Effects of Ethanol-wet Bonding Technique on Root Dentine Adhesion

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Objective: *To investigate the effects of ethanol-wet bonding on the adhesion of experimental hydrophobic and commercial hydrophilic adhesives to root dentine.*

Methods: *A total of 43 single-rooted integrated human premolars were selected and sectioned. Of the 86 initially obtained specimens, 66 were randomly and equally divided into water-wet bonding and ethanol-wet bonding groups (n = 33). The specimens of each group were subdivided into three subgroups (n = 11) based on different adhesives: two experimental hydrophobic adhesives (Bis-GMA/TEGDMA, BT; and UDMA/TEGDMA, UT) and one commercial hydrophilic adhesive (Adper™ Single Bond 2, SB). The root surfaces were ground, acid-etched and rinsed and resin composite applied. After storing in distilled water for 24 h at 37°C, the shear bond strength (SBS) of each specimen was measured. A sample from each subgroup was randomly selected and prepared for scanning electron microscopy (SEM) analysis. The remaining 20 specimens were used in the contact angle (CA) experiment, and the values of CA were measured. SBS was analysed with two-way ANOVA/Tukey's multiple comparison test and CA with independent sample* t *test.*

Results: *A significant increase in SBS to root dentine was observed in the ethanol-wet bonding group compared with the traditional water-wet bonding group. The experimental hydrophobic adhesives (UT group) with ethanol-wet bonding presented the highest SBS (22.44 ± 3.32 MPa). CA increased significantly after the dentine surfaces were dried, especially for the water-saturated group.*

Conclusion: *The adhesion to root dentine surfaces with ethanol-wet bonding may be superior to water-wet bonding.*

Key words: *contact angle, dental adhesives, ethanol, root dentine, shear bond strength*

Water-wet bonding using hydrophilic and acid resin monomers has substantially improved initial bonding[1]. Traditional water-wet bonding can fully expand the dentine matrix and provide relatively wide interfibrillar spaces to allow maximal infiltration of the resin monomer. However, a water-saturated dentine matrix is too weak to resist evaporation stress during solvent evaporation, leading to a dramatic shrinkage of the dentine matrix[2]. When the interfibrillar space becomes smaller, the resin monomers cannot infiltrate deep into the dentine matrix, resulting in the exposure of unprotected collagen fibrils. Therefore, the quality of resin–dentine adhesion is affected. The residual water in the dentine matrix after solvent evaporation leads to collagen fibril hydrolysis, which possibly deteriorates the durability of dental bonding[3,4].

Contemporary adhesives are hydrophilic and permeable to water from the underlying bonded dentine, leading to the degradation of resin–dentine bonds. These properties also affect the mechanical features of polymerised adhesives. To enhance the durability of resin–dentine bonds, future dentine adhesives should be rendered less hydrophilic[5-7]. Hydrophobic resins are

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inclined to exhibit phase separation if water is present in dentine. This may be avoided by replacing the water in the demineralised collagen matrix with ethanol[2].

The concept of ethanol-wet bonding, where the water in water-saturated acid-etched dentine is replaced with ethanol to create ethanol-saturated dentine[8], originated from the principal similarities in the bonding procedures and soft tissue embedding[9,10]. Ethanol can replace water in the dentine matrix by chemical dehydration, which reduces the shrinkage of the dentine matrix and achieves better resin–dentine adhesion. Since ethanol is compatible with hydrophobic monomers, this technique may prevent phase separation of hydrophobic monomers applied to ethanol-saturated dentine. The ultimate goal of ethanol-wet bonding is to infiltrate interfibrillar spaces and dentinal tubules with hydrophobic dimethacrylate resins to decrease hydrolysis and to enhance adhesion to the dentine[2,9].

It is considered that adhesion to radicular dentine is more unpredictable than that to coronal dentine[11,12]. Although some positive results have been obtained for ethanol-wet bonding on coronal dentine, the biocompatibility of dehydrated (highly concentrated) alcohol and water contamination are the main concerns in the application of the wet bonding technique to vital pulp tooth dentine[13,14]. Presumably, ethanol-wet bonding may be suitable for the dry, non-vital conditions of root canal dentine after endodontic treatment, regardless of the biocompatibility of dehydrated alcohol.

Wettability is one of the most important factors in adhesion[15]. Adhesion requires high wettability so that the adhesive spreads spontaneously on the dentine matrix. Measurement of wettability can be expressed in terms of the contact angle, which has an inverse relationship with wettability.

In this study, three groups of adhesives, comprising two experimental hydrophobic adhesives mimicking the commercial two-step etch-rinse adhesive and one commercial adhesive, were applied onto root dentine through the water-wet and ethanol-wet bonding techniques to compare the influence of technique on shear bond strength (SBS). Samples were also analysed by scanning electron microscopy (SEM).

The null hypothesis is that there is no difference in the bonding of different adhesive systems to root dentine with water-wet and ethanol-wet bonding techniques.

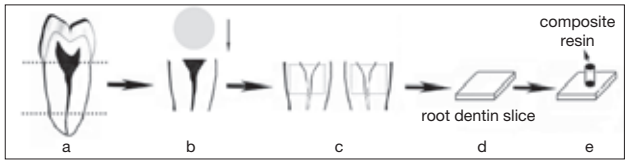

Fig 1 Schematic diagram of sample preparation. a) The crowns and the roots of the apical third of each premolar were removed using a low-speed diamond saw. b) The remaining root segments were sectioned buccolingually into halves. c) Two root slices were obtained from each root. d) The root dentine surface was treated with the ethanol-wet or water-wet technique before a suitable amount of adhesive was applied to the root dentine. e) A composite resin cylinder was constructed with a height of 3 to 4 mm.

Materials and methods

Tooth preparation

A total of 43 single-rooted premolars were collected after the donors' informed consent was obtained under a protocol approved by the Ethics Committee of Wuhan University. The teeth were extracted for orthodontic reasons, stored in 1% chloramine-T at 4°C and used within 1 month. The crowns and the roots of the apical third of each premolar were removed using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The remaining root segments were sectioned buccolingually into halves (Fig 1). Finally, two root slices were obtained from the same root. The root dentine surface was ground with a 600-grit silicon carbide paper for 60 s to create a standardised smear layer. The root sections were divided randomly into six groups (n = 11): two bonding techniques × three different adhesives.

- Group 1: BT (Bis-GMA/TEGDMA) with water-wet bonding technique
- Group 4: BT with ethanol-wet bonding technique
- Group 2: UT (UDMA/TEGDMA) with water-wet bonding technique
- Group 5: UT with ethanol-wet bonding technique
- Group 3: SB (Adper™ Single Bond 2) with water-wet bonding technique
- Group 6: SB with ethanol-wet bonding technique.

Bonding procedures

Experimental adhesives – BT and UT – were formulated by combining 50 wt% resin monomer mixtures with 50 wt% ethanol (Table 1).

For Groups 1 to 3 (water-wet bonding technique), all dentine surfaces were acid-etched with 35% phosphoric acid gel (Scotchbond™ etchant, 3M ESPE, St. Paul, MN, USA) for 15 s and then rinsed for 15 s with deionised water. Excess water was blotted using filter paper until the surface appeared glistening without pooling of water.

Table 1 Composition of the experimental and commercial adhesives

Group	Solvent	Resin composition	Corporation
BT	Ethanol	35% Bis-GMA; 14.25% TEGDMA; 0.5% EDMAB; 0.25% CQ; 50% ethanol	–
UT	Ethanol	35% UDMA; 14.25% TEGDMA; 0.5% EDMAB; 0.25% CQ; 50% ethanol	–
SB	Ethanol and water	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water and ethanol	3M ESPE

Abbreviations:
Bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]-phenyl propane
UDMA: 1,6-bis(methacryloyloxy-2-ethoxycarbonylamino)-2,4,4-trimethyl-hexane
TEGDMA: triethyleneglycol dimethacrylate
EDMAB: ethyl-4-N,N-dimethyl aminobenzoate
CQ: camphorquinone
HEMA: hydroxyethyl methacrylate

For Groups 4 to 6 (ethanol-wet bonding technique), the water-moist dentine sections after acid etching were treated with 100% ethanol for 20 s to replace the water in the dentine matrix. Excess ethanol was then removed using filter paper, leaving an ethanol-wet dentine surface.

During bonding, a suitable amount of each adhesive was applied to the dentine through microbrush agitation for 15 s. A second application of fresh adhesive was then made, resulting in a total application time of 30 s. Excess solvent was evaporated with a gentle air stream for 10 s at a distance of 150 mm, and then the adhesive was light cured for 20 s using a light-curing unit (Spectrum, Dentsply, Konstanz, Germany) with a power output of 600 mW/cm^2. Resin composite buildups were made with three 1.5 mm increments of Valux™ plus composite (3M ESPE) that were individually light cured for 40 s. All bonded samples were incubated in 37°C water for 24 h.

SBS testing

A composite resin cylinder was constructed with a height of 3 to 4 mm and a diameter of 2.8 mm after using adhesive on the dentine surface. After storing in distilled water at 37°C for 24 h, the SBS of each specimen was measured using a universal testing machine (Instron, Norwood, MA, USA) at a cross-head speed of 1 mm/min. The load, recorded in newtons, was divided over the calculated surface area and the SBS was calculated in MPa. The results were statistically analysed using two-way ANOVA and Tukey's multiple comparison test ($\alpha = 0.05$).

SEM evaluation

A sample from each group was selected randomly and prepared for SEM analysis. After storing for 24 h at 37°C, the samples were sectioned axially through the restoration. The bonding interface was etched with 6% hydrochloric acid for 10 s, washed with deionised water, treated with 5% sodium hypochlorite for 5 min, washed with deionised water and then gently air dried for 10 s. The specimens were dried and sputter-coated with gold and then the interfaces were observed under an SEM (S-650, Hitachi, Tokyo, Japan).

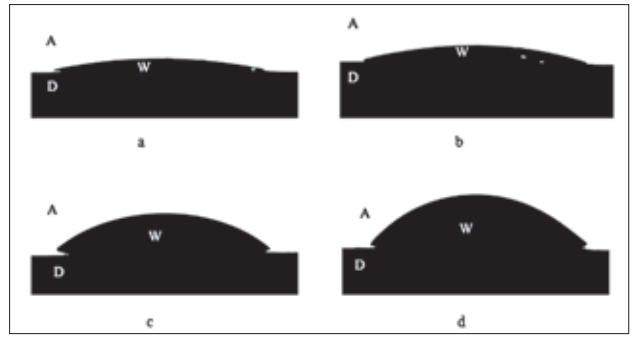

Fig 2 Images of the contact angle on: a) ethanol-wet root dentine surface, b) water-wet root dentine surface; and images of the contact angle after air-drying on: c) ethanol-saturated root dentine surface, d) water-saturated root dentine surface. (A: air; D: dentine; W: water)

Contact angle testing

A total of ten single-root premolars were used in contact angle testing (Fig 2). The root dentine sections were prepared as before (Table 2). The two dentine sections from each root were divided medially into two groups ($n = 10$).

All flat dentine surfaces were carefully kept as clean as possible to ensure credible results. The hydration

Table 2 Groups in the contact angle testing

Group	Method	Treatment	
		Wet dentine	Dry dentine
1	Water	The ground dentine section was etched (15 s) and rinsed (15 s); CA was tested and CA1w was obtained	The water-wet dentine was air-dried for 60 s; CA was tested and CA1d was obtained
2	Ethanol	The ground dentine section was etched (15 s) and rinsed (15 s) then covered with ethanol for 20 s; CA was tested and CA2w was obtained	The ethanol-wet dentine was air-dried for 60 s; CA was tested and CA2d was obtained

Abbreviations:
CA: contact angle

state of group 1 was carefully controlled before the contact angle test images were obtained. The testing was then repeated for group 2. All contact angle measurements were made at 25°C.

Wettability was quantified by contact angle measurements using deionised water pendant drops placed using a microsyringe. Pendant drops of deionised water with a volume of 1 µl were placed on each specimen.

Drop images were acquired by dynamic testing, and the same dentine surface location was photographed five times every second. The contact angles were measured using the θ/2 method by the same operator (under the single-blind method). The means of the five contact angles of each sample were calculated and then analysed using an independent sample *t* test.

Statistical analysis

Two-way ANOVA with a general linear model was used to examine the effects of the dental adhesives and bonding techniques on SBS. Multiple comparisons were performed by Tukey's test with the statistical significance set at $\alpha = 0.05$. The contact angle values were analysed using an independent sample *t* test with $\alpha = 0.05$. All statistical analyses were carried out using SPSS (version 13.0 for Windows, SPSS, Chicago, IL, USA).

Results

Shear bond strength

The mean SBS values (MPa) and standard deviations are shown in Table 3. For the two experimental adhesive groups (BT and UT), SBS was significantly higher in the groups treated with 100% ethanol than that in the groups treated with water ($P = 0.000$). However, SBS of the commercial adhesive group (SB) showed no significant difference between the water-wet and ethanol-wet groups ($P = 0.098$).

The UT experimental hydrophobic adhesive with ethanol-wet bonding exhibited the highest SBS at 22.44 ± 3.32 MPa. For the ethanol-wet bonding groups, SBS showed no differences among UT, BT, and SB groups ($P > 0.05$). For the water-wet bonding groups, the SB group exhibited the highest SBS at 15.71 ± 2.91 MPa, which is significantly higher than the BT version ($P = 0.019$).

Contact angle

The mean values of the contact angle (degrees) and the standard deviations are shown in Table 4. For the wet dentine surface, there were no differences between the contact angles of the water-wet group (18.74 ± 7.91 degrees) and the ethanol-wet group (24.48 ± 9.23 degrees) ($P = 0.152$). After the dentine surfaces were dried, the contact angle of the water-wet group exhibited the highest value, 57.66 ± 11.70 degrees, which was significantly higher than that of ethanol-wet group (38.85 ± 8.74 degrees) ($P = 0.002$).

Scanning electron microscopy

In Figure 3, parts a, c and e show the interfaces of the ethanol-saturated dentine surface infiltrated with BT, UT and SB, respectively. Parts b, d and f show the interfaces of the water-saturated dentine surface infiltrated with BT, UT and SB, respectively. As can be seen in the images, when the adhesive materials were infiltrated into the ethanol-saturated dentine, the resin tags were more condensed and regular. In Figures 3a and 3c (experimental hydrophobic adhesives with ethanol-wet

Table 3 Shear bond strength (MPa) for each adhesive with different bonding techniques

	Water	Ethanol
BT	10.73 ± 2.98	19.23 ± 1.86*
UT	11.28 ± 3.40	22.44 ± 3.32#
SB	15.71 ± 2.91$	19.81 ± 3.04

*$P < 0.05$ versus BT water group; #$P < 0.05$ versus UT water group; $$P < 0.05$ versus BT water group; $n = 10$, two-way ANOVA and Tukey's multiple comparison test.

Table 4 Contact angle (degrees) of the root dentine surface after ethanol or water treatment

	Wet dentine	Dry dentine
Water	24.48 ± 9.23	57.66 ± 11.70*
Ethanol	18.74 ± 7.91	38.85 ± 8.74

*$P < 0.05$ versus ethanol-wet group with dry dentine, $n = 10$, independent sample t test.

bonding), almost every dentine tubule was infiltrated with the resin tags, but the resin tags in the UT version (Fig 3c) were more evenly distributed.

Discussion

Early dentine adhesives were relatively hydrophobic and were applied to dry dentine substrates without first removing the smear layer. As a result, the bond strengths were very low. Bonding was substantially improved by the application of hydrophilic adhesives and use of the water-wet technique. In comparison, contemporary adhesives are now easier to use. However, the simplification of bonding steps has not improved the quality or the durability of resin–dentine bonds[16-18]. Recent studies have shown that the application of hydrophobic Bis-GMA/TEGDMA co-monomer blends onto acid-etched dentine is possible using ethanol-wet bonding[9,19]. It was found that resin monomers seem to penetrate ethanol-saturated dentine more thoroughly than water-saturated dentine[8]. Furthermore, resin–dentine bonds with the ethanol-wet bonding technique are more durable than those with water-wet bonding[20].

The chemical compositions of the two experimental adhesives (BT and UT) are shown in Table 1. In a study by Sadek et al, the 50/50 version (50 wt% ethanol and 50 wt% resin monomer mixture) of the experimental hydrophobic primer exhibited the highest tensile strength in ethanol-saturated acid-etched dentine compared with the 20/80, 30/70, 40/60 and 75/25 versions[9]. Consequently, the 50/50 proportion was chosen for use as the experimental adhesive protocol in this study. The bonding procedure simulated that used for an ethanol-based hydrophilic adhesive, SB, a two-step etch-and-rinse adhesive regarded as an excellent dentine adhesive[19].

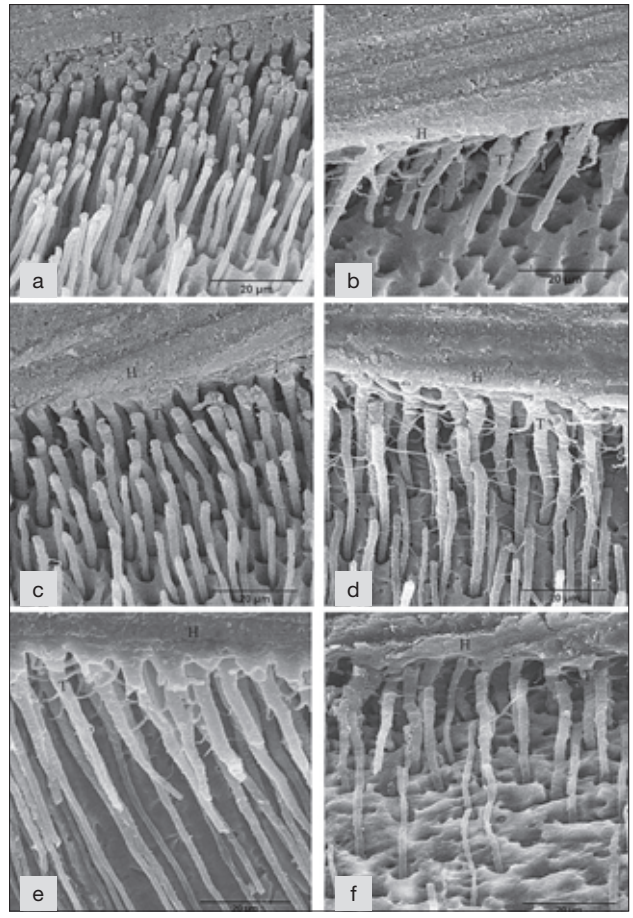

Fig 3 Scanning electron micrographs of root dentine bonding interface. Parts a, c and e show the adhesives BT, UT and SB, respectively, applied to root dentine with the ethanol-wet bonding technique; parts b, d and f show the adhesives BT, UT and SB, respectively, applied to root dentine with the water-wet bonding technique. The images of the ethanol-saturated root dentine bonding interface (a, c, e) show the hybrid layer is more condensed and resin tags better distributed and more regular compared with the water-saturated root dentine bonding interface (b, d, f). (H: hybrid layer; T: resin tag)

In the present study, the bonding technique had a significant influence on the strength of the resin–dentine bond. For the two experimental adhesives, the SBS was significantly increased in the ethanol-wet bonding groups compared with the water-wet bonding groups (Table 3). Therefore, the null hypothesis, that there is no difference in the bonding of different adhesive systems to root dentine with the water/ethanol-wet bonding technique, is rejected.

Pashley et al postulated that the collapse of demineralised dentine matrices is an active process involving the rapid and spontaneous development of new hydrogen bonds (H-bonds) between adjacent collagen peptides[8]. Adhesives cannot re-expand the matrix unless they can break the hydrogen bonds. Hoy's solubility parameters theory provided a new insight into the mechanism of adhesion to dentine.

Hoy's solubility parameters include dispersive forces (δ_d), polar forces (δ_p), hydrogen bonding forces (δ_h) and total cohesive forces (δ_t) (equivalent to Hildebrand's solubility parameter)[21-23]. δ_h values are: 14.8 $(J/cm^3)^{1/2}$ for dry collagen; 40.4 $(J/cm^3)^{1/2}$ for water; and 20.0 $(J/cm^3)^{1/2}$ for ethanol[8]. Although water and ethanol can both break the interpeptide H-bonds among dry collagen, water possibly does it more rapidly and completely than ethanol. Although water can break the H-bonds of collagen fibrils completely, the dentine matrix is very compliant. During water evaporation, the dentine matrix is too weak to resist evaporation stress, resulting in dramatic shrinkage and a loss of resin uptake. Compared with water, ethanol breaks interpeptide H-bonds only partially. During ethanol evaporation, the remaining interpeptide H-bonds maintain the interspace conformation of collagen fibrils. Consequently, there is less shrinkage of the dentine matrix, allowing more resin monomer to infiltrate. The increased infiltration of resin monomer is crucial for optimal hybrid layer formation as the collagen fibres are enveloped with a protective layer of resin[24]. An optimal hybrid layer is thought to produce a high bond strength[15]. This may be responsible for the higher initial SBS of adhesion to root dentine achieved with ethanol-wet bonding than with water-wet bonding.

In the present experiment, two-way ANOVA revealed that the SBSs of the experimental hydrophobic adhesives with ethanol-wet bonding (groups 4 and 5) were significantly higher than those achieved with water-wet bonding ($P = 0.000$). SEM results showed that almost all open dentine tubules were infiltrated with resin tags in the ethanol-wet bonding groups. In addition, the resin tags were much longer and more regular compared with those in the water-wet bonding groups. Ethanol-wet bonding may also improve the infiltration of adhesive into dentine tubules and achieve better interface quality compared with water-wet bonding[25,26]. The greater infiltration of the hydrophobic bonding agent and better collagen encapsulation may lead to more durable resin–dentine bonds because of the improved resistance to hydrolytic attack[20,26].

When the dentine surfaces were wetted with water or ethanol, the contact angles were similar; no significant differences were found between them ($P > 0.05$). However, after removing water or ethanol, the contact angle of the water-saturated dentine was significantly higher than that of the ethanol-saturated dentine ($P < 0.05$). It is known that optimal wettability of solid surfaces is the primary requirement in adhesion[15]. To achieve optimal wettability, surface free energy must be maximised; that is, the adhesive must exhibit a low contact angle with the dentine[27]. The wettability of deionised water spreading on the water-saturated dentine surface was weaker than that on the ethanol-saturated dentine, especially after the dentine was dried. Spontaneous spreading of a liquid on a solid surface expresses the wettability of the surface by the liquid. High wettability involves intimate adhesive–dentine contact and, therefore, enhanced adhesion[15,28]. It is possible that the differences in shrinkage of the water-saturated and the ethanol-saturated dentine matrices after air drying are responsible for the variance. Thus, ethanol-saturated dentine may be beneficial for resin monomers, allowing rapid infiltration and so creating a high-quality hybrid layer. As a result, the initial SBSs of the ethanol-wet bonding groups were significantly higher than those of the water-wet bonding groups. These results may also indicate that the water-wet method is more susceptible to the effects of moisture on the root dentine surface than is the ethanol-wet method.

Among the groups that used water-wet bonding, group 3 had the highest SBS, which was significantly higher than in group 1 ($P < 0.05$). Hydrophilic resin monomers are used in dentine adhesives to enhance their wetting properties and to avoid phase separation[29]. It is possible that the resin–dentine bond of the experimental hydrophobic adhesives could be compromised by phase separation due to the adhesives meeting the residual water in the dentine matrix. This problem can be avoided by ethanol-wet bonding because hydrophobic resin monomers are compatible with ethanol. Actually, the initial SBS of hydrophobic adhesives on root dentine significantly increases with ethanol-wet bonding compared with water-wet bonding.

The rapid evaporation of ethanol might have an adverse effect on the dentine bond, especially under

root canal conditions. Ethanol-wet bonding involves more steps and is more time-consuming than conventional water-wet bonding. Therefore, more evidence is needed to evaluate this technique before its clinical application.

Conclusion

Within the limitations of this study, the initial bonding of hydrophobic adhesives to root dentine using ethanol-wet bonding may be superior to that with water-wet bonding. Further studies should be performed to confirm the durability of root dentine bonding with the ethanol-wet bonding technique.

Clinical relevance

The application of hydrophobic adhesives with ethanol-wet bonding may be of potential benefit to root dentine adhesion.

Acknowledgements

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References

1. Kanca J 3rd. Resin bonding to wet substrate. 1. Bonding to dentin. Quintessence Int 1992;23:39–41.
2. Becker TD, Agee KA, Joyce AP et al. Infiltration/evaporation-induced shrinkage of demineralized dentin by solvated model adhesives. J Biomed Mater Res B Appl Biomater 2007;80:156–165.
3. Nakajima M, Okuda M, Pereira PN, Tagami J, Pashley DH. Dimensional changes and ultimate tensile strengths of wet decalcified dentin applied with one-bottle adhesives. Dent Mater 2002;18:603–608.
4. Wang Y, Spencer P. Hybridization efficiency of the adhesive/dentin interface with wet bonding. J Dent Res 2003;82:141–145.
5. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic? J Can Dent Assoc 2003;69:726–731.
6. Yiu CK, King NM, Pashley DH et al. Effect of resin hydrophilicity and water storage on resin strength. Biomaterials 2004;25: 5789–5796.
7. Ito S, Hashimoto M, Wadgaonkar B et al. Effects of resin hydrophilicity on water sorption and changes in modulus of elasticity. Biomaterials 2005;26:6449–6459.
8. Pashley DH, Tay FR, Carvalho RM et al. From dry bonding to water-wet bonding to ethanol-wet bonding. A review of the interactions between dentin matrix and solvate resins using a macromodel of the hybrid layer. Am J Dent 2007;20:7–20.
9. Sadek FT, Pashley DH, Nishitani Y et al. Application of hydrophobic resin adhesives to acid-etched dentin with an alternative wet bonding technique. J Biomed Mater Res A 2008;84:19–29.
10. Tay FR, Pashley DH, Kapur RR et al. Bonding BisGMA to dentin – A proof of concept for hydrophobic dentin bonding. J Dent Res 2007;86:1034–1039.
11. Ferrari M, Vichi A, Grandini S. Efficacy of different adhesive techniques on bonding to root canal walls: an SEM investigation. Dent Mater 2001;17:422–429.
12. Vichi A, Grandini S, Davidson CL, Ferrari M. An SEM evaluation of several adhesive systems used for bonding fiber posts under clinical conditions. Dent Mater 2002;18:495–502.
13. Sadek FT, Pashley DH, Ferrari M, Tay FR. Tubular occlusion optimizes bonding of hydrophobic resins to dentin. J Dent Res 2007;86: 524–528.
14. Sadek FT, Castellan CS, Braga RR et al. One-year stability of resin-dentin bonds created with a hydrophobic ethanol-wet bonding technique. Dent Mater 2010;26:380–386.
15. Marshall SJ, Bayne SC, Baier R, Tomsia AP, Marshall GW. A review of adhesion science. Dent Mater 2010;26:e11–e16.
16. 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;79:1385–1391.
17. 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;24:3795–3803.
18. Bradna P, Vrbova R, Dudek M, Roubickova A, Housova D. Comparison of bonding performance of self-etching and etch-and-rinse adhesives on human dentin using reliability analysis. J Adhes Dent 2008;10:423–429.
19. Nishitani Y, Yoshiyama M, Donnelly AM et al. Effects of resin hydrophilicity on dentin bond strength. J Dent Res 2006;85: 1016–1021.
20. Hosaka K, Nishitani Y, Tagami J et al. Durability of resin-dentin bonds to water- vs. ethanol-saturated dentin. J Dent Res 2009;88: 146–151.
21. Asmussen E, Uno S. Solubility parameters, fractional polarities, and bond strengths of some intermediary resins used in dentin bonding. J Dent Res 1993;72:558–565.
22. Miller RG, Bowles CQ, Chappelow CC, Eick JD. Application of solubility parameter theory to dentin- bonding systems and adhesive strength correlations. J Biomed Mater Res 1998;41:237–243.
23. Nishitani Y, Yoshiyama M, Hosaka K et al. Use of Hoy' s solubility parameters to predict water sorption/solubility of experimental primers and adhesives. Eur J Oral Sci 2007;115:81–86.
24. Pashley DH, Carvalho RM. Dentine permeability and dentine adhesion. J Dent 1997;25:355–372.
25. Sauro S, Toledano M, Aguilera FS et al. Resin-dentin bonds to EDTA-treated vs. acid-etched dentin using ethanol wet-bonding. Dent Mater 2010;26:368–379.
26. Shin TP, Yao X, Huenergardt R, Walker MP, Wang Y. Morphological and chemical characterization of bonding hydrophobic adhesives to dentin using ethanol wet bonding technique. Dent Mater 2009;25:1050–1057.
27. Adamson AW, Gast AP. Physical Chemistry of Surfaces, ed 6. New York: Wiley, 1997.
28. Rosales-Leal JI, Osorio R, Holgado-Terriza JA, Cabrerizo-Vílchez MA, Toledano M. Dentin wetting by four adhesive systems. Dent Mater 2001;17:526–532.
29. Spencer P, Wang Y. Adhesive phase separation at the dentin interface under wet bonding conditions. J Biomed Mater Res 2002;62: 447–456.