



Evaluating The Effect of pH of Dentin Bonding Agents on Dentin in Relation to the Push-Out Bond Strength of Composites in Class I Cavities *in-vitro*

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INTRODUCTION: Today, the popularity of amalgam as restorative material has decreased due to poor esthetic characteristics and mercury contamination with composite resin materials becoming a choice among the both dentists and patients.

AIM: The objective of this *in-vitro* study was to evaluate the effect of pH of dentin bonding agents (Adper Easy Bond and Xeno V) on dentin in relation to push out bond strength of composite restored with Filtek bulk fill posterior restorative composites.

MATERIALS AND METHOD: Sixty caries free human molars with no cracks or previous restorations were used for investigation. Standard box-type Class-I Cavities of 3.5 mm x 3.5 mm and depth of 4 mm were prepared on the occlusal surface of the molars. Teeth were randomly divided into 2 groups namely Group A, Group B of 30 teeth each, according to the dentin adhesives applied i.e Adper Easy bond and Xeno V. Two bonding agents with different pH were selected for the study. One was Adper Easy Bond with Ultra mild pH of 2.7. Other was Xeno V Self etch with strong pH of <1.3. Push-out test was performed by placing specimens within a centralizing ring to ensure a centered application of the load, resting on another ring, with a central hole slightly larger than the restoration diameter. The test was performed with a universal Testing Machine to express bond strength in mega pascals (MPa), load value in Newton (N). After testing the push-out bond strength, the samples were analyzed under a stereomicroscope.

RESULTS: Adper Easy bond with an ultra mild pH 2.7 is better as compared to Xeno V Self etch with strong pH of <1.3. The most common mode of failure observed with Adper was mixed fracture and cohesive dentin fracture, while in Xeno V, adhesive failure between resin and dentin and cohesive resin fracture was observed. The mean maximum force was significantly more among Adper compared to Xeno V.

CONCLUSION: Comparison of dentin bonding agents in this *in vitro* study revealed that the push out bond strength of Adper Easy performed better than Xeno V.

KEYWORDS: Bonding agents, Dentin, Strength, *in-vitro*

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INTRODUCTION

Amalgam was introduced to the United States in the 1830s. Today, the popularity of amalgam as restorative material has decreased due to poor esthetic characteristics and mercury contamination, this material was mainly used as it still cost-effective when used as direct restorative material.¹ This material has gained popularity due to its good mechanical properties such as, easy handling, low technique sensitivity, wear resistance and low cost. The most common reason for replacing amalgam are marginal degradation and secondary caries.² With the advancement composite resin materials were introduced in dentistry more than 50 years ago, since then these materials have been continuously evolving. It has become a choice among the patient as it not only helps in maintaining the ideal tooth form and function but also esthetics.³

Among all the direct filling restorative materials, composite is the most known material esthetically. Initially these materials were used as anterior

restorative materials, but later due to improvement in technologies the possibility of restoring posterior teeth with composite was introduced. However, there are certain drawbacks of composite such as post operative sensitivity, discoloration of the restoration, marginal fracture, recurrent caries, gross fracture of the restoration, lack of maintaining contact, accumulation of plaque around the restoration.⁴ With development in technology has improved the physical properties of resin based composite and has also expanded their use in clinics. Therefore newer composites have been introduced with better properties and different restoration techniques like bulk fill, flowable and incremental.² The most widely accepted method of placement of composite is incremental method. According to this technique composite should be placed in layers to reduce the polymerization shrinkage and to achieve depth of cure.^{5,6}

Adhesion refers to the forces or energies between atoms or molecules at an interface that hold two phases



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together. Adhesive restorations better transmit and distribute functional stresses across the bonding interface to the tooth and have the potential to reinforce weakened tooth structure.⁷ A strong bond between dental material and tooth structure is very important so as to achieve esthetic and biologic properties. The bonding between enamel and resin is due to micromechanical bond, However bonding of resin to dentin is difficult due to complex dentin structure. Bonding with dentin is achieved by acid etching which removes the inorganic matrix of dentin leaving organic substance but when the tooth is dried the collagen in the organic substance shrinks and collapse in order to prevent the collapse the moist and wet bonding technique should be used to preserve the integrity of collagen matrix.(bonding).⁸ In order to achieve a balance various desensitizing solution have been used as rewetting agents which is said to enhance the bond strength and also reduces the postoperative sensitivity.^{9,10}

MATERIALS AND METHOD

Sixty caries free human molars with no cracks or previous restorations were fabricated. These molars were cleaned and stored in distilled water. All the molars were flattened on their occlusal surface to create an occlusal plane perpendicular to the axial axis. During sample preparations, standard box-type Class-I Cavities of 3.5 mm x 3.5 mm and depth of 4 mm were prepared with the help of air-rotor and bur on the occlusal surface of the molars.

Cavity Restoration: In order to compare the bond strength, the teeth were randomly divided into 2 groups namely Group I, Group II of 30 teeth each, according to the dentin adhesives applied i.e Adper Easy bond (3M ESPE, St Paul, MN, USA) and Xeno V (Dentsply, DeTrey, Germany).

Two bonding agents with different pH were selected for the study. One was Adper Easy Bond with Ultra mild pH of 2.7. Other was Xeno V Self etch with strong pH of <1.3.

Groups: Group A (n=30) restored with Adper Easy bond and Group B (n=30) restored with Xeno V Self etch adhesives. The bonding agents were used according to manufacturer's instructions (table 3)

Push-out test: To access the bond strength of restoration to the lateral walls of the occlusal cavity, the specimens were placed within a centralizing ring to

ensure a centered application of the load, resting on another ring, with a central hole slightly larger than the restoration diameter. The load was applied on the apical coronal direction using a punch tip, which passed through a guide cylinder to ensure a central load application. The test was performed with a universal Testing Machine and bond strength was recorded for statistical analysis.

Fractured slices were carefully removed and observed under a stereo microscopic at 20x to categorize the type of failure as follows:

Type I: Adhesive failure between resin composite and dentin.

Type II: Cohesive resin fracture

Type III: Mixed fracture: pressure of fragments of dentinal tissues or resin composite adhered to interface.

Type IV: Cohesive dentin fracture

The results were statistically analyzed using the Chi squared test, and the unpaired and independent student's t-test.

RESULTS

When Group A i.e (Adper Easy bond) was compared with Group B i.e.(Xeno V) the maximum failure in Group A were Type IV with 36.7% and minimum was Type II and Similarly the maximum failure in Group B was Type I 43.3% followed by Type II 13.3%. as seen in table 1.

	Group A	Group B
Type I failure	9 30.0%	13 43.3%
Type II failure	0 0.0%	4 13.3%
Type III failure	10 33.3%	6 20.0%
Type IV failure	11 36.7%	7 23.3%
Chi-square value = 23.215, p-value = 0.001*		

Table 1. Comparison of type of failure between Group A and Group B

The maximum force was compared between Adper and Xeno V. The mean maximum force (in Newton) was significantly more among Adper (with p-value 0.019) compared to Xeno V (table 2).

Upon evaluating the data statistically using unpaired t-

Groups	Max force (Newtons)				
	Mean	Std. Deviation	Mean Difference	t-test value	p-value
Adper	273.06	134.18	53.10	4.420	0.019*
Xeno-V	219.97	115.52	54.98	4.98	0.876

Table 2. Comparison of mean maximum force between Adper Easy bond and Xeno V

test the mean maximum force (in newtons) was compared between Adper and Xeno-V. The mean maximum force (in newtons) was significantly more among Adper compared to Xeno-V (table 3).

DISCUSSION

The maximum push-out bond strength was seen with Adper Easy bond with Ultra mild pH OF 2.7 as compared to Xeno V with pH(<1.3) in all the age groups. The results showed that the push out bond strength of Adper Easy Bond performed better than Xeno V.

The above results sheds light on the use of Adhesives with different composition and their dependence on pH. We propose that pH dependent efficiency depends on presence and absence of HEMA. To compare, we have used two different types of adhesives depending on the presence or absence of HEMA. Adper Easy Bond contains HEMA (2-hydroxyl ethyl methacrylate) which is absent in case of Xeno V.¹¹ Two bonding agents with different pH were selected for the study. One was Adper Easy Bond with Ultra mild pH 2.7 and other was Xeno V Self etch with strong pH <1.3.¹¹

We observed that Adper Easy Bond responded strongly to pH 2.7 and showed significant enhancement of push-out bond strength which range from 212.44-346.61 MPa in different age groups (20-44 years, 44-79 years, 80 years and above) except higher age group (80 years and above) while as Xeno V Self etch did not show enhancement of push-out bond strength in different age groups. The reason for superior adhesion properties of Adper Easy Bond is because of presence of HEMA whereas Xeno V Self etch is not composed of HEMA.¹² The higher potential of HEMA for hydrophilicity makes it potentially stronger adhesion promoting monomer. Further pH of 2.7 enhances this HEMA dependent hydrophilicity of Adper Easy Bond by increasing wettability. Another reason that promotes bonding strength to dentin is increasing substrate penetrability and diffusibility, pH seems to

modulate both these factors and also helps to keep demineralized collagen wet. The potential of Adper Easy Bond to attain higher bond strength at pH 2.7 is partly because of presence of ethanol as solvent. The pH 2.7 seems to favor solvent properties by keeping ethanol in hydroxide form, thereby allowing efficient removal of water from dentin spaces and collapsing of hydrogen, thus enlarging the inter-fibrillar spaces and allowing more resin infiltration. On the other hand Xeno V Self etch uses acetone as solvent. Acetone is poor in removing water from dentin spaces and also water is necessary for Xeno V Self etch demineralization process leading to presence of excess water. Excess water seems to be reason for its poor bond strength.^{12,13}

The analysis of failure modes in this study revealed that most of the failure with Group A i.e (Adper Easy bond) when compared with Group B i.e.(Xeno V) it was observed that maximum failure in Group A were Type IV (Cohesive dentin fracture) with 36.7% and minimum was Type III (Mixed fracture: pressure of fragments of dentinal tissues or resin composite adhered to interface) and Similarly the maximum failure in Group B was Type I (Adhesive failure between resin composite and dentin) 43.3% followed by Type II (cohesive resin fracture) 13.3%. as seen in table 1.

The mixed type of failure could be due to lack of proper adaptation of the materials to cavity walls with void formation owing to its fluctuating viscosity combined with low shrinkage and contraction stress upon curing of composite. Adhesive type of failure may be attributed to the heavy viscosity of Filtek™ Bulk fill composite, which might hinder the appropriate adaptation of the material to the cavity walls, resulting in void formation at the tooth restoration interface and Cohesive type of failure as observed may be due to the incorporation of voids or contamination between composite layers.¹⁴

CONCLUSION

Within the limitations of this study, it can be concluded that:

-The maximum push-out bond strength was seen with Adper Easy bond with ultra mild pH OF 2.7 as compared to Xeno V (with pH<1.3). The results showed that the push out bond strength of Adper Easy Bond performed better than Xeno V.

	Adper™ Easy Bond Self-Etch Adhesive (3M ESPE)	XENO® IV One-Component Light Cured Self-Etching Dental Adhesive* (Dentsply)
Composition	<ul style="list-style-type: none"> • 2-Hydroxyethyl Methacrylate (HEMA) <ul style="list-style-type: none"> • Bis-GMA • Methacrylated phosphoric esters <ul style="list-style-type: none"> • 1.6 hexanediol dimethacrylate • Methacrylate functionalized Polyalkenoic acid (Vitrebond™ Copolymer) • Finely dispersed bonded silica filler with 7nm primary particle size <ul style="list-style-type: none"> • Ethanol • Water • Initiators based on camphorquinone <ul style="list-style-type: none"> • Stabilizers 	<ul style="list-style-type: none"> • Mono-, Di- and Trimethacrylate resins <ul style="list-style-type: none"> • PENTA • Photoinitiators <ul style="list-style-type: none"> • Stabilizers • Cetylamine hydrofluoride <ul style="list-style-type: none"> • Acetone Water
Indications	<ul style="list-style-type: none"> • All classes of fillings light-curing composite or compomer • Core build-ups made of light-curing composite <ul style="list-style-type: none"> • Repair of composite or compomer fillings <ul style="list-style-type: none"> • Root surface desensitization • Intraoral repair of existing composite, porcelain to metal, and all ceramic restorations • Cementation of indirect restorations made of composite or compomer, ceramic and metal using RelyX™ ARC Adhesive Resin Cement 	<ul style="list-style-type: none"> • Adhesive for direct, light-cured composite and compomer restorations.
Manufacturer's Claims	<ul style="list-style-type: none"> • One-bottle, one-coat convenience • Bond performance equal to or greater than other one-bottle self-etch adhesives <ul style="list-style-type: none"> • Reduced risk of post-operative sensitivity • Faster procedure for less risk of contamination • Moisture tolerant - works on wet or dry enamel and dentin <ul style="list-style-type: none"> • Low nanoleakage • Available in unit-dose delivery 	<ul style="list-style-type: none"> • Etching, priming, bonding in a single component self-etch dental adhesive • High bond strengths to dentin and enamel <ul style="list-style-type: none"> • Minimal post-operative sensitivity • Less technique sensitive than total etch systems <ul style="list-style-type: none"> – no over-drying or over-etching of dentin • Superb sealing ability including impregnating the smear layer and dentin tubules <ul style="list-style-type: none"> • Bottle and Unit Dose delivery • Releases fluoride
Application Technique	<ol style="list-style-type: none"> 1. Apply adhesive to tooth surface for a total of 20 seconds 2. Air thin the adhesive for 5 seconds 3. Light cure for 10 seconds 	<ol style="list-style-type: none"> 1. Apply generous amounts onto cavity surfaces and actively scrub for 15 seconds (20 seconds for larger restorations) 2. Repeat step 1 3. Uniformly spread adhesive by a gentle stream of air pressure until there is no more flow 4. Light cure for at least 10 seconds
Storage Conditions	To achieve the maximum amount of shelf life, store the product at 2-8°C/36-46°F. Refrigeration is not required if the product is depleted within six months.	Store with original cap only, kept out of direct sunlight and stored in a well ventilated place at refrigerated temperature between 2°C/35°F and 8°C/46°F.
Packaging	5 ml Bottle Unit Dose Applicators	4.5 mL Bottle Unit Dose

Table 3. Information regarding Adper Easy bond and Xeno V

-When group A i.e (Adper Easy bond) was compared with group B i.e.(Xeno V) the maximum failure in group A were Type IV with 36.7% and minimum was Type II and similarly, the maximum failure in group B was Type I 43.3% followed by Type II 13.3%.

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