

# **Effect of Tooth Bleaching on the Shear Bond Strength of a Fluoride-Releasing Sealant**

by

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# 1 INTRODUCTION

An attractive smile has been a driving force for patients to seek for orthodontic treatment in the recent years. This does not only pertain to better tooth alignment, but also to better color of their teeth. Home bleaching techniques have been the most marketed cosmetic systems that have been quite accessible to patients since their development. High concentration in-office bleaching products are offered at dental office and kiosks in shopping malls. At-home bleaching products, on the other hand, are sold over the counter and patients regulate the frequency of their use. Among the questions that are of interest to orthodontics is their potential effect on orthodontic bonding.

Demands of more effective and faster results in bleaching have driven manufactures to develop more potent products with more aggressive protocols. In 1997, Kugel suggested the use of the combination of in-office bleaching and an at-home touch up bleaching technique. Since then, this protocol has become more popular in many dental offices. Adult patients who are concerned with the esthetics of the color of their teeth, often also seek for better alignment of their teeth. With highly commercialized and easy access to whitening products, it is quite likely that adult patients who seek for orthodontic treatments may also have had bleaching done previously. Determination of how a combination of in-office bleaching and an at-home touch up protocol may affect the shear bond strength may be helpful to the clinician

Another area of concern to orthodontists is the often irreversible and unesthetic iatrogenic effect of orthodontic treatment, namely white spot formation. White spot lesions are frequently detected in a number of orthodontic patients. The prevalence is reported to be 13% to 75% (Gorelick, Geiger and Gwinnett, 1982; Mitchell, 1992) and highest in teens (Kukleva, Shetkova and Beev, 2002). This undesirable complication can develop quite rapidly and is most frequently located on the cervical and middle thirds of the buccal surfaces of the maxillary lateral incisors, mandibular canines, and first premolars (Årtun and Brobakken, 1986). A survey conducted by Derks, Kuijpers-

Jagtman and Frencken *et al* (2007) reported that 68% of the orthodontists considered it to be necessary to develop a practice guideline to prevent enamel demineralization and the formation of white spots. In recent years, the development of highly filled sealants that are capable of releasing fluoride has triggered some interest among orthodontists. In terms of preventive measures, it is also valuable to know if tooth bleaching will affect the bond strength of the fluoride-releasing sealant.



## 2 LITERATURE REVIEW

### 2.1 Evolution of fluoride delivery systems in orthodontics

Fluorides are widely known for their beneficial role in prevention of demineralization during orthodontic treatment. They significantly influence the rate of demineralization even at the level as low as 0.02 to 0.06ppm (Ten Cate, 1999). It is usually recommended for orthodontic patients to acquire fluorides in the form of toothpaste and/or mouth rinse daily. However, this delivery system depends heavily on the individual patient's compliance and is found to be quite ineffective (Geiger, Gorelick and Gwinnett, 1988). They assessed the patient's compliance in a preventive fluoride-rinse program and found that the degree of compliance was poor in half of their patients. In-office topical fluoride treatments have also been investigated and shown to be ineffective (O'Reilly and Featherstone, 1997). They reported that significant demineralization could develop within only 4 weeks while orthodontic patients may not be seen until 6-8 weeks later.

Frequent application of fluorides directly affects the rate of demineralization, and it is ineffective to rely on patient's compliance. Hence, a number of delivery systems that are able to release fluoride slowly and are independent of patient's compliance have been recommended. Fluoride-releasing adhesives (e.g. glass ionomer cements) were rigorously investigated for their efficacy in protecting enamel from demineralization, but studies reported mixed results (Mitchell, 1992; Turner, 1993; Bank, Burn A and O'Brien *et al* 1997; Marcusson Norevall, and Persson, 1997; Gaworski, Weinstein and Borislow *et al*, 1999; Millett, Nunn and Welbury *et al*, 1999; Gorton and Featherstone, 2003; Pascotto, Navarro and Filho *et al* 2004). Long-term clinical results of these adhesives have not often been successfully shown (Turner, 1993; Bank, Burn A and O'Brien *et al* 1997; Gaworski, Weinstein and Borislow *et al*, 1999; Millett, Nunn and Welbury *et al*, 1999; Gorton and Featherstone, 2003). Fluoride varnish was also investigated for topical use in orthodontic patients (Schmit, Staley and Wefel *et al*; 2002). However, they found in their *in-vitro* study that fluoride varnish does not prevent demineralization on enamel surfaces.

Unfilled and lightly filled sealants were also studied for their ability to prevent demineralization but clinically they have not demonstrated adequate success (Banks and Richmond, 1994; Wenderoth, Weinstein and Borislow, 1999). Sealants are known to bond poorly to smooth enamel surfaces and are easily worn away by mechanical (i.e. tooth brushing) and chemical (i.e. acid) means. Wenderoth, Weinstein and Borislow (1999) used a light-cured fluoride-releasing sealant bonded adjacent to the brackets of twenty patients to examine its effect on demineralization, but found no difference between the control and the experimental groups. Their results confirmed previous finding by Banks and Richmond (1994) and by other *in vitro* studies (Joseph, Rossouw and Basson, 1994; Frazier, Southard and Doster, 1996) on either unfilled or lightly filled sealants. A recent *in vitro* study by Hu and Featherstone (2005) on highly filled, light-cured sealant (Pro Seal, Reliance Orthodontic Products, Itasca, Ill) showed some promising results on this fluoride regimen. They evaluated the efficacy of applying Pro Seal sealant onto the buccal tooth surfaces of extracted human third molars to prevent demineralization and compared the results with those of other groups (e.g. the enamel surface untreated, surface etched, fluoride varnish and light-cured unfilled sealant). They found the demineralization in the Pro Seal group to be significantly less than other groups even in the presence of severe acid challenge. They also found that Pro Seal sealant was significantly resistant to toothbrush abrasion. Buren, Staley and Wefel *et al* (2008) also investigated Pro Seal's effectiveness in inhibition of enamel demineralization using polarized light microscopy. They found that Pro Seal reduced lesion depth by 97% compared with the controls. Soliman, Bishara and Wefel *et al* (2006) demonstrated in another *in vitro* study that Pro Seal was capable of releasing fluoride ions for 17 weeks and had the ability to be recharged with fluoride ions using a foaming solution of acidulated phosphate fluoride. Another advantage being a sealant is that Pro Seal forms a mechanical barrier between plaque and enamel surface under and around orthodontic brackets (Bishara, Oonsombat and Soliman *et al*, 2005). Without the need for patient compliance, Pro Seal sealant has showed to be a promising regimen to inhibit enamel demineralization.

### **2.1.1 Pro Seal Sealant and Its Effect on Shear Bond Strength**

Pro Seal sealant (Reliance Orthodontic Products, Itasca, Ill) is a fluoride-releasing light-cure filled enamel sealant that claims to protect enamel against demineralization during orthodontic treatment with fixed appliances.

According to the manufacturer, Pro Seal can withstand toothbrush abrasion when applied to the labial surface (Reliance Orthodontic Products Manual, 2007).

Shear bond strength of Pro Seal sealant has been investigated and reported in the literature. Bishara, Oonsombat and Soliman *et al* (2005) studied the effect of Pro Seal sealant on shear bond strength when used with Transbond XT adhesive and compared the results with Transbond XT primer plus Transbond XT adhesive, as the control on metal brackets precoated with APC II adhesive. They found no significant difference in shear strength between Pro Seal sealant ( $4.8 \pm 2.3$  MPa) and Transbond XT primer ( $4.9 \pm 2.1$  MPa) after half an hour. Paschos, Okuka and Ilie *et al* (2006) investigated the influence of Pro Seal on shear bond strength when used in addition to and in place of the bonding primer. They found that after 24 hours, Pro Seal showed no negative effect on shear-peel bond strength in either setting. Lowder, Foley and Banting (2008) investigated the use of Pro Seal in combination with four different adhesive systems with metal orthodontic brackets. According to their results when used with Light Bond ( $15.9 \pm 2.9$  MPa) and Transbond XT ( $13.1 \pm 2.1$  MPa), Pro Seal sealant produced significantly higher bond strength than the use with Blugloo ( $10.1 \pm 2.2$  MPa) and APC plus ( $11.3 \pm 2.2$  MPa). However, all four settings produced above 10 MPa in mean shear bond strength. However, these studies investigated the SBS of Pro Seal within 24 hours. Currently there is no published data reporting the long term evaluation of SBS of Pro Seal. Furthermore, the material's effectiveness on bleached enamel has not been demonstrated either.

## **2.2 Tooth Bleaching Agents**

Tooth whitening was first introduced in the late 1870s (Fasanaro, 1992). Early products involved the use of pyrozone 25% and electricity to treat endodontically treated discolored teeth or hypochloric acid to treat fluorosis.

Hydrogen peroxide was first used in 1930s to treat fluorosis staining in combination with ether and heat. Up until late 1980s, tooth whitening remained unpopular as high concentration of hydrogen peroxide was the main method in the treatment of endodontically related discoloration, tetracycline staining, and fluorosis. In 1989, Haywood and Heymann introduced the first home bleaching system using carbamide peroxide applied in a custom fitted tray. This system allows patients to regulate the frequency of their use at home.

Bleaching techniques are now available in many forms; in-office, at home, or special and regular toothpaste. The main ingredient is either carbamide peroxide or hydrogen peroxide. In-office bleaching is applied with high concentrations of hydrogen peroxide (25%- 35%) or carbamide peroxide (35%-45%), a heat source, and a rubber dam to protect the gingival tissues. At-home bleaching often employs a various concentration, from 5 to 30% of carbamide peroxide in a custom fitted tray that patients wear through the night and is usually indicated for the treatment of the superficial enamel. This system is most effective when there is mild yellow, orange, or light brown staining, or in the presence of mild fluorosis and enamel mottling.

Carbamide peroxide, in its undiluted form, can be as equivalently concentrated as 35% hydrogen peroxide. But at such concentrations, iatrogenic effects such as chronic inflammation, tooth hypersensitivity, and pre-neoplastic lesions may occur. The current home bleaching systems use diluted carbamide peroxide, which is equivalent to 30% to 35% in potency to that of hydrogen peroxide. In addition, carbamide peroxide contains carbopal which is an additive that thickens the bleaching material, improves adhesion, and prolongs the oxygen release of the peroxide (Bishara, Oonsombat and Soliman *et al*, 2005).

## **2.3 Tooth Bleaching in Orthodontics**

Tooth bleaching has been shown to have caused morphological change to tooth surfaces (Josey, Rossouw and Basson, 1996; Ptocnik, Kosec, and Gaspersic, 2000; Bishara, Oonsombat, Soliman *et al*, 2005). Josey, Rossouw and Basson (1996) found that bleaching caused the loss of prismatic form, with the enamel appearing etched. Another study investigating the subenamel layer after application of 10% of carbamide peroxide using a scanning

electron microscopy by Potocnik, Kosec, and Gaspersic (2000) revealed local changes in the enamel structure similar to those observed with initial caries. Furthermore, calcium-to-phosphorus ratio decreased. However, these changes were not considered clinically significant. Bishara, Oonsombat, and Soliman *et al* (2005) investigated the effects of in-office and at-home bleaching on the enamel surface. They found that the enamel surface appeared mildly etched after bleaching in both techniques. After the bleached enamel was etched with 37% phosphoric acid they showed that the honeycomb pattern was irregular in both procedures. This effect was more apparent with in-office bleaching than at-home bleaching regimen.

Changes in the enamel surface may result in variation in shear bond strength of resin material to the enamel surface, which can be significant during clinical procedures that involve the bonding orthodontic brackets. Previous studies have shown a reduction in shear bond strength of adhesive resin after the teeth were bleached (Miles, Pontier and Hahiraei, 1994; Bulut and Turkan and Demirbaş, 2006). Miles, Pontier and Bahiraei (1994) reported in their *in vitro* study that the immediate tensile bond strength after 72 hours application of home bleach agent on human premolars ranged from 0 to 13 MPa, while the tensile bond strengths of the control group ranged from 5 to 15 MPa. Bulut, Turkun and Demirbaş (2006) showed in their *in vitro* study that the premolar treated with 10% carbamide peroxide for one week exhibited a lower shear bond strength ( $14.2 \pm 2.4$  MPa) 24 hours after bonding when compared to the control group ( $20.6 \pm 2.9$  MPa). On the contrary, Oztas, Bağdelen and Kılıçoğlu *et al* (2011) found no significant difference between bleached and unbleached enamel with respect to composite bond strength. They reported that 24-hour shear bond strengths of the home bleached groups (24 hours and 14 days period waiting prior to bonding after completion of bleaching sessions) on human premolar exhibited comparable shear bond strengths ( $14.552 \pm 4.622$  MPa and  $13.359 \pm 2.845$  MPa respectively) to that of the control groups ( $12.459 \pm 2.648$  MPa).

The amount of residual oxygen on the bleached enamel surface was reported to have a negative influence on polymerization of the bonding agent (Spyrides, Perdigao and Pagani *et al*, 2000; Lai, Tay and Cheung *et al*; 2002). This often results in a decrease in the number of resin tags, thereby lowering the bond strength. Several methods

were suggested to reduce the residual oxygen in the surface of bleached enamel. Sung, Chan and Mito *et al* (1999) recommended the use of alcohol-based bonding agents. Oztas, Bağdelen and Kılıçoğlu *et al* (2006) suggested a thorough water rinse, then air compression for 30 seconds after every bleaching procedure. Cvitko, Denehy and Swift *et al* (1991) recommended the removal of the superficial layer of enamel. Lai, Tay and Cheung (2002) and Kaya, Türkün and Arici (2008) reported reversal of compromised bonding in bleached enamel by using antioxidant gels right after bleaching for 60 minutes. Other authors suggested delaying bonding orthodontic brackets until two weeks after bleaching (Spyrides, Perdigao and Pagani *et al*, 2000; Cavalli, Reis and Giannini *et al*, 2001; Bishara, Oonsombat and Soliman *et al*, 2005). These studies all used carbamide peroxide as their bleaching agents with at-home technique.

In 1997, Kugrel recommended the simultaneous use of at-home and in-office systems to bleach teeth, because the combination increased the effectiveness of tooth whitening. The surge of popularity of this technique has driven manufacturers to further develop more potent and higher concentrated products. Currently there is no study that reports the effect of this technique on shear bond strength.

## **2.4 Bond Strength Testing in Orthodontics**

Bracket bond failure is one of the most frustrating incidents within any orthodontic practice, resulting in increased treatment time, additional costs in materials and increased visits by the patient. Knowing where the bond failure has occurred can assist orthodontists in modifying their bonding technique and in counseling the patient on care of their appliances. Because the location of the bond failure may indicate the probable cause, it is important to understand the significance of "bond strength" in a clinical application. The tensile and shear bond tests are the most commonly used *in vitro* tests to evaluate the adhesion between the bracket and enamel surface in orthodontics (Brantley and Eliades, 2001).

### **2.4.1 Shear Testing**

Of all the adhesive tests used in dentistry, the shear bond strength (SBS) has been one of the most popular bonding tests ever devised due to its simple design. In clinical shear testing, the bonded bracket is loaded by a blade in tension or compression or by a wire loop in tension, so that the bracket slides parallel to the enamel surface (Brantley and Eliades, 2001).

Pure shear loading is difficult to achieve, and most shear testing also includes components of peeling, tension, and torsion because the experimental design of the specimen and the manner of loading are such that stresses experienced at the adhesive interface are extremely complex (Wiltshire and Noble, 2010). Bond failure is more likely due to the interfacial tensile stresses generated as a consequence of the bending action rather than genuine shear. The shear bond strength test can be problematic when the adhesive bond strength is too high because the fracture may involve enamel or dentin. The reason for this is that the stresses generated by the bending action will predispose any crack that forms to deviate into the dentin or enamel when confronted with a strong adhesive bond (Brantley and Eliades, 2001).

### **2.4.2 Tensile Testing**

The concept of tensile testing is analogous to using a dumbbell specimen design. The idea behind this design is that all measurements are taken in the central part of the specimen, well away from the clamping site, such that a uniform stress field is generated and the local tensile stress can be calculated simply from the load divided by the cross-sectional area. In clinical tensile testing, the bracket is pulled perpendicularly from the enamel substrate (Brantley and Eliades, 2001).

### **2.4.3 Testing Machine**

There are two types of machines that are used to test shear and tensile strength of orthodontic materials. These machines are classified as screw-driven (have a large screw located at each end of the crosshead) or servo-hydraulic (use the pressure of oil pumped into a hydraulic piston to move the crosshead). These machines are often referred to as universal testing machines because they can be used to test tension, compression, torsion or bending (Brantley and Eliades, 2001).

### **2.4.4 Debonding Force and Bond Strength**

Debonding force is determined from the load drop on the mechanical machine and reported in units of newtons (N), kilogram (kg), or pounds (lb). Bond strength is defined as the force of debonding divided by the area of the bonded interface measured in units of megapascals (MPa), kilograms per square centimeter (kg/cm<sup>2</sup>), pounds per square inch (lb/in<sup>2</sup> or psi) (Wiltshire and Noble, 2010).

### **2.4.5 Optimum Bonding Strength**

It is difficult to estimate the optimal bond strength of an adhesive in the oral environment. This is because the orthodontic brackets are subjected to masticatory forces which are often a mixture of shear, peel, shear-peel and tensile force (Wiltshire and Noble, 2010). This is complicated by differential forces exerted in different regions in the mouth. The average masticatory force on the anterior brackets is about 5MPa whereas it is 20MPa on the posterior brackets. This is further complicated by individual variations in masticatory forces, teeth morphology and oral habits. Reynolds (1975) recommended 6 to 8 MPa as reasonable bond strength for orthodontic purposes. However, this suggestion was made over 35 years ago based on a bracket base area of 16mm<sup>2</sup>. Since then, there have been recent advances in bracket materials and design, adhesives, computer technology and testing systems. Rather than focusing on an arbitrary numerical “clinical acceptable bond strength”, Wiltshire and Noble (2010) suggested to pay more attention to potential damage to the enamel, especially when the bond strength is too



high. They recommended a mean bond strength of at least 3 - 4MPa *in vitro* for minimal reliable clinical bonding. When analyzing bond strengths studies, it is also important to consider the range of values and the coefficients of variation (standard deviation/mean x100), which is preferably less than 20-30%.

#### **2.4.6 Bond Strength Comparison from Different Studies**

Caution must be applied when comparing shear bond strength data from experiments carried out in different laboratories. Minor modifications in the method used can potentially have a profound influence on the outcome of the experiment. This problem is exacerbated by other variables that can come into play when bonding to enamel/dentine (Al-Salehi and Burke, 1997; Tay and Pashley, 2004). It is reasonable to compare the adhesive performance of different bonding agents within the same laboratory. Comparison of data from different laboratories is fraught with danger, unless one can be sure that exactly the same experimental conditions were used.

Bond testing using teeth involves many variables that can affect the measured bond strength. According to Stanford *et al* (1997), these variables may include the type of tooth (e.g incisor, molar, human, bovine); fluoride content of tooth; disinfection and storage media of tooth before bonding; elapsed time of storage following debonding; type of loading (shear, tension, peel, or torsion); configuration of specimen testing jig; crosshead speed of mechanical testing machine; bonding area of the bracket. At present, there is no standard methodology for the evaluation of bond strength to tooth enamel or other surfaces for orthodontic adhesives. Stanford *et al* (1997) suggested that data from different studies must be interpreted carefully and one must consider other factors.

### **3. PURPOSE**

The purpose of this study was to investigate the effects of in-office plus at-home touch up bleaching technique on the shear bond strength of Transbond XT adhesive to enamel surface with or without Pro Seal sealant using curved stainless steel lingual buttons.

#### **4. NULL HYPOTHESES**

1. There is no difference between the mean shear bond strength of Transbond XT primer and Pro Seal sealant applied to either bleached or non-bleached enamel over time.
2. There is no difference between Adhesive Remnant Index (ARI) scores between Transbond XT primer and Pro Seal sealant applied to either bleached or non-bleached enamel over time.

## 5. MATERIALS AND METHODS

### 5.1 Materials used in the study

#### 5.1.1 Transbond XT Light Cure Adhesive System

This system (Figure 5.1) is composed of primer, adhesive paste, and etching gel (3M Unitek, Monrovia, CA). The adhesive paste is a composite resin and contains 10-20% wt Bisphenol A diglycidylether methacrylate, 5-10% wt Bisphenol A bis (2-hydroxyethyl ether) dimethacrylate (bis-EMA), 70-80% wt silane treated quartz and less than 2% silane treated silica. The primer is an unfilled light cured resin and is made of Bis-GMA and Triethylene glycol dimethacrylate (TEGDMA) in a 1:1 ratio, and the photoinitiator. The etching gel (Figure 5.2) is composed of 35% phosphoric acid in water and amorphous silica (Material and Safety Data Sheet: Transbond XT Light Cure Adhesive, 2008).

**Figure 5.1: Transbond XT Light Cure Adhesive System**



**Figure 5.2: Transbond XT etching gel 35% phosphoric acid**



### 5.1.2 Pro Seal Sealant

Pro Seal sealant (Reliance Orthodontic Products, Itasca, Ill) contains ethoxylated bisphenol A diacrylate (10–50%), urethane acrylate ester (10–40%), and polyethyleneglycol diacrylate (10–40%). The exact percentages of both products are a trade secret (Bishara *et al*, 2005).

**Figure 5.3: Pro Seal Sealant L.E.D**



### 5.1.3 Bleaching Agents

Opalescence® Quick 45 PF (Figure 5.4) is an in-office bleaching system. It is a clear, high-viscosity, sticky, 45% carbamide peroxide gel with pH ~6.5. It also contains potassium nitrate and fluoride (Opalescence® Quick Material Manual, 2010).

**Figure 5.4: Opalescence Quick 45 PF**



Opalescence® Quick 20 PF (Figure 5.5) is an at-home bleaching system. It is a clear, high-viscosity sticky, 20% carbamide peroxide gel with pH~6.5. It also contains potassium nitrate and fluoride (Opalescence® Quick Material Manual, 2010).

**Figure 5.5: Opalescence Quick 20 PF**



#### **5.1.4 Artificial Saliva**

The composition of the artificial saliva was [g/L]: Methyl-p-hydroxybenzoate, 2.00; sodium carboxymethyl cellulose, 10.0; KCl, 0.625;  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ , 0.059;  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ , 0.166;  $\text{K}_2\text{HPO}_4$ , 0.804;  $\text{KH}_2\text{PO}_4$ , 0.326, pH of 6.75) (McKnight-Hanes and Whitford, 1992).

#### **5.1.5 Orthodontic Buttons**

One hundred and sixty curved stainless steel lingual buttons (#30-000-01, GAC International, Central Islip, NY) were used with the diameter of 3.33mm (surface area of approximately  $8.70 \text{ mm}^2$ ).

**Table 5.1 Shear Bond Material Manufacturer and Batch Number**

Material	Manufacturer	Reference Number	Lot
<b>Bonding Agents</b>			
Transbond XT	3M Unitek, Monrovia, California	712-030	BL/8EX
Adhesive paste		712-031	
Primer		712-039	
35% etching gel		712-039 9802	8KY
LED Pro Seal sealant	Reliance Orthodontic Products, Itasca, Ill		SUK
<b>Bleaching Materials</b>			
45% PF Carbamide Peroxide	Opalescence Quick, Ultradent, Utah	REF/UP 5346	
25% PF Carbamide Peroxide			
<b>Bonding Materials</b>			
Curved stainless steel lingual buttons	GAC International, Central Islip, NY	30-000-01	A597
Loading apparatus gauge	Federal: Miracle Movement 0.001" C81S, Providence, RI		
Diamond saw	Buehler, Lake Bluff, Ill		
Cooper Rings			
Mini LED Blue Ray - Light curing unit	American Orthodontist		
Pumice Preppies	Whip Mix, Louisville, KY		
Bosworth Fastray	Bosworth, Illinois	0921375	
Monomer liquid			C13504
Polymer powder			C14783
<b>Debonding Materials</b>			
Universal Testing Machine	Zwick GmbH, Ulm, Germany		

Bencor Multi-T testing apparatus	Danville Engineering, San Ramon, CA		
Light Microscope	Nikon SMZ800		
Canon XTi digital camera	Canon XTi 10 megapixels		
<b>Chemicals</b>			
Chloramine-T trihydrate 98%	Acros Organics, New Jersey		A0236347
<b>Other</b>			
Digital Caliper	Mastercraft		
Laser Level	Stanley FatMax Level, 24-in	57-5344-8	
Incubator 37°C	Thelco/Canlab Model 2, Precision Scientific, Chicago, IL		

## 5.2 Experimental Method

IRB approval for this study was obtained through the Bannatyne campus research ethics board of the University of Manitoba (Appendix).

One hundred and sixty extracted intact human molars were used in this study. Half of these teeth were first bleached with 45% of carbamide peroxide gel (Opalescence Quick, Ultradent, Utah) applied on their buccal surfaces on the first application for half an hour, followed with five applications of 6 hours of 20% carbamide peroxide gel in 24 hours interval, then stored in artificial saliva at 37°C in incubator for two weeks before bonding.

Group 1 (Control) comprised of forty unbleached teeth that were bonded with lingual buttons (GAC International, Central Islip, NY) using Transbond XT primer and adhesive (3M Unitek, California).



Group 2 had forty unbleached teeth that were bonded with lingual buttons using Pro Seal sealant (Reliance Orthodontic Product Inc, Ill) and Transbond XT adhesive.

Group 3 included forty bleached teeth that were bonded with lingual buttons using Transbond XT primer and adhesive.

Group 4 comprised of forty bleached teeth that were bonded with lingual buttons using Pro Seal sealant and Transbond XT adhesive.

The teeth were stored in artificial saliva in a covered dish at 37°C in an incubator before the tests. The shear bond strength tests were performed at two different time intervals; 24 hours and 3 months. Table 5-2 summarizes the experimental procedure.

**Table 5-2: Summary of the study**

Enamel Surface Treatment	Groups	Bonding Systems with Transbond XT Adhesive	Subgroups	Test Intervals	Number of Teeth
Etched Unbleached Teeth	1	Transbond XT primer	1a	24hrs	20
			1b	3 months	20
	2	Pro Seal	2a	24hrs	20
			2b	3 months	20
Etched Bleached Teeth	3	Transbond XT primer	3a	24hrs	20
			3b	3 months	20
	4	Pro Seal	4a	24hrs	20
			4b	3 months	20
				<b>TOTAL</b>	<b>160</b>

### **5.2.1 Tooth Collection and Storage**

The teeth were collected from four maxillofacial and oral surgery clinics in the city of Winnipeg and were stored in 0.5% Chloramine T solution. The criteria for tooth selection included intact buccal enamel, not being subjected to pretreatment agents, no cracks, and no caries. The teeth selected for this study were also inspected by the primary author carefully to only include the teeth presenting with similar surface flatness.

### **5.2.2 Tooth preparation**

The teeth were rinsed with tap water, and their roots were removed with a water-cooled diamond saw. The teeth were randomly divided into two groups. The first group were subjected to bleaching. The second group were stored in artificial saliva in incubator at 37°C until the bonding procedure. The artificial saliva was refreshed daily.

### **5.2.3 Bleaching Procedure**

The bleaching protocol used in this study involved a combination of one in-office plus five take home sessions procedure using Opalescence Quick PF (Ultradent, Utah). The manufacturer's recommendations were followed: 45% Carbamide peroxide gel was applied for 30 minutes followed by five 6 hours of 20% Carbamide peroxide gel applications in 24-h intervals.

The teeth in the bleached group were polished with non-fluoridated pumice and rubber prophylactic cups for 10 seconds, rinsed with water and then air dried. Carbamide peroxide bleaching gel (45%) was applied to the buccal surfaces of the teeth directly from the syringe in a layer of approximately 1 mm thickness and the teeth were kept at 37°C and in a humid environment for 30 minutes. The bleaching gel was rubbed off and thoroughly rinsed with water, then stored in artificial saliva at 37°C in the incubator.

Twenty-four hours after the first application, 20% carbamide peroxide bleaching gel was applied on the buccal surfaces from the syringe in a layer of approximately 1 mm thickness. The teeth were kept at 37°C and in 100% humidity for 6 hours. The bleaching gel was rubbed off and thoroughly rinsed with water. The teeth were then stored in artificial saliva at 37°C in the incubator. This procedure was repeated 4 more times in 24-h intervals. After the last bleaching session, the bleached teeth were stored in artificial saliva at 37°C in the incubator for two weeks. The artificial saliva was changed daily.

#### **5.2.4 Bonding Procedure**

Two weeks after the completion of bleaching procedure in the experimental group, bleached and unbleached teeth were randomly divided into two groups.

*Group 1 (Control)* - Forty unbleached teeth were bonded with Transbond XT primer and adhesive (3M Unitek, Monrovia, California). Forty unbleached teeth were etched with Transbond XT 37% phosphoric acid gel (3M Unitek, Monrovia, California) for 30 seconds. The teeth were then rinsed with water spray for 20 seconds and dried with an oil-free air spray for 20 seconds until the enamel appeared frosty. Transbond XT primer was applied on the enamel surface and light-cured for 20 seconds. The metal lingual buttons were then applied with Transbond XT adhesive paste placed on the etched enamel surface. A light finger pressure was applied by a bracket holder pushing on the buttons until the buttons touched the surface of the enamel. Excess adhesive was removed with a scaler and the samples were cured for 40 seconds with Ortholux LED light.

*Group 2-* forty unbleached teeth were bonded with Pro Seal sealant (Reliance Orthodontic Product Inc, Itasca, Ill) and Transbond XT adhesive (3M Unitek, Monrovia, California). Forty unbleached teeth were etched with 37% phosphoric acid gel for 30 seconds. The teeth were then rinsed with water spray for 20 seconds and dried with an oil-free air spray for 20 seconds until the enamel appears frosty. Pro Seal sealant was applied on the enamel

surface and light-cured for 20 seconds. The metal lingual buttons applied with Transbond XT adhesive paste were placed on the etched enamel surface. A light pressure was applied by a bracket holder on the buttons until the buttons touched the surface of the enamel. Excess adhesive was removed and the samples were cured for 40 seconds.

*Group 3* – forty bleached teeth bonded with Transbond XT primer and adhesive (3M Unitek, Monrovia, California). Forty bleached teeth were bonded with Transbond XT primer with the same protocol as in Group 1.

*Group 4* – forty bleached teeth were bonded with Pro Seal sealant (Reliance Orthodontic Product Inc, Itasca, Ill) and Transbond XT adhesive (3M Unitek, Monrovia, California). Forty bleached teeth were bonded with Pro Seal with the same protocol as in Group 2.

The teeth were stored immediately upon the completion of the bonding procedure in artificial saliva at 37°C in an incubator.

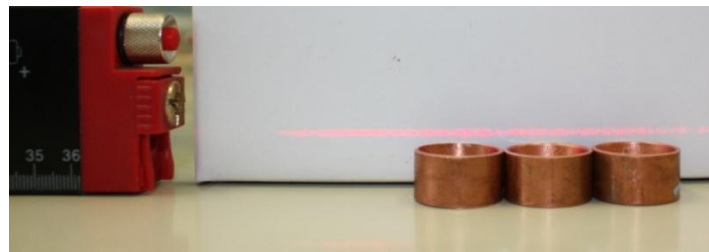
### **5.2.5 Debonding Procedure**

Each group was randomly divided into two subgroups. First subgroup of each group was tested 24 hours after bonding, and the latter was stored in artificial saliva and tested three months after bonding.

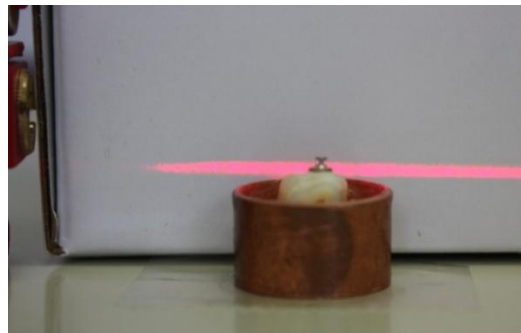
Twenty four hours after the bonding procedure, the teeth were embedded with Bosworth fastray acrylic in cooper rings of 22.5 mm in diameter. The cooper rings were applied with Vaseline prior to acrylic placement. The laser level was positioned on a flat table projecting a horizontal light on a white box. Each pre-vaselined ring was placed parallel to the horizontal laser beam. Each tooth was held inside each ring with sticky wax so that a parallel relationship with the base of the button and the laser beam was achieved (Figure 5.6 and Figure 5.7). Bosworth

fastray acrylic was mixed according to the manufacturer's recommendation and was poured into the rings. After ten minutes, the embedded teeth were removed and were mounted in the Universal testing machine in the Bencor Multi-T Loading Apparatus (Figure 5.8 and Figure 5.9). A direct sharp shearing blade was loaded to the enamel-adhesive-bracket interface parallel to the height of contour in an occluso-gingival direction (Figure 5.10). The speed of the crosshead was set at 0.5 mm/min and the shear bond strength was measured using 1KN load cell. The shear bond strength values were recorded by the computer. The same procedure was repeated for the samples in the three month period.

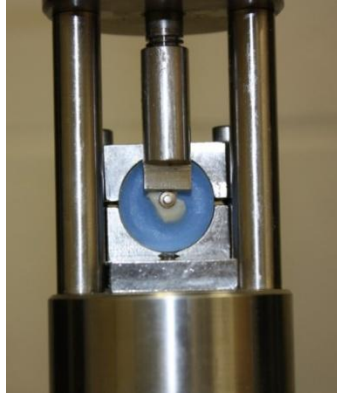
**Figure 5.6: Laser Level with Copper Rings Set Up**



**Figure 5.7: Cut Tooth Held by Wax in Copper Ring before Acrylic Placement**



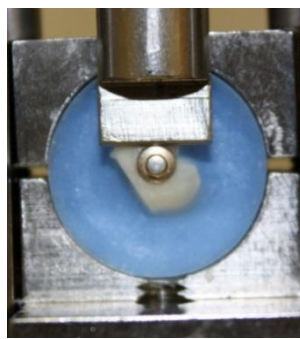
**Figure 5.8: Bonded Tooth Embedded in Acrylic Mounted in Universal Testing Machine**



**Figure 5.9: Universal Testing Machine**



**Figure 5.10: Bencor Multi-T Loading Apparatus**



### **5.2.6 Adhesive Remnant Index**

Evaluation of bond-failure site was carried out with a protocol similar to the one used previously by Årtun and Bergland (1984). The debonded surface of each tooth was examined under a light-microscope with 10x magnification and a score was given to each tooth. Three months later, half of the samples were randomly selected from each group and the adhesive remnant index scores were re-evaluated by the same operator.

#### **Adhesive Remnant Index (ARI) score (1-5):**

- 1 – 100% left on tooth + bracket impression
- 2 – >90% left on tooth
- 3 – 10-90% left on tooth
- 4 – <10% left on tooth
- 5 – 0% left on tooth

### **5.2.7 Statistical Analysis of Data**

Descriptive statistics, including the mean, standard deviation, and minimum and maximum values were calculated for the eight groups of teeth tested. Comparisons of means of shear bond strength values were made with an analysis of variance (ANOVA) test. If significant differences were present, simple t-tests were performed to determine which means were significantly different from each other. The Fisher's Exact Test was used to determine significant differences in the ARI scores among the eight groups. The significance used for all the tests was predetermined at a probability value of 0.05 or less.

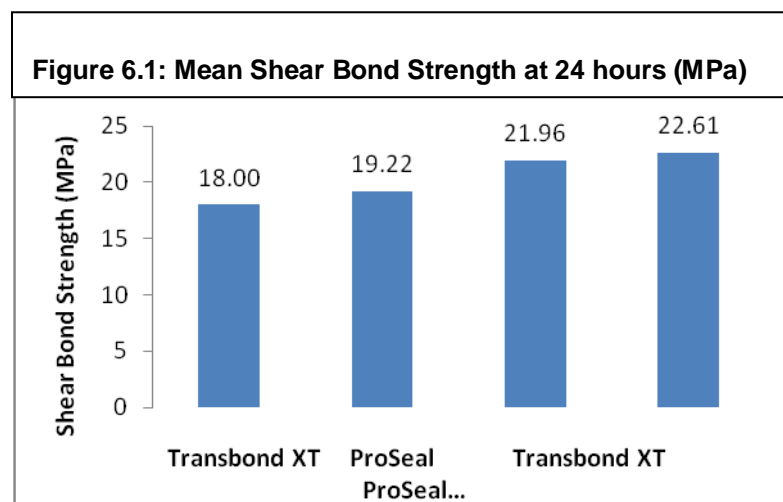
## 6. RESULTS

### 6.1 Shear Bond Strength at 24 hours

In the unbleached groups, the mean shear bond strengths were  $18.00 \pm 4.14$  MPa and  $19.22 \pm 3.43$  MPa for the Transbond XT primer and Pro Seal, respectively. The mean shear bond strengths in the bleached groups were relatively higher;  $21.96 \pm 2.87$  MPa and  $22.61 \pm 4.42$  MPa for Transbond XT primer and Pro Seal, respectively. The ANOVA test indicated a significant difference between the four groups ( $p < 0.0011$ ). Further simple t-tests indicated significant differences for any possible comparison between the subgroups of bleached and unbleached groups. However, no significant difference was observed between Transbond XT primer and Pro Seal in either bleached or unbleached groups.

**Table 6.1: Descriptive Data of Shear Bond Strength at 24 hours**

Groups		Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Range (MPa)	Coefficient of Variation
Unbleached	Transbond XT Primer	20	18.00	4.14	10.13	28.95	18.82	23.00%
	Pro Seal	20	19.22	3.43	13.85	25.25	11.4	17.85%
Bleached	Transbond XT Primer	20	21.96	2.86	16.81	26.71	9.9	13.02%
	Pro Seal	20	22.61	4.42	15.68	32.16	16.48	19.54%



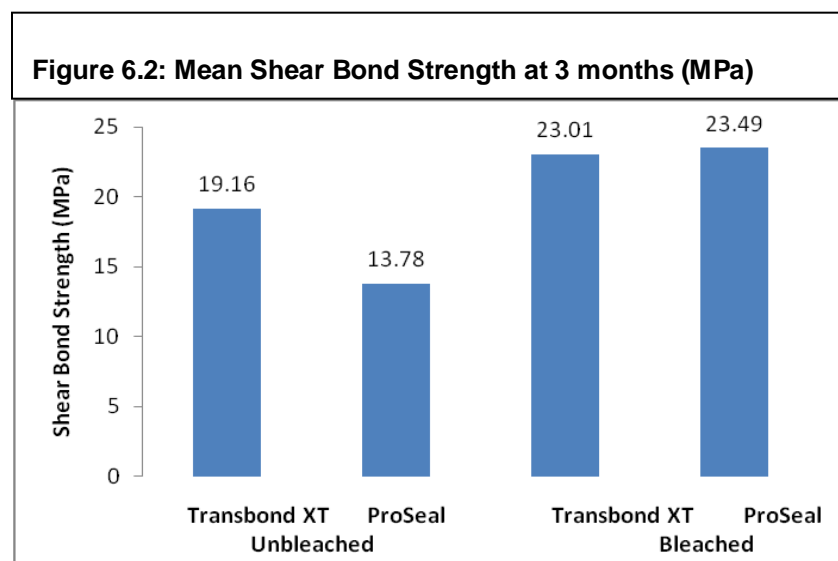


## 6.2 Shear Bond Strength at 3 months

The Transbond XT primer in the unbleached group maintained its shear bond strength after three months ( $19.16 \pm 4.58$  MPa) while the Pro Seal in this group showed a decreased mean of SBS ( $13.78 \pm 4.23$  MPa) compared to the 24-h period. In the bleached group both Transbond XT primer ( $23.01 \pm 5.32$  MPa) and Pro Seal ( $23.49 \pm 4.41$  MPa) maintained their shear bond strength. The ANOVA analysis showed that there is a significant difference between these four groups. The comparison of Pro Seal and the Transbond XT primer in the unbleached group yielded a significant difference ( $P < 0.000428$ ). However, there was no significant difference between the Transbond XT and the Pro Seal in bleached group. The comparison of Transbond XT in the unbleached group to both subgroups of the bleached groups also showed a significant difference ( $p < 0.018797$ ).

**Table 6.2: Descriptive Data of Shear Bond Strength at 3 months**

Mode	Groups	Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Range (MPa)	Coefficient of Variation
Unbleached	Transbond XT Primer	20	19.16	4.58	10.78	26.16	15.38	23.90%
	Pro Seal	20	13.78	4.23	7.16	21.24	14.08	30.69%
Bleached	Transbond XT Primer	20	23.01	5.32	9.04	32.71	23.67	23.12%
	Pro Seal	20	23.49	4.41	13.11	28.74	15.39	18.77%



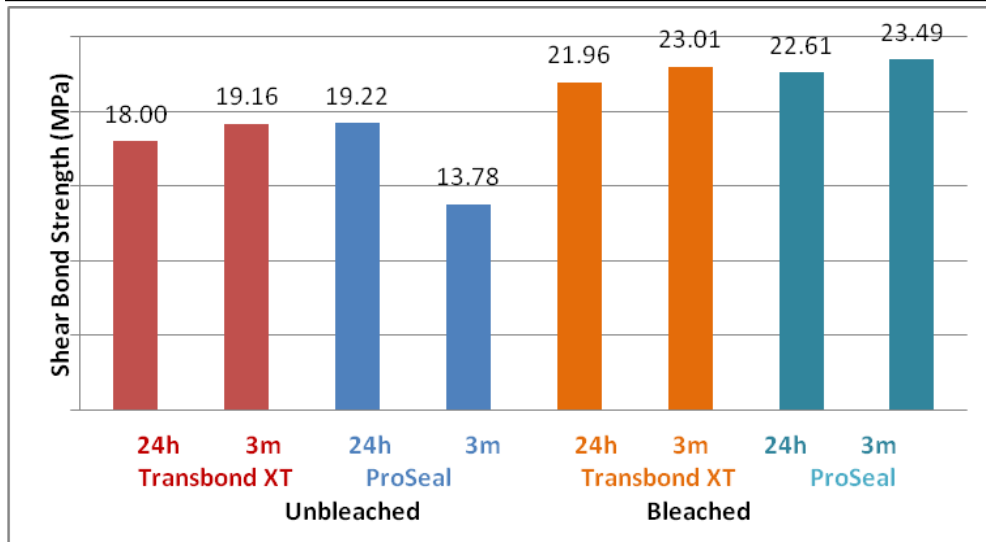
### 6.3 Shear Bond Strength after 3 months

A summary of changes in shear bond strength over 3 months is presented in Table 6.3. Analysis of variance showed there was a significant difference among the groups ( $p < 0.0001$ ). The mean shear bond strength of the Pro Seal sealant group of the unbleached teeth was significantly lower than the other groups. When comparing the mean SBS of each group from the 24-hour interval to correspondent one in the three month interval, the only group that showed a significant difference was the Pro Seal in the unbleached group ( $p < 0.0001$ ). The mean shear bond strength after three months was significantly lower than the one in 24-hour period. The general trend for the other groups was that the shear bond strength slightly increased over three months period, although the difference was not significant ( $p > 0.05$ ).

**Table 6.3: Descriptive data of shear bond strength over 3 months**

Storage Interval	Groups		Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Range (MPa)	Coefficient of Variation
24 hours	Unbleached	Transbond XT Primer	20	18.00	4.14	10.13	28.95	18.82	23.00%
		Pro Seal	20	19.22	3.43	13.85	25.25	11.4	17.85%
	Bleached	Transbond XT Primer	20	21.96	2.86	16.81	26.71	9.9	13.02%
		Pro Seal	20	22.61	4.42	15.68	32.16	16.48	19.54%
3 months	Unbleached	Transbond XT Primer	20	19.16	4.58	10.78	26.16	15.38	23.90%
		Pro Seal	20	13.78	4.23	7.16	21.24	14.08	30.69%
	Bleached	Transbond XT Primer	20	23.01	5.32	9.04	32.71	23.67	23.12%
		Pro Seal	20	23.49	4.41	13.11	28.74	15.39	18.77%

**Figure 6.3: An Overview of Mean Shear Bond Strength over 3 months (MPa)**



## 6.4 Adhesive Remnant Index

**Table 6.4: Frequency of ARI Scores over 3 months**

Storage Interval	Mode	Bonding Agent	ARI Score					Total
			1	2	3	4	5	
24 hours	Unbleached	Transbond XT primer	5	5	8	1	1	20
		Pro Seal	6	8	6	0	0	20
	Bleached	Transbond XT primer	1	4	14	1	0	20
		Pro Seal	6	7	7	0	0	20
3 months	Unbleached	Transbond XT primer	7	4	7	2	0	20
		Pro Seal	6	9	5	0	0	20
	Bleached	Transbond XT primer	1	4	11	3	1	20
		Pro Seal	3	2	13	1	1	20

### Adhesive Remnant Index Scores:

- 1 - 100% left on tooth
- 2 - > 90% left on tooth

3 - 10-90% left on tooth

4 - < 10% left on tooth

5 - 0% left on tooth

(Årtun and Bergland, 1984)

In orthodontics, low adhesive remnant index (ARI) scores are desirable. The more adhesive remnants remaining on the surface of the tooth, the lower the chance that the enamel is damaged during debonding procedure. It is therefore bond failure at bracket-adhesive interface is favourable for patients.

In the unbleached group, the Transbond XT primer group exhibited about 25% of the teeth with the score of 1 at 24 hours. This number increased to 35% at 3 months, while the Pro Seal group maintained the score of 1 at 30% from 24 hours to 3 months. Overall, at 24-hour test, 50% and of the teeth in the Transbond XT primer group and 70% in the Pro Seal sealant group left more than 90% of the adhesive on the enamel surface. While at 3-month test, 55% of the teeth in the Transbond XT primer group and 75% in the Pro Seal group exhibited more than 90% of the adhesive on the enamel surface.

In the bleached group, Transbond XT primer only had about 5% of the teeth with the score of 1 at both 24-hour and 3 month periods. On the other hand, Pro Seal sealant dropped from 30% to 15% of the teeth with the score of 1, from 24 hours to 3 months. At 24-hour interval, 65% of the teeth in Pro Seal sealant group left more than 90% of the adhesive on the enamel surface, while only 25% of those in the Transbond XT primer. At 3-months interval, only 25% of the teeth in both the Transbond XT primer and the Pro Seal sealant group exhibited 90% of the adhesive left on the enamel surface.

Overall, the majority of the bond failures in both unbleached and bleached groups were adhesive/cohesive in nature as most of the ARI scores are 1, 2 and 3. When compared the ARI scores of all the groups using Fisher's Exact Test at 24-hour interval, there was no significant difference among them ( $p>0.05$ ). However, there was a

significant difference among the groups at 3-month interval ( $p < 0.0257$ ). The bleached teeth at 3-months had significantly less adhesive left on the enamel surface.

## 7. DISCUSSION

### 7.1 Effect of Bleaching on Shear Bond Strength

Opalescence Quick PF was the choice of bleaching protocol in this study because it is a newly developed product that contains potassium nitrate and fluoride. The manufacturer claims that with the inclusion of potassium nitrate and fluoride, tooth sensitivity would be reduced (Opalescence® Quick Material Manual, 2010). Thus achievement of desired results may be much faster. Traditional office bleaching protocol employs three applications of 30 minute treatments at one-week intervals due to the felt sensitivity. With the recent addition of potassium nitrate and fluoride, treatments do not have to be a week apart. Patients can complete the whole procedure in three consecutive days. However, sole in-office treatment increases chair time and the need for supervision. Moreover, three separate dental appointments may not be practical for either patients or clinicians. Therefore, a combination of one in-office session with multiple at-home touch-up treatments is often recommended. Manufacturer of Opalescence Quick recommends a 30-45 minutes use of 45% carbamide peroxide in-office followed by 5 to 8 sessions of 6-8 hours of 20% carbamide peroxide in a custom fitted tray at night to achieve an effective result. In this study, we simulated one in-office session of 30 minutes followed by five 6-hour at-home treatments.

It was interesting to note that the shear bond strength of the bleached groups were higher than those of the unbleached groups at both 24 hour and 3 months debonding intervals. A possible explanation for this may be that by delaying the bonding procedure two weeks after the bleaching protocol may possibly have eliminated most, if not all, of the residual oxygen, which in turn may have allowed normal polymerization of the resin tags. It was argued that early bonding following bleaching would result in polymerization inhibition of the composite due to the delayed release of oxygen (Dishman, Covey and Baughan 1994; Lai, Tay and Cheung *et al*, 2002). Moreover, the use of artificial saliva as the storage medium during and between bleaching sessions may have promoted remineralization due to calcium and phosphate ions content (Basting, Rodriguez and Serra *et al*, 2003). Additionally, after treating with phosphoric acid etching, the bleached enamel surface might have been more porous or “overetched” than normally etched enamel (Josey, Meyers, and Romaniuk *et al*, 1996; Bishara,

Oonsombat , and Soliman *et al*, 2005). A combination of these overall effects might have consequently increased the number of resin tags and translated into better shear bond strength. Bishara, Oonsombat, and Soliman *et al* (2005) stated that waiting one week before bonding resulted in similar shear bond strength with the control group. However, the mean shear bond strength of those after two weeks waiting prior to bonding was higher than both the control and one week interval groups. This observation was true for both in-office and at-home protocols used in their study. Similar finding was confirmed by Uysal, Basciftci, and Usümez *et al* (2003).

Our results for shear bond strength of the bleached groups were in agreement with previous studies in which there was no reduction in mean shear bond strength occurred at least one week following bleaching with carbamide peroxide (Bishara, Oonsombat , and Soliman *et al*, 2005; Bulut and Turkan and Demirbaş, 2006; Oztas, Bağdelen and Kılıçoğlu *et al*, 2011). Bishara, Oonsombat, and Soliman *et al* (2005) showed that there was no significant difference in immediate shear bond strengths between the control group and bleached groups regardless at-home or in-office protocol. In their studies, the mean shear bond strength for the control group was  $5.6 \pm 1.8$  MPa, while the means shear bond strength for the at-home groups were  $5.2 \pm 3.6$  MPa and  $7.2 \pm 3.2$  MPa for the 7- and 14-day waiting periods, respectively. The means shear bond strength for their in-office groups were  $5.1 \pm 5.3$  MPa and  $6.6 \pm 2.6$  MPa for the 7- and 14-day waiting periods, respectively. It must be noted that the mean shear bond strengths for their 2-week waiting period was higher than those of the control and the 1-week waiting period in both at-home and in-office bleached groups. This finding was similar to our results as the mean shear bond strength for our 2-week waiting period ( $21.96 \pm 4.14$  MPa) was also higher than that of the control group ( $18.00 \pm 2.86$  MPa). It must also be noted that our mean shear bond strengths were much higher than theirs since our test interval was 24 hours and 3 months rather than immediate. A possible explanation for this higher shear bond strength was bond maturation due to water sorption equilibration after 24 hours (Wiltshire and Noble, 2010). Bulut and Turkan and Demirbaş (2006) also reported no significant difference in shear bond strength between the control group ( $20.6 \pm 2.9$  MPa) and the at-home bleached group after one week waiting period prior to bonding ( $19.7 \pm 2.7$  MPa). Similarly, Oztas, Bağdelen and Kılıçoğlu *et al* (2011) showed there was no difference in shear

bond strength between the control groups and the bleached group after 2-week waiting using both metals and ceramic brackets.

The bleaching protocol used in this study was more intense and the concentrations were much higher than the studies cited above. The amount of residual oxygen from a high concentration of carbamide peroxide bleaching agents has not been thoroughly investigated. It is well known that the variation in shear bond strengths of bleached teeth was suggested to correlate with the amount of residual oxygen on the tooth surface and the amount of calcium on the enamel surface. The residual oxygen within the dentinal tubules interferes with the curing resin tags and thus reduces the number resin tags (Spyrides, Perdigao and Pagani *et al*, 2000; Lai, Tay and Cheung *et al*; 2002). The loss of mineral has also been suggested to reduce bond strength (Potocnik, Kosec, and Gaspersic, 2000; Basting, Rodrigues, and Serra *et al*, 2003; Basting Rodrigues, and Serra, 2004). The amount of residual oxygen often depends on the brand of the bleaching agent, type of solvent in the adhesive (Sung, Chan and Mito *et al*, 1999), and the duration of application (Turkun, Sevgican, and Pehlivan *et al*, 2002).

Several methods were suggested to reduce the residual oxygen on the bleached enamel surface. Sung, Chan and Mito *et al* (1999) recommended the use of alcohol-based bonding agents. Oztas, Bağdelen and Kılıçoğlu *et al* (2011) suggested a thorough rinse with water followed by compressed air application for 30 seconds after every bleaching procedure. Cvitko, Denehy, and Swift *et al* (1991) recommended removal of the superficial layer of enamel. Lai, Tay and Cheung *et al* (2002) and Kaya, Türkün, and Arici *et al* (2008) demonstrated reversal of compromised bonding in bleached enamel by using antioxidant gels right after bleaching for 60 minutes. Other authors suggested delaying bonding orthodontic brackets until two weeks after bleaching (Spyrides, Perdigao and Pagani *et al*, 2000; Cavalli, Reis AF, Giannini *et al*, 2001; Bishara, Oonsombat, and Soliman *et al*, 2005). According to this rationale after the two-week period, the residual oxygen should completely be eliminated from the enamel surface. It must be noted that the bleaching protocol used in these studies was home bleaching with low concentrations of carbamide peroxide.



However, it may not be practical to remove superficial enamel or to apply antioxidant gels for 60 minutes after bleaching in a clinical setting. Therefore, we chose to wait for two weeks after bleaching prior to the bonding procedure in this study. Moreover, the teeth were thoroughly rinsed with water and were stored in artificial saliva after each bleaching session that might have helped with the restoration of mineral loss.

## **7.2 Effect of Bleaching on Surface Enamel**

Changes of enamel surface have been reported in the literature as a consequence of bleaching. Josey, Rossouw and Basson (1996) found that home bleaching caused a significant loss of enamel mineral that sustained over a 12 weeks period in artificial saliva. When etched, bleached enamel surface showed loss of prismatic form with the enamel appearing overetched under the scanning electron microscope. Another study showed that the enamel surface appeared similar to initial caries lesions (Potocnik, Kosec, and Gaspersic (2000). Furthermore, Bishara, Oonsombat, and Soliman *et al* (2005) demonstrates that under scanning electron microscope both the office and home bleaching had a mild etching effect on the enamel surface with the partial loss of the perichymata layer and with the appearance of a rudimentary honeycomb that is typically seen after acid etching. This might explain the cause for high mean shear bond strength observed in the bleached groups in this study. In a different study, Phan and Wiltshire (2010) investigated the effect of bleaching alone without etching on shear bond strength using the same bleaching protocol. We found mean shear bond strength to be  $11.40 \pm 4.17$  MPa. This is higher than clinically accepted mean shear bond strength suggested by both Reynolds (1975), and Wiltshire and Noble (2010).

## **7.3 Shear Bonding Strength Testing**

Minimizing variation from one test to another is one of the most important goals of bond strength studies. This often is measured in coefficient of variation (standard deviation/mean). Powers, Kim and Turner (1997) suggested the coefficient of variation should be in the range of 20% to 30% for the data to be reliable. The coefficients of variation in all groups at the 24 hour period were compatible with this suggestion ranging from 13.02% to 23% in

this study. The coefficient of variation of the data after three months showed a greater range but was still within the suggested values, ranging from 18.77% to 30.69%.

When analyzing the results from bonding studies, it is also important to consider the minimum value in the series together with the mean values. Wiltshire and Noble (2010) suggested a minimum value of 3-4 MPa in each test series for clinically reliable bond strengths to occur. In the 24 hour interval test series, the lowest shear bond strength in this study was 10.13 MPa in the Transbond XT primer group on unbleached teeth. On the contrary, the lower shear bond strength in the three month test series was in the Pro Seal sealant group at 7.16 MPa. Both values were still above the clinically minimal suggested range.

The shear bond strength testing at 24 hours has a significant importance in orthodontics. It often implicates the durability of the bond resisting the initial tension applied by the archwire and chewing forces. Twenty four hour interval is also point of the highest shear bond strength of the entire treatment time demonstrated by Oesterle and Shellhart (2008). This may be due to the fact that most of the polymerization of the resin composite is completed by 24 hours (Klocke, Shi and Vaziri *et al*, 2004; Turk, Elekdag-Turk and Isci *et al*, 2007), and bond maturation due to water sorption equilibration after 24 hours (Wiltshire and Noble, 2010).

In our 24-hour debonding interval, the mean shear bond strengths of Transbond XT and Pro Seal were relatively high in both unbleached ( $18.00 \pm 4.14$  MPa and  $19.22 \pm 3.43$  MPa respectively) and bleached groups ( $21.96 \pm 2.86$  MPa and  $22.61 \pm 4.42$  MPa respectively). There was no significant difference between Transbond XT primer and the Pro Seal in either bleached or unbleached groups. However, significant differences were found between bleached and unbleached groups in comparison of both Transbond XT primer and Pro Seal values. ( $p=0.0011$ ). At the 24-hour period both the bleached and unbleached groups demonstrated higher bond strengths than suggested by Reynolds (1997) and Wiltshire and Noble (2010).

It is often difficult and yet not practical to compare the shear bond strength values from one study to another due to variation in the experimental protocol. Several factors that can potentially influence the bond strength may include the loading type, type of teeth, surface area of bracket and/or buttons, temperature changes, disinfection method and storage media of the teeth before bonding, morphology of the tooth surface and as well as the mineral content of the teeth that are used. It is, therefore a more logical approach to compare our results with studies that were carried out in the same environment under similar conditions. Ho, Bonstein, and Akyalcin *et al* (2010) under a very similar experimental setting reported the shear bond strength of Transbond XT primer with Transbond XT adhesive after 24 hours as  $16.65 \pm 6.04$  MPa, which is comparable to our current study at  $18.00 \pm 4.14$  MPa. The only difference between the two studies was the use of artificial saliva as the storage medium before and after bonding versus distilled water in their study.

The shear bond strength testing at the 3-month time period provides more information on the long term durability of the bonding procedure. After 3 months, it was found that the Transbond XT primer was able to maintain its shear bond strength in both the unbleached and bleached groups (At 24 hours,  $18.00 \pm 4.14$  MPa and  $21.96 \pm 2.86$  MPa respectively versus at 3 month,  $19.16 \pm 4.58$  MPa and  $23.01 \pm 5.32$  MPa respectively). There was no significant difference between 24h and 3-months periods for the mean SBS of Transbond XT primer in either bleached or unbleached groups ( $P > 0.4075$ ). This is in agreement with the results of Ho, Bonstein, and Akyalcin *et al* (2010) in which they found the shear bond strength of Transbond XT primer of  $16.65 \pm 6.04$  MPa at 24 hours and  $15.31 \pm 4.17$  MPa at 3 months. Similarly, Oesterle and Shellhart (2008) showed that there was no significant difference between the 24-hour shear bond strength of Transbond XT primer ( $20.99 \pm 2.11$  MPa) and the strengths at 1 month ( $20.54 \pm 1.58$  MPa) and at 6 months ( $17.77 \pm 1.66$  MPa).

## **7.4 Enamel Fracture during Debonding**

High shear bond strength may become problematic during debonding. Although enamel fracture during bracket debonding does not occur often and is not reported extensively, several precautions should be taken. According to

the literature, enamel fracture is reported to occur with shear bond strengths as low as 9.7 MPa (Retief, 1974). In this study, 15% (3/20) of each subgroup of the bleached groups exhibited enamel fractured at the 3-month period. The means shear bond strength for Transbond XT primer and Pro Seal were  $23.01 \pm 5.32$  MPa and  $23.49 \pm 4.41$  MPa, respectively. This may be beneficial in restorative dentistry but can create enamel fracture during orthodontic debonding. In a similar study by Oztas, Bağdelen and Kılıçoğlu *et al* (2011), 2 samples out of 60 ceramic brackets and 1 sample out of 60 metal brackets demonstrated enamel fracture during debonding on bleached teeth. A possible reason for the higher percentage in the current study can be explained by the higher concentration of carbamide peroxide.

The effect of bleaching on the integrity of the enamel and dentin have been investigated and reported in the literature. Seghi and Denry (1992) found that a period 12 hour at-home bleaching significantly lowered the fracture toughness of the enamel by 30% (from  $1.62 \pm 0.22$  MN/m<sup>3/2</sup> to  $1.14 \pm 0.22$  MN/m<sup>3/2</sup>). They also found that the abrasion resistance was also significantly lower in the bleached groups. Tam and Noroozi (2007) found significant decreases in the mean fracture toughness of the dentin after 2- and 8-week direct (19-34% and 61-68%, respectively) and indirect (up to 17% and 37%, respectively) application. The authors concluded that the reduction in dentin fracture toughness caused by bleaching was greater for the direct application method, with a longer application time, and at a higher bleach concentration.

In our study, the application method was direct and at a higher bleaching concentration. This may explain why the enamel fracture only occurred in the bleached groups, both for Transbond XT primer and Pro Seal, but not in any of the unbleached groups. Patients should be cautioned against prolonged use of these materials to avoid damage to the structure of the dentin. Teeth that are already weakened and restored could potentially exhibit enamel attrition or cuspal fractures.

## 7.5 Bond Strength of Pro Seal Sealants

Highly filled sealants have been advocated by previous studies due to their potential to prevent white spot formations (Hu and Featherstone, 2005; Soliman, Bisharab, and Wefel *et al*, 2006; Buren, Staley and Wefel *et al*, 2008). Currently, there is no data available in the literature investigating the influence of Pro Seal on the shear bond strength after bleaching in either short or long term follow-up.

During the bonding procedure, we carefully followed the manufacturer's instructions on preparing the teeth and curing. The teeth were also chosen so that there was no dentin exposed or fluorosed or chalky appearance to avoid using conditioners prior to applying Pro Seal as advised by the manufacturer.

At the 24-hour period, the shear bond strength of Pro Seal sealant with Transbond XT adhesive was comparable to bonding with Transbond XT primer and adhesive in both unbleached group ( $19.22 \pm 3.43$  MPa versus  $18.00 \pm 4.14$  MPa) and bleached group ( $22.61 \pm 4.42$  MPa versus  $21.96 \pm 2.86$  MPa). There was no statistically significant difference when compared the shear bond strength of the Pro Seal sealant with Transbond XT primer within each group. These results are in agreement with previous studies (Bishara, Oonsombat and Soliman *et al*, 2005; Paschos, Okuka and Ilie *et al*, 2006). Bishara, Oonsombat and Soliman *et al* (2005) showed in their immediate *in vitro* study that Pro Seal sealant did not reduce shear bond strength when compared to the control groups ( $4.8 \pm 2.3$  MPa versus  $4.9 \pm 2.1$  MPa). Similarly, Paschos, Okuka and Ilie *et al* (2006) found no statistically significant difference between Pro Seal sealant and Transbond XT primer when both were used with APC II adhesive ( $10.8 \pm 2.9$  MPa versus  $12.3 \pm 4.0$  MPa).

Upon evaluation the effect of bleaching on Pro Seal sealant, the bond strength of the bleached group with Pro Seal sealant was statistically significantly higher than the unbleached group with Pro Seal sealant ( $p=0.0093$ ). This

finding was shown in both time intervals. A possible explanation for this is that the surface of bleached enamel might have been “overetched” or more porous. Consequently, this may increase the penetration of the Pro Seal sealant, hence the number of resin tags.

Long-term evaluation of Pro Seal has not been reported in the literature. This is particularly important in orthodontics since orthodontic treatment usually lasts more than 24 months. The longest incubation reported in a study was 30 days and was by Lowder, Foley and Banting (2008). In a different laboratory setting with different testing protocols, Lowder, Foley and Banting (2008) showed that Pro Seal sealant when combined with Transbond XT adhesive could demonstrate comparable shear bond strength with their control group ( $13.1 \pm 2.1$  MPa versus  $13.9 \pm 2.8$  MPa) after 30 days incubation. On the contrary, the incubation period in our study was over a 3 month period. Pro Seal sealant when combined with Transbond XT adhesive used on the unbleached teeth exhibited lower shear bond strength ( $13.78 \pm 4.23$  MPa) at 3 month. When compared with the shear bond strength at the 24 hour interval ( $19.22 \pm 3.43$  MPa), the difference was statistically significant ( $p < 0.0001$ ). Despite a large decrease in shear bond strength, this was still higher than the clinically acceptable bond strength suggested by both Reynolds (1975) and minimal reliable clinical bond strength suggested by Wiltshire and Noble (2010).

## **7.6 Adhesive Remnant Index Score**

Information regarding the bond failure location is critically important in orthodontics. Ideally, failure at the bracket-adhesive interface is desirable leaving complete adhesive material on the enamel, which would prevent enamel damaged by debonding force. This constitutes low ARI scores, preferably score 1.

In the unbleached groups, Transbond XT primer exhibited about 25% of the teeth with the score of 1 at the 24 hour period but this increased to 35% at the 3 month period. This was 30% Pro Seal sealant for both 24 hour and 3 month intervals. Overall, the majority of the bond failure in both groups left more than 90% of the adhesive on the enamel surfaces. This finding was in line with Paschos, Okuka and Ilie *et al* (2006).

In the bleached groups, Transbond XT primer maintained with just 5% of the teeth with the score of 1 at 24 hour to 3 months, while the score for Pro Seal sealant dropped down from 30% (24 hour period) to 15% (3 months). The majority of the ARI scores in both the Transbond XT primer and the Pro Seal sealant at either 24 hour or 3 month intervals were of 1, 2 and 3. This adhesive/cohesive failure mode was also found in other studies that waited at least 2 weeks after bleaching prior to bonding (Uysal, Basciftci, and Usümez *et al* 2003; Bulut, Kaya and Turkun 2005; Abe, Endo and Shimooka, 2011).

## **7.7 Limitations and Recommendations from the current study**

In our study, we decided not to test the bond strength immediately or at 6 months, 12 months or 24 months after orthodontic bracket bonding. Immediate testing is often done to determine if the bond strength is strong enough to withstand the force of the initial archwire. Testing in six, twelve and 24 months after bonding would reveal the long term adhesive property of the enamel-adhesive, or adhesive-bracket interface, and cohesive nature of the bonding adhesives, teeth and the orthodontic brackets. In addition to the natural aging process of the orthodontic adhesive, brackets are normally subjected to forces from mastication and heavy archwires as treatment progresses. Therefore testing bond strength over this period of time will be very clinically useful. If the bond strength decreases rapidly over this period, it may not be able to withstand all the forces in the oral environment, thus unnecessary bracket debonding can occur. If the strength is too high, enamel fracture can occur during debonding.

Another criterion that was excluded from our study was to investigate the effect of temperature change over the incubation period. The oral environment would continually be subjected to temperature change due to hot and cold food or drinks from daily intake. Reports from previous studies indicated that shear bond strengths were significantly lower after subjected to 2000, 5000, 10000, and 20000 thermal cycles (Elekdag-Turk, Turk and Isci *et*

*al*, 2008; Turk, Elekdag-Turk, and Isci *et al*, 2010). In contrast, Saito, Sirirungrojying and Meguro *et al* (2005) reported no significant differences in shear bond strength after 0, 2000 and 5000 thermal cycles. It is often difficult to determine the nature of temperature change in each person. In addition, there is an absence in agreement and standardization of thermal cycling protocol in the literature. Thermal cycling was thus excluded from this study.

Our study was one of the first to employ both an in-office and at-home bleaching protocol. Scanning electron microscopic images of the bleached enamel using this protocol were not taken. While it would have been useful to record such images as it could have provided valuable information regarding the roughness of the enamel surface due to the bleaching protocol, in our study, we aimed to concentrate only on the study of the effect of the bleaching protocol on the shear bond strength. Thus recording images were excluded from our study as our major focus in this research.

It is our opinion from the findings in this study that an in-office plus at-home touch up bleaching technique is safe for orthodontic bonding after a two weeks hiatus. However, caution must be applied with teeth that are heavily restored or already compromised during debonding due to potential enamel fracture as shown in 15% of the bleached teeth after 3 months storage.



## 8. CONCLUSIONS

Based on this *in vitro* study on the effect of an in-office plus at-home bleaching on shear bond strength of composite resin with or without Pro Seal sealant, the following conclusions are drawn:

1. At the 24 hour interval, neither adhesive showed better bond strengths *in vitro*
2. After 3-months Transbond outperformed ProSeal *in vitro*
3. Despite lower shear bond strength values *in vitro* at 3-months, the values attained by ProSeal were nevertheless within the range of values considered to be "clinically acceptable"
4. Extrapolating to the clinical situation from our *in vitro* results, clinicians are cautioned against the potential for enamel fracture on bleached teeth during debonding.

## 9. RAW DATA

### Shear Bond Strength after 24 hours (MPa)

Unbleached		Bleached	
Transbond XT	Pro Seal	Transbond XT	Pro Seal
19.95	14.26	19.73	32.16
20.14	21.95	23.08	23.35
15.71	19.61	22.29	24.41
13.85	23.36	17.09	30.14
18.53	17.92	24.19	16.78
15.05	16.2	22.71	20.57
28.95	16.5	26.71	23.57
17.75	24.36	18.51	16.47
12.03	17.34	22.47	21.02
23.27	25.25	24.83	25.85
17.25	13.85	25.01	20.01
17.35	23.64	21.62	18.81
10.13	16.69	19.7	27.74
14.38	15.38	20.8	20.47
21	20.89	16.81	26.34
19.82	19.84	20.78	22.77
19.81	17.44	22.83	24.59
20.95	18.38	24.41	19.24
16.93	18.63	26.36	15.68
17.14	22.87	19.22	22.32

### Shear Bond Strength after 3 months (MPa)

Unbleached		Bleached	
Transbond XT	Pro Seal	Transbond XT	Pro Seal
17.74	8.63	28.55	28.12
23.15	13.84	28.53	24.93
14.05	10.44	19.66	27.4
19.02	17.21	22.26	20.3
18.33	8.77	19.5	26.6
18.93	20.09	21.52	25.07
20.17	10.17	27.85	13.11
20.16	21.24	25.45	17.35
13.52	16	28.52	26.98
24.52	20.79	18.26	21.36
12.48	14.66	24.26	15.11
16.65	15.61	21.96	22.16

24.82	16.61	9.04	27.14
25.64	11.61	18.78	20.88
19.91	11.14	17.41	24.93
21.85	16.47	25.77	21.67
13.49	14.11	26.53	28.5
26.16	7.16	23.37	27.77
21.74	12.29	32.71	25.52
10.78	8.66	20.26	24.84

Adhesive Remnant Index 24 hours			
Unbleached		Bleached	
Transbond XT Primer	Pro Seal	Transbond XT Primer	Pro Seal
3	1	3	2
1	3	3	2
2	1	3	2
3	2	2	3
5	1	3	3
3	2	3	3
2	3	4	1
4	2	2	1
3	2	3	2
1	3	1	1
2	2	2	2
2	3	3	2
1	3	3	1
3	3	2	3
1	1	3	1
3	2	3	3

3	2	3	3
3	1	3	2
1	2	3	1
2	1	3	3

Adhesive Remnant Index 3 months			
Unbleached		Bleached	
Transbond XT Primer	Pro Seal	Transbond XT Primer	Pro Seal
3	1	3	2
3	3	3	3
4	1	3	5
2	3	3	3
2	2	2	3
3	2	3	3
3	2	5	3
2	2	3	1
3	2	3	3
1	2	3	4
1	1	4	3
1	1	3	3
1	3	4	3
1	2	4	3
1	2	1	3
1	3	2	2
2	3	2	3
3	2	2	1
3	1	3	3
4	1	3	1

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## 11. APPENDIX

### 11.1 Ethics Approval

 <b>UNIVERSITY OF MANITOBA</b>	<b>BANNATYNE CAMPUS Research Ethics Boards</b>	P126-770 Bannatyne Avenue Winnipeg, Manitoba Canada R3E 0W3 Tel: (204) 789-3255 Fax: (204) 789-3414
	<b>APPROVAL FORM</b>	

<b>Principal Investigator:</b> Dr. X. Phan <b>Supervisor:</b> Dr. S. Akyalcin	<b>Ethics Reference Number:</b> H2010:249 <b>Date of Approval:</b> July 27, 2010 <b>Date of Expiry:</b> July 27, 2010
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**Protocol Title:**            **Effect of Bleaching on Shear Bond Strength of Fluoride-releasing Sealant**

**The following is/are approved for use:**


- **Proposal dated July 15, 2010**

The above underwent expedited review and was **approved as submitted** on July 27, 2010 by Dr. John Arnett, Ph.D., C. Psych., Health Research Ethics Board, Bannatyne Campus, University of Manitoba on behalf of the committee per your submission dated July 15, 2010 (received July 19, 2010). The Research Ethics Board is organized and operates according to Health Canada/ICH Good Clinical Practices, Tri-Council Policy Statement, and the applicable laws and regulations of Manitoba. The membership of this Research Ethics Board complies with the membership requirements for Research Ethics Boards defined in Division 5 of the *Food and Drug Regulations of Canada*.

**This approval is valid for one year only.** A study status report must be submitted annually and must accompany your request for re-approval. Any significant changes of the protocol and informed consent form should be reported to the Chair for consideration in advance of implementation of such changes. The REB must be notified regarding discontinuation or study closure.

This approval is for the ethics of human use only. For the logistics of performing the study, approval must be sought from the relevant institution, if required.

Sincerely yours,



John Arnett, Ph.D., C. Psych.  
Chair, Health Research Ethics Board  
Bannatyne Campus

**Please quote the above Ethics Reference Number on all correspondence.**  
Inquiries should be directed to REB Secretary  
**Telephone:** (204) 789-3255 / **Fax:** (204) 789-3414

## 11.2 Journal Article and Submission Confirmation

### EFFECT OF TOOTH BLEACHING ON SHEAR BOND STRENGTH OF A FLUORIDE-RELEASING SEALANT

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# EFFECT OF TOOTH BLEACHING ON SHEAR BOND STRENGTH OF A FLUORIDE-RELEASING SEALANT

## ABSTRACT

**Objective:** To evaluate the effect of an in-office plus at-home bleaching protocol on shear bond strength of orthodontic buttons when using a fluoride-releasing sealant.

**Methods and Materials:** One hundred and sixty extracted human molars were randomly divided into bleached (N=80) and unbleached groups (N=80). The bleached group was treated with 45% carbamide peroxide for half an hour, followed with five applications of 20% carbamide peroxide at 24 hour intervals. After two weeks, lingual buttons were bonded on the teeth in both groups using either Transbond XT primer or Pro Seal sealant. The teeth were then stored in artificial saliva and subjected to shear testings at 24 hours and 3 months using a Zwick Universal Test Machine.

**Results:** The ANOVA analysis of the 24-hour results indicated a significant difference between the four subgroups ( $p < .0011$ ). Further simple t-tests indicated that the differences were significant only between bleached and unbleached subgroups ( $p < .0011$ ). The 3-month results showed the mean shear bond strengths of the unbleached group using Pro Seal sealant was statistically significantly lower than the others although still greater than clinically minimal suggested bond strengths. Interestingly, 15% of the bleached teeth exhibited enamel fracture at the 3-month testing.

**Conclusion:** At 24 hours, both Pro Seal sealant and Transbond XT primer appear to be a reliable choice on both bleached and unbleached teeth. However, at the 3-month period, Pro Seal sealant yielded significantly lower shear bond strength on unbleached teeth, nevertheless well within the range of values considered to be "clinically acceptable".

## INTRODUCTION

White teeth are commonly associated with confidence and good hygiene habits. As a result, demands of more effective and faster results in dental bleaching have driven manufactures to develop more potent products with more aggressive protocols. The most common active ingredient in bleaching gels is carbamide peroxide, a chemical agent that penetrates the porosities of tooth enamel and oxidizes stain deposits. The traditional office bleaching protocol employs three applications of 30-minute treatments at one-week intervals due to the potential for sensitivity. With the recent addition of potassium nitrate and fluoride to the whitening gels<sup>1</sup>, treatments no longer have to be as frequent. Thus, patients can complete the whole procedure in three consecutive days. Currently, a combination of one in-office session with multiple at-home touch-up treatments is often recommended.

Many adults who seek orthodontic treatment may have had teeth whitening done previously, and therefore the layer of enamel could have been structurally affected. An area of concern to orthodontists is the often irreversible and unesthetic iatrogenic effect of orthodontic treatment on the tooth surface. If dental plaque accumulates around brackets, decalcification or white spot lesions will develop on the enamel. Unfortunately, this problem is frequently detected in orthodontic patients. The prevalence is reported to be as low as 13% to as high as 75%.<sup>2,3</sup> Recently, highly filled, fluoride-releasing sealants have been advocated due to their potential to prevent white spot formations.<sup>4,5,6</sup> This has triggered some interest among orthodontists, since this material may be useful to protect the susceptible area beneath and adjacent to bonded attachments.

In terms of using preventive measures such as a fluoride-releasing sealant, it is valuable to know if tooth bleaching protocols will affect the bond strength of orthodontic brackets. Therefore, the main goal of this study was to investigate the effects of an “in-office plus at-home touch up bleaching technique” on the shear bond strength of orthodontic attachments bonded with fluoride-releasing sealants.

## MATERIAL AND METHODS

One hundred and sixty extracted human molars were collected from four independent maxillofacial and oral surgery clinics. The criteria for tooth selection included intact buccal enamel with similar flatness. Ethics approval for this study was obtained from Health Research Ethics Board at the University of Manitoba prior to the start to the experiment. Half of the teeth were subjected to bleaching, whereas the other half was incubated in artificial saliva at 37° until the bonding procedure. The artificial saliva was refreshed daily. The bleached teeth were polished with a non-fluoridated pumice and water slurry using rubber prophylactic cups for 10 seconds, rinsed with water and then air dried. Carbamide peroxide gel (45%) (Opalescence Quick PF ,Ultradent, Utah) was applied to the buccal surfaces of the teeth directly from the syringe in a layer of approximately 1 mm thickness and the teeth were kept at 37°C at 100% humidity for 30 minutes. After the bleaching gel was rubbed off and thoroughly rinsed with water, the teeth were incubated in artificial saliva at 37°C. Twenty-four hours later, the teeth were bleached again with 20% carbamide peroxide for 6 hours. The bleaching gel was rubbed off and again thoroughly rinsed with water. The teeth were again incubated in artificial saliva at 37°C. This procedure was repeated 4 more times at 24-h intervals and the bleached teeth were finally incubated in artificial saliva at 37°C for two weeks prior to bonding.

The experimental design is summarized in Figure 1. The bleached and unbleached teeth were randomly divided into four groups and then submitted to different bonding procedures as follows:

*Group 1 (Control)* - Forty unbleached teeth were etched with Transbond XT 37% phosphoric acid gel (3M Unitek, Monrovia, California) for 30 seconds. The teeth were then rinsed with water spray for 20 seconds and dried with an oil-free air spray for 20 seconds until the enamel appeared frosty. Transbond XT primer (3M Unitek, Monrovia, California) was applied on the enamel surface and light-cured for 20 seconds. Metal lingual buttons (GAC International, Central Islip, NY) of 3.33 mm diameter with a mesh-base were then bonded with Transbond XT adhesive paste and placed on the etched enamel surface. A light finger pressure was applied until the buttons touched the surface of the enamel. Excess adhesive was removed with a scaler and the samples were cured for 40 seconds with an Ortholux LED light (3M, Unitek, Monrovia, California) at a distance of approximately 5 mm.

*Group 2*- Forty unbleached teeth were submitted to a similar protocol as described for group 1; however, Pro Seal sealant (Reliance Orthodontic Product Inc, Itasca, Ill), a fluoride-releasing sealant, was used in place of Transbond XT primer.

*Group 3* – Forty bleached teeth subjected to the same protocol described for group 1.

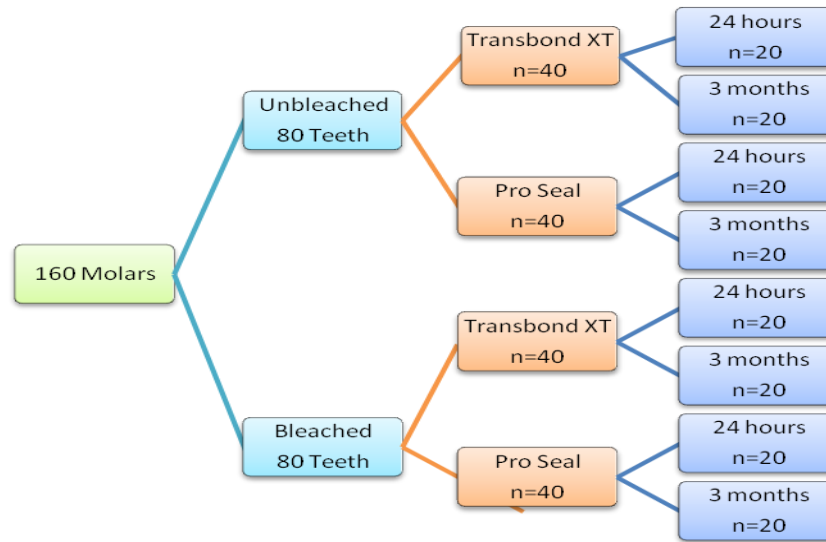
*Group 4* – Forty bleached teeth subjected to the same protocol described for group 2.

The teeth were incubated immediately upon the completion of the bonding procedure in artificial saliva at 37°C. Each group was then randomly divided into two subgroups. The first subgroup of each group was tested 24 hours after bonding, and the latter was tested 3 months after bonding. The shear bond strength was tested using the Zwick Universal Testing Machine (Zwick GmbH, Ulm, Germany). Briefly, a direct sharp shearing blade was loaded to the enamel-adhesive-bracket interface parallel to the height of contour in an occluso-gingival direction. The speed of the crosshead was set at 0.5mm/min and the shear bond strength was measured using a 1KN load cell.

Evaluation of bond-failure sites was carried out with a protocol similar to the one used previously by Årtun and Bergland.<sup>7</sup> The debonded surface of each tooth was examined under a light-microscope with 10x magnification and an adhesive remnant index (ARI) score was given to each tooth. Three months later, the samples were randomly selected from each group and the ARI scores were re-evaluated by the same operator to establish intra-examiner reliability.



## Summary of the study



## STATISTICAL ANALYSIS

Descriptive statistics, including the mean, standard deviation, and minimum and maximum values were calculated for the eight subgroups of teeth tested. Comparisons of mean shear bond strength values were made with an analysis of variance (ANOVA) test. If significant differences were present, simple t-tests were performed to determine which means were significantly different from each other. The Fisher's exact test was used to determine significant differences of the adhesive remnant index (ARI) scores among the eight subgroups. The significance used for all the tests was predetermined at  $p \leq 0.05$ .

## RESULTS

The mean shear bond strengths at 24 hours are summarized in Table 1. The ANOVA test indicated significant differences between the four groups ( $p < .0011$ ). Further simple t-tests indicated significant differences for any possible comparison between the subgroups of bleached and unbleached groups. However, no significant difference was observed between Transbond XT primer and Pro Seal in either bleached or unbleached groups ( $p > .05$ ).

Table 1: Descriptive data of shear bond strength after 24 hours

Groups		Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Coefficient of Variation
Unbleached	Transbond XT Primer	20	18.00	4.14	10.13	28.95	23.00%
	Pro Seal	20	19.22	3.43	13.85	25.25	17.85%
Bleached	Transbond XT Primer	20	21.96	2.86	16.81	26.71	13.02%
	Pro Seal	20	22.61	4.42	15.68	32.16	19.54%

Table 2 summarizes the mean shear bond strength at 3 months. The ANOVA analysis showed that there is a significant difference between these four groups ( $P<.0001$ ). The comparison of Pro Seal sealant and the Transbond XT primer in the unbleached group yielded a statistically significant difference ( $P<.000428$ ). However, there was no significant difference in bleached group.

Table 2: Descriptive data of shear bond strength after 3 months

Mode	Groups	Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Coefficient of Variation
Unbleached	Transbond XT Primer	20	19.16	4.58	10.78	26.16	23.90%
	Pro Seal	20	13.78	4.23	7.16	21.24	30.69%
Bleached	Transbond XT Primer	20	23.01	5.32	9.04	32.71	23.12%
	Pro Seal	20	23.49	4.41	13.11	28.74	18.77%

When comparing the results of each group from the 24-hour interval to the 3-month interval, the only subgroup that showed statistically significant difference was the Pro Seal in the unbleached group ( $p<.0001$ ). The shear bond strength after 3 months (13.78MPa) was significantly lower than at 24 hours (19.22MPa) after bonding. The general trend for other groups was that the shear bond strength increased slightly over the 3 months period, although the differences were not statistically significant ( $p>.05$ ).

Upon inspection of the mode of bond failure, the majority of the failures in the unbleached teeth left more than 90% of adhesive on the enamel surfaces while the mode of failure in the bleached groups was more mixed adhesive/cohesive. Fisher's Exact Test indicated no significant difference among the ARI scores the at 24-hour interval. However, there was a significant difference among the groups at the 3-month interval ( $p < .0257$ ). The bleached teeth at the 3-month period had significantly less adhesive remaining on the enamel surface.

## DISCUSSION

It was interesting to note that the mean shear bond strengths of the bleached teeth were higher than those of the unbleached teeth at both 24-hour and 3-month debonding intervals. A possible explanation for this may be the fact that the bonding procedure was delayed for two weeks after the bleaching protocol had finished; thus, allowing enough time for the elimination of the residual oxygen that normally interferes with the polymerization of the resin tags.<sup>8,9</sup> Early bonding following bleaching could result in polymerization inhibition of the composite due to the delayed release of oxygen.<sup>10,11</sup> Moreover, the use of artificial saliva as the storage medium during and between bleaching sessions may have promoted remineralization due to the calcium and phosphate ion content in the artificial saliva.<sup>12</sup> It is also important to emphasize that the bleached enamel surface might have become more porous or "overetched" than regular etched enamel after being treated with phosphoric acid.<sup>13,14</sup> A combination of these overall effects might have consequently increased the number of resin tags and translated into higher shear bond strengths. The results for shear bond strength of the bleached groups in this study are in agreement with previous studies where there was no reduction in mean shear bond strength at least one week following bleaching with carbamide peroxide.<sup>15,16,17</sup>

When analyzing the results from bonding studies, it is important to consider the minimum value in the series together with the mean values. Wiltshire and Noble<sup>18</sup> suggested a minimum value of 3-4 MPa in each test series for clinically reliable bond strengths to occur. In the 24-h interval test series, the lowest

shear bond strength in the present study was 10.13 MPa found in the Transbond XT primer group on unbleached teeth. However, the lowest shear bond strength in the 3-month test series was 7.16 MPa, found in the Pro Seal sealant group. Both values are above the recommended clinically minimal range suggested by Wiltshire and Noble.<sup>18</sup> Consistency of the tests is another important consideration in bonding studies. This is often measured using the coefficient of variation (standard deviation/mean). Powers *et al*<sup>19</sup> suggested the coefficient of variation should be in the range of 20% to 30% for the data to be reliable. The coefficients of variation in all groups were compatible with this recommendation ranging from 13.02% to 23% at the 24-hour test and 18.77% to 30.69% at 3-months in our study.

High shear bond strength may indicate problems during debonding. Although enamel fracture during bracket debonding does not occur often and has not been reported extensively in the literature, several precautions should nevertheless be taken. Retief<sup>21</sup> reported that enamel fracture may occur with shear bond strengths as low as 9.7 MPa. In our study, 15% (3/20) of each subgroup of the bleached groups exhibited enamel fracture at the 3-month period suggesting that this may be a risk factor for enamel fracture during orthodontic debonding. This *in vitro* finding may raise a red flag for enamel fracture during orthodontic debonding *in vivo*. In a similar study, Öztas *et al*<sup>17</sup> reported that 2 samples out of 60 ceramic brackets and that 1 sample out of 60 metal brackets demonstrated enamel fracture during debonding on bleached teeth. A possible reason for the higher percentage in the present study may be due to the higher concentration of carbamide peroxide employed in the Opalescence Quick PF whitening system.

Currently, there is no data available in the literature investigating the influence of tooth bleaching on Pro Seal sealant in either short or long term follow-up. This study has shown that Pro Seal sealant performed comparably to the Transbond XT primer on bleached enamel surfaces at both time intervals. On unbleached enamel surfaces, our results are in agreement with previous studies.<sup>15,20</sup> Bishara *et al*<sup>15</sup> showed in their *in vitro* study that Pro Seal sealant did not reduce immediate shear bond strength when compared to Transbond XT primer. Similarly, Paschos *et al*<sup>20</sup> found no statistically significant difference between Pro Seal sealant and Transbond XT primer when used with adhesive precoated brackets.

The long-term evaluation of fluoride-releasing sealants has not been reported in the literature either. This is particularly important in orthodontics since orthodontic treatment usually lasts about 24 months. The longest incubation period reported in the literature was 30 days but in a different laboratory setting with different testing protocols and they showed that Pro Seal sealant when combined with Transbond XT adhesive could demonstrate comparable shear bond strengths with the Transbond XT primer ( $13.1 \pm 2.1$  MPa versus  $13.9 \pm 2.8$  MPa, respectively) after 30 days incubation.<sup>21</sup> On the contrary, the incubation period in the present study was over 3 months. Pro Seal sealant when combined with Transbond XT adhesive used on the unbleached teeth exhibited lower shear bond strength at 3 months when compared to the 24-hour result ( $p < .0001$ ). Despite a large decrease in shear bond strength, this was still higher than the minimal reliable clinical value suggested by Wiltshire and Noble.<sup>18</sup>

Information regarding the bond failure location is critically important in orthodontics. Ideally, failure at the bracket-adhesive interface is desirable leaving all the adhesive material on the enamel, which would prevent this mineralized tissue from being damaged by debonding forces. In the unbleached groups, the majority of the bond failure in both groups left more than 90% of the adhesive on the enamel surfaces. This finding is in agreement with Paschos *et al.*<sup>21</sup> In the bleached groups, the mode of bond failure was adhesive/cohesive in nature. Indeed, this mostly mixed adhesive/cohesive failure mode was also found in other studies that stored their samples 2 weeks after bleaching, prior to bonding.<sup>16,22</sup>

In summary, the findings in this study suggest that an “in-office plus at-home touch up bleaching technique” is safe for orthodontic bonding after a two weeks hiatus. However, caution must be applied to teeth that are heavily restored or already compromised during debonding due to potential enamel fracture.

## CONCLUSION

Based on this *in vitro* study on the effect of an in-office plus at-home bleaching protocol on shear bond strength of a fluoride-releasing sealant, the following conclusions are drawn:

- 1- At 24 hours, both Pro Seal sealant and Transbond XT primer appear to a reliable choice on either bleached or unbleached teeth for bonding orthodontic brackets. However, at the 3-month period, Pro Seal sealant yielded significantly lower shear bond strength on unbleached teeth, nevertheless well within the range of values considered to be "clinically acceptable".
- 2- Extrapolating to the clinical situation from our *in vitro* results, clinicians are cautioned against the potential for enamel fracture on bleached teeth during debonding.

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