
44. Davidson CL, de Gee AJ. Relaxation of polymerization contraction stress by flow in dental composites. J Dent Res 1984;63:146–8.
45. Yoshikawa T, Sano H, Burrow MF, Tagami J, Pashley DH. Effects of dentin depth and cavity configuration on bond strength. J Dent Res 1999;78:898–905.
46. Hannig M, Friedrichs C. Comparative in vivo and in vitro investigation of interfacial bond variability. Oper Dent 2001;26:3–11.
47. Tay FR, Loushine RJ, Weller RN, et al. Ultrastructural evaluation of the apical seal in roots filled with a polycaprolactone-based root canal filling material. J Endod 2005;31:514–9.
48. Goracci C, Fabianelli A, Sadek FT, Papacchini F, Tay FR, Ferrari M. The contribution of friction to the dislocation resistance of bonded fiber posts. J Endod 2005;31:608–12.
49. Pirani C, Chersoni S, Foschi F, et al. Does hybridization of intraradicular dentin really improve fiber post retention in endodontically treated teeth? J Endod 2005;31:891–4.
50. Roulet JF. Marginal integrity: clinical significance. J Dent 1994;22(Suppl 1):S9–12.
51. Hashimoto M, Ohno H, Endo K, Kaga M, Sano H, Oguchi H. The effect of hybrid layer thickness on bond strength: demineralized dentin zone of the hybrid layer. Dent Mater 2000;16:406–11.
52. Hashimoto M, Ohno H, Sano H, Kaga M, Oguchi H. Degradation patterns of different adhesives and bonding procedures. J Biomed Mater Res 2003;66B:324–30.
53. Hashimoto M, Ohno H, Sano H, Kaga M, Oguchi H. In vitro degradation of resin-dentin bonds analyzed by microtensile bond test, scanning and transmission electron microscopy. Biomaterials 2003;24:3795–803.
54. Hashimoto M, Ohno H, Sano H, et al. Micromorphological changes in resin-dentin bonds after 1 year of water storage. J Biomed Mater Res 2002;63:306–11.
55. Armstrong SR, Vargas MA, Chung I, et al. Resin-dentin interfacial ultrastructure and microtensile dentin bond strength after five-year water storage. Oper Dent 2004;29:705–12.
56. de Oliveira Carrilho MR, Tay FR, Pashley DH, Tjaderhane L, Carvalho RM. Mechanical stability of resin-dentin bond components. Dent Mater 2005;21:232–41.
57. De Munck J, Braem M, Wevers M, et al. Micro-rotary fatigue of tooth-biomaterial interfaces. Biomaterials 2005;26:1145–53.
58. Frankenberger R, Strobel WO, Kramer N, et al. Evaluation of the fatigue behavior of the resin-dentin bond with the use of different methods. J Biomed Mater Res B Appl Biomater 2003;67:712–21.

59. Hashimoto M, Ohno H, Kaga M, Endo K, Sano H, Oguchi H. In vivo degradation of resin-dentin bonds in humans over 1 to 3 years. J Dent Res 2000;79:1385–91.
60. Hashimoto M, Ohno H, Kaga M, Endo K, Sano H, Oguchi H. Resin-tooth adhesive interfaces after long-term function. Am J Dent 2001;14:211–5.
61. Okuda M, Pereira PN, Nakajima M, Tagami J, Pashley DH. Long-term durability of resin dentin interface: nanoleakage vs. microtensile bond strength. Oper Dent 2002;27:289–96.
62. Okuda M, Pereira PN, Nakajima M, Tagami J. Relationship between nanoleakage and long-term durability of dentin bonds. Oper Dent 2001;26:482–90.
63. Jang KT, Chung DH, Shin D, Garcia-Godoy F. Effect of eccentric load cycling on microleakage of Class V flowable and packable composite resin restorations. Oper Dent 2001;26:603–8.
64. Kubo S, Yokota H, Sata Y, Hayashi Y. The effect of flexural load cycling on the microleakage of cervical resin composites. Oper Dent 2001;26:451–9.
65. Frankenberger R, Pashley DH, Reich SM, Lohbauer U, Petschelt A, Tay FR. Characterisation of resin-dentine interfaces by compressive cyclic loading. Biomaterials 2005;26:2043–52.
66. Frankenberger R, Strobel WO, Lohbauer U, Kramer N, Petschelt A. The effect of six years of water storage on resin composite bonding to human dentin. J Biomed Mater Res B Appl Biomater 2004;69:25–32.
67. Pashley DH, Tay FR, Yiu C, et al. Collagen degradation by host-derived enzymes during aging. J Dent Res 2004;83:216–21.
68. Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG, Pashley DH. Nanoleakage: leakage within the hybrid layer. Oper Dent 1995;20:18–25.
69. Suppa P, Breschi L, Ruggeri A, et al. Nanoleakage within the hybrid layer: a correlative FEISEM/TEM investigation. J Biomed Mater Res B Appl Biomater 2005; 73:7–14.
70. Paul SJ, Welter DA, Ghazi M, Pashley D. Nanoleakage at the dentin adhesive interface vs microtensile bond strength. Oper Dent 1999;24:181–8.
71. Santerre JP, Shajii L, Leung BW. Relation of dental composite formulations to their degradation and the release of hydrolyzed polymeric-resin-derived products. Crit Rev Oral Biol Med 2001;12:136–51.
72. Hebling J, Pashley DH, Tjaderhane L, Tay FR. Chlorhexidine arrests subclinical degradation of dentin hybrid layers in vivo. J Dent Res 2005;84:741–6.
73. Pioch T, Stotz S, Buff E, Duschner H, Staehle HJ. Influence of different etching times on hybrid layer formation and tensile bond strength. Am J Dent 1998;11:202–6.
74. Van Landuyt KL, Kanumilli P, De Munck J, Peumans M, Lambrechts P, Van Meerbeek B. Bond strength of a mild self-etch adhesive with and without prior acid-etching. J Dent 2006;34:77–85.
75. Tay FR, Hosoya Y, Loushine RJ, Pashley DH, Weller RN, Low DC. Ultrastructure of intraradicular dentin after irrigation with BioPure MTAD. II. The consequence of obturation with an epoxy resin-based sealer. J Endod 2006;32:473–7.
76. Inoue S, Van Meerbeek B, Abe Y, et al. Effect of remaining dentin thickness and the use of conditioner on micro-tensile bond strength of a glass-ionomer adhesive. Dent Mater 2001;17:445–55.
77. Yoshida Y, Van Meerbeek B, Nakayama Y, et al. Evidence of chemical bonding at biomaterial-hard tissue interfaces. J Dent Res 2000;79:709–14.
78. Yip HK, Tay FR, Ngo HC, Smales RJ, Pashley DH. Bonding of contemporary glass ionomer cements to dentin. Dent Mater 2001;17:456–70.
79. De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Suzuki K, Lambrechts P. Four-year water degradation of a resin-modified glass-ionomer adhesive bonded to dentin. Eur J Oral Sci 2004;112:73–83.
80. Perez CR, Hirata R Jr, Sergio PP. Evaluation of antimicrobial activity of fluoride-releasing dental materials using a new in vitro method. Quintessence Int 2003; 34:473–7.
81. Boeckh C, Schumacher E, Podbielski A, Haller B. Antibacterial activity of restorative dental biomaterials in vitro. Caries Res 2002;36:101–7.
82. Coogan MM, Creaven PJ. Antibacterial properties of eight dental cements. Int Endod J 1993;26:355–61.
83. Van Meerbeek B, Conn LJ Jr, Duke ES, Eick JD, Robinson SJ, Guerrero D. Correlative transmission electron microscopy examination of nondemineralized and demineralized resin-dentin interfaces formed by two dentin adhesive systems. J Dent Res 1996;75:879–88.
84. Berry EA 3rd, Powers JM. Bond strength of glass ionomers to coronal and radicular dentin. Oper Dent 1994;19:122–6.
85. Weiger R, Heuchert T, Hahn R, Lost C. Adhesion of a glass ionomer cement to human radicular dentine. Endod Dent Traumatol 1995;11:214–9.
86. Mjor IA, Smith MR, Ferrari M, Mannocci F. The structure of dentine in the apical region of human teeth. Int Endod J 2001;34:346–53.
87. Ferrari M, Mannocci F, Vichi A, Cagidiaco MC, Mjor IA. Bonding to root canal: structural characteristics of the substrate. Am J Dent 2000;13:255–60.
88. Mannocci F, Pilecki P, Bertelli E, Watson TF. Density of dentinal tubules affects the tensile strength of root dentin. Dent Mater 2004;20:293–6.
89. Bitter K, Paris S, Martus P, Schartner R, Kielbassa AM. A Confocal Laser Scanning Microscope investigation of different dental adhesives bonded to root canal dentine. Int Endod J 2004;37:840–8.
90. Yoshiyama M, Carvalho RM, Sano H, Horner JA, Brewer PD, Pashley DH. Regional bond strengths of resins to human root dentine. J Dent 1996;24:435–42.
91. Yoshiyama M, Matsuo T, Ebisu S, Pashley D. Regional bond strengths of self-etching/self-priming adhesive systems. J Dent 1998;26:609–16.
92. Mannocci F, Innocenti M, Ferrari M. Stereomicroscopic and scanning electron microscopic study of roots obturated with vertically condensed gutta-percha, epoxy resin cement, and dentin bonding agent. J Endod 1998;24:397–400.
93. Burrow MF, Sano H, Nakajima M, Harada N, Tagami J. Bond strength to crown and root dentin. Am J Dent 1996;9:223–9.
94. Prati C, Chersoni S, Mongiorgi R, Pashley DH. Resin-infiltrated dentin layer formation of new bonding systems. Oper Dent 1998;23:185–94.
95. Tay FR, Sano H, Carvalho R, Pashley EL, Pashley DH. An ultrastructural study of the influence of acidity of self-etching primers and smear layer thickness on bonding to intact dentin. J Adhes Dent 2000;2:83–98.
96. Gaston BA, West LA, Liewehr FR, Fernandes C, Pashley DH. Evaluation of regional bond strength of resin cement to endodontic surfaces. J Endod 2001;27:321–4.
97. Muniz L, Mathias P. The influence of sodium hypochlorite and root canal sealers on post retention in different dentin regions. Oper Dent 2005;30:533–9.
98. Bouillaguet S, Troesch S, Wataha JC, Krejci I, Meyer JM, Pashley DH. Microtensile bond strength between adhesive cements and root canal dentin. Dent Mater 2003;19:199–205.
99. Mallmann A, Jacques LB, Valandro LF, Mathias P, Muench A. Microtensile bond strength of light- and self-cured adhesive systems to intraradicular dentin using a translucent fiber post. Oper Dent 2005;30:500–6.
100. Aksornmuang J, Foxton RM, Nakajima M, Tagami J. Microtensile bond strength of a dual-cure resin core material to glass and quartz fibre posts. J Dent 2004; 32:443–50.
101. Foxton RM, Nakajima M, Tagami J, Miura H. Adhesion to root canal dentine using one and two-step adhesives with dual-cure composite core materials. J Oral Rehabil 2005;32:97–104.
102. Ngoh EC, Pashley DH, Loushine RJ, Weller RN, Kimbrough WF. Effects of eugenol on resin bond strengths to root canal dentin. J Endod 2001;27:411–4.
103. Kanno T, Ogata M, Foxton RM, Nakajima M, Tagami J, Miura H. Microtensile bond strength of dual-cure resin cement to root canal dentin with different curing strategies. Dent Mater J 2004;23:550–6.
104. Foxton RM, Nakajima M, Tagami J, Miura H. Bonding of photo and dual-cure adhesives to root canal dentin. Oper Dent 2003;28:543–51.
105. Lopes GC, Cardoso Pde C, Vieira LC, Baratieri LN. Microtensile bond strength to root canal vs pulp chamber dentin: effect of bonding strategies. J Adhes Dent 2004; 6:129–33.
106. Mannocci F, Sherriff M, Ferrari M, Watson TF. Microtensile bond strength and confocal microscopy of dental adhesives bonded to root canal dentin. Am J Dent 2001;14:200–4.
107. Paque F, Luder HU, Sener B, Zehnder M. Tubular sclerosis rather than the smear layer impedes dye penetration into the dentine of endodontically instrumented root canals. Int Endod J 2006;39:18–25.
108. Tay FR, Pashley DH, Yoshiyama M. Two modes of nanoleakage expression in single-step adhesives. J Dent Res 2002;81:472–6.
109. Braga RR, Ferracane JL. Alternatives in polymerization contraction stress management. Crit Rev Oral Biol Med 2004;15:176–84.
110. Alster D, Feilzer AJ, de Gee AJ, Mol A, Davidson CL. The dependence of shrinkage stress reduction on porosity concentration in thin resin layers. J Dent Res 1992;71:1619–22.
111. Cheong C, King NM, Pashley DH, Ferrari M, Toledano M, Tay FR. Incompatibility of self-etch adhesives with chemical/dual-cured composites: two-step vs one-step systems. Oper Dent 2003;28:747–55.
112. Sanares AM, Itthagarun A, King NM, Tay FR, Pashley DH. Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites. Dent Mater 2001;17:542–56.
113. Tay FR, Pashley DH, Yiu CK, Sanares AM, Wei SH. Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites: part I. Single-step self-etching adhesive. J Adhes Dent 2003; 5:27–40.
114. Asmussen E, Peutzfeldt A. Short- and long-term bonding efficacy of a self-etching, one-step adhesive. J Adhes Dent 2003;5:41–5.
115. Goracci C, Sadek FT, Fabianelli A, Tay FR, Ferrari M. Evaluation of the adhesion of fiber posts to intraradicular dentin. Oper Dent 2005;30:627–35.
116. Tay FR, Suh BI, Pashley DH, Prati C, Chuang SF, Li F. Factors contributing to the incompatibility between simplified-step adhesives and self-cured or dual-cured composites: part II. Single-bottle, total-etch adhesive. J Adhes Dent 2003;5:91–105.
117. Ikemura K, Endo T. Effect on adhesion of new polymerization initiator systems comprising 5-monosubstituted barbituric acids, aromatic sulphonate amides, and

tert-butyl peroxymaleic acid in dental adhesive resin. J applied Polymer Sci 1999;72:1655–68.
118. Nielsen BA, Beeler WJ, Vy C, Baumgartner JC. Setting times of Resilon and other sealers in aerobic and anaerobic environments. J Endod 2006;32:130–2.
119. Tay FR, Frankenberger R, Krejci I, et al. Single-bottle adhesives behave as permeable membranes after polymerization. I. In vivo evidence. J Dent 2004;32:611–21.
120. Chersoni S, Suppa P, Grandini S, et al. In vivo and in vitro permeability of one-step self-etch adhesives. J Dent Res 2004;83:459–64.
121. Chersoni S, Acquaviva GL, Prati C, et al. In vivo fluid movement through dentin adhesives in endodontically treated teeth. J Dent Res 2005;84:223–7.
122. Carvalho RM, Pegoraro TA, Tay FR, Pegoraro LF, Silva NR, Pashley DH. Adhesive permeability affects coupling of resin cements that utilize self-etching primers to dentine. J Dent 2004;32:55–65.
123. Estrela C, Estrela CRA, Barbin EL, et al. Mechanism of action of sodium hypochlorite. Braz Dent J 2002;13:113–7.
124. Morris MD, Lee KW, Agee KA, Bouillaguet S, Pashley DH. Effects of sodium hypochlorite and RC-prep on bond strengths of resin cement to endodontic surfaces. J Endod 2001;27:753–7.
125. Ari H, Yasar E, Belli S. Effects of NaOCl on bond strengths of resin cements to root canal dentin. J Endod 2003;29:248–51.
126. Erdemir A, Ari H, Gungunes H, Belli S. Effect of medications for root canal treatment on bonding to root canal dentin. J Endod 2004;30:113–6.
127. Nikaido T, Takano Y, Sasafuchi Y, Burrow MF, Tagami J. Bond strengths to endodontically-treated teeth. Am J Dent 1999;12:177–80.
128. Lai SC, Mak YF, Cheung GS, et al. Reversal of compromised bonding to oxidized etched dentin. J Dent Res 2001;80:1919–24.
129. Ozturk B, Özer F. Effect of NaOCl on bond strengths of bonding agents to pulp chamber lateral walls. J Endod 2004;30:362–5.
130. Perdigao J, Lopes M, Geraldeli S, Lopes GC, Garcia-Godoy F. Effect of a sodium hypochlorite gel on dentin bonding. Dent Mater 2000;16:311–23.
131. Yiu CK, Garcia-Godoy F, Tay FR, et al. A nanoleakage perspective on bonding to oxidized dentin. J Dent Res 2002;81:628–32.
132. Rueggeberg FA, Margeson DH. The effect of oxygen inhibition on an unfilled/filled composite system. J Dent Res 1990;69:1652–8.
133. Marais JT, Williams WP. Antimicrobial effectiveness of electro-chemically activated water as an endodontic irrigation solution. Int Endod J 2001;34:237–43.
134. Meiers JC, Kresin JC. Cavity disinfectants and dentin bonding. Oper Dent 1996;21:153–9.
135. Perdigao J, Denehy GE, Swift EJ Jr. Effects of chlorhexidine on dentin surfaces and shear bond strengths. Am J Dent 1994;7:81–4.
136. Cunningham MP, Meiers JC. The effect of dentin disinfectants on shear bond strength of resin-modified glass-ionomer materials. Quintessence Int 1997; 28:545–51.
137. el-Housseiny AA, Jamjoum H. The effect of caries detector dyes and a cavity cleansing agent on composite resin bonding to enamel and dentin. J Clin Pediatr Dent 2000;25:57–63.
138. Kazemi RB, Meiers JC, Peppers K. Effect of caries disclosing agents on bond strengths of total-etch and self-etching primer dentin bonding systems to resin composite. Oper Dent 2002;27:238–42.
139. Erdemir A, Eldeniz AU, Belli S, Pashley DH. Effect of solvents on bonding to root canal dentin. J Endod 2004;30:589–92.
140. Fuentes V, Ceballos L, Osorio R, Toledano M, Carvalho RM, Pashley DH. Tensile strength and microhardness of treated human dentin. Dent Mater 2004;20:522–9.
141. Sim TP, Knowles JC, Ng YL, Shelton J, Gulabivala K. Effect of sodium hypochlorite on mechanical properties of dentine and tooth surface strain. Int Endod J 2001; 34:120–32.
142. Eldeniz AU, Erdemir A, Belli S. Effect of EDTA and citric acid solutions on the microhardness and the roughness of human root canal dentin. J Endod 2005; 31:107–10.
143. Macchi RL, Capurro MA, Herrera CL, Cebada FR, Kohen S. Influence of endodontic materials on the bonding of composite resin to dentin. Endod Dent Traumatol 1992;8:26–9.
144. Woody TL, Davis RD. The effect of eugenol-containing and eugenol-free temporary cements on microleakage in resin bonded restorations. Oper Dent 1992;17: 175–80.
145. Watanabe EK, Yamashita A, Imai M, Yatani H, Suzuki K. Temporary cement remnants as an adhesion inhibiting factor in the interface between resin cements and bovine dentin. Int J Prosthodont 1997;10:440–52.
146. Wolanek GA, Loushine RJ, Weller RN, Kimbrough WF, Volkmann KR. In vitro bacterial penetration of endodontically treated teeth coronally sealed with a dentin bonding agent. J Endod 2001;27:354–7.
147. Peutzfeldt A, Asmussen E. Influence of eugenol-containing temporary cement on efficacy of dentin-bonding systems. Eur J Oral Sci 1999;107:65–9.
148. Capurro MA, Herrera CL, Macchi RL. Influence of endodontic materials on the bonding of glass ionomer cement to dentin. Endod Dent Traumatol 1993;9:75–6.
149. Peters OA, Schonenberger K, Laib A. Effects of four Ni-Ti preparation techniques on root canal geometry assessed by micro computed tomography. Int Endod J 2001;34:221–30.
150. Peters OA. Current challenges and concepts in the preparation of root canal systems: a review. J Endod 2004;30:559–67.
151. Sevimay S, Oztan MD, Dalat D. Effects of calcium hydroxide paste medication on coronal leakage. J Oral Rehabil 2004;31:240–4.
152. Lambrianidis T, Margelos J, Beltes P. Removal efficiency of calcium hydroxide dressing from the root canal. J Endod 1999;25:85–8.
153. Kim SK, Kim YO. Influence of calcium hydroxide intracanal medication on apical seal. Int Endod J 2002;35:623–8.
154. Wang CS, Debelian GJ, Teixeira FB. Effect of intracanal medicament on the sealing ability of root canals filled with Resilon. J Endod 2006;32:532–6.
155. Ezzie E, Fleury A, Solomon E, Spears R, He J. Efficacy of retreatment techniques for a resin-based root canal obturation material. J Endod 2006;32:341–4.
156. de Oliveira DP, Barbizam JV, Trope M, Teixeira FB. Comparison between gutta-percha and Resilon removal using two different techniques in endodontic retreatment. J Endod 2006;32:362–4.
157. Schirrmeister JF, Meyer KM, Hermanns P, Altenburger MJ, Wrbas KT. Effectiveness of hand and rotary instrumentation for removing a new synthetic polymer-based root canal obturation material (Epiphany) during retreatment. Int Endod J 2006;39:150–6.
158. Eldeniz AU, Erdemir A, Belli S. Shear bond strength of three resin based sealers to dentin with and without the smear layer. J Endod 2005;31:293–6.
159. Economides N, Liolios E, Kolokuris I, Beltes P. Long-term evaluation of the influence of smear layer removal on the sealing ability of different sealers. J Endod 1999; 25:123–5.
160. Economides N, Kokorikos I, Kolokouris I, Panagiotis B, Gogos C. Comparative study of apical sealing ability of a new resin-based root canal sealer. J Endod 2004; 30:403–5.
161. Cobankara FK, Adanir N, Belli S, Pashley DH. A quantitative evaluation of apical leakage of four root-canal sealers. Int Endod J 2002;35:979–84.
162. Khayat A, Jahanbin A. The influence of smear layer on coronal leakage of Roth 801 and AH26 root canal sealers. Aust Endod J 2005;31:66–8.
163. Clark-Holke D, Drake D, Walton R, Rivera E, Guthmiller JM. Bacterial penetration through canals of endodontically treated teeth in the presence or absence of the smear layer. J Dent 2003;31:275–81.
164. Goldman M, Goldman LB, Cavaleri R, Bogis J, Lin PS. The efficacy of several endodontic irrigating solutions: a scanning electron microscopic study: part 2. J Endod 1982;8:487–92.
165. Lee KW, Williams MC, Camps JJ, Pashley DH. Adhesion of endodontic sealers to dentin and gutta-percha. J Endod 2002;28:684–8.
166. Tagger M, Tagger E, Tjan AH, Bakland LK. Shearing bond strength of endodontic sealers to gutta-percha. J Endod 2003;29:191–3.
167. Gogos C, Stavrianos C, Kolokouris I, Papadoyannis I, Economides N. Shear bond strength of AH-26 root canal sealer to dentine using three dentine bonding agents. J Dent 2003;31:321–6.
168. Gesi A, Raffaelli O, Goracci C, Pashley DH, Tay FR, Ferrari M. Interfacial strength of Resilon and gutta-percha to intraradicular dentin. J Endod 2005;31:809–813.
169. Mannocci F, Ferrari M. Apical seal of roots obturated with laterally condensed gutta-percha, epoxy resin cement, and dentin bonding agent. J Endod 1998; 24:41–4.
170. Mannocci F, Innocenti M, Bertelli E, Ferrari M. Dye leakage and SEM study of roots obturated with Thermafill and dentin bonding agent Endod Dent Traumatol 1999;15:60–4.
171. Pommel L, About I, Pashley D, Camps J. Apical leakage of four endodontic sealers. J Endod 2003;29:208–10.
172. Suprabha BS, Sudha P, Vidya M. A comparative evaluation of sealing ability of rootcanal sealers. Indian J Dent Res 2002;13:31–6.
173. Schafer E, Olthoff G. Effect of three different sealers on the sealing ability of both Thermafil obturators and cold laterally compacted gutta-percha. J Endod 2002; 28:638–42.
174. Sevimay S, Kalayci A. Evaluation of apical sealing ability and adaptation to dentine of two resin-based sealers. J Oral Rehabil 2005;32:105–10.
175. Shipper G, Orstavik D, Teixeira FB, Trope M. An evaluation of microbial leakage in roots filled with a thermoplastic synthetic polymer-based root canal filling material (Resilon). J Endod 2004;30:342–7.
176. Cobankara FK, Adanr N, Belli S. Evaluation of the influence of smear layer on the apical and coronal sealing ability of two sealers. J Endod 2004;30:406–9.
177. Ferguson DB, Marley JT, Hartwell GR. The effect of chlorhexidine gluconate as an endodontic irrigant on the apical seal: long-term results. J Endod 2003;29:91–4.
178. Bergmans L, Moisiadis P, De Munck J, Van Meerbeek B, Lambrechts P. Effect of polymerization shrinkage on the sealing capacity of resin fillers for endodontic use. J Adhes Dent 2005;7:321–9.

179. Zmener O, Banegas G, Pameijer CH. Bone tissue response to a methacrylate-based endodontic sealer: a histological and histometric study. J Endod 2005;31:457–9.
180. Zmener O. Tissue response to a new methacrylate-based root canal sealer: preliminary observations in the subcutaneous connective tissue of rats. J Endod 2004;30:348–51.
181. Bouillaguet S, Wataha JC, Lockwood PE, Galgano C, Golay A, Krejci I. Cytotoxicity and sealing properties of four classes of endodontic sealers evaluated by succinic dehydrogenase activity and confocal laser scanning microscopy. Eur J Oral Sci 2004;112:182–7.
182. Kardon BP, Kuttler S, Hardigan P, Dorn SO. An in vitro evaluation of the sealing ability of a new root-canal-obturation system. J Endod 2003;29:658–61.
183. Sipert CR, Hussne RP, Nishiyama CK, Torres SA. In vitro antimicrobial activity of Fill Canal, Sealapex, Mineral Trioxide Aggregate, Portland cement and EndoRez. Int Endod J 2005;38:539–43.
184. Tay FR, Loushine RJ, Monticelli F, et al. Effectiveness of resin-coated gutta-percha cones and a dual-cured, hydrophilic methacrylate resin-based sealer in obturating root canals. J Endod 2005;31:659–64.
185. Zmener O, Pameijer CH. Clinical and radiographic evaluation of a resin-based root canal sealer. Am J Dent 2004;17:19–22.
186. Teixeira FB, Teixeira EC, Thompson JY, Trope M. Fracture resistance of roots endodontically treated with a new resin filling material. J Am Dent Assoc 2004; 135:646–52.
187. Stuart CH, Schwartz SA, Beeson TJ. Reinforcement of immature roots with a new resin filling material. J Endod 2006;32:350–3.
188. Williams C, Loushine RJ, Weller RN, Pashley DH, Tay FR. A comparison of cohesive strength and stiffness of Resilon and gutta-percha. J Endod 2006;32:553–5.
189. Cobankara FK, Ungor M, Belli S. The effect of two different root canal sealers and smear layer on resistance to root fracture. J Endod 2002;28:606–9.
190. Aptekar A, Ginnan K. Comparative analysis of microleakage and seal for 2 obturation materials: Resilon/Epiphany and gutta-percha. J Can Dent Assoc 2006;72:245–9.
191. Shipper G, Teixeira FB, Arnold RR, Trope M. Periapical inflammation after coronal microbial inoculation of dog roots filled with gutta-percha or Resilon. J Endod 2005;31:91–6.
192. Tay FR, Pashley DH, Williams MC, et al. Susceptibility of a polycaprolactone-based root canal filling material to degradation. I. Alkaline hydrolysis. J Endod 2005;31:593–8.
193. Tay FR, Pashley DH, Yiu CK, et al. Susceptibility of a polycaprolactone-based root canal filling material to degradation. II. Gravimetric evaluation of enzymatic hydrolysis. J Endod 2005;31:737–41.
194. Versiani MA, Carvalho-Junior JR, Padilha MI, Lacey S, Pascon EA, Sousa-Neto MD. A comparative study of physicochemical properties of AH Plus and Epiphany root canal sealants. Int Endod J 2006;39:464–71.
195. Melker KB, Vertucci FJ, Rojas MF, Progulske-Fox A, Belanger M. Antimicrobial efficacy of medicated root canal filling materials. J Endod 2006;32:148–51.
196. Hiraishi N, Papacchini F, Loushine RJ, et al. Shear bond strength of Resilon to a methacrylate-based root canal sealer. Int Endod J 2005;38:753–63.
197. Tay FR, Hiraishi N, Pashley DH, et al. Bondability of Resilon to a methacrylate-based root canal sealer. J Endod 2006;32:133–7.
198. Hiraishi N, Loushine RJ, Vano M, et al. Is an oxygen inhibited layer required for bonding of resin-coated gutta-percha to a methacrylate-based root canal sealer? J Endod 2006;32:429–33.
199. Leonard JE, Gutmann JL, Guo IY. Apical and coronal seal of roots obturated with a dentine bonding agent and resin. Int Endod J 1996;29:76–83.
200. Timpawat S, Sripanaratanakul S. Apical sealing ability of glass ionomer sealer with and without smear layer. J Endod 1998;24:343–5.
201. Eick JD, Robinson SJ, Byerley TJ, Chappelow CC. Adhesives and nonshrinking dental resins of the future. Quintessence Int 1993;24:632–40.
202. Eick JD. Smear layer: materials surface. Proc Finn Dent Soc 1992;88(Suppl 1): 8–14.
203. Stansbury JW, Trujillo-Lemon M, Lu H, Ding X, Lin Y, Ge J. Conversion-dependent shrinkage stress and strain in dental resins and composites. Dent Mater 2005;21:56–67.
204. Stansbury JW. Synthesis and evaluation of new oxaspiro monomers for double ring-opening polymerization. J Dent Res 1992;71:1408–12.
205. Ferracane JL. Current trends in dental composites. Crit Rev Oral Biol Med 1995;6:302–18.
206. Yesilsoy C. Radiographic evidence of absorption of Hydron from an obturated root canal. J Endod 1984;10:321–3.