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Review Article

A review of ethanol wet-bonding: Principles and techniques

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ABSTRACT

Conventional water wet-bonding technique has been advocated by many scientists, but the excess water will induce suboptimal polymerization of dental adhesives, phase separation, and nanoleakage, which will influence the longevity of resin-dentin interfaces. Recent studies have put forward a new concept, ethanol wet-bonding. This technique can increase in dentin bond durability. This review focuses on the principles of ethanol wet-bonding, its surface treatment methods.

Key words: Bond strength, dentin, durability, ethanol wet-bonding, wet-bonding

INTRODUCTION

Despite significant improvements of adhesive systems, resin-tooth interface remains the weakest area of composite resin restorations. Durable and reliable dentin bonding has not been achieved yet.[1] Traditional water-wet-bonding technique has been advanced to improve initial bond strength of etch-and-rinse adhesives,[2] as water is an excellent solvent to re-expand collapsed demineralized dentin matrices prior to resin infiltration.[3] However, excess water often causes suboptimal polymerization of infiltrated resin monomers. In addition, water is not a proper solvent for resin monomers, as their miscibility is limited in the water, resulting in phase separations of hydrophobic resins.[4] Hereafter, poor quality hybrid layers made with a conventional water wet-bonding technique are quite susceptible to biodegradation over time in a harsh oral environment.[1] A hydrophilic tissue also results in poor dentin bond durability as hydrophilic adhesives absorb more water and are less durable than more hydrophobic adhesives over time.[5]

The importance of interactions of solvents, solvated resins with demineralized dentin matrices, should be emphasized to address and solve an issue of dentin bond durability.[3] In this context, ethanol wet-bonding was introduced as a proof of concept by Tay *et al.* in order to address a sound solution for improving resin-dentin bond durability in 2007.[6] This innovative research reported that bonding to hydrophobic resin monomer blends to dentin, which is a hydrophilic tissue, and might become reality when ethanol wet-bonding is utilized. Later research also confirmed that ethanol wet-bonding enhances resin infiltration-promoting higher quality hybrid layers in comparison with conventional water wet-bonding.[7] The crucial aim of ethanol wet-bonding is to infiltrate the interfibrillar spaces and dentinal tubules with hydrophobic dimethacrylate resins, which mimic the filling of tissue spaces with hydrophobic epoxy resins in tissue embedding.[3] In this study, the principle of ethanol wet-bonding techniques, application protocols, and research are reviewed.

PRINCIPLES OF ETHANOL WET-BONDING

The ethanol wet-bonding concept is derived from the tissue embedding techniques in which hydrated organic tissues are chemically dehydrated with ethanol

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for a few hours and then are embedded into epoxy resin.[3,8] Similarly, in the dentin bonding process, water within the demineralized dentin matrices can be replaced gradually with resin monomers with the use of ethanol promoting hydrophobic resin infiltration into a resultant dentinal hybrid layer.[3,6]

It is accepted that the collapse of demineralized dentin matrices is an active process, involving the rapid and spontaneous development of new hydrogen bonds between adjacent collagen peptides.[3] As solubility parameters are able to rank the ability of chemicals to perform a hydrogen bond, principles of ethanol-wet-bonding are explained by using Hoy's triple solubility parameter theory in literature.[3,8] Hoy's triple solubility parameters consist of dispersive forces (δd), polar forces (δp), hydrogen bonding forces (δh), and total cohesive forces (δt).[3]

Solubility parameters for hydrogen bonding forces (δh) are used to predict how any solvent or adhesive resin can re-expand a collapsed dried, acid-etched dentin. When demineralized dentin matrices collapsed (dried dentin), to re-expand matrices again, a solvent or resin monomer blends with a higher hydrogen bonding force than 14.8 $(Jcm^3)^{1/2}$ is needed.[3] Ethanol (δh = 20.0 $[Jcm^3]^{1/2}$) and water (δh = 40.4 $[Jcm^3]^{1/2}$) are successful in breaking interpeptide hydrogen bonds allowing the matrix to soften to the point that it can expand.[3] Most monomers used in adhesive dentistry have δh values below those of dried dentin. Thus, in their neat form, such resins cannot expand dried, acid-etched dentin. Water-wet-bonding expands the dried dentin maximally because water has a very high δh value of 40 $(Jcm^3)^{1/2}$. Unfortunately, not all adhesive monomers are soluble in the water.[3] Dimethacrylates, such as bisphenol A-glycidyl methacrylate, are not water-soluble and can undergo phase separations in the water-wet dentin. Therefore, bonding to water-wet dentin using more hydrophobic resin monomer is the out of reach goal to obtain more durable dentin bonding.

In water wet-bonding, demineralized dentin matrices are expanded with water with considerably higher δh value than 14.8 $(Jcm^3)^{1/2}$; resin filtration and evaporation of solvent processes are performed. Ethanol with lower δh value than those of water it is not able to re-expand collapsed matrices, to a point, which water can do.[3] The problems with water wet-bonding are water is not a proper solvent for most resin monomers used in the adhesive systems along with the collapse of matrices, due to softening matrices following evaporation of water, resulting in poor resin infiltration.[3] On the other hand, it was observed that if ethanol was used to replace rinse water from acid-etched matrices, the resulting collapse of matrices was very limited and resin infiltration into the hybrid layer appeared very high.[3] This indicates that ethanol-saturation of the water-saturated matrix does not soften the matrix so much that it cannot shrink when ethanol is evaporated, and ethanol is a proper solvent for resin monomers.

Solubility parameter theory has also been used to predict the miscibility of two different solutions by comparing their total cohesive forces (δt). It predicts that if there is <5 $(Jcm^3)^{1/2}$ between the solubility parameter for the total cohesive energy (δt) of a solution and a second solution or a substrate that the solution will wet the substrate and cause it to swell enough to permit entry of the solution. It was shown that discrepancies in δt values of hydrophobic resin monomers with water-wet matrices are >5 $(Jcm^3)^{1/2}$ while solubility parameters of hydrophobic resin match better with those of ethanol-saturated dentin matrices.[3]

ETHANOL WET-BONDING TECHNIQUES

Convention of water-saturated demineralized dentin matrices to ethanol-saturated demineralized dentin matrices can be achieved by treating acid-etched dentin surfaces with a series of increasing ethanol concentrations (50, 70, 80, 95, and 100% 3 times each, for 30 s), totaling 3–4 min.[8,9] This ethanol dehydration process is called "full chemical dehydration protocol" by Sadek *et al*.[8] and consumes more time and is too complex to perform properly in a clinic routine. It may be considered contrary to a tendency to simplify bonding procedures that currently exist.[10] Therefore, it may be necessary to seek more user-friendly ethanol wet-bonding methods.

Because dentin matrix is a highly cross-linked network structure, some researchers believe that using only 100% ethanol is enough to achieve the same effect.[11] Currently, different studies using different ethanol dehydration protocols, including using 100% ethanol 2 times, each for 10 s,[7] using 100% ethanol for 20 s,[6,11] 5 min[12] or 1 min,[13,14] exist in the literature. Sadek *et al*. compared five different ethanol dehydration protocols in terms of dentin bond durability after 6-month of water storage and does not suggest using 100% ethanol for 1 or 3 times

for 30 s, because of increased nanoleakage and the reduction of bond durability.[15] However, using a series of increasing ethanol concentrations for 15 s or 30 s provided a similar effect.[15]

The simulation of pulpal pressure is an important parameter when considering efficiency of ethanol dehydration protocols *in vitro*. The studies using user-friendly ethanol dehydration protocols, that is, 100% ethanol for 60 s, or 20 s generally ignore the presence of pulpal pressure or use tubular occlusion agents to prevent water contamination from pulpal pressure.[16] However, it was shown that the absence of pulpal pressure, both with simplified or full-dehydration protocols of ethanol wet-bonding, provided similar dentin bond strength.[17] Furthermore, Sauro *et al*. claimed that 100% ethanol for 1- or 5-min applications will achieve a similar effect. About 100% ethanol for 1 min still can replace water while maintaining that the collagen fibrils do not collapse and promote infiltration of resin monomers.[14]

The selection of proper ethanol dehydration protocol for ethanol wet-bonding in terms of dentin bond durability is currently unclear. Kuhn *et al*. reported that reduced nanoleakage within *in vivo* hybrid layers made with ethanol wet-bonding using full-dehydration protocol when compared to conventional water wet-bonding.[18] Thus, evidence can suggest that a longer ethanol dehydration time is better. Further studies are needed to assess efficiency of shorter ethanol dehydration protocols under *in vivo* conditions.

STUDIES ON ETHANOL WET-BONDING TECHNIQUES

Effects of ethanol on demineralized dentin matrices

As mentioned above, when water rinsed acid-etched dentin surfaces were treated with 100% ethanol, it was observed that matrices, just slightly, collapsed. In addition, ethanol dehydrates, hence shrinks proteoglycans filling the interfibrillar spaces, resulting in significantly wide interfibrillar spaces that serve as infiltration highways for resin monomers when compared to water-wet-bonding.[3,13] Thus, in spite of the slight shrinkage that occurs during ethanol replacement, the ethanol-stiffened collagen matrix is prevented from collapsing while being suspended in the ethanol that is a less hydrophilic chemical dehydrant. This is a prerequisite for resin infiltration. Widening interfibrillar spaces were observed within conventional dentinal hybrid layers by Hosaka *et al*. using 100% ethanol application for 1 min without simulating pulpal pressure using transmission electron microcopy.[13] However, whether the same effect could be achieved with simplified ethanol application protocols under pulpal pressure simulation, is currently unknown.

Effects of ethanol wet-bonding on dentin bond strength

The *in vitro* studies have shown that the application of ethanol wet-bonding can improve initial bond strengths of both of hydrophilic and hydrophobic resin monomer blends or adhesive systems[6,8,9,11,14] or did not affect current status.[19,20] However, a current *in vivo* study on ethanol-wet-bonding reported that the immediate benefits of the ethanol-bonding, observed in the laboratory setting, was not confirmed when the same protocol was performed *in vivo*. Despite that, they also reported that reduced nanoleakage was seen in adhesive interfaces produced with the ethanol-wet-bonding technique.[18] It was claimed that the observed benefits of ethanol wet-bonding on initial bonding effectiveness of resin adhesives may be contributed to relatively increased interfibrillar spaces achieved by ethanol wet-bonding.[3,21]

Effects of ethanol wet-bonding on dentin bond durability

The mechanisms for poor durability of dentin bonds made with conventional water-wet-bonding are: (1) Hydrophilic nature of adhesive resin which absorbs more water,[22] leading to the plasticization effect, thereby reducing the mechanical properties of the resin component of an adhesive joint[5] and (2) activation of an endogenous dentin matrix, metalloproteinase, following acid-etching, or even application of adhesive resin monomers. After resin infiltration, a complete resin encapsulation of collagen fibrils is not achieved, resulting in exposed collagen fibrils, which are vulnerable to matrix metalloproteinase proteolytic enzyme activity.[23]

Therefore, to improve resin-dentin durability, hydrophilicity of the adhesive used should be reduced.[5] Because hydrophobic resins have higher stability in the aqueous environment as compared to the hydrophilic resins, they can improve durability of bonding interfaces.[24] To achieve this, dentin that is hydrated tissue should be conditioned as a compatible substrate with more hydrophobic resin monomers during the first step. Ethanol can replace rinse water after acid-etching, thus lowering hydrophilicity of matrices, at the same time, stabilizing matrices and

promoting resin infiltration. Further, since most of hydrophobic monomers are mixable in ethanol and not in water, bonding to dentin with hydrophobic adhesives with reduced water adsorption and increased durability could be achieved.[7]

Hosaka *et al.* evaluated the effects of ethanol wet-bonding (100% ethanol for 60 s) on dentin bond durability of five increasingly hydrophilic experimental resin blends in comparison with water wet-bonding after a 12-month water storage. They stated that increases in bond strength and durability in ethanol wet-bonding might be due to a higher resin uptake and better resin sealing of the collagen matrix, thereby minimizing endogenous collagenolytic activities.[13] In another study, Sadek *et al.* assessed the effect of a 12-month water storage on the dentin bond durability of an experimental hydrophobic adhesive in comparison with a commercial hydrophilic adhesive with water wet-bonding. They reported that coaxing hydrophobic resins into acid-etched dentin using ethanol-wet-bonding preserves resin-dentin bond integrity.[24]

On the other hand, ethanol can increase the penetration of resin and provide better encapsulation of collagen fibrils with resin to avoid matrix metalloproteinases.[13] Antimatrix metalloproteinase benefits of ethanol-wet-bonding were assessed and confirmed by Sadek *et al.* in comparison with the use of chlorhexidine that is a nonspecific antimatrix metalloproteinase agent.[25] It was reported that bonds made to ethanol-saturated dentin did not change after an 18-month water storage with preservation of hybrid layer integrity, whereas bonds made to chlorhexidine diacetate pretreated acid-etched dentin with commercial adhesives with water wet-bonding were not preserved after 18 months, with severe hybrid layer degradation.

Previous studies on ethanol-wet-bonding generally used an experimental resin monomer blends where exact chemical compositions are known, thus solubility parameters can be calculated. However, bond durability of commercial resin adhesive systems to dentin with ethanol wet-bonding were researched by several studies. Yesilyurt *et al.* found that simplified ethanol wet-bonding improved aged dentin bond strength of two simplified etch-and-rinse adhesive, but not significantly, in comparison with water-wet-bonding after a 12-month water storage.[26] Similarly, Li *et al.* found that simplified ethanol wet-bonding improves resin-dentin durability of a commercial adhesive.[27] It should be mentioned that both of these studies used 100% ethanol for 60 s to replace rinse water from acid-etched dentin, but the pulpal pressure was not simulated in both studies.

CONCLUSION

Application of 100% ethanol onto deep dentin of vital pulps may arise some concerns among clinicians regarding to vitality of dental pulp.[3] It was shown that application of 100% ethanol for 60 s on directly exposed human pulp did not increase pulpal damage compared to a water-wet-bonding technique and produced only mild pulp injury that was similar to the pulpal damage produced by a contemporary water-wet-bonding technique.[28] Ethanol wet-bonding can promote an infiltration of hydrophobic dimethacrylate resins into the interfibrillar spaces and dentinal tubules to improve stability of resin-dentin interfaces *in vitro*. However, there is also some concerns regarding the effectiveness of ethanol-wet-bonding on actual performances of resin adhesive systems *in vivo* in terms of water contamination from dental pulp.[29] It was shown that prevention of ethanol-saturated matrices from pulpal water contamination with intrapulpal pressure is more difficult than was previously thought. However, it should be noted that caries-affected dentin have much lower permeabilities and administration of local anesthetics containing vasoconstrictors, decreased pulpal fluid pressure. Thus, during most dentin bonding procedures, pulpal pressure is closer to zero. The best answer to these concerns will come from further long-term *in vivo* dentin bond durability studies with different ethanol application protocols and different increasingly hydrophobic adhesives. Thus far, researchers and clinicians should consider ethanol-wet-bonding, especially a simplified technique, as an *in vitro* technique for better dentin bond durability than a clinical solution.

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Conflicts of interest
There are no conflicts of interest.

REFERENCES

1. Spencer P, Ye Q, Misra A, Goncalves SE, Laurence JS. Proteins, pathogens, and failure at the composite-tooth interface. J Dent Res 2014;93:1243-9.
2. Kanca J 3rd. Improving bond strength through acid etching of dentin and bonding to wet dentin surfaces. J Am Dent Assoc 1992;123:35-43.
3. Pashley DH, Tay FR, Carvalho RM, Rueggeberg FA, Agee KA, Carrilho M, *et al.* From dry bonding to water-wet bonding to

ethanol-wet bonding. A review of the interactions between dentin matrix and solvated resins using a macromodel of the hybrid layer. Am J Dent 2007;20:7-20.
4. Spencer P, Wang Y. Adhesive phase separation at the dentin interface under wet bonding conditions. J Biomed Mater Res 2002;62:447-56.
5. Tay FR, Pashley DH. Have dentin adhesives become too hydrophilic? J Can Dent Assoc 2003;69:726-31.
6. Tay FR, Pashley DH, Kapur RR, Carrilho MR, Hur YB, Garrett LV, *et al.* Bonding BisGMA to dentin – A proof of concept for hydrophobic dentin bonding. J Dent Res 2007;86:1034-9.
7. Shin TP, Yao X, Huenergardt R, Walker MP, Wang Y. Morphological and chemical characterization of bonding hydrophobic adhesive to dentin using ethanol wet bonding technique. Dent Mater 2009;25:1050-7.
8. Sadek FT, Pashley DH, Nishitani Y, Carrilho MR, Donnelly A, Ferrari M, *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.
9. Sadek FT, Pashley DH, Ferrari M, Tay FR. Tubular occlusion optimizes bonding of hydrophobic resins to dentin. J Dent Res 2007;86:524-8.
10. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, *et al.* Buonocore memorial lecture. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 2003;28:215-35.
11. Nishitani Y, Yoshiyama M, Donnelly AM, Agee KA, Sword J, Tay FR, *et al.* Effects of resin hydrophilicity on dentin bond strength. J Dent Res 2006;85:1016-21.
12. Sauro S, Watson TF, Mannocci F, Miyake K, Huffman BP, Tay FR, *et al.* Two-photon laser confocal microscopy of micropermeability of resin-dentin bonds made with water or ethanol wet bonding. J Biomed Mater Res B Appl Biomater 2009;90:327-37.
13. Hosaka K, Nishitani Y, Tagami J, Yoshiyama M, Brackett WW, Agee KA, *et al.* Durability of resin-dentin bonds to water- vs. ethanol-saturated dentin. J Dent Res 2009;88:146-51.
14. Sauro S, Toledano M, Aguilera FS, Mannocci F, Pashley DH, Tay FR, *et al.* Resin-dentin bonds to EDTA-treated vs. acid-etched dentin using ethanol wet-bonding. Dent Mater 2010;26:368-79.
15. Sadek FT, Mazzoni A, Breschi L, Tay FR, Braga RR. Six-month evaluation of adhesives interface created by a hydrophobic adhesive to acid-etched ethanol-wet bonded dentine with simplified dehydration protocols. J Dent 2010;38:276-83.
16. Ayar MK. Letter to the Editor regarding "Depletion of water molecules during ethanol wet-bonding with etch and rinse dental adhesives". Mater Sci Eng C Mater Biol Appl 2015;50:141-2.
17. Ayar M. Ethanol application protocols and microtensile dentin bond strength of hydrophobic adhesive. Tanta Dent J 2014;11:206-12.
18. Kuhn E, Farhat P, Teitelbaum AP, Mena-Serrano A, Loguercio AD, Reis A, *et al.* Ethanol-wet bonding technique: Clinical versus laboratory findings. Dent Mater 2015;31:1030-7.
19. Ayar MK. Effect of simplified ethanol-wet bonding on microtensile bond strengths of dentin adhesive agents with different solvents. J Dent Sci 2014;10:270-4.
20. Guimarães LA, Almeida JC, Wang L, D'Alpino PH, Garcia FC. Effectiveness of immediate bonding of etch-and-rinse adhesives to simplified ethanol-saturated dentin. Braz Oral Res 2012;26:177-82.
21. Carvalho RM, Mendonça JS, Santiago SL, Silveira RR, Garcia FC, Tay FR, *et al.* Effects of HEMA/solvent combinations on bond strength to dentin. J Dent Res 2003;82:597-601.
22. Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, *et al.* Water sorption/solubility of dental adhesive resins. Dent Mater 2006;22:973-80.
23. Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM, *et al.* Collagen degradation by host-derived enzymes during aging. J Dent Res 2004;83:216-21.
24. Sadek FT, Castellan CS, Braga RR, Mai S, Tjäderhane L, Pashley DH, *et al.* One-year stability of resin-dentin bonds created with a hydrophobic ethanol-wet bonding technique. Dent Mater 2010;26:380-6.
25. Sadek FT, Braga RR, Muench A, Liu Y, Pashley DH, Tay FR. Ethanol wet-bonding challenges current anti-degradation strategy. J Dent Res 2010;89:1499-504.
26. Yesilyurt C, Ayar MK, Yildirim T, Akdag MS. Effect of simplified ethanol-wet bonding on dentin bonding durability of etchand-rinse adhesives. Dent Mater J 2015;34:441-8.
27. Li F, Liu XY, Zhang L, Kang JJ, Chen JH. Ethanol-wet bonding technique may enhance the bonding performance of contemporary etch-and-rinse dental adhesives. J Adhes Dent 2012;14:113-20.
28. Scheffel DL, Sacono NT, Ribeiro AP, Soares DG, Basso FG, Pashley D, *et al.* Immediate human pulp response to ethanol-wet bonding technique. J Dent 2015;43:537-45.
29. Sauro S, Watson TF, Mannocci F, Tay FR, Pashley DH. Prevention of water contamination of ethanol-saturated dentin and hydrophobic hybrid layers. J Adhes Dent 2009;11:271-8.

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