THE EFFECT OF CROSSHEAD SPEED, LOAD CELL CONFIGURATION AND CURING TIME ON THE SHEAR BOND STRENGTH OF ORTHODONTIC BRACKETS

By

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Abstract

Objective: Evaluate the effect of crosshead speed, load cell configuration and curing time on shear bond strengths**.**

Methods: 160 human molars were divided into equal groups of 20 second and 40 second photopolymerization times and then into 1kN or 10kN load cell groups. Each of the groups were divided into 0.5mm/min or 5mm/min crosshead speeds.

Results: Regarding photopolymerization time (20s vs. 40s) and crosshead speeds (0.5mm/min vs. 5.0mm/min) there were no significant differences in SBS ($p>0.05$). The load cell configuration (1kN vs. 10kN) however showed statistically significant differences ($p<0.05$) with the 1kN producing higher bond strengths.

Conclusion: Only load cell configuration affected the shear bond strengths.

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Table of Contents

List of Tables and Figures

1. INTRODUCTION

Since the introduction of different bracket adhesive materials to the orthodontic market, both *in vivo* and *in vitro* studies have been conducted to test their effectiveness and working characteristics. As new bonding agents have been introduced, research focused on this area and the resultant publication rate of papers on orthodontic bonding increased considerably. This is illustrated in the steadily increasing number of bonding papers appearing in orthodontic journals (Eliades & Brantley, 2000).

Studies involving orthodontic adhesive materials have mainly focused on shear bond strength studies. The results obtained from these studies show the strength of the adhesive between the tooth and bracket. Typically higher bond strengths indicate a better adhesive material. To conduct the actual tests, brackets are bonded to extracted teeth, tightly secured into a tooth test ring using acrylic and then mounted onto a testing machine. The machine contains a steel rod attached to a crosshead and once the rod is activated it contacts the mounted bracket and shears it off. A computer electronically connected to the Universal Test Machine records the strength of the adhesive of each test in megapascals. The speed of the crosshead and load cell weight can be changed and as a result may affect the overall results (Eliades & Brantley, 2000).

Although many bonding studies have been conducted, there lacks a universally accepted protocol to conduct these studies. As a result, it is difficult and sometimes impossible to compare the materials and techniques used in the studies as well as the results within themselves (Bishara, Soliman, Laffoon, & Warren, 2005). Fox (1994) found that even changing one test parameter could significantly affect the interpretation of the results. In addition, it was observed that there was a large variation in the methodology used for orthodontic material testing of bond strength.

From this it was suggested that researchers should adopt a standard methodological approach that included tooth type, surface enamel preparation, storage medium, testing equipment and technique, sample size, statistical analysis, and bond strength units (Fox, McCabe, & Buckley, 1994). Finnema et al. (2010) reported that many studies on *in vitro* orthodontic bond strength fail to report test conditions that could significantly affect their outcome. In their systematic review and meta-analysis, a summary of factors is given that can affect the *in vitro* bond strength of orthodontic brackets. Experimental conditions that significantly influence in-vitro bond strength are water storage of the bonded specimens, photopolymerization time, and crosshead speed (Finnema, Ozcan, Post, Ren, & Dijkstra, 2010).

New dental products are constantly being introduced to the orthodontic market and are continually being tested for their effectiveness. In order for clinicians to critically evaluate new products and better serve their patients, it remains essential that thorough testing of new materials be conducted by independent sources, in addition to the potentially biased "in house" tests undertaken by manufacturers. Materials need to bond sufficiently strong, yet not too strong to cause enamel damage on debonding. The bond should not deteriorate during orthodontic treatment over time, it should be non-toxic, non-allergenic and preferably anticariogenic. Only eventual standardized testing of orthodontic products will allow accurate evaluation, comparison and full disclosure scrutiny of new and evolving products (W. A. Wiltshire, 2012a).

2. LITERATURE REVIEW

2.1 Evolution of Bonding

Michael G Buonocore (1955) revolutionized the field of dentistry by introducing the acid etch technique that allowed adhesion of an acrylic filling material to enamel surfaces (BUONOCORE, 1955). The basis of bonding materials, the bisphenyl A glycidyl dimethacrylate (Bis-GMA) was synthesized and introduced by Bowen in 1956 and eventually led to the first successful production of composite resin for filling teeth (BOWEN, 1956). The popularity of the bonding technology improved significantly and eventually Newman (1965), introduced direct bonding of orthodontic attachments as an alternative to banding as a viable clinical option (Newman, 1965). Over the past 45 years, many advances and innovations in adhesion and bonding have occurred, from multi-step to one-step bonding procedures, and all with claims of improved adhesivity (W. A. Wiltshire & Noble, 2010). Subsequently, orthodontic bonding developed as an excellent alternative to banding and eventually over the years its properties, applications, and techniques of use significantly increased. Eliminating the need for banding of individual teeth, allowed clinicians to increase treatment efficiency, patient comfort, elimination of pretreatment tooth separation, improve oral hygiene and esthetics, and reduce chair time (W. A. Wiltshire & Noble, 2010).

Retief et al. (1970) brought clarification to the bonding mechanics by explaining the process of adhesion, wetting and contact angles that allowed a more focused approach to studying orthodontic bonding. The group also initiated much of the research in comparing orthodontic adhesives and were able to illustrate that fresh adhesive outperformed a similar old sample (Retief, Dreyer, & Gavron, 1970). Conventional laboratory bond strength testing has been described in numerous peer-reviewed journals. It is important to review the methodology before applying the results of a project clinically. Bond strength studies, irrespective whether shear bond strength (SBS) or tensile bond strength (TBS), use the "mechanical mouth," better known scientifically as the Universal Testing Machine (P. Emile, 2010). This testing device provides standardized bond strength results which can be compared between various studies and can allow new adhesive products to be manufactured and introduced to the marketplace based on its improved properties. A standardized approach in laboratory testing of new self-etching primer systems is quite difficult to achieve due to variations in methodologies and techniques.

2.2 Bond Testing in Orthodontics

Ensuring an adequate bond between the bracket base and the tooth is important to the clinical success of orthodontic treatment. The bracket must be able to withstand the masticatory forces during the length of the treatment; otherwise, treatment can be unnecessarily delayed and costly for the patient and orthodontist. On the other hand, if the bond strength between the bracket interface and tooth is too strong, the enamel surface can be damaged during debonding (Proffit WR, Fields HW, Sarver DM, 2007). It is important to initiate preliminary *in vitro* studies of new bonding agents to provide clinical insight as to how they may actually perform. Without the input from *in vitro* studies, research in orthodontic bonding will not progress and orthodontics will not be able to utilize and take advantage of possibly more effective and efficient bonding agents entering the market.

In general, studies on orthodontic bonding are classified according to mode of load application and testing environment. Load applications are shear, tension or torsion and testing environments are either *in vitro, in vivo, or ex vivo.* Due to the relative simplicity of experimental configuration and presumably increased reliability of simulating debonding that occurs during treatment, shear testing is the most popular mode of load application in an *in vitro* environment (Eliades T, 2000).

2.2.1 Shear Testing

In shear testing, the load can be applied parallel to the base of the bracket by a blade in compression or by a wire loop in tension (T. R. Katona & Long, 2006). However, pure shear testing is extremely difficult to obtain due to the complexities of the forces involved and instead is a combination of shear, peel and shear peel forces. Bond failure occurs due to a combination of bending of the bracket base resulting in tensile stress and shearing. When the adhesive bond strength is too high, fractures in the enamel and dentine can occur due to bending action that will predispose any crack that forms to deviate into the dentine or enamel, which would be considered problematic in a clinical situation. Overall, it is difficult, if not impossible to have pure shear testing and replicate the *in vivo* masticatory forces, but laboratory studies can provide the clinician an understanding of how biomaterials may perform in the complex oral environment (W. A. Wiltshire & Noble, 2010).

2.2.2 Tensile Testing

In tensile testing, the bracket is pulled off perpendicular to the bracket base. 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 (Phan, Akyalcin, Wiltshire, & Rody, 2011a).

2.2.3 Shear-Peel Bond Strength

In this method of testing, the debonding force is applied at some distance from and perpendicular to the adhesive-attachment junction. This method results in a combination of shear stress and some component of "peel" force being applied to the adhesive interfaces. The distance of the debonding apparatus from the attachment-adhesive junction will determine the amount of shear and peel force being applied. It is difficult to determine the exact amount of each force, however, it is well understood that studies reporting shear bond strengths are in fact testing shear-peel bond strength since it is difficult to get shear bond strength alone (T. R. Katona, 1994; T. R. Katona, 1997).

2.2.4 Fracture Toughness Testing

The ability of a material to resist fracture (breakage) is the mechanical property that most distinguishes ceramics from metals. This ability is called fracture toughness. A shallow scratch on the surface of a ceramic will drastically reduce the load required for fracture, whereas the same scratch on a metal surface will have little, if any, effect on fracture under load. In orthodontics this testing is mainly undertaken to compare ceramic (poly crystalline or monocrystalline) and metal brackets. If a scratch of the same dimension is made on the surface of a metal and ceramic bracket, and they are flexed, the ceramic rods will break. Typical values of fracture toughness for stainless steel are in the 80-95 MPa range, and 2.4-4.5 MPa range for ceramics (Scott, 1988).

2.2.5 Debonding Force and Bond Strength

Force is known to be measured in Newtons (N) and one N is required to accelerate a mass of 1Kg at a rate of $1 m/s^2$. Forces are often expressed in kilo Newtons (kN) (1 kN= 1000N), or pound (lb) or pound force (lb_f) (1N ≈ 0.22481 lb_f) (W. A. Wiltshire & Noble, 2010). In orthodontic bond strength testing terms, use is often made of the Pascal (Pa), or metric unit of pressure or stress (1 Pa = 1 N acting on an area of 1 m^2). Pound per square inch (psi) as a unit of stress is also frequently used in bond strength reporting (1 psi = 1 lb_f/in^2) (1 psi = 6894.76 Pa) (W. A. Wiltshire & Noble, 2010). Megapascals (MPa) is currently accepted as the preferred unit for reporting bond strengths. Bond strength can also be reported as *bond force* in units of Newtons (N), kilograms (kg) or pounds (lbs). Bond strength is the bond force divided by the area of the bonded interface (e.g. 1 Pa = 1 N/m^2, 1 MPa = 1 N/mm^2). Thus, experimental studies using a universal testing machine (e.g. Zwick GmBH) can measure the force (N) needed to debond a bracket with a known bracket base area (mm^2) to give a bond strength value in N/mm^2 or MPa (Powers, Kim, & Turner, 1997).

2.2.6 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)(Phan, Akyalcin, Wiltshire, & Rody, 2011a). Electromechanical or universal testing machines are most commonly used for static testing in a tensile or compression mode. Additional test types include tensile, compression, shear, flexure, peel, tear, cyclic, and bend tests. Capacities for these systems range from low-load forces of 0.5 kN (112 lbf) up to high-capacity 600 kN (135,000 lbf). These systems are frequently configured for automated testing. In addition to the testing equipment, specimen preparation is an extremely important factor to consider when evaluating the repeatability of results. Specimens with nicks, cuts, and non-parallel edges will have an adverse impact on repeatability of results (Instron® Products: By

Product Type, Electromechanical Systems, 2012). The two most common testing machines in the field of dentistry and orthodontics are Instron, Norwood, MA (Figure 2.1) and Zwick Universal Testing Machines, Zwick GmBH, Ulm, Germany (Figure 2.2) (W. A. Wiltshire, 2012a).

Figure 2.1: Instron Universal Testing Machine 3360 Series

Figure 2.2: Zwick GmBH Universal Testing Machine

2.2.7 Minimum recommended bond strength

The posterior region of the mouth can produce a biting force of about 20MPa and this may be an indication of the minimum bond strength required. In addition to the masticatory forces, the clinical bond strength of a bracket needs to be strong enough to resist the applied orthodontic forces placed by the orthodontist but weak enough to allow for debonding at the end of treatment. Reynolds (1975) proposed that "clinically acceptable" bond strengths should be in the 6-8 MPa range. This recommendation was deduced if a typical bracket has a bonding area of 16mm^2 and the average force transmitted to a bracket during function is between 40 – 120 N (1 MPa = 1 N/mm^2). Thus, the minimum bond strength needed to withstand the applied force of 120 N is 7.5 MPa (Powers et al., 1997; Reynolds, 1975). However, the Reynolds study was published 30 years ago and testing systems, computerization, and products have changed significantly.

According to Wiltshire & Noble (2010), an "ideal bond strength" is difficult to define because each individual differs in their masticatory forces, eating habits, and intra-oral environment. They recommended that to achieve the minimal reliable clinical bond strength, *in vitro* bond strengths should be at least 3-4 MPa. It is also important to note that during *in vitro* testing, it is essential to not only look at the means but to also examine the range of values; in particular the low end of the range. (Wiltshire & Noble, 2010) This recommendation was based on clinical trials using glass ionomers to bond orthodontic brackets. It was demonstrated that there was no significant differences in failures rates between the glass ionomer (3.3%) and conventional orthodontic resin (1.6%). (Fricker, 1994)

2.3 Testing Standardization

For useful standards to be developed for orthodontic products, test protocols for important physical properties need to be standardized (Stanford, Wozniak, & Fan, 1997). Despite this abundance of studies on dental materials in the scientific literature, it is often difficult to meaningfully compare the performance of these products because of the lack of a universally accepted protocol to conduct these experiments. It has been reported that changing one of the test parameters could significantly affect the results as well as the interpretation of the outcome (Bishara et al., 2005).

In 1970, the American National Standards Committee (ASC) MD156 was created for the development of standards in dentistry with the American Dental Association (ADA) as Secretariat. As of this date, all ADA specifications for dental materials, instruments, and equipment are submitted and then approved as American National Standards. As part of this program, a manufacturer or distributor of a product submits data showing that the product meets the requirement values of the appropriate specification. The ADA then confirms this compliance by testing the marketed product in its evaluation laboratories. Currently, two ASC MD 156 working groups are active in the development of standards for orthodontic products. These working groups evaluate test methods reported by researchers and determine appropriate tests that best fit the need of the standard. A proposed test method is then evaluated by round robin testing in several laboratories, using products chosen to be representative of safe and effective products for that category. It is well recognized in ASC MD156 that to facilitate the development of a suitable test method, standardization of test protocols must come first. Similar activities are conducted on an international basis by the International Organization for Standardization (ISO) TC106 Dentistry.

Currently, a review of recent publications in the orthodontic literature involving laboratory testing shows that there is a lack of standardized test protocols in evaluation of orthodontic products, including bond strength testing (Stanford et al., 1997).

2.4 Bond Strength Testing Standarization

The major classification of bonding studies can be divided into three categories:

1. Test environment: *in vivo, in vitro, ex vivo*
2. Substrate: *enamel, composite resin, porcelain, amalgam*
3. Loading mode: *shear, tensile, torsion, shear-peel*

In-vitro studies possibly allow for more standardization procedures since clinically it is almost impossible to distinguish the adhesive potential of a specific bonding system independent of many other variables (Eliades & Brantley, 2000). Many factors influencing adhesive shear and tensile bond strengths have been studied, including time elapsed between bonding and debonding, whether the bonded samples were subjected to thermal stresses, whether contamination occurred during the bonding procedure, the type of curing light, the composition of the adhesive, the use of fluorides on teeth, the type and concentration of etchant, etching time, the type of brackets, the preparation of enamel, and the use of bleaching prior to bonding (Bishara et al., 2005; Finnema et al., 2010).

2.5 Literature on Bond Strength Testing Standardization

Finnema (2010) conducted a systematic review of the available literature regarding *in-vitro* orthodontic shear bond strength testing. To date, this publication has been the most comprehensive review of bond strength testing conditions. Results from this paper showed bond strength testing was negatively influenced when the test teeth were stored in water. Water storage on average decreased bond strength by 10.7 MPa, assuming that the other predictors remain constant. Although this was the most pronounced effect of an experimental condition, this finding was mainly influenced by one relatively large study sample in which artificial saliva was used as a storage medium for specimens. It was reported that bond strengths were significantly higher when teeth were stored in artificial saliva. Analogously, each second of photopolymerization time increased the bond strength by 0.077 MPa. The studies in the meta-analysis showed considerable variations in photopolymerization time: from 2 to 50 seconds, however, most studies reported 40 seconds for polymerizing adhesive and this corresponds to the routine clinical standard. Although the results indicated increasing photopolymerization time yields higher bond strengths, the most optimal time for polymerizing still needs to be determined in future studies. When crosshead speed increased by 1 mm per minute, bond strength increased by 1.3 MPa. However, the studies used in the meta-analysis reported conflicting results with no obvious explanations. The discrepancy in the results indicates that additional research is required for this important parameter. Overall, they suggested because of developments in adhesive dentistry and the increasing numbers of bond strength studies, uniform guidelines for standardization of the experimental conditions of in-vitro bond strength research is clearly indicated (Finnema et al., 2010). The guidelines from this study were used to select the testing conditions used in this study, including storage medium for teeth, cross-head speed, photopolymerization time, and load cell configuration.

Bishara et al. (2005) attempted to standardize testing conditions when they conducted a study to determine the effect of crosshead speed of the testing machine on shear bond strength while standardizing all other variables. They found changing the crosshead speed from 5.0 to 0.5mm/min increased shear bond strength by approximately 57% and also decreased the ratio of the standard deviation to the mean by half, from 66% to 33%. They also suggested that identifying and standardizing other testing parameters included in shear bond testing would make the results more useful for comparative purposes. Though this study presented some very valuable information regarding the speed at which the crosshead should contact the bracket, it failed to standardize other important variables such as the load cell configuration and photopolymerization time (Bishara et al., 2005).

While depth of cure is an important consideration for the restorative dentist, it is much less of a concern for the orthodontist because the layer of composite that bond brackets to teeth is very thin. Manufacturers have recommended light-curing times of 20-40 second for polymerizing composite restorative materials 2mm thick but the thickness of orthodontic adhesive is considerably less and therefore shorter polymerization times might be adequate (Swanson, Dunn, Childers, & Taloumis, 2004). Swanson *et al.* (2004) demonstrated lower shear bond strengths for 10 second vs. 40 second curing with light emitting diode (LED) light curing units, however, found that all experimental groups had mean SBS's greater than 8MPa. Mavropoulos, A (2005) also found a curing time of 10 seconds to be sufficient to bond metallic brackets to incisors using intensive LED curing units, however, higher curing times (5sec vs 40 sec) did show higher bond strengths. (Mavropoulos, Staudt, Kiliaridis, & Krejci, 2005). Similarily, with a Resin Modified Glass Ionomer Cement (RMGIC) enhanced with 37% phosphoric acid etching and 40 s light-curing time, did not show difference in SBS when the light-curing time was increased, regardless of the acid used (Maruo et al., 2010).

From the studies conducted by Finnema, K.J. (2010) and Bishara *et al.* (2005), it has been suggested to use artificial saliva for the storage medium, photopolymerization times ranging from 20 to 40 seconds, and use cross-head speeds ranging from 0.5mm/min to 5.0mm/min

(Bishara et al., 2005; Finnema et al., 2010). These studies however failed to address the important issue of load cell configuration and from the studies analyzed in Table 2.1 and from Wiltshire, W.A. (2012) load cell configuration is possibly an important factor in shear bond strength testing and should be further investigated (W. A. Wiltshire, 2012a).

The aim of the present study was not to find testing conditions that would produce the highest bond strengths. For example, the goal of this study is not to decrease cross-head speed or load cell configuration and thus achieve higher bond strengths. Instead we desire to find test parameters that are most clinically relevant. Wiltshire W.A. (1994) conducted a study to compare the shear bond strengths of mesh-backed orthodontic buttons bonded to human enamel using a glass ionomer marketed for direct bonding in orthodontics, both in conjunction with, as well as without, enamel etching and to compare the results with a no-mix composite bonding resin. Results showed the no-mix bonding resin had significantly higher shear bond strength than the glass ionomer cement (26.6±6 MPa vs. 4.4±1.8). Enamel etching with 37% orthophosphoric acid increased the mean shear bond strength of the glass ionomer, however, not significantly (5.5±1.8) (W. A. Wiltshire, 1994). Though this study had much lower bond strengths for glass ionomer than resin, stronger bond strengths are not necessarily better, however, clinically relevant bond strengths are most valuable (Kusy, 1994). The requirement is that the bond strength is sufficient to keep the bracket bonded for the duration of orthodontic treatment, yet weak enough to be able to be debonded at the end of treatment, or during a repositioning appointment, without any macro or micro damage to the enamel at debond (W. A. Wiltshire, 2012a).

Table 2.1: Shear bond strength studies showing their test parameters used in present study

Study	Study	Crosshead Speed	Load Cell	Storage Medium	Curing Time	Average SBS ± S.D. (MPa)	Range	Test Condition
(Pinto et al., 2011)	SBS	0.5mm/min	NS	Distilled Water	40 sec	Transbond Resin: 12.70±3.35	NS	Bracket to human enamel
(Phan, Akyalcin, Wiltshire, & Rody, 2011a)	SBS	0.5mm/min	1kN	Saliva	20 sec	24hrs Unbleached: 18.0±4.4	10.13-28.95	Button to human enamel
(Ho ACS, Bonstein T, Akyalcin S, et al., 2010)	SBS	0.5mm/min	1kN	De-ionized Water	20 sec	Tranbond Resin: 16.65±6.04	2.63-26.87	Button to human enamel
(Maruo et al., 2010)	SBS	0.5mm/min	NS	Distilled Water	40 sec	RMGIC: 3.60±0.98	NS	Bracket to human enamel
(Yuasa et al., 2010)	SBS	0.5mm/min	NS	Distilled Water	20 sec	Transbond: 9.8	NS	Bracket to human enamel
(Vilar et al., 2009)	SBS	0.5mm/min	NS	Distilled Water	20 sec	Transbond: 11.22±1.68	NS	Bracket to bovine enamel
(Scougall Vilchis et al., 2009)	SBS	0.5mm/min	NS	Distilled Water	30 sec	Transbond Resin: 19.0±6.7	7.6-29.2	Bracket to human enamel
(Nemeth, Wiltshire, & Lavelle, 2006)	SBS	0.5mm/min	10kN	Distilled Water	30 sec	Transbond Resin: 10.57±2.83	7.10-15.73	Button to human enamel
(Mavropoulos et al., 2005)	SBS	0.5mm/min	NS	Saliva (24hours)	5-40 sec	Resin: 9.5±4.3 – 19.26.8	NS	Bracket to bovine enamel
(Swanson et al., 2004)	SBS	0.5mm/min	NS	Saliva (24hours)	10-40sec	Resin: 8.1±6.3-18.6±5.8	NS	Bracket to human Enamel
(Bishara, Gordan, VonWald, & Jakobsen, 1999)	SBS	0.5mm/min	NS	Distilled Water	20 sec	Transbond: 10.4±2.8	3.4-17.1	Bracket to human enamel
(W. A. Wiltshire, 1994)	SBS	0.5mm/min	20kN	Distilled Water	NS	Resin: 26.6± 6 GI: 5.5±1.8	16-31	Buttons to human enamel
(Summers, Kao, Gilmore, Gunel, & Ngan, 2004)	SBS	1mm/min	NS	Distilled Water	40 sec	Resin: 18.46±2.95	15.44-23.47	Bracket to human enamel
(Abdelnaby & Al-Wakeel Eel, 2010)	SBS	2 mm/min	NS	Distilled Water	20 sec	Transbond Resin: 11.2±3.1	NS	Bracket to human enamel
(Halpern & Rouleau, 2010a)	SBS	2 mm/min	5 kN	Distilled Water	40 sec	Transbond: 7.24	NS	Bracket to human enamel
(Banerjee & Banerjee, 2011)	SBS	5.0mm/min	NS	Saliva	40 sec	Transbond Resin: 14.12	NS	Bracket to human enamel
(Bishara et al., 2005)	SBS	0.5mm/min 5.0mm/min	NS	Distilled Water	20 sec	Transbond Resin: 0.5mm/min: 12.2±4.0 5.0mm/min: 7.0±4.6	2.8-18.5	Bracket to human enamel

NS= Not Stated

From Table 2.1 we can see that there are many similarities and yet many differences that exist within bond strength testing study parameters. To compare each of the above studies is difficult due to the different testing conditions. All of the studies included the crosshead speed of the Universal Testing Machine, storage medium for teeth and curing time for the adhesives used, however, very few studies included the load cell configuration. In fact, the studies which included the load cell configuration were those conducted at the University of Manitoba, Winnipeg, and from these a noticeable difference in bond strengths was observed. Comparing the same adhesives, we can see that when increasing the load cell (1 vs. 5 vs. 10kN) the bond strengths decrease (Ho et al., 2011; Phan, Akyalcin, Wiltshire, & Rody, 2011a)(Halpern & Rouleau, 2010a). Generally, the SBS for crosshead speeds of 0.5mm/min tend to be higher than a crosshead speed of 2-5mm/min (Table 2.1). Photopolymerization time does not show any trend that allows us to make any conclusions regarding SBS's, however, the range for curing times usually varies between 20-40 seconds. Although most studies examined have used distilled water as a storage medium and there is no difference in SBS's, artificial saliva is the preferred storage medium as suggested by the recent systematic review (Finnema et al., 2010). Furthermore, there is an expert who has developed an unique recipe for artificial saliva at the University of Manitoba, Department of Oral Biology (Dr. Colin Dawes) and this storage medium was used in two previous studies at the University of Manitoba (McNeill, Wiltshire, Dawes, & Lavelle, 2001; Phan, Akyalcin, Wiltshire, & Rody, 2011b).

Overall, the need for standardization in testing protocols is emphasized (table 2.1) to obtain valuable interpretation and use of data generated by researchers. Standardization of test protocols will lead to improved standards for orthodontic products and ultimately higher-quality products for orthodontists and their patients and improved interpretation of data from benchside to the clinical situations.

3. PURPOSE

Even with many of the variables accounted for, one has to determine whether the mechanics of the testing itself may influence the results. Therefore the purpose of this study was to determine the effect of changing the crosshead speed of the testing machine, the load cell configuration (a lesser dimension load cell may be more clinically relevant), and the light-curing time, on the shear bond strength of orthodontic brackets while standardizing other variables, such as tooth type, adhesive system, brackets, and debonding time.

4. NULL HYPOTHESIS

1. There is no difference in mean shear bond strength values when changing the cross head speed in orthodontic bonding.
2. There is no difference in mean shear bond strength values when changing the load cell configuration in orthodontic bonding.
3. There is no difference in mean shear bond strength values when changing photopolymerization time in orthodontic bonding.

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 Artificial Saliva

The artificial saliva contained KCl (1.04 g/L), NaH_2PO_4 (0.68 g/L), $NaHCO_3$ (0.42 g/L), $CaCl_2$ (0.03 g/L), and $MgCl_2$ (0.01 g/L). The pH of the artificial saliva was 6.95 and stored in an incubator at 37°C (McNeill et al., 2001). The artificial saliva was made up by Dr. Colin Dawes at the University of Manitoba, Department of Oral Biology.

5.1.3 Orthodontic Buttons

One hundred and sixty curved stainless steel lingual orthodontic buttons (#30-000-01, GAC International, Central Islip, NY) were used with the diameter of 3.31mm (surface area of approximately 8.60 mm^2. The Zwick computer required the input of the surface area of the base of button. With this information, and the load upon failure, it recorded the shear-peel bond strength in MPa (megapascals). Consistency in diameter was achieved by measuring the diameter of 20 buttons and then averaging the diameter.

Table 5.1 Shear Bond Material Manufacturer and Batch Number

Material	Manufacturer	Reference Number	Lot
Bonding Agents			
Transbond XT	3M Unitek, Monrovia, California	712-035	CR3BW
Adhesive paste		712-035	N220837
Primer		712-034	CQ8C1
35% etching gel		712-039 9802	8KY
Bonding Materials			
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		
Copper Rings			
Mini LED Blue Ray - Light curing unit	American Orthodontist	149220-003	
Bosworth Fastray	Bosworth, Illinois	0921375	
Monomer liquid			C13504
Polymer powder			C14783
Debonding Materials			
Universal Testing Machine	Zwick GmBH, Ulm, Germany		
Bencor Multi-T testing apparatus	Danville Engineering, San Ramon, CA		
Chemicals			
Chloramine-T trihydrate 98%	Acros Organics, New Jersey		A0236347
Other			
Digital Caliper	Mastercraft		
Laser Level			
Incubator 37°C	Thelco/Canlab Model 2, Precision Scientific, Chicago, IL		

5.2 Experimental Method

Prior to collecting results for the study, Research ethics board (REB) approval was obtained from the Bannatyne campus University of Manitoba (Appendix 11.1).

5.2.1 Tooth Collection, Storage, and Preparation

One hundred and sixty lower first, second, and third molar teeth were collected from four maxillofacial and oral surgery clinics in Winnipeg and were stored in 0.5% Chloramine T. The criteria for tooth selection included characteristics like: intact buccal enamel, similar buccal surface contour, not subjected to any pretreatment agents, no cracks, and no caries on any surface. Prior to the bonding process, roots of all included teeth were removed using the diamond saw.

5.2.2 Bonding Procedure

All 160 teeth were cleansed and polished with residue free, nonfluoridated, nonflavored pumice (Preppies) in a pumice and water slurry for 10 seconds with a slow speed dental hand piece and rubber prophylactic cup (Nemeth et al., 2006). Teeth were then etched with 37% phosphoric acid gel for 30 seconds and then rinsed with water spray for 20s and dried with an oil-free air spray for 20 seconds until the enamel appeared frosty. Adhesive primer Transbond XT (3M Unitek, Monrovia, California) was applied on the enamel surface with a bond applicator and cured for 20 seconds. The metal lingual buttons were then applied with Transbond XT adhesive paste and 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 20 or 40 seconds, depending on which group they belonged, with LED light. The teeth were stored in artificial saliva for 24 hours in a covered dish at 37°C in an incubator before the tests.

5.2.3 Debonding Procedure

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 copper 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 5.3 and Figure 5.4). 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 Zwick Universal testing machine in a Bencor Multi-T Loading Apparatus (Danville Engineering, San Ramon, CA) (Figure 5.6 and Figure 5.7). A knife-edged shearing blade was used and when a direct sharp shearing force is applied to the enamel-adhesive-bracket interface parallel to the height of contour in an occluso-gingival direction, the button debonds (Figure 5.7). The apparatus was developed in 1989 by Dr. Cornel Driessen at the University of Pretoria, South Africa, and the first article was published in 1994 using this device for orthodontic bond strength testing (W. A. Wiltshire, 1994). The speed of the crosshead was set at 0.5 mm/min and the shear bond strength was measured using a 1KN load cell. The shear bond strength values were recorded by the computer. The same debonding procedures were performed for each of the groups outlined in Table 5-1. In order to change the cross-head speed, the default was changed in the computer and the load was changed by removing the 1kN load cell from the machine and attaching the 10kN load cell. Once the load cell was changed the testing machine was re-calibrated to the new load cell.

Table 5-2: Tooth preparation, load cell configuration (kN) and cross-head speeds (mm/min)

Photopolymerization Time	**Load Cell Configuration**	**Cross-Head Speed (mm/minute)**	**Number of Teeth**
20 second cure	1 kN	0.5	20
		5.0	20
	10 kN	0.5	20
		5.0	20
40 second cure	1 kN	0.5	20
		5.0	20
	10 kN	0.5	20
		5.0	20
		TOTAL	**160**

Figure 5.3: Laser Level with Copper Rings Set Up

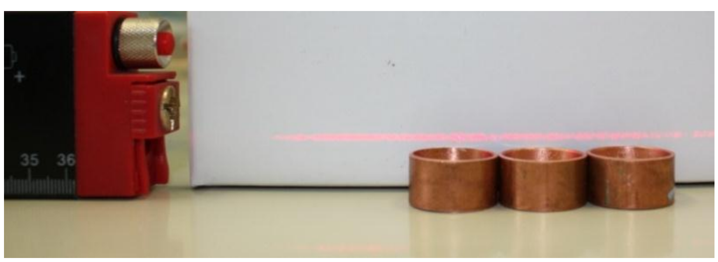


Figure 5.4: Cut Tooth Held by Wax in Copper Ring before Acrylic Placement

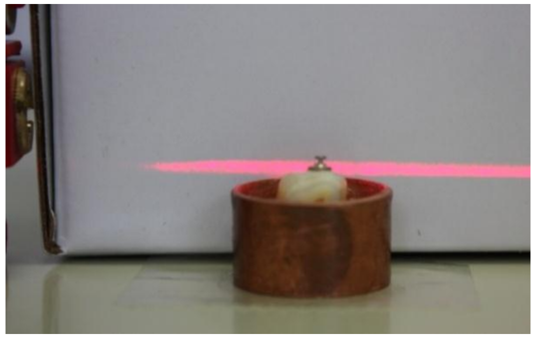


Figure 5.5: Bonded Tooth Embedded in Acrylic Mounted in the Zwick Universal Testing Machine

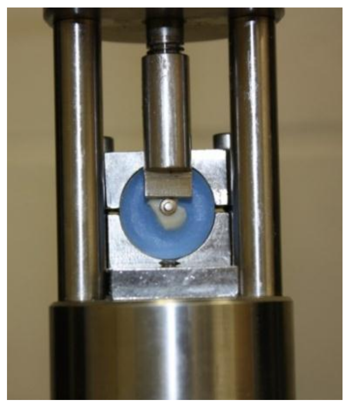
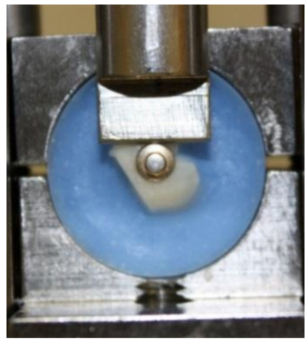


Figure 5.6: Zwick Universal Testing Machine

Figure 5.7: Bencor Multi-T Loading Apparatus with the knife-edged shearing blade in position prior to performing the debond test



5.2.4 Statistical Analysis of Data

All statistical analyses were performed with the Statistical Analysis Software (SAS 9.2 for Windows 10.0.1, SAS Institute Inc., Cary, NC, USA). Descriptive statistics, including the mean, standard deviation, and minimum and maximum values and coefficients of variation were calculated for the groups of teeth tested. Comparisons of means of shear bond strength values were done with Students t-tests. Multiple regression analysis for SBS was also performed. Significance was predetermined at $p \leq 0.05$.

6. RESULTS

6.1 Shear Bond Strengths after 24 hours

The descriptive statistics for each of the test conditions are presented in Table 6.1. Observation and interpretation of the minimum bond strengths, provide clinical meaning to the bond strengths measured during *in vitro* studies. Reynolds (1975) first recommended a minimum value of 6-8 MPa (Reynolds, 1975). However, since then, there have been many advances in materials, computer technology and testing systems. Recently, Wiltshire & Noble (2010) recommended that the minimal reliable clinical bond strength should be at least 3-4 MPa. They decided on this value when evaluating clinical studies *in vivo* on glass ionomers and relating the clinical studies to *in vitro* studies (W. A. Wiltshire & Noble, 2010).

The coefficient of variation was also calculated and presented with the data (Table 6.1). The goal for bonding studies is to achieve a coefficient of variation (standard deviation/mean) in the range of 20-30% (Powers et al., 1997) or even less, if possible (W. A. Wiltshire, 2012b). Results show that all the measured mean bond strengths were above the minimally accepted magnitudes (W. A. Wiltshire & Noble, 2010) and the coefficients of variation were within or close to the acceptable range (Powers et al., 1997).

Table 6.1: Descriptive data of shear bond strengths

Photo-polymerization Time (s)	Load Cell Configuration (kN)	Crosshead Speed (mm/min)	Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Δ max-min	Coefficient of Variation (%)
20	1	0.5	20	23.51	3.36	17.14	30.88	13.74	14.29
		5.0	20	21.88	3.96	14.84	28.84	14.00	18.10
	10	0.5	20	14.32	2.31	9.09	17.89	8.8	16.13
		5.0	20	12.07	3.42	5.97	19.32	13.35	28.33
40	1	0.5	20	22.62	3.01	16.69	29.52	12.83	13.31
		5.0	20	20.01	3.13	14.23	24.92	10.69	15.64
	10	0.5	20	15.05	3.08	9.57	21.14	11.57	20.47
		5.0	20	14.41	4.51	6.28	18.85	12.57	31.30

Judging from Table 6.1 it is evident that with a 1kN load cell configuration, irrespective of photopolymerization time or crosshead speed, the average shear bond strength was in the same order of magnitude (between 20.01MPa and 23.51 MPa) (Refer to table 6.4 for further information). Similarly, judging from table 6.1 it is also evident that with a 10kN load cell configuration, irrespective of photopolymerization time, the average shear bond strength was in the same order of magnitude (between 12.1 MPa and 15.1 MPa) (Refer to Table 6.4 for further information).

Table 6.1 also shows that the minimum acceptable SBS values were achieved with a 10 kN load cell at 20 sec and 40 sec photopolymerization times and 0.5 and 5.0 mm/min crosshead speeds (between 5.97 MPa and 9.57 MPa).

On the other hand, the highest maximum SBS's were recorded in the entire test range with a 1 kN load cell configuration at 20 sec and 40sec photopolymerization times and 0.5 and 5.0 mm/min crosshead speeds (between 24.92 and 30.88 MPa)(Table 6.1).

The coefficient of variation showed that for the 1kN load cell configuration and 0.5mm/min crosshead speeds, the coefficients of variation were the lowest (14.29% and 13.31%). The results under these conditions were the most accurate and had the least spread of values of SBS's around the mean value. In contrast, a load cell configuration of 10kN and 5mm/min crosshead speed resulted in higher coefficient of variations (28.33% and 31.30%) and less accurate representation of overall SBS values.

6.2 Statistical Analysis of subgroups

T-test analysis was performed to determine whether there were significant differences between the tested groups. All significant differences were pre-determined at a probability value of 0.05 or less.

In the photopolymerization subgroup, the overall mean shear bond strengths were 17.95 ± 3.26 MPa and 18.02 ± 3.43 MPa for the 20 sec and 40 sec, respectively. While, the overall mean shear bond strengths were higher for the 40 sec versus the 20 sec curing time group, there was no statistically significant difference ($p > 0.05$) (Table 6.2). Although, the 40 sec test condition showed higher shear bond strengths than the 20 sec group overall, it is clear that when the load cell configuration was 1kN, the 20 sec curing groups showed higher shear bond strengths (23.51 vs. 22.62 and 21.88 vs. 20.01). Similarily, when the load cell configuration was 10kN the 40 sec curing groups showed higher bond strengths (14.32 vs. 15.01 MPa and 12.07vs. 14.41MPa) (Table 6.2).

Table 6.2: Statistical difference between 20 second vs. 40 second photopolymerization time

Load Cell (kN) and Crosshead Speed (mm/min)	Mean ± SD (MPa)		p-value
	Time = 20 sec	Time = 40 sec	
Load = 1, Speed = 0.5	23.51±3.36	22.62±3.01	0.38
Load = 1, Speed = 5.0	21.88±3.96	20.01±3.13	0.11
Load = 10, Speed = 0.5	14.32±2.31	15.05±3.08	0.40
Load = 10, Speed = 5.0	12.07±3.42	14.41±4.51	0.07
Average	17.95 ± 3.26	18.02 ± 3.43	0.88

Meanwhile, in the crosshead speed subgroups, the overall mean SBS were 18.88 ± 2.94 MPa and 17.09 ± 3.76 MPa for 0.5mm/min and 5mm/min, respectively (Table 6.3). Overall, there were no significant differences in crosshead speeds (0.5mm/min vs. 5mm/min) on shear bond strength ($p>0.05$). When comparing the crosshead speeds (0.5mm/min vs. 5.0mm/min) the mean shear bond strengths were higher for the 0.5mm/min subgroup, however, significant differences ($p<0.05$) in crosshead speeds were only found at (time=20, load 10) and (time=40, load=1)(Table 6.3). Evaluation of the confidence intervals (CI) of mean differences proved that there was no significance. Mean differences of (time=20, load=10) = 2.2 with CI (0.4-4.1). Mean differences of (time=40, load=1) = 2.6 with CI (0.6-4.6).

Table 6.3: Statistical differences between crosshead speed 0.5mm/min and 5.0mm/min

Photopolymerization time (sec) and Load Cell (kN)	Mean ± SD (MPa)		p-value
	Speed = 0.5mm/min	Speed = 5.0mm/min	
Time = 20, Load = 1	23.51±3.36	21.88±3.96	0.16
Time = 20, Load = 10	14.32±2.31	12.07±3.42	0.02*
Time = 40, Load = 1	22.62±3.01	20.01±3.13	0.01*
Time = 40, Load = 10	15.05±3.08	14.41±4.51	0.60
Average	18.88 ± 2.94	17.09 ± 3.76	0.12

*$p<0.05$

In the load cell configuration subgroup, the overall mean shear bond strengths were 22.05 ± 3.37 MPa and 13.96 ± 3.33 for the 1kN load cell and 10kN load cell groups. The load cell configuration (1kN vs. 10kN) showed highly statistically significant differences in SBS's when considering crosshead speed and photopolymerization time ($p<0.05$) (Table 6.4).

Table 6.4: Statistical differences between load cell configuration 1kN vs. 10kN

Photopolymerization Time (sec) and Crosshead Speed	Mean ± SD		p-value
	Load=1kN	Load=10kN	
Time=20, Speed=0.5	23.51±3.36	14.32±2.31	*
Time=20, Speed=5	21.88±3.96	12.07±3.42	*
Time=40, Speed=0.5	22.62±3.01	15.05±3.08	*
Time=40, Speed=5	20.01±3.13	14.41±4.51	*
Average	22.05 ± 3.37	13.96 ± 3.33	*

*all values highly significant

Further analysis of the data was attempted using a multiple regression model for SBS, however, this failed to demonstrate any further relationships between the data, yet was able to confirm the findings from the stratified method of statistical analysis.

7. DISCUSSION

This study evaluated the effect of changing the crosshead speed of the testing machine, the load cell configuration, and the light curing time on the shear bond strength of orthodontic brackets while controlling other variables, such as storage medium, tooth type, adhesive system, brackets, and debonding time. Crosshead speed and photopolymerization time were selected based on a systematic review conducted by Finnema (2010) and load cell configuration was selected since the institution where the study was conducted had a 1kN load cell and 10kN load cell and when the two were used in different studies (Ho ACS, Bonstein T, Akyalcin S, et al., 2010; Nemeth et al., 2006; Phan, Akyalcin, Wiltshire, & Rody, 2011b) lower bond strengths were achieved with the 10kN load cell vs. 1kN. The goal for the study was not to achieve the highest bond strengths and the variables that are most important, but, instead find the variables that were most clinically relevant and then test them against each other (W. A. Wiltshire, 2012a). Testing the most clinically relevant variables and determining the most optimal test conditions would allow us to better evaluate orthodontic shear bond strength studies and standardize testing conditions (Fox et al., 1994; W. A. Wiltshire, 2012b). Although, bond strength testing has been conducted for over 40 years, no standardization of testing exists. The literature regarding test condition standardization is limited and typically testing follows manufacturer's recommendations and methodologies used in previous studies. Fox (1994) suggested that researchers should adopt a standard methodological approach that includes tooth type, surface enamel preparation, storage medium, testing equipment and technique, sample size, statistical analysis and bond strength units, however, this study focused on clinically relevant variables (Fox et al., 1994).

7.1 Shear Bond Strength

Overall, the results from this study found that photopolymerization time and crosshead speeds did not affect orthodontic shearbond strength, however, load cell configuration was an important factor of *in-vitro* bond strength testing.

7.2 Photopolymerization Time

Findings indicated that photopolymerization time did not affect the overall shear bond strength. When the curing time was 20 sec the average shear bond strength was lower than the 40 sec subgroup (17.95 ± 3.26 MPa and 18.02 ± 3.43 MPa), however, this was not statistically significant ($p > 0.05$). When analyzing each subgroup separately there was still no statistically significant relationship between 20 and 40 sec curing times. However, mean shear bond strengths were higher when the load cell was 1kN with speeds of 0.5mm/min and 5.0mm/min for 20 sec vs. 40 sec curing times (Table 6.2). On the other hand when a 10kN load cell was used SBS's were higher for 40 sec vs. 20 sec curing times at both speeds of 0.5mm/min and 5.0mm/min. Although, these relationships were not significant they do show that photopolymerization times variably affect orthodontic SBS's and perhaps are not a test factor for times between those ranges. Finnema *et al.* (2010) had found that photopolymerization times significantly affected *in vitro* bond strength and that each additional second of photopolymerization increased bond strength by 0.077 MPa. In our study, for test conditions at 1kN for both crosshead speeds (0.5 and 5.0 mm/min) this was not found to be the case, however, with a 10 kN load cell, this was true. On average, for the 10kN subgroup each additional second of curing increased the SBS's by 0.076 MPa and was almost identical to the value in the systematic review. Although, the systematic review found increasing curing times resulted in increased SBS's, they were unable to suggest the most optimal time for polymerizing the adhesive (Finnema et al., 2010). Swanson (2004) conducted a study comparing different light units (LED and Halogen) while standardizing other test conditions similar to our study. The results from their study found statistically significant differences between 10 sec vs. 40 sec curing times, where 40 sec curing times had higher shear bond strengths (Swanson et al., 2004). Similarly, Mavropoulos (2005) found a curing time of 10 sec was sufficient to bond metallic brackets to incisors using intensive LED curing units, however, 40 sec of curing resulted in higher bond strengths (Mavropoulos et al., 2005). Mauro *et al.* (2010) assessed the influence of etching and light-curing time on the shear bond strength (SBS) of a resin-modified glass ionomer cement (RMGIC) upon debonding of orthodontic brackets. They found RMGIC SBS enhanced with 37% phosphoric acid etching and 40 sec light-curing time, but this did not occur when the light-curing time was increased, therefore light-curing time did not affect SBS (Maruo et al., 2010).

Reynolds (1975) recommended minimum shear bond strength values between 6-8 MPa and all 4 experimental subgroups produced bond strengths that appear to be able to withstand normal orthodontic forces (Reynolds, 1975). Results of this study also demonstrated higher bond strengths as curing time was increased. This can easily be explained by the higher conversion rate of monomer to polymer at increased polymerization times (Mavropoulos et al., 2005). The data of this *in vitro* study should be compared only with groups in the study, and the laboratory findings should not be extrapolated to the clinical situation. Studies appear to indicate that mean bond strengths recorded *in vivo* following comprehensive orthodontic treatment are significantly lower than bond strengths recorded in vitro (Pickett, Sadowsky, Jacobson, &

Lacefield, 2001). However, *in vitro* studies are an important first step prior to confirming any clinical situation.

7.3 Crosshead Speed

The findings of this study indicated that variations in the crosshead speed of the testing machine can influence the test results; however, this was not significant overall. More specifically, by slowing the crosshead speed of the Zwick machine during shear bond testing of the orthodontic brackets from 5.0mm/min to 0.5mm/min, the mean shear bond strength increased from 17.09 to 18.88 MPa, an increase of only 9.4%. In other words crosshead speed did not affect the shear bond strength. Klocke *et al.* (2005) investigated the influence of cross-head speed on debonding force of orthodontic brackets and selected speed of 0.1mm/min, 0.5mm/min, 1mm/min and 5.0mm/min. Their results, similar to those from the present study, showed cross-head speed variation between 0.1 and 5 mm/min did not seem to influence debonding force measurements of brackets bonded to enamel with a composite adhesive (Klocke & Kahl-Nieke, 2005a). Bishara *et al.* (2005) conducted a similar study where they tested the effect of changing crosshead speed from 5.0 to 0.5mm/min and their results did not support the present findings. Their study found that changing the crosshead speed from 5.0 to 0.5 mm/min significantly increased the shear bond strength from 7.0 to 12.2 MPa, an increase of 57%. Furthermore, the ratio between the mean standard deviation for 5.0mm/min testing speed was 66%, whereas for the slower 0.5mm/min testing speed, it was 33%. In other words, there was an increase in the shear bond strength values and a decrease in relative variation (Bishara et al., 2005). Eliades *et al.* (2004) examined the effect of crosshead speed on the bond strength of brackets bonded to enamel. Crosshead speeds of a standard 1 mm/min and a fast 200 mm/min, which better approximates the actual jaw velocity during chewing, were selected. The results indicated that an increased crosshead speed resulted in decreased shear bond strength. Furthermore, they stated that this was probably due to the induction of a stiff body response and elimination of the visoelastic properties of the resin (Eliades, Katsavrias, Zinelis, & Eliades, 2004). Finnema *et al* (2010) reported in her systematic review that an increase in crosshead speed of 1mm/min yielded an increase in average bond strength of 1.3 MPa. From the studies presented in Table 2.1 it is evident that crosshead speeds of 0.5mm/min (8.1-26.6 MPa) typically produced higher shear bond strengths than those at higher crosshead speeds (7.0-18.46 MPa) (Refer to Table 2.1 in introduction chapter). Also, from the studies in Table 2.1 we can see that most studies use 0.5mm/min as their crosshead speeds since this produces more consistent results. The consistency of results was evident in our study if you examine the coefficients of variation. From Table 6.1 we can see at 0.5mm/min there was less variation of the shear bond strength values (13.31-20.47%) compared to 5.0mm/min (15.64-31.30%). This shows that our results at 0.5mm/min are more accurate and there is more confidence regarding the shear bond strength values.

An interesting issue raised by Eliades (2004) was regarding the clinical relevance of the reported bond strength values and how they come in to question since the standard crosshead speeds cited in the literature are irrelevant to the velocity of tooth occlusion during chewing. Although the reported crosshead speeds range from 0.1mm/min-200mm/min, it must be noted that these rates are many orders of magnitude lower than the actual masticatory velocity in humans. A complete masticatory cycle (sequential opening and closing) of a healthy individual lasts 800ms, with the closing movement having duration of less than 400 ms, which translates to over 2000mm/min and this value is much higher than the standard crosshead speed used in bond strength testing. However, using crosshead speeds observed in human chewing cycles is not practical in laboratory testing since testing is restricted to the upper limit of the effective crosshead speed of the testing apparatus (Eliades et al., 2004). The present study aims to find test conditions that are clinically relevant; however, when standardizing crosshead speed in the laboratory we demonstrated this is not relevant. That said, it is difficult, if not impossible to reproduce and replicate the actual clinical situation *in vitro*. Seldom, if ever, for example, is a sustained, perfect shear force produced on an orthodontic bracket and furthermore, replication of the human chewing cycle has not been effectively mimicked intra-orally (W. A. Wiltshire, 2012a).

7.1.3 Load Cell Configuration

The third experimental condition shown to significantly affect outcomes of bond strength testing was load cell configuration. An increase in load cell configuration from 1kN to 10kN resulted in significantly lower bond strengths (22.05 vs. 13.96MPa) ($p<0.05$). Even for all 4 subgroups tested, increases in load cell configuration resulted in significantly lower bond strengths ($p<0.01$). To date no study has been conducted that supports or refutes the findings of this test condition. In fact, reporting of the load cell configuration is limited (Halpern & Rouleau, 2010c; Ho ACS, Bonstein T, Akyalcin S, et al., 2010; Phan, Akyalcin, Wiltshire, & Rody, 2011a; W. A. Wiltshire, 1994) and has not been considered a factor in the majority of the bond strength testing literature. As mentioned previously, Wiltshire (2012) felt this was an important test factor since the Department of Orthodontics, University of Manitoba, Canada, had two different load cells (1kN and 10kN) that were producing different results under similar test conditions. Ho *et al.* (2010) and Phan (2011) conducted studies using similar test conditions as the present study (1kN load cell, 0.05mm/min crosshead speed, 20sec curing time) and the results showed shear bond strengths of 16.7 and 18.0 MPa, whereas our study had a bond strength of 23.51MPa (Ho ACS, Bonstein T, Akyalcin S, et al., 2010; Phan, Akyalcin, Wiltshire, & Rody, 2011a). Comparing these studies with Nemeth (2006) who used a 10kN load cell, 0.05mm/min crosshead speed and 30sec curing time and found a shear bond strength of 10.6Mpa, we can note that the shear bond strengths are lower when the load cell configuration is increased (Nemeth et al., 2006). Similarly, Halpern (2010) used a 5kN load cell, with a 2mm/min crosshead speed and 40 second curing time and found an even lower bond strength (7.24MPa) than the above studies at 1kN load cell configuration(Halpern & Rouleau, 2010b). On the other hand, when Wiltshire (1994) used a 20kN load cell with a 0.05mm/min crosshead speed and 40sec curing time, the shear bond strength was much higher (resin: 26.6MPa) than all the studies mentioned above. However, it must be noted that the same resin (Tranbond XT, 3M Unitek, Monrovia, CA) was not used and could be the reason for the major difference in shear bond strengths (W. A. Wiltshire, 1994). With respect to occlusal forces of mastication generated in the human, one might argue in favor of higher load cell usage. On the flip side, the lighter load cell may improve the test sensitivity.

The results of the study also found that the coefficients of variation, a measure of the variability about the mean, were highest for the 10kN load cell and 5.0mm/min crosshead speed (28.33 and 31.30%) and this was above the recommended 20-25% (W. A. Wiltshire, 2012b). Furthermore, the lowest coefficient of variation was achieved when a more sensitive 1kN load and slower crosshead speed of 0.05mm/min were used (14.29 and 13.31%). This demonstrates that when using a 1kN load cell the results are more consistent and uniform than for a 10kN load cell. Lighter load cells tend to produce higher bond strengths and higher bond strengths are what manufacturers may be interested in, in order to market their products. If adhesives display higher bond strengths, the clinician may assume that this results in fewer debonds and may be more inclined to purchasing that product. In order to be more stringent it would make more sense to use a heavier load cell which would result in lower bond strengths, but may be a better indicator of the clinical applicability of a product. Independent researchers may be more in favor of using a higher load cell configuration and manufacturers may use a lower load cell configuration, but, overall, from this study we suggest to use both 1 and 10 kN load cells. Results would be more uniform with a 1kN load cell but a 10kN load cell would produce lower values.

The major finding of this study shows that load cell configuration is an important test parameter that rarely appears in the literature. In order to adequately compare products this parameter needs to be stated otherwise studies cannot be accurately evaluated and clinicians may have higher expectations of an orthodontic adhesive.

7.5 Limitations of the Study

To our knowledge, this is the first investigation on the influence of load cell configuration in orthodontic bond strength testing. In contrast, other studies have been conducted on crosshead speeds and photopolymerization time (Banerjee & Banerjee, 2011; Bishara et al., 2005; Eliades, Eliades, Bradley, & Watts, 2000; Eliades et al., 2004; Klocke & Kahl-Nieke, 2005a; Maruo et al., 2010; Mavropoulos et al., 2005; Summers et al., 2004; Swanson et al., 2004). That being said, during shear bond strength testing cross head speed and load cell may influence the adhesive interface in a similar way. Higher crosshead speeds can be similar to heavier load cells and lower cross head speeds may act like lighter load cells. Assuming the two parameters behave similarly, Hara *et al.* (2001) found that cross-head speeds of 0.50 and 0.75mm/min produce more adhesive failure and those are preferable in SBS testing. They further stated that relatively high cross-head speeds may develop abnormal stress distributions during the shear testing, including cohesive failures in the tooth substrate or in the resin-based composite which would influence the bond strength values achieved (Hara, Pimenta, & Rodrigues, 2001). For adhesive failures elastic behavior of the adhesive resin might increase the shear bond strength and slower crosshead speeds may allow an extended recovery period during which stress and strain are compensated for by the elasticity. At lower speeds the resin behaves like a viscous material, deforming more as pressure is applied, where at higher impact velocity this phenomenon plays a minor role, resulting in lower shear bond strengths (Lindemuth, J.S. 2000). Overall, slower crosshead speeds and lighter load cells may increase the shear bond strength values and higher crosshead speeds and heavier load cells may decrease the shear bond strength values. This study was not interested in determining the Adhesive Remnant Index (ARI) or the fracture rate, however, we did not observe any enamel fractures and all debonds were adhesive or cohesive.

The present study aimed to ensure the debonding force location was at the enamel/adhesive interface and not on the bracket. This was done using a Bencor Multi-T Loading Apparatus with the knife-edged shearing blade. The blade was in a perpendicular position prior to performing the debond test. Although care was taken to ensure the location was consistent, this could have varied. Debonding force location can have a significant influence on shear bond strength measurements and bond failure pattern. Mean shear bond strengths are higher when the force is applied closer to the tooth adhesive interface than further away (Klocke & Kahl-Nieke, 2005b).

8. CONCLUSIONS

Based on this *in vitro* study on the SBS of when changing test parameters of photopolymerization time, crosshead speed, and load cell configuration, we can conclude that:

1. Photopolymerization time does not affect orthodontic SBS testing
2. Crosshead speed does not affect orthodontic SBS testing
3. Load cell configuration does affect orthodontic SBS testing, where the more sensitive 1kN load cell configuration produced higher bond strengths

9. RECOMMENDATIONS

- Future studies on orthodontic bond strength testing should state the load cell used.
- 1 kN and 10kN load cells used in together will be the most accurate evaluation method.
- Bond strength testing of new products is an important method for the clinician to estimate the efficacy of new adhesives and clinicians should overview the results of such studies prior to product selection.
- Independent and unbiased University-based studies are urged when new products enter the marketplace.

10. RAW DATA

Table 10-1: Raw data for 24 hour shear bond strengths

Photopolymertization time	Load Cell Configuration	Cross-Head Speed	Shear Bond Strength
20	1	0.5	23.91
20	1	0.5	18.72
20	1	0.5	26.22
20	1	0.5	24.81
20	1	0.5	30.88
20	1	0.5	24.02
20	1	0.5	17.14
20	1	0.5	22.29
20	1	0.5	18.71
20	1	0.5	21.52
20	1	0.5	26.31
20	1	0.5	25.37
20	1	0.5	28.11
20	1	0.5	24.18
20	1	0.5	20.73
20	1	0.5	25.32
20	1	0.5	23.58
20	1	0.5	23.63
20	1	0.5	24.88
20	1	0.5	19.94
20	1	5	21.22
20	1	5	23.28
20	1	5	28.84
20	1	5	16.67
20	1	5	12.34
20	1	5	24.28
20	1	5	22.76
20	1	5	25.21
20	1	5	20.2
20	1	5	21.38
20	1	5	24.46
20	1	5	27.61
20	1	5	14.84
20	1	5	18.91
20	1	5	23.66
20	1	5	23.04
20	1	5	22.87

20	1	5	21.7
20	1	5	20.64
20	1	5	23.72
20	10	0.5	15.04
20	10	0.5	11.5
20	10	0.5	15.8
20	10	0.5	15.41
20	10	0.5	13.32
20	10	0.5	10.7
20	10	0.5	13.27
20	10	0.5	17.49
20	10	0.5	10.78
20	10	0.5	15.2
20	10	0.5	16.13
20	10	0.5	14.79
20	10	0.5	17.89
20	10	0.5	9.09
20	10	0.5	16.32
20	10	0.5	15.4
20	10	0.5	13.18
20	10	0.5	15.32
20	10	0.5	15.09
20	10	0.5	14.72
20	10	5	13.1
20	10	5	9.75
20	10	5	19.32
20	10	5	7.37
20	10	5	5.97
20	10	5	10.64
20	10	5	6.21
20	10	5	8.28
20	10	5	11.31
20	10	5	10.64
20	10	5	15.83
20	10	5	14.34
20	10	5	13.88
20	10	5	13.27
20	10	5	11.49
20	10	5	11.86
20	10	5	13.99
20	10	5	14.91

20	10	5	15.61
20	10	5	13.72
40	1	0.5	23.8
40	1	0.5	18.67
40	1	0.5	20.5
40	1	0.5	21.95
40	1	0.5	21.88
40	1	0.5	16.69
40	1	0.5	26.69
40	1	0.5	22.42
40	1	0.5	23.41
40	1	0.5	24.52
40	1	0.5	29.52
40	1	0.5	18.69
40	1	0.5	24.41
40	1	0.5	25.44
40	1	0.5	21.29
40	1	0.5	20.85
40	1	0.5	25.73
40	1	0.5	23.22
40	1	0.5	22.14
40	1	0.5	20.65
40	1	5	18.38
40	1	5	20.65
40	1	5	21.92
40	1	5	24.92
40	1	5	19.3
40	1	5	20.32
40	1	5	18.22
40	1	5	17.53
40	1	5	18.92
40	1	5	14.23
40	1	5	15.97
40	1	5	16.46
40	1	5	24.87
40	1	5	23.25
40	1	5	19.14
40	1	5	22.5
40	1	5	15.65
40	1	5	21.87
40	1	5	23.25

40	1	5	22.84
40	10	0.5	21.14
40	10	0.5	20.73
40	10	0.5	18.11
40	10	0.5	17.22
40	10	0.5	10.81
40	10	0.5	18.97
40	10	0.5	13.46
40	10	0.5	15.34
40	10	0.5	14.92
40	10	0.5	17.5
40	10	0.5	9.57
40	10	0.5	12.34
40	10	0.5	12.99
40	10	0.5	15.02
40	10	0.5	14.76
40	10	0.5	13.75
40	10	0.5	12.79
40	10	0.5	13.27
40	10	0.5	15.02
40	10	0.5	13.38
40	10	5	15.46
40	10	5	25.16
40	10	5	17.23
40	10	5	14.43
40	10	5	18.85
40	10	5	20.29
40	10	5	13.73
40	10	5	22.13
40	10	5	11.56
40	10	5	12.97
40	10	5	12.48
40	10	5	11.76
40	10	5	13.79
40	10	5	12.52
40	10	5	12.56
40	10	5	6.28
40	10	5	9.16
40	10	5	15.75
40	10	5	11.79
40	10	5	10.38

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12. APPENDIX

12.1 Ethics Approval

UNIVERSITY OF MANITOBA

BANNATYNE CAMPUS
Research Ethics Boards

P126-770 Bannatyne Avenue
Winnipeg, Manitoba
Canada R3E 0W3
Tel: (204) 789-3255
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APPROVAL FORM

Principal Investigator: Dr. V. Cheba

Ethics Reference Number: H2010:394
Date of Approval: November 25, 2010
Date of Expiry: November 25, 2011

Protocol Title: The Effect of Cross-Head Speed, Load Cell Weight, and Preparation of Enamel on Shear Bond Strength of Orthodontic Brackets

The following is/are approved for use:

- **Proposal submitted November 18, 2010**

The above underwent expedited review and was **approved as submitted** on November 25, 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 November 18, 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 form the relevant institution, if required.

Sincerely yours,

[redacted]

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

www.umanitoba.ca/medicine/ethics

12.2 Journal Approval

RE: Affiliate Academic papers- Angle East 2013 Page 1 of 3

RE: Affiliate Academic papers- Angle East 2013

מירי - DR. HAISRAELI SHALISH MIRIAM [Mshalish@hadassah.org.il]

Sent: 05 June 2012 07:58

To: Billy Wiltshire

Cc: CTrotman@umaryland.edu; santoro.orthodontics@gmail.com; drAr@i.email.ne.jp; pngan@hsc.wvu.edu

Dear Billy,

Thank you for sending me the abstract.

I am glad to let you know that the academic committee members approved the topic and the outline of your paper.
We will wait for the submission of your paper by **November 15, 2012.**

Warm regards,

Miri

Dr. Miri Haisraeli-Shalish

Chair, Academic Committee, Angle East

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11.3 Journal Article Submitted to Angle Orthodontics

The effect of crosshead speed, load cell configuration and curing time on the shear bond strength of orthodontic brackets

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Abstract

Objective: The purpose of this study was to evaluate the effect of crosshead speed, load cell configuration and curing time on the shear bond strength of orthodontic bracket

Materials and Methods: One hundred and eighty human molars were randomly divided into two equal groups of 20 sec and 40 sec photopolymerization time and stored in artificial saliva at 37°C in incubator for two weeks before bonding. The teeth were bonded using regular primer and Transbond XT (R) adhesive (3M Uniktek). Each group was further divided into 1kN or 10kN load cell groups and each of those groups were divided into 0.5mm/min or 5mm/min crosshead speeds. The test assemblies were subjected to shear-testing 24 hours after bonding using a Zwick Universal Test Machine in a Bencor Multi-T testing castle.

Results: In terms of the photopolymerization(Sfondrini, Cacciafesta, Scribante, Boehme, & Jost-Brinkmann, 2006) time (20sec vs. 40sec) there were no significant differences in SBSs considering load cell configuration and crosshead speeds ($p>0.05$). When comparing the crosshead speeds (0.5mm/min vs. 5.0mm/min) there were significant differences ($p<0.05$) in crosshead speeds at (time=20, load 10) and (time=40, load=1). However, evaluation of the confidence intervals (CI) of mean differences, there was no significance. Overall, there were no significant differences in crosshead speeds (0.5mm/min vs. 5mm/min) on SBS's. The load cell configuration (1kN vs. 10kN) showed statistically significant differences in SBS's when considering crosshead speeds and photopolymerization time ($p<0.05$). In the load cell configuration subgroup, shear bond strengths attained were 21.11 ± 3.47 MPa and 13.9±3.33 MPa for 1kN and 10kN load cells respectively.

Conclusion: Changing the crosshead speeds and photopolymerization time does not affect the shear bond strengths. However, the more sensitive 1kN load cell configuration produced higher bond strengths.

Introduction

Since the introduction of different bracket adhesive materials to the orthodontic market, both *in vivo* and *in vitro* studies have been conducted to test their effectiveness and working characteristics. As new bonding agents have been introduced, research focused on this area and the resultant publication rate of papers on orthodontic bonding increased considerably. This is illustrated in the steadily increasing number of bonding papers appearing in the leading orthodontic journals (Eliades & Brantley, 2000).

Orthodontic adhesive materials have mainly been subjected to shear bond strength studies. The results obtained from these studies show the strength of the adhesive between the tooth and bracket. Typically higher bond strengths indicate a better adhesive material. To conduct the actual tests, brackets are bonded to extracted teeth, tightly secured into a tooth test ring using acrylic and then mounted onto a testing machine. The machine contains a steel rod attached to a crosshead and once the rod is activated it contacts the mounted bracket and shears it off. A computer electronically connected to the Universal Test Machine records the strength of the adhesive of each test in megapascals. The speed of the crosshead and load cell weight can be changed and as a result may affect the overall results (Eliades & Brantley, 2000).

Although many bonding studies have been conducted, there lacks a universally accepted protocol to conduct these studies. As a result, it is difficult and sometimes impossible to compare the materials and techniques used in the studies as well as the results within themselves (Bishara, Soliman, Laffoon, & Warren, 2005). Fox (1994) found that even changing one test parameter could significantly affect the interpretation of the results. In addition, it was observed that there was a large variation in the methodology used for orthodontic material testing of bond strength. From this it was suggested that researchers should adopt a standard methodological approach that included tooth type, surface enamel preparation, storage medium, testing equipment and technique, sample size, statistical analysis, and bond strength units (Fox, McCabe, & Buckley, 1994). Finnema et al. (2010) reported that many studies on *in vitro* orthodontic bond strength fail to report test conditions that could significantly affect their outcome. In their systematic review and meta-analysis, a summary of factors is given that can affect the *in vitro* bond strength of orthodontic brackets. Experimental conditions that significantly influence in-vitro bond strength are water storage of the bonded specimens, photopolymerization time, and crosshead speed (Finnema, Ozcan, Post, Ren, & Dijkstra, 2010).

New dental products are constantly being introduced to the orthodontic market and continually being tested for their effectiveness. In order for clinicians to critically evaluate new products and better serve their patients, it remains essential that thorough testing of materials new to the market be tested by independent sources, in addition to the potentially biased "in house" tests undertaken by manufacturers. Materials need to bond sufficiently strongly, yet not too strong to cause enamel damage on debonding, the bond should not deteriorate during orthodontic treatment over time, it should be non-toxic and non-allergenic and preferably anticariogenic. Only eventual standardized testing of orthodontic products will allow accurate evaluation, comparison and full disclosure scrutiny of new and evolving products (W. A. Wiltshire, 2012a).

Finemma (2010) conducted a systematic review of the available literature regarding *in-vitro* orthodontic shear bond strength testing. To date this publication has been the most comprehensive review of bond strength testing conditions. Results from this paper showed bond strength testing was negatively influenced when the teeth were stored in water. Water storage on average decreased bond strength by 10.7 MPa, assuming that the other predictors remain constant. Although this was the most pronounced effect of an experimental condition, this finding was mainly influenced by 1 relatively large study sample in which artificial saliva was used as a storage medium for specimens. It was reported that bond strengths were significantly higher when teeth were stored in artificial saliva. Analogously, each second of photopolymerization time increased the bond strength by 0.077 MPa. The studies in the meta-analysis showed considerable variations in photopolymerization time: from 2 to 50 seconds, however, most studies reported 40 seconds for polymerizing adhesive and this corresponds to the routine clinical standard. Although the results indicated increasing photopolymerization time yields higher bond strengths, the most optimal time for polymerizing still needs to be deduced in future studies. When crosshead speed increased by 1 mm per minute, bond strength increased by 1.3 MPa. However, the studies used in the meta-analysis reported conflicting results with no obvious explanations. The discrepancy in the results indicates that more research is required for this important parameter. Overall, they suggested because of developments in adhesive dentistry and the increasing numbers of bond strength studies, uniform guidelines for standardization of the experimental conditions of in-vitro bond strength research is clearly indicated(Finnema et al., 2010). The guidelines from this study were used to select the testing conditions used in this study, including storage medium for teeth, cross-head speed, photopolymerization time, and load cell configuration.

Bishara et al. (2005) attempted to standardize testing conditions when they conducted a study to determine the effect of crosshead speed of the testing machine on shear bond strength while standardizing all other variables. They found changing the crosshead speed from 5.0 to 0.5mm/min increased shear bond strength by approximately 57% and also decreased the ratio of the standard deviation to the mean by half, from 66% to 33%. They also suggested that identifying and standardizing other testing parameters included in shear bond testing would make the results more useful for comparative purposes. Though this study presented some very valuable information regarding the speed at which the crosshead should contact the bracket, it failed to standardize other important variables such as the load cell configuration and photopolymerization time (Bishara et al., 2005).

While depth of cure is an important consideration for the restorative dentist, it is much less of a concern for the orthodontist because the layer of composite that bond brackets to teeth is very thin. Manufacturers have recommended light-curing times of 20-40 second for polymerizing composite restorative materials 2mm thick but the thickness of orthodontic adhesive is considerably less and therefore shorter polymerization times might be adequate (Swanson, Dunn, Childers, & Taloumis, 2004) Swanson *et al.* (2004) demonstrated lower shear bond strengths for 10 second vs. 40 second curing with light emitting diode (LED) light curing units, however, found that all experimental groups had mean SBS's greater than 8MPa. Mavropoulos, A (2005) also found a curing time of 10 seconds to be sufficient to bond metallic brackets to incisors using intensive LED curing units, however, higher curing times (5sec vs 40 sec) did show higher bond strengths. (Mavropoulos, Staudt, Kiliaridis, & Krejci, 2005).

From the studies conducted by Finnema, K.J. (2010) and Bishara *et al.* (2005), it has been suggested we use artificial saliva for the storage medium, photopolymerization times ranging from 20 to 40 seconds, and use cross-head speeds ranging from 0.5mm/min to 5.0mm/min (Bishara et al., 2005; Finnema et al., 2010). These studies however failed to address the important issue of load cell configuration and we have added this parameter in our present study since we believe that increasing the load cell would reduce the bond strengths (W. A. Wiltshire, 2012a)

Even with many of the variables accounted for, one has to determine whether the mechanics of the testing itself may influence the results. Therefore the purpose of this study is to determine the effect of changing the crosshead speed of the testing machine, the load cell configuration (a lesser dimension load cell may be more clinically relevant), and the light-curing time, on the shear bond strength of orthodontic brackets while standardizing other variables, such as tooth type, adhesive system, brackets, and debonding time.

Materials and Methods:

One hundred and sixty lower first, second, and third molar teeth were collected from four maxillofacial and oral surgery clinics in Winnipeg and were stored in 0.5% Chloramine T. The criteria for tooth selection included characteristics like: intact buccal enamel, similar buccal surface contour, not subjected to any pretreatment agents, no cracks, and no caries on any surface. Prior to the bonding process, roots of all included teeth were removed using the diamond saw.

All 160 teeth were cleansed and polished with residue free, nonfluoridated, nonflavored pumice (Preppies) in a pumice and water slurry for 10 seconds with a slow speed dental hand piece and rubber prophylactic cup. Teeth were then etched with 37% phosphoric acid gel for 30 seconds and then rinsed with water spray for 20s and dried with an oil-free air spray for 20 seconds until the enamel appeared frosty. Adhesive primer Transbond XT (3M Unitek, Monrovia, California) was applied on the enamel surface with a bond applicator and cured for 20 seconds. The metal lingual buttons were then applied with Transbond XT adhesive paste and placed on the etched enamel surface. One hundred and sixty curved stainless steel lingual orthodontic buttons (#30-000-01, GAC International, Central Islip, NY) were used with the diameter of 3.31mm (surface area of approximately 8.60 mm^2). 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 20 or 40 seconds, depending on which group they belonged, with LED light. The teeth were stored in artificial saliva (McNeill et al., 2001) for 24 hours in a covered dish at 37°C in an incubator before the tests.

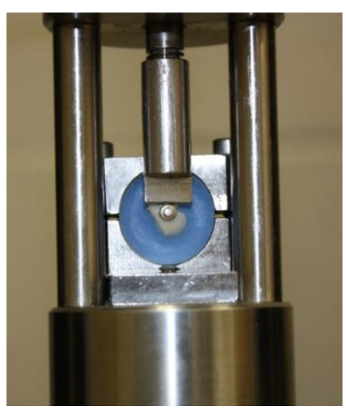
Debonding Procedure

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 copper 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. 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 Zwick Universal testing machine in a Bencor Multi-T Loading Apparatus (Danville Engineering, San Ramon, CA) (Figure 1). A knife-edged shearing blade was used and when a direct sharp shearing force is applied to the enamel-adhesive-bracket interface parallel to the height of contour in an occluso-gingival direction, the button debond. The speed of the crosshead was set at 0.5 mm/min and the shear bond strength was measured using a 1KN load cell. The shear bond strength values were recorded by the computer. The same debonding procedures were performed for each of the groups outlined in Table 5-1. In order to change the cross-head speed, the default was changed in the computer and the load cell configuration was changed by removing the 1kN load cell from the machine and attaching the 10kN load cell. Once the load cell was changed the testing machine was re-calibrated to the new load cell.

Table 1: Tooth preparation, load cell configuration (kN) and cross-head speeds (mm/min)

	Photopolymerization Time	**Load Cell Configuration**	**Cross-Head Speed (mm/minute)**	**Number of Teeth**
1	20 second cure	1 kN	0.5	20
			5.0	20
2		10 kN	0.5	20
			5.0	20
3	40 second cure	1 kN	0.5	20
			5.0	20
4		10 kN	0.5	20
			5.0	20
			TOTAL	**160**

Figure 1: Bonded Tooth Embedded in Acrylic Mounted in the Zwick Universal Testing Machine



Statistical Analysis of Data

All statistical analyses were performed with the Statistical Analysis Software (SAS 9.2 for Windows 10.0.1, SAS Institute Inc., Cary, NC, USA). Descriptive statistics, including the mean, standard deviation, and minimum and maximum values and coefficients of variation were calculated for the groups of teeth tested. Comparisons of means of shear bond strength values were done with Students t-tests. Significance was predetermined at $p \leq 0.05$.

Results

Table 2 : Descriptive data of shear bond strengths

Photo-polymerization Time (s)	Load Cell Configuration (kN)	Crosshead Speed (mm/min)	Sample Size (N)	Mean (MPa)	Std Dv	Min (MPa)	Max (MPa)	Δ max-min	Coefficient of Variation (%)
20	1	0.5	20	23.51	3.36	17.14	30.88	13.74	14.29
		5.0	20	21.88	3.96	14.84	28.84	14.00	18.10
	10	0.5	20	14.32	2.31	9.09	17.89	8.8	16.13
		5.0	20	12.07	3.42	5.97	19.32	13.35	28.33
40	1	0.5	20	22.62	3.01	16.69	29.52	12.83	13.31
		5.0	20	20.01	3.13	14.23	24.92	10.69	15.64
	10	0.5	20	15.05	3.08	9.57	21.14	11.57	20.47
		5.0	20	14.41	4.51	6.28	18.85	12.57	31.30

Judging from Table 2 it is evident that with a 1kN load cell configuration, irrespective of photopolymerization time or crosshead speed, the average shear bond strength was in the same order of magnitude (between 20.01MPa and 23.51 MPa) (Refer to table 3 for further information). Similarly, judging from table 2 it is also evident that with a 10kN load cell configuration, irrespective of photopolymerization time, the average shear bond strength was in the same order of magnitude (between 12.1 MPa and 15.1 MPa) (Refer to Table 3 for further information).

Table 2 also shows that the minimum acceptable SBS values were achieved with a 10 kN load cell at 20 sec and 40 sec photopolymerization times and 0.5 and 5.0 mm/min crosshead speeds (between 5.97 MPa and 9.57 MPa).

On the other hand, the highest maximum SBS's were recorded in the entire test range with a 1 kN load cell configuration at 20 sec and 40sec photopolymerization times and 0.5 and 5.0 mm/min crosshead speeds (between 24.92 and 30.88 MPa)(Table 2).

The coefficient of variation showed that for the 1kN load cell configuration and 0.5mm/min crosshead speeds, the coefficients of variation were the lowest (14.29% and 13.31%). The results under these conditions were the most accurate and had the least spread of values of SBS's around the mean value. In contrast, a load cell configuration of 10kN and 5mm/min crosshead speed resulted in higher coefficient of variations (28.33% and 31.30%) and less accurate representation of overall SBS values.

6.2 Statistical Analysis of subgroups

T-test analysis was performed to determine whether there were significant differences between the tested groups. All significant differences were pre-determined at a probability value of 0.05 or less.

In the photopolymerization subgroup, the overall mean shear bond strengths were 17.95 ± 3.26 MPa and 18.02 ± 3.43 MPa for the 20 sec and 40 sec, respectively. While, the overall mean shear bond strengths were higher for the 40 sec versus the 20 sec curing time group, there was no statistically significant difference ($p > 0.05$) (Table 3). Although, the 40 sec test condition showed higher shear bond strengths than the 20 sec group overall, it is clear that when the load cell configuration was 1kN, the 20 sec curing groups showed higher shear bond strengths (23.51 vs. 22.62 and 21.88 vs. 20.01). Similarily, when the load cell configuration was 10kN the 40 sec curing groups showed higher bond strengths (14.32 vs. 15.01 MPa and 12.07vs. 14.41MPa) (Table 3).

Table 3: Statistical difference between 20 sec vs. 40 sec photopolymerization time

Load Cell (kN) and Crosshead Speed (mm/min)	**Mean ± SD (MPa)**		**p-value**
	Time = 20 sec	**Time = 40 sec**	
Load = 1, Speed = 0.5	23.51±3.36	22.62±3.01	0.38
Load = 1, Speed = 5.0	21.88±3.96	20.01±3.13	0.11
Load = 10, Speed = 0.5	14.32±2.31	15.05±3.08	0.40
Load = 10, Speed = 5.0	12.07±3.42	14.41±4.51	0.07
Average	17.95 ± 3.26	18.02 ± 3.43	0.88

Meanwhile, in the crosshead speed subgroups, the overall mean SBS were 18.88 ± 2.94 MPa and 17.09 ± 3.76 MPa for 0.5mm/min and 5mm/min, respectively (Table 4). Overall, there were no significant differences in crosshead speeds (0.5mm/min vs. 5mm/min) on shear bond strength ($p>0.05$). When comparing the crosshead speeds (0.5mm/min vs. 5.0mm/min) the mean shear bond strengths were higher for the 0.5mm/min subgroup, however, significant differences ($p<0.05$) in crosshead speeds were only found at (time=20, load 10) and (time=40, load=1)(Table 4). Evaluation of the confidence intervals (CI) of mean differences proved that there was no significance. Mean differences of (time=20, load=10) = 2.2 with CI (0.4-4.1). Mean differences of (time=40, load=1) = 2.6 with CI (0.6-4.6).

Table 4: Statistical differences between crosshead speed 0.5mm/min and 5.0mm/min

Photopolymerization time (sec) and Load Cell (kN)	Mean ± SD (MPa)		p-value
	Speed = 0.5mm/min	Speed = 5.0mm/min	
Time = 20, Load = 1	23.51±3.36	21.88±3.96	0.16
Time = 20, Load = 10	14.32±2.31	12.07±3.42	0.02*
Time = 40, Load = 1	22.62±3.01	20.01±3.13	0.01*
Time = 40, Load = 10	15.05±3.08	14.41±4.51	0.60
Average	18.88 ± 2.94	17.09 ± 3.76	0.12

*p<0.05

In the load cell configuration subgroup, the overall mean shear bond strengths were 22.05 ± 3.37 MPa and 13.96 ± 3.33 for the 1kN load cell and 10kN load cell groups. The load cell configuration (1kN vs. 10kN) showed highly statistically significant differences in SBS's when considering crosshead speed and photopolymerization time ($p<0.05$) (Table 5).

Table 5: Statistical differences between load cell configuration 1kN vs. 10kN

Photopolymerization Time (sec) and Crosshead Speed	Mean ± SD		p-value
	Load=1kN	Load=10kN	
Time=20, Speed=0.5	23.51±3.36	14.32±2.31	*
Time=20, Speed=5	21.88±3.96	12.07±3.42	*
Time=40, Speed=0.5	22.62±3.01	15.05±3.08	*
Time=40, Speed=5	20.01±3.13	14.41±4.51	*
Average	22.05 ± 3.37	13.96 ± 3.33	*

*all values highly significant

Discussion

Overall, the results from this study found that photopolymerization time and crosshead speeds did not affect orthodontic shearbond strength, however, load cell configuration was an important factor of *in-vitro* bond strength testing.

Photopolymerization Time

Findings indicated that photopolymerization time did not affect the overall shear bond strength. When the curing time was 20 seconds the average shear bond strength was lower than the 40 second subgroup (17.95 ± 3.26 MPa and 18.02 ± 3.43 MPa), however, this was not statistically significant ($p > 0.05$). When analyzing each subgroup separately there was still no statistically significant relationship between 20 and 40 second curing times. However, mean shear bond strengths were higher when the load cell was 1kN with speeds of 0.5mm/min and 5.0mm/min for 20 sec vs. 40 second curing times (Table 6.2). On the other hand when a 10kN load cell was used SBS's were higher for 40 sec vs. 20 sec curing times at both speeds of 0.5mm/min and 5.0mm/min. Although, these relationships were not significant they do show that photopolymerization times variably affect orthodontic SBS's and perhaps are not a test factor for times between those ranges. Finnema *et al.* (2010) had found that photopolymerization times significantly affected *in vitro* bond strength and that each additional second of photopolymerization increased bond strength by 0.077 MPa. In our study, for test conditions at 1kN for both crosshead speeds (0.5 and 5.0 mm/min) this was not found to be the case, however, with a 10 kN load cell, this was true. On average, for the 10kN subgroup each additional second of curing increased the SBS's by 0.076 MPa and was almost identical to the value in the systematic review. Although, the systematic review found increasing curing times resulted in increased SBS's, they were unable to suggest the most optimal time for polymerizing the adhesive (Finnema et al., 2010). Swanson (2004) conducted a study comparing different light units (LED and Halogen) while standardizing other test conditions similar to our study. The results from their study found statistically significant differences between 10 sec vs. 40 sec curing times, where 40 second curing times had higher shear bond strengths (Swanson et al., 2004). Similarily, Mavropoulos (2005) found a curing time of 10 seconds was sufficient to bond metallic brackets to incisors using intensive LED curing units, however, 40 seconds of curing resulted in higher bond strengths (Mavropoulos et al., 2005). Mauro *et al.* (2010) assessed the influence of etching and light-curing time on the shear bond strength (SBS) of a resin-modified glass ionomer cement (RMGIC) upon debonding of orthodontic brackets. They found RMGIC SBS enhanced with 37% phosphoric acid etching and 40 s light-curing time, but this did not occur when the light-curing time was increased, therefore light-curing time did not affect SBS (Maruo et al., 2010).

Reynolds (1975) recommended minimum shear bond strength values between 6-8 MPa and all 4 experimental subgroups produced bond strengths that appear to be able to withstand normal orthodontic forces (Reynolds, 1975). Results of this study also demonstrated higher bond strengths as curing time was increased. This can easily be explained by the higher conversion rate of monomer to polymer at increased polymerization times (Mavropoulos et al., 2005). The data of this *in vitro* study should be compared only with groups in the study, and the laboratory findings should not be extrapolated to the clinical situation. Studies appear to indicate that mean bond strengths recorded *in vivo* following comprehensive orthodontic treatment are significantly lower than bond strengths recorded in vitro (Pickett, Sadowsky, Jacobson, & Lacefield, 2001). However, *in vitro* studies are an important first step prior to confirming any clinical situation.

Crosshead Speed

The findings of this study indicated that variations in the crosshead speed of the testing machine can influence the test results; however, this was not significant overall. More specifically, by slowing the crosshead speed of the Zwick machine during shear bond testing of the orthodontic brackets from 5.0mm/min to 0.5mm/min, the mean shear bond strength increased from 17.09 to 18.88 MPa, an increase of only 9.4%. In other words crosshead speed did not affect the shear bond strength. Klocke *et al.* (2005) investigated the influence of cross-head speed on debonding force of orthodontic brackets and selected speed of 0.1mm/min, 0.5mm/min, 1mm/min and 5.0mm/min. Their results, similar to those from the present study, showed cross-head speed variation between 0.1 and 5 mm/min did not seem to influence debonding force measurements of brackets bonded to enamel with a composite adhesive (Klocke & Kahl-Nieke, 2005a). Bishara *et al.* (2005) conducted a similar study where they tested the effect of changing crosshead speed from 5.0 to 0.5mm/min and their results did not support the present findings. Their study found that changing the crosshead speed from 5.0 to 0.5 mm/min significantly increased the shear bond strength from 7.0 to 12.2 MPa, an increase of 57%. Furthermore, the ratio between the mean standard deviation for 5.0mm/min testing speed was 66%, whereas for the slower 0.5mm/min testing speed, it was 33%. In other words, there was an increase in the shear bond strength values and a decrease in relative variation (Bishara et al., 2005). Eliades *et al.* (2004) examined the effect of crosshead speed on the bond strength of brackets bonded to enamel. Crosshead speeds of a standard 1 mm/min and a fast 200 mm/min, which better approximates the actual jaw velocity during chewing, were selected. The results indicated that an increased crosshead speed resulted in decreased shear bond strength. Furthermore, they stated that this was probably due to the induction of a stiff body response and elimination of the visoelastic properties of the resin (Eliades, Katsavrias, Zinelis, & Eliades, 2004). Finnema *et al* (2010) reported in her systematic review that an increase in crosshead speed of 1mm/min yielded an increase in average bond strength of 1.3 MPa. From the studies presented in Table 2.1 it is evident that crosshead speeds of 0.5mm/min (8.1-26.6 MPa) typically produced higher shear bond strengths than those at higher crosshead speeds (7.0-18.46 MPa) (Refer to Table 2.1 in introduction chapter). Also, from the studies in Table 2.1 we can see that most studies use

0.5mm/min as their crosshead speeds since this produces more consistent results. The consistency of results was evident in our study if you examine the coefficients of variation. From Table 6.1 we can see at 0.5mm/min there was less variation of the shear bond strength values (13.31-20.47%) compared to 5.0mm/min (15.64-31.30%). This shows that our results at 0.5mm/min are more accurate and there is more confidence regarding the shear bond strength values.

An interesting issue raised by Eliades (2004) was regarding the clinical relevance of the reported bond strength values and how they come in to question since the standard crosshead speeds cited in the literature are irrelevant to the velocity of tooth occlusion during chewing. Although the reported crosshead speeds range from 0.1mm/min-200mm/min, it must be noted that these rates are many orders of magnitude lower than the actual masticatory velocity in humans. A complete masticatory cycle (sequential opening and closing) of a healthy individual lasts 800ms, with the closing movement having duration of less than 400 ms, which translates to over 2000mm/min and this value is much higher than the standard crosshead speed used in bond strength testing. However, using crosshead speeds observed in human chewing cycles is not practical in laboratory testing since testing is restricted to the upper limit of the effective crosshead speed of the testing apparatus (Eliades et al., 2004). The present study aims to find test conditions that are clinically relevant; however, when standardizing crosshead speed in the laboratory we demonstrated this is not relevant. That said, it is difficult, if not impossible to reproduce and replicate the actual clinical situation *in vitro*. Seldom, if ever, for example, is a sustained, perfect shear force produced on an orthodontic bracket and furthermore, replication of the human chewing cycle has not been effectively mimicked intra-orally (W. A. Wiltshire, 2012a).

Load Cell Configuration

The third experimental condition shown to significantly affect outcomes of bond strength testing was load cell configuration. An increase in load cell configuration from 1kN to 10kN resulted in significantly lower bond strengths (22.05 vs. 13.96MPa) ($p<0.05$). Even for all 4 subgroups tested, increase in load cell configuration resulted in significantly lower bond strengths ($p<0.01$). To date no study has been conducted that supports or refutes the findings of this test condition. In fact, reporting of the load cell configuration is limited (Halpern & Rouleau, 2010c; Ho ACS, Bonstein T, Akyalcin S, et al., 2010; Phan, Akyalcin, Wiltshire, & Rody, 2011a; W. A. Wiltshire, 1994) and has not been considered a factor in the majority of the bond strength testing literature. As mentioned previously Wiltshire (2012) felt this was an important test factor since the Department of Orthodontics, University of Manitoba, Canada, had two different load cells (1kN and 10kN) that were producing different results under similar test conditions. Ho *et al.* (2010) and Phan (2011) conducted studies using similar test conditions as the present study (1kN load cell, 0.05mm/min crosshead speed, 20sec curing time) and the results showed shear bond strengths of 16.7 and 18.0 MPa, whereas our study had a bond strength of 23.51MPa (Ho ACS, Bonstein T, Akyalcin S, et al., 2010; Phan, Akyalcin, Wiltshire, & Rody, 2011a). Comparing these studies with Nemeth (2006) who used a 10kN load cell, 0.05mm/min crosshead speed and 30sec curing time and found a shear bond strength of 10.6Mpa, we can note that the shear bond strengths are lower when the load cell configuration is increased (Nemeth et al., 2006). Similarly, Halpern (2010) used a 5kN load cell, with a 2mm/min crosshead speed and 40 second curing time and found an even lower bond strength (7.24MPa) than the above studies at 1kN load cell configuration(Halpern & Rouleau, 2010b). On the other hand, when Wiltshire (1994) used a 20kN load cell with a 0.05mm/min crosshead speed and 40sec curing time, the shear bond strength was much higher (resin: 26.6MPa) than all the studies mentioned above. However, it must be noted that the same resin (Tranbond XT, 3M Unitek, Monrovia, CA) was not used and could be the reason for the major difference in shear bond strengths (W. A. Wiltshire, 1994). With respect to occlusal forces of mastication generated in the human, one might argue in favor of higher load cell usage. On the flip side, the lighter load cell may improve the test sensitivity.

The results of the study also found that the coefficients of variation, a measure of the variability about the mean, were highest for the 10kN load cell and 5.0mm/min crosshead speed (28.33 and 31.30%) and this was above the recommended 20-25% (W. A. Wiltshire, 2012b). Furthermore, the lowest coefficient of variation was achieved when a more sensitive 1kN and slower crosshead speed of 0.05mm/min was used (14.29 and 13.31%). This just shows that when using a 1kN load cell the results are more consistent and uniform than for a 10kN load cell. Lighter load cells tend to produce higher bond strengths and higher bond strengths are what manufacturers may be interested in, in order to market their products. If adhesives display higher bond strengths, the clinician may assume that this results in fewer debonds and may be more inclined to purchasing that product. In order to be more stringent it would make more sense to use a heavier load cell which would result in lower bond strengths, but may be a better indicator of the clinical applicability of a product. Independent researchers may be more in favor of using a higher load cell configuration and manufacturers may use a lower load cell configuration, but, overall, from this study we suggest to use both 1 and 10 kN load cells. Results would be more uniform with a 1kN load cell but a 10kN load cell would produce lower values.

The major finding of this study shows that load cell configuration is an important test parameter that rarely appears in the literature. In order to adequately compare products this parameter needs to be stated otherwise studies cannot be accurately evaluated and clinicians may have higher expectations of an orthodontic adhesive.

Conclusions

Based on this *in vitro* study on the SBS of when changing test parameters of photopolymerization time, crosshead speed, and load cell configuration, we can conclude that:

1. Photopolymerization time does not affect the orthodontic SBS testing
2. Crosshead speed does not affect orthodontic SBS testing
3. Load cell configuration does affect orthodontic SBS testing, where the more sensitive 1kN load cell configuration produced higher bond strengths

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