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An *in-vitro* Investigation of the Effect of a Water Additive and a New Acidic Primer on the Tensile Bond Strength of Composite Resin to Human Enamel and Dentine.

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Abstract - *The aim of this study was to investigate the effect of a water additive on the bond strength of a composite restorative material to enamel and dentine which have undergone a range of surface treatments regimes. Human enamel and dentine specimens were prepared and divided into 3 surface treatment groups. Composite resin restorative material was bonded to the surface prepared enamel and dentine specimens. The tensile bond strength of the composite resin was measured using a Lloyd's testing machine. The tensile bond strength of composite resin to etched enamel surfaces using a standard etch technique was significantly greater than that to dentine ($p < 0.05$). For those specimens subjected to the addition of the water additive 'Alpron', the bond strength to the enamel was still significantly greater than to the dentine ($p< 0.005$). The addition of 'Alpron' to the water significantly improved the bond strength of composite to enamel and dentine when etched and placed with 'Prime&BondNT' ($p < 0.05$). There was no significant difference in bond strength of enamel and dentine specimens bonded using Xeno III ($p> 0.05$). However, the bond strength of dentine specimens bonded with Xeno III was significantly greater than other dentine specimens bonded with Prime&BondNT ($p< 0.05$). For those groups of specimens which included the water additive 'Alpron' in the regime, the tensile bond strength of the composite resin restorative material to enamel and dentine was greater than for those non water additive groups. The tensile bond strength of composite resin bonded to enamel surfaces prepared with 'Alpron' was significantly higher than that bonded to dentine surfaces.*

KEY WORDS: Primer, Water Additive, Bond Strength, Composite Resin

INTRODUCTION

It is well documented that water from dental unit water lines may be heavily contaminated with microrganisms. The source of bacterial contamination has been identified as micro-colonies of proliferating bacteria, fungi and protozoa forming a biofilm on the inner surface of the water lines [1,2]. A number of products are now available on the market for the control of biofilm and effluent contamination of dental unit water lines [1,3,4]. There are however some concerns of the effect of these agents on the bond strength of restorative materials to enamel and dentine [5,6].

The use of the acid etch technique as a means achieving chemical and micromechanical bonds between resin based restorative materials and enamel and is now a recognised and documented technique [7,8]. The bonding of resin based restorative materials to dentine is however more challenging and long term bonding is less predictable than that to enamel. There have however been significant advances in dentine bonding technology with the development of adhesive monomers which penetrate into the superficially demineralised dentine [9]. The plethora of dentine bonding agents available for purchase, coupled with their many and varied means of application, can lead to confusion. Manufacturers have now however attempted to reduce the multiple steps required to achieve dentine bonding and this has been reflected in the development of the "one bottle" adhesives. One such new product on the dental market is Xeno III (Dentsply, De Trey GmbH, Konstanz, Germany). The manufacturers have claimed that surface treatment of enamel and dentine with Xeno III provides improved adhesion to dentine. For some of the surface treatments, for example, Prime and Bond NT (Dentsply, De Trey GmbH, Konstanz, Germany), the recommended regime includes rinsing the tooth surface with water but for other treatments, for example Xeno III, this stage is not included. It is for the former group of surface treatments that the water additives may have an effect on the bond strength of the restorative material to the tooth.

The aim of this study was to investigate the effect of surface treatment on the bond strength of a composite restorative material to enamel and dentine. The hypothesis was that the effect of the surface treatment regime would have an effect on the bond strengths.

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Table 1. *Materials used in the investigation together with their manufacturers.*

Dental Material	*Components*	*Manufacturer*
Spectrum Lot number: 0404001158 Expiry 2006-01	▪ Polymerisable monomers (Bis-GMA-adduct & Bis-EMA) ▪ TEGDMA ▪ Photo-initiator ▪ Stabilisers ▪ Bariumaluminiumborosilicate filler particles (mean particle size <1 microns in diameter) ▪ Highly dispersed silicon dioxide filler particles (<0.04 micron in diameter)	Dentsply De Trey GmbH Konstanz Germany
Prime&BondNT Lot number: 0406000178 Expiry 2006-05	▪ Di- and trimethacrylate resins Amorphous silica (Nanofiller) ▪ PENTA ▪ Photoinitiators ▪ Stabilisers ▪ Cetylamine hydrofluoride ▪ Acetone diluent	Dentsply De Trey GmbH Konstanz Germany
Xeno III *Liquid A*	▪ 2-hydroxyethyl methacrylate (HEMA) ▪ Purified water ▪ Ethanol ▪ 2,6-Di-tert-butyl-p hydroxy toluene ▪ Highly dispersed silicon dioxide (Nanofiller)	Dentsply De Trey GmbH Konstanz Germany
Liquid B Lot number: 0406001766 Expiry 2006-01	▪ Phosphoric acid modified methacrylate (Pyro- EMA) ▪ Pentamethacryloxyethyl cyclo-phosphazen mono fluoride (PEM-F) ▪ Urethane dimethacrylate (UDMA) ▪ 2,6-Di-tert-butyl-p-hydroxy toluene (BHT) ▪ p-Dimethylamino ethyl benzoate (EPD) ▪ Camphorquinone	
Conditioner 36 Lot number: 0312000627 Expiry 2008-10	▪ Phosphoric acid ▪ Highly dispersed silicon dioxide ▪ Detergent ▪ Pigment ▪ Water	Dentsply De Trey GmbH Konstanz Germany
Alpron Lot number: 504205 Expiry 2006-03	▪ Phenylalanine ▪ Tosylchlcramide sodium	Dental Produkte GmbH St. Georgen Germany

Table2. *Surface treatment procedure for each group of specimens.*

Group n=15	*Surface*	*Surface Treatment*
1	Enamel	Enamel Control: Etched for 15 seconds, washed with tap water using triple syringe for 15 seconds, air dried for 2 seconds, Prime&BondNT and composite resin placed
2	Enamel	Etched for 15 seconds, washed for 15seconds with Alpron containing water, air dried for 2 seconds, Prime&BondNT and composite resin placed
3	Enamel	Xeno III - self etching primer adhesive prior to the application of composite resin
4	Dentine	Dentine Control: Etched for 15 seconds, washed with tap water using triple syringe for 15 seconds, air dried for 2 seconds, Prime&BondNT and composite resin placed
5	Dentine	Etched for 15 seconds, washed for 15seconds with Alpron containing water, air dried for 2 seconds, Prime&BondNT and composite resin placed
6	Dentine	Xeno III - self etching primer adhesive prior to the application of composite resin

Table 3. *The tensile bond strengths achieved between composite and the three different surface-treated human enamel and dentine. Values connected by a vertical bar are not significantly different.*

Test Group	*Mean Tensile Bond Strength (MPa) (n = 15)*	*Standard Deviation*
Group 1 ; Enamel – Control	20.54	6.49
Group 2 ; Enamel – 'Alpron'	25.79	7.55
Group 3 ; Enamel – 'Xeno III'	24.26	6.06
Group 4 ; Dentine – Control	8.00	3.63
Group 5 ; Dentine – 'Alpron'	11.21	9.28
Group 6 ; Dentine – 'Xeno III'	20.09	7.48

MATERIALS AND METHOD

The materials used in this study are presented in Table 1.

Preparation of the enamel surface

Enamel specimens were obtained from recently extracted non-carious human molar teeth. The extracted teeth were cleaned with a hand scaler to remove tissue remnants and stored in a disinfection solution containing 500 ppm available chlorine. The solution was prepared by dissolving half a sodium dichloro-isocyonurate tablet (Haz-Tabs, Guest Medical Limited, Kent UK) in one litre of water. The roots of each tooth were sectioned using an air rotor dental handpiece, discarded and the crowns hemi-sected in the longitudinal mesio-distal axis of the crown.

Each crown was carefully examined to preserve the flat enamel surfaces and a series of buccal and palatal/lingual surfaces were produced. After sectioning pulpal remnants were removed using an excavator. A diamond disc held in a slow-speed straight hand-piece was used to cut several shallow grooves of approximately 1mm depth into the internal surfaces of the sections of crowns to aid their retention in poly (methyl methacrylate) acrylic resin. The grooves were prepared on the inner aspect of the dentine from the direction of the pulp chamber to create a series of multi-directional undercuts. Each section of the crown was secured in a flask with adhesive and the jig assembled. The flask was filled with auto-polymerising acrylic resin and left in warm water for 30 minutes to allow polymerisation to occur. Once polymerised the blocks of acrylic resin containing the specimens of enamel were removed from flask.

The enamel surfaces were then ground flat manually on wet 600 Å grade abrasive paper at 200rpm using a Kemet lapping machine (Kemet, Maidstone, Kent UK) and then polished on the same machine using 1200 Å paper. The surface of the enamel available for bonding was checked regularly during the lapping procedure with a notched aluminium ring designed to demarcate the area required for bonding. These specimens were then ready for the bonding procedures and subsequent tensile bond strength testing. Specimens were stored in reverse osmotic distilled water at all subsequent stages between testing.

Preparation of the dentine surface

Following tensile bond strength testing of all the enamel specimens, the samples were re-lapped on wet 1200 Å grade abrasive paper until a flat dentine surface was obtained. These specimens were then ready for a subsequent bonding procedure and tensile bond strength testing. The specimens were stored in distilled water at all subsequent stages.

Surface Treatment and Bonding

Six test groups were prepared for tensile bond strength testing. The surface treatment regimes are shown in Table 2. The same light curing machine with an output of 700mW/cm^2 (Spectrum Curing Light, Dentsply, Weybridge, UK) was used for all groups, to eliminate variance. The light curing machine was tested with a light meter (Demetron Research Corp, Kerr, Conneticut, USA) before use for each group to ensure output energy was sufficient.

1. Groups 1 and 4 - Acid-etch preparation

The bonding jig was made up of an aluminium tube in which the specimen was compressed by a small spring against a plastic ring. The plastic ring was the same diameter as the specimen acrylic resin block with an internal cone shaped countersunk hole, the smaller end of which measured 2.5mm in diameter. When held against the enamel or dentine surfaces, the smaller end of the countersunk hole provided a bonding area of 2.5mm in diameter. When this cone was filled with composite resin, the plastic ring became bonded to the enamel or dentine surface.

Once secured in the bonding jig, the enamel or dentine was acid-etched for 15 seconds using the manufacturer's recommended etchant (36% phosphoric acid gel), washed for 15 seconds and then air dried for 2 seconds without desiccating the surface. Prime&BondNT was applied to the surface of enamel or dentine using the manufacturer's recommended applicator, left undisturbed for 20 seconds then gently blown to remove the solvent leaving a glossy finish. The adhesive was immediately light cured for 10 seconds. The cone shaped internal hole in the plastic ring was filled with the composite resin, Spectrum, to a depth of 4mm and light cured for 40 seconds. All specimens were prepared using the same shade of composite resin and resin from the same compule was used for each specimen.

On removal of the holding rod, the acrylic resin block was extracted from the bonding jig with the plastic ring bonded to the enamel or dentine surface. All the specimens were prepared in one session and placed in thermocycling apparatus. Specimens were cycled with a dwell time of 20 seconds in distilled water at 55°C followed by 20 seconds in distilled water at 5°C for 24 hours prior to bond strength testing (Figure 1). The specimens were thermocycled at extremes of temperature to which natural teeth are normally subjected with the resultant effect of artificially aging the specimens.

2. Groups 2 and 5 - Acid-etched preparation using 'Alpron' water-line additive

In these groups, the acid-etched surface was washed for 15 seconds with water containing 'Alpron' a water-line additive using the manufacturer's recommended concentration of 10ml of 'Alpron' in 990ml of water .

3. Groups 3 and 6 -Xeno III' acidulated primer preparation

The enamel and dentine surfaces were self etched, primed and Xeno III applied. This acidulated primer was applied using the manufacturer's recommended applicator. This was left undisturbed for 20 seconds then gently blown to remove the solvent leaving a glossy finish. The adhesive was immediately light cured for 10 seconds.

Tensile Bond Strength Testing

Immediately after thermocycling, the specimens were tested to failure on the Lloyds Materials Testing Machine (Lloyds Machine Model 2000R, Lloyds Instruments PLC, Southampton, UK) with a cross head speed of 1mm/minute. The force at failure was recorded by the load cell and noted as the load in Newtons (N). From the load and the known bonding area of 2.5mm diameter (radius 1.25mm) the bond

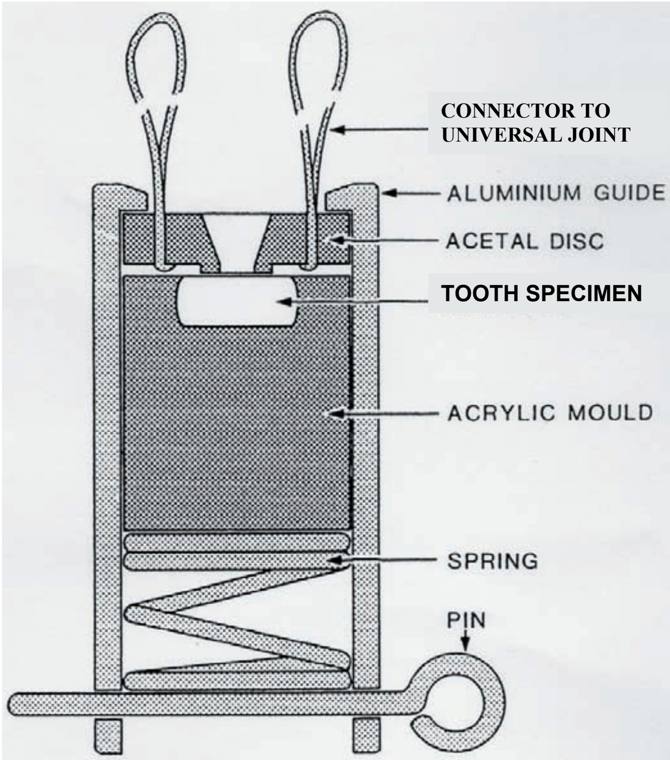

Figure 1. *The specimen mounted in the test jig prior to tensile bond testing.*

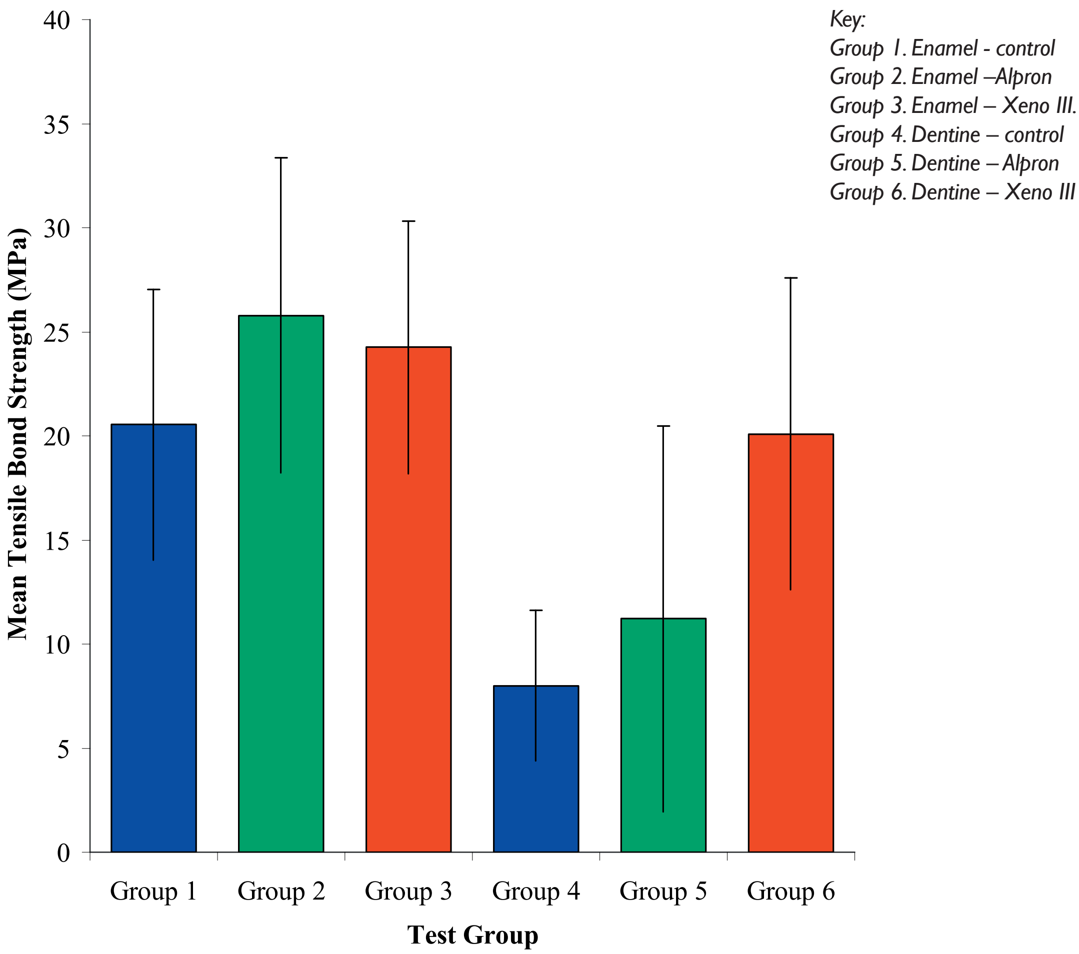

Figure 2. *Mean tensile bond strength of composite resin to enamel and dentine.*

strength was calculated in Mega Pascals (MPa). For this calculation the following formula was used: Bond Strength (MPa) = Force (N) / Area [$\pi \times$ radius $\times$ radius] (m^2).

RESULTS

The results are presented in Table 3 and Figure 2. The results were subjected to statistical analysis. A two way analysis of variance demonstrated a significant

difference in the bond strength for groups of enamel and dentine specimens ($p<0.001$). Similarly, significant differences were identified for the surface treatment regimens ($p< 0.0003$).

The bond strength values in each of the groups were compared using multiple range tests. The tensile bond strength of composite resin to etched enamel surfaces using a standard technique was significantly greater than that to dentine ($P < 0.05$). For those specimens subjected to the addition of the water additive, the bond strength to the enamel was still significantly greater than to the dentine ($P < 0.005$).

The addition of 'Alpron' to the water significantly improved the bond strength of composite to enamel and dentine when etched and placed with 'Prime&BondNT' ($P < 0.05$).

There was no significant difference in bond strength of enamel and dentine specimens bonded using Xeno III ($P > 0.05$). However, the bond strength of dentine specimens bonded with Xeno III was significantly greater than other dentine specimens bonded with Prime&BondNT ($P < 0.05$).

DISCUSSION

Adhesive techniques have enabled dentistry to become more conservative in terms of cavity design. Large cavity preparations and their extension for prevention have been replaced by more tooth conserving methods as adhesive restorative materials can now be placed in non-retentive cavities [10-12]. There are today, a number of surface treatments marketed to enhance the bond strength between restorative materials and enamel and dentine. It is generally accepted that satisfactory bond strengths can be achieved between restorative materials and enamel, however bonding to dentine is less predictable.

The aim of this study was to investigate the effect of a water additive on the bond strength of a composite restorative material to enamel and dentine specimens which have undergone a range of surface treatments regimes. With regards to the preparation of the enamel and dentine specimens, the teeth were hand prepared by an individual experienced in specimen preparation. It is appreciated however that the properties of enamel and dentine vary with its position on the tooth and can also vary according to histological structure. Cuspal enamel is stronger than on the side of the tooth and is stronger under compression in a direction parallel to the rods than in a direction perpendicular to the rods. It is also appreciated that it is not practical or in fact possible, to take all of the enamel and dentine specimens from teeth from the same individual. It is therefore expected that there will be some variation in the results which can be attributed to the varying properties of different teeth.

The results of the study have shown that for those specimens in Group 2, which included the water additive 'Alpron' in the treatment regime, the tensile bond strength of composite resin bonded to enamel was increased significantly compared to the other treatment groups.

For those specimens treated with Xeno III, there was no significant difference in bond strength of enamel and dentine specimens bonded using Xeno III ($p>0.05$). However, the bond strength of dentine specimens bonded with Xeno III was significantly greater than other dentine specimens bonded with Prime&BondNT ($p< 0.05$).

The results of this study provide support to the manufacturer's claims that surface treatment of enamel and dentine with Xeno III provides improved adhesion to dentine. A possible explanation for this could be due to simultaneous etching and penetration by Xeno III, which contains two new monomers, Pyro-EMA and PEM-F that become active when exposed to water. Pyro-EMA forms free phosphoric acid groups and demineralises the tooth surface. PEM-F enhances the etching effect of Pyro-EMA, and acts as a strong cross-linking monomer, which helps contribute to the cohesive strength of Xeno III (Dentsply, Xeno III Information Sheet). Thereby, increasing micromechanical retention.

The ability for Xeno III to exhibit increased bonding strengths to dentine may also be aided by the presence of a smear layer found on the surface of dentine that is generated during cavity preparation. It has been suggested that the smear layer contains hydroxyapatite crystals and since Xeno III penetrates and dissolves the smear layer, calcium ions may be released from these crystals. These ions are possibly mopped up by PEM-F. The free phosphoric acid groups formed from the hydrolysed Pyro-EMA are then able to penetrate further into the dentine tubules. This may then mean that longer resin tags can be formed resulting in a thicker hybrid zone, which equates to better bonding strengths.

The controls used in this current study clearly demonstrated that the tensile bond strength of composite resin is significantly weaker to dentine than enamel. This is well documented and is due to many factors including its complex histological structure, the constant outward hydrostatic pressure from the dentinal tubules and the presence of a smear layer [13].

An important finding in the results of this study are that specimens treated with Xeno III demonstrated a greater tensile bond strength to enamel and dentine than those specimens subjected to the traditional method of etching and bonding. It is possible therefore that Xeno III may be valuable in cases where large amounts of dentine are exposed, such as in patients with moderate tooth wear resulting from attrition or erosion. The elimination of the need to wash the preparation may also be of use in the cementation of veneers or resin-retained bridges where moisture control is critical for its retention in the mouth [14,15].

CONCLUSIONS

The following conclusions can be drawn from the results of the study:

When using traditional etching methods, the tensile bond strength of composite resin restorative material is stronger to enamel, than dentine tooth surfaces. For those groups of specimens which included the water additive 'Alpron' in the regime, the tensile bond strength of the composite resin restorative material to enamel and dentine was greater than for those non water additive groups. The tensile bond strength of composite resin bonded to enamel surfaces prepared with 'Alpron' was significantly higher than that bonded to dentine surfaces. Those dentine specimens treated with Xeno III had a greater tensile bond strength of composite resin to dentine, when compared to those specimens subjected to traditional etching techniques. There was no significant difference between the tensile bond strength for enamel and dentine specimens surface treated with with Xeno III.

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