

**ORTHODONTIC BOND STRENGTHS OF
A SELF-ADHERING RESIN TO ENAMEL,
RESTORATIVE COMPOSITE AND PORCELAIN**

By

ANDREW J. BERNAS

**A thesis submitted to the Faculty of Graduate Studies of the University of Manitoba in
partial fulfillment of the requirements of the degree of**

MASTER OF SCIENCE

(Orthodontics)

Department of Preventive Dental Science

University of Manitoba

Winnipeg

Copyright © 2013 by Andrew J. Bernas

Abstract

Title: Orthodontic bond strengths of a self-adhering resin to enamel, restorative composite and porcelain.

Introduction: As new adhesive products become available in restorative dentistry, investigating their potential application for orthodontic use is warranted. Vertise Flow (Kerr) is a self-adhering flowable resin and is being marketed for use as a sealant, porcelain repair and small class I restorations. It has potential for use as an orthodontic adhesive.

Objective: Determine if Vertise Flow (Kerr) is suitable for bonding fixed orthodontic appliances to enamel, restorative resin composite and porcelain with minimal surface preparation.

Methods: Shear Bond Strengths (SBS) from six (6) groups of fifteen (15) bonded stainless steel lingual buttons (Ormco) were obtained over three time points (24hr, 7 days, and 3 months). The six test groups were: 1. Vertise Flow to enamel (T_1) with coarse pumice debridement, 2. Transbond XT (3M, Unitek) to enamel (T_c) with phosphoric acid etching [control], 3. Vertise Flow to Herculite Ultra (Kerr) (C_c) with coarse pumice debridement, 4. Vertise Flow to Filtek Supreme Ultra (3M, ESPE) (C_1) with coarse pumice debridement, 5. Vertise Flow (Kerr) to porcelain (P_1) with diamond bur roughening, and 6. Transbond XT (3M Unitek) to porcelain (P_c) with hydrofluoric acid etching. Samples were stored in distilled water and incubated at 37°C. The buttons were then debonded with a Zwick Universal Testing machine using a 10 kN load cell with a crosshead speed of 0.5mm/min. Debonded buttons were evaluated based on a modified Adhesive Remnant Index (ARI). Statistical assessment of the data was performed

using parametric and non-parametric tests, with $p < 0.05$ as the threshold for statistical significance.

Results: The mean SBS obtained in all groups at each timepoint were $>4\text{MPa}$ and varied between 8.69MPa and 27.44MPa . Statistical differences were found within the composite and porcelain groups at T1, and the enamel and composite groups at both T2 and T3. Nearly half of the sample (47.2%) achieved an ARI score of 5 (100% adhesive left on button base).

Conclusion: Vertise Flow potentially provides clinically acceptable bond strengths to enamel, restorative resin composite and porcelain with minimal surface preparation. Furthermore, upon removal, minimal adhesive clean-up is required thus saving valuable chair time. Based on the results in this study, future *in vivo* investigation is suggested.

Acknowledgements

- My supervising committee
 - Dr. William Wiltshire (Supervisor)
 - Dr. Igor Pesun (External Examiner)
 - Dr. Miloš Lekić (Internal Examiner)
- Dr. Lisa Gazdzinski, Dr. Mark Poustie, Family and Friends for their support and advice
- Dr. Xiem Phan and Ken Chizick for assistance with the materials and methods.
- Companies for their generous donation of products
 - Todd Lachance (3M Unitek)
 - Ryan Lucas (Kerr)
 - Carmen Allegranza (Ormco)
 - Jim McGuire (Vident)

Table of Contents

1. INTRODUCTION	1
2. LITERATURE REVIEW	3
2.1. Evolution of Bonding	3
2.2. Bond Strength Testing in Orthodontics.....	5
2.2.1. Shear/Peel Testing	5
2.2.2. Tensile Testing.....	7
2.2.3. Torsional Testing	7
2.2.4. Testing Machine.....	8
2.2.5. Debonding Force and Bond Strength.....	8
2.2.6. Recommended Bond Strength	9
2.3. Bond Strength Testing Standardization.....	10
2.3.1. Thermocycling	11
2.3.2. Effect of Storage Medium.....	12
2.4. Conventional bonding to Porcelain	13
2.5. Conventional bonding to composite resin	16
2.6. Conventional bonding to enamel.....	17
2.7. Self-Adhering Bonding and Flowable Composites.....	20
3. PURPOSE	24

4. NULL HYPOTHESES	25
5. MATERIALS AND METHODS	26
5.1. Materials used in the study	26
5.1.1. Adhesive materials	26
5.1.1.1. Transbond XT Light Cure Adhesive System (3M, Unitek)	26
5.1.1.2. Vertise Flow (Kerr)	28
5.1.2. Substrate Materials	29
5.1.2.1. Herculite Ultra (Kerr)	29
5.1.2.2 Filtek Supreme Ultra (3M ESPE)	30
5.1.2.3. Porcelain	31
5.2. Experimental Method	34
5.2.1 Ethics	34
5.2.2. Tooth collection	34
5.2.3. Orthodontic Buttons	34
5.2.4. Sample Preparation and Storage	36
5.2.4.1. Enamel Samples	36
5.2.4.2. Restorative Resin Composite Samples	37
5.2.4.3. Porcelain Samples	38
5.2.5. Bonding Procedure	39

5.2.6.	Debonding Procedure.....	42
5.2.7.	Adhesive Remnant Index.....	45
5.2.8.	Statistical Analysis.....	46
6.	RESULTS.....	48
6.1.	Shear Bond Strength.....	48
6.1.1.	24 hours.....	48
6.1.2.	After 7 days.....	52
6.1.3.	After 3 months.....	55
6.2.	Statistical Analysis of Subgroups.....	59
6.3.	Adhesive Remnant Index.....	62
7.	DISCUSSION.....	65
7.1.	Shear Bond Strength.....	65
7.1.1.	Enamel.....	68
7.1.2.	Restorative Resin Composite.....	70
7.1.3.	Porcelain.....	72
7.2.	Potential Clinical Application and Performance.....	74
7.3.	Limitations of the current study.....	76
8.	CONCLUSIONS.....	77
9.	RECOMMENDATIONS.....	78

10. REFERENCES	79
11. APPENDIX	85
11.1. Ethics Approval	85
11.2. Journal Article and Submission Confirmation	87
12. RAW DATA	107

List of Tables and Figures

Figure 2.2.1: Schematic Illustration: Shear Force	6
Figure 2.2.2: Schematic illustration: Tensile Force	7
Table 2.4: Published SBS values and test parameters for Porcelain used in this study.....	15
Table 2.5: Published SBS values and test parameters for Restorative Resin Composite used in this study	17
Table 2.6: Published SBS values and test parameters for Enamel used in this study.....	19
Table 2.7.1: Published SBS values and test parameters for Flowable composite and/or SEP used in this study	21
Table 2.7.2: Published SBS values and test parameters for Vertise Flow used in this study	23
Figure 5.1.1.1: Transbond XT Light Cure Adhesive System	27
Figure 5.1.1.2 Vertise Flow (Kerr)	28
Figure 5.1.2.1 Herculite Ultra	29
Figure 5.1.2.2 Filtek Supreme Ultra	30
Figure 5.1.2.3: Porcelain Block	31
Table 5.1: Materials used in this experiment	32
Figure 5.2.3: Flat Lingual Button	35
Table 5.2.3.1: Surface areas obtained from ImageJ software (sq.mm).....	35
Table 5.2.3.2: Average Surface Area of Lingual Button (sq.mm).....	36
Figure 5.2.2.1: 34% Phosphoric Acid Etch.....	40
Figure 5.2.2.2: 9.4% Hydrofluoric Acid Etch.....	41

Table 5.2: Experiment Design	42
Figure 5.2.3.1: Bencor Multi-T Apparatus	44
Figure 5.2.3.2: Zwick Universal testing machine and computer	45
Figure 5.2.7: Representative Samples of Adhesive Remnant Index Scoring	46
Table 6.1.1: Descriptive Statistics at 24 hours.....	48
Figure 6.1.1.2: 24 hours Average Shear Bond Strengths with one standard deviation	51
Table 6.1.2: Descriptive Statistics after 7 days.....	52
Figure 6.1.2.2: 7 day Mean Bond Strength with one standard deviation.....	54
Table 6.1.3: Descriptive Statistics after 3 months	55
Figure 6.1.3.1: Quartile and Extreme Distribution of 3 months Shear Bond Strength.....	57
Figure 6.1.4.2: 3 month Mean Bond Strengths with one standard deviation.....	58
Table 6.2: Subgroup Analysis within each Group	59
Figure 6.2: Mean bond strengths with one standard deviation with Multiple Comparisons	61
Table 6.3: Adhesive Remnant Index Score Tally	63
Figure 6.3 ARI Subgroup statistical multiple comparisons – median with range (Kruskal-Wallis)	64

1. INTRODUCTION

Successful bonding of attachments to enamel, restorative resin and porcelain in modern orthodontics, is crucial for successful treatment outcomes. Constantly evolving and improving, generations of bonding materials have been developed to improve on existing techniques and protocols. Vertise Flow (Kerr) was developed by incorporating the self-etching technology of a seventh generation primer, OptiBond (Kerr), into a flowable composite resin (Kerr, 2012). It is marketed as suitable for bonding to mineralized tooth structure and restorative substrates including porcelain and composite (Kerr, 2012). It is currently being sold for use as a pit and fissure sealant material, restorative composite (liner for large restorations and small cavities) and porcelain repair (Kerr, 2012). It thus has potential as an orthodontic adhesive for bonding attachments.

Prior to conducting clinical trials, *in vitro* studies are essential to provide data which can be compared to well established successful *in vitro* outcomes, in order to determine whether the application of a new product may be clinically useful.

This study was designed to explore the prospect of using Vertise Flow (Kerr) as an orthodontic bonding resin. In contrast to conventional bonding techniques, which are time consuming and involve multiple steps, Vertise Flow (Kerr) claims to simplify significantly bonding, which would be advantageous in a busy orthodontic practice. The results collected from similar previous *in vitro* studies show the strength of an adhesive between the substrate and the attachment and can give an indication of how the product will perform *in vivo*. This

will give an indication of its potential usefulness in the clinical environment. Vertise Flow's (Kerr) potential in the orthodontic market can be explored.

2. LITERATURE REVIEW

2.1. Evolution of Bonding

Since the breakthrough of acid-etching technology by Buonocore in 1955, bonding has evolved substantially and is an integral part of modern dentistry and orthodontics (Buonocore, 1955; Brantley & Eliades, 2001). The bonding mechanism of bonding materials can be categorized into two categories, micromechanical or chemical (Brantley & Eliades, 2001). Techniques of surface alteration depend on the bonding surface. In orthodontics, suppliers provide attachments (brackets and buttons) with a base that has a roughened surface in an attempt improve the bond at the interface between the attachment and the adhesive (Lopez, 1980; Rossouw, 2010). In dentistry, substrates that may require bonding of orthodontic attachments are either restorative materials (amalgam, composite resin, gold, porcelain) and/or mineralized tooth (cementum, dentine, enamel). Methods of increasing substrate surface area may include the application of an acid which dissolves the surface of the substrate creating an irregular roughened surface (Brantley & Eliades, 2001; Rossouw, 2010). This acid can either be applied as a separate step or be incorporated with other components of the bonding material (Bourke & Rock, 1999; Rossouw, 2010). Alternatively, the substrate surface can be mechanically roughened with air abrasion (Wolf *et al.* 1993; Halpern & Rouleau, 2010; Bayram *et al.*, 2011; Girish *et al.*, 2012) or rotary handpieces equipped with diamond burs (Pratt *et al.*, 1989; Özden *et al.*, 1994; Bayram *et al.*, 2011; Girish *et al.*, 2012), abrasive discs (Hulterström & Bergman, 1993), or green stones (Eustaquio *et al.* 1988; Kao *et al.* 1988; Huang & Kao (2001)).

Adhesives have undergone several modifications since the contribution of acid-etching of Buonocore in 1955 (Buonocore, 1955). The first generation of adhesives (e.g. Concise (3M)) were chemically cured with two phases (Brantley & Eliades, 2001). They were time-consuming and labourious to use (Brantley & Eliades, 2001). The second generation (e.g. System 1 (Ormco), Rely-a-bond (Reliance) and Unite (3M)), were chemically cured one-phase adhesive system requiring no mixing (Brantley & Eliades, 2001). The incorporation of light curing resins launched the third generation of adhesives in the early 1980s (Brantley & Eliades, 2001). By the mid to late 1980s, the fourth generation of bonding materials came onto the market. The three components (etch, primer and adhesive) were packaged separately and the generation was coined the multiple-bottle bonding agent. Further simplification in the 1990s by combining the primer and adhesive into a single bottle resulted in the fifth generation. This generation is now accepted as conventional bonding and has been reported to be the gold standard in bonding (Turgut *et al.*, 2011). The sixth and seventh generations, commonly known as self-etchant primers (SEP), simplified bonding by eliminating a separate acid etching step. The sixth generation still required the mixing of the primer and adhesive with no separate etching required (Farah and Powers, 2004; Powers et al, 2006). The seventh generation are single component bottles that require no mixing or etching (Farah and Powers, 2004). The latest adaptation, self-adhering flowable composites, incorporate the technology of the seventh generation into a flowable composite nanofilled resin (Kerr, 2012).

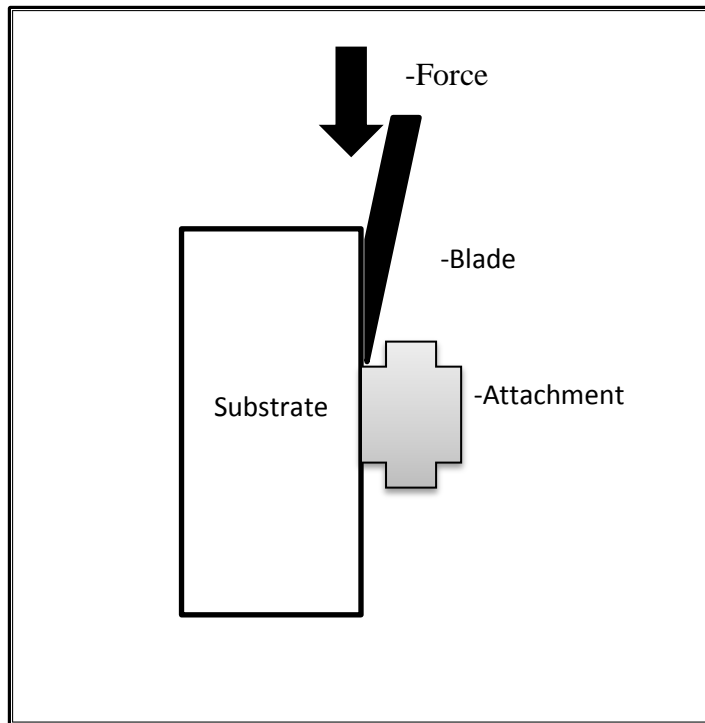
2.2. Bond Strength Testing in Orthodontics

Recently, measuring bond strength *in vivo* has become possible but continues to be challenging (Pickett *et al.*, 2001; Hajrassie & Khier, 2007; Prietsch *et al.*, 2007). *In vitro* bond strength study provides easier means to obtain bond strength data. An attachment can be debonded by way of one to three load applications: *shear/peel*, *tensile*, and *torsional* (Brantley & Eliades, 2001). The direction and placement of the force application is important as the magnitude of the force required to fracture the bond may differ (Fox *et al.*, 1994). Simulating the oral environment in the laboratory requires that the investigator report which method of debonding has been performed in the experiment (Fox *et al.*, 1994).

2.2.1. Shear/Peel Testing

In shear testing, the debonding force is applied with either a point source or line at the interface between the attachment and the adhesive, so that the attachment slides parallel off the substrate (Powers, Kim and Turner, 1997). Commonly a sharp blade is used (Figure 2.2.1).

Figure 2.2.1: Schematic Illustration: Shear Force

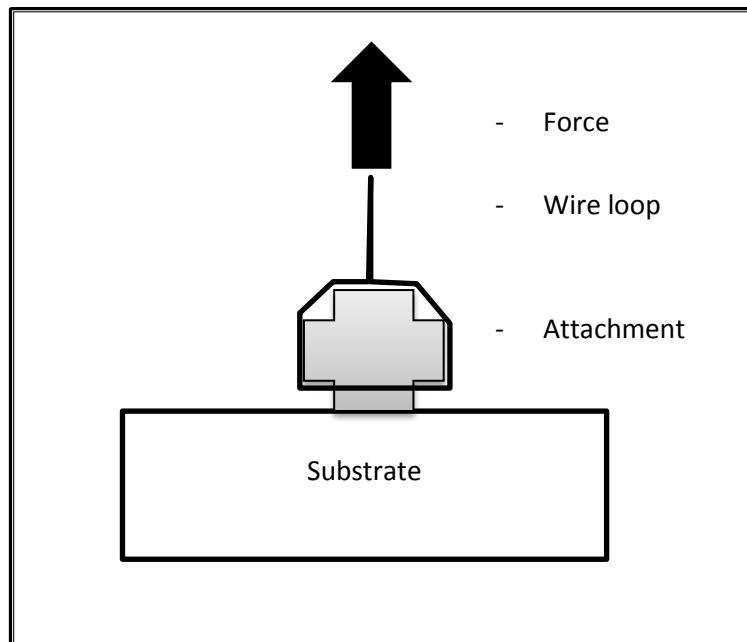


This method would best represent a true shear force, something quite unlikely to occur clinically (Brantley & Eliades, 2001). Unfortunately, pure shear loading is difficult to obtain and will likely include components of peeling, tension and torsion force applied to the adhesive interfaces (Powers, Kim & Turner, 1997). The precise amount contributed by each component will depend on the distance the force is applied from the adhesive-attachment interfaces. It is difficult to precisely determine the relative contributions of shear and peel that are being exerted at the adhesive interfaces. Typically, in the patient, the debonding force would be applied to the attachment and then disseminated to the adhesive interfaces. The unavoidable inherent bending resulting from this force application prevents a pure shear load to the attachment (Katona, 1994). Shear testing is prevalent in bond strength testing.

2.2.2. Tensile Testing

In tensile testing, the debonding force is applied perpendicular to the bonding substrate, “pulling” the attachment away from the adhesive interfaces (Figure 2.2.2). This technique, although reported as tensile strength, should be considered tensile-peel strength, which is considered amongst some investigators to be equivalent to “shear-peel” strength (Katona, 1997; Phan *et al.*, 2011).

Figure 2.2.2: Schematic illustration: Tensile Force



2.2.3. Torsional Testing

The debonding force is applied in a torquing manner and the attachment is rotated or ‘twisted’ off (Katona, 1997). Torsion is expressed in units of Newton-meter (N·m). (Katona, 1997).

2.2.4. Testing Machine

Two types of mechanical testing machines are used in orthodontic material testing: screw-driven and servohydraulic (Brantley & Eliades, 2001). In either type, a crosshead is driven with an established load towards the specimen being tested. A load cell senses the force applied and relays this raw data to computer software which will typically plot the force applied over the time or distance of the crosshead movement. If desired, stress versus strain plots can be generated from the raw data using software that has been calibrated with the bondable surface area of the attachment (Brantley & Eliades, 2001).

Both types of mechanical testing machines are referred to as constant strain-rate machines, since the speed of the crosshead can be accurately controlled (Brantley & Eliades, 2001). Crosshead speeds used in orthodontic testing typically range from 0.1 to 5 mm per minute (Cheba, 2012).

2.2.5. Debonding Force and Bond Strength

The magnitude of a bond strength is reported in megapascals (MPa), kilogram per square centimeter (kg/cm^2) or pounds per square inch (lb/in^2). It could also be reported as a bond force in units of Newtons (N), kilograms (kg) or pounds (lbs). Bond strength is the bond force over the surface area of the bonded interface (e.g. $1 \text{ Pa} = 1 \text{ N}/\text{m}^2$, $1 \text{ MPa} = 1 \text{ N}/\text{mm}^2$). (Powers *et al*, 1997)

2.2.6. Recommended Bond Strength

The magnitude of a “bond strength” is really only meaningful when it relates to what is acceptable clinically for the attachment of orthodontic appliances. Minimum tensile bond strengths of 5.9 to 7.9 MPa have been reported to be adequate to resist orthodontic forces (Reynolds, 1979; Powers, Kim & Turner, 1997). A bond strength of 6-8 MPa has been stated as adequate in many studies (Reynolds, 1975; Wiltlock et al, 1994). Reynolds (1975) was the first to report this value and was based on a typical bracket area of 16mm^2 withstanding a force of 40-120N (Powers et al. 1997). Consequently, the bond strength value of 6-8 MPa remains a commonly quoted minimum value. Contrary to this value, some glass ionomer cements have a minimum bond strength value around 3-4 MPa and have proven to be effective clinically (Wiltshire & Noble, 2010). This lower value is validated by no significant bond failures between glass ionomer bonding (3.3%) and conventional resin bonding (1.6%) (Fricker, 1994). Applying this observation, a lower minimum bond strength value than previously accepted is warranted.

Higher bond strength values may provide too strong of a connection to the substrate and cause damage upon removal (Kusy, 1994; Proffit & Fields, 2000; Cerhrel *et al.*, 2005). Experimentally, fractures with enamel have occurred with bond strengths as low as 13.8 MPa (Retief, 1974), while fractured within porcelain have been reported with bond strengths as low as 10 to 12.4 MPa (Major, Koeler & Manning 1995; Zacharison, Zacharison & Büyükyılmaz, 1996). It is important to note that when reviewing *in vitro* testing that not only the mean but also the range values are interpreted (Wiltshire & Noble, 2010).

Regardless of the direction of force application, bond failures can be either cohesive or adhesive in nature. Cohesive failures occur within the material itself whereas adhesive failures occur between an interface (attachment-adhesive or adhesive-substrate) (Powers, Kim & Turner, 1997). Bond strength failure location is also important as damage to the substrate surface can be minimized if the failure occurs at the attachment-adhesive interface (Pickett *et al.*, 2001). A disadvantage of cohesive fracture is increased adhesive clean-up from the substrate surface (Pickett *et al.*, 2001).

2.3. Bond Strength Testing Standardization

There are many criticisms of bond strength testing in dentistry and orthodontics (Van Noort *et al.*, 1989; Solderholm, 1991; Fox, McCabe and Buckley, 1994). Such criticisms center around the fact that none of the testing methods available accurately represents the clinical situation. Different forces applied in combinations of the aforementioned debonding methods place different stresses on the attachment/adhesive/substrate interface (Katona, 1997).

Furthermore, standardization of the testing variables (load cell, crosshead speed, testing method, thermocycling, quality of enamel, fluoride content, storage medium, type of attachment, size of attachment, bonding surface of attachment, testing machine, time interval post-bonding) has not been universally accepted. There is a lack of standardized test protocols in evaluation of orthodontic products (Stanford *et al.*, 1997). Cheba *et al.* (2012) examined the effect of crosshead speed, load cell configuration and curing time testing

parameters and concluded that neither photopolymerization nor crosshead speed affect shear bond strength, however, load cell configuration did with lighter load cells (1 kN in his investigation) producing higher bond strengths.

With so many inherent confounding parameters in *in vitro* shear bond strength investigation, it is important to match as many parameters as possible for direct comparison of data obtained amongst different centers (Phan, 2011).

2.3.1. Thermocycling

Thermocycling attempts to provide a simulated oral environment to replicate the temperature range that an adhesive would experience in function (Gale & Darvell, 1999; Bishara *et al.*, 2003; Daub *et al.*, 2006; Elekdag-Turk *et al.*, 2008). Normal body temperature, 37°C, is where the majority of function occurs, however during eating exposed temperature may fluctuate to lower temperatures, when drinking cold beverage or eating food such as iced cream, and to hot temperatures, when drinking hot beverages or eating food such as hot soup. The effect of thermal change on a material is microcontraction when cooled and microexpansion when heated (Gale & Darvell, 1999). This may weaken the bond over time as the different interfaces expand and contract at different rates (Gale & Darvell, 1999). A criticism of thermocycling is that the thermal change is not accurate for what the adhesive will experience intra-orally (Gale & Darvell, 1999; Bishara *et al.*, 2003). Although the adhesive may be exposed to a range of temperatures, the exposure is for a brief duration and would rarely oscillate from one thermal extreme to the other (Gale &

Darvell, 1999). There is a lack of standard protocols to follow as to how to perform the thermocycling (Gale & Darvell, 1999). Different thermocycling regimes have been used varying with number of cycles (200, 500, 750, 1500, 2000, 2500, 5000, 6000, 10,000 and 20,000), temperature ranges (low: 5°C, 10°C and high: 45°C, 50°C or 55°C) and immersion periods (dwell time: 30s transfer time: 4s, 5s, 15s) (Arici & Arici, 2003; Daub *et al.*, 2006; Turk *et al.*, 2010; Costa *et al.*, 2011)

The literature is mixed in terms of the importance of thermocycling on bond strength (Finnema *et al.*, 2010). After a day or so, a full cure (complete reaction of unreacted monomer) of an adhesive is achieved (Bishara *et al.*, 1999). Thereafter, the bond strength degrades over time, regardless of thermocycling (Ferracane *et al.*, 1998). However, it is apparent that the rate of degradation does increase in the presence of thermocycling. Currently, the literature provides mixed conclusions on the clinical impact of thermocycling. Studies have shown that thermocycling has a significant effect (Bishara *et al.*, 2003; Elekdag-Turk *et al.*, 2008; Goracci *et al.*, 2013), whereas others have shown no difference (Arici & Arici, 2003; Daub *et al.*, 2006; Turk *et al.*, 2010; Costa *et al.*, 2011).

2.3.2. Effect of Storage Medium

The storage medium in *in vitro* studies may influence the SBS outcome (Ferracane *et al.*, 1998; Finnema *et al.*, 2010). Maintaining a moist environment is important to prevent desiccation and allow for water equilibration of substrates. Distilled or deionized water has

been and continues to be used as a storage medium for samples. Ferracane *et al.* (1998) investigated the long-term effect of aging in water at 37°C on the physical properties of composites for 1 day, 6, 12 and 24 months and found a 20-30% reduction in fracture toughness of the composites after 6 months with minimal change thereafter. Saliva analogues have been formulated to better simulate the oral environment (Finnema *et al.*, 2010). Despite continued advances, it is impossible for *in vitro* studies to precisely duplicate the oral environment in a controlled setting. Factors that are difficult to replicate *in vitro* include pH, microflora, temperature variation, as well as forces delivered from engaged archwires and occlusion (Power *et al.*, 1997; Stanford *et al.*, 1997; Brantley & Eliades, 2001).

2.4. Conventional bonding to Porcelain

The structure of porcelain is stronger than enamel (Shillingburg, 1997). This impacts on the methods used to alter the surface structure of porcelain prior to bonding. A strong acid, hydrofluoric acid, is often used to etch porcelain (Major, Koeler and Manning, 1995; Jarotski, 2000; Huang & Kao, 2001). Not only is the acid stronger, but the duration of its application to the surface is longer (e.g. 2 mins) than acids commonly used (phosphoric acid) for etching enamel (e.g. 10-30sec) (Bourke & Rock, 1999). Alternatively, mechanical roughening of the porcelain surface with either fine particle sandblasting, diamond bur or green stones, prior to bonding has been reported (Bourke & Rock, 1999; Huang & Kao, 2001; Girish, 2012)

Silane treatment of the roughened porcelain has been suggested to improve bonding to porcelain (Major, Koeler and Manning, 1995). This procedure is beneficial when bonding veneers or all ceramic crowns where bonding permanence is a desirable, but its necessity for orthodontic bonding has been disputed. Potentially clinically acceptable bond strengths are possible without silane coating the porcelain (Bourke & Rock, 1999). In fact, in a study conducted by Jarotski (2000) at the University of Manitoba, when silane was incorporated in the methodology, higher incidence of porcelain fracture was observed.

Table 2.4: Published SBS values and test parameters for Porcelain used in this study

Study	Test parameters	C.H.S.	Load Cell	Storage Medium	N	Average SBS	Range	Test Condition
Costa et al (2012)	Silane HF 24hr	1mm/ min	N.R.	Distilled water	20	7.62- 13.81MP a	N.R.	Metal brackets Adh: Transbond XT/Fuji Ortho LC
Girish et al (2012)	Sandblast Silane Diamond bur HF 24hr	-	-	Artificial Saliva	10	-	-	Metal Brackets
Huang & Kao (2001)	Thermo. HF Green stone Silane 24h/7d	1mm/ min	N.R.	Distilled water	8	10.9-182 kg/cm ²	N.R.	Composite brackets Adh: Unite
Jarotski (2000)	24h/6m HF Silane Pumice	0.5mm/ min	10kN	Distilled water	10	3.97- 17.08 MPa (24hr) 2.16- 16.64MP a (6m)	2.17- 21.06 MPa (24hr) 1.40- 21.81 MPa	Metal brackets Adh: Concise
Bourke & Rock (1999)	Sandblast HF H ₂ PO ₄ Thermo Silane	5mm/ min	200N	water	10	0- 18.69MP a	N.R.	Metal brackets Adh: Right-on
Major et al (1995)	HF Silane 24hr	0.5mm/ min	50kN @10%	Distilled water	10	0.41- 13.53MP a	0- 19.44 MPa	Metal brackets Adh: Phase II/Rely-a- bond

N.R.: Not Reported; Thermo: Thermocycled; Adh: Adhesive

2.5. Conventional bonding to composite resin

Many adhesives used in orthodontics are resin based or have resin included in the formulations. Although, theoretically, resin adheres to resin chemically, increasing the surface area by means of surface micromechanical preparation has been suggested to allow for reliable bonding to restorative resin composite (Bayram *et al.*, 2011).

A recent *in vitro* study by Bayram *et al.* (2011) investigated the bond strength of resin composite adhesive (Transbond XT) to artificially aged restorative resin composite with a variety of surface preparations. Their finding showed that mechanical surface alteration by means of diamond bur, particle abrasion and acid treatment with hydrofluoric acid performed superior to acid treatment with phosphoric acid treatment or no additional preparation. Noteworthy from this study were reported mean SBS of 3.71 ± 1.22 MPa with 38% phosphoric acid treatment and 2.77 ± 0.34 MPa with no treatment, suggesting the requirement for some surface preparation be conducted when bonding to composite (Bayram *et al.*, 2011). The results obtained from this study were lower than reported values from Lai *et al* (1999). Lai *et al* (1999) obtained a SBS of 26.8MPa and 24.4 MPa after thermocycling for metal brackets bonded with Transbond XT and System 1+, respectively.

Table 2.5: Published SBS values and test parameters for Restorative Resin Composite used in this study

Study	Test parameters	C.H.S.	Load Cell	Storage Medium	N	Average SBS	Range	Test Condition
Bayram et al (2011)	H ₃ PO ₄ HF Sandblast Diamond Thermo.	1mm/ min	N.R.	Distilled water	15	2.77- 10.61MPa	2.08- 15.77 MPa	Metal brackets Adh: Transbond XT
Lai et al (1999)	Thermo	-	-	-	12	26.8MPa	-	Metal bracket Adh: Transbond XT

N.R.: Not Reported; Thermo: Thermocycled; Adh: Adhesive

2.6. Conventional bonding to enamel

Conventional bonding to enamel may be complicated by the presence of bleaching or fluorosis (Noble *et al.*, 2008; Wiltshire & Noble, 2010; Phan *et al.*, 2012). Techniques to increase the surface area have been previously discussed and include: air abrasion, acid etching and self-etching primers. The gold standard for enamel bonding has been reported to be the fifth generation (e.g. Transbond XT) (Turgut *et al.*, 2011). Bond strength values are well established and have withstood clinical scrutiny (Table 2.6).

Fifth generation adhesives systems follow similar application methodology. Firstly, the acid, typically phosphoric acid (H₃PO₄), dissolves a superficial layer (approximately 3-10µm

thick) of enamel and creates a roughened surface with improved wettability in preparation for the application of the primer-adhesive solution (Powers *et al.*, 2006; Rossouw, 2010). After the enamel conditioner is rinsed away and the surface is lightly dried, the primer-adhesive solution is applied and flows into the enamel irregularities resulting in a resin tags creating a micro-mechanical lock once polymerized. (Powers *et al.*, 2006). Primers are essentially hydrophilic monomers dissolved in a solvent (e.g. acetone, water, or ethanol-water with a photoinitiator, camphorquinone) while adhesives are hydrophobic, dimethacrylate oligomers (e.g. Bis-GMA) usually diluted with a lower-molecular weight monomer (e.g. triethylene glycol dimethacrylate -TEGDMA). (Powers *et al.*, 2006).

Table 2.6: Published SBS values and test parameters for Enamel used in this study

Study	Test parameters	C.H.S.	Load Cell	Storage Medium	N	Average SBS	Range	Test Condition
Goracci (2013)	30min/ 24hr+ Thermo.	1mm/ min	N.R.	Deionized water	20	9.8 MPa	N.R.	Metal Adh: Transbond XT (conv)
Cheba (2012)	24hr	0.5mm/ min 5mm/ min	1kN 10kN	Artificial Saliva	20	12.07- 23.51MPa	5.97- 30.88 MPa	Metal buttons Adh: Transbond XT
Işman et al (2012)	24hr	1mm/ min	N.R.	Distilled water	12	9.86 MPa	N.R.	Metal brackets Adh: Transbond XT
Banerjee & Banerjee (2011)	24hr	5mm/ min	N.R.	Distilled water	10	14.6MPa	N.R.	Metal brackets Adh:Transbond XT/Fuji Ortho LC/ Relybond/ Orthobond LC/Enlight
Phan et al (2011)	24hr/3m	0.5mm/ min	1kN	Artificial Saliva	20	18- 19.6MPa	10.13- 28.95 MPa	Metal buttons Adh: Transbond XT
Ho et al (2010)	5min/24 hr/3m	0.5mm/ min	1kN	Deionized water	15	11.22- 16.65MPa	2.63- 26.87 MPa	Metal buttons Adh: Transbond XT

N.R.: Not Reported; Thermo: Thermocycled; Adh: Adhesive

2.7. Self-Adhering Bonding and Flowable Composites

The sixth and seventh generation of adhesives are made up of self-etching primers (SEP) and were developed to reduce chair time and technique sensitivity (Turgut *et al.*, 2010; Ho, 2011). The distinction between the two generations is the presence and absence of mixing the components (Farah & Powers, 2004). SEPs are considered bicomponent hydrophilic adhesive and have shown to have a shallower etch pattern than conventional etching suggesting less enamel loss to tooth and as a result lower bond strength (Table 2.7.1) (Paschos *et al.*, 2008). The main ingredient in SEP (e.g. Transbond Plus) is methacrylated phosphoric acid esters (Grubisa *et al.*, 2004).

Flowable resin composites have been studied for the purpose of orthodontic bonding (Table 2.7.1). Tecco *et al* (2005) reported mean SBS varying from 25.52-34.80 MPa and range values of 12.93-108.7 MPa for flowable composites (Denfil Flow with and without intermediate unfilled resin (Vericom Laboratories Ltd., Anyang, Korea) and Dyrac Flow) on etched enamel. D'Attilio *et al.* (2005) using the same flowable composite (Denfil Flow), with a liquid resin intermediate, on etched enamel, reported a SBS of 24.98 MPa with a range of 14.72-35.14 MPa.

Table 2.7.1: Published SBS values and test parameters for Flowable composite and/or SEP used in this study

Study	Test parameters	C.H.S.	Load Cell	Storage Medium	N	Avg. SBS	Range	Test Condition
Albaladejo et al (2011)	72hr	0.5mm/min	N.R.	Deionized water	20	6-15.1 MPa	N.R.	Metal brackets to enamel Adh: Admira Flow; Tetric Flow; Filtek Supreme; Transbond Supreme
Turgut et al (2011)	24hr	5mm/min	N.R.	Deionized water	12	~2-7MPa	~2-12 MPa	Metal brackets Adh: Transbond XT w/ SEP; Clearfil Majesty Flow w/Clearfil S3; Adper Easy w/Filtek Sup. Flow; Grandio Flow w/Futura Bond NR
Ho et al (2010)	5min/24hr/3m	0.5mm/min	1kN	Deionized water	15	3.46-13.2 MPa	2.78-25.18 MPa	Metal buttons Adh: iBond; Transbond Plus; G Bond
Paschos et al (2008)	Thermo . 30d	0.5mm/min	N.R.	Deionized water	25	10.6-13.2 MPa	N.R.	Metal brackets Adh: Transbond Plus; Transbond XT; iBond
Ryou et al (2008)	24hr	1.0mm/min	N.R.	water	10	7.2-10.9 MPa	N.R.	Metal brackets Adh: Grandio Flow; UniFil Flow: UniFil LoFlo; DenFil Flow; Transbond XT; Filtek Z250
Attar et al. (2007)	48hr	5mm/min	N.R.	Deionized water	14	9-9.55 MPa	5.1-16.45 MPa	Metal brackets to enamel Adh: Clearfil tri-S
D'Attilio et al (2005)	72hr Peel	1.0mm/min	N.R.	Deionized water	40	24.9-8 MPa	14.72-35.14 MPa	Metal brackets to enamel Adh: Denfil Flow; Transbond XT
Tecco et al (2005)	72hr	1.0mm/min	N.R.	Deionized water	20	23.2-35.8 MPa	12.93-108.7 MPa	Metal brackets to enamel Adh: Denfil Flow; Dyrac Flow; Denfil Flow/Denfil Primer; Transbond XT
Grubisa et al (2004)	Thermo .	2.4mm/min	500N	Distilled water	65-70	7.3-9.8 MPa	0.8-22.0 MPa	Metal brackets Adh: Transbond SEP; Enlight

N.R.: Not Reported; Thermo: Thermocycled; Adh: Adhesive

Self-adhering flowable resins are formulated by combining a self-etching primer with a flowable nanofilled resin (Kerr, 2012). There are currently two self-adhering flowable composites (Maxcem Elite, and Vertise Flow) that have been investigated for use in orthodontic bonding. Load cell configuration used during the testing of these materials has never been reported.

Işman *et al.* (2012) investigated the 24hr shear bond strength of Vertise Flow and Maxcem Elite with or without etching to human enamel. They reported bond strength values of 2.55 ± 0.77 MPa for Vertise Flow without etching, 7.89 ± 1.17 MPa for Vertise Flow with etching, 4.67 ± 2.94 MPa Maxcem Elite without etching and 7.82 ± 2.56 MPa for Maxcem Elite with etching. Goracci *et al.* (2013) investigated the thirty (30) minute shear bond strength and post-thermocycling shear bond strength values of Vertise Flow to human enamel with and without etching compared with Transbond XT adhesive with a Transbond self-etching primer or a conventional primer. Initial bond strength values for Vertise Flow were encouraging both with etching (11.86 ± 4.17 MPa) and without etching (10.13 ± 2.86 MPa). However, thermocycling at 5 and 55°C for 1,000 cycles lasting 65 s (dwell time, 30s; transfer time 5 s) resulted in a substantial drop in bond strength values (without etching 2.99 ± 1.2 MPa and with etching 6.56 ± 1.05 MPa) (Goracci *et al.*, 2013).

The performance of self-adhering flowable composites for use in the bonding of orthodontic attachments has not been assessed over a longer term or to different substrates.

Table 2.7.2: Published SBS values and test parameters for Vertise Flow used in this study

Study	Type	C.H.S.	Load Cell	Storage Medium	Curing Time	N	Avg SBS ± s.d MPa	Range	Test Condition
Goracci et al (2013)	SBS	1mm/min	N.R.	Deionized water w/ thermo	20s	10	10.13 ± 2.86	N.R.	Bracket to human enamel
Işman et al (2012)	SBS	1mm/min	N.R.	Distilled water	40s	12	2.55 ± 0.77	N.R.	Bracket to human enamel

N.R.: Not Reported; Thermo: Thermocycled

3. PURPOSE

The purpose of this study was to determine if Vertise Flow (Kerr), a self-adhering flowable resin, would be suitable for orthodontic bracket bonding to various restorative materials (porcelain, composite) and to natural tooth enamel with minimal surface preparation of the bonding surface.

4. NULL HYPOTHESES

- 1) Vertise Flow (Kerr) does not provide any significant difference in shear bond strength of orthodontic attachments to restorative resin composite, enamel and porcelain compared to the conventional bonding.
- 2) Vertise Flow (Kerr) does not provide any significant difference in shear bond strength of orthodontic attachments to restorative resin composite, regardless of the manufacturer of the composite.

5. MATERIALS AND METHODS

5.1. Materials used in the study

Table 5.1 lists the materials used in this study.

5.1.1. Adhesive materials

Compositions of the adhesive materials and non-enamel bonding substrates used in this study are outlined below.

5.1.1.1. Transbond XT Light Cure Adhesive System (3M, Unitek)

The Transbond XT Light Cure Adhesive System is comprised of a primer and an adhesive paste (3M Unitek, Monrovia, CA). The primer is an unfilled light cured resin consisting of Bis-GMA and Triethylene glycol dimethacrylate (TEGDMA) in a 1:1 ratio with a photoinitiator. The adhesive paste is a composite resin containing Bisphenol A diglycidylether methacrylate [10-20%wt], Bisphenol A bis (2-hydroxyethyl ether) dimethacrylate (Bis-EMA) [5-10%wt], silate treated quartz [70-80% wt] and less than 2% silane treated silica (MSDS: 3M a, 2010).

Figure 5.1.1.1: Transbond XT Light Cure Adhesive System



5.1.1.2. Vertise Flow (Kerr)

Vertise Flow (Kerr, 1717 West Collins, Orange, CA) is a self-adhering, light cure flowable composite (Kerr, 2010). It is composed of 18-40% methacrylate ester monomers with inert mineral fillers which include the following: Ytterbium Fluoride, Glyceroldimethacrylate dihydrogen phosphate (GPDM), 2-hydroxyethyl methacrylate (HEMA), Monomethyl ether hydroquinone (MEHQ), Ethoxylated bisphenol A dimethacrylate, Bisphenol A diglycidyl Methacrylate, Urethane dimethacrylate, Leucopure EGM, 2-Hydroxy-4-methoxybenzophenone, Amine co-initiator, Camphorquinone, 4-Methoxyphenol, Phosphated polyester, Prepolymerized Filler, Silane treated Barium Aluminoborosilicate glass and colourants. (MSDS: Kerr a 2008; İşman, 2012; Kerr a, 2013).

Figure 5.1.1.2 Vertise Flow (Kerr)



5.1.2. Substrate Materials

5.1.2.1. Herculite Ultra (Kerr)

Herculite Ultra is a nanohybrid restorative composite resin composed of 20-45% wt uncured methacrylate ester monomers, non-hazardous inert mineral fillers, non-hazardous activators and stabilizers. The ingredients of which are: Bisphenol A diglycidyl methacrylate, Triethyleneglycol dimethacrylate, Ethoxylated bisphenol A dimethacrylate, 2-Hydroxy-4-methoxybenzophenone, Camphorquinone, 4-Methoxyphenol, Coumarin derivative, Ethyl-4-(dimethylamino)benzoate, Silicon dioxide, Methacryloxypropyl trimethyloxysilane, Barium glass, pre-polymerized filler, Ytterbium fluoride, and Synthetic iron oxides (MSDS: Kerr b, 2010).

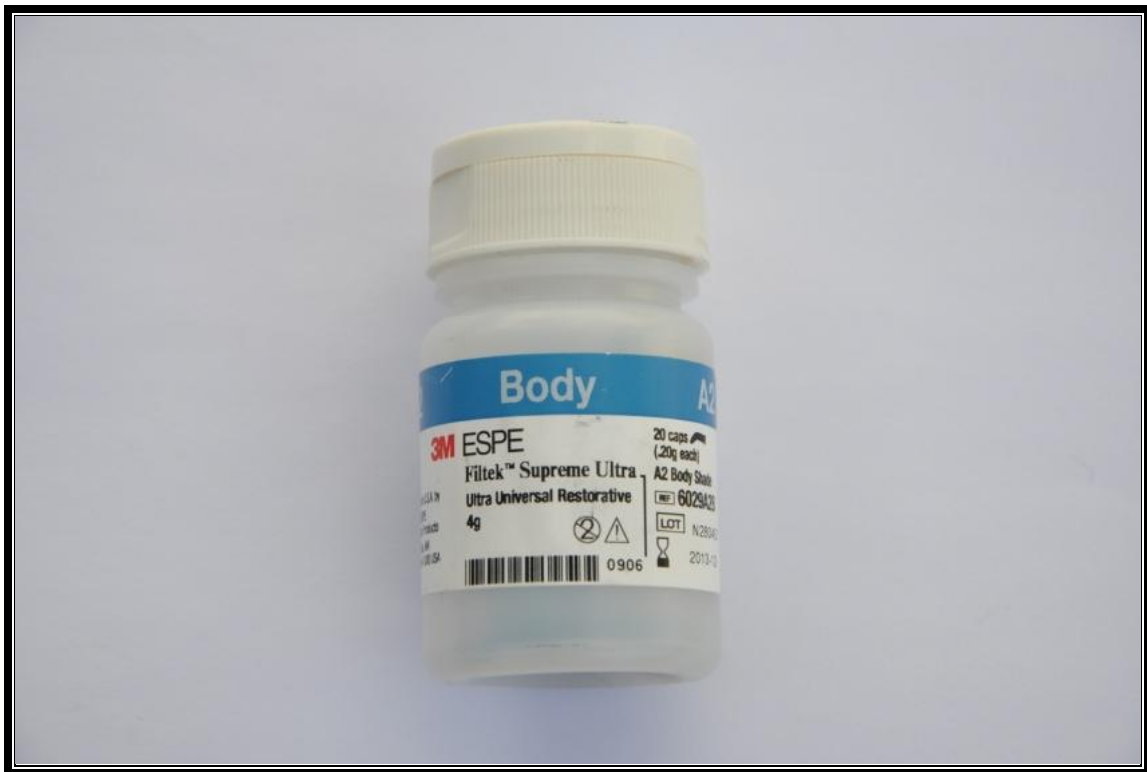
Figure 5.1.2.1 Herculite Ultra



5.1.2.2 Filtek Supreme Ultra (3M ESPE)

Filtek Supreme Ultra is a restorative composite resin composed of silane treated ceramic [65-75% wt], silane treated silica [5-15% wt], bisphenol A polyethylene glycol diether dimethacrylate (BISEMA6) [5-15% wt], diurethane dimethacrylate (UDMA) [5-15% wt], bisphenol A diglycidyl ether methacrylate (BISGMA) [1-10% wt], and triethylene glycol dimethacrylate (TEGDMA) [$<5\%$ wt] (MSDS: 3M, 2008b).

Figure 5.1.2.2 Filtek Supreme Ultra



5.1.2.3. Porcelain

Porcelain blocks (Figure 5.1.2.3) used for the fabrication of crowns in a Cerec machine were obtained from Vident. The Vitablocs are composed of fine-structure feldspar ceramic (Vident.com, 2013). The main constituent of feldspar is silicon dioxide, present in the form of $\text{Na}_2\text{O Al}_2\text{O}_3 6\text{SiO}_2$ and $\text{K}_2\text{O Al}_2\text{O}_3 6\text{SiO}_2$ (Shillingburg, 1997; MSDS: Vita, 2008).

Figure 5.1.2.3: Porcelain Block



Table 5.1: Materials used in this experiment

Material	Manufacturer	Reference Number	Lot
Sample Preparation			
Copper Rings			
Petroleum Jelly (Vaseline)	Chesebrough-Pond's, USA		
Bosworth Fastray (Custom tray and acrylic base plate material)	Bosworth, Illinois	0921375	
- Monomer Liquid			1107-297
- Monomer Powder			0901-005
Separating Disc	Great Lakes Ortho	086-013	42626330
Substrate			
Herculite Ultra	Kerr, Orange, CA	34348	4296373
Filtek Supreme Ultra	3M ESPE, St Paul, MN	6029A2B	N280457
Vitablocs (Porcelain)	Vident, Brea, CA	n/a	5426, 6011, 6135, 6273, 6722, 7236, 7265, 7278, 7374, 7392, 7551, 7763, 7868, 22440, 23390, 27780, 28560
Substrate Surface Preparation			
34% Phosphoric Acid Etch	Denstply	546151	050322
9.6% Hydrofluoric Acid Etch	Pulpdent, Watertown, MA	PEG-3	120314
Diamond Bur (coarse)	Brasseler	6847KRDC.31.016	n/a

Material	Manufacturer	Reference Number	Lot
Pumice	n/a		
Used in Bonding			
Vertise Flow	Kerr, Orange, CA	34243	3700959
Transbond XT	3M Unitek, Monrovia, CA	712-035	CZ6T0
- Primer		712-034	N261942
- Adhesive Paste		712-305	N255786
Flat stainless steel lingual buttons	Sybron Dental Specialties Ormco	300-0097	11L137L
Debonding Equipment			
Universal Testing Machine	Zwick GmBH, Ulm, Germany		
Bencor Multi-T testing apparatus	Danville Engineering, San Ramon CA		
Other			
EZCal Digital Caliper	iGaging		
Blue Ray 3 Microflash LED (light emitting diode) curing device	American Orthodontics	851-940	n/a
Incubator 37°C	Thelco/Canlab Model 2, Precision Scientific, Chicago, IL		
Composite Instrument	Thompson Composite	TD15x #15	13
Plastic Containers	Ziploc		
	Gladware		

5.2. Experimental Method

5.2.1 Ethics

Ethics approval was not required for this study as the collected biological materials were anonymous. The discussion with the University of Manitoba Ethics Board can be found in Appendix 12.1.

5.2.2. Tooth collection

Extracted human teeth were collected from a local oral surgeon. Upon collection, the teeth were stored in distilled water. Ninety (90) molar teeth were selected from the sample based on being non-carious and non-restored. The molar teeth were cleaned of any attached soft tissue using a universal scaler and had the roots sectioned apical to the cemento-enamel junction using a diamond disc in a straight attachment slow speed handpiece. The sectioned teeth were then randomly divided into six containers each containing fifteen (15) teeth and immersed in distilled water.

5.2.3. Orthodontic Buttons

Three hundred flat ovoid stainless steel orthodontic buttons were obtained from Ormco. Four (4) randomly selected buttons were digitally photographed (Figure 5.2.3) using a Nikon D90 Digital SLR Camera with an AF-S VR Micro-Nikkor 105mm f/2.8G IF-ED lens and measured along the width of the button base using a digital caliper (EZCal, iGaging). The

photographs were then used to determine the average surface area of the button base. The measured width of the bracket base was used to calibrate the digital photographs with image analysis software, ImageJ. The perimeter of the button base was digitally traced, over 5 repetitions, and the surface area was derived within the software. The experimental surface area used in the shear peel testing was obtained from the average of the ImageJ surface area outputs. The average surface area was calculated to be 11.03 sq.mm and is summarized in Table 5.2.3.1 and Table 5.2.3.2.

Figure 5.2.3: Flat Lingual Button



Table 5.2.3.1: Surface areas obtained from ImageJ software (sq.mm)

	Button 1	Button 2	Button 3	Button 4
Tracing 1	11.223	10.968	10.890	10.676
Tracing 2	11.287	11.067	10.849	10.770
Tracing 3	11.233	11.287	11.047	10.748
Tracing 4	11.417	11.070	11.021	10.797
Tracing 5	11.257	11.104	11.104	10.829
Average	11.2834	11.1054	10.9822	10.764

Table 5.2.3.2: Average Surface Area of Lingual Button (sq.mm)

Button 1	11.2834
Button 2	11.1054
Button 3	10.9822
Button 4	10.764
Overall Average	11.03375

The calculated average surface area (SA_{avg}) of 11.03375 mm² was then used to determine the diameter of a representative circular bracket base. The representative diameter of a round bracket base was required for the software of the Zwick Testing Machine. Using the following formula:

$$d = (4 \times SA_{avg} / \pi)^{1/2}$$

a diameter of 3.75mm was calculated to be the equivalent round base to the ovoid buttons.

5.2.4. Sample Preparation and Storage

5.2.4.1. Enamel Samples

The enamel samples were then embedded in a chemically cured acrylic custom tray material (Bosworth Fastray) with the most flat surface exposed, parallel to a level surface, formed using copper moulds that coincide with the diameter of the Bencor Multi T testing apparatus. Petroleum jelly was placed on the inside surface of the copper moulds to facilitate removal after the acrylic had set. The level surface was evaluated at eye-level as the sample was rotated 360°. The prepared tooth samples were divided into two (2) groups:

- 1) forty-five (45) samples further subdivided into three (3) groups of fifteen (15) to be used with the test material (Vertise Flow) and;
- 2) forty-five (45) further subdivided into three (3) groups of fifteen (15) to be used with the control material (Transbond XT).

The samples were then placed into separate labelled plastic containers for each test material (2), filled with distilled water and stored in an incubator (Thelco/Canlab Model 2) at 37 °C.

5.2.4.2. Restorative Resin Composite Samples

In a similar manner, forty-five (45) restorative resin samples of Kerr Herculite Ultra and forty-five (45) samples of 3M ESPE Filtek Supreme Ultra were prepared in chemically cured custom tray (Bosworth Fastray) material in copper moulds. Petroleum jelly was placed on the inside surface of the copper mould to facilitate removal after the acrylic had set. A well was created in the setting acrylic using a 10mm dowel (the non-writing terminus of a pencil). Resin was then loaded incrementally using a composite unidose cartridge dispenser into the wells, adapted to the wells using a condenser, and cured every 2mm increments for twenty (20) seconds according to manufacturer requirements using a Blue Ray 3 microflash LED light. The restorative resin composite samples from each company were prepared to a flat surface parallel to a level surface using a Thompson Composite TD15x #15 Lot No. 13 instrument. The level surface was evaluated at eye-level as the sample was rotated 360°. The prepared restorative composite resin samples were divided into two (2) groups:

- 1) forty-five (45) samples further subdivided into three (3) groups of fifteen (15) to be used with the control material (Herculite Ultra) and;
- 2) forty-five (45) further subdivided into three (3) groups of fifteen (15) to be used with the test material (Filtek Supreme Ultra).

The samples were then placed into separate labelled plastic containers for each test material (2), filled with distilled water and stored in an incubator (Thelco/Canlab Model 2) at 37 °C.

5.2.4.3. Porcelain Samples

A single porcelain unit was used to fabricate an acrylic loading jig from which subsequent porcelain blocks could be inserted and placed in the Bencor testing castle. The jig was made so that the terminal surface of the porcelain block was parallel to a level surface. This was accomplished by pressing a porcelain unit through the setting acrylic until it made contact with the level surface. Ninety (90) porcelain samples were divided into two (2) groups:

- 1) forty-five (45) samples further subdivided into three (3) groups of fifteen (15) to be used with the test material (Vertise Flow) and;
- 2) forty-five (45) further subdivided into three (3) groups of fifteen (15) to be used with the control material (Transbond XT).

The samples were then placed into separate labelled plastic containers for each test material (2), filled with distilled water and stored in an incubator (Thelco/Canlab Model 2) at 37 °C.

5.2.5. Bonding Procedure

270 flat lingual buttons were bonded using one of 6 adhesive/substrate combinations i.e. 15 samples per group. The shear bond strength was then measured after three (3) different time periods:

T1: Immediate (24 hours)

T2: 7 days

T3: 3 months

The six test groups were: 1. Vertise Flow to enamel (Tooth Test -T_t), 2. Transbond XT to enamel (Tooth Control - T_C), 3. Vertise Flow to Herculite Ultra (Composite Control – C_c), 4. Vertise Flow to Filtek Supreme Ultra (Composite Test - C_t), 5. Vertise Flow to porcelain (Porcelain Test - P_t), and 6. Transbond XT to porcelain (Porcelain Control -P_c). The overall experiment control was the Transbond XT to enamel (Tooth Control - T_C). Preparation of the bonding surface each groups was as follows: 1.(T_t) Coarse pumice debridement, 2. (T_c) Phosphoric acid etching, 3. (C_c) Coarse pumice debridement, 4. (C_t) Coarse pumice debridement, 5. (P_t) Diamond bur roughening, and 6. (P_c) Hydrofluoric acid etching. For groups involving coarse pumice debridement (Groups 1. T_t, 3. C_c, and 4. C_t), non-fluoridated coarse pumice in a slurry of water was applied to the substrate using a slow-speed handpiece equipped with a prophylactic cup for fifteen (15) seconds. The surface was then rinsed with water for ten (10) seconds and air dried five (5) seconds. 34% Phosphoric acid etch (T_c) (Figure 5.2.2.1) was applied for ten (10) seconds, rinsed with water for ten (10) seconds and air dried to a light chalk appearance.

Figure 5.2.2.1: 34% Phosphoric Acid Etch



The primer was applied, air thinned and cured using the American Orthodontic Blue Ray microflash LED curing light for ten (10) seconds. A coarse diamond bur (P_1) was equipped in a high-speed handpiece and was applied with light pressure to roughen the porcelain surface and remove the glaze. Efforts were made to not damage or reduce the porcelain surface unnecessarily. 9.4% Hydrofluoric acid (P_c) (Figure 5.2.2.2) was applied for two (2) minutes, rinsed with water for thirty (30) seconds and air dried five (5) seconds to a light chalk appearance.

Figure 5.2.2.2: 9.4% Hydrofluoric Acid Etch



The primer was applied with a brush provided by the manufacturer, air thinned for three (3) seconds and cured using the American Orthodontic Blue Ray Microflash LED curing light for ten (10) seconds.

With each group, the desired adhesive, either Transbond XT or Vertise Flow, was applied to the orthodontic button base and transferred to the appropriate substrate for each test group. A condenser instrument was applied to the button head with light pressure to seat the button to the substrate. Excess flash material was removed using an explorer instrument. The American Orthodontics Blue Ray Microflash LED curing light was used to cure the adhesive for twenty (20) seconds, rotating around the button every four (4) seconds.

Table 5.2 provides a summary of the experimental design showing how the six (6) test groups were subdivided into three subgroups to test each of the different adhesive/substrate combinations at the three time points.

Table 5.2: Experiment Design

Group	Adhesive and Substrate	Subgroups	Time prior to debond	Number of Samples
1 (T _t)	Vertise Flow to Tooth	1	24 hours	15
		2	7 days	15
		3	3 months	15
2 (T _c)	Transbond XT to Tooth (control)	1	24 hours	15
		2	7 days	15
		3	3 months	15
3 (C _c)	Vertise Flow to Herculite (control)	1	24 hours	15
		2	7 days	15
		3	3 months	15
4 (C _t)	Vertise Flow to Filtek Supreme	1	24 hours	15
		2	7 days	15
		3	3 months	15
5 (P _t)	Vertise Flow to Porcelain	1	24 hours	15
		2	7 days	15
		3	3 months	15
6 (P _c)	Transbond XT to Porcelain (control)	1	24 hours	15
		2	7 days	15
		3	3 months	15
TOTAL				270

Table Note: 1. Tooth Test (T_t), 2. Tooth Control (T_c), 3. Composite Control (C_c), 4. Composite Test (C_t), 5. Porcelain Test (P_t), 6. Porcelain Control (P_c)

5.2.6. Debonding Procedure

Randomized selection of samples from each group were loaded into the Bencor Multi-T testing apparatus (Figure 5.2.3.1) and placed on the Zwick Universal Testing machine (Figure 5.2.3.2). A 10kN load cell operating with a crosshead speed of 0.5mm per minute

and an attachment diameter of 3.75mm were the computer software parameters used for each trial. The Zwick Universal Testing machine consists of a steel rod attached to a crosshead which when activated contacts the mounted bracket and shears it off. A computer electronically connected to the Universal Test Machine records the strength of the bond of each trial in megapascals. The machine was set to run until separation of the button from the substrate was achieved. The value of the shear bond strength obtained from the computer was recorded on an Excel (Microsoft) spreadsheet. Trials were performed at three (3) time points: less than 24hrs, 7 days, and 3 months.

Figure 5.2.3.1: Bencor Multi-T Apparatus

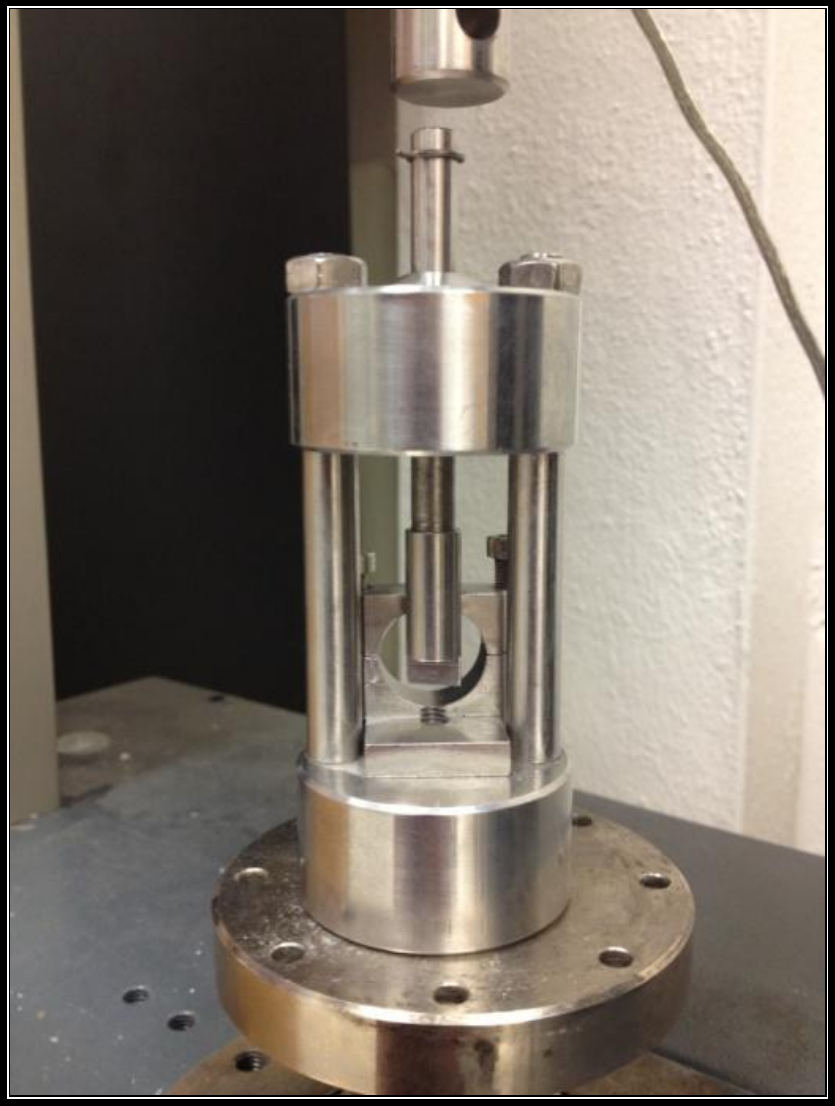


Figure 5.2.3.2: Zwick Universal testing machine and computer



5.2.7. Adhesive Remnant Index

Ten debonded buttons were evaluated by two examiners to reduce bias in the interpretation of the Adhesive Remnant Index (ARI) scores. Inter-examiner and intra – examiner reliabilities were excellent at 100%.

Ten (10) buttons per subgroup, two-thirds of the sample, were evaluated to obtain the ARI scores. This was due to some buttons being non-retrievable or lost during the testing. The amount of adhesive remaining on the button bonding surface after debond was examined

by a single evaluator under direct vision and appointed a score according to the Adhesive Remnant Index adapted from Bishara et al. 1999 (Figure 5.2.7).

Adhesive Remnant Index score (1-5):

Score 1 = 0% left on bracket; 100% left on substrate

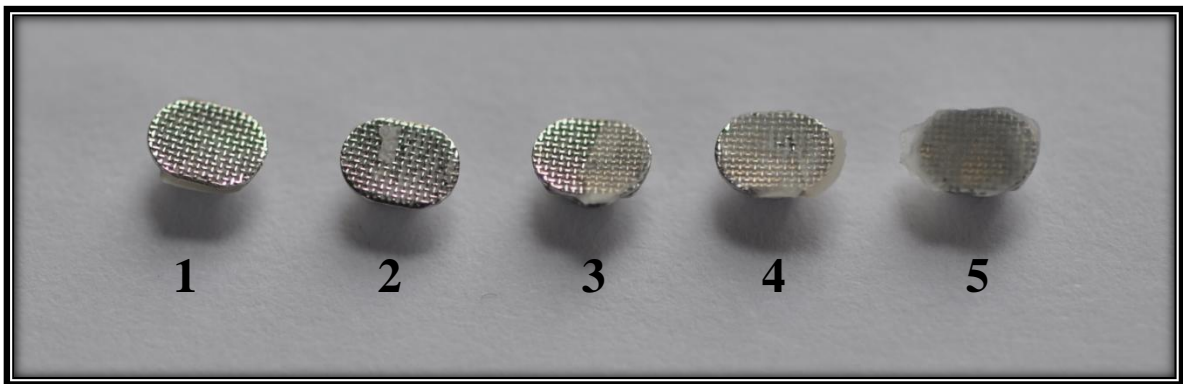
Score 2 = <10% left on bracket; >90% left on substrate

Score 3 = 10-90% left on bracket; 10-90% left on substrate

Score 4 = >90% left on bracket; <10% left on substrate

Score 5 = 100% left on bracket; 0% left on substrate

Figure 5.2.7: Representative Samples of Adhesive Remnant Index Scoring



5.2.8. Statistical Analysis

Three (3) trials recorded a shear bond strength value of 0 MPa because the attachments debonded as they were being loaded into the Bencor Multi-T testing apparatus and were excluded from the analysis.

Statistical analysis was calculated using GraphPad Prism version 6.00 for Windows (GraphPad Software, La Jolla California USA, www.graphpad.com) software. Descriptive statistics and the D'Agostino & Pearson omnibus normality test were performed to determine which statistical tests were applicable to a subgroup. Statistical significance was established at a threshold of $p < 0.05$. Statistical assessment was performed on the SBS data obtained for each group (Tooth $T_t:T_c$, Composite $C_t:C_c$, and Porcelain $P_t:P_c$) at each time point (T1, T2, and T3) and within each subgroup (T_t , T_c , C_t , C_c , P_t , P_c). Median ARI scores and significance within each subgroup were also determined. Statistical significance was determined for subgroups displaying a Gaussian properties using unpaired t tests and applying Welch's correction when applicable. Non-Gaussian subgroups were analysed using Mann-Whitney tests. Multiple subgroups comparisons were performed using analysis of variance (ANOVA) with Tukey's multiple comparisons test for Gaussian subgroups and Kruskal-Wallis test with Dunn's multiple comparisons test for non-Gaussian subgroups.

6. RESULTS

6.1. Shear Bond Strength

6.1.1. 24 hours

Table 6.1.1 and Figure 6.1.1.1 summarize the SBS at 24hrs.

Table 6.1.1: Descriptive Statistics at 24 hours

Group	n	Mean (MPa)	Std. Dev. (MPa)	Min (MPa)	Max (MPa)	Coeff. Var. (%)	p-value
1 (T _t)	15	8.69	3.33	4.54	14.7	38.3	p>0.05
2 (T _c)	15	11.08	6.39	2.55	23.5	57.6	
3 (C _c)	14	27.44	8.47	13.91	41.6	30.8	p<0.01
4 (C _t)	15	17.45	4.83	9.06	24.43	27.7	
5 (P _t)	14	21.86	8.42	11.44	34.94	38.5	p<0.01 ^a
6 (P _c)	15	13.98	4.22	9.83	24.15	30.2	

Table Note: 1. Vertise Flow to Tooth (T_t), 2. Transbond XT to Tooth (T_c) 3. Vertise Flow to Herculite Ultra (C_c) 4. Vertise Flow to Filtek Supreme Ultra (C_t) 5. Vertise Flow to Porcelain (P_t) 6. Transbond XT to Porcelain (P_c)

^a Mann-Whitney test used.

All data sets were normally distributed with the exception of P_c. Statistical significance was found in composite and porcelain categories, p=0.0009 and p=0.0027 respectively. Vertise Flow had weaker average bond strength but higher minimum bond strength to tooth compared to its control, Transbond XT to enamel. Vertise Flow had higher bond strength to Herculite Ultra and porcelain both in terms of mean and minimum values. Figure 6.1.1.2 illustrates the average values obtained with error bars denoting one standard deviation. The coefficients of variation were within acceptable values (~30%), except for Groups T_t, T_c, and P_t. The most consistent group was C_t.

Figure 6.1.1.1: Quartile and Extreme Distribution of 24 hours Shear Bond Strength

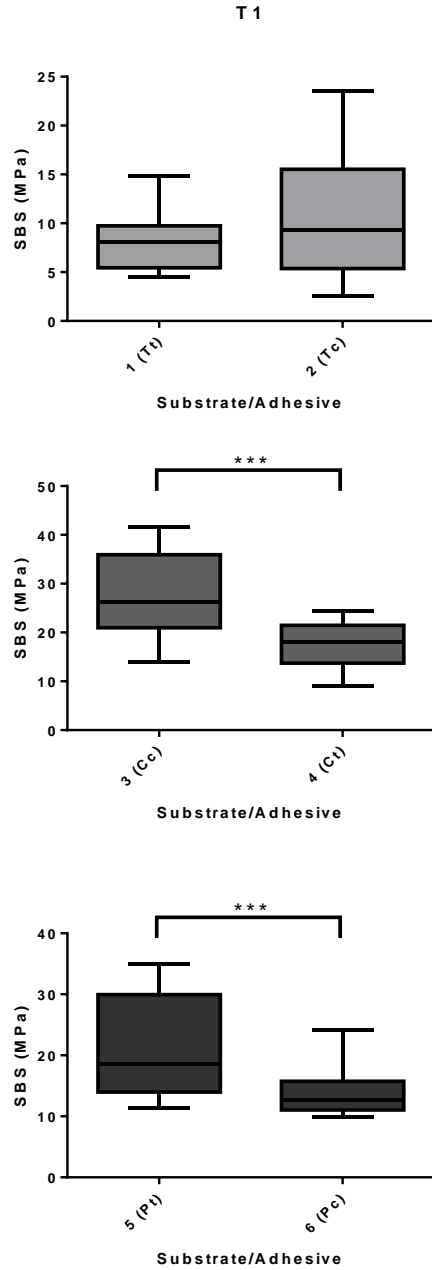


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc); Triple asterix denotes significance $p < 0.01$.

Figure 6.1.1.2: 24 hours Average Shear Bond Strengths with one standard deviation

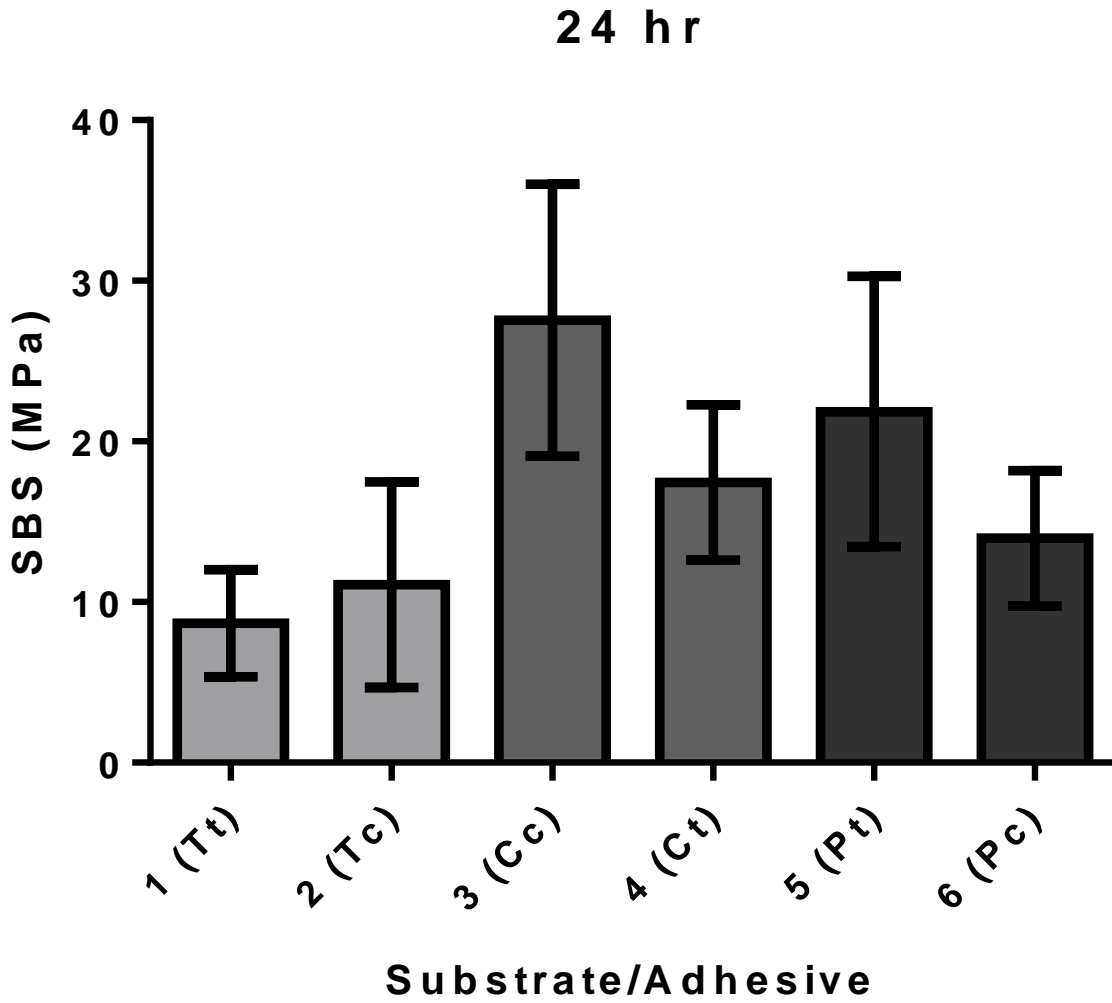


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc)

6.1.2. After 7 days

Table 6.1.2 and Figure 6.1.2.1 summarize the SBS at 7 days.

Table 6.1.2: Descriptive Statistics after 7 days

Group	n	Mean (MPa)	Std. Dev. (MPa)	Min (MPa)	Max (MPa)	Coeff. Var. (%)	p-value
1 (T_t)	15	8.89	1.85	5.05	12.25	20.8	p<0.01
2 (T_c)	15	22.81	9.89	9.90	43.14	43.4	
3 (C_c)	15	22.88	11.68	6.54	47.66	51.0	p<0.05 ^a
4 (C_t)	15	17.26	9.04	8.49	42.20	52.4	
5 (P_t)	15	21.20	5.14	8.55	27.61	24.2	p>0.05
6 (P_c)	15	17.37	5.74	9.34	29.53	33.0	

Table Note: 1. Vertise Flow to Tooth (T_t), 2. Transbond XT to Tooth (T_c) 3. Vertise Flow to Herculite Ultra (C_c) 4. Vertise Flow to Filtek Supreme Ultra (C_t) 5. Vertise Flow to Porcelain (P_t) 6. Transbond XT to Porcelain (P_c)

^a Mann-Whitney Test used.

All data sets were normally distributed with the exception of C_t. Statistical significance was found between categories involving enamel and composite as the bonding substrate, p<0.01 and p=0.499 respectively. Vertise Flow had weaker mean SBS to tooth enamel but stronger mean SBS to Herculite Ultra and porcelain. With respect to minimum values, Vertise Flow had lower SBS to all substrates. The coefficients of variation were within acceptable values (~30%), except for Groups T_c, C_c and C_t. The most consistent group was T_t. Figure 6.1.2.2 illustrates the average values obtained with error bars denoting one standard deviation.

Figure 6.1.2.1: Quartile and Extreme Distribution of 7 days Shear Bond Strength

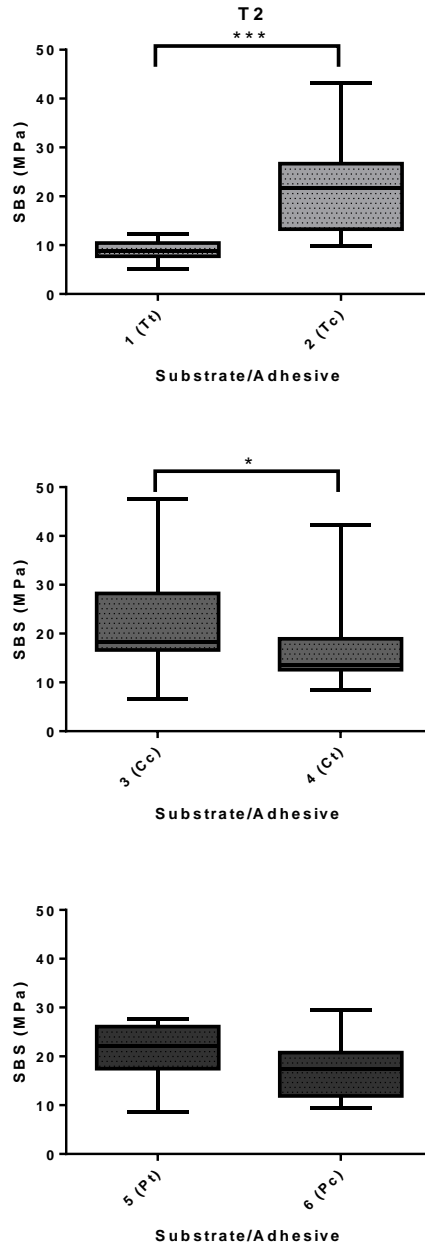


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc); Single asterix denotes significance $p < 0.05$; Triple asterix denotes significance $p < 0.01$.

Figure 6.1.2.2: 7 days Mean Bond Strength with one standard deviation

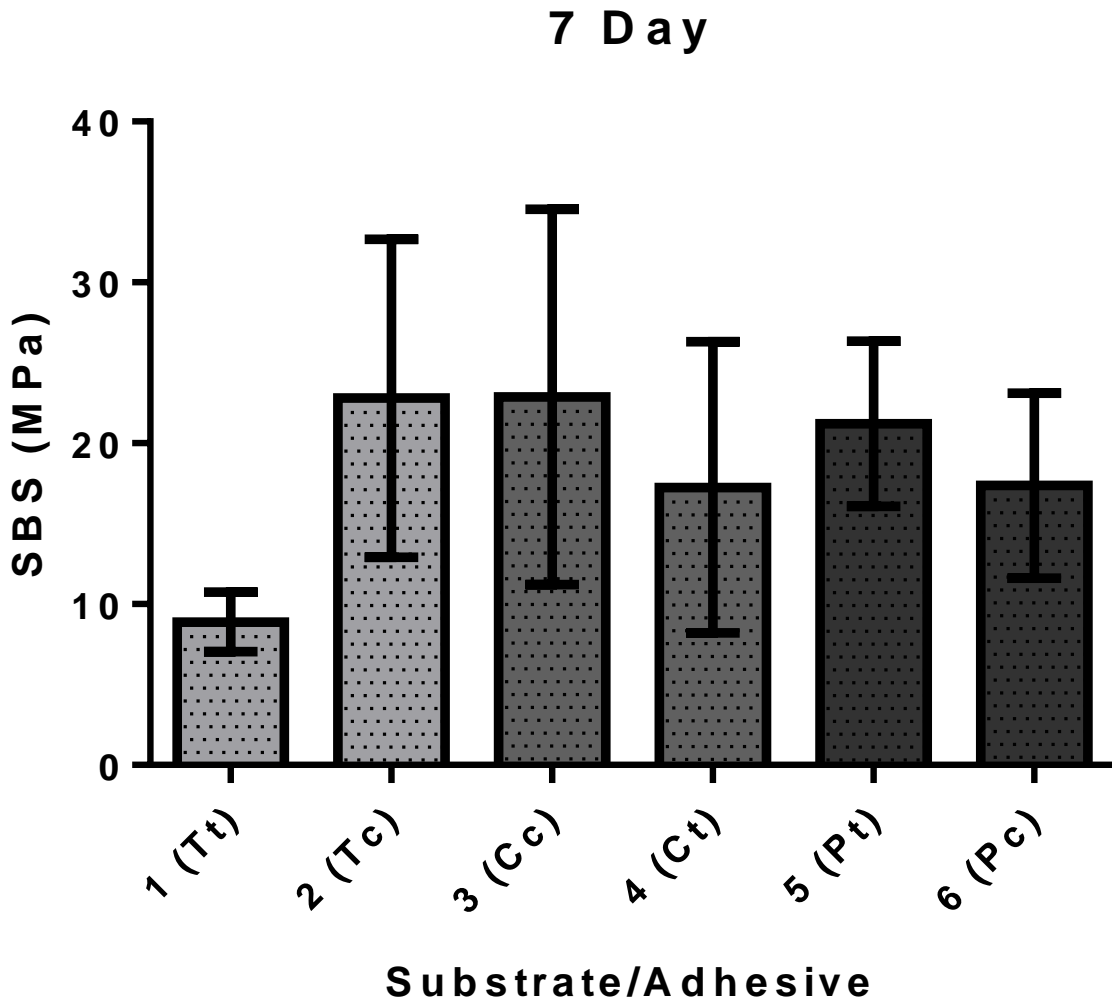


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc)

6.1.3. After 3 months

Table 6.1.3 and Figure 6.1.3.1 summarize the SBS at 3 months.

Table 6.1.3: Descriptive Statistics after 3 months

Group	n	Mean (MPa)	Std. Dev. (MPa)	Min (MPa)	Max (MPa)	Coeff. Var. (%)	p-value
1 (T _t)	15	10.85	4.40	4.58	18.43	40.5	p<0.02
2 (T _c)	15	19.10	10.87	5.99	39.29	56.9	
3 (C _c)	14	18.96	8.36	4.58	34.94	44.1	p<0.05
4 (C _t)	15	13.13	4.47	2.96	21.17	34.1	
5 (P _t)	15	12.20	5.30	3.21	22.97	43.4	p>0.05
6 (P _c)	15	14.70	5.75	6.48	28.49	39.1	

Table Note: 1. Vertise Flow to Tooth (T_t), 2. Transbond XT to Tooth (T_c) 3. Vertise Flow to Herculite Ultra (C_c) 4. Vertise Flow to Filtek Supreme Ultra (C_t) 5. Vertise Flow to Porcelain (P_t) 6. Transbond XT to Porcelain (P_c)

All data sets were normally distributed. Statistical significance found in the tooth and composite categories, $p=0.014$ and $p=0.031$ respectively. Vertise Flow continued to have lower mean bond strength compared to its control. No statistical significance was found for the category involving porcelain as the bonding surface, $p=0.226$. Vertise Flow continued to have higher mean bond strength to Herculite Ultra but this time had a lower mean shear bond strength to porcelain compared to control, Transbond XT to porcelain. Vertise Flow had the lowest minimum shear bond strength value for each substrate pairing. All bond strengths obtained exceeded 4 MPa suggesting clinical longevity at 3 months, however Vertise Flow to

Herculite Ultra encroached closely to the value, 4.58 MPa. Figure 6.1.3.2 illustrates the average values obtained with error bars denoting one standard deviation.

Figure 6.1.3.1: Quartile and Extreme Distribution of 3 months Shear Bond Strength

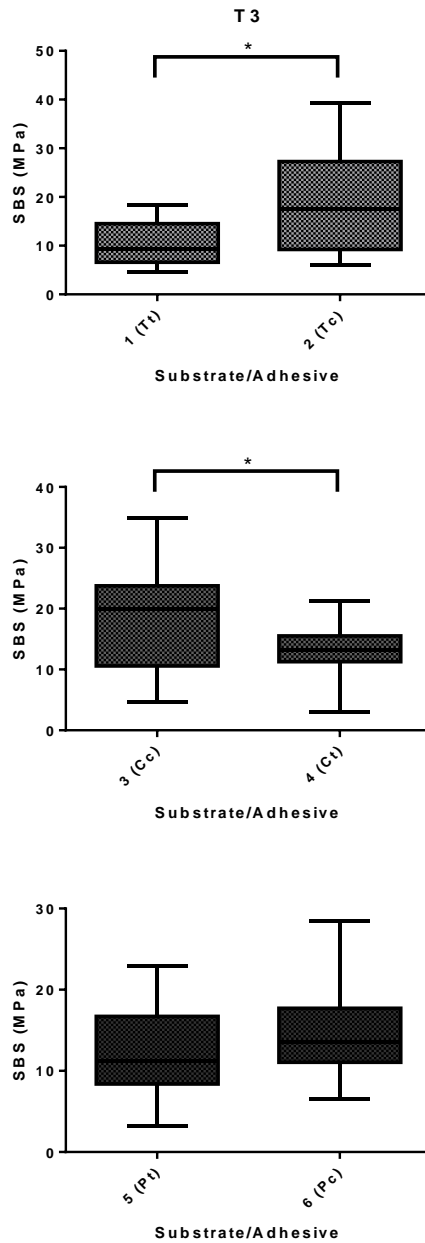


Figure note:1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc); Asterix denotes significance $p < 0.05$.

Figure 6.1.4.2: 3 months Mean Bond Strengths with one standard deviation.

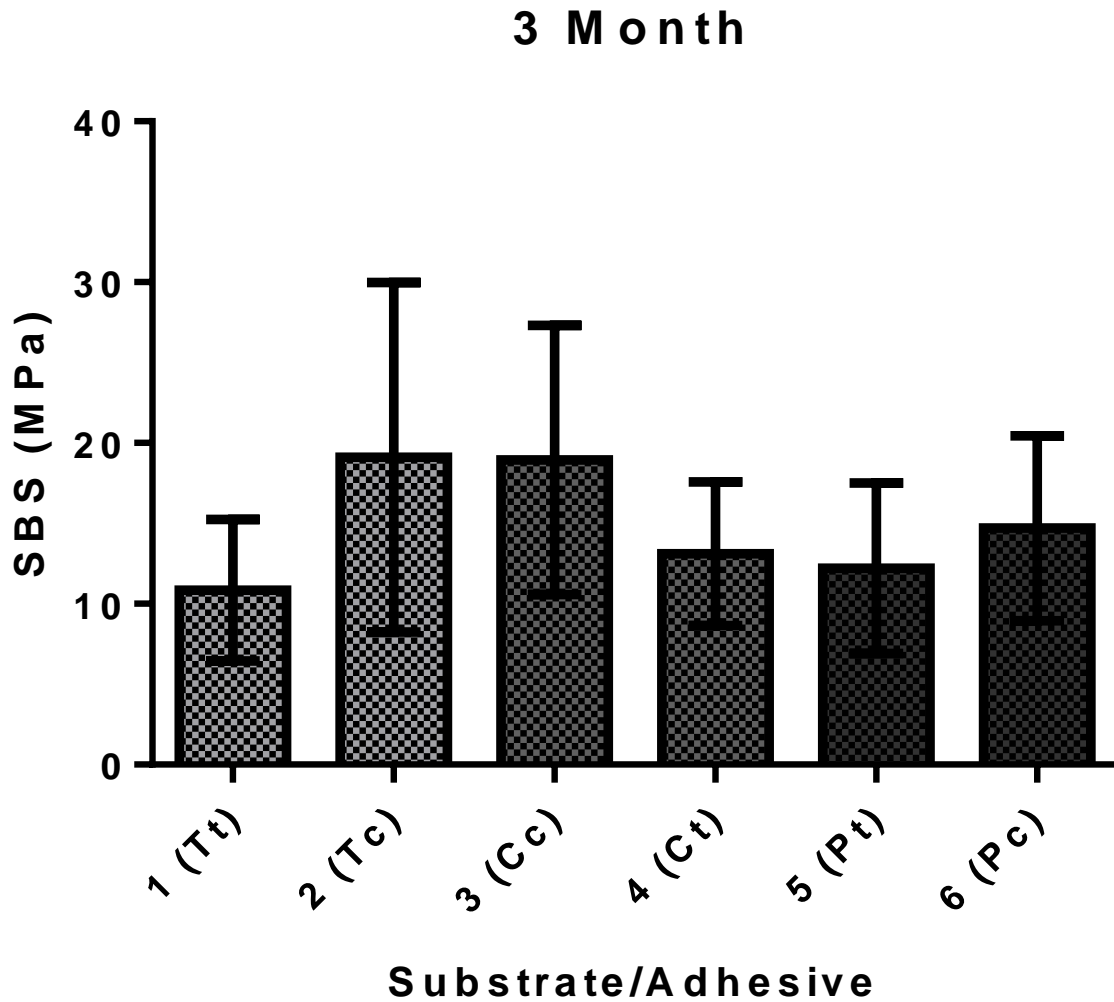


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc)

6.2. Statistical Analysis of Subgroups

Figure 6.2 summarizes the within group statistical significance.

Table 6.2: Subgroup Analysis within each Group

Group	Time Point	Mean SBS (MPa)	Std. Dev. (MPa)
1 (T _i)	T1	8.69	3.33
	T2	8.89	1.85
	T3	10.85	4.40
2 (T _e)	T1	11.08	6.39
	T2	22.81	9.89
	T3	19.10	10.87
3 (C _e)	T1	27.55	8.47
	T2	22.88	11.68
	T3	18.96	8.36
4 (C _i)	T1	17.45	4.83
	T2	17.26	9.04
	T3	13.12	4.47
5 (P _i)	T1	21.86	8.42
	T2	21.20	5.14
	T3	12.20	5.30
6 (P _e)	T1	13.98	4.22
	T2	17.37	5.74
	T3	14.70	5.75

Vertise Flow to enamel showed a progressive increase in SBS that was not statistically significant ($p > 0.05$). Transbond XT to enamel showed a highly significant increase in SBS as the adhesive matured in the first week (T2) ($p < 0.01$), which decreased insignificantly thereafter (T3). Vertise Flow to restorative resin composites, Herculite Ultra and Filtek Supreme Ultra, showed similar progressive insignificant declining trend. Vertise Flow to porcelain had a similar trend to Transbond XT to enamel, showing a maturation within the first week; however, it was not significant. At T3, Vertise Flow to porcelain showed a significant drop in SBS, $p < 0.01$. Transbond XT to porcelain also displayed a similar trend to

Transbond XT to enamel with a significant increase in SBS at one week (T2) which degraded insignificantly thereafter. The net SBS was statistically insignificant to initial SBS values.

Figure 6.2: Mean bond strengths with one standard deviation with Multiple Comparisons

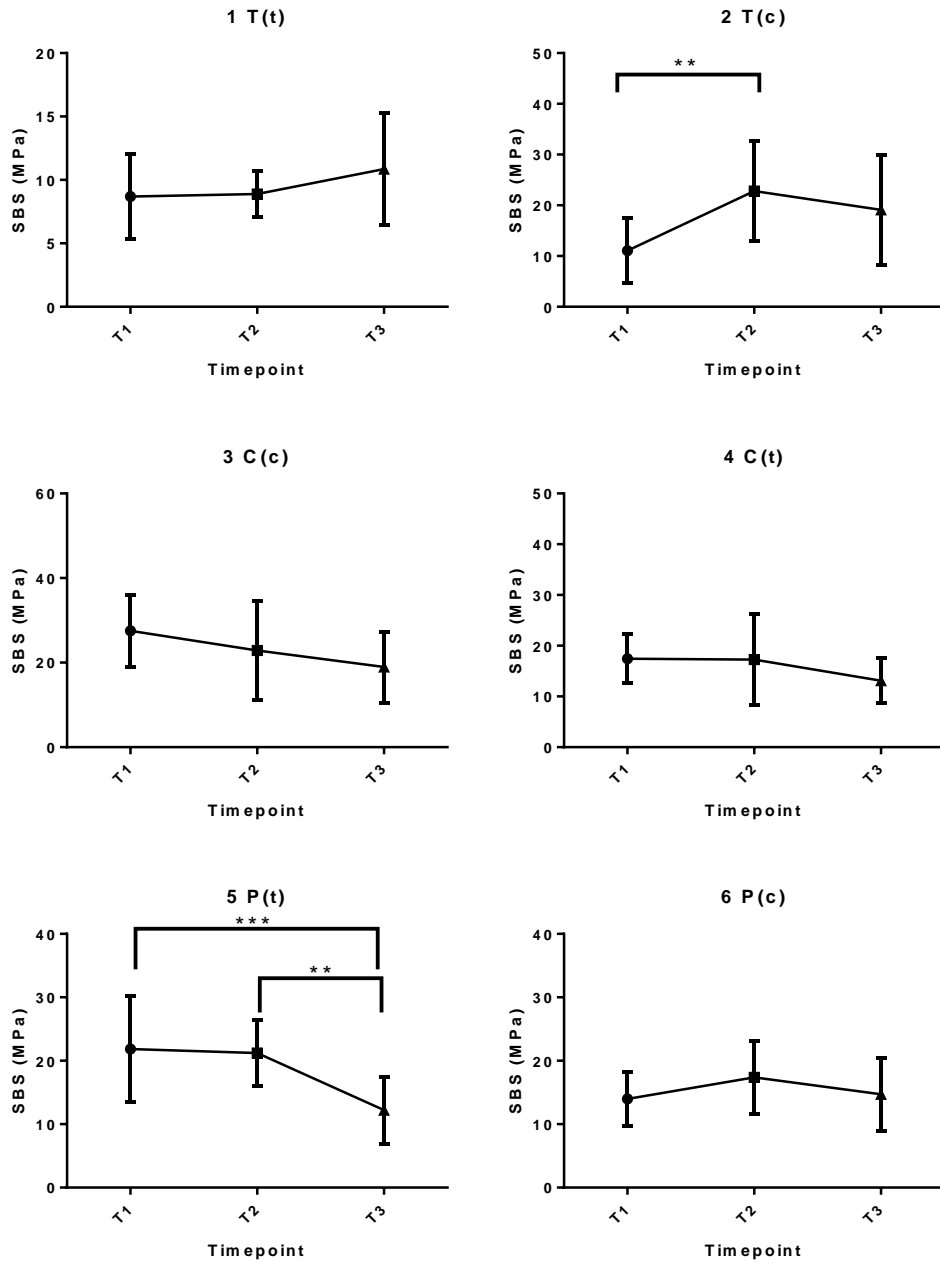


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc); Asterix denotes significance p<0.05.

6.3. Adhesive Remnant Index

Fractures were all adhesive-cohesive in nature with most of the values, 93.3%, being between 3 and 5, i.e. most of the adhesive remaining on the button base. The lowest median score, 3, was achieved at T1:C_c, T2:T_c and T3:C_t, while the highest median score, 5, was achieved at T1:T_t, T2:T_t, T3:T_t, T1:T_c, T1:P_c, T2:P_c, T3:P_c and T3:P_t. Table 6.3 shows the frequency of ARI scores in this study. Figure 6.3 shows the results of the Kruskal-Wallis Test with Dunn's multiple comparisons test. The median values for ARI scores were similar within each group at each time point. The only significant difference ($p < 0.05$) occurred in T_c from T1 to T2 with a change from 5 to 3.

Table 6.3: Adhesive Remnant Index Score Tally

Group	Timepoint	ARI ¹ Score					Total
		1	2	3	4	5	
1 (T_t)	T1	0	1	0	1	8	10
	T2	0	0	0	1	9	10
	T3	0	0	0	3	7	10
2 (T_c)	T1	0	1	0	2	7	10
	T2	1	2	3	2	2	10
	T3	0	0	5	1	4	10
3 (C_c)	T1	1	3	3	1	2	10
	T2	0	0	3	4	3	10
	T3	0	1	4	2	3	10
4 (C_t)	T1	0	1	2	5	2	10
	T2	0	0	3	3	4	10
	T3	0	1	6	1	2	10
5 (P_t)	T1	0	0	2	5	3	10
	T2	0	0	4	2	4	10
	T3	0	0	2	1	7	10
6 (P_c)	T1	0	0	2	2	6	10
	T2	0	0	3	1	6	10
	T3	0	0	0	4	6	10

¹ARI: Score 1 = 0% adhesive left on button base; 100% adhesive left on substrate; Score 2 = <10% adhesive left on button base; >90% adhesive left on substrate; Score 3 = 10-90% adhesive left on button base; 10-90% adhesive left on substrate; Score 4 = >90% adhesive left on button base; <10% adhesive left on substrate; Score 5 = 100% adhesive left on button base; 0% adhesive left on substrate

Note: 1. Vertise Flow to Tooth (T_t), 2. Transbond XT to Tooth (T_c) 3. Vertise Flow to Herculite Ultra (C_c) 4. Vertise Flow to Filtek Supreme Ultra (C_t) 5. Vertise Flow to Porcelain (P_t) 6. Transbond XT to Porcelain (P_c)

Figure 6.3 ARI Subgroup statistical multiple comparisons – median with range (Kruskal-Wallis)

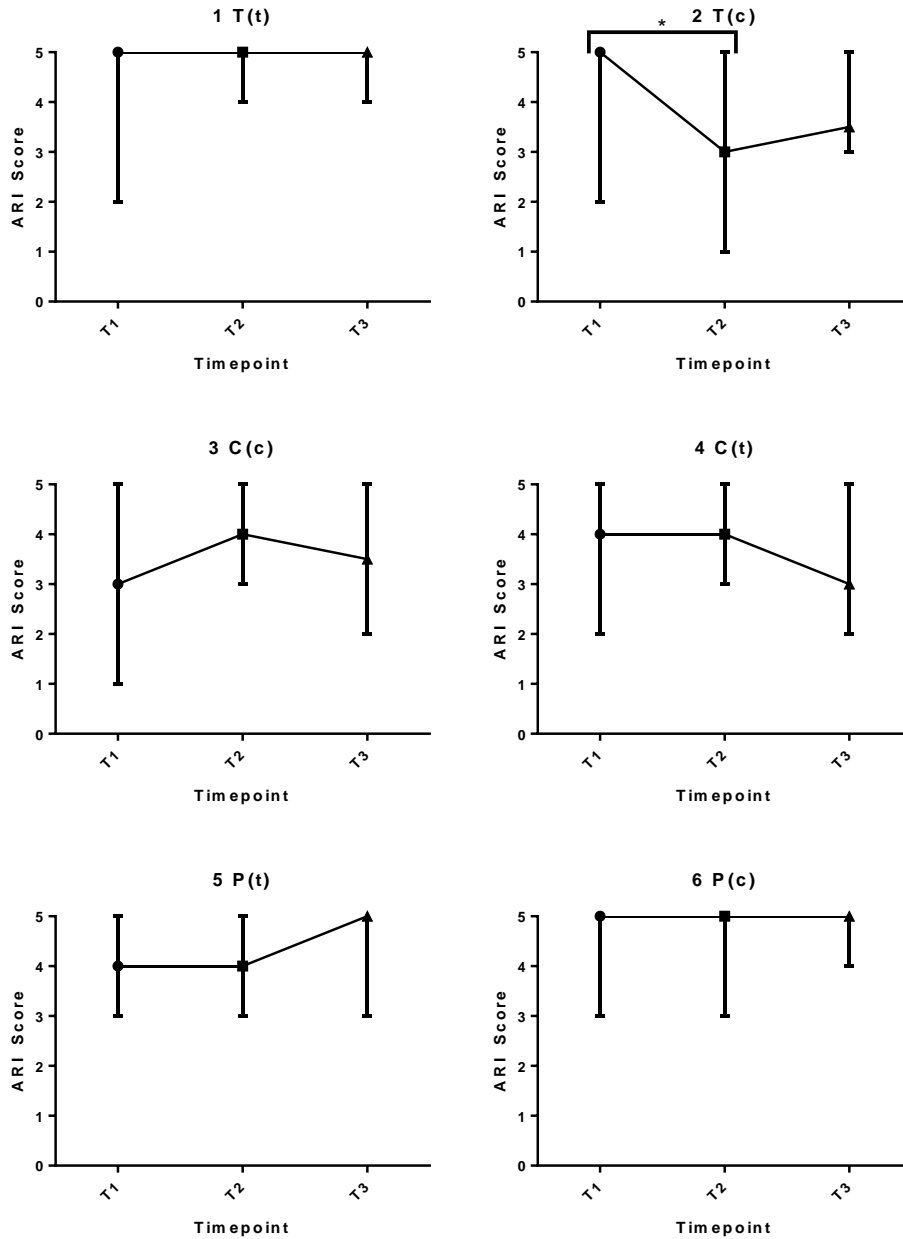


Figure note: 1. Vertise Flow to Tooth (Tt), 2. Transbond XT to Tooth (Tc) 3. Vertise Flow to Herculite Ultra (Cc) 4. Vertise Flow to Filtek Supreme Ultra (Ct) 5. Vertise Flow to Porcelain (Pt) 6. Transbond XT to Porcelain (Pc); Asterisk denotes significance $p < 0.05$.

7. DISCUSSION

Understandably, it is desirable to find methods to save chair-time during orthodontic treatment. The introduction of self-adhering flowable resin composite has the potential to address this objective. The claimed advantage of this product is the ability to adhere to various substrates with minimal surface preparation including mineralized tooth structure, restorative composite resin and porcelain. The present study intended to investigate the orthodontic shear bond strength of Vertise Flow, a self-adhering flowable resin composite, to three substrates (enamel, restorative resin composite, and porcelain), with minimal surface preparation.

7.1. Shear Bond Strength

For the purpose of bonding orthodontic attachments, an ideal shear bond strength of 6-8 MPa has been suggested (Reynolds, 1975). This number has been challenged by the successful clinical performance of glass ionomer cements with *in vitro* SBS outcomes in the 3-4 MPa range (Fricker, 1994; Fricker, 1998; Wiltshire & Noble, 2010). Traditionally, orthodontic adhesives do not perform with a static SBS value, but rather, dynamically change over time. Light photons initiate radical formation and the hardening, by means of crosslinking unreacted monomers, of light cured resins. This reaction continues until there is no further monomer available to react. Complete maturation is not instantaneous but progresses hours to days following the initial set. This is evident with higher SBS values being obtained after 24 hours than those obtained immediately following the initial cure.

Once complete maturation has been achieved, degradation of the bond begins with water sorption, or other means, and gradual decreases in SBS occur. This is enhanced by thermal exposure with expansion and contraction of the adhesive interfaces. This process is replicated in the laboratory by thermocycling the samples. Furthermore, the acquisition of the SBS value can vary depending on the testing parameters used. According to a recent thesis by Cheba (2012), shear bond strength in *in vitro* studies can be influenced by load cell configuration and not by amount of light curing or crosshead speed.

Interpretation of both the mean shear bond strength value and the range expressed of an orthodontic adhesive in an experiment are important for study comparison.

In this study, Transbond XT to enamel was used as both a control for tooth substrate and as the overall control. Transbond XT has been used in other SBS studies at the University of Manitoba (Ho *et al.*, 2010; Phan *et al.*, 2011; Cheba *et al.*, 2012). The mean and range 24hr SBS values (11.08 ± 6.39 MPa; [2.55-23.5 MPa]) for Transbond XT to enamel in this study using the Zwick Universal Testing machine with a 10kN load cell and a crosshead speed of 0.5mm/min were similar to the results achieved by Nemeth *et al.* (2006) (10.57 ± 2.83 MPa; [7.11-15.73 MPa]) and lower than the results achieved with the same machine and configuration by Cheba *et al.* (2012) (14.32 ± 2.31 MPa; [5.97-19.32 MPa]). Furthermore, in the study Phan *et al.* (2011) conducted investigating the SBS of Transbond XT and ProSeal to bleached and unbleached teeth, a 1kN load cell and a crosshead speed of 0.5mm/min configuration of the Zwick machine also yielded a higher mean SBS (18 ± 4.14 MPa; [10.13-

28.95 MPa]). Ho *et al.* (2010), with similar testing parameters to Phan *et al.* (2011), achieved immediate (< 5mins) results in close agreement to the present study (11.22 ± 1.98 MPa; [7.91-14.92 MPa]) but higher at 24hr (16.65 ± 6.04 MPa; [2.63-26.87 MPa]). The second and third time points in this study, 7 days and 3 months, had higher mean SBS and range values, 22.24 ± 2.24 MPa [11.51-42.49 MPa] and 18.56 ± 2.62 MPa [7.52-36.71 MPa] respectively, than the immediate results achieved in the other studies likely due to the adhesive having matured by T2. Nemeth *et al.* (2006) had evaluated the SBS of Transbond XT to enamel at six months and achieved value of 12.23 ± 3.14 MPa [9.10-17.67 MPa]. Outside of the University of Manitoba studies, SBS of Transbond XT have been obtained. Isman 2013 reported a 24hr SBS of 9.86 ± 3.20 MPa using an AGS-X Universal Testing machine (Shimadzu, Kyoto, Japan) with unknown load cell and a crosshead speed of 1mm/min configuration parameters. Goracci *et al.* (2013) reported a SBS of 9.80 ± 2.28 MPA, 30 minutes after bonding.

The minor differences in SBS for Transbond XT to enamel achieved in the different experiments outlined when compared to the result in this experiment may be attributed to three possibilities:

- 1) enamel maturation – the collection of the teeth used in this study was anonymous.

Whether the extracted tooth had erupted or had been intra-osseous prior to extraction was unknown.

- 2) the load cell used, i.e. 1kN would yield higher SBS than the 10kN

- 3) the first time point (up to 24 hrs) in the present experiment spanned a greater time frame as the six groups were randomized. Some attachments were debonded closer to five minutes while others were approaching 24hrs. This would result in a lower average.

On the whole, based on the experiment control (Transbond XT to enamel), the results from this experiment are consistent with the results achieved in previous studies performed at the University of Manitoba and abroad. The SBS values obtained in the other groups tested are valid for comparison.

7.1.1. Enamel

This was the first study at the University of Manitoba investigating Vertise Flow (Kerr) as a potential orthodontic bonding adhesive. To date, only two other studies have been conducted for this purpose. The 24hrs mean and range SBS values obtained with Vertise Flow to enamel was 8.69 ± 3.33 MPa and 4.54-14.70 MPa. This was lower than the value obtained by Goracci *et al.* (2013) after 30mins (10.13 ± 2.86 MPa) and higher than the 24hr SBS for Vertise Flow (2.55 ± 0.77 MPa) reported by Işman *et al.* (2012). Both investigators, Goracci *et al.* (2013) and Işman *et al.* (2012), achieved higher mean SBS, 11.86 ± 4.17 MPa and 7.89 ± 1.17 MPa respectively, closer to the value achieved in this study, when etching the enamel with 37% phosphoric acid for 15s prior to bonding. Both external studies used premolar teeth that had been cleaned with pumice of unspecified grit (10s Goracci *et al.* (2013); 15s Işman *et al.* (2012)). Goracci *et al.* (2013) did not report the testing machine

configuration used or the range SBS values obtained in their investigation. Işman *et al.* (2012) also did not report the range of SBS values obtained but did report a using a AGS-X Universal Testing machine (Shimadzu, Kyoto, Japan) with unknown load cell and a crosshead speed of 1mm/min configuration parameters. The initial SBS of Vertise Flow to enamel in this study is in agreement the study conducted by Goracci *et al.* (2013) (both found no statistical difference despite this study having Vertise Flow with a lower bond strength than Transbond XT and Goracci's *et al.* (2013) study the opposite) and differ from Işman *et al.* (2012). Işman *et al.* (2012) found Vertise flow, without acid etching, to have a significantly lower initial bond strength compared to Transbond XT. The effect of thermocycling in Goracci's *et al.* (2013) study was significant at lowering the SBS of Vertise Flow, with a mean reduction of 7.14 MPa. This effect was not investigated in the current study.

The differences in SBS for Vertise Flow to enamel achieved in the three studies may be attributed to:

- 1) enamel maturation
- 2) load cell used
- 3) Universal Testing machine calibration
- 4) time elapsed prior to debond
- 5) surface preparation technique

This is the first study to investigate the orthodontic SBS of Vertise Flow at time points greater than 24hrs. It would appear that Vertise Flow does not mature in the same manner as Transbond XT. Contrary to the control, Vertise Flow did not follow the same trend in SBS but instead had a very gradual increase, although insignificant, over time.

The SBS of Vertise Flow to enamel is comparable to the results obtained with glass ionomer cements. Fricker reported no clinical significance in bonding failures between glass ionomer cements and composite resin adhesive over a 12 month period (Fricker, 1994). The resin-modified glass ionomer cements without acid etching have reported mean SBS ranging from 4.4 MPa to 7.49 MPa (Wiltshire, 1994; Banerjee & Banerjee, 2011). Based on this, the results from the current study suggest that it is warranted to reject the null hypothesis and conclude that Vertise Flow is suitable for the bonding of metal orthodontic attachments to enamel.

7.1.2. Restorative Resin Composite

To date, this is the first study to investigate the orthodontic SBS of self-adhering flowable resin to restorative composite. In a study conducted in Turkey, restorative resin composite (Filtek Supreme XT, 3M ESPE, St Paul, MN, USA) underwent accelerated ageing prior to having orthodontic attachment bonded with Transbond XT over several surface preparations (Bayram *et al.*, 2011). The control group (no surface treatment) with thermocycling (1000 cycles, 5-55°C) obtained a 7 day mean and range SBS values of 2.77 ± 0.34 MPa [2.08-3.25 MPa] (Bayram *et al.*, 2011). This value is drastically lower than the results obtained in this

study with Vertise Flow. In contrast, Lai *et al.* (1999) reported a bond strengths of 26.8 MPa, after thermocycling, for a metal brackets bonded to microfilled resin composite with Transbond XT. The present study showed significant differences in the initial SBS values between the two restorative resin composites. This may be attributed to the minor differences in composition of the two restorative resin composites.

In a similar manner to the tooth group, Vertise Flow did not show bond 'typical' bond maturation. Instead, a steady decline in bond strength was observed. This difference persisted, though not significantly, with both groups having progressively lower SBS than the previous time point. The rate of decline in bond strength was greater for Herculite Ultra than Filtek Supreme Ultra and may be due to the initial bond strength of Herculite Ultra being greater than that of Filtek Supreme Ultra.

The differences in SBS among the two groups in this study and other reported SBS values to composite may be attributed to:

- 1) The precise composition of the restorative resins.
- 2) Adhesive used
- 3) Restorative resin artificial Ageing
- 4) Thermocycling

This is the first experiment investigating the SBS for self-adhering flowable resin at time points greater than 24 hours. The SBS achieved in this study suggest that Vertise Flow is suitable for bonding metal orthodontic attachments to restorative resin composite.

7.1.3. Porcelain

To date, this is the first study to investigate the orthodontic SBS of self-adhering flowable resin to porcelain. Using similar test parameters to this study, Jarotski (2000) reported 24 hour mean and range SBS values of an autocure orthodontic bonding resin (Concise, 2M Unitek) to hydrofluoric acid etched porcelain of 9.25 ± 2.91 MPa [5.90-15.48 MPa]. Costa *et al.* (2012) reported 24 hour SBS of 9.81 ± 1.1 MPa and 11.60 ± 1.1 MPa for Transbond XT to porcelain etched with 10% hydrofluoric acid for 20 and 60 seconds respectively. These results are lower than the result achieved in this experiment for Transbond XT to porcelain (13.98 ± 4.22 MPa [9.83-24.15 MPa]). This may be due to the minor differences in porcelain and adhesive compositions, with respect to Jarotski's (2000) study, and due to the amount of time etching, with respect to the Costa *et al.* (2012) study. Bourke and Rock (1999) found significant differences in 24 hour SBS achieved with various surface preparations of porcelain and using a composite resin adhesive (Right On). In their study, they reported a SBS of 3.52 ± 0.24 MPa when the porcelain was prepared with only hydrofluoric acid for 3 minutes. In this study, Vertise Flow had statistically higher SBS to porcelain compared to control, Transbond XT to Porcelain, at 24 hours. At 7 days, this difference no longer persisted and by 3 months Vertise Flow had a statistically insignificant lower SBS. The

surface preparation of the two groups were not the same (diamond roughening versus hydrofluoric acid etching) and may have contributed to the difference.

Silane treatment has been suggested as a way to improve bonding to porcelain (Bourke & Rock, 1999; Jarotski, 2000; Costa, 2012). The reported range of 24 hour mean SBS values for silane treated, hydrofluoric acid etched porcelain with a composite resin adhesive is 10.29-18.69 MPa (Bourke & Rock, 1999; Jarotski, 2000; Costa, 2012). This range is still lower, but is closer to, the 24hour SBS achieved in this study with Vertise Flow to diamond-roughened porcelain.

Both Transbond XT and Vertise Flow had ‘typical’ maturation patterns. The maturation of Vertise Flow from T1 to T2 was insignificant and at T3, a significant drop in SBS from T2 was observed. This reduction, from T2 to T3, is concerning as the rate of degradation was the greatest amongst all the groups tested. The cause for this drastic in SBS drop is unknown, but could be due to continued bond degradation due to watersorption equilibration.

The differences in SBS for Vertise Flow to porcelain achieved in the three studies may be attributed to:

- 1) Porcelain composition
- 2) load cell used
- 3) Universal Testing machine calibration
- 4) surface preparation technique

5) adhesive used

This is the first experiment investigating the SBS for self-adhering flowable resin at time points greater than 24 hours. In the study by Jarotski (2000), he reported a mean and range 6 month SBS of hydrofluoric acid etched porcelain of 9.34 ± 1.93 MPa [6.67-13.28 MPa]. This is lower than both the porcelain groups in this study. The SBS values achieved in this study suggest that Vertise Flow is suitable for bonding metal orthodontic attachments to porcelain.

7.2. Potential Clinical Application and Performance

Although not directly quantified in this study, it is within reason to presume that the bonding method with Vertise Flow was faster than conventional bonding methods. The challenges with the product relate to its fluidity. In its current formulation, Vertise Flow is more fluid than the resin composite adhesives commonly used for orthodontic attachment bonding, e.g. Transbond XT paste. When placing an attachment, minimal amount of material is necessary. If too much adhesive is present on the attachment bonding surface when placed, the resin will spread and allow the metal base to be encased/swallowed by the excess material. This makes the removal of this 'flash' material more difficult. The fluidity also makes the attachment slide easily on the substrate surface. In this investigation, gravity was assisting securing the attachment in place. Clinically, this may not always be the case. The author would predict that using Vertise Flow in its room temperature consistency may pose a challenge at securing a bracket in its ideal position on posterior teeth. Downward flow when using flowable composites for orthodontic bonding has been previously described as a

drawback (Turgut *et al.*, 2011). Refrigerating the product when not in use is suggested to increase the shelf life of the product. This reduces the fluidity of the product but may impact on its chemical reactivity if used directly to bond and its clinical advantage in terms of ease of use. It would therefore be desirable for the manufacturer to provide a more viscous formulation for orthodontic bonding use.

The results in this study encourage the use of Vertise Flow for orthodontic bonding to restorative resin composites and porcelain. Bonding to these substrates can at times be frustrating. The success of Vertise Flow, in this experiment, in bonding to these substrates warrants its place in the arsenal for restorative resin composite and porcelain bonding.

The interface between the substrate and Vertise Flow was most often the site of debond failure. If the site of failure had been more evenly distributed across both interfaces (attachment-adhesive; adhesive-substrate), one may expect that inadvertent damage to the substrate may have occurred as the adhesive remaining on the attachment may have microfractured the substrate on failure. The outcome in this study was advantageous as it suggests that less damage occurs to substrate on debond. This also reduced iatrogenic damage to the substrate upon the removal of residual adhesive post-debond; another time-saving experience.

7.3. Limitations of the current study

- 1) This study did not investigate the effect Vertise Flow with multiple surface preparations for a given substrate. Acid etching has been shown to increase the initial SBS (Işman, 2012; Goracci, 2013).
- 2) The present study did not investigate the impact of bleached, fluorosed, or hypercalcified enamel. Reduced SBS values have been reported for altered enamel. This can contribute difficulty in bonding orthodontic attachments (Wiltshire & Noble, 2010).
- 3) The present study also did not include thermocycling. Thermocycling has been shown to result in lower SBS, and may be too rigorous a test method compared to the *in vivo* situation.
- 4) The present study did not use an artificial saliva storage medium. Saliva may hasten degradation of an adhesive intraorally and contribute to lower SBS.
- 5) Scanning Electron Microscopy was not used in the evaluation of surface damage to substrate. Visually it is difficult to quantify the extent of surface damage the various bonding methods impose on a substrate. However, the main purpose of the study was to evaluate the bond strength and not microscopic surface characteristics during debonding.

8. CONCLUSIONS

From this study the following conclusions can be drawn:

- 1) Metal orthodontic attachments were successfully bonded *in vitro* with Vertise Flow (Kerr) to enamel after storage in 37°C distilled water for a period of up to 3 months, using minimal surface preparation.
- 2) Metal orthodontic attachments were successfully bonded *in vitro* with Vertise Flow (Kerr) to restorative resin composites (Herculite Ultra (Kerr) and Filtek Supreme Ultra (3M ESPE)) after storage in 37°C distilled water for a period of up to 3 months, using minimal surface preparation.
- 3) Metal orthodontic attachments were successfully bonded *in vitro* with Vertise Flow (Kerr) to porcelain (Vitabloc) after storage in 37°C distilled water for a period of up to 3 months, using minimal surface preparation.
- 4) The results of this *in vitro* study show potential for this self-adhering flowable resin to be translated to the clinical situation for bonding of metal orthodontic attachments to human enamel, restorative resin composite and porcelain.
- 5) Vertise Flow may reduce bonding time and chair time in the orthodontics if translated to the clinical situation.

9. RECOMMENDATIONS

9.1. Product Modification

Due to the fluidity of the product, determining whether the product will perform as adequately at lower temperatures is suggested. This may improve the workability of the product and ease the removal of excess material around the bracket base upon initial placement.

9.2. Future Research

Based on the results of this *in vitro* study, a clinical trial utilizing Vertise™ Flow to attach fixed appliances *in vivo* is suggested. Also, investigating the performance of Vertise Flow with different surface preparation techniques is suggested to see if improved SBS can be achieved. Lastly, orthodontic treatment often extends past three months. Investigating a longer term more equivalent to the clinical scenario (24-30 months) is advisable.

10. REFERENCES

- Albaladejo A, Montero J, de Diego RG, López-Valverde A. (2011). Effect of adhesive application prior to bracket bonding with flowable composites. *Angle Orthod* 81(4): 716-720.
- Attar N, Taner TU, Tülümen E, Korkmaz Y. (2007) Shear bond strength of orthodontic brackets bonded using conventional vs one and two step self-etching/adhesive systems. *Angle Orthod* 77(3): 518-23.
- Arici S, Arici N. (2003) Effects of thermocycling on the bond strength of a resin-modified glass ionomer cement: an *in vitro* comparative study. *Angle Orthod* 73:692-696.
- Banerjee S, Banerjee R. (2011) A comparative evaluation of the shear bond strength of five different orthodontic bonding agents polymerized using halogen and light-emitting diode curing lights: An *in vitro* investigation. *Indian J Dent Res* 22:731-2.
- Bayram M, Yeşilyurt C, Kuşgöz A, Ülker M, Nur M. (2011) Shear bond strength of orthodontic brackets to aged resin composite surfaces: effect of surface conditioning. *Eur J Orthod* (33): 174-179.
- Bishara SE, VonWald, L, Olsen ME, Lafoon JF. (1999) Effect of time on the shear bond strength of glass ionomer and composite orthodontic adhesives. *Am J Orthod Dentofac Orthop* 116: 616-20.
- Bishara SE, Ajlouni R, Laffoon JF. (2003) Effect of thermocycling on the shear bond strength of cyanoacrylate orthodontic adhesive. *Am J Orthod Dentofac Orthop* 123: 21-4.
- Bourke BM, Rock WP. (1999) Factors affecting the shear bond strength of orthodontic brackets to porcelain. *Br J Orthod* 26:285-90.
- Brantley WA, Eliades, T. (2001) *Orthodontic Materials: Scientific and Clinical Aspects*. Thieme New York, NY: Georg Thieme Verlag. x, 310 p.p.
- Buonocore MG. (1955) A simple method for increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 34(6): 849-853.
- Cehreli ZC, Kecik D, Kocadereli I. (2009) Effect of self-etching primer and adhesive formulations of the shear bond strength of orthodontic brackets. *Am J Orthod Dentofac Orthop* 135(6): 782-6.
- Cheba V. (2012) The effect of crosshead speed, load cell configuration and curing time on the shear bond strength of orthodontic brackets. (Unpublished MSc. Ortho.). University of Manitoba, Winnipeg.
- Costa AR, Correr AB, Puppini-Rontani RM, Vedovello SAS, Valdrighi HC, Correr-Sobrinho L, Vedovello Filho M. (2011) Effects of Thermocycling and Light Source on the Bond Strength of Metallic Brackets to Bovine Teeth. *Braz Dent J* 22(6): 486-489.

- D'Attilio M, Traini T, Di Iorio D, Varvara G, Festa F, Tecco S. (2005) Shear bond strength, bond failure, and scanning electron microscopy analysis of a new flowable composite for orthodontic use. *Angle Orthod* 75:410-415.
- Daub J, Berzins DW, Linn BJ, Bradley TG. (2006) Bond strength of direct and indirect bonded brackets after thermocycling. *Angle Orthod* 76: 295-300.
- Elekdag-Turk S, Turk T, Isci D, Ozkalayci. (2008) Thermocycling effects on shear bond strength of a self-etching primer. *Angle Orthod* 78(2): 351-56.
- Eustaquio R, LaForrest DG, Moore BK. (1988) Comparative tensile strengths of brackets bonded to porcelain with orthodontic adhesive and porcelain repair systems. *Am J Orthod Dentofac Orthop* (94): 421-425.
- Farah J, Powers J. (2004) The Dental Advisor: Self-etching bonding agents. *J Can Dent Assoc* 70 (7): 446-7.
- Ferracane JL, Berge HX, Condon JF. (1998) In vitro again of dental composites in water effect of degree of conversion, filler volume, and filler/matrix compiling. *J. Biomed Mater Res* 42:465-72.
- Finnema KJ, Ozcan M, Post WJ, Ren Y, Dijkstra PU. (2010). *In vitro* orthodontic bond strength testing: A systematic review and meta-analysis. *Am J Orthod Dentofac Orthop*: 137(5): 615-622e.3.
- Fox NA, McCabe JF, Buckley JG. (1994) A critique of bond strength testing in orthodontics. *Br J Orthod* 21(1): 33-43.
- Fricker JP. (1994) A 12-month clinical evaluation of a light-activated glass polyalkenoate (ionomer) cement for the direct bonding of orthodontic brackets. *Am J Orthod Dentofac Orthop* 105:502-5.
- Fricker JP. (1998) A new self-curing resin-modified glass-ionomer cement for the direct bonding of orthodontic brackets *in vivo*. *Am J Orthod Dentofac Orthop* 113: 384-6.
- Gale MS, Darvell BW. (1999) Thermal cycling procedures for laboratory testing of dental materials. *J Dent* 27: 89-99.
- Girish PV, Dinesh U, Bhat, CS, Shetty PC. (2012). Comparison of shear bond strength of metal brackets bonded to porcelain surface using different surface conditioning methods: an *in vitro* study. *J Contemp Dent Pract* 13(4): 487-93.
- Grubisa HSI, Heo G, Raboud D, Glover KE, Major PW. (2004) An evaluation and comparison of orthodontic bond strength achieved with self-etching primers. *Am J Orthod Dentofac Orthop* 126: 213-9.

- Goracci C, Margvelashvili M, Giovannetti A, Vichi A, Ferrari M. (2013) Shear bond strength of orthodontic brackets bonded with a new self-adhering flowable resin composite. *Clin Oral Invest* 17: 609-617.
- Hajrassie MKA, Khier SE. (2007) *In-vivo* and *in-vitro* comparison of bond strengths of orthodontic brackets bonded to enamel and debonded at various times. *Am J Orthod Dentofacial Orthop* 131-884-90.
- Halpern RM, Rouleau T. (2010) The effect of air abrasion preparation on the shear bond strength of an orthodontic bracket bonded to enamel. *Eur J Orthod.* (32): 224-227.
- Ho ACS, Akyalçin S, Bonstein T, Wiltshire WA. (2011). *In vitro* shear force testing of two seventh generation self-etching primers. *J Orthod* 38:269-274.
- Ho ACS. (2010). Shear bond strength of two new self-etching primers. (Unpublished M.Sc. Ortho) University of Manitoba, Winnipeg.
- Huang TH, Kao CT. (2001). The shear bond strength of composite brackets on porcelain teeth. *Eur J Orthod.* 23:433-439.
- Hulterström AK, Bergman M. (1993) Polishing systems for dental ceramics. *Acta Odonto Scand.* (51): 229-234.
- Işman E, Karaarslan EŞ, Okşayan R, Tunçdemir AR, Üşümez S, Adanir N, Cebe MA. (2012) Inadequate shear bond strength of self-etch, self-adhesive systems for secure orthodontic bonding. *Dent Mater J* 31(6): 947-953.
- Jarotski TJ. (2000) Twenty-four hour and six month evaluation of porcelain surface preparation and orthodontic bond strength. (Unpublished MSc. Ortho.) University of Manitoba, Winnipeg.
- Kao EC, Boltz EC, Johnston WM. (1988) Direct bonding of brackets to porcelain veneer laminates. *Am J Orthod Dentofac Orthop* 94: 458-468.
- Katona TR. (1994) The effects of load location and misalignment on shear/peel testing of direct bonded orthodontic brackets – a finite element model. *Am J Orthod Dentofac Orthop* 106-395-402.
- Katona TR. (1997) A comparison of the stresses developed in tension, shear peel, and torsion strength testing of direct bonded orthodontic brackets. *Am J Orthod Dentofac Orthop* 112: 244-251.
- Kerr. (2012) Vertise Flow Technique Guide. Kerr Sybron Dental Specialties. 80263 Rev.0.
- Kusy RP. (1994) Commentary on Dr. Wiltshire's article: When is stronger better?- Letter to editor. *Am J Orthod Dentofac Orthop* 106:17A.

- Lai PY, Woods MG, Tyas MJ. (1999). Bond strengths of orthodontic brackets to restorative resin composite surfaces. *Aust Orthod J* Apr 15(4):235-45.
- Lopez J. (1980) Retentive shear strengths of various bonding attachment bases. *Am J Ortho*. Jun; 77(6): 669-78.
- Major PW, Koehler JR, Manning KE. (1995) 24-hour shear bond strength of metal orthodontic brackets bonded to porcelain using various adhesion promoters. *Am J Orthod Dentofac Orthop* 108: 322-329.
- MSDS: 3M (2010). Material Safety Data Sheet. Transbond XT Light Cure Adhesive Kit. 3M Unitek. Dec. 22, 2010.
- MSDS: 3M (2009). Material Safety Data Sheet. Filtek Supreme Universal Restorative. 3M ESPE. May 7, 2009.
- MSDS: Kerr (2008a). Material Safety Data Sheet. Vertise Flow, Dental restorative material. Kerr Corporation. Nov 12, 2008.
- MSDS: Kerr (2008b). Material Safety Data Sheet. Herculite Ultra, Restorative Composite. Kerr Corporation. October 2008.
- MSDS: Vita (2008). Material Safety Data Sheet. Vitabloc Mark II for CEREC. Vita Zahnfabrik. April 24, 2008.
- Noble J, Karaiskos NE, Wiltshire WA. (2008) *In vivo* bonding of orthodontic brackets to fluorosed enamel using a adhesion promoter. *Angle Orthod* 78(2): 357-60.
- Nemeth BR, Wiltshire WA, Lavelle CLB. (2006) Shear/peel bond strength of orthodontic attachments to moist and dry enamel. *Am J Orthod Dentofac Orthop* 129: 396-401.
- Özden AN, Akaltan F, Can G. (1994) Effect of surface treatments of porcelain on the shear bond strength of applied dual-cured cement. *J Prosth Dent*. (72): 85-88.
- Paschos E, Westphal JO, Ilie N, Huth KC, Hickel R, Rudzki-Janson I. (2008). Artificial saliva contamination effects on bond strength of self-etching primers. *Angle Orthod* 78(4): 715-21.
- Pickett KL, Sadowsky PL, Jacobson A and Lacefield W. (2001) Orthodontic *in vivo* bond strength: comparison with *in vitro* results. *Angle Orthod* 71: 141-8.
- Phan X. (2011). Effect of tooth bleaching on the shear bond strength of a fluoride-releasing sealant. (Unpublished M.Sc. Ortho.). University of Manitoba, Winnipeg.
- Powers JM, Kim HB, Turner DS. (1997) Orthodontic adhesives and bond strength testing. *Seminars in Orthod*. 3: 147-156.

- Powers JM, Sakaguchi RL, Craig RGRdm. (2006) Craig's restorative dental materials. 12th ed. / John M powers and Ronald L Sakaguchi. ed. St. Louis, Mo. ; [London]: Mosby Elsevier; xvii, 632 p.p.
- Pratt RC, Burgess JO, Schwartz RS, Smith JH. (1989) Evaluation of six porcelain repair systems. J Prosth Dent (62): 11-13.
- Prietsch JR, Spohr AM, Lima da Silva IN, Pinheiro Beck JC, Silva Oshima HM. (2007) Development of a device to measure bracket debonding force *in vivo*. Eur J Orthod (29): 564-570.
- Proffit WR, Fields HW. (2000) *Contemporary Orthodontics*. 3th ed. St. Louis, Mo.: Mosby Elsevier; xii, 742 p.p.
- Retief DH. (1974) Failure at the dental adhesive-etched enamel interface. J Oral Rehabil 1: 265-284.
- Reynolds I. (1975) A review of direct orthodontic bonding. Br J Orthod 2:171-178.
- Rossouw PE. (2010) A historical overview of the development of the acid-etch bonding system in orthodontics. Sem Orthod 16:2-23.
- Ryou DB, Park HS, Kim KH, Kwon TY. (2008). Use of flowable composites for orthodontic bracket bonding. Angle Orthod 78(6):1105-09.
- Shillingburg HT. (1997) Fundamentals of Fixed Prosthodontics. 3rd ed. Quintessence Publishing Co. Inc. x, p.p. 582.
- Solderholm KJM. (1991) Correlation of *in vivo* and *in vitro* performance of adhesive restorative materials: a report of the ASC MD156 Task Group on Testing Methods for the Adhesion of Restorative Materials. Dent Mater 4:118-123.
- Stanford SK, Wozniak WT, Fan PL. (1997) The need for standardization of test protocols. Semin Orthod 3(3):206-9
- Tecco S, Traini T, Capui S, Festa F, de Luca V, D'Attilio M. (2005) A new one-step dental flowable composite for orthodontic use: An *in vitro* bond strength study. Angle Orthod 75:672-677.
- Turgut MD, Attar N, Korkmaz Y, Gokcelik A. (2011) Comparison of shear bond strengths of orthodontic brackets bonded with flowable composites. Dent Mater J 30: 66-71.
- Turk T, Elekdag-Turk S, Isci D, Cakmak F, Ozkalayci N. (2010) Shear bond strength of self-etching primer after 10,000 and 20, 000 thermal cycles. J Adhes Dent 12:117-122.
- Van Noort R. (1989) Principles of adhesion. In: Van Noort, editor. Introduction to dental materials. St. Louis: Mosby, p. 61-71.

Vident.com. (2013) Vitablocks® triluxe forte. url:<http://vident.com/products/cadcam/triluxe-forte/>. Access date: April 17, 2013.

Wiltshire WA. (1994) Shear bond strengths of glass ionomer for direct bonding in Orthodontics. Am J Orthod Dentofac Orthop. 106(2):127-30.

Wiltshire WA, Noble, J. (2010) Clinical and Laboratory Perspective of Improved Orthodontic Bonding to Normal, Hypoplastic and Fluorosed Enamel. Sem in Orthod Vol 16 (1) Mar: 55-65.

Wolf DM, Powers JM, O'Keefe KL. (1993) Bond strength of composite to etched and sandblasted porcelain. Amer J Dent 6:155-158.

Wood DP, Jordan RE, Way DC, Galil KA. (1986) Bonding to porcelain and gold. Am J Ortho 89: 194-204.

Zachrisson YO, Zachrisson BU, Büyükyılmaz T. (1996) Surface preparation for orthodontic bonding to porcelain. Am J Orthod Dentofac Orthop 109: 420-430.

11. APPENDIX

11.1. Ethics Approval

Ethics approval from the University of Manitoba Research and Ethics Board was not required as the human materials used in this project were anonymous.

As per an email correspondence with Shelley Rempel-Rossum of University of Manitoba Research and Equity Board:

It would appear that the extracted teeth you used in your research project were "anonymous" (i.e. in no way could be linked to any individual). If this is indeed the case it would appear that prior ethics approval is not required as per Article 2 of the Tri-Council Policy Statement 2: Ethical Conduct for Research Involving Humans.

Article 2 "REB review is not required for research that relies exclusively on secondary use of anonymous information, or anonymous human biological materials, so long as the process of data linkage or recording or dissemination of results does not generate identifiable information."

Application: "Secondary use refers to the use in research of information or human biological materials originally collected for a purpose other than the current research purpose. Anonymous information and human biological materials are distinct from those that have been coded, and also from those that have been anonymized (see Section A of Chapters 5 and 12)."

Typed of Biological Material (as defined in TCPS2) Human biological materials that may reasonably be expected to identify an individual, alone or in combination with other available information, are considered identifiable biological materials (or biological materials that are identifiable) for the purposes of this Policy.

The following categories, similar to those found in Chapter 5 in regard to categories of information, provide guidance for assessing the extent to which human biological materials could be used to identify an individual:

- * Identified human biological materials - the materials are labelled with a direct identifier (e.g., name, personal health number). Materials and any associated information are directly traceable back to a specific individual.
- * Coded human biological materials - direct identifiers are removed from the materials and replaced with a code. Depending on access to the code, it may be possible to reidentify specific individuals (e.g., a principal investigator retains a key that links the coded material with a specific individual if re-linkage is necessary).
- * Anonymized human biological materials - the materials are irrevocably stripped of direct identifiers, a code is not kept to allow future re-linkage, and risk of re-identification of individuals from remaining indirect identifiers is low or very low.
- * Anonymous human biological materials - the materials never had identify attached to them and risk of identification of individuals is low or very low.

11.2. Journal Article and Submission Confirmation

Detailed Status Information

Manuscript #	062613-475
Current Revision #	0
Submission Date	2013-06-26 21:21:19
<u>Current Stage</u>	Under Review
Title	Orthodontic shear bond strengths of a new self-adhering resin to enamel, restorative composite and porcelain
Running Title	Ortho SBS of a self adhering resin to 3 substrates
Manuscript Type	Original Article
Special Section	N/A
Corresponding Author	William Wiltshire (University of Manitoba)
Contributing Authors	Andrew Bernas , Milos Lekic , Igor Pesun
Financial Disclosure	I have no relevant financial interests in this manuscript.
Abstract	Objective: Determine if Vertise Flow (VF), a new self-adhering flowable composite resin, is suitable for bonding fixed orthodontic appliances to enamel, restorative composite and porcelain with minimal surface preparation. Materials and Methods: Shear bond strengths (SBS) from six groups of fifteen bonded metal lingual buttons were obtained over three time points (T1: 24hrs, T2:7 days, and T3:3 months). Samples had been stored in distilled water and incubated at 37°C and 100% RH. The buttons were debonded with a Zwick Universal Testing machine using a 10 kN load cell and a crosshead speed of 0.5mm/min. After debonding, the buttons were visually evaluated based on a modified Adhesive Remnant Index (ARI). Results: The mean SBS obtained in all groups at each time point were >4 MPa and varied

	between 8.69 MPa and 27.44 MPa. Statistical differences were found within the composite and porcelain groups at T1, and the enamel and composite groups at both T2 and T3. Nearly half of the sample (47.2%) achieved an ARI score of 5 (100% of adhesive left on the button base). Conclusion: VF potentially provides clinically acceptable bond strengths to enamel, restorative resin composite and porcelain with minimal surface preparation. Furthermore, upon debond, minimal adhesive clean-up is required, thus saving valuable chair time. Based on the results of this study, future in vivo investigation is warranted.
Assistant Editor	Not Assigned
Key Words	Shear Bond Strength, Orthodontic, Self-adhering flowable composite
Conflict of Interest	I have no conflict of interest that I should disclose.

Stage	Start Date	End Date	Approximate Duration
Under Review	2013-06-27 11:44:47		
Initial QC Complete	2013-06-27 11:44:47		
Initial QC Started	2013-06-26 21:33:21		
Author Approved Converted Files	2013-06-26 21:33:21		
Waiting for Author Approval of Converted Files	2013-06-26 21:31:41		
File Conversion Complete	2013-06-26 21:31:41		

Waiting for File Conversion	2013-06-26 21:29:57		
	2013-06-26 21:27:28		
Manuscript Submitted	2013-06-26 21:27:28		
Manuscript Files Submitted	2013-06-26 21:27:28		
Preliminary Manuscript Data Submitted	2013-06-26 21:21:21		

AUTHORS

Bernas AJ^a, Wiltshire WA^a, Lekic M^a and Pesun I^b

^a Department of Orthodontics, University of Manitoba

^b Department of Restorative Dentistry, University of Manitoba

Corresponding Author: Wiltshire WA

TITLE

Orthodontic shear bond strengths of a new self-adhering resin to enamel, restorative composite and porcelain.

ABSTRACT

Objective: Determine if Vertise Flow (VF), a new self-adhering flowable composite resin, is suitable for bonding fixed orthodontic appliances to enamel, restorative composite and porcelain with minimal surface preparation.

Materials and Methods: Shear bond strengths (SBS) from six groups of fifteen bonded metal lingual buttons were obtained over three time points (T1: 24hrs, T2:7 days, and T3:3 months). Samples had been stored in distilled water and incubated at 37°C and 100% RH. The buttons were debonded with a Zwick Universal Testing machine using a 10 kN load cell and a crosshead speed of 0.5mm/min. After debonding, the buttons were visually evaluated based on a modified Adhesive Remnant Index (ARI).

Results: The mean SBS obtained in all groups at each time point were >4 MPa and varied between 8.69 MPa and 27.44 MPa. Statistical differences were found within the composite and porcelain groups at T1, and the enamel and composite groups at both T2 and T3. Nearly half of the sample (47.2%) achieved an ARI score of 5 (100% of adhesive left on the button base).

Conclusion: VF potentially provides clinically acceptable bond strengths to enamel, restorative resin composite and porcelain with minimal surface preparation. Furthermore, upon debond, minimal adhesive clean-up is required, thus saving valuable chair time. Based on the results of this study, future *in vivo* investigation is warranted.

INTRODUCTION

Since the introduction of the acid-etching technique by Buonocore¹ in 1955, bonding has evolved substantially and is now an integral part of modern orthodontics². Several generations of bonding agents have been developed. The most recent, self-etchant primers (SEP), have simplified bonding by eliminating the additional acid etching step³. SEPs are bicomponent hydrophilic adhesives and have a shallower etch pattern than conventional etching, with less enamel loss but consequently, lower shear

bond strengths (SBS)⁴. The main component in SEPs is methacrylated phosphoric acid esters⁵. Self-adhering flowable composites (SAFC) incorporate this SEP technology into a flowable composite nanofilled resin (Vertise Flow Technical Bulletin).

There are currently two SAFCs which have been researched from an orthodontic perspective: Maxcem Elite (Kerr, Scafati, Italy) and Vertise Flow (VF, Kerr, Orange, CA)^{6,7}. The performance of SAFC for use in the bonding of orthodontic attachments has not been assessed over an extended time period or to different substrates. The potential ease of use and versatility of this product, with subsequent chair-time saving in modern orthodontic practice, warrants its *in vitro* investigation for possible translation to the clinical situation.

The purpose of this study was to determine if VF would be suitable for orthodontic attachment bonding to enamel and restorative materials (porcelain/composite) with minimal surface preparation.

MATERIALS AND METHODS

Ninety (90) extracted human molars were obtained from an oral surgeon. The teeth were free of decay and restorations. The teeth were cleaned, the roots removed and crowns embedded in a self-curing acrylic block. Similarly, 90 restorative resin block samples (45 Filtek Supreme Ultra (3M, ESPE), 45 Herculite Ultra (Kerr)) and 90 porcelain blocks (VitaBloc, Vident) were prepared.

270 flat lingual buttons (Ormco), each with a calculated average surface area of 11.03 mm², were bonded using one of 6 adhesive/substrate combinations (Table 1). Transbond XT adhesive system (TBxt, 3M, Unitek, Monrovia, CA) was used as the control for the tooth and porcelain categories. To seat the button to the substrate, a condenser instrument was applied to the button head with light finger pressure. Flash material was removed using an explorer instrument. The samples were then stored in 37°C distilled water at 100% relative humidity until debonding.

A randomized selection of fifteen samples from each group were loaded into the Bencor Multi-T testing apparatus and placed on the Zwick Universal Testing machine configured with a 10kN load cell operating with a crosshead speed of 0.5mm/min. Debonding trials were performed at three time points: 24hrs, 7 days, and 3 months.

After debonding, ten buttons from each subgroup were examined under direct vision by a single examiner and were assigned scores based on a modified Adhesive Remnant Index (ARI) adapted from Bishara et al⁸. Examiner reliability was assessed by repeat measurements on a random sample of ten buttons evaluated by two independent examiners. Inter-examiner and intra-examiner reliability was excellent at 100%.

Table 1: Group Preparation Protocols

	Group	Adhesive & Substrate	Surface Preparation	Rinse	Air Dry	Cure	Time Point	N
Tooth	1 T_t	Vertise Flow to Enamel	Coarse Pumice (15s)	10s	5s	20s	24 hr	15
							7 day	15
	2 T_c	Transbond XT to Enamel (control)	H ₃ PO ₄ (10s)	10s	5s	20s	24 hr	15
7 day							15	
	3 C_c	Vertise Flow to Herculite Ultra (control)	Coarse Pumice (15s)	10s	5s	20s	24 hr	15
							7 day	15
	4 C_t	Vertise Flow to Filtek Supreme	Coarse Pumice (15s)	10s	5s	20s	24 hr	15
							7 day	15
	5 P_t	Vertise Flow to Porcelain	Diamond Bur	10s	5s	20s	24 hr	15
							7 day	15
	6 P_c	Transbond XT to Porcelain (control)	HF (2mins)	30s	5s	20s	24 hr	15
							7 day	15
							3 month	15
							Total	270

Note: T- Tooth, C- Composite, P- Porcelain, t- test, c-control

Statistical Analysis

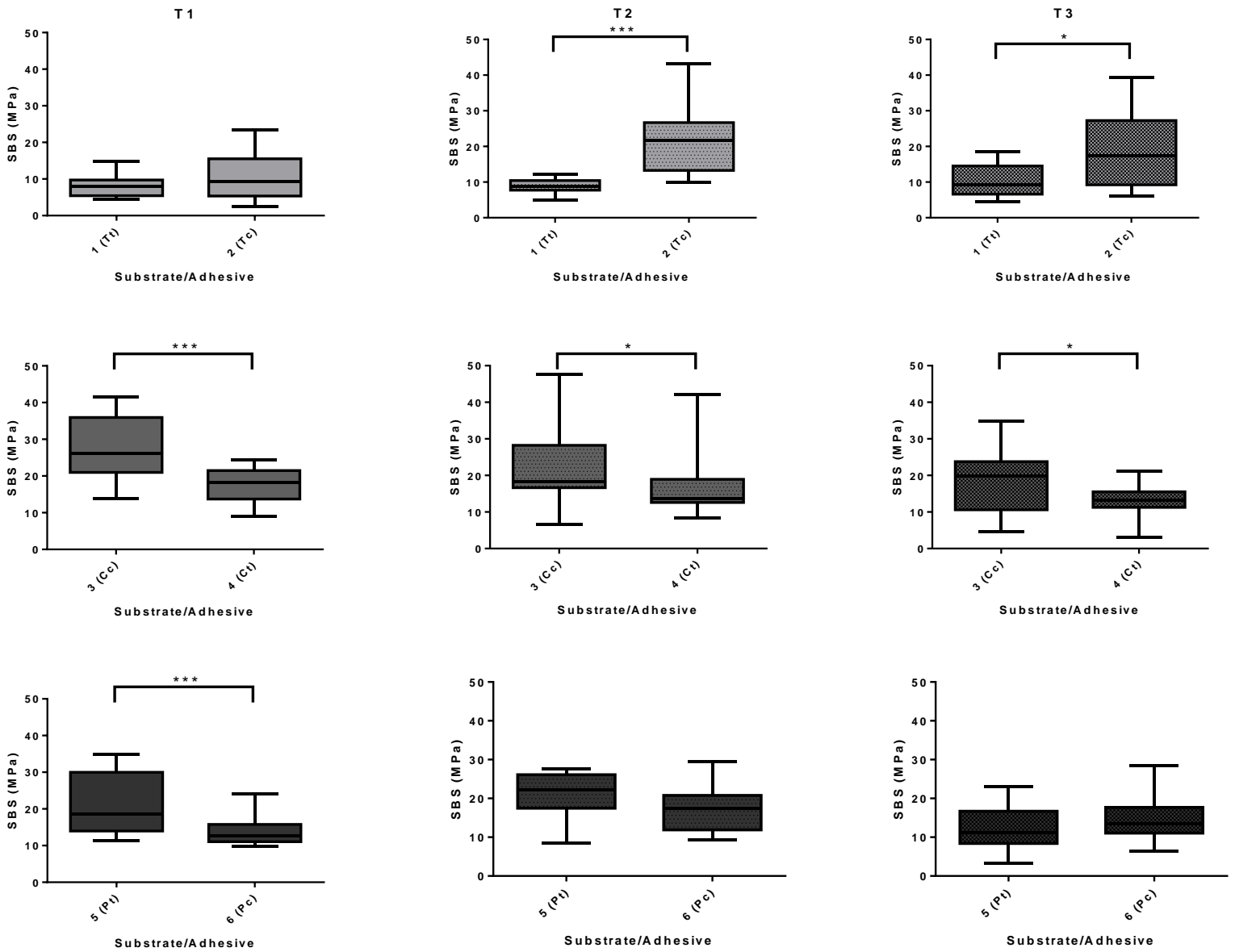
Statistical analysis was calculated using GraphPad Prism version 6.00 for Windows (GraphPad Software, La Jolla California USA, www.graphpad.com) software. Descriptive statistics and the D'Agostino & Pearson omnibus normality test were performed to determine which statistical tests were applicable to a subgroup. Statistical significance was established at a threshold of $p < 0.05$. Statistical assessment was performed on the SBS data obtained for each substrate category (T-Tooth, C-Composite, and P-Porcelain) at each time point (T1, T2, and T3) and within each group for the control (c) and test (t) (T_t, T_c, C_t, C_c, P_t, P_c). Median ARI scores and significance within each group were also determined.

Statistical significance was determined for subgroups displaying Gaussian properties using unpaired t tests and applying Welch's correction when applicable. Non-Gaussian subgroups were analysed using Mann-Whitney tests. Multiple subgroup comparisons were performed using analysis of variance (ANOVA) with Tukey's multiple comparisons test for Gaussian subgroups and Kruskal-Wallis' test with Dunn's multiple comparisons test for non-Gaussian subgroups.

RESULTS

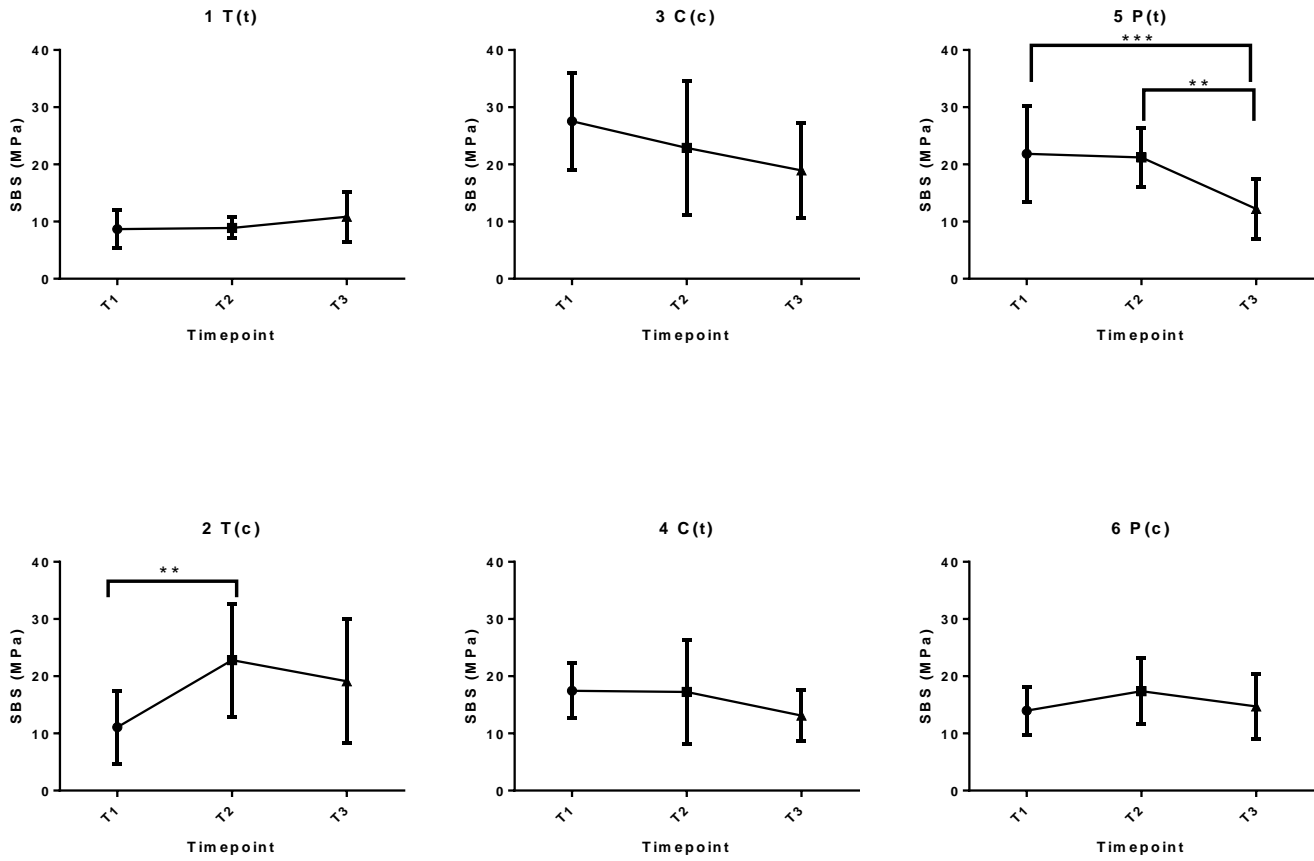
Data sets were normally distributed with the exception of T1: P_c and T2: C_t. Mean SBS obtained ranged from 8.69-22.88 MPa (Table 2). Significant differences were found in the tooth category at T2 and T3, in the composite category at all three time points, and in the porcelain category at T1 (Figure 1). Significant differences ($p < 0.05$) were found within the T_c group from T1 to T2 and within the P_t group from T1 to T3 and T2 to T3 (Figure 2). Table 3 shows the frequencies of each subgroup's ARI scores. 47.2% of the sample achieved an ARI score of 5 (100% adhesive remained on the button base) with the only significant median change being in T_c from T1 to T2 (Figure 3).

Figure 1 Quartile Shear Bond Strengths and Statistical Analysis



Note: T- Tooth, C- Composite, P- Porcelain, t- test, c-control (Asterisks denote significance: * p<0.05; ** p<0.02; *** p<0.01)

Figure 2 Within Group Statistical Analysis



Note: T- Tooth, C- Composite, P- Porcelain, t- test, c-control (Asterisks denote significance: * p<0.05; ** p<0.02; *** p<0.01)

Table 2: Shear Bond Strengths

	Group	Time Point	N	Mean (MPa)	Std. Dev. (MPa)	Min (MPa)	Max (MPa)	Coeff. Var. (%)
Tooth	1 T_t	T1	15	8.69	3.33	4.54	14.7	38.3
		T2	15	8.89	1.85	5.05	12.25	20.8
		T3	15	10.85	4.40	4.58	18.43	40.5
	2 T_c	T1	15	11.08	6.39	2.55	23.5	57.6
		T2	15	22.81	9.89	9.90	43.14	43.4
		T3	15	19.10	10.87	5.99	39.29	56.9
Composite	3 C_c	T1	14	27.44	8.47	13.91	41.6	30.8
		T2	15	22.88	11.68	6.54	47.66	51.0
		T3	14	18.96	8.36	4.58	34.94	44.1
	4 C_t	T1	15	17.45	4.83	9.06	24.43	27.7
		T2	15	17.26	9.04	8.49	42.20	52.4
		T3	15	18.96	8.36	4.58	34.94	44.1
Porcelain	5 P_t	T1	14	21.86	8.42	11.44	34.94	38.5
		T2	15	21.20	5.14	8.55	27.61	24.2
		T3	15	12.20	5.30	3.21	22.97	43.4
	6 P_c	T1	15	13.98	4.22	9.83	24.15	30.2
		T2	15	17.37	5.74	9.34	29.53	33.0
		T3	15	14.70	5.75	6.48	28.49	39.1

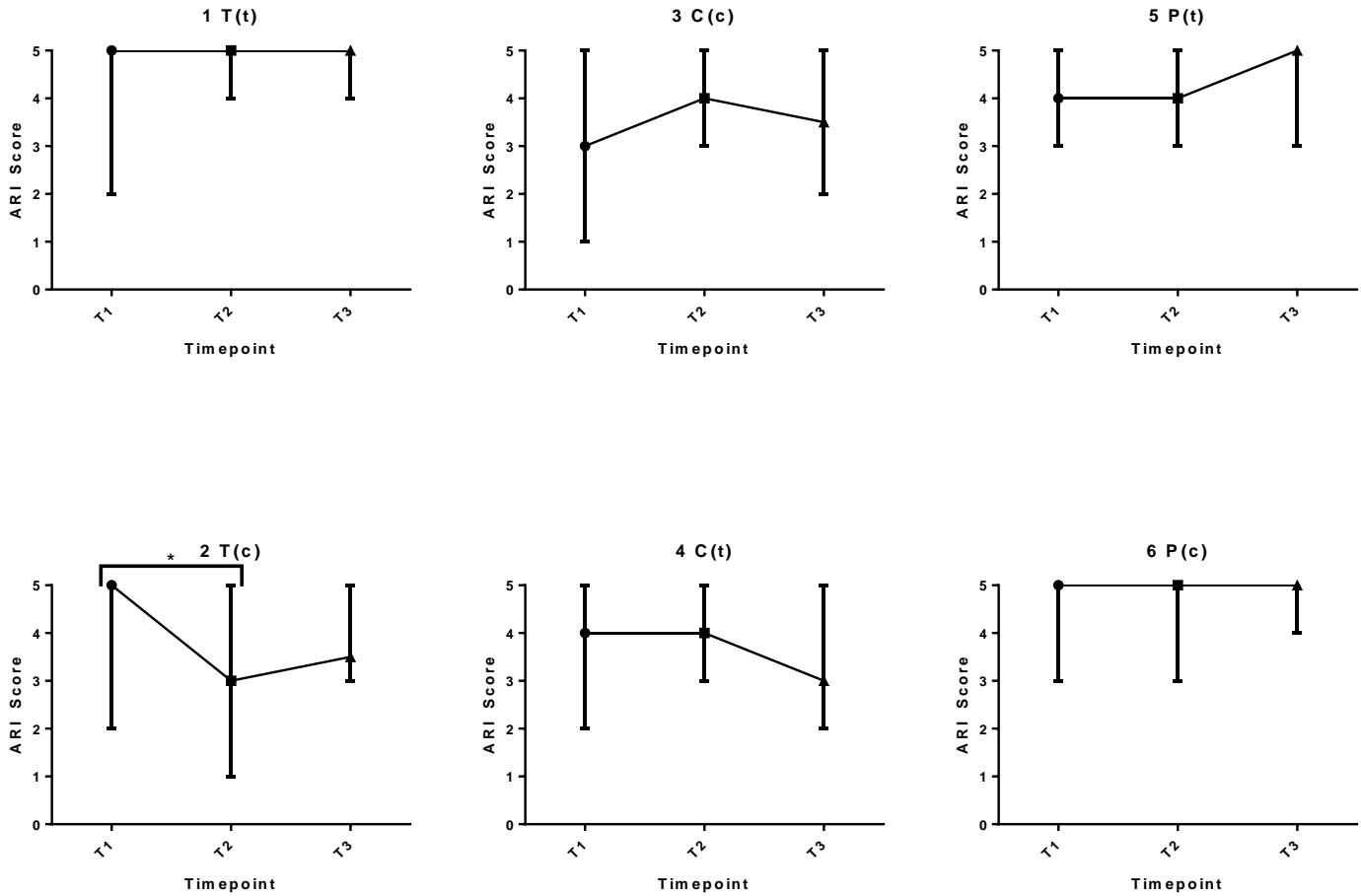
Note: T- Tooth, C- Composite, P- Porcelain, t- test, c-control

Table 3: Frequency Table of Adhesive Remnant Index Scores

	Group	Time point	ARI ¹ Score				
			1	2	3	4	5
Tooth	1 T _t	T1	0	1	0	1	8
		T2	0	0	0	1	9
		T3	0	0	0	3	7
	2 T _c	T1	0	1	0	2	7
		T2	1	2	3	2	2
		T3	0	0	5	1	4
Composite	3 C _c	T1	1	3	3	1	2
		T2	0	0	3	4	3
		T3	0	1	4	2	3
	4 C _t	T1	0	1	2	5	2
		T2	0	0	3	3	4
		T3	0	1	6	1	2
Porcelain	5 P _t	T1	0	0	2	5	3
		T2	0	0	4	2	4
		T3	0	0	2	1	7
	6 P _c	T1	0	0	2	2	6
		T2	0	0	3	1	6
		T3	0	0	0	4	6

¹ARI: Score 1 = 0% adhesive left on button base; Score 2 = <10% adhesive left on button base; Score 3 = 10-90% adhesive left on button base; Score 4 = >90% adhesive left on button base; Score 5 = 100% adhesive left on button base;

Figure 3: Kruskal-Wallis with Dunn's multiple comparisons of ARI scores



Note: T- Tooth, C- Composite, P- Porcelain, t- test, c-control

DISCUSSION

It is advantageous to find methods to save chair-time during orthodontic treatment. The introduction of self-adhering flowable resin composite has the potential to address this objective. According to Reynolds, bonding orthodontic attachments requires a minimum SBS of 6-8 MPa⁹. This has been

challenged by Wiltshire and Noble¹⁰ due to the successful clinical performance of glass ionomer cements *in vivo*, which show *in vitro* SBS testing outcomes in the 3-4 MPa range¹⁰⁻¹³.

Enamel

To date, only two other studies have investigated the potential use of SAFC for orthodontic use. The 24hrs mean and range SBS values obtained in our study with VF to enamel was 8.69 ± 3.33 MPa [4.54-14.70 MPa]. This was lower than the value obtained by Goracci et al⁷ after 30mins (10.13 ± 2.86 MPa) and higher than the 24hr SBS for VF (2.55 ± 0.77 MPa) reported by İşman et al⁶. Acid-etching with 37% phosphoric acid prior to using SAFC has been shown to increase SBS^{6,7}, however would be more time-consuming clinically and may defeat the original intention of using a SEP.

Thermocycling is known to reduce SBS¹⁴⁻¹⁶. This reduction has also been shown with SAFC⁷. When translated to the clinical scenario, thermocycling is quite rigorous and does not accurately portray the intra-oral thermal exposure the test adhesive would encounter^{16,17}. The present study stored samples at 37°C at 100% relative humidity, a more accurate representation of the thermal exposure experienced by the adhesive over the long term.

The SBS of VF to enamel is comparable to the results obtained with glass ionomer cements^{11,18}. Fricker¹³ reported no clinical significance in bonding failures between glass ionomer cements and composite resin adhesive over a 12 month period *in vivo*. Based on this finding, the results from the current study suggest that Vertise Flow is suitable for the bonding of metal orthodontic attachments to enamel.

Restorative Resin Composite

The present study showed significant differences in the initial SBS values between the two restorative resin composites. This may be attributed to the differences in composition of the two restorative resin composites. The difference in rate of decline of SBS between the two groups may be due to the initially higher SBS value of C_c. The SBS values for VF in the present study are in agreement with reported values for restorative resin composite bonding with TBxt^{19,20}. The SBS values to restorative composite resin attained *in vitro*, show that the SAFC Vertise Flow, has potential for orthodontic bonding to composite resin substrates.

Porcelain

In the present study, VF had statistically higher SBS to porcelain compared to control, TBxt to porcelain, at 24 hours. At 7 days, this difference no longer persisted and by 3 months, VF had a similar SBS to the control ($p > 0.05$). The surface preparation of the two groups were not the same (diamond roughening versus hydrofluoric acid etching) and may have contributed to the difference. Costa²¹ reported 24 hour SBS of 9.81 ± 1.1 MPa and 11.60 ± 1.1 MPa for TBxt to porcelain etched with 10% hydrofluoric acid for 20 and 60 seconds respectively. These results are lower than the result achieved in this study for TBxt to porcelain (13.98 ± 4.22 MPa [$9.83-24.15$ MPa]). This may be due to the different amounts of etching time. Bourke and Rock²² found significant differences in 24 hour SBS achieved with various surface preparations of porcelain and using a composite resin adhesive (Right On). In their study, they reported a SBS of 3.52 ± 0.24 MPa when the porcelain was prepared with only hydrofluoric acid for 3 minutes.

Silane treatment has been suggested as a way to improve bonding to porcelain^{21,22}. The reported range of 24 hour mean SBS values for silane treated, hydrofluoric acid etched porcelain with a composite resin adhesive is 10.29-18.69 MPa^{21,22}. This range is still lower, but is closer to, the 24hour SBS achieved in the present study with VF to diamond-roughened porcelain.

The reduction in SBS seen in the P_t group, from T2 to T3, is concerning as the rate of degradation was the greatest amongst all the groups tested. The cause for this drastic drop in SBS is unknown, but could be due to continued bond degradation as a result of watersorption equilibration. However, at this time point the SBS of VF was still in the order of magnitude of the control group (p>0.05). Further studies on the longer term SBS performance of VF to porcelain is advisable.

Potential Clinical Application and Performance

Although not directly quantified in this study, it is within reason to presume that the bonding method with VF would be faster than conventional bonding methods. The challenges with the product relate to its fluidity. In its current formulation, VF is more fluid than the resin composite adhesives commonly used for orthodontic attachment bonding, e.g. TBxt. When placing an attachment, a minimal amount of material is necessary. If too much adhesive is present on the attachment bonding surface when placed, the resin will spread and allow the metal base to be flooded by the excess material. This would make the removal of this 'flash' material more difficult. The fluidity also makes the attachment slide easily on the substrate surface. In this investigation, gravity was assisting securing the attachment in place. Clinically, this may not always be the case. VF in its room temperature consistency may pose a challenge at securing a bracket in its ideal position on posterior teeth. Downward flow when using flowable composites for orthodontic bonding has been previously described as a drawback²³.

The interface between the substrate and VF was most often the site of debond failure. The outcome in this study was advantageous as it suggests that less damage occurs to substrate on debond. This also reduces iatrogenic damage to the substrate upon the removal of residual adhesive post-debond; another time-saving experience.

Based on the results of this *in vitro* study, a clinical trial utilizing VF to attach fixed appliances or fixed retainers *in vivo* is suggested.

CONCLUSIONS

From this study the following conclusions can be drawn:

- Metal orthodontic attachments were successfully bonded *in vitro* with VF to enamel, restorative resin composites (Herculite Ultra and Filtek Supreme Ultra) and porcelain, after storage in 37°C distilled water for a period up to 3 months, using minimal surface preparation.
- The results of this *in vitro* study show potential for this self-adhering flowable resin to be translated to the clinical situation for bonding of metal orthodontic attachments to human enamel, restorative resin composite and porcelain.

- VF may reduce bonding time and chair time in the orthodontics as well as cause less surface substrate destruction, if translated to the clinical situation.

REFERENCES

1. Buonocore M. A simple method for increasing the adhesion of acrylic filling material to enamel surfaces. *Journal of dental research*. 1955;34(6):849–853.
2. Rossouw PE. A Historical Overview of the Development of the Acid-Etch Bonding System in Orthodontics. *Seminars in Orthodontics*. 2010;16(1):2–23.
3. Powers JM, Kim HB, Turner DS. Orthodontic adhesives and bond strength testing. *Seminars in orthodontics*. 1997;3(3):147–56.
4. Paschos E, Westphal J, Ilie N, Christine K, Hickel R, Rudzki-janson I. Artificial Saliva Contamination Effects on Bond Strength of Self-etching Primers. 2008;78(4):716–721.
5. Grubisa HS., Heo G, Raboud D, Glover KE, Major PW. An evaluation and comparison of orthodontic bracket bond strengths achieved with self-etching primer. *American Journal of Orthodontics and Dentofacial Orthopedics*. 2004;126(2):213–219.
6. İşman E, Karaarslan ES, Oksayan R, et al. Inadequate shear bond strengths of self-etch, self-adhesive systems for secure orthodontic bonding. *Dental Materials Journal*. 2012;31(6):947–953.
7. Goracci C, Margvelashvili M, Giovannetti A, Vichi A, Ferrari M. Shear bond strength of orthodontic brackets bonded with a new self-adhering flowable resin composite. *Clinical oral investigations*. 2013;17(2):609–17.
8. Bishara SE, Vonwald L, Olsen ME. Effect of time on the shear bond strength of glass ionomer and. *American Journal of Orthodontics and Dentofacial Orthopedics*. 1999;116:616–620.
9. Reynolds I. A review of direct orthodontic bonding. *British journal of orthodontics*1. 1975;2:171–178.

10. Wiltshire WA, Noble J. Clinical and Laboratory Perspectives of Improved Orthodontic Bonding to Normal, Hypoplastic, and Fluorosed Enamel. *Seminars in Orthodontics*. 2010;16(1):55–65.
11. Wiltshire WA. Shear bond strengths of a glass ionomer for direct bonding in orthodontics. *American journal of orthodontics and dentofacial orthopedics*. 1994;106(2):127–30.
12. Fricker JP, Dip G, Adult E. for the direct bonding of orthodontic brackets in vivo. 1998:384–386.
13. Fricker JP. A 12-month clinical evaluation of a light-activated glass polyalkenoate (ionomer) cement for the direct bonding of orthodontic brackets. *American journal of orthodontics and dentofacial orthopedics*. 1994;105(5):502–5.
14. Elekdag-Turk S, Turk T, Isci D, Ozkalayci N. Thermocycling effects on shear bond strength of a self-etching primer. *The Angle orthodontist*. 2008;78(2):351–6.
15. Costa AR, Correr AB, Puppini-Rontani RM, et al. Effects of thermocycling and light source on the bond strength of metallic brackets to bovine teeth. *Brazilian dental journal*. 2011;22(6):486–9.
16. Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *Journal of dentistry*. 1999;27(2):89–99.
17. Bishara SE, Ajlouni R, Laffoon JF. Effect of thermocycling on the shear bond strength of a cyanoacrylate orthodontic adhesive. *American journal of orthodontics and dentofacial orthopedics : official publication of the American Association of Orthodontists, its constituent societies, and the American Board of Orthodontics*. 2003;123(1):21–4.
18. Banerjee R, Banerjee S. A comparative evaluation of the shear bond strength of five different orthodontic bonding agents polymerized using halogen and light-emitting diode curing lights: An *in vitro* investigation. *Indian Journal of Dental Research*. 2011;22(5):731–732.
19. Lai PY, Woods MG, Tyas MJ. Bond strengths of orthodontic brackets to restorative resin composite surfaces. *Australian Orthodontic Journal*. 1999;15(4):235–245.
20. Bayram M, Yesilyurt C, Kusgöz A, Ulker M, Nur M. Shear bond strength of orthodontic brackets to aged resin composite surfaces: effect of surface conditioning. *European journal of orthodontics*. 2011;33(2):174–9.
21. Costa AR, Correr AB, Puppini-Rontani RM, et al. Effect of bonding material, etching time and silane on the bond strength of metallic orthodontic brackets to ceramic. *Brazilian dental journal*. 2012;23(3):223–7.

22. Bourke BM, Rock WP. Factors affecting the shear bond strength of orthodontic brackets to porcelain. *British journal of orthodontics*. 1999;26(4):285–90.
23. Turgut MD, Attar N, Korkmaz Y, Gokcelik A. Comparison of shear bond strengths of orthodontic brackets bonded with flowable composites. *Dental Materials Journal*. 2011;30(1):66–71.

12. RAW DATA

T1: IMMEDIATE

	1	2	3	4	5	6
	VF Tooth	VF K comp	VF 3M comp	VF porc	TB Tooth	TB porc
1	13.77	35.78	24.43	27.62	8.34	13.01
2	9.74	13.91	17.32	16.43	5.27	24.15
3	6.72	41.6	11.25	27.97	17.8	11.54
4	7.25	36.37	21.69	30.01	10.85	11.24
5	4.54	29.98	9.92	17.81	23.5	14.54
6	7.65	28.36	20.13	33.53	5.36	10.77
7	14.87	39.14	9.06	34.94	15.54	9.83
8	5.45	22.82	18.16	17.68	8.27	15.02
9	5.44	24.07	18.71	0	22.11	22.18
10	14.17	17.68	13.72	11.44	12.25	11.06
11	9.09	24.11	21.46	29.95	6.49	12.71
12	8.05	21.62	19.99	19.32	9.29	10.43
13	9.23	19.08	16.3	13.06	2.55	15.81
14	9.32	0	15.49	11.94	13.95	15.75
15	5.03	31.11	24.06	14.27	4.65	11.62

T2: 7 DAYS

	1	2	3	4	5	6
	VF Tooth	VF K comp	VF 3M comp	VF porc	TB Tooth	TB porc
1	8.84	16.64	29.16	17.49	19.7	22.29

2	6.31	47.66	27.87	16.23	23.75	11.88
3	7.64	17.33	12.38	22.24	43.14	11.57
4	12.25	35.42	12.6	8.55	42.49	25.94
5	5.05	27.45	13.43	23.76	21.64	17
6	11.18	43.37	12.7	23.4	11.51	19.67
7	9.65	15.56	9.3	26.63	12.57	9.34
8	8.07	28.22	14.97	26.11	26.67	17.81
9	8.52	17.05	8.49	27.07	27.37	29.53
10	9.36	20.44	13.4	18.78	9.9	20.79
11	10.47	9	42.2	22.3	21.24	13.07
12	10.43	22.22	18.92	20.28	26.3	10.79
13	8.35	18.24	15.03	16.6	13.26	18.63
14	7.72	18.06	14.9	21	18.99	14.81
15	9.44	6.54	13.61	27.61	23.6	17.44

T3: 3 MONTHS

	1	2	3	4	5	6
	VF Tooth	VF K comp	VF 3M comp	VF porc	TB Tooth	TB porc
1	4.58	34.94	6.55	8.94	11.15	13.53
2	14.53	18.41	2.96	22.97	27.27	11.46
3	16.1	19.7	11.27	19.05	27.1	17.7
4	8.07	8.69	18.91	18.48	7.52	8.27
5	6.22	21.4	16.4	8.38	17.47	16.45
6	13.02	24.91	14.18	10.98	23.47	6.48

7	6.6	21.37	15.52	15.29	9.02	28.49
8	7.96	20.21	12.74	9.93	36.71	16.32
9	8.7	9.48	13.18	16.7	5.99	22.87
10	9.33	10.96	11.04	6.86	17.69	11.85
11	13.25	0	15.5	11.69	39.29	19.62
12	12.8	17.6	21.17	3.21	11.23	11.83
13	6.34	4.58	11.88	11.38	13.41	11.05
14	18.43	29.76	11.76	7.99	9.22	13.93
15	16.82	23.37	13.71	11.21	30.01	10.68

Adhesive Remnant Index

time point 1

	VFT-1	3MT-5	VFK-2	VF3M-3	VFP-4	3MP-6
1	2	4	3	4	4	5
2	4	5	2	3	5	3
3	5	2	1	2	4	5
4	5	4	2	4	3	5
5	5	5	2	5	4	5
6	5	5	5	4	4	5
7	5	5	3	4	5	3
8	5	5	3	3	4	4
9	5	5	4	4	3	5
10	5	5	5	5	5	4

time point 2

	1	2	3	4	5	6
1	5	1	3	3	4	5
2	4	2	4	3	3	4
3	5	3	5	3	5	3
4	5	4	4	5	3	3
5	5	5	5	4	5	3
6	5	3	4	5	5	5

7	5	5	5	4	3	5
8	5	3	3	4	5	5
9	5	2	3	5	3	5
10	5	4	4	5	4	5

time point 3

	1	2	3	4	5	6
1	4	3	5	3	3	5
2	5	3	3	4	5	5
3	5	3	2	3	3	4
4	4	5	4	3	4	5
5	4	4	5	2	5	5
6	5	3	5	3	5	4
7	5	5	3	3	5	5
8	5	3	3	3	5	5
9	5	5	4	5	5	4
10	5	5	3	5	5	4

Group	Timepoint	1	2	3	4	5
1 (T _t)	T1	0	1	0	1	8
	T2	0	0	0	1	9
	T3	0	0	0	3	7
2 (T _c)	T1	0	1	0	2	7
	T2	1	2	3	2	2
	T3	0	0	5	1	4
3 (C _c)	T1	1	3	3	1	2
	T2	0	0	3	4	3
	T3	0	1	4	2	3
4 (C _t)	T1	0	1	2	5	2
	T2	0	0	3	3	4
	T3	0	1	6	1	2
5 (P _t)	T1	0	0	2	5	3
	T2	0	0	4	2	4
	T3	0	0	2	1	7
6 (P _c)	T1	0	0	2	2	6
	T2	0	0	3	1	6
	T3	0	0	0	4	6