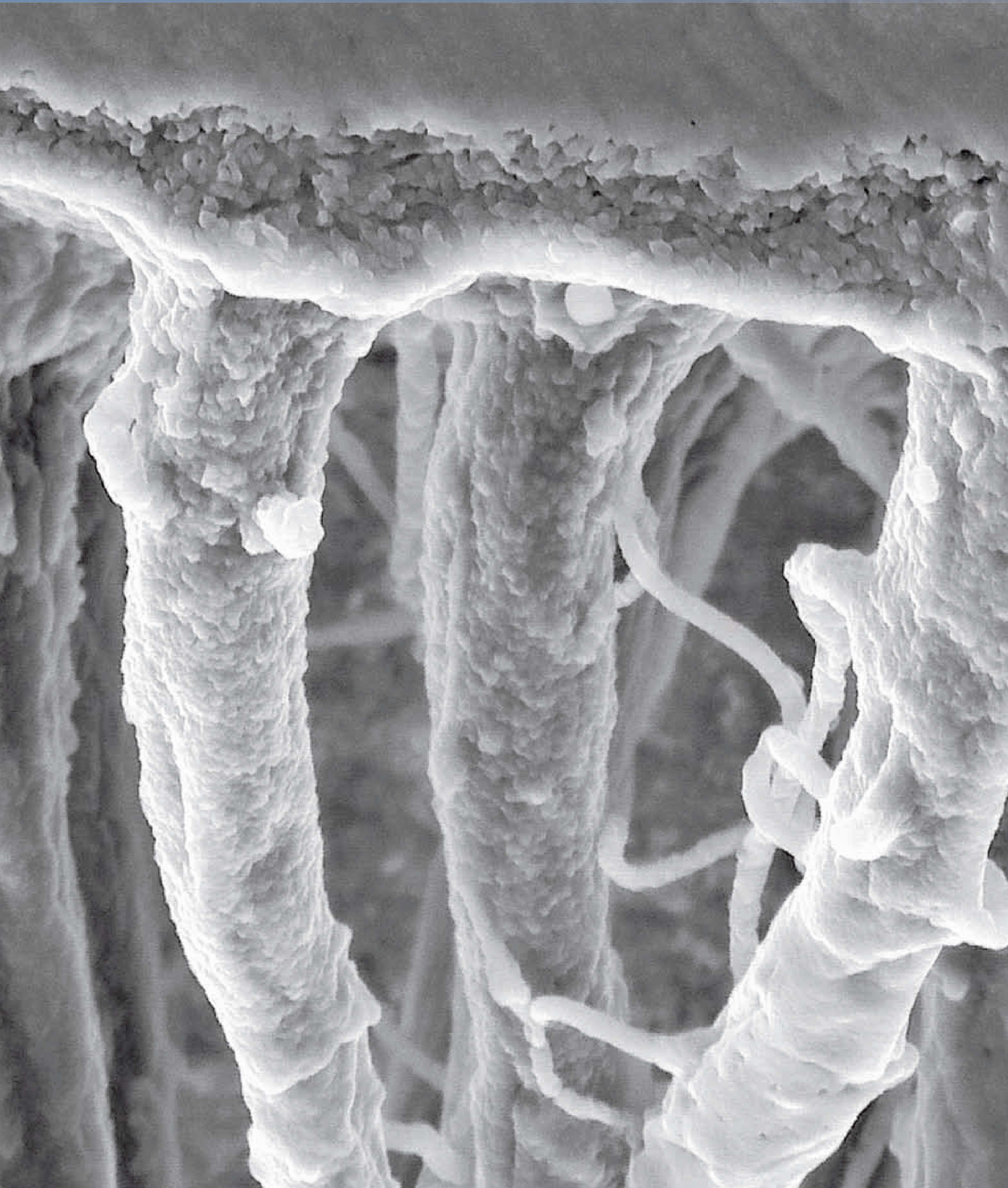
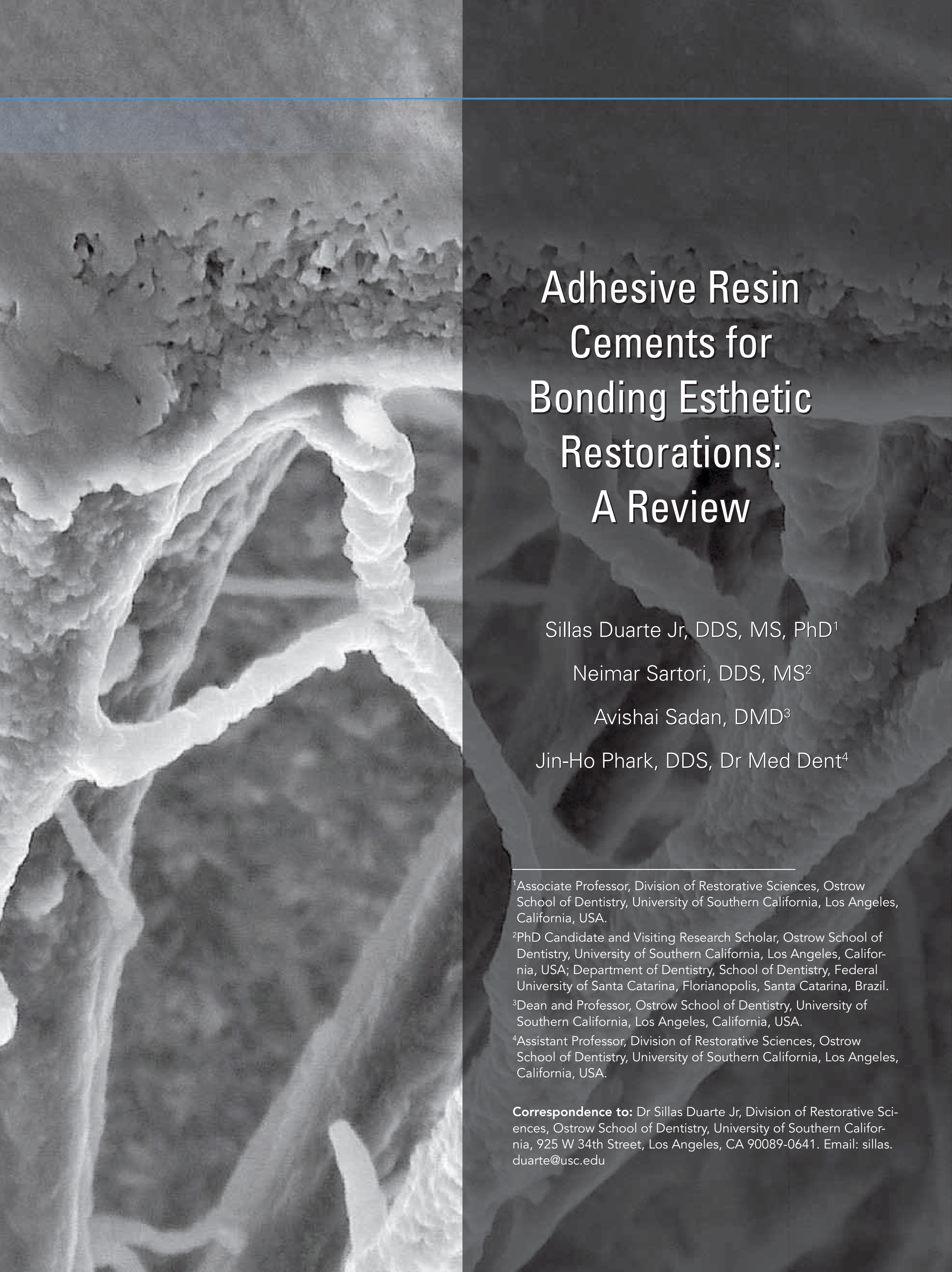
Biomaterials Update





Adhesive Resin Cements for Bonding Esthetic Restorations: A Review

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Table 1 Material Properties of Various Dental Cements

Interaction between substrates	Cement type	Film thickness (µm)	Strength (MPa)	
			Compressive	Tensile
Nonadhesive	Zinc phosphate	25–35	96–133	3.1–4.5
Chemical bonding	Polycarboxylate	19–25	57–99	3.6–12
	Glass-ionomer	11–35	93–226	42.53
	Resin-modified glass-ionomer	11–21	85–160	13–25
	Phosphate-modified composite resin (self-adhesive)	13–50	212–291	34
Micromechanical bonding	Self-cured composite resin	24.3–50	292	62
	Light-cured composite resin	5–10	345–400	77.4
	Dual-cured resin cements	16.4	279–352	40–56

Cement is a substance that produces a solid union between two surfaces. In dentistry, three types of luting cements are available based on their interaction with the substrate: nonadhesive luting cements (eg, zinc phosphate cements), chemically bonded cements (eg, polycarboxylate, glass ionomer–based, and phosphate-modified resin cements), and micromechanically bonded cements (eg, polyfunctional dimethacrylate–based cements) (Table 1). The adhesive properties of dimethacrylate-based cements are determined not primarily by the cement itself, but by the type of coupling adhesive system. Since most esthetic restorations require adhesive cementation, clinicians must understand the performance of different adhesive resins to produce long-lasting restorations.

BONDING MECHANISMS OF ADHESIVE RESIN CEMENTS

Most resin cements require pretreatment of the dental substrate to promote bonding to the dental tissues. This pretreatment can be obtained by the application of an etch-and-rinse or self-etch dentin adhesive system, depending on the manufacturer or the characteristics of the resin cement. Recently, self-adhesive resin cements were also introduced as an alternative to multistep resin-based luting cements. Therefore, resin cements can be classified into one of three groups according to the bonding characteristics: etch-and-rinse, self-etch, and self-adhesive resin cements.

Modulus of elasticity (GPa)	Solubility	Flexural strength (MPa)	Trade name
13	0.2%	15–98	HY-Bond Zinc Phosphate (Shofu, San Marcos, CA, USA)
5–6	0.06%	14.7–16.5	Durelon (3M ESPE, St Paul, MN, USA) HY-Bond Polycarboxylate (Shofu)
7–8	1%	7.8–24.8	Ketac Cem (3M ESPE) GC FujiCEM (GC America, Alsip, IL, USA)
2.5–7.8	79 µg/mm^3	27–100	RelyX Luting (3M ESPE) RelyX Luting Plus (3M ESPE) GC Fuji PLUS (GC America)
4.5–6.6	3–33 µg/mm^3	42–99	RelyX Unicem 2 (3M ESPE) Maxcem Elite (Kerr, Orange, CA, USA) Biscem (Bisco, Schaumburg, IL, USA) SpeedCem (Ivoclar Vivadent, Schaan, Liechtenstein)
6.5	0.89%	100	Panavia 21 (Kuraray, Kurashiki, Okayama, Japan) C&B Cement (Bisco)
4.5	0–12 µg/mm^3	107–123	Variolink Veneer (Ivoclar Vivadent) Choice 2 (Bisco) RelyX Veneer (3M ESPE)
6–9.6	0–128 µg/mm^3	110–131	Panavia F 2.0 (Kuraray) RelyX ARC (3M ESPE) NX3 (Kerr) Multilink Automix (Ivoclar Vivadent) Variolink II (Ivoclar Vivadent) Calibra (Dentsply, Milford, DE) Duo-Link (Bisco)

Etch-and-Rinse Resin Cements

Etch-and-rinse resin cements combine a dentin adhesive system with methacrylate-based resin cement (Table 2). The manufacturers of most resin cements recommend the use of two-step etch-and-rinse adhesives or "one-bottle" adhesive systems. These systems combine the primer and adhesive resin into one solution, which is theoretically more user-friendly than multi-bottle etch-and-rinse adhesives. However, clinicians must reevaluate the application technique of two-step etch-and-rinse adhesives to achieve similar bond strengths to those of multistep etch-and-rinse adhesives.

Etch-and-rinse adhesives include phosphoric acid that etches enamel and dentin simultaneously. The application of acid to dentin results in removal of the smear layer, demineralization of dentin up to 5 to 8 µm, widening of the dentin tubuli, and exposure of the collagen fibers (Fig 1). Following this procedure, three layers can be distinguished: *(1)* a superficial smeared collagen layer, *(2)* an intermediate densely packed fibrillar layer, and *(3)* a deeper area with some scattered mineral crystals and a few randomly exposed collagen fibrils.[1] Hydrophilic monomers permeate the small spaces created within the dentin collagen network, resulting in resin-enveloped collagen fibrils and formation of a resin-dentin interdiffusion zone (Fig 2).[2]

Etch-and-rinse adhesives must be used with a wet bonding technique to expand the acid-etched dentin matrix and avoid the collapse of the collagen network

Table 2 **Etch-and-Rinse Resin Cements**

Resin cement	Polymerization mode	Cement composition	Adhesive system
RelyX ARC	Dual-cured	Paste A: silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol	Adper Single Bond Plus
		Paste B: silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide	
RelyX Veneer Cement	Light-cured	Silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer	Adper Single Bond Plus
NX3 Nexus Third Generation	Dual- or light-cured	Uncured methacrylate ester monomers, inert mineral fillers, activators and stabilizers, radiopaque agent	OptiBond Solo Plus
Calibra	Dual- or Light-cured	Base: barium boron fluoroalumino silicate glass, bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, colorants are inorganic iron oxides	Prime & Bond NT
		Catalyst: barium boron fluoroalumino silicate glass, bis-GMA resin, polymerizable dimethacrylate resin, hydrophobic amorphous fumed silica, titanium dioxide, benzoyl peroxide	
C&B Cement	Self-cured	Base: bis-GMA, ethoxylated bis-GMA, triethyleneglycol dimethacrylate, fused silica, glass filler, sodium fluoride.	One-Step Plus or
		Catalyst: bis-GMA, triethyleneglycol dimethacrylate, fused silica	All-Bond 3
Choice 2 Veneer Cement	Light-cured	Strontium glass, amorphous silica, bis-GMA	One-Step Plus
			All-Bond 3
Duo-Link	Dual-cured	Base: bis-GMA, triethyleneglycol dimethacrylate, urethane dimethacrylate, glass filler	One-Step Plus
		Catalyst: bis-GMA, triethyleneglycol dimethacrylate, glass filler	All-Bond 3
Variolink II	Dual-cured	Dimethacrylates, bis-GMA, triethylene glycoldimethacrylate, urethanedimethacrylate, benzoyl peroxide, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments	Excite F DSC
			Syntac Classic
Variolink Veneer	Light-cured	Dimethacrylates, urethanedimethacrylate, decandiole dimethacrylate, inorganic fillers, ytterbiumtrifluoride, initiators, stabilizers and pigments	Syntac Classic
			Excite F
Duo Cement Plus	Dual-cured	Base: bis-GMA, TEGDMA	One Coat Bond
		Catalyst: bis-GMA, TEGDMA, dibenzoyl peroxide, benzoyl peroxide	

TEGDMA = triethylene glycol dimethacrylate; bis-GMA = bisphenol glycidyl methacrylate; HEMA = hydroxyethyl methacrylate; BPDM = biphenyl dimethacrylate; SiO_2 = silicon dioxide; GDMA = glycerol dimethacrylate/maleate adduct; NTG-GMA = -tolylglycine-glycidyl methacrylate.

*As per the manufacturer.

Adhesive composition	Manufacturer
Ethyl alcohol, bis-GMA, 2-hydroxyethyl methacrylate, glycerol 1,3 dimethacrylate, copolymer of acrylic and itaconic acids, diurethane dimethacrylate, water	3M ESPE
Ethyl alcohol, bis-GMA, 2-hydroxyethyl methacrylate, glycerol 1,3 dimethacrylate, copolymer of acrylic and itaconic acids, diurethane dimethacrylate, water	3M ESPE
Ethyl alcohol, alkyl dimethacrylate resins, barium aluminoborosilicate glass, fumed silica, sodium hexafluorosilicate	Kerr
Acetone, urethane dimethacrylate resin, dipentaerythritol pentaacrylate phosphate, polymerizable dimethacrylate resins, polymerizable trimethacrylate resins	Dentsply, Milford, DE, USA
Self-cure activator: aromatic sodium sulfinate, acetone, ethanol	
Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, dental glass	Bisco
Part A: Ethanol, NTG-GMA salt	
Part B: bis-GMA, HEMA, BPDM	
Resin: bis-GMA, urethane dimethacrylate, triethyleneglycol dimethacrylate, glass filler	
Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, dental glass	Bisco
Part A: Ethanol, NTG-GMA salt	
Part B: bis-GMA, HEMA, BPDM	
Resin: bis-GMA, urethane dimethacrylate, triethyleneglycol dimethacrylate, glass filler	
Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, dental glass	Bisco
Part A: Ethanol, NTG-GMA salt	
Part B: bis-GMA, HEMA, BPDM	
Resin: bis-GMA, urethane dimethacrylate, triethyleneglycol dimethacrylate, glass filler	
Dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, potassium fluoride, initiators and stabilizers	Ivoclar Vivadent
Primer: Water, acetone, maleic acid, and dimethacrylate	
Adhesive: Water, glutaraldehyde, maleic acid, and polyethyleneglycoldimethacrylate	
Primer: Water, acetone, maleic acid, and dimethacrylate	Ivoclar Vivadent
Adhesive: Water, glutaraldehyde, maleic acid, and polyethyleneglycoldimethacrylate	
Dimethacrylates, alcohol, phosphonic acid acrylate, HEMA, SiO_2, potassium fluoride, initiators and stabilizers	
2-hydroxyethyl methacrylate, GDMA, urethane dimethacrylate	Coltène/Whaledent, Altstätten, Switzerland

(Figs 3a and 3b).[3] However, excessive water in interfibrillar spaces will compete with the adhesive monomers, diluting their concentration and preventing optimal polymerization (Figs 4a and 4b).[4] Water within the collagen network leads to rapid degradation of the bonded interfaces. Therefore, some strategies will now be suggested to improve bonding to dental tissues.

Two-step etch-and-rinse adhesives require multiple coatings—more than recommended by the manufacturer—to achieve acceptable micromechanical interlocking of monomers into the microretentive collagen network.[5] In addition, vigorous application improves clinical retention and bond strength.[6,7]

After dentin adhesive application, meticulous solvent evaporation must be performed. Incomplete solvent evaporation increases permeability and decreases bond strength.[4] Residual water trapped within the collagen network will lead to incomplete polymerization of the adhesive monomers. The solvent evaporation process must also be more prolonged than advocated by the manufacturer. Complete evaporation of the solvent is almost impossible to attain.[8,9]

Simplified adhesives are permeable to fluid movements across the cured adhesive layer (Fig 4b).[10] Fluid transudation has been observed on bonded surfaces for both vital and endodontically treated teeth.[11] The transudation of dentinal fluids significantly affects the bonding of dual-cured resin cements.[10] Water droplets trapped along the interface may plasticize the polymer, resulting in catastrophic failure of the restoration.[10,11] Application of an additional hydrophobic resin coating over the simplified adhesive may decrease adhesive permeability and increase bond stability (Fig 5).

The polymerization of an adhesive system yields adequate mechanical and physical properties. Successful polymerization of a given adhesive is dependent on its composition and the distance from the light tip. However, especially for indirect restorations, the use of a self- or dual-cured adhesive may be considered when effective light polymerization is uncertain. To address this problem, some etch-and-rinse resin cements include chemical co-initiators or activators to convert the light-polymerized adhesive into a self- or dual-cured adhesive. But the use of an activator has limited effect in improving the coupling of dual-cured adhesives with self- or dual-cured composites.[12,13] In addition, self-polymerization alone is not advised since the degree of conversion is significantly lower than when the same adhesive is used in dual-cure mode.[14] Therefore, dentin adhesives used in conjunction with resin cements must be light polymerized, irrespective of the activation mode. Furthermore, extending the curing time beyond 20 seconds is highly recommended.[15,16]

Self-Etch Resin Cements

The demand for resin cements that are less technique sensitive and more user-friendly pushed manufacturers to substitute etch-and-rinse adhesive with self-etch adhesive. Self-etch adhesives consist of non-rinsing acidic monomers that simultaneously etch and prime dentin and enamel. Self-etch adhesives are available as one- or two-step adhesives. Two-step self-etch adhesives comprise a self-etching primer and a hydrophobic adhesive resin, whereas one-step self-etch adhesives combine etchant, primer, and bonding in a single solution. Self-etch adhesives simultaneously demineralize and infiltrate the dental substrate. The etching characteristics are dependent on the pH of the acidic solutions. Ultra-mild self-etch adhesives ($pH > 2.5$) provide nano-interaction with dental substrates. Mild self-etch adhesives ($pH \approx 2.0$) feature a submicron hybrid layer with less-pronounced resin tag formation (Figs 6a and 6b). Strong self-etch adhesives ($pH \leq 1.0$) result in an interfacial ultramorphology resembling that produced typically by total-etch adhesives, with the formation of abundant resin tags (Figs 7a and 7b).[17]

Since self-etch adhesives do not require rinsing and drying, the smear layer is not removed but impregnated by the acidic monomers. Intertubular collagen is then exposed, and the removed minerals are replaced by resin monomers, creating micromechanical interlocking within the collagen interstices. The collagen fibrils are not completely deprived of hydroxyapatite, in contrast with total-etch adhesives.[18] For that reason, chemical interaction between functional monomers (10-methacryloyloxydecyl dihydrogen phosphate [MDP]) or some acids (polyalkenoic acids) and hydroxyapatite is also observed and may improve bond durability (Table 3).[17] Despite the limited chemical bonding, micromechanical interlocking is still the main source of bonding for self-etch adhesives.

The effectiveness of self-etch adhesive systems varies considerably and is affected by composition,[19,20] shelf life,[21] and aging.[20,22,23] One-step self-etch adhesives

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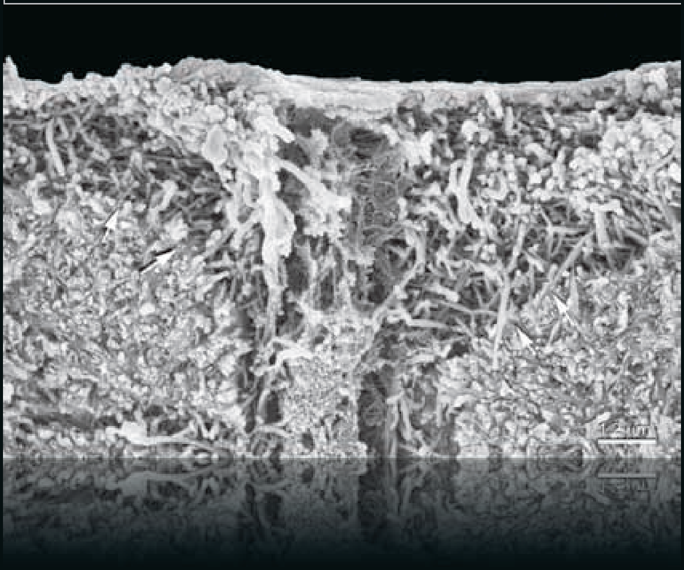

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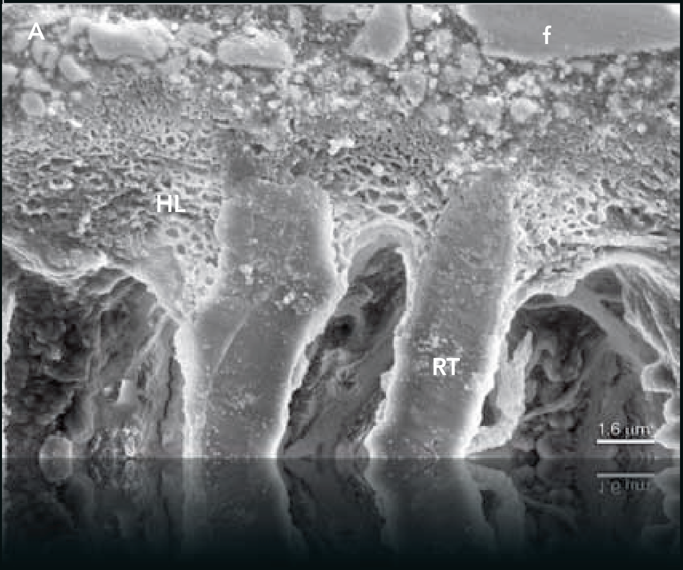
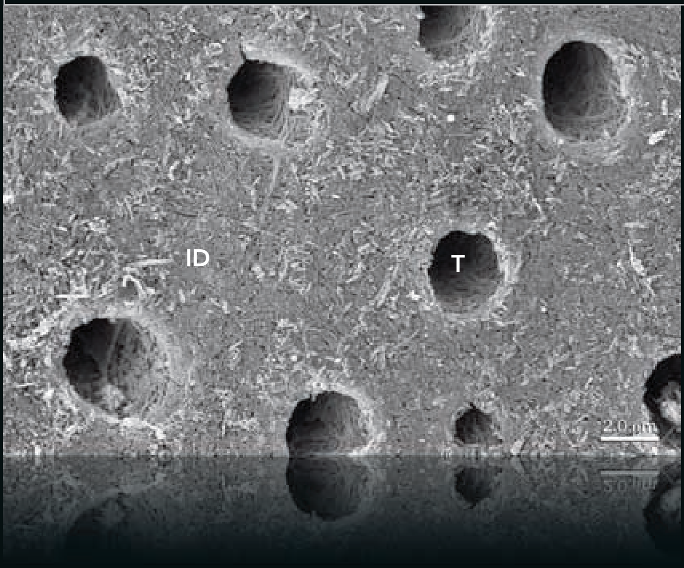


3a and 3b



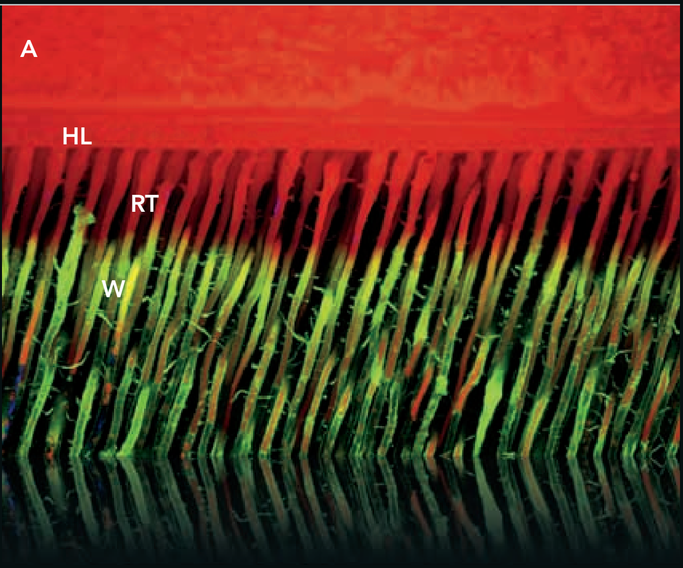


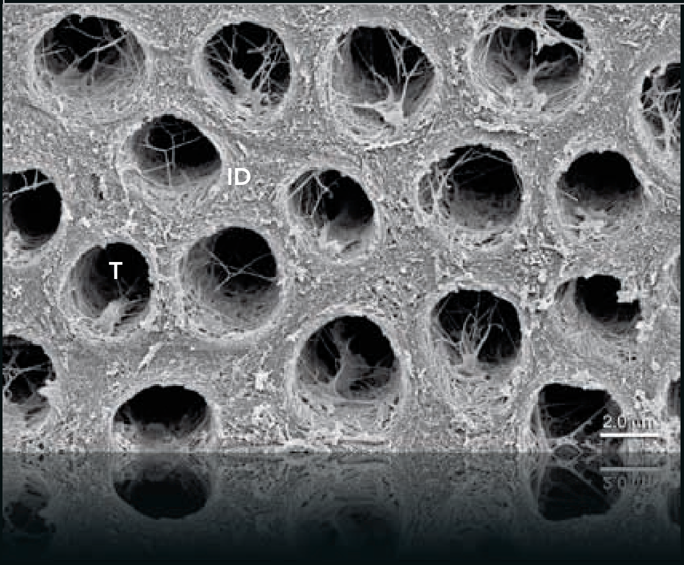
Fig 1 Field-emission scanning electron microscopy (FeSEM) showing a longitudinal view of etched dentin (magnification ×10,000). White arrows = dentin decalcification.

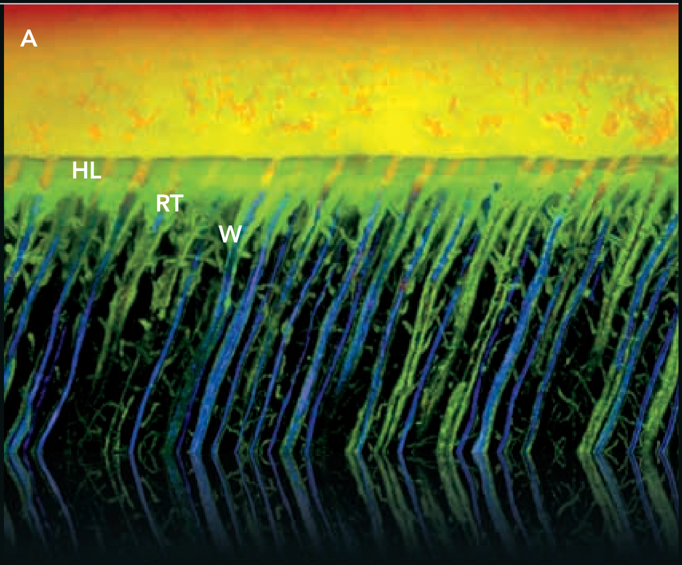
Fig 2 FeSEM showing the hybrid layer of a highly filled adhesive (magnification ×15,000). HL = hybrid layer; RT = resin tags; A = adhesive layer; f = filler.

Fig 3a Acid-etched superficial dentin (magnification ×5,000). Note the large amount of intertubular dentin. ID = intertubular dentin; P = peritubular dentin; T = tubule.

Fig 3b Confocal laser scanning microscopy (CLSM) showing full hybridization of an etch-and-rinse adhesive applied on superficial dentin (magnification ×100). The adhesive system *(red)* and water *(green)* were stained to facilitate visualization. Note that the hybrid layer and resin tags are distinct from the intratubular water. A = adhesive layer; HL = hybrid layer; RT = resin tags; W = water.

4a and 4b





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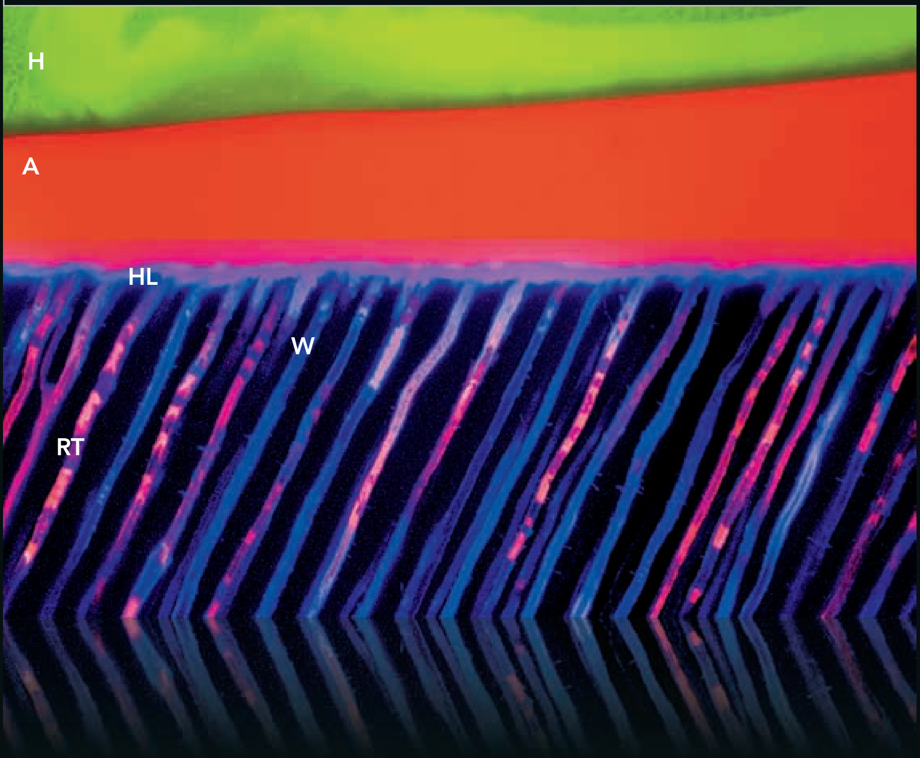
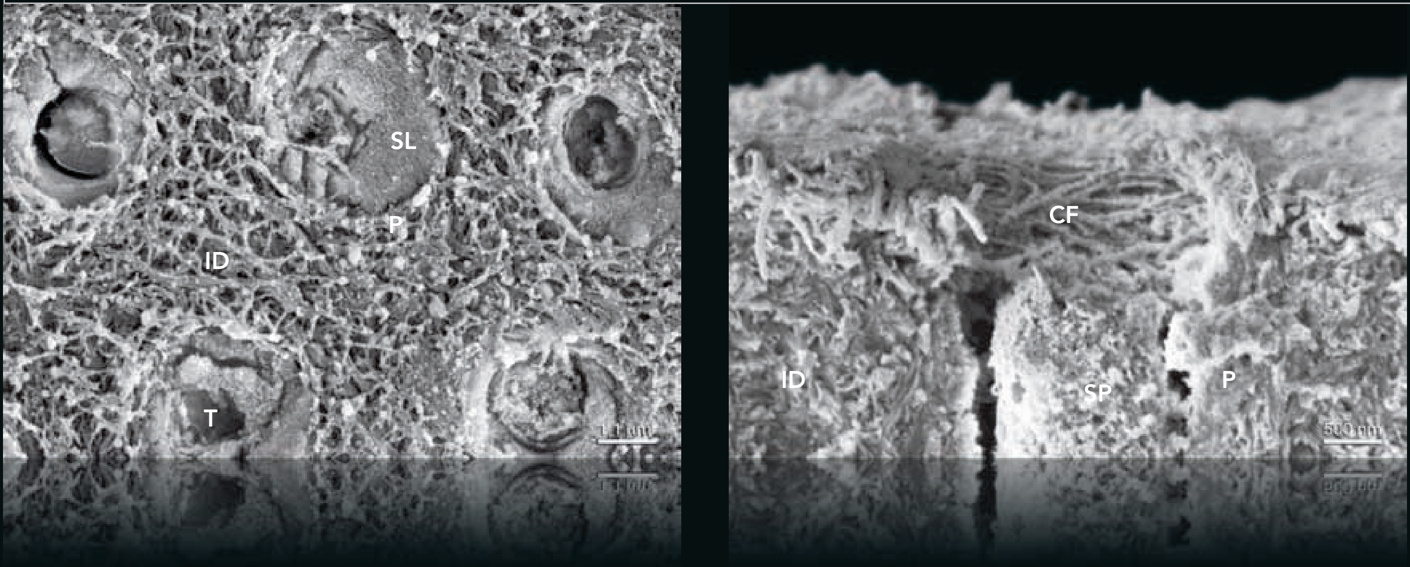


Fig 4a Acid-etched deep dentin showing a reduced intertubular dentin area and enlarged tubules (magnification ×5,000).

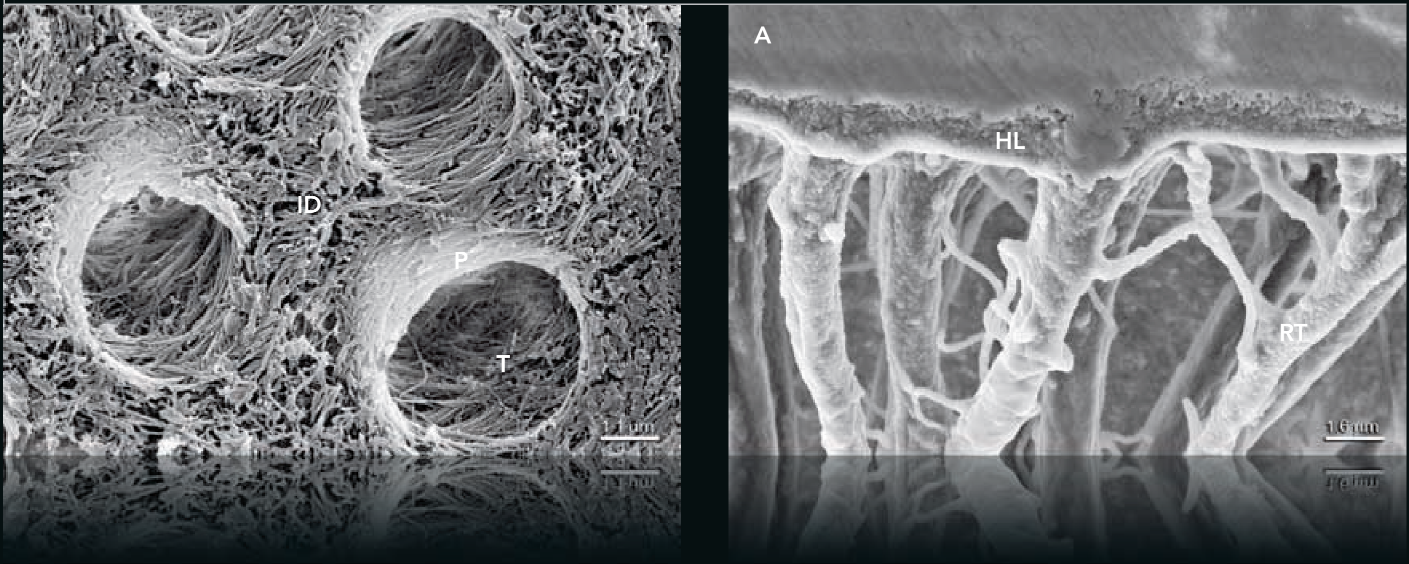
Fig 4b CLSM showing fluid transudation through the resin tags and hybrid layer to the adhesive layer (magnification ×100). Simplified adhesives are permeable to fluid movements across the cured adhesive layer. Note that water *(green)* is heavily concentrated around the resin tags and at the bottom of the hybrid layer and flows toward the adhesive layer *(yellowish-green)*.

Fig 5 CLSM showing a hydrophobic resin *(green)* applied over a simplified etch-and-rinse adhesive *(red)*. Note the decreased adhesive permeability and lack of water penetration *(blue)* beyond the hybrid layer. H = hydrophobic resin layer; A = adhesive layer; HL = hybrid layer; RT = resin tags; W = water.

6a and 6b



7a and 7b



Figs 6a and 6b FeSEM showing decalcification of dentin after application of a mild self-etch adhesive. *(a)* Intertubular collagen is exposed, while the smear layer is still within the tubules (magnification ×20,000). *(b)* Longitudinal view showing superficial decalcification of the dentin and smear plug inside of the tubule (magnification ×50,000). ID = intertubular dentin; P = peritubular dentin; T = tubule; SM = smear layer; CF = collagen fibers; SP = smear plug.

Figs 7a and 7b *(a)* FeSEM showing aggressive dentin etching with a strong self-etch adhesive. Ultramorphology resembles that of dentin etched with phosphoric acid (magnification ×20,000). *(b)* FeSEM showing the hybrid layer obtained with a strong self-etch adhesive (magnification ×15,000). Note the similarity to an etch-and-rinse adhesive, except for the smaller hybrid layer.

Table 3 Self-Etch Resin Cements

Resin cement	Polymerization mode*	Cement composition
RelyX ARC	Dual-cured	Paste A: Silane-treated ceramic, TEGDMA, bis-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol
		Paste B: Silane-treated ceramic, TEGDMA, BIS-GMA, silane-treated silica, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide
NX3 Nexus Third Generation	Dual- and light-cured	Uncured methacrylate ester monomers, inert mineral fillers, activators and stabilizers, radiopaque agent
Clearfil Esthetic Cement	Dual-cured	Bisphenol A diglycidylmethacrylate, triethylene glycol dimethacrylate, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated barium glass filler, colloidal silica, dl-camphorquinone, catalysts, accelerators, pigments, other
Panavia 21	Self-cured	Base: Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated titanium oxide, silanated barium glass filler, catalysts, accelerators, pigments, other
		Catalyst: 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated silica filler, colloidal silica, catalysts, other
Panavia F 2.0	Dual-cured	Paste A: 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler, silanated colloidal silica, dl-camphorquinone, catalysts, initiators, other
		Paste B: Sodium fluoride, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, catalysts, accelerators, pigments, other
C&B Cement	Self-cured	Base: bis-GMA, ethoxylated bis-GMA, triethyleneglycol dimethacrylate, fused silica, glass filler, sodium fluoride
		Catalyst: bis-GMA, triethyleneglycol dimethacrylate, fused silica
Duo-Link	Dual-cured	Base: bis-GMA, triethyleneglycol dimethacrylate, urethane dimethacrylate, glass filler
		Catalyst: bis-GMA, triethyleneglycol dimethacrylate, glass filler
Multilink Automix	Self-cured	Dimethacrylates, HEMA, benzoyl peroxide, inorganic fillers, ytterbium trifluoride, initiators, stabilizers, pigments
ParaCem Universal DC	Dual-cured	Base: bis-GMA, TEGDMA, sodium fluoride
		Catalyst: bis-GMA, TEGDMA, dibenzoyl peroxide, benzoyl peroxide, sodium fluoride

TEGDMA = triethylene glycol dimethacrylate; bis-GMA = bisphenol glycidyl methacrylate; HEMA = hydroxyethyl methacrylate.

*As per the manufacturer.

Adhesive system	Adhesive composition	Manufacturer
Adper Easy Bond	bis-GMA, 2-hydroxyethyl methacrylate, ethanol, water, phosphoric acid-6-methacryloxy-hexylesters, silane treated silica, 1,6-hexanediol dimethacrylate, copolymer of acrylic and itaconic acid, (dimethylamino) ethyl methacrylate, camphorquinone, 2,4,6-trimethylbenzoyldiphenylphosphine oxide	3M ESPE
OptiBond All-In-One	Acetone, ethyl alcohol, uncured methacrylate ester, monomers, TWA, inert mineral fillers, ytterbium fluoride, photoinitiators, accelerators, stabilizers, water	Kerr
OptiBond XTR	Primer: Acetone, ethyl alcohol, HEMA, GPDM, mono- and di-functional methacrylate monomers, camphorquinone	
	Adhesive: monomers, ethyl alcohol, camphorquinone, barium glass nano-silica, sodium hexafluorosilicate	
Clearfil DC Bond	Liquid A: 2-hydroxyethyl methacrylate, bisphenol A diglycidylmethacrylate, dibenzoyl peroxide, 10-methacryloyloxydecyl dihydrogen phosphate, colloidal silica, dl-camphorquinone, initiators, other	Kuraray
	Liquid B: ethanol, water, accelerators, catalysts	
ED primer	Liquid A: 2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, N-methacryloyl-5-aminosalicylic acid, water, accelerators	Kuraray
	Liquid B: N-methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators	
ED primer II	Liquid A: 2-hydroxyethyl methacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, N-methacryloyl-5-aminosalicylic acid, water, accelerator	Kuraray
	Liquid B: N-methacryloyl-5-aminosalicylic acid, water, catalysts, accelerators	
All-Bond SE	Part I: Ethanol, sodium benzene sulfinate	Bisco
	Part II: Hydroxyethyl methacrylate, bis(glyceryl 1,3 dimethyacrylate) phosphate, biphenyl dimethacrylate	
All-Bond SE	Part I: Ethanol, sodium benzene sulfinate	Bisco
	Part II: Hydroxyethyl methacrylate, bis(glyceryl 1,3 dimethyacrylate) phosphate, biphenyl dimethacrylate	
Multilink Primer	Primer A: Mixture of water and initiators	Ivoclar Vivadent
	Primer B: Phosphonic acid acrylate, HEMA, methacrylate modified polyacrylic acid, stabilizer	
ParaBond	Conditioner: 2-hydroxyethyl methacrylate, acrylamido sulphonic acid	Coltène/ Whaledent
	Adhesive A: 2-hydroxyethyl methacrylate, ethanol, ethyl alcohol, benzoyl peroxide, dibenzoyl peroxide	
	Adhesive B: ethanol, ethyl alcohol	

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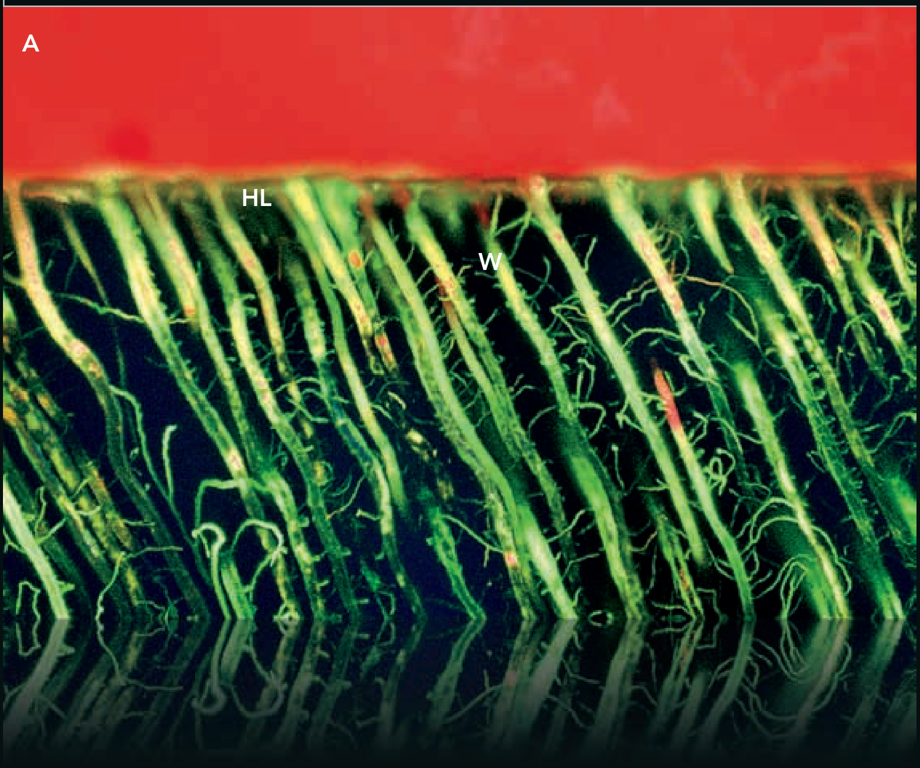

Fig 8 CLSM showing water permeation through the hybrid layer of a one-step self-etch adhesive (magnification ×100). Self-etch adhesives act as a semi-permeable membrane, allowing diffusion of water through the bonded interfaces even after polymerization.

showed the highest annual failure rate compared to two-step self-etch and etch-and-rinse adhesives.[24,25] The application of all-in-one adhesives is not necessarily simpler or less time consuming,[19,26] and their sealing properties are still problematic.[27] The clinical performance of newer one-step self-etch adhesives has shown some improvement.[28,29] However, caution is advised when bonding one-step self-etch adhesive to dual-cured resin cements because of the adverse chemical interaction between the acidic adhesive and resin cement.[30–32] In addition, water from dentin can mix with the hydrophilic co-monomers during evaporation of solvent, creating nanoleakage pathways within the hybrid and adhesive layers.[33] As a result, these adhesives act as a semi-permeable membrane with blisters filled with water and incompletely polymerized monomers, allowing diffusion of water through the bonded interfaces even after polymerization (Fig 8).[34,35] This process is deleterious to the restorations since water accumulation jeopardizes the longevity of the bonded interface.[34]

The placement of a hydrophobic resin coat seems to improve the sealing ability of one-step self-etch adhesive.[36] However, one-step self-etch adhesive is still technique sensitive.[37,38] Two-step self-etch adhesives are more stable and reliable and should be preferred.

Despite all recent advances in the bonding of self-etch adhesives, acceptable long-term enamel bonding is only achieved by pretreatment with phosphoric acid.

Self-Adhesive Resin Cements

Self-adhesive resin cements can bond to dental tissues without previous etching procedures or the application of bonding adhesive (Table 4). Their application is accomplished in one step, which makes them clinically attractive. After mixing, the phosphoric acid

Table 4 **Self-adhesive Resin Cements**

Resin cement	Polymerization mode	Cement composition	Manufacturer
RelyX Unicem 2 Automix	Dual-cured	Base: Silane-treated glass powder, 2-propenoic acid, 2-methyl-, 1,1′-[1-(hydroxymethyl)-1,2-ethanediyl] ester, reaction products with 2-hydroxy-1,3- propanediyl dimethacrylate and phosphorus oxide, TEGDMA, silane-treated silica, sodium persulfate, glass powder, tert-butyl peroxy-3,5,5-trimethylhexanoate	3M ESPE
		Catalyst: Silane-treated glass powder, substituted dimethacrylate, silane-treated silica, 1-benzyl-5-phenyl-barbic-acid, calcium salt, sodium p-toluenesulfinate, 1,12-dodecane dimethycrylate, calcium hydroxide	
Maxcem Elite	Dual-cured	Uncured methacrylate ester monomers, inert mineral fillers, ytterbium fluoride, activators, stabilizers, colorants	Kerr
Clearfil SA Cement	Dual-cured	Bisphenol A diglycidylmethacrylate, sodium fluoride, triethylene glycol dimethacrylate, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, dl-camphorquinone, initiators, accelerators, catalysts, pigments, other	Kuraray
BisCem	Dual-cured	Base: bis-GMA, uncured dimethacrylate monomer, glass filler	Bisco
		Catalyst: Phosphate acidic monomer, glass filler	
SpeedCem	Self- or light-cured	Dimethacrylates, methacrylated phosphoric acid ester, benzoyl peroxide, inorganic fillers, copolymer, ytterbium trifluoride, initiators, stabilizers and pigments	Ivoclar Vivadent

TEGDMA = triethylene glycol dimethacrylate; bis-GMA = bisphenol glycidyl methacrylate.

*As per the manufacturer.

methacrylate is able to demineralize the hard tissues. However, despite the initial low pH (pH < 2.0), the enamel and dentin demineralization is only superficial.[39,40] An increase in pH (up to 7.0) is observed as a consequence of the reaction between the phosphate groups and alkaline fillers and the hydroxyapatite from enamel and dentin, neutralizing the resin's inherent acidity.[41] The bonding mechanism of these newly developed resins relies more on chemical bonding than on micromechanical retention. The acid groups chelate the calcium ions of the hydroxyapatite, promoting chemical adhesion.[42] In addition, carboxylic groups of polyalkenoic acid (found in RelyX Unicem, 3M ESPE) form ionic bonds with calcium present in the hydroxyapatite, positively influencing the chemical bonding.[43]

Self-adhesive resin cements are able to partially dissolve the smear layer without removing the smear plug within the dentinal tubules.[44] A thick smear layer may negatively influence the bond strength of self-adhesive cements, since the chemical bond is achieved with hydroxyapatite. Acid etching the dentin with phosphoric acid before the application of self-adhesive resin cement is detrimental to bond strength and must be avoided.[39,45] Conversely, the application of mild acidic agents, such as 25% polyacrylic acid (same dentin conditioner used for glass-ionomer cements), might remove the superficially loose bound fraction of the smear layer, thus improving adhesion.[46,47] However, the effect of mild acidic conditioner on self-adhesive resin cements must be validated clinically. Pretreatment of enamel with strong acid, such as 35% phosphoric acid, is highly recommended.[32,45]

Most self-adhesive resin cements yield bond strengths lower than etch-and-rinse resin cements or 10-MDP self-etch resin cements.[48,49] With the exception of RelyX Unicem, most self-adhesive resin cements maintain low pH for a long time after setting, which can adversely influence the bonding.[41]

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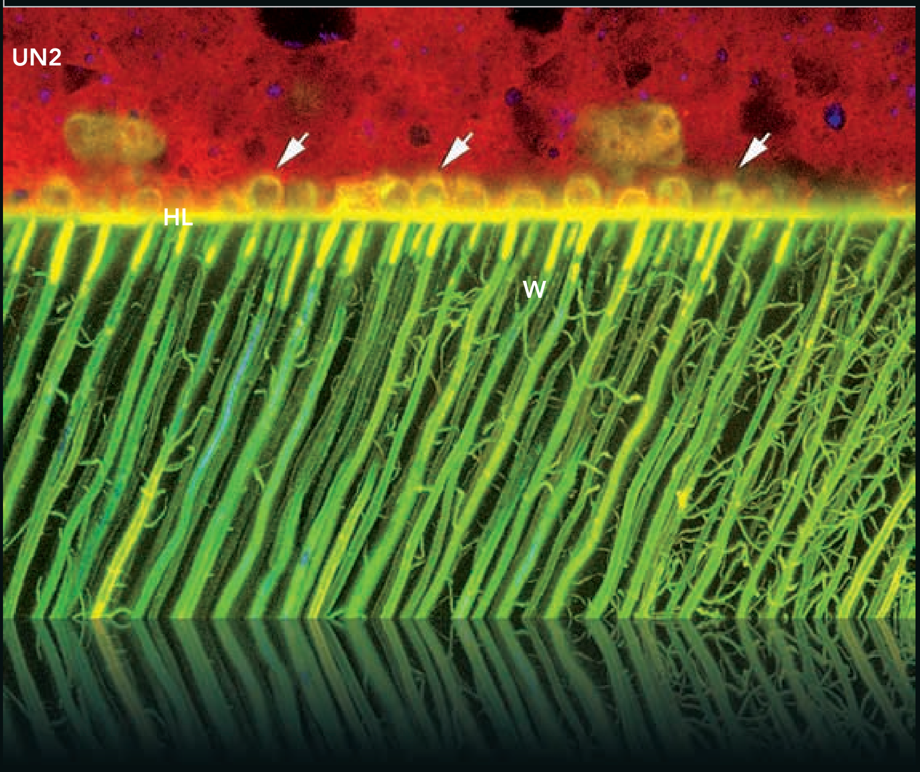


Fig 9 CLSM of self-adhesive resin cement showing water blisters protruding from the interface of the dentin and self-adhesive resin cement in a deep preparation. White arrows = water blisters; HL = pseudo hybrid layer; UN2 = Unicem 2; W = water.

During cementation, self-adhesive resin cements must be seated under pressure to ensure maximum contact of the cement with the dentin.[40] Insufficient seating pressure leads to a lack of intimate contact between the resin and tooth substrate, resulting in poor adaptation or low bond strength.[39,50]

Water degradation is still a problem for self-adhesive cements. Fluid permeation during the initial setting period deteriorates the bonding quality of the cement.[51] Findings from our laboratory at the Herman Ostrow School of Dentistry, University of Southern California Biomaterials Laboratory, California, USA, showed water blisters protruding from the dentin/self-adhesive resin cement bonded interface in a deep preparation (Fig 9). These water blisters may soften the resin cement and weaken the bond strength.

A recent clinical trial revealed good performance of a self-adhesive cement (RelyX Unicem) over 38 months for luting alloy–based restorations.[52] Another clinical investigation showed promising results when self-adhesive cement was used to adhesively cement lithium disilicate inlay restorations.[53] However, long-term clinical trials are needed to fully recommend self-adhesive resin cements as substitutes for etch-and-rinse resin cements for onlay, inlays, or porcelain veneers.

IMPROVING THE LONGEVITY OF EXPOSED RESIN CEMENT MARGINS

Low-viscosity composite resins can be used as resin cements to retain indirect restorations and to achieve an adequate seal between the restoration and tooth substrate. However, regardless of the marginal adaptation, a certain amount of resin cement at the margin of the indirect restoration will be exposed to the oral environment. Over time, the exposed cement will be subjected to water sorption,[54] subsurface degradation,[55] and wear processes that may result in marginal ditching.[56] All of these shortcomings lead to cement wear gap formation and marginal discoloration.

Wear of dental restorative materials and resin cements is a complex phenomenon involving both the material and the working environment.[57] Environmental factors influencing material wear usually include the type of load and counterbody,[57–59] applied force,[60] type and abrasiveness of abrasive medium,[57,61] and contact duration.[60]

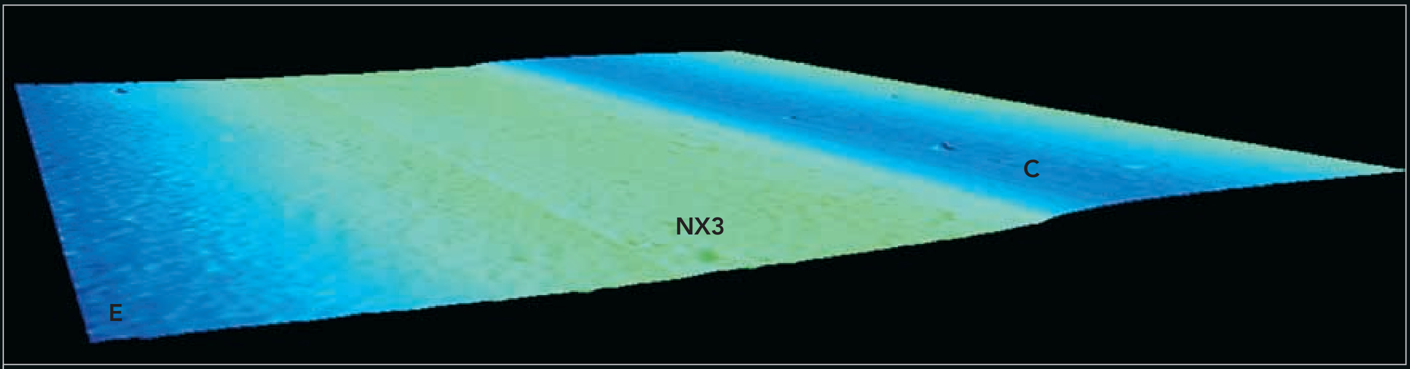
Wear of resin cements is influenced by the filler type and size,[58,62–64] filler load,[65] silane coupling agent,[65–67] nature of the matrix, degree of porosity, and degree of conversion.[68] The width of the exposed cement surface, determined by the marginal gap between the restoration and preparation, also significantly influences wear of the cement.[69]

The use of toothpaste with lower abrasiveness and less force during brushing results in less deterioration.[60,62] In vitro wear studies using a three-body wear model simulating the food bolus between contacting teeth reported higher wear values in comparison to simulations of toothbrush wear.[57–59] Tooth brushing movement parallel to the margin of the restoration results in increased wear by abrading the filler particles and resin matrix of the cement. Brushing movement perpendicular to the restoration margin results in less vertical wear by washing out only the resin matrix around the filler particles.[57] Resin cements with larger filler particles were shown to exhibit increased wear in comparison to resin cements with smaller filler particles.[58,62] A recent evaluation performed at our laboratory showed that preheated microhybrid composite used as a luting agent exhibited reduced wear compared to that of methacrylate- or phosphate-based resin cements (Figs 10 to 17).

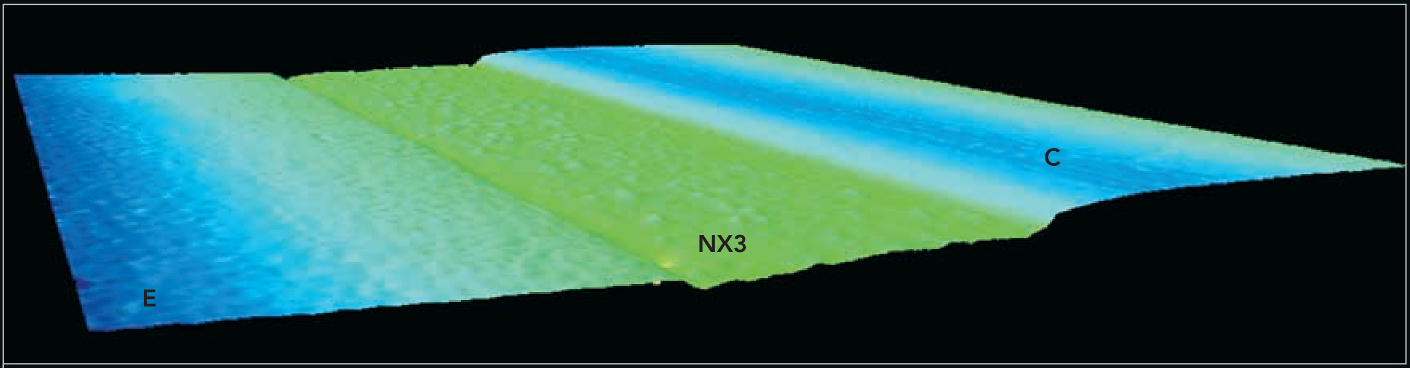
Preheating

In recent studies, the positive effect of preheating on the bond strength of composite materials was reported. The data showed that preheating to 55°C or 60°C reduced viscosity, improved flowability, and decreased film thickness of restorative composite resins.[70,71] Furthermore, preheating of light-cured composite resins resulted in significantly less microleakage at the cervical margins compared to that of flowable and non-preheated composites.[71] As a result of the enhanced monomer conversion, preheating was claimed to positively affect properties such as surface hardness, flexural modulus, fracture toughness, tensile strength,[70,71] and wear resistance,[72] which may also be clinically relevant for luting agents. Neither repeated nor extended preheating affected the degree of conversion.[73] However, recent investigations showed that preheating composite resin might increase polymerization shrinkage.[74,75]

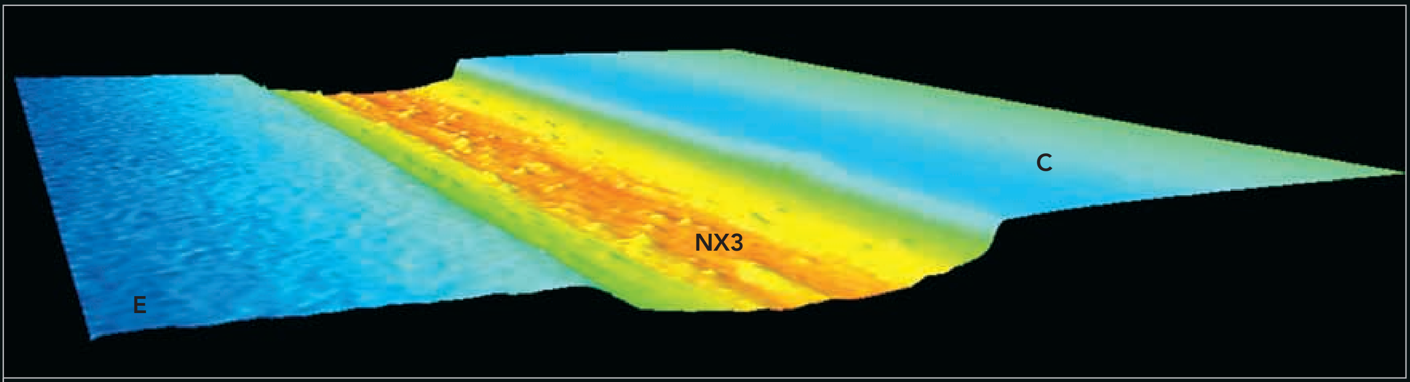
The applicability of preheating procedures to luting agents has also been investigated. For self-etch or self-adhesive resin cements, it was shown that warming the cements from refrigerator temperature to room or body temperature before use improved adhesion.[76] Preheating composite resins to 37°C and 54°C improved the adaptation to preparation walls.[77,78] However, temperatures higher than 37°C increased cuspal movement and may lead to postoperative sensitivity.[78] Resin cements were practically unusable at 60°C due to the accelerated setting mechanism, which meant that the cement was already set prior to dispensing.[76,79] Unfortunately, it is impossible to predict heated composite resin film thickness irrespective of the brand, filler shape, or volumetric filler loading (Figs 18a to 18c).[77] Therefore, while preheating a composite resin to slightly higher than body temperature has potential benefits, clinicians should be aware that increased film thickness might interfere with the bonding procedures of all-ceramic restorations.



10a



10b



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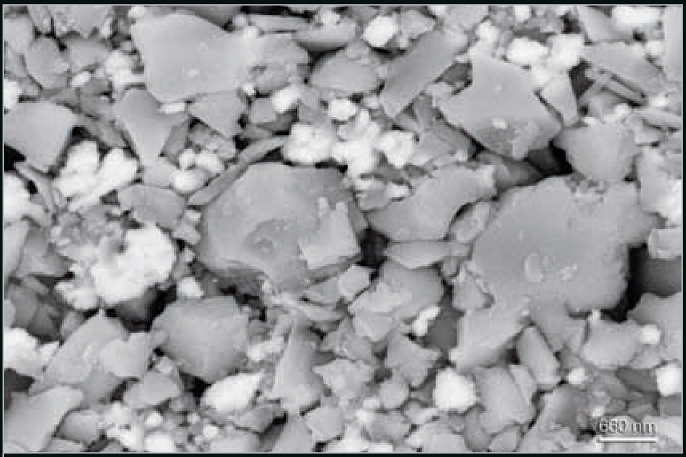

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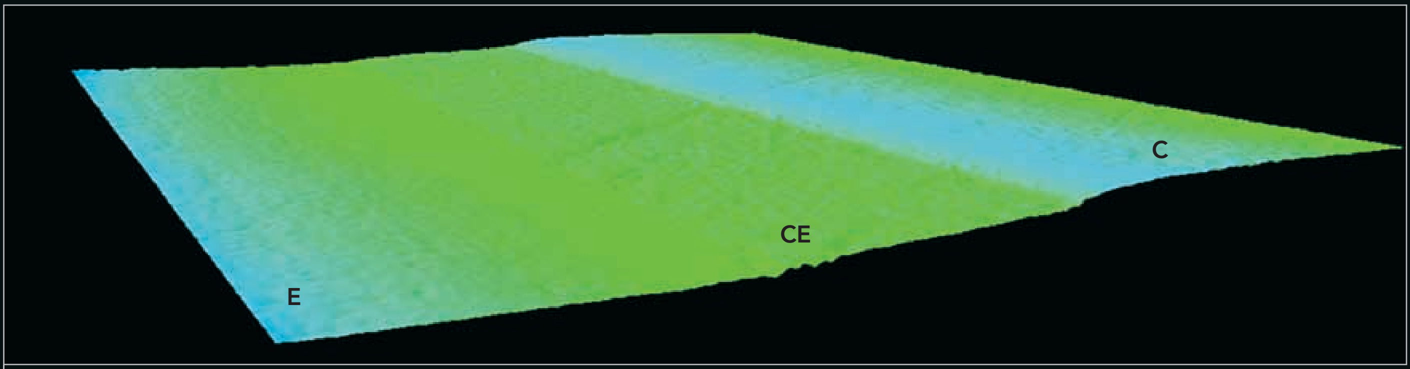
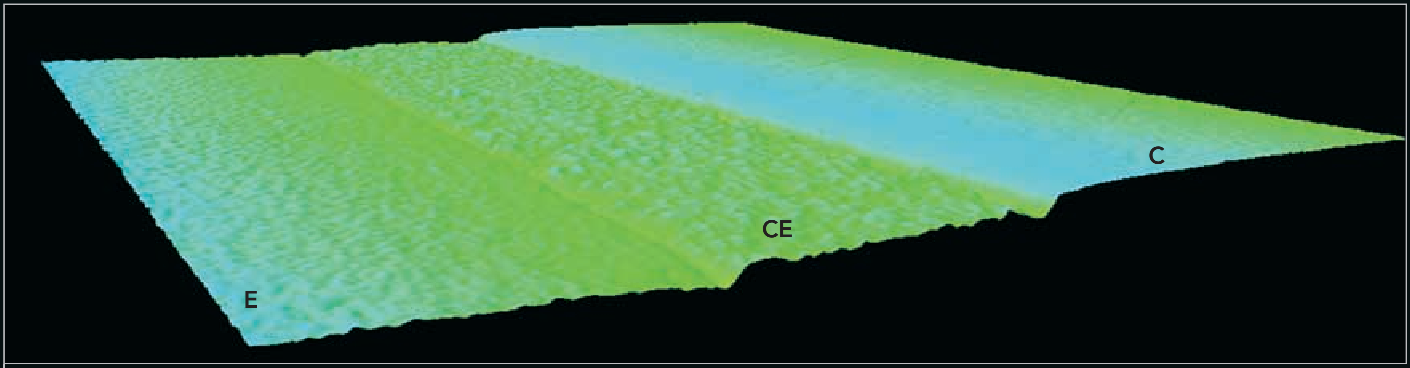
Figs 10a to 10c CLSM showing sequential wear of resin cement (Nexus 3, Kerr) exposed at the margin of a bonded ceramic restoration under simulated tooth brushing. *(a)* Baseline (no tooth brushing); *(b)* 20,000 tooth-brushing cycles; *(c)* 100,000 tooth-brushing cycles. E = enamel; NX3 = Nexus 3; C = ceramic.

Fig 11 FeSEM showing fillers of Nexus 3 after resin matrix removal (magnification ×35,000).



12a



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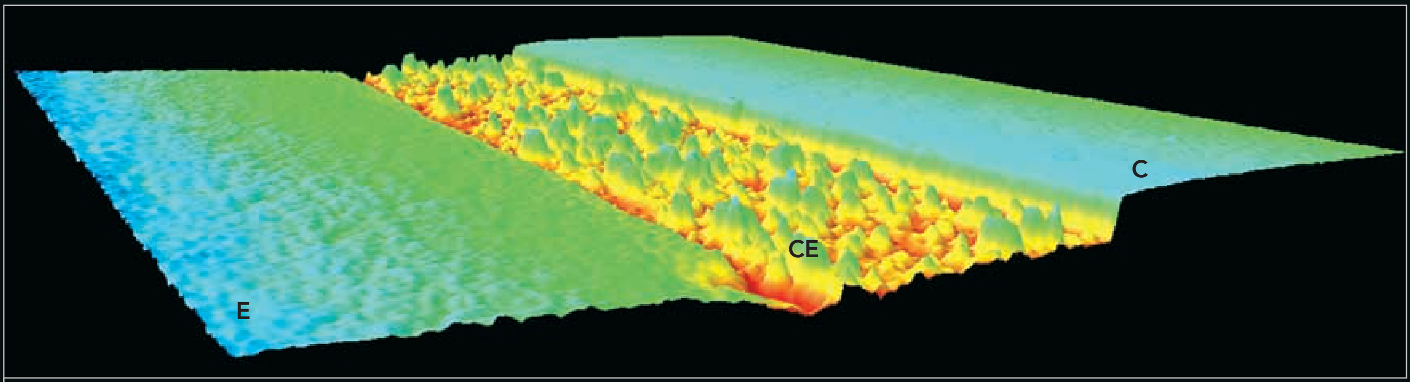
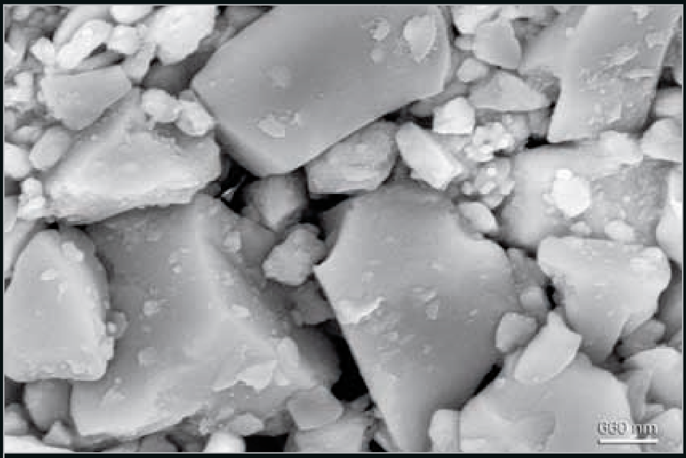


12c

Figs 12a to 12c CLSM showing sequential wear of resin cement (Clearfil Esthetic Cement, Kuraray) exposed at the margin of a bonded ceramic restoration under simulated tooth brushing. *(a)* Baseline (no tooth brushing); *(b)* 20,000 tooth-brushing cycles; *(c)* 100,000 tooth-brushing cycles. E = enamel; CE = Clearfil Esthetic Cement; C = ceramic.

Fig 13 FeSEM showing fillers of Clearfil Esthetic Cement after resin matrix removal (magnification ×35,000).



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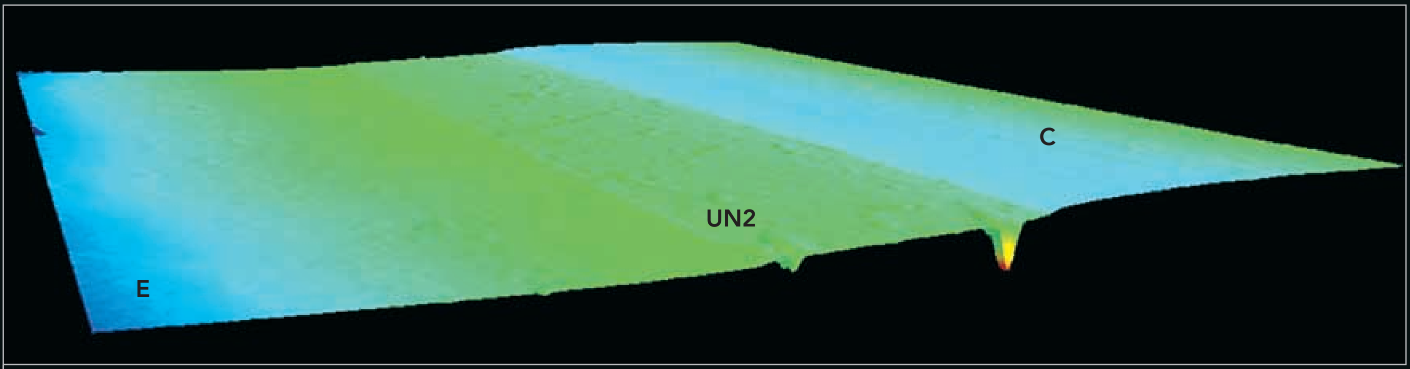

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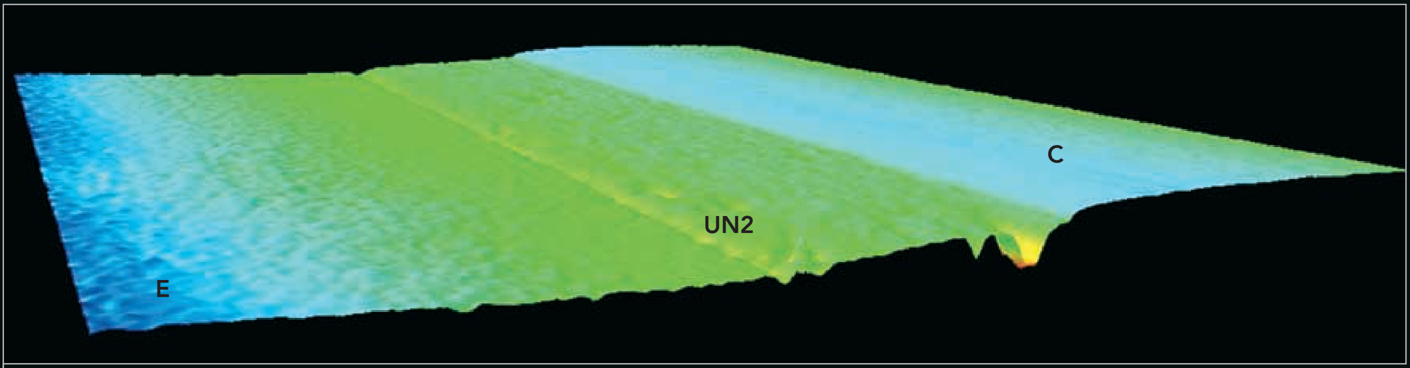

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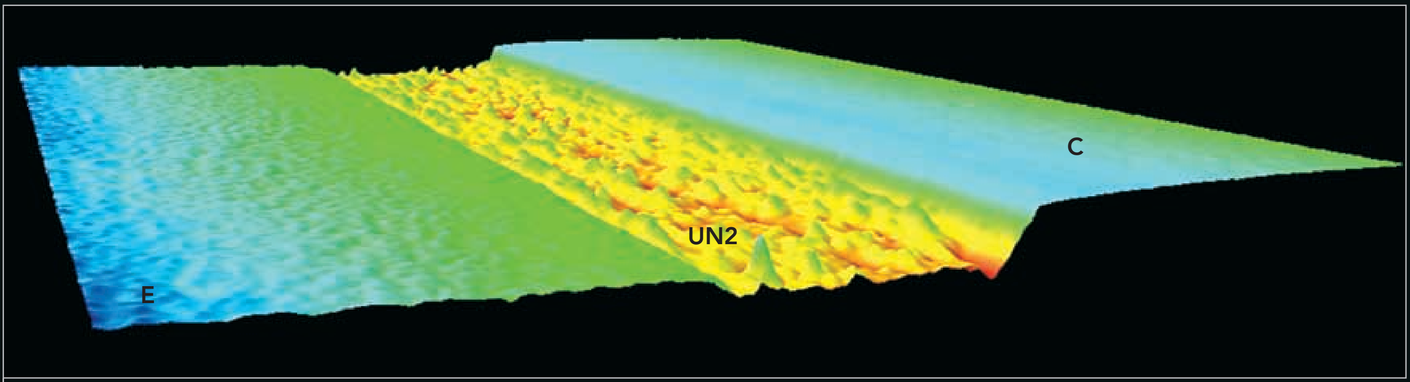
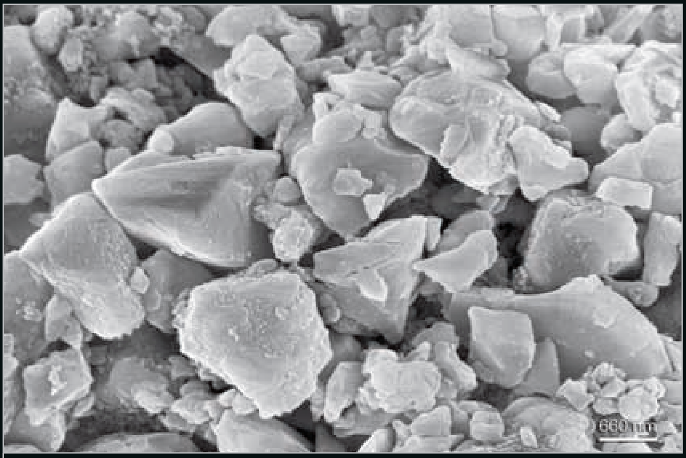


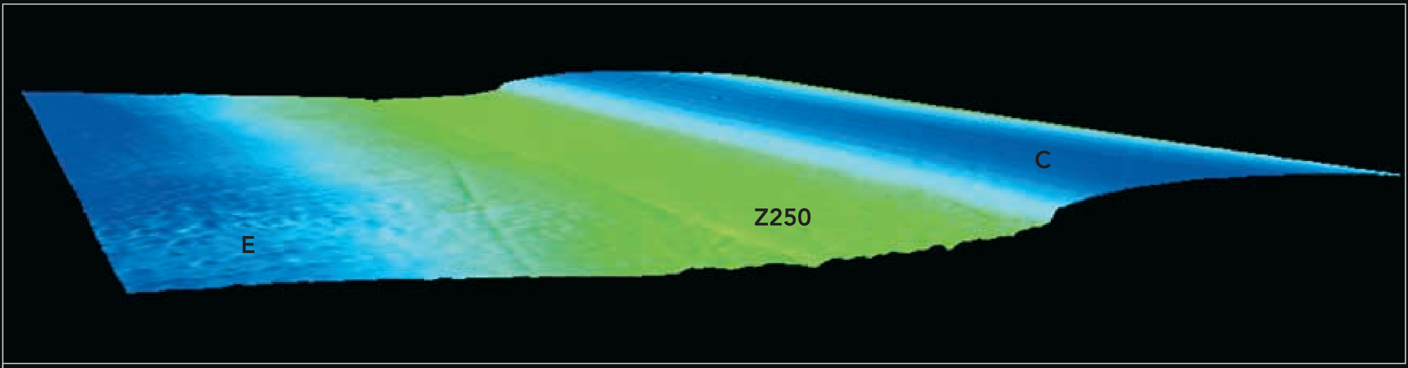
14c



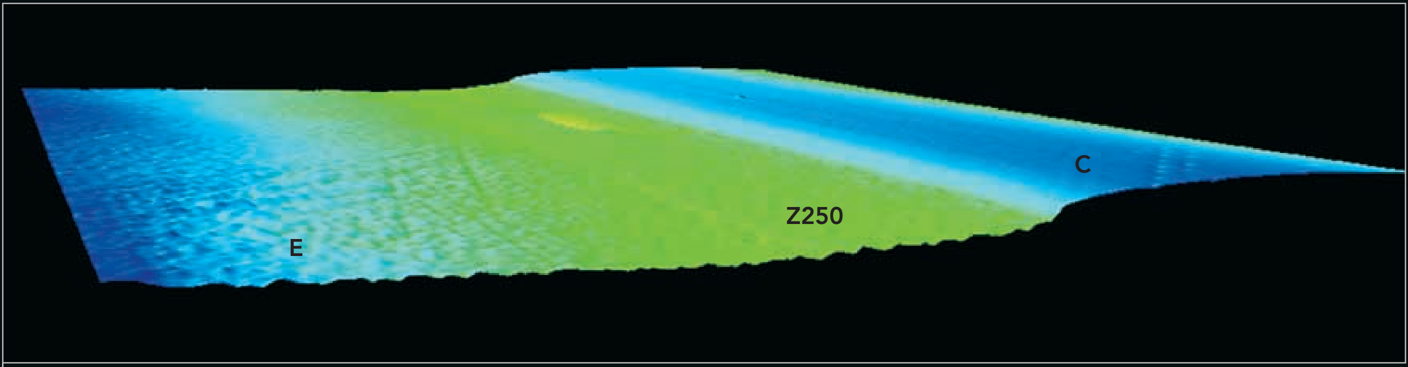
15

Figs 14a to 14c CLSM showing sequential wear of resin cement (Unicem 2, 3M ESPE) exposed at the margin of a bonded ceramic restoration under simulated tooth brushing. *(a)* Baseline (no tooth brushing); *(b)* 20,000 tooth-brushing cycles; *(c)* 100,000 tooth-brushing cycles. E = enamel; UN2 = Unicem 2; C = ceramic.

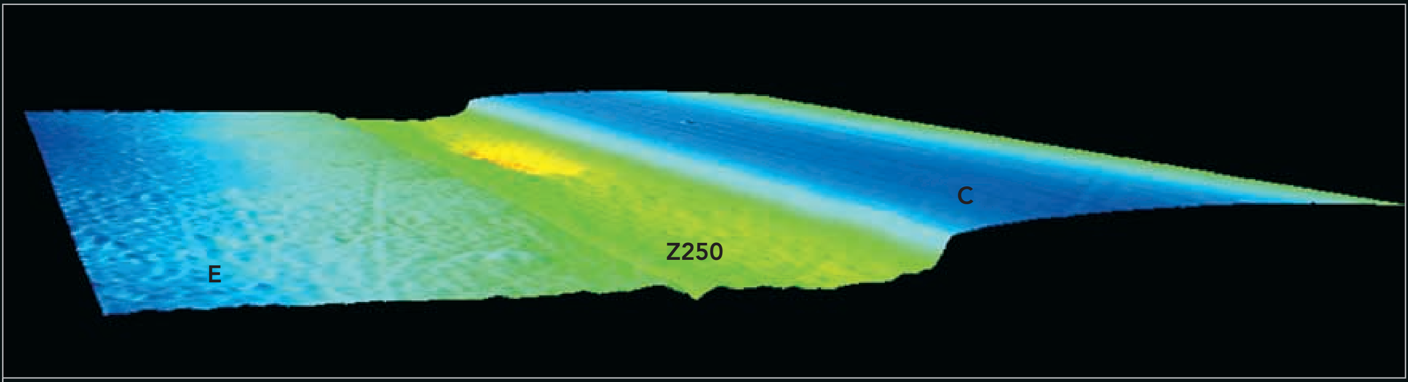
Fig 15 FeSEM showing fillers of Unicem 2 after resin matrix removal (magnification ×35,000).



16a



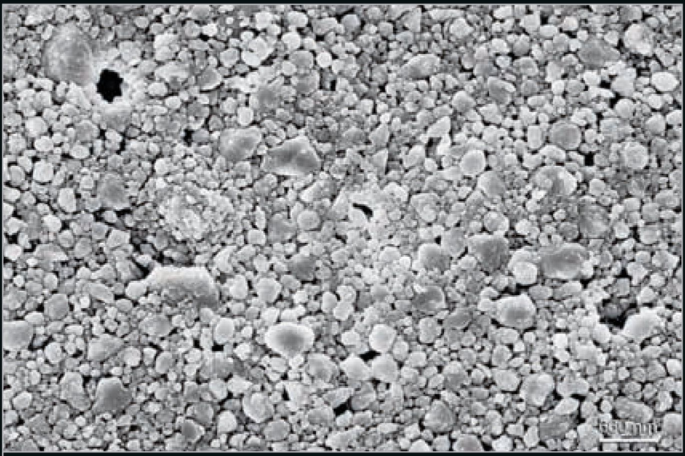
16b



16c

Figs 16a to 16c CLSM showing sequential wear of pre-heated microhybrid conventional composite resin (Filtek Z250, 3M ESPE) exposed at the margin of a bonded ceramic restoration under simulated tooth brushing. *(a)* Baseline (no tooth brushing); *(b)* 20,000 tooth-brushing cycles; *(c)* 100,000 tooth-brushing cycles. E = enamel; Z250 = Filtek Z250; C = ceramic.

Fig 17 FeSEM showing fillers of Filtek Z250 after resin matrix removal (magnification ×35,000).



17

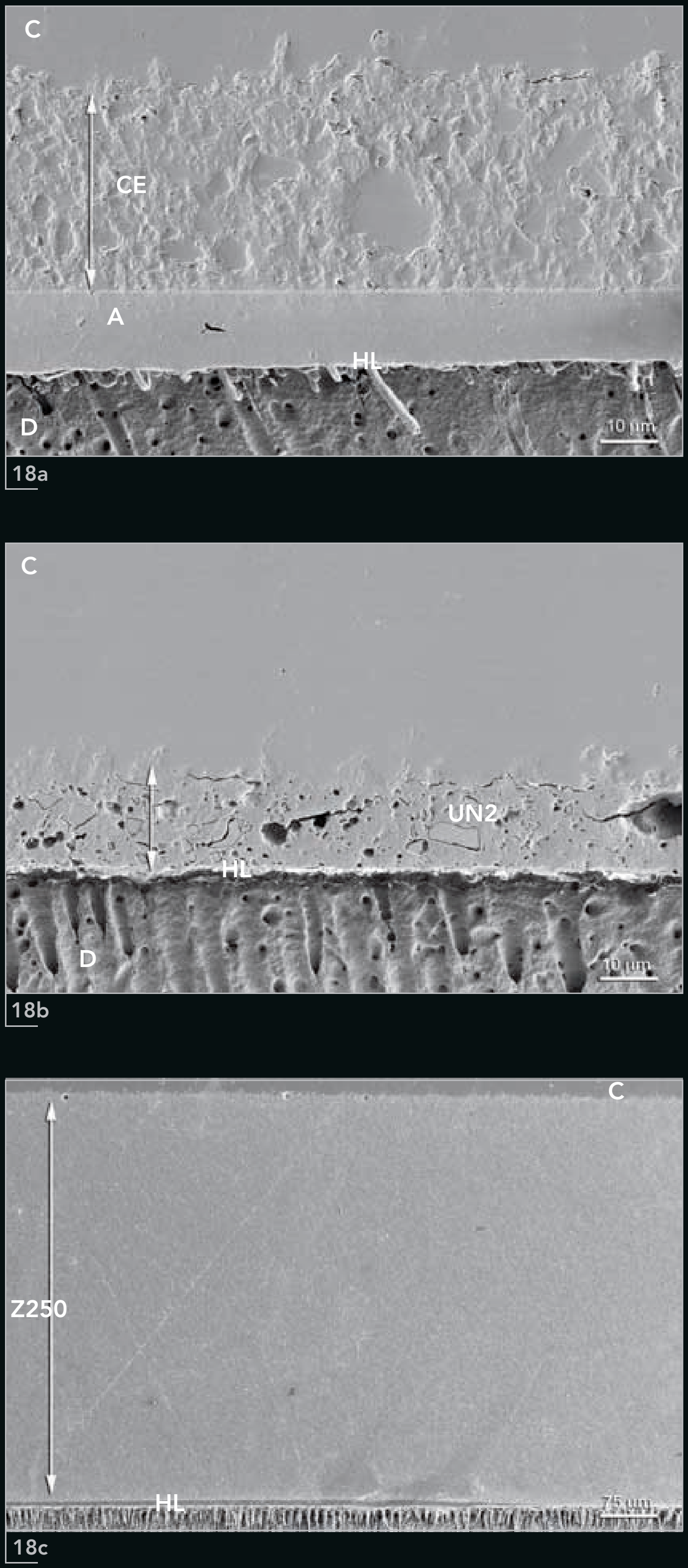


Fig 18 FeSEM showing the film thickness of different resin cements and preheated composite resin. *(a)* Clearfil Esthetic Cement (magnification ×1,500); *(b)* Unicem 2 (magnification ×1,500); *(c)* preheated Filtek Z250 (magnification ×350). D = dentin; A = adhesive layer; CE = Clearfil Esthetic Cement; UN2 = Unicem 2; Z250 = Filtek Z250; HL = hybrid layer; white arrow = film thickness.

IMPROVING BONDING EFFECTIVENESS OF ADHESIVE RESIN CEMENTS

All bonded interfaces are subject to degradation. The following steps can be taken to reduce these deleterious effects:

- Leave sclerotic dentin during abutment preparation. The presence of mineral deposits in the dentin tubules reduces dentin permeability.[80]
- Freshly cut dentin may be prebonded with dentin bonding agent (Fig 19).[81] The application of a dentin adhesive to freshly prepared dentin decreases dentin permeability.[82] However, not all adhesives are suitable for this task.[83] Only highly filled etch-and-rinse dentin adhesive (Optibond FL, Kerr) provides acceptable long-term bonding effectiveness.[84,85]
- Avoid eugenol-containing temporary cements and thoroughly clean the preparation before bonding. Eugenol-containing temporary cement reduces the bond strength of etch-and-rinse, self-etch, and self-adhesive resin cements.[86,87] Furthermore, a recent study showed that even dentin contaminated with eugenol-free temporary cement decreased the bonding of adhesive cements.[88] Therefore, more important than the type of temporary cement used is proper cleaning of the preparation before bonding. Cleaning can be effectively performed with low-pressure and small-particle air abrasion, followed by strong water spray.[85,87] The long-term effect of aluminum oxide cleaning and tribochemical coating on prebonded dentin is detrimental to the bonded interface (Figs 20a and 20b).
- Before cementation, apply local anesthesia with vasoconstrictors. Local anesthesia decreases transdentinal flow by 70%.[89] Consequently, the pulpal pressure diminishes during adhesive restorative procedures.[90]
- Chlorhexidine may be used after acid etching for etch-and-rinse dentin adhesives. Chlorhexidine, which is an antibacterial agent with matrix metalloproteinases inhibiting properties,[91] preserves the collagen integrity of the hybrid layer created by etch-and-rinse adhesives,[92] thus diminishing its degradation.[93] Conversely, chlorhexidine negatively affects the integrity of self-etch or self-adhesive resin cements bonded to dentin and must be avoided with these materials.[94]
- Application of a hydrophobic resin coat improves the bond strength of self-etch adhesives. Hydrophobic resin coating increases the hydrophobicity of the adhesive layer. The adhesive interface will be less permeable to water movement,[30,95] less susceptible to water degradation,[96] and thus more compatible with self- and dual-cured resin cement.[95] However, excessive film thickness of the hydrophobic layer may interfere with the fit of the indirect restoration.[51]
- Increase the seating pressure. This procedure suppresses the absorption of water and globule formation, thus reducing water infiltration from the underlying dentin into the bonded interface and enhancing the quality of the adhesive interface.[97] Additionally, increased seating pressure reduces the amount of porosities at the interface, improving the adaptation and bond strength.[39,97]
- Apply ultrasonic vibration (Figs 21a to 21d). When ultrasound is used during cementation, it affects the thixotropic properties of the luting agents, decreases the viscosity, and increases the temperature and seating speed.[98,99] This leads to a uniform, densely packed cement layer with less porosity,[98,99] which is especially important for self-adhesive resin cements that need close contact with dental tissues to create acceptable bond strengths.

CONCLUSION

The durability and clinical success of bonded esthetic restorations are intimately related to the bond strength of the adhesive resin–based luting materials. Proper understanding of the principles and limitations of these materials and procedures will ensure successful, long-lasting restorations.

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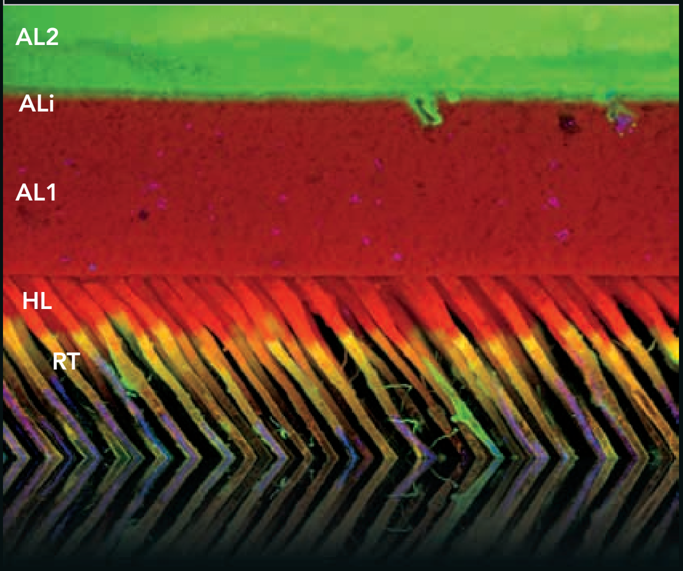

Fig 19 CLSM showing prebonded dentin after preparation and after adhesive cementation. There is minimal interaction of the second adhesive layer with the original hybrid layer. The adhesive layer interface is the area susceptible to adhesive failure. HL = hybrid layer; AL1 = first adhesive layer (after prebonding); AL2 = second adhesive layer (after cementation), ALi = adhesive layer interface; RT = resin tags.

20a

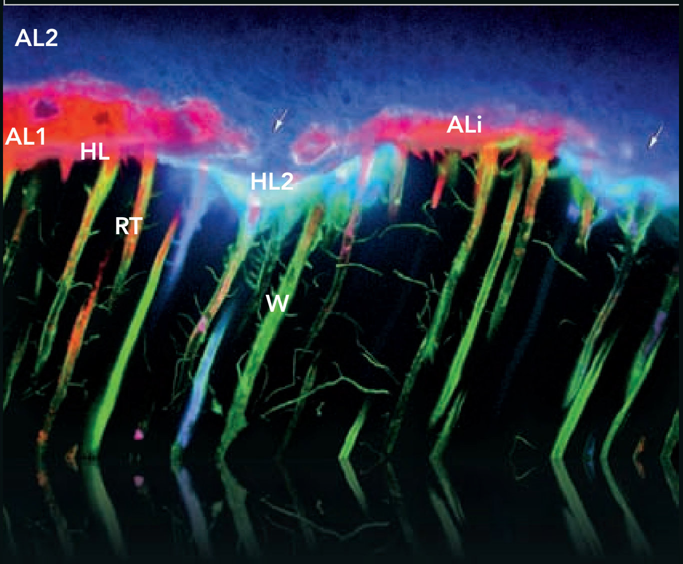

Fig 20a CLSM showing the effects of aluminum oxide cleaning on prebonded dentin after preparation and after adhesive cementation. Aluminum oxide air abrasion (white arrows) resulted in partial removal of the original hybrid layer (HL), followed by the formation of a new ghost-like hybrid layer (HL2). The adhesive layer interface (ALi) was also modified, allowing for the incorporation of aluminum oxide powder even after cleaning. AL1 = first adhesive layer; AL2 = second adhesive layer; RT = resin tags; W = water.

20b

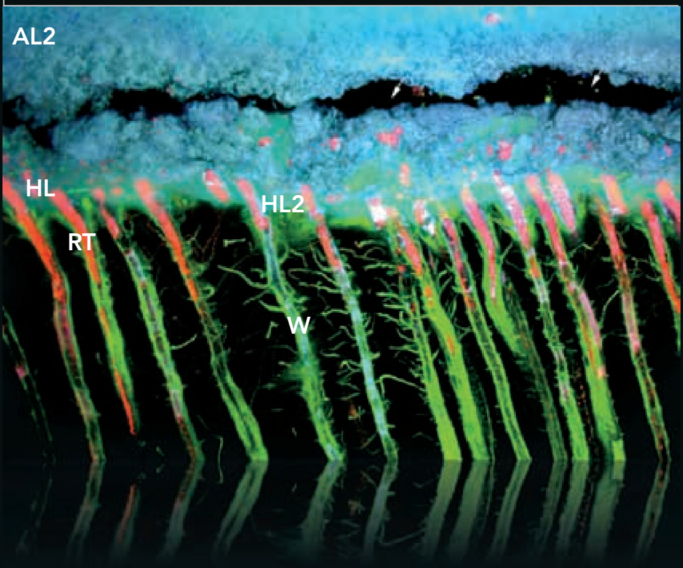

Fig 20b CLSM showing the effects of tribochemical coating on prebonded dentin. Tribochemical coating resulted in removal of the first hybrid layer (HL) and formation of a new ghost-like hybrid layer (HL2) susceptible to dentinal fluid transudation. The first adhesive layer (AL1) was partially removed, and microgaps (white arrows) were found at the new adhesive layer (AL2). ALi = adhesive layer interface; RT = resin tags; W = water.

21a

21b

21c

Figs 21a to 21d Clinical sequence of a conservative adhesive treatment of eroded maxillary posterior teeth: *(a)* Preoperative view; *(b)* wax-up; *(c)* ceramic onlays on the cast; *(d)* ceramic onlays after adhesive cementation. (Ceramist: Jose Carlos Romanini, Londrina, Brazil.)

21d

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