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Effect of Placement Agitation and Drying Time on Dentin Shear Bond Strength: An *In Vivo* Study

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Clinical Relevance

Primer agitation and primer drying time both affect dentin shear bond strength *in vivo*.

SUMMARY

The current study measured the bond strength of a self-etch system to dentin with and without agitation and with varying drying times of primer in vital dentin. The null hypotheses tested were that primer agitation and primer drying time did not affect the dentin shear bond strength. Sixty human maxillary and mandibular premolars scheduled to be extracted for orthodontic reasons were selected. The adhesive/resin combination used was Clearfil SE (Kuraray, Osaka, Japan)/TPH Spectrum (Dentsply DeTrey, Konstanz, Germany). The occlusal surfaces of the teeth were flattened using straight fissure diamond abrasive points ISO-012. Samples were divided into six groups of 10 teeth each. Primer was applied following the manufacturer's instructions with and without agitation followed by air-drying time of 0 (without air drying), five and 10 seconds. Clearfil SE Bond was applied and cured for 10 seconds. TPH Spectrum composite, shade A2 (Dentsply DeTrey), was placed over cured adhesive and was cured for 40 seconds. The teeth were restored to their original anatomy. The teeth were extracted after one week and the samples were kept in distilled water until testing at room temperature. The samples were tested in shear at a 1 mm/minute crosshead speed using an LR100 Universal Testing Machine. The data was analyzed using ANOVA and Tukey post-hoc tests (*p*<0.05). Varying the drying time of the primer from zero (0) to 10 seconds did not significantly affect the bond strength values *in vivo*. At five seconds drying time, agitation significantly improved the

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shear bond strength to dentin. Agitation showed no effect when primer was not dried or it was dried for 10 seconds.

INTRODUCTION

Although enamel adhesion is a predictable and established entity in contemporary restorative dentistry, an adequate bond to dentin is more difficult to achieve. This is partly due to the biologic characteristics of dentin, namely, its high organic content, its tubular structure with the presence of odontoblastic processes, the continuous moist condition due to the presence of dentinal fluid, intratubular pressure, permeability of the dentin and the presence of a smear layer formed immediately after cavity preparation.[1]

Based on the underlying adhesion strategy, three mechanisms of adhesion—total etch, self-etch and glass ionomer—are currently in use with modern adhesive systems.[2-3] The drawbacks of the total-etch system are the risk of over-etching the dentin, requiring a post-conditioning rinse phase, sensitivity to an over-dry or over-wet surface and the involvement of multiple steps.[3] Glass ionomer systems, though, are the only material that are self-adhesive to tooth tissue without any surface pretreatment; they have low bond strength due to the limited capacity of high molecular weight polycarboxyl base polymers infiltrating into dentin.[4] To reduce technique sensitivity and the materials-related factors that affect bond strength, a self-etch approach involving either the one-step or two-step application has been developed.[2,5] However, these bonding agents are often associated with incorporation of the remaining water and acidic solvents and the smear layer with bonding system. Such a solvent surplus may directly weaken bond integrity, provide channels for nanoleakage or it may affect the polymerization of infiltrated monomers. The resultant interfacial structure also becomes more hydrophilic and, thus, more prone to hydrolytic degeneration.[2]

Despite the adhesive approach per se, the result of the resin dentin interaction is often incomplete hybridization of the dentin surface, leaving the collagen fibrils unprotected and vulnerable to hydrolytic degradation that is also susceptible to other degradation promoting factors, such as residual solvent of the adhesives or insufficiently removed surface water.[6] Bond strength and durability seem to rely on the quality of the hybrid layer (on the proper impregnation of the dentin substrate), rather than on the thickness or morphology of the hybrid layer/resin tags.[7]

Different clinical approaches have been proposed to improve monomer infiltration, reduce the rate of water sorption and reduce collagen degradation. Use of an additional layer of hydrophobic resin agent, multiple layer applications, enhanced solvent evaporation, prolonged curing time, use of MMP inhibitors and use of electric current[7] and an active rubbing of primer with moderate pressure using brushes or sponges, may improve and accelerate the monomer inter-diffusion process.[3]

One of the most critical steps in the etch-and-rinse and self-etch approach is the priming step, because it allows for infiltration and hybridization at the dentin bond interface. The active agitation of self-etching bonding agents may improve bonding by enhancing removal of the smear layer[8] and the interaction of acid monomers with tooth structure and dispersing the etching byproducts into the hybrid layer. While some studies reported a significant effect of agitation on the improvement of dentin bond strength with total-etch systems,[9] other studies did not find this improvement statistically significant.[10] Chan and others[9] observed complete dissolution and dispersion of the smear layer after agitation in mild self-etch adhesives. Velasquez and others[12] reported significant improvement in dentin shear bond strength after agitation in all three self-etch adhesives—Clearfil SE BOND, Xeno III and Adhe SE—at the 20-second application time, while there was no effect of agitation at 30 seconds and limited effect at the 10-second application.

Air drying of primed dentin is another simple approach correlated with enhanced solvent evaporation to avoid phase separation within the adhesive agent.[7] Primed tooth surfaces are usually air dried to evaporate solvents and disperse water, because the remaining liquid primer on the adhesive surface may act as an inhibitor of polymerization of the bonding agent.[13] Excess water competes for space with HEMA molecules within the collagen network, resulting in lower HEMA density inside the collagen mesh, thus lowering bond strength.[14] While Bonilla and others[15] reported maximum bond strength with a dry time of one second with both the self-etch and total-etch systems, Chiba and others[13] reported maximum bond strength with a dry time of five seconds with the self-etch systems. Furthermore, Miyazaki and others[10] found that drying time varied for two total-etch systems tested. For Imperva Bond, maximum bond strength was obtained with 10-30 seconds, and for ScotchBond Multi Purpose, maximum bond strength was obtained with one-to-five seconds, while Miyazaki and others[16] found variable drying time of primer for three self-etch systems. For Liner Bond II and FluoroBond, a minimum drying time of 10 seconds was required, while Mac Bond was least affected by drying time.

The differing conditions *in vivo* and *in vitro* can influence the adaptation of bonding agents to the dentinal surface. It has been suggested that the hydrostatic pulpal pressure, dentinal fluid flow and increased dentinal wetness in vital dentin can affect the intimate interaction of certain dentin adhesives with dental tissue.[14,17-18]

Previous studies[17,19-29] have observed significantly lower bond strength with simulated physiologic hydrostatic pressure. Hebling and others[30] observed that the impact of intrapulpal pressure on bond strength seems to be more adhesive dependent than dentin morphological characteristics related to depth. Mason and others[31] observed that, with new hydrophilic dentin bonding systems, shear bond strengths *in vitro* were not substantially different from *in vivo* application.

The reliability and validity of bond strength determination on dentin bonding have been questioned, and laboratory tests have generally been considered to be unreliable and not predictive of clinical behavior.

Thus, the current study evaluated the combined effects of placement agitation and primer drying time on the shear bond strength of dentin in *in vivo* conditions. The null hypotheses tested were that primer drying time did not affect the dentin shear bond strength and primer agitation had no effect on dentin shear bond strength.

METHODS AND MATERIALS

The current study was performed on 60 human maxillary and mandibular caries-free premolars scheduled to be removed for orthodontic purposes from patients of similar age groups, ranging in age from 14 to 20 years, with an average age of 17 to 18 years. Approval from the post-graduate board of study was taken before the procedure, and the procedure was explained to the patient. After informed consent was obtained from each patient, the occlusal surfaces of the teeth were wet ground using a straight fissure diamond bur (Dentsply DeTrey) ISO 012 in an air water-cooled high-speed handpiece at a speed of 150,000 rpm to obtain a flat dentin surface. The entire occlusal surface was flattened and an occlusal clearance of 2 mm was obtained from the deepest fissure. Adequate isolation was obtained using cotton rolls and high vacuum suction. The teeth were divided into six groups of 10 teeth each using Clearfil SE bond in all the groups according to the manufacturer's instructions, with a varying drying time of the primer and application method. An equal number of maxillary and mandibular teeth were included in each group. The maximum width and length of the flat surfaces were calculated, and they were in the range of 9-10 mm buccopalatally and 7-8.5 mm mesiodistally for the maxillary premolars and 7.5-8.5 and 7-8 mm, respectively, for the mandibular premolars.

The primer was applied with a 3M brush for 20 seconds and dried with a three-way syringe from a distance of 5 cm using an air pressure of 80 psi for 0, 5 and 10 seconds with and without agitation. In the agitation group, the primer was vigorously rubbed using a 3M brush for the entire duration of the 20-second application time by a single operator using similar pressure in all samples. Bond was then applied and made as thin as possible with a gentle stream of dry air and it was cured for 10 seconds according to the manufacturer's direction using a QTH light with an output of 600 mW/cm^2. The flat tooth surface was restored to its original occlusal anatomy with the hybrid composite Spectrum TPH in increments of 1 mm placed over the cured adhesive and cured for 40 seconds with the same visible light-curing unit. The restorations were checked for any occlusal discrepancy and polished using the Enhance disk system.

The teeth were extracted one week after placing the restorations using elevators and forceps without touching the restoration tooth interface. For mechanical loading, metallic rings one-inch in diameter and two-inches high were used. The rings were filled with auto-polymerizing acrylic resin up to their uppermost margin. All the specimens were placed in the rings with the roots covered with acrylic resin. The specimens were placed perpendicular to the acrylic resin surface. The mounted rings were stored in distilled water until testing. Bond strength between the restorative material and tooth surface was measured in the shear mode using a Universal Testing Machine, number LR100. The specimens were mounted in a jig, while a straight knife-edge rod was applied at the tooth-restoration interface at a crosshead speed of 1 mm/minute. Load was applied until restoration failure occurred. The maximum force to debond the area was estimated in kgf.

Statistical Analysis

The results were analyzed by calculating the mean shear bond strength and standard deviation for each group. The statistical analysis was done using the one-way ANOVA, two-way ANOVA and post-hoc Tukey tests to evaluate differences among the experimental groups at a significance level of p=0.05.

Table 1: *Manufacturer's Recommended Technique*

	Clearfil SE Bond
Tooth Surface	Dry
Application	Dispense the necessary amount of primer into a well immediately before application.
	Apply primer to cavity walls and leave it in place for 20 seconds.
	Evaporate the volatile ingredients with a mild air stream. Avoid pooling.
	Dispense the necessary amount of bond into a well.
	Apply bond to the cavity.
Drying	Make the bond as thin as possible using a gentle air stream.
Curing	10 seconds

Table 2: *Shear Bond Strength Values in kgf*

	Without Agitation (Group I)			With Agitation (Group II)		
Sample #	**Zero Seconds**	**Five Seconds**	**10 Seconds**	**Zero Seconds**	**Five Seconds**	**10 Seconds**
	(A)	**(B)**	**(C)**	**(A)**	**(B)**	**(C)**
1	31.92	35.49	38.05	29.68	50.45	36.61
2	36.69	31.16	26.93	39.74	53.52	35.44
3	38.63	30.60	34.93	28.97	53.64	35.06
4	37.33	38.18	31.17	39.54	49.56	30.23
5	30.21	29.04	33.78	29.17	51.56	27.91
6	38.33	33.24	32.75	38.65	47.65	34.43
7	36.33	33.25	35.87	37.65	53.72	33.43
8	35.68	34.19	34.76	29.87	47.43	28.76
9	37.68	31.19	28.67	25.77	54.61	26.76
10	32.80	32.56	30.79	35.16	78.26	41.87
Average Value	35.56	32.89	32.17	33.42	54.04	33.05

Table 3: *Results of Shear Bond Testing to Dentin with Standard Deviation for Each Group*

	Without Agitation (I)	With Agitation (II)
Dry Time	**SBS (kgf)**	**SBS (kgf)**
Zero (0) seconds (A)	35.56 ± 2.35[a]	33.42 ± 5.25[a]
Five (5) seconds (B)	32.89 ± 2.63[a]	54.04 ± 8.88[b]
Ten (10) seconds (C)	32.17 ± 3.07[a]	33.05 ± 4.64[a]

**Values with the same letter are not statistically significant at* p*=0.05.*

RESULTS

The results of the shear bond strength tests with different air-drying times and application of primer onto the dentin surface are shown in Tables 2 and 3 and Figure 1. The dentin bond strengths varied with air-drying time and agitation. The values obtained in the various groups were: 35.56 ± 2.35 (IA), 32.89 ± 2.63 (IB), 32.17 ± 3.07 (IC), 33.42 ± 5.25 (IIA), 54.04 ± 8.88 (IIB), 33.05 ± 4.64 (IIC) (Table 3). The results of the two-way ANOVA indicates a statistically significant overall model (F=24.595, p=0.000). The variable agitation and drying times were also statistically significant (F=23.834, p=0.000) and (F=24.976, p=0.000), respectively. The interaction between agitation and drying time were also found to be statistically significant (F=31.706, p=0.000). While evaluating shear bond strength to dentin, agitation and primer drying time significantly interacted at five seconds (Table 3). In the non-agitation group, no significant difference in the mean shear bond strength was found (p>0.05). The decreasing order of the mean shear bond strength value was: IIB > IA > IIA > IIC > IB > IC. No significant difference was found between groups IIA and IIC, while significant differences were found between groups IIA–IIB and IIB–IIC (p<0.05).

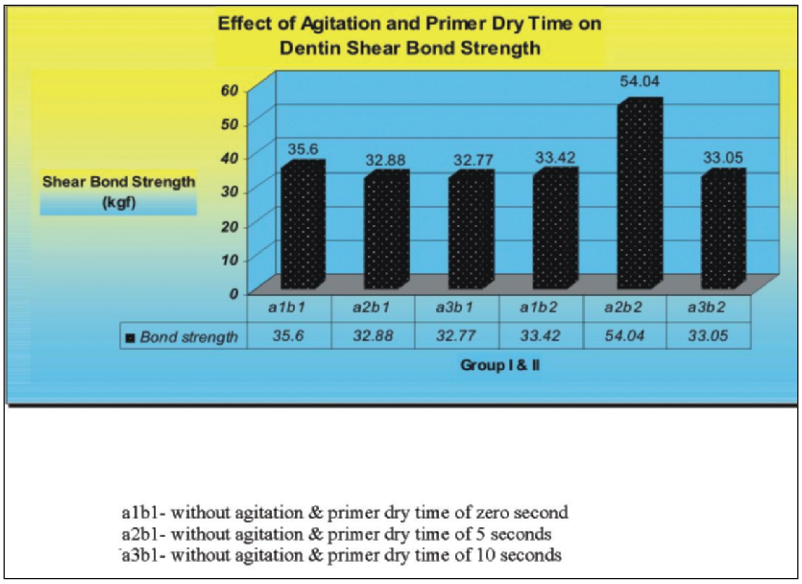

Figure 1.

DISCUSSION

Adhesive systems in restorative dentistry allow for the use of conservative preparations, the reduction of microleakage in the tooth-restoration interface, the prevention of recurrent caries, marginal discoloration and the reduction of post-operative sensitivity. Therefore, one of the primary objectives of researchers is to achieve a strong, durable and predictable union between restorative materials and the tooth surface.[32]

In the current study, the shear bond strength was tested instead of the tensile bond strength. The shear bond strength would seem to have an advantage over the tensile bond strength in that it appears to more likely produce failure at the tooth adhesion interface and represents the bond strength between the tooth and the material rather than the strength of the material or tooth.[33] The major disadvantage of microtensile bond strength is rather labor-intensive, technically demanding and a relatively fragile sample preparation technique. It also induces micro fractures at the interface during specimen preparation, which may weaken the bond and thus reduce the actual bond strength.[2]

The current study depicted no effect of drying time on dentin bond strength when samples were not agitated. Significantly higher bond strength was observed at the five-second air-drying time when samples were agitated for 20 seconds. However, the effect was not significant when drying was done for 10 seconds or not done. The ranking of bond strengths from highest to lowest yielded the following results: IIB > IA > IIA> IIC >IB > IC.

The results of current study, which show no effect of air drying in the non-agitation group, are contrary to previous *in vitro* studies that reported higher bond strength with the air drying of primer in self-etch adhesives.[15-16,34-35] These differences could be related to the different bonding substrates (vital versus non-vital). As opposed to laboratory conditions, vital dentin is an intrinsically hydrated tissue penetrated by a network of 1 µm to 2.5 µm diameter fluid-filled dentinal tubules. The flow of fluid from the pulp to the dentin enamel junction is the result of a slight but constant pulpal pressure. Pulpal pressure has a magnitude of 25 to 30 mm Hg or 34 to 40 cm H_2O.[36]

Clearfil SE bond contains an aqueous mixture of phosphoric acid ester monomer (10–MDP) with a pH relatively higher than that of phosphoric acid-etching gels.[37] Although the pH of a 34%-37% phosphoric acid gel is in the range of 0.5 to 1.0, the pH of Clearfil SE primer is 1.9 to 2.0.[38-39] Longer contact of primer would have sufficiently demineralized the smear layer to substantially increase permeability, thus effecting the adaptation and/or pooling of fluid. When bonded under dentin perfusion, self-etch allowed water movement across adhesives, and none of the self-etch adhesive was found to be any more effective in sealing dentin than the original smear layer.[40]

In the current study, in *in-vivo* conditions, the effect of drying time was only significant when the primer was actively agitated. These results suggest that the effect of evaporation of the remaining solvent would not have been apparent in the non-agitation group as in vital dentin; besides the smear layer, the outward dentinal fluid flow and tubular contents would not have allowed the effective infiltration and contact of primer to dentin. The significantly better bond strength with five-second air drying time in the agitation group could be related to better solvent evaporation and dispersion of water.[13]

Better bonding with active agitation could be attributed to better removal of the smear layer, dispersal of fluid content and better diffusion of dentin primer into the collagen-rich partially-demineralized zone, which helps in achieving the micromechanical and chemical interaction with the underlying dentin.[10]

Active agitation of self-etching bonding agents may improve bonding by enhancing the interaction of acid monomers with tooth structure and dispersing etching byproducts into the hybrid layer.[12] With continuous agitation, smear layers were completely dispersed or dissolved, and thicker hybrid layers with upstanding collagen fibrils were observed.[11]

Chan and others found bond strength to dentin with a thick smear layer increased significantly with agitation and, on SEM evaluation, they noted passive application resulted in a hybridized smear layer, while agitation resulted in the smear layer being completely dissolved or dispersed into the adhesive.[11]

Reis and others[41] concluded that the rubbing action can improve the kinetics and allow for better monomer diffusion inward, while solvents are diffusing outward, which may explain the high bond strength values obtained under slight and vigorous agitation. It is likely that vigorous agitation improved the removal of residual water, which increased the degree of conversion and cross-linking of the polymer and, consequently, the mechanical properties of the resin inside the hybrid layer.

The reversal of bond strength, even in the agitation group with a longer drying time, could be related to a greater increase in fluid filtration due to longer primer contact and/or more evaporation of tubular content. It is also possible that the remaining primer becomes saturated with air that could, in turn, inhibit polymerization of the bonding agent.[10] Longer drying time might also change the monomer/water ratio and, subsequently, result in phase separation and blistering.

Considering the findings of the current study, the benefit of air drying was only apparent when samples were actively agitated. These data suggest that incorporation of active agitation may be an essential step for self-etch primers. However, optimum drying time with agitation for different self-etch primers in vital dentin needs further investigation, as the effect may differ with mild and moderate pH self-etch primers. Since the current study has been performed in young, caries-free premolars scheduled for orthodontic extraction, these results may overestimate clinical conditions and require further long-term studies on different boding substrates, such as caries affected dentin.

CONCLUSIONS

Within the limits of this *in vivo* study, it can be postulated that the air drying of primer has no significant effect on dentin shear bond strength *in vivo*. The effect of agitation was pronounced only when the primer was dried for a period of five seconds. No significant effect of agitation was seen when the primer was not dried or was dried for 10 seconds.

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