The Comparison of Ethyl-Alcohol-Wet Bonding Techniques on the Shear Bond Strength of Resin Cements; An *in vitro* Study

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Abstract

Aims: The aim of this *in vitro* study was to assess the shear bond strengths of hydrophobic and hydrophilic resin cements to dentin, after application of ethyl-alcohol-wet and water-wet bonding techniques. Materials and Methods: Seventy flat dentin surfaces were etched, rinsed, blot-dried, and kept moist before applying the water-wet bonding technique. In addition to these procedures, the surfaces were re-wetted with 100% ethanol solution for 30 seconds for the application of the ethanol-wet bonding technique. They were then bonded with adhesives. After construction of resin composite build-ups with resin cements, the specimens were held in water for 1 day. An Instron device was used to measure bond strength and environmental scanning electron microscopy (ESEM) was used to evaluate dentin surfaces treated with adhesive. Analysis of variance (ANOVA) and the three factor with interaction model were used for statistical analysis. Results: The mean bonding values of the experimental groups generally were statistically insignificant (P>.05). However, resin cement–ethanol interaction was significant (P= .027). According to ESEM images, it was clear that the groups with high bond strength values had more resin in the dentinal tubules. Conclusion: Higher bond strengths were achieved when dentin was bonded with ethanol with hydrophobic resins, or when dentin was bonded moist with water with hydrophilic resins.

Key Words: Ethyl alcohol, Bonding, Dentin, Resins

Introduction

Today's dentistry widely employs dental adhesives to ensure the retention of composite restoration and to decrease associated marginal microleakage [1,2]. Bonding systems and cementing agents have been developed to simplify the application procedure and to decrease sensitivity to operator technique. Adhesion makes possible the close joining of two materials, and contiguity is likely to be physical or chemical. Etching, priming, and bonding resin application procedures provide successful resin–dentin adhesion [3]. However, it has not been verified that the effectiveness of the bond to human dentin is influenced by the simplification of application procedures [4]. Melo et al. [5] demonstrated that a self-etching adhesive system led to lower bonding to dentin than the "total etched" adhesive system, which was once used along with two dual-polymerized cements. Carvalho et al. [6] demonstrated that resin/dentin bond strengths were considerably influenced by the type of solvent in the primer solutions and the formerly dried demineralized matrix.

It was reported that dentin–resin bonding that employsethyl-alcohol-wet bonding is less vulnerable to deterioration [7]. However, it has never been validated that this would generate higher bond strengths when comparing the water-wet technique (WT) with the ethyl-alcohol-wet bonding technique (ET).

Kanca [8] advanced the "wet-bonding" technique with the discovery of water or water-HEMA (2-Hydroxyethyl methacrylate) primers. Another recently developed technique, "ethanol-wet bonding," employs ethanol rather than water to reinforce the demineralized dentin collagen matrix [9,10], whereby the ethanol and water blend and thus, in principle, water is removed. Resin–dentin bond strength can theoretically be improved by ethanol-wet bonding (ET), reducing water sorption via a polymerized hydrophobic adhesive [9], Acid-etched dentin becomes less hydrophilic with ethanol dehydration, which makes it possible to use comparatively hydrophobic monomers to infiltrate a contracted but non-collapsed demineralized collagen network suspended in ethyl alcohol. This is the logical basis underlying the technique. Until now, ET's contribution to the durability of hydrophobic or hydrophilic dentin–resin bonds has not been evaluated.

The first adhesive was made from bisphenol glycidyl methacrylate (bis-GMA); this hydrophobic bonding system does not achieve bonding with the wet dentin. Later studies have led to the development of hydrophilic adhesives such as HEMA. It is very important that the dental adhesives can tolerate water especially after the development of the acid-etching method, which released water onto the cavity surface [11].

The purpose of this study was to compare resin–dentin bond strengths of hydrophobic vs hydrophilic resins bonded to acid-etched dentin saturated with water or ethanol. The hypothesis of this study was that bonding strength increased with the addition of ethanol.

Materials and methods

In this study, 70 maxillary central incisors, which had no caries or restorations, were immersed in 0.9% NaCl immediately after extraction.The water was replaced weekly to avoid microbial growth. The root and crown surface was rinsed clean of debris and other material. Using a standard high-speed dental handpiece (W&H DENTALWERK, A-5111 Bürmoos), each root was sectioned 2-3 mm below the cemento-enamel joint. In preparation for bond strength testing, the crowns of the teeth were placed in self-cure acrylic resin (Imicryl, Konya, Turkey) with the labial surfaces facing upward (*Figure 1*). Flat surfaces were prepared on the mounted teeth at the mid-coronal level under water-cooling with the same high-speed dental handpiece. Exposed dentin

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surfaces were ground with 600-grit sandpaper under water, to form a smear layer.Then, 35% phosphoric acid was applied to all prepared dentin surfaces for 15s. After the acid etching, the surfaces were rinsed with distilled water for 10s. Redundant water was blotted from the surfaces with absorbent paper (Kimwipes, Kimberly); however, humidity was still observed on the dentin. The 70 specimens were then allocated to groups: 30 specimens for ET; 30 specimens for WT; 10 specimens as control. The control specimens were also demineralized with acid-etching, and humidity was still observed on the dentin, but ethanol and bonding agents were not used in the control group.

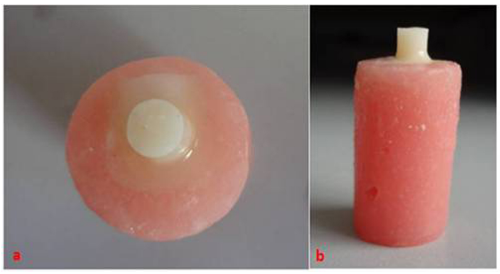

Figure 1. *One of the samples.*

100% ethanol solvent was used for the ET technique. Following a 15s waiting period, two drops of 100% ethanol were applied to the surfaces, following acid etching and rinsing of the dentin surface. The surface was left to air-dry for 5s before two more drops of ethyl alcohol were applied; a further wait of 15s was taken in order to keep the dentin humid. The redundant ethanol was then cleaned from the surface with absorbent paper. Two layers of adhesive from each adhesive system (Tetric N Bond, Adper Single Bond 2, Heliobond) were applied with mild air drying for 10s following each layer of application."Adper Single Bond 2 adhesive" can be applied in direct and indirect restorations [12]. A two-step "total etched" single bond adhesive, which is classified as a water-based adhesive [13], was used in the current study (Adper Single Bond 2, 3M ESPE). *Table 1* describes the adhesives and their compositions. Thus, the study employed two separate bonding techniques, ET and WT, with three adhesives and two resins. The groups in this study are shown in *Table 2*.

Table 1. *Materials used in this study.*

	Name	Content	Manufacturer	Lot number	Code
Adhesive System					
	Clearfil	Paste A: Bis-GMA, TEGDMA, MDP, hydrophobic aromatic di methacrylate monomers, silanated barium glass filler, others	Kuraray	0020AA	C
		Paste B: Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, silanated barium glass filler, silanated colloidal silica, others	Tokyo, Japan		
	Panavia F	Paste A: MDP, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica filler, photo initiator, chemical initiator, HEMA, MDP	Kuraray Tokyo, Japan	Paste A: 00410A	P
		Paste B: methacrylate monomer, photo initiator, chemical initiator		Paste B: 00210B	
Conditioner					
	Scotchbond Etchant	35% phosphoric acid	3M ESPE,USA		
Bonding Agents					
	Heliobond	Bis-GMA, TEGDMA, catalysts, stabilizers	Ivoclar vivadent	M35797	H
	Adper single	Bis-GMA, HEMA, dimethacrylates, polyalquenoic acid copolymer, initiators, water and ethanol	3M ESPE, USA	9XP	A
	Tetric N	Bis-GMA, urethane dimethacrylate, dimethacrylate, hydroxyethyl methacrylate, phosphonic acid acrylate, Nano-fillers (SiO2), Ethanol	Ivoclar vivadent	P11348	T
bisGMA = bisphenol-A-glycidyl dimethacrylate; TEGDMA = triethylene glycol dimethacrylate; HEMA= 2-hydroxyethyl methacrylate; MDP= 10-Methacryloyloxydecyl dihydrogen phosphate					

Light polymerization then followed for 20s (Henry Shein Inc., Melville, NY 11747). Then, 70 prepared composite cylindrical blocks (Ivoclar Vivadent), 4mm in height and 4 mm in diameter, were cemented using Panavia F or Clearfil and kept below 10 N for 10s. After 2s of light polymerization, redundant cement was removed from the margins and 20s of light polymerization was applied using hand instruments. Samples were immersed in distilled water at 37°C for a day prior to testing.

Table 2. Groups used in this study.

	Adhesive	Bonding technique	Resin cement
G1	Tetric N bond	ET	Panavia
G2	Adper single bond	ET	Panavia
G3	Heliobond	ET	Panavia
G4	Tetric N bond	WT	Panavia
G5	Adper single bond	WT	Panavia
G6	Heliobond	WT	Panavia
G7	Tetric N bond	ET	Clearfil
G8	Adper single bond	ET	Clearfil
G9	Heliobond	ET	Clearfil
G10	Tetric N bond	WT	Clearfil
G11	Adper single bond	WT	Clearfil
G12	Heliobond	WT	Clearfil
G13	-	WT	Panavia (control group)
G14	-	WT	Clearfil (control group)

The bonded specimens were placed in an apparatus and attached to a universal testing machine. A shear test was carried out at a crosshead speed of 1mm/min until bonding failure. Bond-breaking force was recorded in N, and bond strengths were calculated in MPa.After the shear test, a perpendicular section was taken from some specimens for ESEM analysis.

ANOVA and the three factor with interaction model were evaluated. An enviromental scanning electron microscope (ESEM, FEI, Quanta FEG-250) was used to produce a high resolution image.

Results

Shear bond strength values are demonstrated in *Tables 3 and 4*. The mean bonding values of the experimental groups generally were statistically insignificant (P > .05). Resin, alcohol, and adhesive types were not significant. Bond strength generally did not show a change using the bonding techniques, cements, and adhesives type (P >.05). However, resin cement–ethanol interaction was significant (P= .027). When using Panavia resin cement, the bonding values were higher with the application of WT than with the application of ET. Conversley, when using Clearfil resin cement, the bonding values were higher with the application of ET than with the application of WT.

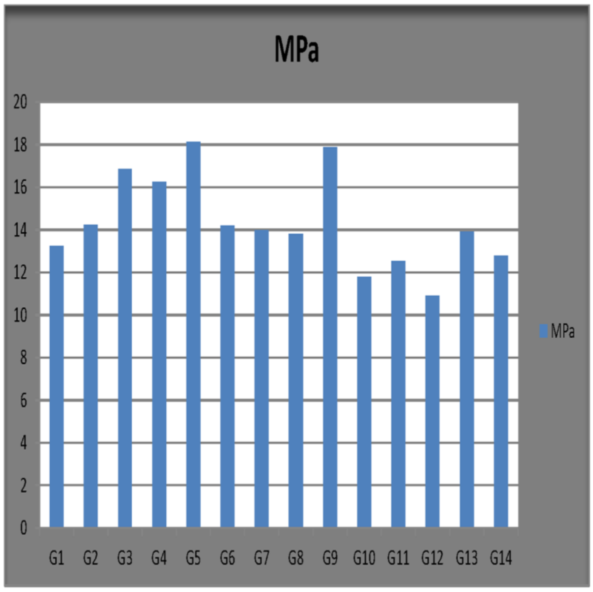

Figure 2. Mean values of bonding.

After the application of WT, higher bonding strength was observed with Panavia resin cement. After the application of ET, higher bonding values were observed for Clearfil resin cement. *Figure 2* shows mean shear bond strength values by group (n = 5).

Table 3. The minimum, maximum, mean and standard deviation values.

Resin	Bond	Alcohol	Mean	S.D.
P	T	+ (ET)	13.23	3.66
		- (WT)	16.26	4.58
	A	+(ET)	14.22	4.38
		-(WT)	18.12	5.01
	H	+(ET)	16.85	4.42
		-(WT)	14.2	3.67
	Control	-(WT)	13.9	3.65
C	T	+(ET)	13.98	4.43
		-(WT)	11.78	2.67
	A	+(ET)	13.82	5.09
		-(WT)	12.53	3.86
	H	+(ET)	17.9	6.25
		-(WT)	10.91	2.27
	Control	-(WT)	12.79	3.04

Secondary electron and back-scattered electron images were evaluated in some groups (*Figures 3A–E*). In *Figures 3A, 3C and 3D*, it is clear that the groups with resin in the dentinal tubules have high bonding values.

However, it does not seem to be the same in Figures 3B and 3E because these groups have low bonding values. In the light of these results, it can be noted that ethanol increases bonding and the WT may also show the same success when a hydrophilic resin is used (*Figure 4*).

Table 4. *Bonding values.*

Source	Type III sum of squares	df	Mean square	F	Sig.
Corrected model	314.001a	13	24.154	1.374	0.201
Intercept	13808.63	1	13808.63	785.609	0
Resin	36.597	1	36.597	2.082	0.155
Alcohol	16.017	1	16.017	0.911	0.344
Bond	17.174	3	5.725	0.326	0.807
Resin * alcohol	90.774	1	90.774	5.164	0.027
Alcohol * bond	109.469	2	54.735	3.114	0.052
Resin * bond	29.806	3	9.935	0.565	0.64
Resin * alcohol * bond	0.627	2	0.314	0.018	0.982
Error	984.31	56	17.577		
Total	15652.106	70			
Corrected total	1298.311	69			
[a] R squared = .242 (adjusted R squared = .066)					

Discussion

Saturating the dentin matrix with ethanol creates favorable conditions for hydrophobic methacrylates (such as bis-GMA) to diffuse into interfibrillar spaces, forming a hybrid layer and producing a higher mechanical property [14]. Since all the results are similar statistically, the hypothesis of the current study was rejected. A reasonable explanation for the greater penetration of dentinal tubules with resin tags could be related to the presence of bis-GMA methacrylates in the composition of G9 formulations when using Clearfil resin cement, and could be related to the presence of HEMA in the composition of G5 formulations when using Panavia resin cement.

With adhesives improved by adding hydrophilic HEMA to the production process, dentin is wetted by the adhesives and more humidity is tolerated, so HEMA significantly improves bond strengths [15]. The current study supports this: wet bonding results in higher bonding values when hydrophilic resin cement Panavia was applied with the Adper Single Bond containing HEMA. Also, similar to the current study, Hosaka et al. [16] reported that in resins that are less hydrophilic, according to Hoy's solubility parameters, ethanol resulted in greater bonding strengths than water. In the current study, significantly higher bonding values resulted with the Panavia resin cement using WT, and with Clearfil SA cement using ET. Greater bonding was observed after the application of ET with Clearfil SA cement because Clearfil SA cement is more hydrophobic; therefore, lower bond strength on dentin resulted with the use of the "Heliobond" adhesive with Clearfil resin cement.

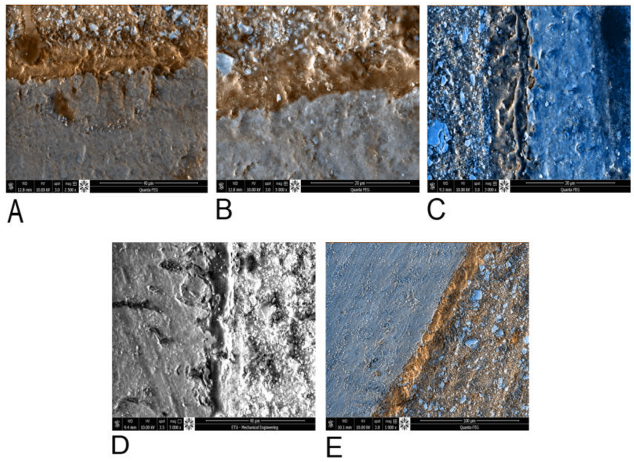

Figure 3. *A: ESEM micrograph of the dentin surface treated with ethanol, Heliobond, and Panavia resin cement, respectively, in Group 3. B: ESEM micrograph of a sample from control Group 14 , including only Clearfil resin cement, and is alcohol- or bonding-agent-free. C: ESEM micrograph of dentin surface treated with Adper Single Bond 2 and Panavia resin cement in Group 5. D: ESEM micrograph of dentinal tubules filled with resin tags in Group 9 treated with ethanol, Heliobond, and Clearfil resin cement. E: ESEM micrograph of resin cement–dentin interface luted using the water-wet technique with Heliobond adhesive and Clearfil SA cement and bonded on etched and rinsed dentin, where a thick adhesive layer is visibly observed (Group 12).*

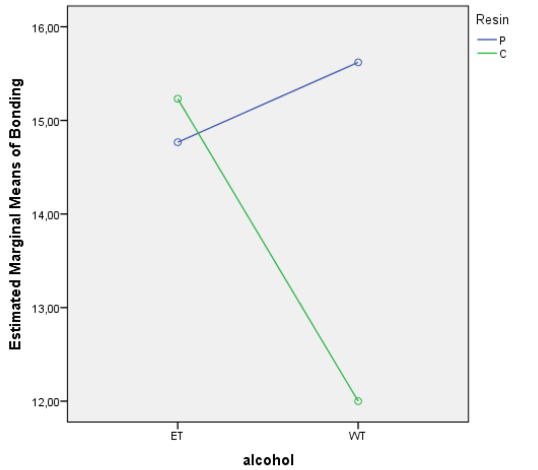

Figure 4. *Estimated Marginal Means of Bonding.*

Panavia as a "self-etching" adhesive cement facilitates the implementation of the system and simplifies the application procedure.Such adhesive systems include methacrylate, and the acidic material in the bonding surface are phosphoric acids. In the demineralized dentine surface formed by the lower pH, good bonding micromechanics are achieved [17].

Resin cement already contains phosphoric acid, and no significant difference between the control and other groups can be explained in this way.

Dentin sensitivity, microleakage, and low resin–dentin bond strengths are problems associated with "dry" bonding. Kanca [18] discovered that higher bond strengths were produced by wet bonding with water in comparison to dry bonding. Dentin remains completely hydrated because free space exists for resin infiltration in the non-collapsed dentin matrix [19]. Resin–dentin bond strength is increased by this technique. The dentin surface is wetted with a resin to create strong adhesion. The liquids can wet the surface. The substrate's surface energy must be higher than the adhesive's surface tension to achieve high wettability. Close contact and increased adhesion is achieved with high wettability [20]. The wet-bonding technique has been advised for over 10 years [18]. Orellana et al. [21] demonstrated that adhesive bonding is increased by a moist substrate. Unfortunately, the maintenance of moist demineralized dentin is difficult to achieve in daily clinical practice, since the ideal degree of moisture varies depending on the solvent present in each adhesive system. This can lead to bonding failures and may also turn the adhesive interface into a permeable membrane, which is enormously sensitive to the degrading impacts of water [22].

Several studies demonstrated that the endurance of resin–dentin bonds is enhanced by ethanol-wet-bonding [8,23]. Shin et al. [2] showed that in ET, adhesives penetrate deeper into dentin, are more resistant to acid-bleach treatment, and better encapsulate collagen fibrils in comparison to WT. According to Chen et al. [24], favorable bond strength is produced by dentin saturated with ethanol for more than 2 minutes before bonding a hydrophobic adhesive to dentin, thereby decreasing the micro permeability of bonding interfaces under simulated pulp pressure [24]. Sauro et al. [25] applied resin adhesives to root canal dentin, using WT (control) or ET. For all adhesives tested, the ET yielded higher bond strength values. Duan et al. [26] observed that ET might be more effective for adhesion to root dentin surfaces than WT. Additionally; WT might not provide practical clinical advantages over dry bonding *in vivo*. When 100% ethyl alcohol (1–5 minutes) was used for rinsing, a considerable reduction was observed in dentin roughness and fibril diameter. Absolute ethanol caused the collapse and contraction of collagen fibrils, but the matrix was not collapsed by increased ethanol concentrations, which contracted the fibrils less than absolute ethanol-rinses [27]. The influence of simplified ET on bonding to mid-coronal dentin was assessed by Guimarães et al. [7] who concluded that ET provides equivalent bonding to dentin compared with WT, regardless of the adhesive system tested. No difference was found in mean bond strengths when WT and ET were compared. Also, no variation was detected among different dimensional features in ET or a traditional three-step etch-and-rinse adhesive by Tay et al. [9].

ESEM that allows for the option of collectingof specimens that are "wet," uncoated, or both, by allowing for a gaseous environment in the specimen chamber.

Higher bonding values were observed with Clearfil SA cement, which has low water absorption when the tooth surface is wetted with ethanol. The bonding values of Panavia resin cement, which is more hydrophilic than Clearfil, are higher when the dentin surface is wetted with water.

In this study, a reasonable explanation for the equivalence of bond strength findings of dentin among the groups could be related to both HEMA and bis-GMA methacrylates, which are present in the composition of general formulations. However, the cementing agent and alcohol interaction was statistically significant and affected the strength of the bond to the dentin. The results show that ET and WT might offer practical clinical advantages *in vivo* according to type of adhesive and resin cement used. Certainly, more research is required to examine how ET affects the endurance of resin–dentin bonds.

Conclusion

Within the limitations of this *in vitro* study, regarding the difference between the two adhesive cementation treatments, one was not more effective than the other and there was no addition to today's clinical knowledge of the ideal bond strength. It brings into question the use of ET while employing adhesive cements for luting objects. In light of this study's results, many factors, such as cements and adhesives, might affect bonding in clinical situations and ethanol can increase the bonding if a hydrophobic adhesive and resin are used. Water-wet technique may also show the same success when a hydrophilic adhesive and resin are used. The present study needs to be repeated using more hydrophobic and hydrophilic resins. Future long-term experiments on nanoleakage and bond strength over time should test those hypotheses. Furthermore, these results need to be confirmed under*in vivo*conditions.

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