# AN ANALYSIS OF INTERNAL VOIDS OF ORTHODONTIC ADHESIVES VIA MICRO-CT

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## ABSTRACT

**Objectives:** While bracket debonding has been explored through shear bond-strength tests with debonding linked to the type of material used, the technique of adhesive application, and contamination, the contribution of internal voids in orthodontic adhesives is unknown. Voids may result in fracture or bond-failure, either within the adhesive or at the tooth-adhesive-bracket interfaces. The aim of this thesis is to quantify the internal volumetric voids and bonding strength of three generations of bracket adhesives.

**Methods:** Extracted third molars were bonded with three groups of orthodontic brackets including conventionally-pasted (CP), pre-coated (PC), or pre-coated flash-free (FF) (n=5 per group). The three-dimensional internal structure of the adhesive was evaluated with Micro Computed Tomography (micro-CT) using the Skyscan micro-CT (maximum resolution of 5 microns). Data from the micro-CT were analyzed with SkyScan software to perform 3D reconstructions, image processing, and qualitative and quantitative analysis of the adhesive's structure. The amount of void was determined by measuring the percentage of voids at the bonded interfaces ($V_{int}$) and within the adhesive ($V_{bulk}$). The total amount of void was also calculated ($V_{tot}= V_{int}+ V_{bulk}$). Differences in void between the groups were assessed using one-way ANOVA with post-hoc Tukey tests ($\alpha=0.05$). The bonding strength of the three adhesives systems was evaluated via shear bond strength tests.

**Results:** Our void quantification results showed that FF brackets had a statistically higher ($p<0.05$) amount of void per volume ($V_{tot}=26.99\pm7.26\%$) compared to CP ($13.75\pm4.48\%$)

and PC (9.65±4.56%) for all evaluated locations. To determine the distribution of voids along the adhesive of bonded samples, we compared the quantities of voids at interfaces and within the bulk. The amount of void at the bonded interfaces ($V_{int}$) was statistically higher ($p<0.05$) for FF compared to CP and PC. On the contrary, the FF group had a statistically significant lower percentage volume of void within the bulk of the adhesive compared to CP and PC. There were no significant differences of the void between CP and PC groups ($p>0.05$) for any the analyzed locations ($V_{int}$, $V_{bulk}$). Our results indicate the majority of voids were found at bonded interfaces ($V_{int}$) compared to within the bulk ($V_{bulk}$) for all three groups, with statistically significant ($p<0.05$) differences for CP and FF. Our bonding strength evaluation revealed the pre-coated group (PC) to have on average the highest bond strength compared to conventionally-pasted (CP) and pre-coated flash-free (FF) groups.

**Conclusions:** The overall amount of void in the pre-coated flash-free adhesive brackets is significantly higher compared to conventionally-pasted and pre-coated groups. The majority of the void was identified to be located at the bonded interfaces (tooth/adhesive and bracket/adhesive) rather than within the bulk of the adhesive for all groups. Our bonding strength results indicate the pre-coated brackets to have the highest bond strength compared to pre-coated flash-free and conventionally-pasted. Our preliminary set of results indicate an inverse relationship between bonding strength and amount of void of brackets adhesives, with the pre-coated exhibiting the highest bond strength and least void. However, due to a limited sample size additional data are needed to validate these conclusions and find solid relationships between adhesive voids and bonding strength.

## ACKNOWLEDGEMENTS

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# CHAPTER 1

# INTRODUCTION

Orthodontic brackets are an integral part in the treatment of dental malocclusion. In combination with wires, brackets provide a means by which a practitioner can move teeth and align the dentition. In the first generation of brackets, adhesive needed to be applied separately onto the bracket. To eliminate a step in the bonding procedure and reduce the time of patients in the chair, brackets were designed to be pre-coated or pre-pasted with adhesive (Sfondrini 2002). Recently, advancements in the orthodontic field include brackets that are considered "flash-free," i.e. there is no need to remove excess material, eliminating yet another step during the application of brackets to a tooth (Foresch 2016). While these different bracket-adhesive technologies are all currently in use, there may be a lack of understanding of how these fundamental differences affect bonding properties.

To understand and quantify the bonding strength of brackets, different testing methods have been proposed including shear bond, tensile, and torsion (Sunilkumar 2013). Debonding of brackets is attributed to many causes, including the type of adhesive material used, the amount and size of filler of the adhesive, curing parameters, the enamel surface preparation, and microleakage (Shahabi 2014, Boruziniat 2015, Sharma 2014). However, little attention has been given to the potential influence of adhesive voids on bonding strength. Voids of an adhesive at a bonded interface can result in decreased bonding surface area and an increase in stress concentration that leads to premature failure. Additionally, voids within the bulk of the bonding material may allow for fracture within the material

itself due to a rise in stress. As a result, debonding of the adhesive at the bonded interfaces (tooth-adhesive or bracket-adhesive) or adhesive bulk fracture can occur. There is no agreement and significant variation of bonding strength based on the different types of brackets and adhesives, etching process, and even force during bracket placement (Finnema 2010). There has not been a study investigating interface voids of a bonded bracket and its potential relation to bond strength. Findings from this investigation may suggest a possible association between the type of adhesive-bracket and bonding failures during orthodontic treatment.

This study seeks to determine the voids present in the adhesives of conventionally-pasted, pre-coated, and, newly, pre-coated "flash-free" brackets via micro-computed tomography of the bonded interfaces and its possible impact on bonding strength.

# CHAPTER 2

# REVIEW OF THE LITERATIVE

## 2.1 History of Fixed Orthodontics

The design of fixed oral appliances has changed much since the early days of modern orthodontics. In order to move and align teeth, early fixed appliances were brackets welded to gold or stainless-steel circumferential bands (Gange 2015). These bands attached to teeth by cement and were introduced in 1871 by Magil (Sanders 1997). This system of orthodontic bands, however, did not come without challenges. In addition to the possibility of gingival trauma during band fitting, decalcification could occur under these circumferential bands during treatment if they were not sealed properly (Gange 2015).

Since the 1960s, bonding the bracket directly to tooth enamel has become the prevailing way to apply orthodontic forces/moments to the tooth. Dr. George Newman from New Jersey and Professor Fujio Miura from Tokyo Medical and Dental University in Japan pioneered the bonding of orthodontic brackets to enamel, allowing for orthodontic treatment without the need for bands (Gange 2015, Green 2014). Since then, direct bonding of orthodontic brackets, especially on anterior teeth, has offer improved esthetics, easier placement, increased patient comfort, avoidance of interproximal separators needed for banding and the issue of interproximal space after debonding, and decreased decalcification (Sanders 1997).

## 2.2 Pre-coated Orthodontic Brackets

Beginning in the 1960s, conventionally-pasted orthodontic brackets became the principal way to attach a bracket to a tooth (Figure 1). In this system, adhesive is placed on the bracket followed by bonding the bracket on the tooth. To eliminate a step in the bonding process of brackets, companies developed appliances with the cement or adhesive already applied in the bracket. Pre-coated orthodontic brackets were created in 1992 by 3M Unitek, where the adhesive is already pasted onto the back of each bracket (Sfondrini 2002) (Figure 2). The claimed advantages of pre-coated brackets are consistent quality and quantity of adhesive, reduced waste during bonding, easier clean-up following bonding, and improved asepsis (Foresch 2016). Although conventional and pre-coated brackets attain the same goal of bonding onto a tooth, one of the main differences is that the pre-coated adhesive by 3M Unitek contains more filler than the conventionally-pasted Transbond$^{TM}$ XT adhesive – 80% compared to 77% respectively (Hassan 2010). By increasing the filler, it is possible to increase the viscosity of the adhesive and allow better adhesion between the bracket and the tooth during initial bonding and bracket positioning (Bishara 1997). The other adhesives' ingredients are also the same but in different percentages – with the pre-coated brackets containing 12% BisGMA and 8% Bis EMA while the conventional Transbond$^{TM}$ XT contains 14% BisGMA and 9% Bis EMA.


**Figure 1:** Conventionally-pasted Orthodontic Bracket, 3M Unitek


**Figure 2:** Pre-coated Orthodontic Bracket, 3M Unitek

Clinical performance of pre-coated brackets, when compared to traditionally pasted brackets, have been shown to perform within acceptable limits. Studies have shown both that conventional and pre-coated brackets are equally effective in maintaining orthodontic brackets on a tooth (Kula 2002). However, a study illustrated that pre-coated brackets that

used the same adhesive had a significantly lower shear bond strength than those on conventionally-pasted (uncoated) brackets (Bishara 1997). While this may be an area of contention and disagreement, evaluating factors related to the differences between these adhesive systems may clarify the differences in results found in these bonding studies.

### 2.3 Pre-coated "Flash-free" Brackets

In traditional bracket bonding, flash, i.e. excess adhesive, around a bracket may cause irritation to the gingiva and increase plaque accumulation that could lead to enamel decalcification. The pre-coated "flash-free" orthodontic brackets developed by 3M Unitek in 2014 added another step forward in reduction of the time needed to bond orthodontic brackets (Figure 3). In prior bracket/adhesive bonding, excess bonding material had to be removed prior to curing, increasing the time needed to attach an orthodontic bracket to a tooth. In addition to being pre-coated with adhesive like the pre-coated system that was previously introduced, the "flash-free" system was created to also eliminate the need of excess adhesive removal. A study in the Angle Orthodontist concluded that the flash-free adhesive system, by eliminating the need for clean-up of excess adhesive, "significantly reduced" the time needed for orthodontic bracket bonding by 64.25% in the lower jaw and 42.3% in the upper jaw when compared to the bonding process of conventional brackets (Foresch 2016). In addition to time-savings, however, these new brackets also must be hygienic and held at a minimum to the same standard as conventional orthodontic brackets. According to 3M, however, this not an issue with their flash-free brackets. A non-woven, polypropylene fiber mat is placed on the base of the bracket and socked with a low-viscosity resin. This mat is meant to be slightly compressible when being pressed to the

tooth, but also holds back excess adhesive that is squeezed out during bracket application with the claim that the morphology and seal of the excess flash-free resin is supposed to inhibit plaque accumulation and microleakage (Foersch 2016).


**Figure 3:** Pre-coated Flash-free Orthodontic Bracket, 3M Unitek

Despite the laudable promises of flash-free brackets, this technology needs to be thoroughly evaluated to ensure that, despite the reduced bonding time, undesirable clinical consequences are avoided. A recent study looked at this bracket system and discussed that the flash-free adhesives reduced time needed for bonding and did not require clean-up of excess adhesive, but called for further clinical studies on this technology, particularly the marginal integrity (Foresch 2016). A comparison of marginal integrity found that the brackets bonded with the flash-free adhesive had a smooth, non-textured surface that conformed to the enamel surface while the conventional adhesive had a more “ruffled” surface with greater irregularity of transition from adhesive to enamel surface (Grunheid 2015). In addition, their microleakage testing showed a non-statistically significant difference between the two conventional and flash-free adhesives.

This study will refer to this bracket technology as “flash-free” for brevity, but the reader should note that all flash-free brackets are also pre-coated/pre-pasted with adhesive.

## 2.4 Ceramic Brackets

To improve patients’ aesthetics during treatment, there have been advances in the materials used to fabricate orthodontic brackets. In contrast to conventional metal brackets, manufacturers have looked for materials where a bracket can have a color closer to a patient’s teeth. The first of these brackets were plastic. However, they were prone to staining and slot distortion (Theodorakopoulou 2004). The first ceramic brackets – a combination of monocrystalline sapphire and polycrystalline ceramic – became available in the 1980s, providing a viable esthetic alternative (Theodorakopoulou 2004). Ceramic brackets that are chemically bonded to the teeth have shown to have a stronger bond strength than conventional metal brackets (Theodorakopoulou 2004). Ceramic brackets are stiffer and more rigid compared to metallic ones, which can lead to fracture of the bracket itself when debonding. A study showed that metal brackets deform 20% under mechanical stress before fracturing, while ceramic brackets deform less than 1% before fracturing under the same load conditions (Bishara & Trulove 1990). In addition, enamel damage during the debonding of ceramic brackets continues to be a matter of concern for the clinician (Bishara 2008). Despite the drawbacks of ceramic brackets, they are likely to continue growing as an esthetic choice by patients.

## 2.5 Bracket Debonding & Bond Strength

Orthodontic bracket debonding is a prevalent issue in the field of orthodontics, with failure of the total bonded brackets ranging from as low as 6% to nearly 18% (Millett 1998, Bherwani 2008). Loss of brackets during orthodontic care can result in increased treatment time, operator time, material cost, and patient discomfort (Wiltshire 2010). Success rates of bracket bonding are related to the bonding agent, technique used, etching time and concentration, and orthodontic bracket base (Bherwani 2008, Vijayakumar 2014). The most common locations of potential bracket debonding include the interfaces between the bracket and the adhesive and between the enamel and adhesive. However, failure can also occur at the bulk of the resin (Wang 2004).

Bond failure is different between metal and ceramic brackets. Odegaard and Segner found that while metal brackets had bond failure more often at the bracket-adhesive interface, ceramic bracket bond failure was more prevalent at the enamel-adhesive interface (Odegaard & Segner 1988). This difference in bond failure location could have potential implications for the importance of the voids distribution within the resin adhesive, enamel fracture during debonding, and for the maximum force required for bracket failure or debonding.

A current challenge for material scientists is to create a bond that is strong enough to retain the bracket during treatment but can allow debonding at the end of treatment without damage to the enamel surface (Theodorakopoulou 2004). A systematic review and meta-analysis that included results from 24 studies, including both metal and ceramic brackets, reported that bracket shear bond strength values ranged from 3.5 to 27.8 MPa (Finnema 2010). In addition, they stated that while *in-vitro* bond testing is a valuable tool

for initial screening and selection of materials, they have not been successful in predicting the bond strength under *in-vivo* conditions.

A systematic review by Finnema *et al*. summarized that there is "no overview on bracket bond strength from which general conclusions can be drawn. Because of the lack of standardization, the growing number of *in-vitro* studies being published can only be evaluated individually" (Finnema 2010). Due to all of these factors, standardization becomes difficult and individual studies may need to thoroughly investigate all of these variables. Bonding conditions of orthodontic brackets to teeth have been used in many different studies. The surface used (human vs. animal tooth), *in-vitro* or *in-vivo*, and pretreatment of the enamel to be studied may all have an effect on bond strength based on the differences in study design (Finnema 2010). Moreover, testing of bond strength may be evaluating different values – i.e. tensile, torsion or shear strength testing. The addition of stress concentrations by defects or voids may lead to premature failure. No studies have analyzed a bonded bracket for defects and in relation to debonding (Finnema 2010).

## 2.6 Internal Void of Adhesives

Voids in dental resins are created when air or moisture is trapped with the material, which can occur either during manufacturing or during material placement into the cavity during dental restorations (Nazari 2013, Purk 2007). Composite resins, in addition to being used for composite dental restorations, are also the most popular materials for orthodontic adhesive (Kula 2002). Voids affect the mechanical properties of a composite used for dental restorations. They can lower the fatigue resistance and contribute to failure by lowering the load-bearing capacity of the material in the oral environment (Nazari 2013).

Voids within the resin can also inhibit polymerization and weaken the bond strength as was seen in the earlier generation of self-polymerizing or chemically-cured adhesives (Majid 2016, Heravi 2013). A study of class II composite restorations associated greater bond strength with fewer voids, with voids more strongly predicting bond strength than location or condition of the restoration (Purk 2007). Studies have evaluated the voids of bulk filled resins in use for restorative dentistry, but no research has been done to look at the voids of bonded orthodontic adhesives. There is a need to measure the quantity and distribution (e.g. location) of voids formed after bonding brackets resin adhesives and find a possible association with bonding strength.

## 2.7 Micro-computed Tomography (micro-CT)

An experimental tool to quantify the location and size of the voids of materials is with micro-computed tomography (micro-CT) (Nazari 2013). Micro-CT is a technology with the ability to provide 3D structural analysis that was could not be previously accomplished via 2D imaging methods (Cosmin 2013). Micro-CT works by using X-rays to create high resolution cross-sectional images of a few microns in size (Cosmin 2013). One main advantage of micro-CT is the capability to measure voids within bonded materials. Orthodontic brackets use resins as adhesives or bonding agents. Micro-CT allows for an evaluation of the volume and location of voids without debonding the orthodontic bracket from the tooth. Utilizing micro-CT and analysis software, the data can be reconstructed in 3-dimensions, the image can be processed, and the samples of interest can be analyzed to view these voids. Micro-CT evaluation can provide the internal structure

of orthodontic adhesives bonded to a tooth, which later can be subjected to bonding test and highlight potential correlation.

# CHAPTER 3

# AIMS OF THE INVESTIGATION

The aim of this study is to determine the internal 3D structure of bracket adhesives bonded to a tooth and find potential relationships with the voids and bonding strength. This study may provide insight into other studies related to bracket bond failure. Specific aims include:

- To determine void quantity between different technologies of bracket-adhesive combinations including conventionally-pasted, pre-coated, and flash-free brackets
- To identify the location and distribution of these voids at the bonded interfaces versus within the bulk of the adhesive
- To evaluate the bonding strength of different bracket technologies and relate with the voids

# SIGNIFICANCE

There has been no evidence evaluating the internal void distribution of different bracket-adhesive technologies (i.e. conventional, pre-coated, and flash-free), and its potential relation to bonding strength. The information gathered in this study may provide orthodontists with a clinically significant rationale for potential bracket debonding due to voids that affect the tooth-adhesive-bracket interface. In addition, this research may determine that the bracket-adhesive combinations have an impact on the force a bracket can withstand before debonding and, therefore, help the manufacturer improve its product.

# CHAPTER 4

# MATERIALS AND METHODS

## 4.1 Material Selection

This study selected three bracket/adhesive technologies with different qualities including conventional (CC), pre-coated (PC) and flash-free (FF). The three different dental bracket-adhesive groups from 3M Unitek (St. Paul, MN) were bonded to teeth. The brackets either needed adhesive added (conventionally-pasted), had adhesive already on the bracket where the excess adhesive needed to be removed after placing the bracket (pre-coated), or had adhesive on the bracket where no excess needed to be removed after placement (flash-free). Extracted maxillary and mandibular third molars were utilized for bonding the orthodontic brackets. Of these, the most uniform and flat enamel surface available was selected. The enamel surfaces had to be free of defects, cracks, or other significant surface anomalies in order to be included for bonding. Third molars without a uniform or flat enamel surface were not considered for bonding.

## 4.2 Sample Preparation

First, extracted teeth were collected and stored in a 1:10 solution of diluted bleach in a securely sealed, plastic specimen container clearly labeled "Biohazard." The teeth were sectioned by a highspeed handpiece with a 556 bur under water irrigation to remove the root and create a flat surface on the bottom, back, and sides. Next, the tooth was irrigated with an air-water spray for ten seconds to remove any debris created from the sectioning.

The sectioned teeth meeting inclusion requirements were replaced in the storage solution at room temperature until the sample could be prepared for bonding.

The flat surface selected to be utilized for bonding was cleaned and prepared for bonding with a rubber disposable prophy angle (Henry Schein, Melville, NY) and Nada™ pumice paste (Preventech, Indian Trial, NC) for ten seconds with continuous pressure (Figure 4a). Next, the sample was rinsed by an air-water syringe for five seconds (Figure 4b), then air dried for five seconds (Figure 4c). The exposed enamel surface was treated with Transbond™ Plus Self Etching Primer (3M Unitek, St. Paul, MN) for five seconds (Figure 4d) followed by light air drying for two seconds as recommended by the instructions (Figure 4e). The protocol for the self-etching primer and subsequent air drying follows manufacturer instructions provided by 3M. Up to this step, each tooth sample in every group was prepared in a uniform fashion.

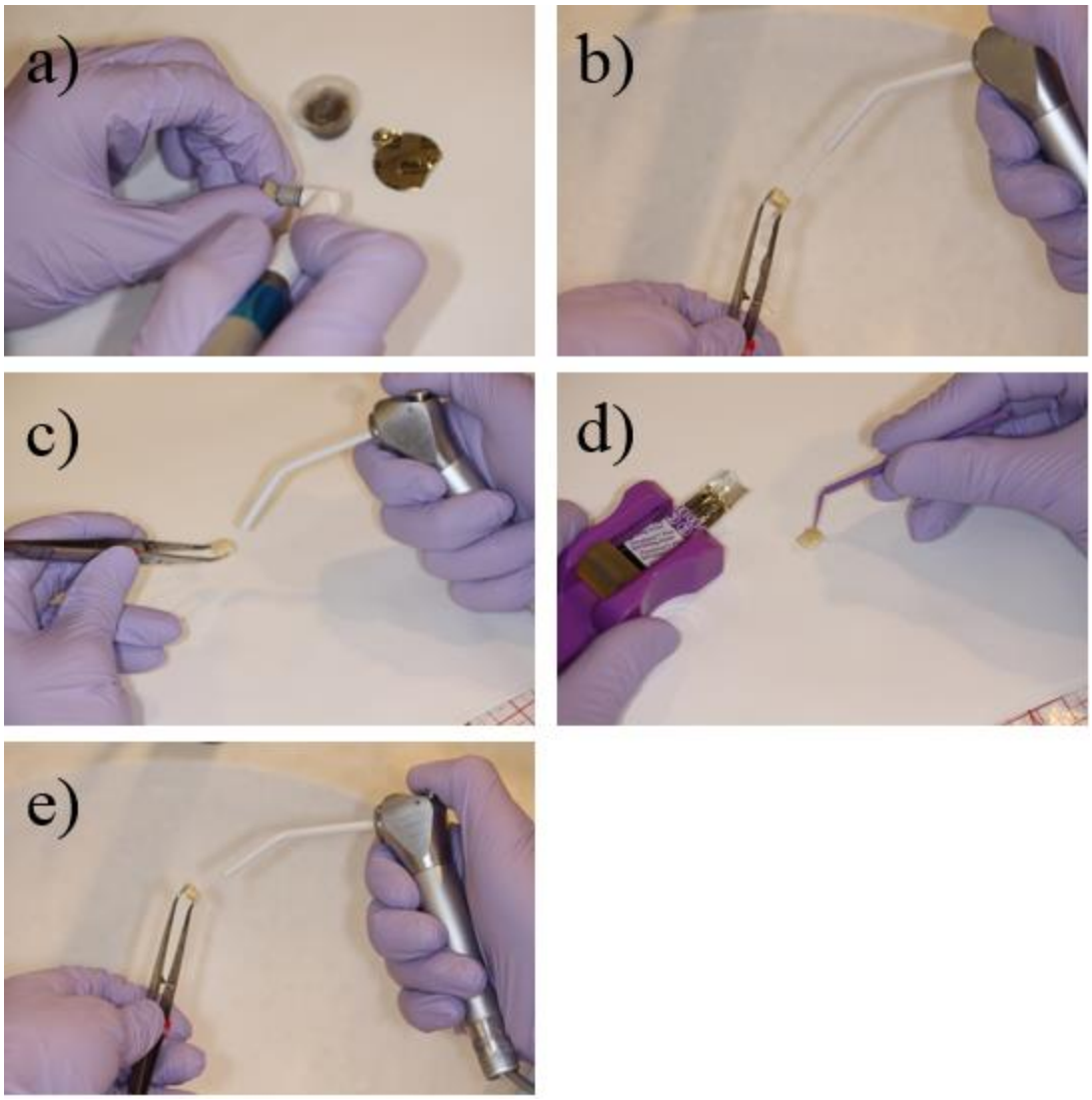


**Figure 4:** Preparation of sample before bracket bonding a) 10 second clean with pumice on rubber wheel, b) 5 second air/water irrigation, c) 5 second air dry, d) 5 second self-etch primer application, e) 2 second light air dry

Maxillary right central orthodontic brackets, due to their size, shape, and relatively flat bonding surfaces, either with conventionally-pasted (CP), pre-coated (PC), or flash-free (FF) cement were utilized for bonding. The brackets without pre-loaded adhesive (CP) received a layer of Transbond$^{TM}$ XT adhesive paste (3M Unitek, St. Paul, MN) filling the entirety of each bracket pad prior to application of the bracket on the tooth. The PC and FF

groups brackets come in sealed, opaque packaging from the manufacturer with the respective adhesive already applied to the bracket.

The brackets were then directly positioned and centered on the primed enamel surface. Firm pressure perpendicular to the bracket was then applied with a ligature director to ensure cement was expressed beyond the outer border of the bracket pad. Any excess cement present beyond the bracket pad edge was removed in the conventional and pre-coated groups with an explorer placed flush against the tooth and then directed away from the bracket. This was followed by light-curing from the mesial and distal of the brackets for three seconds each, six seconds total, with an Ortholux$^{TM}$ Luminous Curing Light (3M Unitek, St. Paul, MN) on the conventionally-pasted and pre-coated metal brackets and six seconds on the mesial and distal, twelve seconds total, for the flash-free brackets. These curing instructions follow the recommended orthodontic direct bonding protocol from 3M. A matrix with each step and comparisons of the bracket-adhesive combinations are illustrated in Figure 5.

This protocol was initially conducted with metal orthodontic brackets. After micro-CT scanning, the differences in densities between the bracket, adhesive, and tooth, due to the radiopacity of the metal bracket, led to obscured results. Due to this, the protocol was adjusted and repeated with ceramic brackets. The light curing protocol was amended to a three second light cure directly through the bracket as instructed in the 3M direct bonding protocol for ceramic brackets for all of the adhesive systems used.

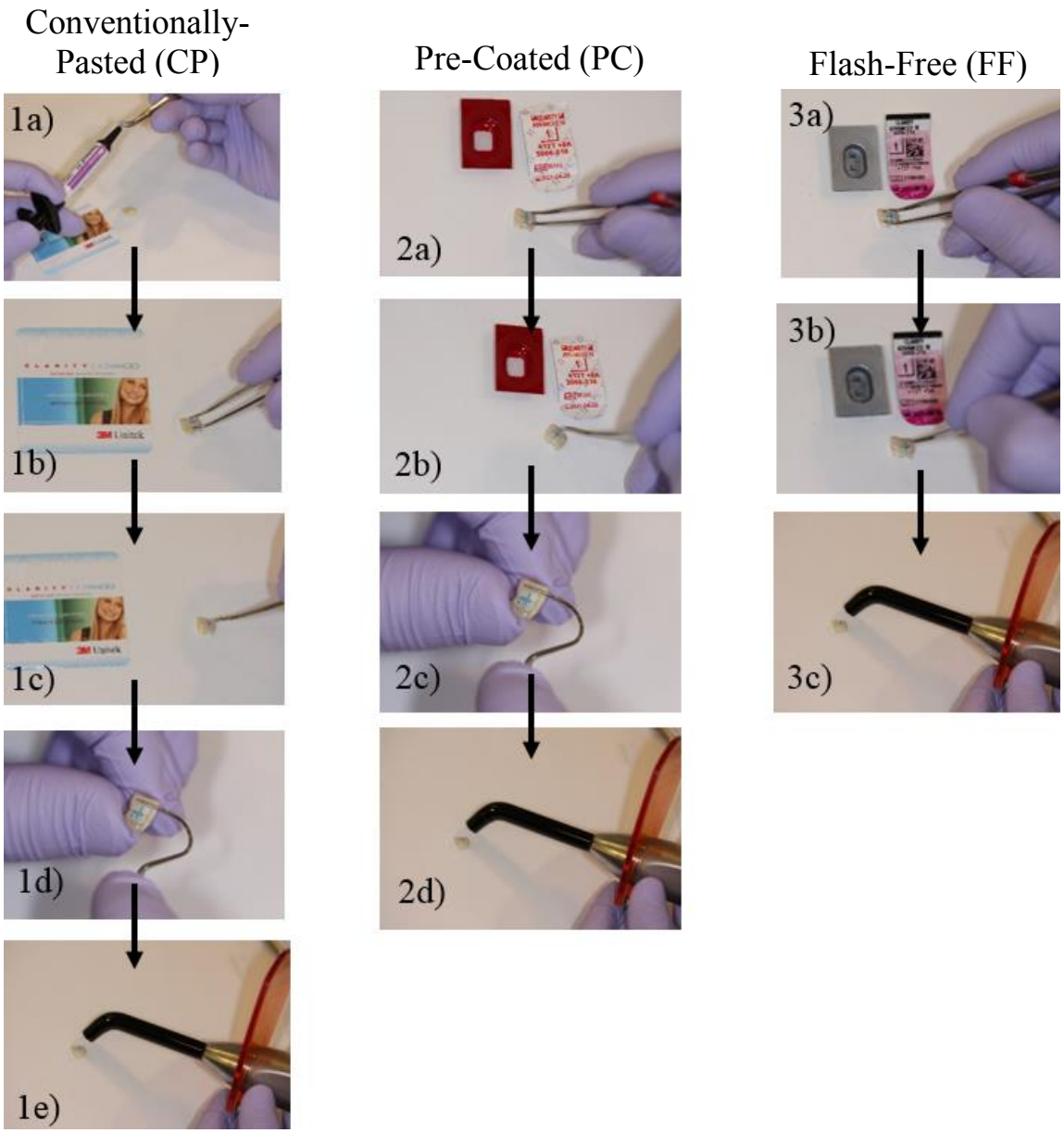


**Figure 5:** Bonding of brackets to extracted tooth. 1a) adhesive applied, 1b) bracket applied, 1c) perpendicular pressure, 1d) excess removed, 1e) light cure, 2a) bracket applied, 2b) excess removed, 2c) perpendicular pressure, 2d) light cure, 3a) bracket applied, 3b) perpendicular pressure, 3c) light cure

### 4.3 Micro-CT Scanning

Prepared samples in each group (n=5) were labeled according to the type of adhesive and number in which the sample is being scanned. As a result, the tested samples were labeled CP1, CP2, CP3, CP4, and CP5 for the conventionally-pasted group, PC1, PC2, PC3, PC4, and PC5 for the pre-coated group, and FF1, FF2, FF3, FF4, and FF5 for the flash-free group. Each sample was individually wrapped in parafilm prior to scanning. Each group was placed in a tube with the bracket pad parallel to the floor (Figure 6).

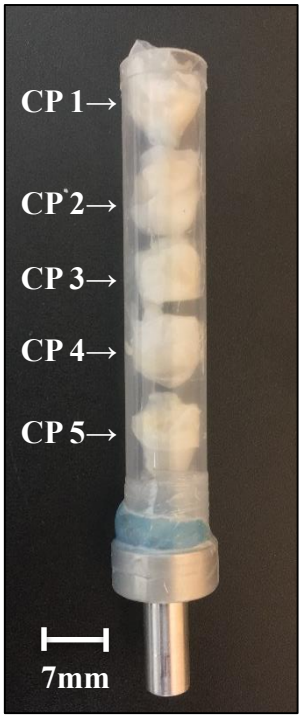


**Figure 6:** A batch of 5 samples (CP group) in tube holder wrapped with parafilm and ready for micro-CT scan

Micro Computed Tomography (micro-CT) using the Skyscan 1172 micro-CT (Skyscan, Kontich, Belgium) was utilized in order to 3-dimensionally scan the adhesive

between the bracket and tooth without having to debond the bracket. Scanning parameters were obtained from the master's thesis of Leonid Epshteyn and used for preliminary testing (Epshteyn 2016). After scanning test samples, the values were adjusted as follows due to differences in scanned materials including densities between the bracket, adhesive, voids, and enamel. Data acquisition values for scanning include a source voltage of 80 kV. Source current was 124 mAs. Camera size (# of rows) is small. Image pixel size was 3 microns. A 0.5mm Aluminum filter was used. Ring artifact correction is 40%. Rotation step was placed at 0.5 and frame averaging was placed at 7. Reconstruction values include ring artifact correction of 10 and beam hardening at 40%. These parameters were used in order to compensate for the differences of density and be able to distinguish adhesive from void during data analysis.

### 4.4 Micro-CT Data Analysis

Data from the micro-CT were analyzed with CTAn Micro-CT software (Bruker, Billerica, MA). This allowed for 3D reconstructions, image processing, and analysis of the samples of interest to quantify data relating to the internal architecture of the samples, void volume, and void location. First, the reconstructed image was loaded in the CTAn program. Next, the regions of interest preview were selected. The region of interest (ROI) was drawn centered on the flattest portion of the tooth and then was saved as a dataset. To eliminate variability in tooth morphology and measure void in a similar volume of adhesive, a uniform region of interest of 2086.9 x 670.6 $\mu m^2$ was utilized in all samples. The ROI had to include the bracket-adhesive and adhesive-tooth interfaces in each slice. This size area was selected to establish a uniform width and the height that included the adhesive and

both interfaces for all 15 samples. Next, each slice was processed to remove the top portion of the ROI that was made up of the bracket and the bottom portion of the ROI that was the tooth. This was saved as the volume of interest (VOI). The VOI was then opened with a top and bottom slice selected where each had to include the top and bottom interfaces of adhesive. 700 slices were evaluated for each sample. A task list was created for batch analysis. Global threshold was placed at Lower Grey = 30 and Higher Grey = 225. Despeckling was done to remove white speckles less than 5 pixels and then 3D analysis was performed. These parameters were chosen to distinguish voids from adhesive and quantify void volume.

While each scan started with a uniform ROI, the thicknesses of the adhesive were different for all samples even though the selected ROI had the same width across. Due to this, the selected quantitative variable for comparison was void percentage. This allowed for acceptable statistical analysis since utilizing a percentage of volume for each adhesive would allow for comparisons despite differences in total volume of the adhesive. The three variables measured were defined (in percentages) as total void ($V_{tot}$), open void where the void contacted an interface (either the bracket or enamel) ($V_{int}$), and closed void where the void was contained entirely inside the bulk of the adhesive ($V_{bulk}$) (Figure 7).

Collected data was statistically analyzed with a Shapiro-Wilk test of normality to indicate if the groups were normally distributed. ANOVA tests were then run to evaluate any potential significant differences for all three variables ($V_{tot,}$ $V_{int,}$ $V_{bulk}$). All of the ANOVA analyses included a Tukey's post hoc analysis and were performed with $p \leq 0.05$ indicating a significant difference.

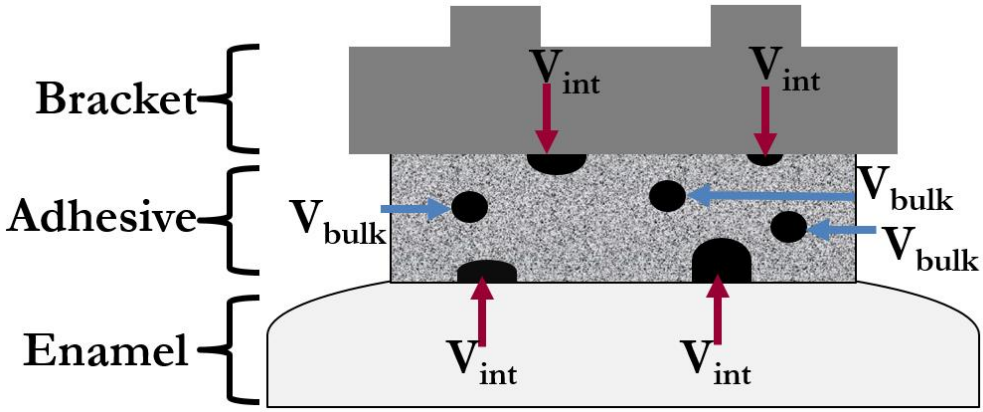


**Figure 7:** Cartoon representation of voids

### 4.5 Bond Strength Test

After scanning, every bonded sample was mounted in an epoxy resin (EpoxyMount, Allied High Tech) with cylinder shape (4×6 $in^2$) following procedures recommended by manufacturer. The samples were fixed in a mechanical universal testing machine (Instron 5569; Norwood, MA) using a vise. Brackets bonded to the tooth were subjected to shear-bond tests (Figure 8). A metal blade was fabricated to provide a force perpendicular to the base of the orthodontic bracket (Figure 9). The metal blade was based off designs of multiple past shear bond strength studies, and the blade generated pressure perpendicular to the bonding line between the bracket and the tooth (Linjawi & Abbassy 2016; Isber 2010; Rambhia 2009). The blade was rested on the adhesive/bracket surface, most common site for force application in debonding studies (Finnema 2010). Using the Instron® machine and software, samples were loaded to failure under quasi-static loading conditions and a strain rate of 0.06 $s^{-1}$. The actuator displacement (mm) and the load (N) were recorded

during test. The maximal force prior to bond failure was identified from the load-displacement curve. The bonding stress (MPa) was calculated by dividing the force by the cross-sectional bracket area (3.65 mm × 3.90 mm).


**Figure 8:** Instron® Machine used for bonding strength evaluation

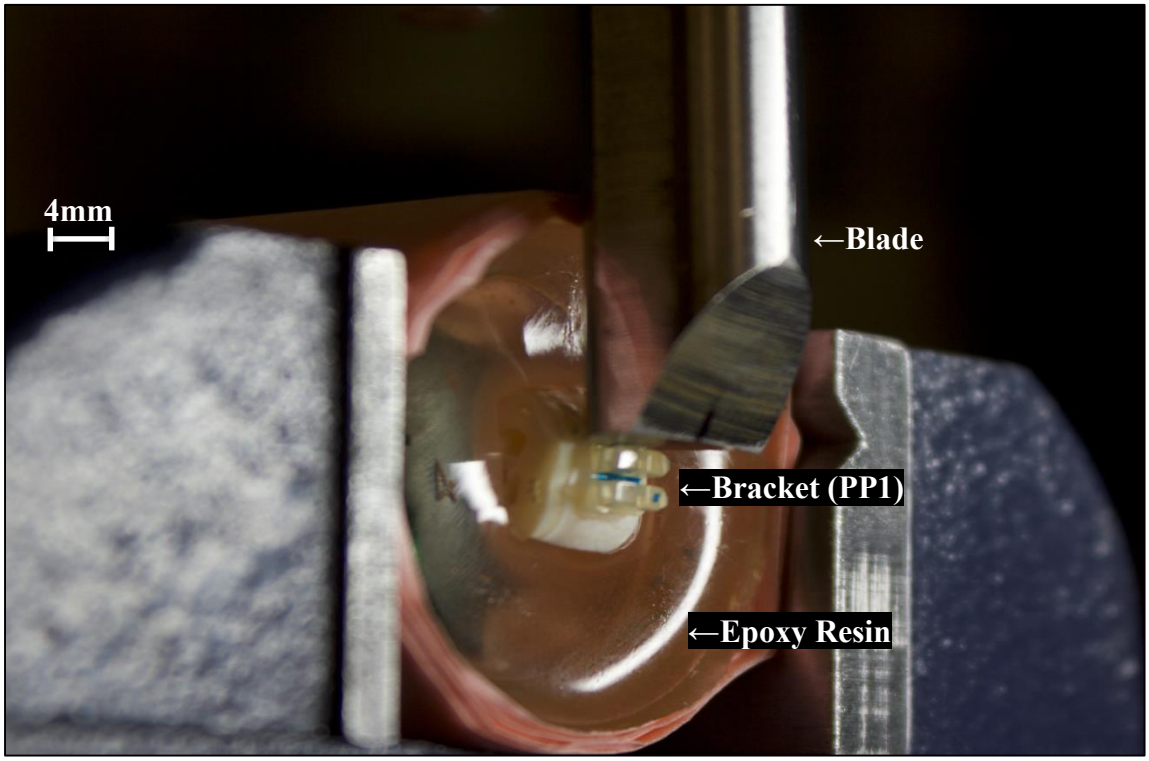


**Figure 9:** Mounted sample (PP1) before shear bonding test

# CHAPTER 5

# RESULTS

## 5.1 Void Results

Scanning the internal three-dimensional structure of the different bracket-adhesive combinations yielded differences in the overall ($V_{tot}$), interface ($V_{int}$), and bulk ($V_{bulk}$) volume of voids. Figure 10 illustrates the micro-CT scan of a representative bracket bonded to the tooth. A higher magnification view of the adhesive denotes the region of interest (ROI) and volume of interest (VOI) with the designations of $V_{int}$ and $V_{bulk}$, which were used to calculate the amount of void in each group. Figures 11, 12, and 13 represent a scanned slice of conventionally-pasted, pre-coated, and flash-free adhesive, respectively, with the bracket and tooth removed in order to quantify the voids per region.

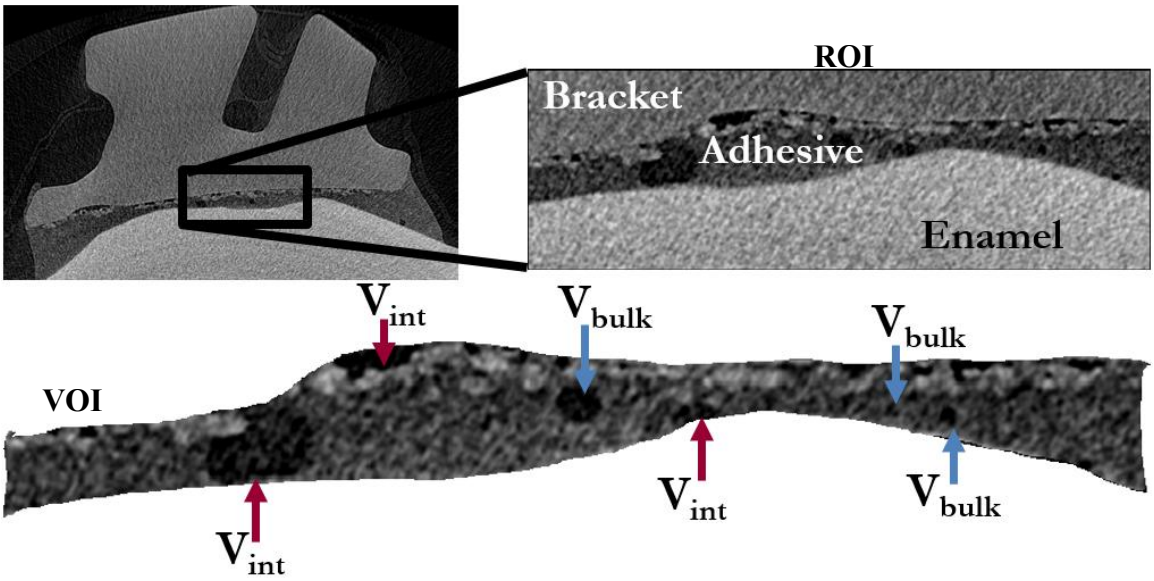


**Figure 10:** Labeled sample after micro-CT scan

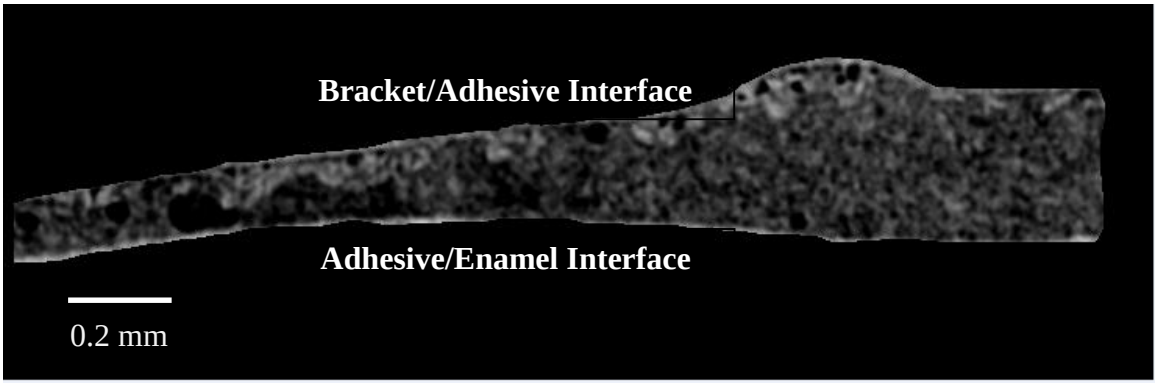

**Figure 11:** Conventionally-pasted (CP) Sample 5 Adhesive VOI Slice 2452

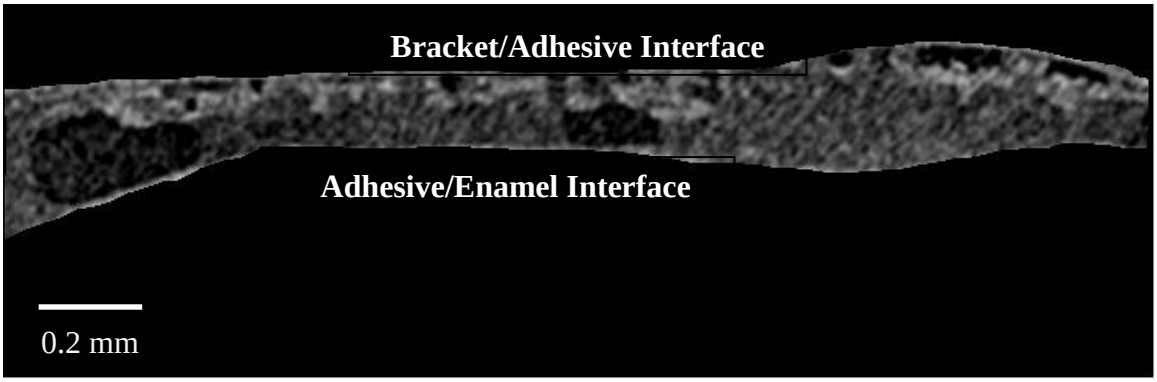

**Figure 12:** Pre-coated (PC) Sample 4 Adhesive VOI Slice 1832

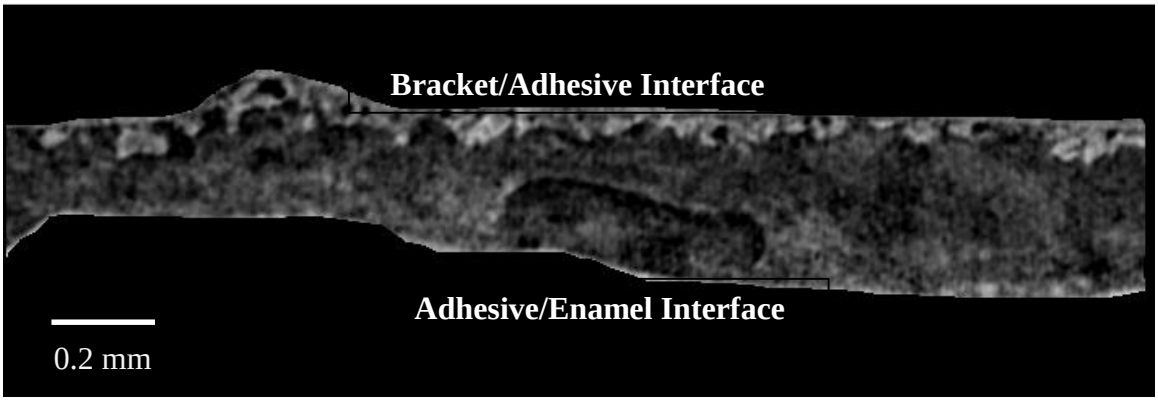

**Figure 13:** Flash-free (FF) Sample 5 Adhesive VOI Slice 2604

Comparing the void between the conventionally-pasted (CP) and pre-coated (PC) groups revealed no statistically significant ($p>0.05$) differences for any of the variables tested ($V_{tot}$, $V_{int}$, $V_{bulk}$). On the contrary, flash-free (FF) brackets had a statistically significant ($p<0.05$) higher amount of void overall ($V_{tot}$) and at the interfaces ($V_{int}$) when compared to the conventionally-pasted and pre-coated groups. Surprisingly, FF had a statistically significant ($p<0.05$) lower volume of void within the bulk of the adhesive ($V_{bulk}$) when compared to CP and PC although this contributed much less to the overall volume of the void. In each group, the majority of void was found at interfaces (adhesive/enamel and adhesive/bracket) rather than within the bulk of the adhesive. Specifically, 85.0%, 71.3%, and 96.8% of the overall void was found at interfaces in the conventionally-pasted, pre-coated, and flash-free groups, respectively. The location of the voids was higher at the interfaces versus the bulk for all groups with statistical significance ($p<0.05$) for CP and FF . These results are illustrated in Figure 14. The quantification of the voids ($V_{tot}$, $V_{int}$, $V_{bulk}$) for each evaluated group is shown in Table 7. Statistical analyses are shown in Tables 1 through 7.

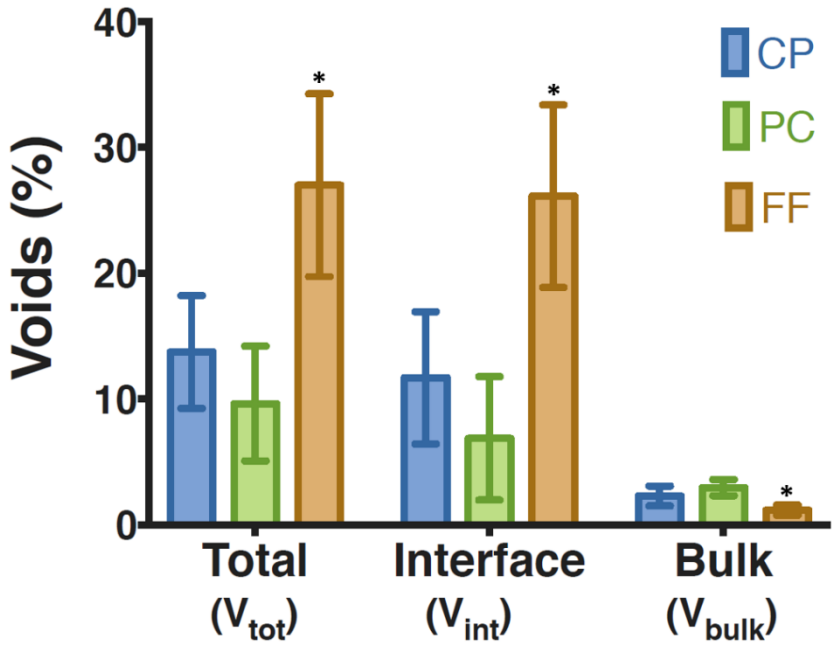


**Figure 14:** Amount of void of each evaluated group and broken down per analyzed location including voids at the bulk of the adhesive ($V_{bulk}$), at the bonded interfaces ($V_{int}$) and the overall amount of voids ($V_{tot}=V_{bulk} + V_{int}$ ). Significant difference between bracket groups (FF, PC and CP) were indicated by asterisk symbol ($p<0.05$)

### 5.2 Bond Strength Results

After scanning and evaluation of the voids under micro-CT, the same samples underwent bond strength testing. The bond strength testing revealed a mean stress prior to debonding of 9.02±0.90 MPa for the conventionally-pasted group, 16.59±2.63 MPa for the pre-coated group, and 8.22±3.69 MPa for the flash-free group. PC, the group with the lowest voids, had the highest bonding strength, while FF, the group with the highest voids, had the lowest bonding strength. These results were based off sample sizes of n=2 for CP group, n=4 for PC group, and n=3 for FF group. Samples with invalid tests due to mounting error, mechanical error, fracture at the wing of the bracket, or fractures of the tooth itself were excluded from the results. Due to the low number of samples, statistical analysis could

not be run. Bond strength results are compared in Figure 15 with the full results in Table 8.

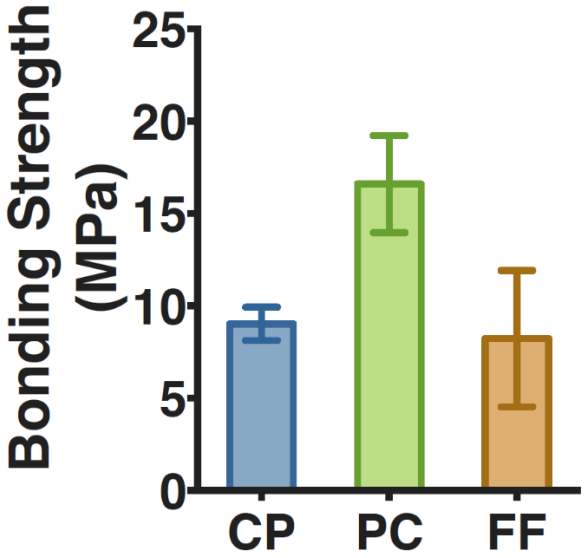


**Figure 15:** Bonding strength for each evaluated bracket group including conventionally-pasted (CP), pre-coated (PC) and flash-free (FF).

# CHAPTER 6

# DISCUSSION

Using micro-CT, this investigation provided the first study of measuring void volume and quantity and distribution of location of voids in bonded orthodontic brackets. Three different bracket-adhesive combinations including conventionally-pasted, pre-coated, and flash-free brackets were evaluated for differences in these areas. In addition, bond strength tests were conducted to find possible correlations between voids and bonding strength. A previous study evaluating Class II composite resin restorations stated that voids were "highly predictive of bond strengths" and found an *in vivo* axial area of voids equal to 13.6 ± 25.6% (n=12) (Purk 2007). The total void volume percentage for each group in our study is covered within this standard deviation, which may indicate that, similar to resin restorations, voids may have an impact on bond strength of orthodontic brackets.

Our results found that pre-coated brackets had the lowest total percentage of volume with voids while the flash-free brackets had the highest. Voids were found to be more present at the interfaces, rather than within the bulk of the material for the three types of brackets systems. Voids were visible at both interfaces on the micro-CT scans including adhesive/enamel and adhesive/bracket. A recent study evaluated the bond condition of conventional and flash-free adhesives with micro-CT, where they utilized a silver nitrate dye to evaluate its penetration into the adhesive (Grunheid 2015). They found no statistically significant differences of the microleakage between conventional and flash free brackets. However, the study only evaluated voids at the adhesive periphery, which is inadequate to evaluate the bond condition. While our study did not covered the entirety of

the adhesive, the centered region of interest ensures that the internal bonded areas of samples are being evaluated, not just around the border as is seen in other studies.

This study revealed the potential relationship between adhesive void and bonding strength for these three different bracket-adhesive combinations. Our bond strength results are consistent with those of previous studies. The bond strength values in all groups obtained from this study are within ranges reported in a systematic review with the lowest value at 8.22 MPa (FF) to the highest at 16.59 MPa (FF) (Finnema 2010). In addition, a recent study found the bonding strength of flash-free brackets is 13.7±2.2 MPa, which is consistent with our bond strength results for flash-free brackets (Lee & Kanavakis 2015).

The preliminary results of this investigation indicate a relationship between adhesive void and bonding strength. Pre-coated brackets had the lowest amount of void and the highest bond strength while the flash-free group had the highest amount of void and the lowest bond strength. The existence of voids in dental materials give rise to stress concentration (Spencer 2010). As a result, the bonding strength may be reduced, resulting in premature failure of the bonded system (Martini 2009). The results of this investigation are consistent with previous findings. In addition, an increased void at the bonded interface ($V_{int}$) reduces the effective adhesive surface area contributing to the reduction of bonding strength (Purk 2007). Using conclusions from separate void and bond strength studies, the two factors together appear to have an association that was previously unstudied in relation to orthodontic adhesives.

### 6.1 Limitations & Sources of Error

The results of this study suggest that the majority of voids in bracket adhesives are located at the bonded interfaces and associated with low bond strength. There may be some possible limitations in this study. Metal brackets were first run to obtain micro-CT scanning parameters, but the metal bracket obscured some of the voids, particularly at the bracket-adhesive interface, and due to the three significant differences in densities (bracket, adhesive, and tooth) the data could not be clearly read. To this end, the protocol was adjusted to use ceramic brackets. Due to their similar density under micro-CT analysis, the analysis of the adhesive and void was much more defined. However, this could have had a negative impact on the bracket debonding values. Unlike a metal bracket, ceramic brackets are inherently more rigid and brittle which can lead to partial or complete failure or fracture of the bracket itself when debonding (Theodorakopoulou 2004). This led to several invalid results when running the shear bond strength tests making the sample size smaller in some groups and decreasing the associations that could be drawn between percentage of void volume and maximum load before debonding.

Another limitation of this study is that the number of samples utilized for bond strength tests was insufficient. We initially chose n=5 based on preliminary data obtained from previous masters' theses on micro-CT analysis (Epshteyn 2016). However, during mechanical testing some samples were removed from the data set due to mounting error, mechanical error, fracture at the wing of the bracket, or fractures of the tooth itself. Although the bond strength test was not the primary aim of this study, a larger sample size and decreased invalidity could assist in making more robust conclusions between void volume and bond strength.

The debonding protocol also introduced potential limitations of our results. The differences in adhesive thickness and overall adhesive width from bracket edge-to-edge with possible excess could influence the bond strength calculation. Differences in adhesive thickness and width could be rectified by looking at void within the entire width of the adhesive, but that was beyond the scope of this study's protocol. This study assumed constant width and thickness of adhesive.

Another potential limitation was the selection of the bracket surface used for bond strength testing. In this study, the mesial or distal surface of the bracket was used as the location for force application due to the necessity of having a flush surface. We prevented any unwanted contact between the blade and the wing of the bracket that interfered with uniform stress application. Since multiple studies with shear bond testing have this force from gingival to occlusal or occlusal to gingiva, there may be a confounding of the findings (Linjawi & Abbassy 2016; Isber 2010). A thinner blade and judicious removal of the flash from the CP and PC samples may also result in lower test invalidity.

Finally, even though all samples were bonded by one practitioner (SB), differences in pressure during bonding may have an effect on void generation. Standardization of pressure with a machine could reduce this possible source of error.

### 6.2 Future Studies

This study provided a new approach an evaluation of internal void of brackets at the bonded bracket-adhesive-tooth interfaces. As a result, future studies are warranted.

While this study did not standardize the pressure related to bracket placement, future studies can explore the influence of operator pressure on voids, adhesive thickness,

and bond strength. In addition, future studies can include a look at which interface more voids are present (i.e. enamel/adhesive vs adhesive/bracket). In addition, future studies should have a closer look at the failure location (i. e. Bulk of adhesive or interfaces) to find possible relations between void and bonding strength. A comparison between metal and ceramic brackets should be considered to identify the influence of voids on failure of brackets.

# CHAPTER 7

## CONCLUSIONS

This study evaluated the internal void and bonding strength of three orthodontic bracket technologies. The following conclusions can be drawn from the results:

1. The void found at the bonded interfaces of the flash free brackets was statistically significant higher compared to conventionally-pasted and pre-coated brackets
2. The amount of void between conventionally-pasted and pre-coated brackets was not statistically significant
3. The majority of the void was present at the bonded interfaces rather than within the bulk of the adhesive for all brackets (conventionally-pasted, pre-coated, and flash-free)
4. The flash-free brackets show the lowest average bonding strength compared to conventionally-pasted and pre-coated brackets but will need a larger sample to statistically confirm these findings and trends

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# APPENDIX

## SHAPIRO-WILK TESTS OF NORMALITY

**Table 1:** Shapiro-Wilk Tests of Normality

| Shapiro-Wilk Tests of Normality | | | | | | | |
|---|---|---|---|---|---|---|---|
| $V_{tot}$ | CP | .200 | 5 | .200* | .972 | 5 | .886 |
| | PC | .178 | 5 | .200* | .940 | 5 | .663 |
| | FF | .225 | 5 | .200* | .924 | 5 | .558 |
| $V_{int}$ | CP | .223 | 5 | .200* | .972 | 5 | .888 |
| | PC | .214 | 5 | .200* | .900 | 5 | .413 |
| | FF | .273 | 5 | .200* | .859 | 5 | .224 |
| $V_{bulk}$ | CP | .244 | 5 | .200* | .961 | 5 | .817 |
| | PC | .188 | 5 | .200* | .950 | 5 | .737 |
| | FF | .259 | 5 | .200* | .875 | 5 | .285 |

*. This is a lower bound of the true significance.

Indicates that all groups have a normal distribution. A failed significance test indicates normalcy.

# ANOVA

**Table 2:** ANOVA

| ANOVA | | | | | | |
|---|---|---|---|---|---|---|
| | | **Sum of Squares** | **df** | **Mean Square** | **F** | **Sig.** |
| **$V_{bulk}$** | Between Groups (Combined) | 8.09 | 2 | 4.048 | 10.06 | .003 |
| | Within Groups | 4.82 | 12 | .402 | | |
| | Total | 12.91 | 14 | | | |
| **$V_{int}$** | Between Groups (Combined) | 1003.03 | 2 | 501.518 | 14.41 | .001 |
| | Within Groups | 417.51 | 12 | 34.793 | | |
| | Total | 1420.55 | 14 | | | |
| **$V_{tot}$** | Between Groups (Combined) | 821.39 | 2 | 410.695 | 13.17 | .001 |
| | Within Groups | 374.09 | 12 | 31.175 | | |
| | Total | 1195.49 | 14 | | | |

ANOVA tests run in order to determine if post-hoc Tukey's tests can be run. All groups have a significant ANOVA, so post-hoc tests can be performed.

## TUKEY'S TEST – TOTAL VOIDS

**Table 3:** Tukey's Test – Total Voids

| (I) group | (J) group | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval | |
|---|---|---|---|---|---|---|
| | | | | | Lower Bound | Upper Bound |
| CP | PC | 4.102 | 3.531 | .497 | -5.318 | 13.523 |
| | FF | -13.239* | 3.531 | .007 | -22.660 | -3.818 |
| PC | CP | -4.102 | 3.531 | .497 | -13.523 | 5.318 |
| | FF | -17.341* | 3.531 | .001 | -26.762 | -7.920 |
| FF | CP | 13.239* | 3.531 | .007 | 3.818 | 22.660 |
| | PC | 17.341* | 3.531 | .001 | 7.920 | 26.762 |

Based on observed means. The error term is Mean Square (Error) = 31.175.
*. The mean difference is significant at the .05 level.
Significant difference ($p<0.05$) between CP and FF groups.
Significant difference ($p<0.05$) between PC and FF groups.
No significant difference ($p>0.05$) between CP and PC groups.

## TUKEY'S TEST – VOIDS AT AN INTERFACE

**Table 4:** Tukey's Test – Voids at an Interface

| (I) group | (J) group | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval | |
|---|---|---|---|---|---|---|
| | | | | | Lower Bound | Upper Bound |
| CP | PC | 4.810 | 3.730 | .427 | -5.141 | 14.763 |
| | FF | -14.433* | 3.730 | .006 | -24.386 | -4.480 |
| PC | CP | -4.810 | 3.730 | .427 | -14.763 | 5.141 |
| | FF | -19.244* | 3.730 | .001 | -29.197 | -9.291 |
| FF | CP | 14.4335420* | 3.73058057 | .006 | 4.4808617 | 24.3862223 |
| | PC | 19.2444660* | 3.73058057 | .001 | 9.2917857 | 29.1971463 |

Based on observed means. The error term is Mean Square (Error) = 34.793.
*. The mean difference is significant at the .05 level.
Significant difference ($p<0.05$) between CP and FF groups.
Significant difference ($p<0.05$) between PC and FF groups.
No significant difference ($p>0.05$) between CP and PC groups.

## TUKEY'S TEST – VOIDS WITHIN THE BULK

**Table 5:** Tukey's Test – Voids within the Bulk

| (I) group | (J) group | Mean Difference (I-J) | Std. Error | Sig. | 95% Confidence Interval | |
|---|---|---|---|---|---|---|
| | | | | | Lower Bound | Upper Bound |
| CP | PC | -.665 | .400 | .260 | -1.735 | .404 |
| | FF | 1.115* | .400 | .041 | .045 | 2.185 |
| PC | CP | .665 | .400 | .260 | -.404 | 1.735 |
| | FF | 1.780* | .400 | .002 | .710 | 2.850 |
| FF | CP | -1.115* | .400 | .041 | -2.185 | -.045 |
| | PC | -1.780* | .400 | .002 | -2.850 | -.710 |

Based on observed means. The error term is Mean Square (Error) = .402.
*. The mean difference is significant at the .05 level.
Significant difference ($p<0.05$) between CP and FF groups.
Significant difference ($p<0.05$) between PC and FF groups.
No significant difference ($p>0.05$) between CP and PC groups.

## VOID LOCATION

**Table 6:** Void Location

| Group | Location | Mean | P Value | 95% Confidence Interval | Std. Error OF Difference |
|---|---|---|---|---|---|
| CP | $V_{bulk}$ | 2.299 | 0.0251* | -16.863 to -1.920 | 2.691 |
| | $V_{int}$ | 11.691 | | | |
| PC | $V_{bulk}$ | 2.964 | 0.1671 | -10.365 to 2.533 | 2.323 |
| | $V_{int}$ | 6.880 | | | |
| FF | $V_{bulk}$ | 1.184 | 0.0015* | -33.822 to -16.061 | 3.198 |
| | $V_{int}$ | 26.125 | | | |

* Statistical Significance ($p<0.05$)
Location of the voids are statistically significantly higher at the interfaces versus the voids for CP and FF

## VOID STATISTICS

**Table 7:** Void Statistics

| Void Statistics | | | | | |
|---|---|---|---|---|---|
| | **Group** | **N** | **Mean** | **Std. Deviation** | **Std. Error Mean** |
| **$V_{bulk}$** | CP | 5 | 2.299 | .793 | .354 |
| | PC | 5 | 2.964 | .640 | .286 |
| | FF | 5 | 1.184 | .408 | .182 |
| **$V_{int}$** | CP | 5 | 11.691 | 5.257 | 2.351 |
| | PC | 5 | 6.880 | 4.903 | 2.192 |
| | FF | 5 | 26.125 | 7.258 | 3.246 |
| **$V_{tot}$** | CP | 5 | 13.754 | 4.479 | 2.003 |
| | PC | 5 | 9.651 | 4.562 | 2.040 |
| | FF | 5 | 26.993 | 7.255 | 3.244 |

Mean and standard deviation void results at the bulk, interfaces, and total for CP, PC, and FF

## STRESS (MPa) RESULTS

**Table 8:** Stress (MPa) Results

| | **Stress (MPa) Results** | | |
|---|---|---|---|
| | **CP** | **PC** | **FF** |
| **Sample 1** | N/A | N/A | N/A |
| **Sample 2** | N/A | 13.30 | N/A |
| **Sample 3** | 8.38 | 16.24 | 4.78 |
| **Sample 4** | N/A | 17.16 | 7.76 |
| **Sample 5** | 9.65 | 19.67 | 12.11 |
| **Average** | **9.02** | **16.59** | **8.22** |
| **Standard Deviation** | **0.90** | **2.63** | **3.69** |

Bond Strength Results: Mean and standard deviation for each sample (1-5) in the three groups – CP, PC, and FF
N/A denotes an invalid test