EFFECTS OF CURING MODES ON DEPTH OF CURE AND MICROTENSILE BOND STRENGTH TO DENTIN OF BULK FILL RESIN COMPOSITES

by

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Abstract

Objectives: To compare microtensile bond strength (μTBS) and depth of cure (DOC) of bulk fill composites cured by monowave (MW) and polywave (PW) LED units at different curing times.

Materials and methods: Three composites were tested: Tetric EvoCeram Bulk Fill (TBF), Filtek Bulk Fill (FBF) and Tetric EvoCeram (T). Flat dentin surfaces treated with adhesive (Adhese, Ivoclar Vivadent) were bonded with cylindrical 4mm bulk composite of each material (n=6) and cured with monowave (Satelec) or polywave (Bluephase Style) curing units at 10s or 20s. After 24 hours, teeth were sectioned in individual 0.9 mm^2 beams and tested for μTBS. In addition, the DOC (IOS 4090) scrape test was performed (n=5) following the same curing protocols. Two-way ANOVA (α=0.05) was performed isolating light curing units.

Results: MW light curing unit showed no statistical significant difference in μTBS for all materials and curing times tested. Likewise, the PW light curing unit did not show differences, except for TBF. Overall, bulk fill composites presented greater DOC and longer curing time resulted in higher DOC for all composites.

Conclusion: Curing mode does not result in differences in μTBS to dentin. However, bulk fill composites exhibit greater DOC than conventional resin composites.

Lay Summary

Introduction: Bulk fill (BF) composites are another class of composites (white fillings) introduced to simplify technique sensitive restoration steps when restoring cavities.

Objectives: We wanted to investigate how much BF composites bond to the dentin of the teeth comparing the use of two different light emitting devices used for material setting when restoring teeth with composites. In addition, we wanted to evaluate materials ability to transmit the light energy from the light emitting devices when applied in thick layer into the tooth at two exposure times for comparison.

Materials and methods: The bond strength of two types of BF composites was tested and compared to conventionally used composite.

Results: BF composites show good bond strength to dentin and high translucency.

Conclusion: BF composites have high translucency that allows light penetration and bonding to the tooth. However, better setting is associated with increased exposure time to light emitting device.

Preface

The input and reinforcement of methodology was done by supervisor Dr. Adriana Manso and committee members Dr. Ricardo Carvalho and Dr. Karen Campbell. Sample collection, sample preparation and data collection was done by Dr. Sara Makhdoom with advice from Dr. Adriana Manso and Dr. Ricardo Carvalho. Statistical analyses were performed by Dr. Adriana Manso and Dr. Sara Makhdoom.

This study was approved by the Clinical Research Ethics Board of The University of British Columbia, protocol ID H16-03262.

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List of Abbreviations

AAPD: The American Academy of Paediatric Dentistry

BAPO: Bis-Alkyl Phosphine Oxide phosphine oxide

BF: Bulk fill

Bis-GMA: Bisphenol A Glycolmethacrylate

CQ: Camphorquinone

DC: Degree of Conversion

DOC: Depth Of Cure

EBPDMA: Ethoxylated Bis-GMA

FBF: Filtek Bulk Fill

LCU: light curing unit

LED: Light emitting diode

MEK: Methyl Ethyl Ketone

μTBS: Microtensile bond strength

MW: Monowave

PPD: 1-phenyl- 1,2-propanedione

PW: Polywave

RBCs: Resin based composites

SBS: Shear Bond Strengths

SEM: Scanning Electron Microscopy

TEGDMA: Triethyleneglycol dimethacrylate

T: Tetric EvoCeram

TBF: Tetric EvoCeram Bulk Fill

TPO: Trimethylbenzoyl- Diphenyl Phosphine Oxide

UDMA: Urethane Dimethacrylate

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Dedication

I dedicate this thesis to my parents for their priceless presence and continuous support throughout my life journey- I will do my best to make you proud of me.

I dedicate this thesis to my small family, my son and my husband- Thank you for your love and care. I am all yours.

I dedicate this thesis to my big family and friends for their sincere presence and support.

Chapter 1: Introduction and Literature Review

Resin based composites (RBCs) have been considered desirable for use as anterior and posterior restoratives as they mimic natural tooth appearance. When applied properly they are relatively stable in the oral cavity, with excellent clinical performance and longevity demonstrated up to 12 years (Opdam et al., 2010). In addition, RBC can be ideal for minimally invasive cavity preparations, as it bonds to tooth structure, thereby allowing shallow preparations without the need for mechanical retentive features to prevent dislodgement.

RBCs are conservative restorative treatment options for primary and permanent teeth as the scope of Paediatric Dentistry practice spans the period during which the dentition is in a state of transition, including individuals up to early adulthood. However, according to The American Academy of Paediatric Dentistry (AAPD) recommendations, RBC restorations are not an ideal option for uncooperative patients (Donly & García-Godoy, 2015; Antony et al., 2008) since isolation is difficult yet essential for long-lasting results. The use of bulk fill (BF) composite materials for paediatric patients could potentially offer a faster, easier and more efficient clinical procedure when restoring with RBCs but still child cooperation and chair time are crucial.

1.1 Resin Composites – An Overview

RBCs are multifaceted materials with a very distinctive composition, introduced for multiple purposes in dentistry. In general, RBCs are a mixture of resin matrix, inorganic fillers, coupling agent and other essential elements such as photoinitiators and pigments. Research in RBCs has been constantly evolving with the aim of improving mechanical

properties, handling characteristics and aesthetic qualities to yield a direct restorative material with satisfactory and long-lasting outcomes. An interesting chronological progress in resin composite research and development has been observed in the technology of filler particles, resin matrix composition, photoinitiator systems and application technique (Ferracane, 2011; Leprince, 2013). Nevertheless, they are very technique sensitive materials and demand excellent isolation for optimum results (Donly, 2015; Swift 2002). Since RBCs were first introduced, many modifications have been adopted in their composition, aiming for a tougher, more resistant material with improved mechanical and optical properties. Fillers (content, size and shapes), polymeric matrices, and photoinitiators account for the predominant component changes (Ferracane; 2011).

More recently, BF composites were introduced, aiming to eliminate the need for incremental filling by increasing the DOC of the material. In order to accommodate curing of 4-5 mm increments at a time, manufacturers improved the conventional RBCs formula by enhancing the photoinitiator system and reducing the resin composite opacity, thus creating a more translucent material to ease the light penetration (Bucuta & Ilie, 2014; Ilie, Bucuta & Draenert, 2013; Czasch, Ilie, 2012). BF composites are commercially available in different viscosities (flowable, restorative) with various clinical application and dispensing systems. Even though the material has garnered considerable clinical interest due to its convenience and time-saving application, the long-term clinical performance compared to conventional RBCs is not well understood.

1.2 Monomers and Filler Particles

The viscosity of the matrix affects the polymerization reaction (Czasch & Ilie, 2012). Monomers with less viscosity allow for more flexible network interaction during

polymerization and subsequent enhancement of monomer conversion (Sideridou, Tserki & Papanastasiou; 2002). Most of the RBCs are Bis-GMA based, which has fewer hydrophilic monomers than others such as UDMA, TEGDMA, and EBPDMA (Sideridou, Tserki & Papanastasiou; 2002). The addition of the low molecular weight monomer to RBCs is a common strategy which results in a more flexible material (Amirouche-Korichi et al, 2009; Ferracane,1986; Frauscher & Ilie,2013). Therefore, these monomers are also added to the BF composite formulations (Zorzin et al., 2015). The dilution effect of UDMA, which resulted in reduction of polymerization shrinkage and delayed gelation (Ilie & Hickel, 2011). In addition to these strategies, some manufacturers added monomers known as "stress relievers" to BF resinous materials (Zorzin et al., 2015) in the attempt to minimize the stresses generated at the interface (Zorzin et al., 2015; Czasch & Ilie, 2012; El-Damanhoury & Platt, 2014; Guo et al., 2016). Guo and collaborators compared flowable BF composite (Filtek Bulk Fill) with conventional packable composite (Filtek Z250) with respect to three important aspects of their filler contents: polymerization stresses, exothermic reaction, and degree of conversion (DC). The authors found good internal adaptation and lower shrinkage stress with flowable BF composite; and also, higher DC and a slightly increased exothermic reaction (Guo et al., 2016).

A recent study by Güler & Karaman, 2014 revealed lower polymerization shrinkage stresses but comparable cuspal deflection when BF composites were compared with conventional RBCs in MOD restorations. In this study, they applied BF as base and as bulk restoration. In contrast, another study observed minimal reduction in cuspal deflection for flowable BF composites applied as a base in high C-factor cavities (Moorthy et al., 2012).

BF resin composite presents adequate DOC and acceptable hardness (Bucuta & Ilie, 2014; Zorzin et al., 2015). The increased filler size and decreased filler content improves the DC and increases translucency with the benefit of reducing volumetric shrinkage and polymerization shrinkage stresses (Finan et al., 2013; Zorzin et al., 2015). However, BF flowable composites became rougher and more prone to abrasion with compromised esthetics and mechanical properties as consequence of those modifications (Czasch & Ilie, 2012). In general, flowable BF composites have less fillers than the packable bulk materials, which results in lower mechanical properties and elastic modulus (Bucuta & Ilie, 2014; Ilie, Bucuta & Draenert, 2013). However, a clinical modification was proposed to line the cavity with a flowable BF material and subsequently cover it with a packable composite (Ilie, Schöner, Bücher & Hickel, 2014). A recent three-year randomized controlled clinical study involving Class I and Class II preparation (van Dijken & Pallesen, 2014) demonstrated high clinical efficiency of a BF flowable composite (SDR™,DENTSPLY) applied in a 4 mm layer and subsequently covered with conventional RBC. In Campos et al., 2014 marginal adaptation of BF using the epoxy resin replicates of class II mesio-occlusal cavities restored in bulk using 4 mm increments compared to 2 mm conventional RBCs showed good potential for adequate marginal adaptation for BF composites at different tooth substrate levels.

1.3 Bulk Fill Resin Composites: Material Modifications

The translucency in resin composite is strongly influenced by filler size and the match between the filler-matrix resin refractive indices. The goal is always to minimize light scattering as more opaque material has a significant impact in the polymerization depth (Fujita et al., 2005; Fujita, Ikemi & Nishiyama, 2011; Park et al, 1999). The translucency of

BF composite has been increased to optimize polymerization reaction, and consequently it is DC. BF composites have some filler modifications (composition, size, shape and volume fraction) to accommodate minimal requirements for ideal polymerization. Larger filler size, lower volume filler fraction, and matched refractive indexes between the fillers and the matrix were strategies used by manufacturers. The rationale is to minimize the scattering of light with less filler- matrix interface (Shortall, Palin & Burtscher, 2008) and thus encourage more light transmission through the BF material (Azzopardi et al., 2009; Arikawa et al., 2007). Each manufacturer has developed strategies to produce BF restorative materials. Several commercially available BF materials have increased filler size (Sonic Fill, Kerr Dental; X-tra Bulk fill, VOCO) while others have added pre-polymerized fillers (Tetric EvoCeram, Ivoclar Vivadent), or decreased filler content (Filtek Bulk Fill, 3M/ESPE) (Bucuta & Ilie, 2014; Ilie, Bucuta & Draenert, 2013). In addition, other components of the resin composites have been modified, such as the incorporation of high molecular weight monomers and new alternative photoinitiators (Czasch & Ilie, 2012; Ilie, Bucuta & Draenert, 2013), which result in improved physical and optical properties, lower polymerization shrinkage, and convenient application (Leprince et al., 2014).

1.4 Photoinitiator Systems

Camphorquinone (CQ) photoinitiator in conjunction with tertiary aromatic amine has been largely used as the photoinitiator/co-initiator system in visible-light activated resin composites (Rueggeberg, 2011; Salgado et al., 2015), using a 470nm wavelength range as optimal for CQ (Price & Felix, 2009; Nomoto, 1997). It is well known that increasing the concentration of CQ can affect the aesthetics of RBCs due to its intense yellow color (Cramer, Stansbury & Bowman, 2011). Even though its undesirable yellowish effect is not an

issue with darker shades, and it can be slightly reduced after the photoactivation process (Price & Felix, 2009), the need for alternative photoinitiators became more evident with the advent and popularity of dental bleaching in dental practices, thus requiring restorative materials that would match with the final tooth color.

Alternatives to CQ have been developed to approximate colorless photoinitiators. Trimethyl-benzoyl-diphenylphosphine Oxide (TPO) and 1-phenyl1-1,2-propanedione (PPD) are examples of recently developed photoinitiators incorporated into resinous materials, especially for bleached and more translucent shades, thus minimizing the yellowish effect of CQ and enhancing the optical properties of cured materials (Santini et al., 2012). There are various photoinitiator systems that exhibit better reactivity and stability with light activation, with improved optical characteristics and DOC compared to CQ (Cramer, Stansbury & Bowman, 2011). An example is benzoylgermanium initiators, which do not need a co-initiator and their maximum peak of light absorption is 450nm (Moszner et al., 2008). Another alternative photoinitiator is PPD (1-phenyl- 1,2-propanedione), a phosphine oxide photoinitator which has no yellowish effect, does not need a co-initiator, and has above the minimal absorption peak of 420nm (Ogunyinka et al., 2007; Schneider et al, 2009). Additionally, PPD photoinitiator has higher polymerization coefficient compared to CQ, which leads to higher DC (Shin & Rawls, 2009). Another example of phosphine oxide derivatives is phenylbis Bis-alkyl Phosphine Oxide phosphine oxide (BAPO) (Meereis et al., 2014) and diphenyl(2,4,6-trimethylbenzoyl) phosphine oxide (TPO) (Mendonca et al., 2008), which also have less yellowing effect and better color stability than CQ (Salgado et al., 2015; Albuquerque et al., 2013). Furthermore, monoacylphos- phine oxide (Lucirin TPO) another photoinitiator which was originally incorporated with the dental adhesives formula

replacing CQ-amine initiator system (Ilie & Hickel 2007), along with Irgacure 819 (bisacylphosphine oxide), and OPPI (p-octyloxy- phenyl-phenyl iodonium hexafluoroantimonate) were also introduced as alternative photoinitiators (Shin & Rawls, 2009).

CQ can absorb light from 450nm to 490nm wavelength range, which matches exactly with the blue light spectra of most monowave (MW) light emitting diode (LED) light curing units (LCUs). However, MW-LED units do not cover the absorption peak of the alternative photoinitiators (ranging from 350nm to 460 nm) (Moszner et al., 2008, Santini, 2010). To overcome this issue, polywave (PW) LED units were designed to expand the wavelength ranges needed to cover both CQ photoinitiator and the alternatives added to RBCs (Santini et al., 2012; Ilie, Bucuta & Draenert, 2013; Jandt & Mills, 2013).

Nevertheless, some BF materials also have alternative photoinitiator systems in addition to the CQ. One example is Ivocerin (Bis-4 (methoxybenzoyl)diethylgermanium Ge-3), an alternative photoinitiator used to enhance the transmission of light, with absorption spectra between 370-460nm (Moszner et al., 2008) on Tetric EvoCeram Bulk Fill (TBF) in addition to CQ/amine system. The addition of alternative photoinitiators on BF composites has been reported as the main factor to the enhanced DOC. Apparently, filler modifications on these bulk materials play a minor role. Ilie and co-authors showed that the incorporation of alternative photoinitiator system in a BF material (TBF) improved the DOC compared to a control (nanohybride composite) with similar filler load (Ilie & Stark, 2014).

1.5 Light Curing Units

The introduction of alternative photoinitiators in resin composites identifies a concern related to whether broadband or dual spectrum LCUs is efficient. The aim is for an ideal

match between photoinitiators and the corresponding LCU, thus resulting in a satisfactory curing outcome (Uhl, Mills & Jandt, 2003, Tsai, Meyers, & Walsh, 2004). Nowadays, LED units are an efficient, fast and less expensive technology for photo polymerization of RBCs compared to previous Argon lasers, xenon plasma arc and quartz-tungsten-halogen (QTH) LCU (Rueggeberg, 2011; Tsai, Meyers, & Walsh, 2004). LED are semiconductor-based photonic devices based on the phenomenon called electroluminescence (Sze & Ng, 2006) which converts electrical energy into optical radiation. These devices are more efficient and produce more curing energy than QTH (Tsai, Meyers, & Walsh, 2004). They are very durable, portable, light in weight, consume less power, produce less heat and have long lifespan (Stahl et al., 2000). They emit blue light in the spectral region between 450-470nm (Jandt & Mills, 2013), which exactly matches the absorption spectra of CQ photoinitiators (Nomoto,1997). Current MW or single peak LED units use narrow emitters and increased energy output to provide a more focused emission of blue light, required to match the ideal absorption peak for CQ photoinitiators (Jandt & Mills, 2013; Santini et al., 2012, Ogunyinka et al., 2007). However, single peak or MW units cannot adequately activate alternative photoinitiators with a different absorption spectra range (390-410nm). The resulting mismatch between the irradiation wavelength of MW units and the alternative photoinitiator absorption peak causes insufficient resin composite curing (Miletic et al., 2012; Rueggeberg, 2011; Jandt & Mills, 2013; Santini et al., 2012; Lucey, Santini, & Roebuck, 2015; Cramer, Stansbury & Bowman, 2011).

To overcome this issue, LED units have been through several changes in an effort to support the incorporation of alternative photoinitiators. This has been achieved by increasing the range of spectral output and the emitter elements (semiconductor chips) of these units

(Cao, 2005; Rueggeberg, 2011; Uhl, Mills & Jandt, 2003). As a result, a dual peak or PW LED unit was developed and utilizes two or more semiconductor chips to cover both short and long wave lengths (390-410 nm in the violet spectrum, and 460-470 nm in blue light spectrum) (Rueggeberg; 2011, Cao, 2005). The double light spectra enable activation of both CQ and the alternative photoinitiator system.

Newer PW LED units such as Bluephase G2 (Ivoclar Vivadent), UltraLume 5 and VALO (Ultradent products inc.) effectively increased the DOC of new resin dental materials with additional irradiance value (Jandt & Mills, 2013; Rencz, Hickel & Ilie, 2012). As a result, both DC and microhardness of these modified RBCs has improved (Santini et al., 2012). Interestingly, not all CQ containing materials cured efficiently with PW LEDs. The result of insufficient curing and lower DC can lead to toxic release of uncured composite monomers (Sigusch et al.,2009; Aranha et al., 2010; Durner et al., 2012). Lucy et al., 2015 highlighted the importance of using the proper light source for the selected material for successful restorative outcomes. They investigated the presence of new photoinitators in combination with CQ for different types of fissure sealants cured by PW and MW LED units. The TPO-containing sealants (Vit-l-escence®) were properly cured with PW LED units. However, the non-TPO sealants were properly cured with MW LEDs yet showed lower conversion when PW LEDs used.

The irradiance energy delivered by a LCU is another factor to be considered. The distance between LCU tip and restoration coupled with exposure time, composite shade, layer thickness and finally on the chemical structure of the material were considered important factors in regards to RBC polymerization (Knezˇevic et al., 2001; Tsai, Meyers, & Walsh, 2004). However, it is currently accepted that DC is a crucial factor in analyzing

composite polymerization, which is dependent on filler size, geometry and filler load along with the monomer composition and presence of photointiators (Alshali, Silikas & Satterthwaite, 2013). Two studies have investigated curing times and polymerization of BF composites. Their findings demonstrated that 20 to 30 seconds of curing time to polymerize the material in 4mm increments can develop clinically acceptable properties in BF materials (Ilie & Stark, 2014; Zorzin et al., 2015).

1.6 Polymerization Shrinkage and Application Technique

An inherent characteristic of a resinous material is shrinkage upon polymerization and stress generation at the bonded interfaces (Lu et al., 2004). These stresses can cause cuspal deflection, micro-cracks at the enamel margins, with the potential for bond failure at the interface (Kim & Park, 2011; Lu et al., 2004; Hannig & Friedrichs, 2000).

Several attempts have been made to reduce or eliminate polymerization shrinkage in resin composites, including modifications in the organic matrix and/or in the inorganic fillers (Condon & Ferracane, 2000; Leprince et al., 2012). The silorane-based composites, which contain siloxane core technology to offset the shrinkage resulted from monomer-polymer chemical interaction during polymerization process are an example of this type of modification. Polymerization occurs via a cationic ring-opening reaction resulting in lower shrinkage compared to the methacrylate-based resins (Ilie & Hickel, 2006).

The deleterious effect of shrinkage, especially in high C-factor cavities, is well-known to impair the durability of the restoration and cause undesirable consequences such as post-operative sensitivity (Briso et al., 2007), marginal staining, recurrent caries (Lutzet al, 1991; Schneider et al., 2010; Lu et al., 2004). These effects often lead to financial burden and patient dissatisfaction, where additional cost for replacement of composite restoration is

required and more tooth structure need to be removed for proper bonding. Thus, some techniques have been adopted to reduce the effects of shrinkage stress such as placement of low modulus flowable RBC (Alomari, Reinhardt & Boyera, 2001; Kwon, Ferracane & Lee, 2012; Radhika et al., 2010) or glass ionomer cement liners (Puckett et al., 1991) along with an incremental layering technique (Versluis et al.,1996; Poskus, Placido & Cardoso, 2004; Park et al., 2008). These techniques were proposed to overcome the effects of composite shrinkage and to assure an acceptable polymerization of RBCs. Optimal polymerization is desired for biocompatibility and durability of RBC restorations (Van Landuyt et al, 2011; Versluis et al.,1996; Kovarik & Ergle,1993; Poskus, Placido & Cardoso, 2004; Sigusch et al.,2009; Aranha et al., 2010; Durner et al., 2012). As the incremental layering technique can minimize cuspal deflection, reduce interfacial stresses (Dietschi et al., 2002) and gap formation (Alomari, Reinhardt & Boyera, 2001; Furness et al., 2014), thereby providing positive impact on the clinical performance of the restoration (Poskus, Placido & Cardoso, 2004; Owens & Johnson, 2005). Even though there are several benefits attributed to the incremental layering technique, it is indeed a time consuming clinical routine.

The BF restorative technique was previously investigated with conventional RBCs (Stavridakis et al., 2007; Puckett et al.,1991; Kwon, 2012; Abbas, 2003; Idriss et al., 2003; Winkler, 1996), however the challenge was to adequately cure the material in increments greater than 2 mm, especially for more opaque composites. More recently, additional modifications in the RBCs intended for posterior restoration created what has been called "bulk fill" material. These composite materials are meant to be applied in up to 4-5mm increments (Bucuta & Ilie, 2014). Theoretically, they propose to have overcome the limitations of conventional RBCs, suggesting adequate polymerization can be achieved in

increments thicker than 2mm. Additionally, they offer a less time-consuming technique and more convenient clinical application, thus being of great value for both, patient and clinician.

1.7 Polymerization and Laboratorial Tests

The depth of cure (DOC) is an alternative, indirect measurement for hardness after polymerization. It has been described as an indirect measurement that can estimate the DC (Moore et al.,2008; Alrahlah, Silikas & Watts, 2014; Flury et al, 2012), routinely measured by Raman and FTIR spectroscopy. There have been several methods implemented to measure the DOC of RBCs. One approach uses optical microscopy to show the quality of curing between cured and uncured parts of a material (DeWald & Ferracane,1987). Vickers (VHN) and Knoop indentations, (Rueggeberg et al., 2000; Flury et al., 2014; Bouschlicher & Rueggeberg, 2004) and ultra-microhardness methods are also implemented (Antunes et al., 2002). A simplified method to determine the DOC is the scraping test (ISO 4049). The physical length of the cured composite is measured after scraping away the uncured part immediately after curing, and then dividing the length by 2 to obtain the DOC. The final measured length should not be less than 0.5 mm from the depth claimed by manufacturer (Moore et al., 2008; Tsai, Meyers & Walsh, 2004). The accuracy of optical microscopy and the scraping method has been questioned due to a potential overestimation of hardness values (Flury et al., 2012).

According to Leprince et al., 2012, microhardness, scrape and DC methods used did not provide the accuracy needed to reflect the DOC at the material transition from rubbery to glassy state. They suggested other techniques such as atomic force microscopy (AFM), electron paramagnetic resonance (EPR) and differential scanning calorimetry (DSC). They found those proposed methods provided more in depth information of elastic modulus and its

relation to DC, cross-linking density and monomer leaching.

BF composites have been designed to improve the depth of light penetration into the resinous materials, thus enhancing DC and DOC. Those changes are mostly in the fillers and photoinitiators as discussed above. However, the impact of those changes under different light curing conditions is not currently clear. There is limited research investigating the impact of MW LED units on the performance of BF restorative materials (Lucy et al., 2015). Therefore, the interaction between the LCUs and BF composites remains an important area of research. A recent study by Menees et al., 2015 evaluated the DOC of BF composites cured by MW and PW LED units. They used simulated 4x4 Class II cavities on permanent molars and custom fabricated molds for IOS 4049 method. They found no significant difference in DOC of BF composites, using MW or PW curing modes. Other previous studies (Santini et al., 2012; Miletic & Santini, 2012) investigated different RBCs properties, such as DC and hardness, on those materials having TPO and/ or other initiators in their formulation, cured by PW or MW units. Their results found significant difference between LCUs; with PW adequately curing composites containing TPO, similar to what was observed by Lucy et al., 2015 on pit and fissure sealants. Some studies have investigated shear bond strengths (SBS) of BF composites in primary and permanent molars (Bucher et al, 2014. Ilie et al,2014) with results showing comparable SBS values for BF and conventional RBCs.

There is high demand for the use of esthetic RBCs restorations among pediatric population. Many parents opt for the use of un-esthetic amalgam restorations on their children's teeth as they are less expensive and more likely covered by dental insurances. Although there is no evidence for harmful effects of amalgam restoration, many parents are not comfortable with the use of these fillings on their children's teeth and prefer tooth-

colored restorations. As mentioned before, childe cooperation is important for success using bonded restorations due to an increased number of technique- sensitive steps. When application protocol and case selection are met, composite restorations show good longevity on primary teeth (Bücher et al., 2014). All previous investigations on the mechanical properties for BF composite are promising and it is important to investigate the curing depth and the quality of bonding to the cavity floor in order to insure fast and efficient bulk restoration for primary and permanent teeth.

1.8 Study Rationale and Objectives

Even though several independent studies have validated the DOC for BF composites (Alshali, Silikas & Satterthwaite; 2013; Zorzin et al., 2015; Leprince et al., 2012; Salgado et al., 2015; Alrahlah, Silikas, Watts, 2014; Flury, 2012; Lucy et al., 2015; Santini et al., 2012; Miletic & Santini, 2012), none have explored the impact of different LED curing units and curing times on the resin-dentin bond strengths with restorative BF resin composites, and the DOC of those BF composites under similar curing protocols. Thus, the objectives of this study were: 1) to evaluate the effect of PW and MW LED curing units, using two different curing times, on the micro-tensile dentin bond strength (μTBS) of restorative BF composites to dentin; 2) to evaluate the DOC of restorative BF composites cured by PW and MW LEDs, in two different curing times.

1.9 Null hypothesis

The null hypotheses of this study are:

N_0 1: There is no difference in the μTBS values of 4mm restorative BF composites cured for 10 or 20 seconds using MW or PW LED curing units.

N_0 2: There is no difference in DOC of restorative BF composites cured for 10 or 20 seconds using MW or PW LED curing units.

N_0 3: There is no difference in the scrape test values before and after immersion in Methyl Ethyl Ketone (MEK).

Chapter 2: Materials and Methods

2.1 Materials

Two commercially available restorative BF composites, Tetric EvoCeram bulk fill restorative (TBF) (Ivoclar Vivadent) and Filtek bulk fill restorative (FBF) (3M/ESPE), and one conventional resin composite, Tetric EvoCeram (T) (Ivoclar Vivadent) as the control were used in this study. The dentin bonding procedure was performed by using the etch-and-rinse technique by the application of 35% phosphoric acid etching followed by the application of a universal dental adhesive AdheSE® (Ivoclar Vivadent) on a flat dentin surface. The materials' classification, composition, manufacturers and lot numbers are listed in Table 2.1.

2.2 Microtensile Bond Strength (μTBS)

Sample size calculations from a pilot study resulted in a minimum sample size of 6 per group to detect a statistically significant difference in bond strengths to dentin, with 80% power and 95% confidence level (SigmaPlot 13, Systat Software Inc.). The study used sound extracted third molars, which were collected and stored in 0.1 % thymol solution at 4°C.

2.3 Tooth collection and preparation

Seventy-two sound human third molars were randomly assigned to twelve groups, with 6 teeth in each group. The teeth were wet polished with #180 SiC abrasive paper in a polishing machine (Precision Lapping/Polishing machine, UNIPOL-1210) to expose a flat dentin surface, parallel to the cemento-enamel junction. The prepared dentin surfaces were checked under light microscope to ensure no enamel was left on the flat surface. Immediately prior to bonding, a standardized smear layer was manually created using a 320-grit extra-fine finishing paper (NORTON) for 10 seconds, under water lubrication.

2.4 Bonding procedures

The prepared teeth were stabilized with wax on a dappen dish prior to the bonding step. The surfaces were etched with 35% phosphoric acid for 15 seconds and then rinsed with water for 15 seconds. The demineralized dentin surface was blot dried with filter paper to achieve uniform surface moisture. A multimode adhesive, AdheSE® (Ivoclar Vivadent) was actively applied with rubbing action for 20 seconds using a microbrush applicator. Solvent evaporation was promoted by air circulation from an approximate distance of 20 cm for 10 seconds. Subsequently, the adhesive was light cured for 10 seconds with the selected light-curing unit at ≅ 960 mW/cm^2. The light output was checked daily with their corresponding radiometers, (Satelec Miniled Supercharged (ACTEOM) radiometer), (Bluephase Meter II radiometer, Ivoclar Vivadent) for MW and PW LCUs respectively. Total energy for 10 and 20 seconds were 9.6 J/cm^2 and 19.2 J/cm^2, respectively (Beolchi et al., 2015).

2.5 Composite build ups

Each of the resin composites tested (TBF, FBF and T) was cured in 4mm increments under four different curing protocols as follows: MW LED unit (Satelec Miniled Supercharged (ACTEOM) with either 10 or 20 seconds curing time; or PW LED unit (LED Bluephase Style (Ivoclar Vivadent) with either 10 or 20 seconds curing time. In order to standardize the height (4 mm) and diameter (7.5 mm) of the composite build-ups and to avoid light exposure on the periphery of the build ups, thus simulating a more realistic clinical scenario, matrices were created with impression material, polyvinyl siloxane (PVS) (Aquasil Ultra LV/XLV Smart Wetting® Regular Set, Dentsply) to hold the bulk of the resin composite applied. The matrix diameter corresponded with the LCU tips for both MW and PW curing units, at 7.5 mm. The elastomeric matrix was manually held on the top of the

bonded dentinal surface while a single increment of the selected BF composite was carefully applied. Each specimen was then cured for the assigned time and LED LCU as described previously. The specimen was immediately stored in distilled water and kept at 37°C for 24 hours (Figure2.1). The specimens were then sliced into beams of approximately 0.9 mm^2 using a slow speed diamond saw at 300rpm (SYJ-150 slow speed diamond saw, MTI corporation) and immediately submitted to mechanical testing. No premature failure occurred during slicing of the beams in preparation to μTBS except for 2 beams out of the 72 sliced teeth.

2.6 Microtensile bond strength test (μTBS)

Each beam was individually stabilized with cyanoacrylate glue (LEPAGE super glue-gel control, Henkel Corporation) on metallic slabs for μTBS (stainless steel OG01; Odeme Dental Research, Brasile), mounted in a Universal Testing Machine (SHIMADZU AutoGraph, AGS-X) and tested in tension at crosshead speed of 0.5mm/min. The force at failure was measured in Newton (N) and then converted to Megapascal (MPa) to determine the bond strength. The force (Kgf) recorded at failure was divided by the corresponding sample surface area (cm^2) and multiplied by 0.0981 to give the final bond strength value in MPa.

After data collection, the mean and standard deviation of each group was described statistically. The bond strength values in MPa were statistically analyzed (SigmaPlot 13.5, Systat Software Inc.) using two-way ANOVA test for each LCU independently. In addition, two-way ANOVA analyses were performed for each resin composite independently, as curing time and LCU as factors.

2.7 Failure mode analysis

After testing, each beam was evaluated under optical microscopy (SKU:HL150-AR) with 20x magnification to determine the mode of failure (cohesive, adhesive or mixed). The composite side and the dentin sides were examined from both vertical and horizontal perspectives and matched with the corresponding fractured piece. Adhesive failure was defined as adhesive remaining attached to the dentin side or to the composite side or on both sides, but including only the dental adhesive material. Cohesive failure in composite was observed when the fractured occurred within the composite build up only; while cohesive failure in dentin was determined when the fracture occurred within the dentin. A mixed failure was identified when at least two failure modes occurred in the same fractured surface. The percentages of the failure modes were calculated.

Random selection of beams was completed to view failure mode under SEM microscopy (Hitachi SU3500). The selected samples were representative of each failure mode. The specimens (dentin side and composite side) were coated with iridium to enable them more conductive for electrons and were stabilized on stubs for SEM analysis. The magnification ranged from X200 to X500 and varied according to the area of interest (Figure3.2).

2.8 Scrape Test

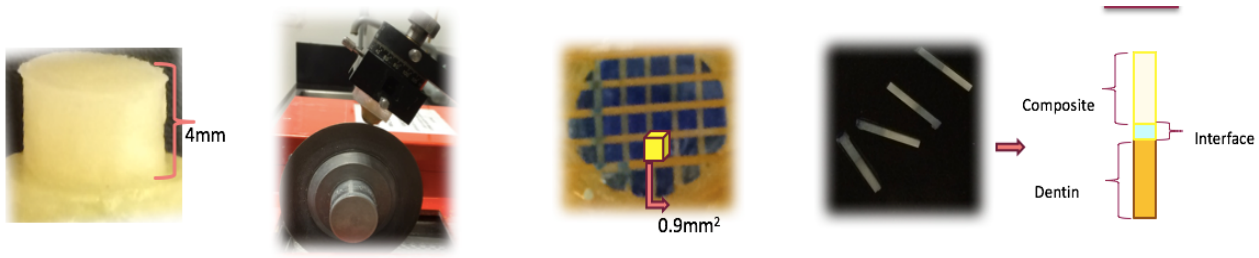
Individual unit-dose capsules of each resin composite were used as molds for the scrape test described by ISO 4049 specification. The capsule's nozzle end was cut off using a double-sided diamond disc (#8861-7CC, Galaxy^{TM,} manufacturer) to obtain a perfectly parallel dark cylinder. The composite inside each capsule was condensed with an instrument against a Mylar strip on a glass slab to achieve a flat surface, then subsequently cured from

this side using the same curing protocols described previously for the μTBS test. The cured resin composite cylinder was then ejected using composite dispensing gun (Septodont) and the uncured material immediately scrapped off using a metallic instrument (756 Premium Instrument, AISI 420, Germany). The length of the remaining resin composite material left after the scraping procedure was measured with digital micrometer and divided by two, as described in ISO 4049, to determine the DOC in millimeters. The resin composite cylinders were immediately stored in a dry, dark container at room temperature prior to immersion in MEK.

An additional analysis of the resin composite samples used for the scrape tests was performed for all groups to assess the presence of poorly polymerized and soft resin composites on the cured samples, as described in previous studies (Leprince et al, 2013; Al Sunbul 2015; Bonfante et al, 2008; Price et al, 2015). Each pre-measured and cured resin composite cylinder was immersed and sonicated (Gyromax 838, Mandel, Amerex Instruments, Inc.) in a 5 ml volume of a solvent, (MEK) (SigmaAldrich) for a period 2 hours.

To determine the ideal immersion time in MEK, a pilot test was performed based on time and sample dimension used in previous studies with similar methodology (Leprince et al,2013; Al Sunbul 2015; Bonfante et al, 2008; Price et al, 2015). After completing the process, the resin composite cylinders were dried and the additional poorly cured resin composite that was softened by the solvent was scrapped off with the spatula again. The final lengths were measured and divided by two for the additional post-MEK immersion DOC data collection. The statistical analysis was performed independently on the pre- and post-MEK immersion DOC data using 2-way ANOVA. An additional statistical analysis, the Paired t-test, was utilized to compare the DOC (in mm) before and after MEK immersion.

Figure 2.1: Microtensile testing

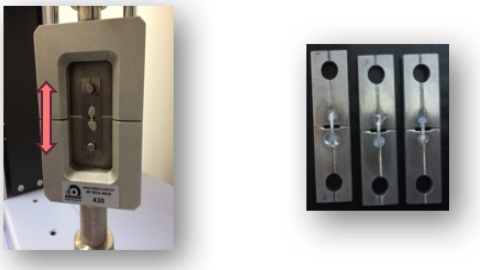


A. 4 mm resin bonded dentin surface.

B. Sectioning after 24 hours storage.

C. Sectioned beams

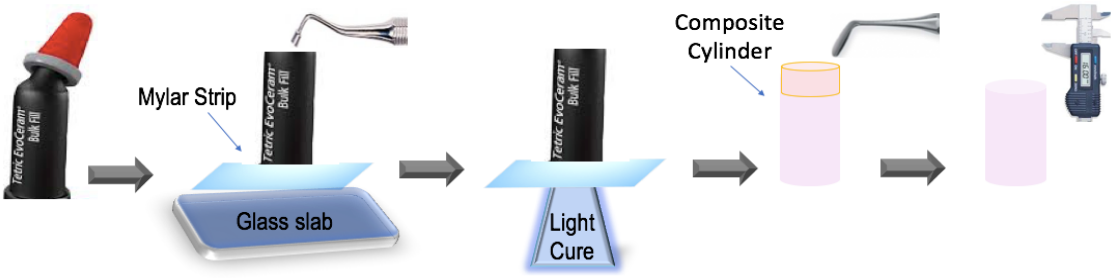
D. Individual sectioned beams



F. Microtensile testing

E. Stabilized beams

Figure 2.2: Graphic illustration for scrape test method.



Cut

Condense

Cure PW/MW

Scrape un-cured composite

Measure length (mm)

Table 2.1: Materials, manufactures, composition and lot numbers

Material	Manufacturer	Composition	Lot numbers
EvoCeram bulk fill restorative (TBF)	Ivoclar Vivadent, Shade IVA	Bis-GMA 5-10% UDMA 5-<10% Ethyoxylated bisphenol A dimethacrylate Photoinitiator: Ivocerin, CQ, Lucerin TPO. Fillers (80% Wt):Ytterbium trifluoride 3-5%, Barium aluminum silicate glass with two different mean particle sizes, an Isofiller, spherical mixed oxide	T327776, T39247, R03284 R08234
Filtek bulk fill restorative (TBF)	(3M/ESPE), Shade A1	Aromatic Urethan Dimethacrylate UDMA Dodecane Dimethycrylate Imethycrylate (DDDMA) Dimethyl Aminobenzoate (EDMAB), Modified methacrylate monomer Fillers (76.5% Wt): Ytterbium fluoride (YbF3), Silane treated silica (trade secret), Silane treated Zirconia Photoinitiators: not disclosed.	N642401, N669295. 4864A1
Tetric EvoCeram(T)	Ivoclar Vivadent, Shade A1	Ethoxylated Bis A Dimethacrylate 10 - 30 % Bis-GMA,UDMA, Triethylene glycol dimethacrylate 5 – 10% Fillers (48.5% Wt): Barium glass filler, Ytterbium trifluoride, mixed oxide, pre-polymers 34 % Wt. silica, fumed 5 - 10 % Photoinitiator: CQ.	T32772.
Adhese Universal	Ivoclar Vivadent	2-hydroxyethyl methacrylate 20-<25%. Bis-GMA 20-<25% ethanol 10-13% 1,10-decandiol dimethacrylate 5-<10% Methacrylated phosphoric acid ester 3-7% CQ 1-3% 2-dimethylaminoethyl methacrylate <1%	T33767
SELECT HV ETCH	BISCO	High viscosity 35% Phosphoric acid etchant.	E-5906

Chapter 3: Results

3.1 Microtensile Bond Strength Test (MPa):

The μTBS results for the samples cured with the PW LCU (Table 3.1) demonstrated that there was a statistically significant lower bond strength value for TBF cured for 20 seconds in comparison to those cured for 10 seconds, but no differences were observed for the other composites, FBF and T in regards to curing time. In contrast, for the μTBS values of samples cured using the MW LCU (Table 3.2) resulted in no statistically significant differences with different curing times for all resins tested.

Table 3.1: Microtensile bond strengths of resin composites (4mm increments) to dentin cured with polywave light curing unit. Data are presented in MPa (SD).

Resin composite	Curing times	
	10 seconds	20 seconds
Tetric EvoCeram Bulk Fill (TBF)	54.8 (13.3) ^{A,a} n=75	46.2 (16.6) ^{B,b} n=75
Filtek Bulk Fill (FBF)	46.75 (22.6) ^{A,b} n=78	49.6 (18.01) ^{A,b} n=86
Tetric EvoCeram (T)	54.0 (12.3) ^{A,a} n=71	55.8 (12.4) ^{A,a} n=76

Superscript letters represent statistical differences. Capital letters compare curing times and lower-case letters compare resin composites.

Table 3.2: Microtensile bond strengths of resin composites (4mm increments) to dentin cured with Monowave light curing unit. Data are presented in MPa (SD).

Resin Composite	Curing times	
	10 seconds	**20 seconds**
Tetric EvoCeram Bulk Fill (TBF)	47.91 (15.1) ^{A,a} n=75	51.1 (16.0) ^{A,a} n=75
Filtek Bulk Fill (FBF)	51.3 (22.2) ^{A,a} n=82	51.7 (19.9) ^{A,a} n=76
Tetric EvoCeram (T)	51.3 (8.5) ^{A,a} n=76	50.5 (12.1) ^{A,a} n=78

Superscript letters represent statistical differences. Capital letters compare curing times and lower-case letters compare resin composites.

Table 3.3: Microtensile bond strength values of Tetric EvoCeram Bulk Fill composite bonded to dentin. Data are presented in MPa, mean (SD).

LCU / Time	Polywave	Monowave
10 seconds	54.8 (13.3) ^{A,a} n=75	47.91 (15.1) ^{B,a} n=75
20 seconds	46.2 (16.6) ^{A,b} n=75	51.1 (16.0) ^{A,a} n=75

Superscript letters represent statistical differences. Capital letters compare light curing units and lower-case letters compare curing times.

Table 3.4: Microtensile bond strength values of Filtek Bulk Fill composite bonded to dentin. Data are represented in MPa (SD).

LCU / Time	Polywave	Monowave
10 seconds	^{A,a} 46.75 (22.6) n=78	^{A,a} 51.3 (22.2) n=82
20 seconds	^{A,a} 49.6 (18.01) n=86	^{A,a} 51.7 (19.9) n=76

Superscript letters represent statistical differences. Capital letters compare light curing units and lower-case letters compare curing times.

Table 3.5: Microtensile bond strength values of Tetric EvoCeram composite bonded to dentin. Data are presented in MPa (SD).

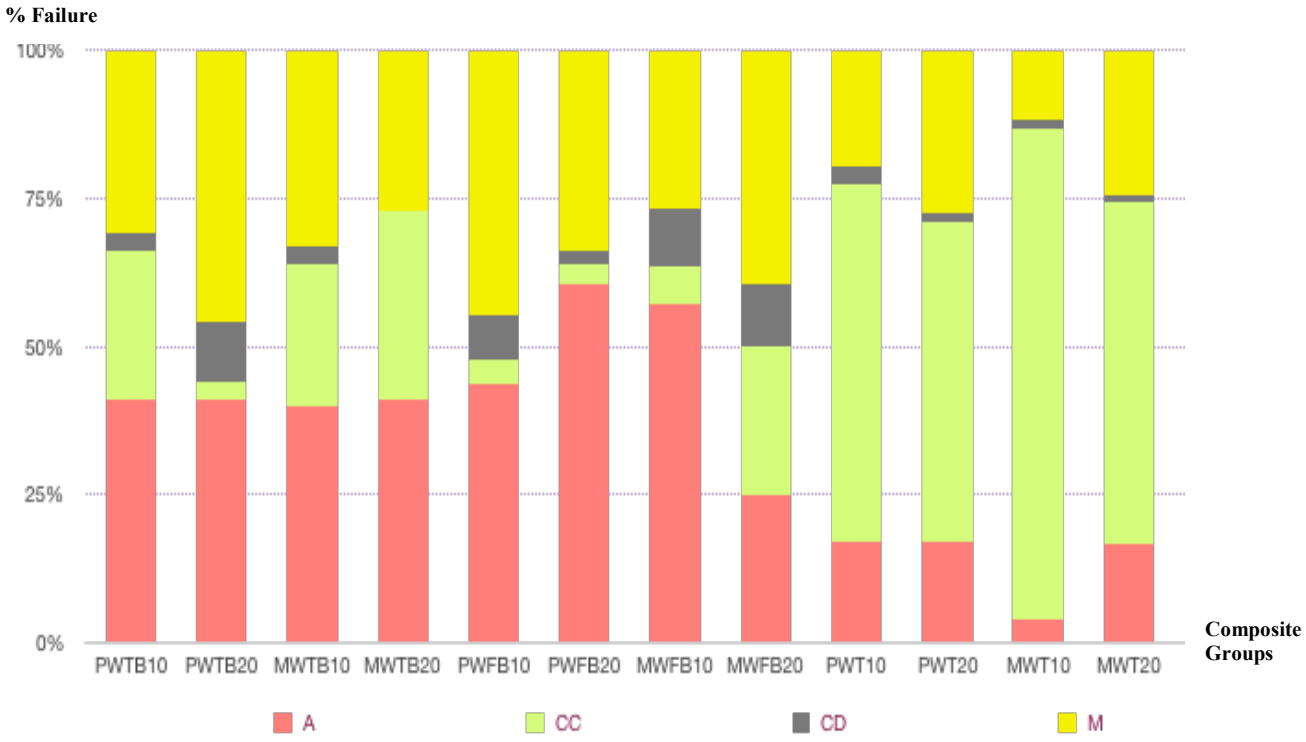
LCU / Time	Polywave	Monowave
10 seconds	^{A,a} 54.0 (12.3) n=71	^{B,a} 51.3 (8.5) n=76
20 seconds	^{A,a} 55.8 (12.4) n=76	^{B,a} 50.5 (12.1) n=78

Superscript letters represent statistical differences. Capital letters compare light curing units and lower-case letters compare curing times.

3.2 Analysis of Failure Modes

The overall failure mode analysis (Figure 3.1) presented high percentage of adhesive and mixed failures for both BF composites tested, regardless of the curing protocol applied. However, for the conventional RBC (control), a higher percentage of cohesive failures in composite were observed (50 to 75% range).

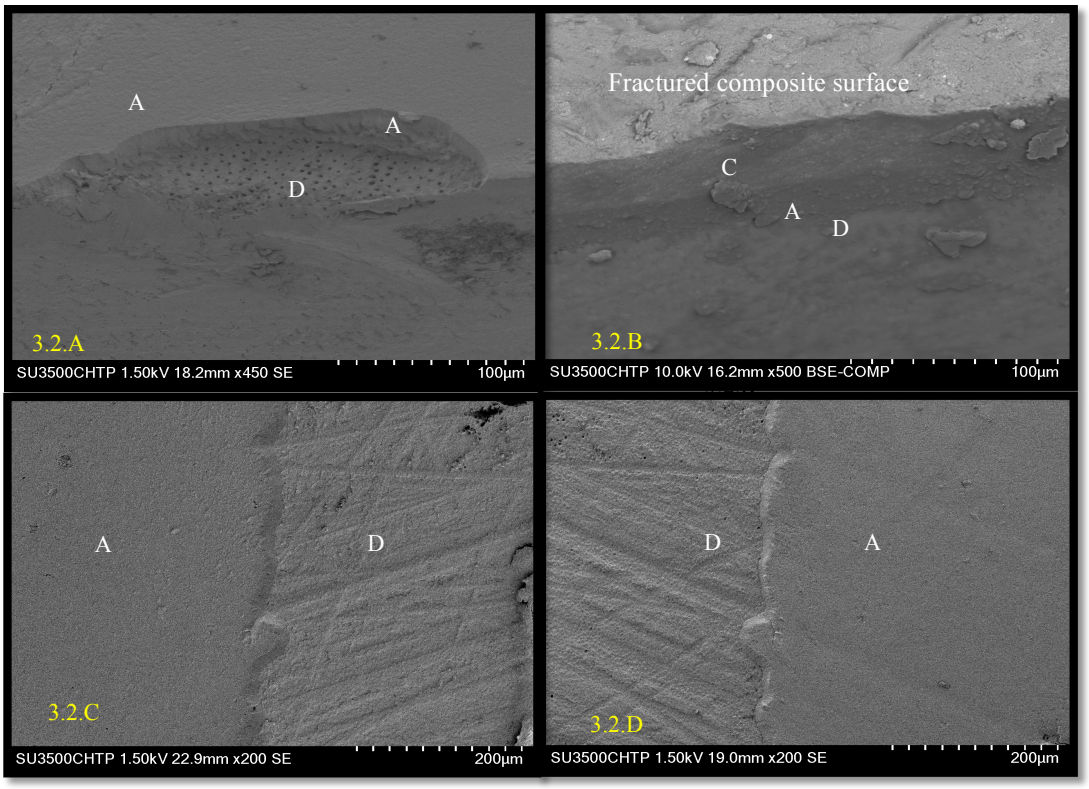
Figure 3.1: Failure modes in percentage for each experimental group.



A: Adhesive failure; **CC**: cohesive in composite failure; **CD**: cohesive in dentin failure; **M**: Mixed failure.

The scanning electron microscopy (SEM) images, representative of each failure mode, are presented in Figure 3.2. A mixed failure involving two failure modes combined (adhesive and cohesive) can be seen in Figure 3.2.A. A common pattern of cohesive failure in composite close to the adhesive interface was often observed (Figure 3.2.B). Adhesive failures were described when the fracture occurred in a single plane or in multiplanes involving only the adhesive and the adjacent bonded substrate, either dentin or composite as seen in Figures 3.2.C and 3.2.D.

Figure 3.2:SEM images of representative samples of each failure mode.



C: Composite; D: Dentin; A: Adhesive
3.2.A: Mixed failure (M) seen in MW TBF cured for 20 seconds. It shows (A) and (D) surfaces involved in the same fracture.
3.2. B: Cohesive failure in composite (CC) seen in MW T cured for10 seconds. The image clearly shows the three distinct substrates: (C), (A) and (D).
3.2.C and 3.2. D: Mirror images showing a two-level fracture along the (A) layer seen in PW FBF cured for 20 seconds, partially de-bonding from the composite and partially from dentin.

3.3 Scrape Tests

For the pre-MEK immersion scrape test (Tables 3.6 and 3.7), the results showed a significantly greater (DOC) for all resin composites cured for 20 seconds with either MW or PW LCUs. In addition, for all curing protocols applied, BF composites presented statistically higher DOC values than the conventional RBC (control). Even though there was no statistically significant difference detected among the two BF composites tested, it was observed for PW curing that TBF presented a higher DOC value for both curing times; while in MW, FBF cured at 10 seconds showed the highest DOC value.

For the post-MEK immersion scrape test (Tables 3.6 and 3.8), the results for PW curing unit were similar to what was observed in the pre-MEK test, with significantly lower DOC for reduced curing time (10 seconds) for all composites; and significantly higher DOC for BF composites in both curing times. However, for MW curing unit, no statistically significant difference was observed when comparing resin composites for both curing times. There was a statistically significant difference with increasing curing time (10 seconds vs. 20 seconds) for all materials tested.

Additionally, the paired t-test results for each composite independently (Table 3.10) showed a significant difference between the DOC pre-MEK versus DOC post-MEK for all composites (TBF, FBF and T) and all curing protocols (MW and PW; 10s and 20s).

Table 3.3: Scrape test results of resin composites (n=5) cured with polywave light curing unit (≅960mW/cm2) pre-MEK immersion. Data are presented in mm (SD).

Curing Time / Resin Composite	10 seconds	20 seconds
Tetric EvoCeram Bulk Fill (TBF)	3.02 (0.075) ^{B,a}	3.63 (0.18) ^{A,a}
Filtek Bulk Fill (FBF)	2.78 (0.12) ^{B,a}	3.53 (0.25) ^{A,a}
Tetric EvoCeram (T)	2.25 (0.22) ^{B,b}	2.74 (0.11) ^{A,b}

Superscript letters represent statistical differences. Capital letters compare curing times and lower-case letters compare resin composites

Table 3.4: Scrape test results of resin composites (n=5) cured with polywave light curing unit (≅960mW/cm2) post-MEK immersion. Data are presented in mm (SD).

Curing Time / Resin Composite	10 seconds	20 seconds
Tetric EvoCeram Bulk Fill (TBF)	2.63 (0.19) ^{B,a}	3.22 (0.17) ^{A,a}
Filtek Bulk Fill (FBF)	2.55 (0.21) ^{B,a}	3.21 (0.19) ^{A,a}
Tetric EvoCeram (T)	2.00 (0.2) ^{B,b}	2.38 (0.14) ^{A,b}

Superscript letters represent statistical differences. Capital letters compare curing times and lower-case letters compare resin composites

Table 3.5: Scrape test results of resin composites(n=5) cured with monowave light curing unit (≅960mW/cm2) Pre-MEK immersion. Data are represented in mm (SD).

Curing Time / Resin Composite	10 seconds	20 seconds
Tetric EvoCeram Bulk Fill (TBF)	2.84 (0.19) ^{B,a}	3.54 (0.06) ^{A,a}
Filtek Bulk Fill (FBF)	2.88 (0.05) ^{B,a}	3.44 (0.21) ^{A,a}
Tetric EvoCeram (T)	2.33 (0.08) ^{B,b}	2.72 (0.08) ^{A,b}

Superscript letters represent statistical differences. Capital letters compare curing times and lower-case letters compare resin composites

Table 3.6: Scrape test results of resin composites (n=5) cured with monowave light curing unit (≅960mW/cm2) post-MEK immersion. Data are represented in mm(SD).

Curing Time / Resin Composite	10 seconds	20 seconds
Tetric EvoCeram Bulk Fill (TBF)	2.45 (0.2) ^{B,b}	3.01 (0.07) ^{A,a}
Filtek Bulk Fill (FBF)	2.67 (0.02) ^{B,b}	3.17 (0.198) ^{A,a}
Tetric EvoCeram (T)	1.98 (0.04) ^{B,b}	2.36 (0.05) ^{A,a}

Superscript letters represent statistical differences. Capital letters compare curing times and lower-case letters compare resin composites

Table 3.7: Scrape test results of each resin composite (n=5) analyzed independently (Paired t-test) pre- and post-MEK immersion. Data are represented in mm (SD).
P= <0.05

LCU	Curing Time	TBF (t-test)		FBF (t-test)		Tetric (t-test)	
		Before MEK immersion	After MEK immersion	Before MEK immersion	After MEK immersion	Before MEK immersion	After MEK immersion
PW	10s	3.02 (0.075)[a]	2.63 (0.19)[b]	2.78 (0.12)[a]	2.55 (0.21)[b]	2.25 (0.22)[a]	2.00 (0.2)[b]
	20s	3.63 (0.18)[a]	3.22 (0.17)[b]	3.53 (0.25)[a]	3.21 (0.19)[b]	2.74 (0.11)[a]	2.38 (0.14)[b]
MW	10s	2.84 (0.19)[a]	2.45 (0.2)[b]	2.88 (0.05)[a]	2.67 (0.02)[b]	2.33 (0.08)[a]	1.98 (0.04)[b]
	20s	3.54 (0.06)[a]	3.01 (0.07)[b]	3.44 (0.21)[a]	3.17 (0.198)[b]	2.72 (0.08)[a]	2.36 (0.05)[b]

Superscript letters represent statistical differences.

Chapter 4: Discussion

BF composites were formulated to simplify the clinical application of direct posterior resin composite restorations. Their composition however differs from conventional RBCs in some aspects, aiming to increase the DOC. The addition of alternative photoinitiators and the increased translucency of the resin are among the modifications made for BF composites to ensure the extended DOC. The BF materials have been constantly studied since they were launched, mostly in regards to mechanical properties such as hardness, DOC, DC and SBS (Alrahlah, Silikas, Watts, 2014, Zorzin et al., 2015, Santini et al; 2012, Salgado et al., 2015, Czasch & Ilie N, 2012, Ilie et al., 2014; Alshali, Silikas & Satterthwaite; 2013).

4.1 Microtensile bond strength and failure mode

This study explored the effect of curing modes of BF composites on the μTBS to dentin. The μTBS test is a useful tool to evaluate bonding performance of adhesive materials to different substrates (Armstrong et al., 2010; Pashley et al., 1999). It was utilized to assess the impact of curing protocols on composite bond strength to dentin in the present study.

The first null hypothesis of the study was rejected as statistically lower bond strength values were observed for TBF, cured for 20 seconds with PW LCU (Table 3.1). The original selection of curing times, 10 and 20 seconds, was based on both manufacturer's recommendations for their BF restorative materials, Ivoclar and 3M/ESPE, respectively. Since those curing times are the most commonly recommended for BF materials available in the market, the intent was to test both curing intervals under similar energy output ($\cong$960mW/cm^2) for each curing unit evaluated. However, the results found were intriguing, especially in regard to the low bond strength values of TBF cured with PW LCU for 20 seconds. This particular BF composite/ LCU combination corresponds exactly to what is

recommended by the manufacturer, (dental adhesive and curing unit from the same manufacturer) with extended curing time (20 seconds). Therefore, one would expect higher bond strength values for TBF cured with PW curing unit at 10 or 20 seconds due to the full compatibility of adhesive-composite-curing unit. However, TBF composite cured for 20 seconds presented the lowest bond strength among all tested groups for PW LCU. We can speculate that the lowest bond strength values for this particular group (TBF cured for 20 seconds) could be a result of a higher modulus of elasticity of a well-cured resin composite (double the recommended curing time), which could have resulted in increased interfacial stresses and significantly lower bond strengths as the consequence. As described in the literature, multiple factors can affect the stress distribution at the bonded interface during tensile tests (Armstrong et al., 2010; Pashley et al., 1999). According to Wakasa et al., 1995; Nakatsuka, Wakasa & Yamaki, 1996, interfacial stresses along the bonded interface increase linearly with increasing elastic modulus of the bonded interface. This suggest that a stiffer composite near the interface could generate higher stresses during testing and cause failure at a lower force.

In contrast, the conventional RBCs tested (control group), recommended for placement in 2mm increments to ensure optimal curing, presented μTBS values comparable to those of BF materials, even when light cured for only 10 seconds in 4mm increments (Table 3.1). Thus, an under cured composite at the interface on conventional RBCs cured at short time (10 seconds) in bulk restorative technique (4 mm increment) could result in an elastic behavior of the composite (Kamagain et al., 2015), especially closer to the interface, thus resulting in “apparent” high bond strength due to yielding of the resin before fracture. This consideration can be supported by the observations in the failure mode data (Figure 3.1),

where a high percentage of cohesive failures in composite were observed for the conventional RBC (control). Interestingly, most of those aforementioned failures happened close to the adhesive interface (Figure 3.2) and more distant from the light source, which can be associated to a reduced DOC and the resulting elastic behavior of the composite during the μTBS test.

Furthermore, our specimens were stored for 24 hours, which is the most common procedure for all μTBS tests. However, this time corresponds to a minimum termination time for maximum curing (Pilo & Cardash, 1992), which could have resulted in an additional polymerization of both adhesive and composite build-ups, thus eliminating major bond strength differences between the two curing times (10 and 20 seconds) and among the resin composites (BF and conventional RBC) tested. Interestingly, a significant increase in the DC and hardness of BF materials after 24 hours storage has been observed (Alshali, Silikas & Satterthwaite; 2013; Alrahlah, Silikas & Watts, 2014).

Some studies have investigated μTBS of BF composites aiming to understand the impact of the interfacial polymerization stresses in bond strengths to dentin (Van Ende et al., 2013; Kamagain et al., 2015; Nayif et al, 2007). Van Ende et al., 2013 is one of the recent studies that looked at μTBS of BF composites compared to conventional RBCs in different C-factor cavity configurations. No significant difference in the μTBS value was found in high or low C-factor cavities for BF composites, contrary to the conventional ones tested. This cavity approach was not considered for the present study, where flat dentin surfaces were bonded instead, aiming to remove a potential confounding factor and evaluating exclusively the effects of curing protocols on bond strength to dentin.

Interestingly, the resin composites evaluated in this study demonstrated different

behavior under similar curing protocols (Tables 3.3, 3.4 and 3.5). FBF composite was the least sensitive to curing protocol in regard to μTBS values, presenting no statistically significant differences for all curing modes evaluated. It seems that the use of PW or MW LCUs have no impact on the bonding performance of FBF. However, for Ivoclar resin composite, the PW LCU resulted in overall improved bond strength values. This is in alignment to what has been recommended by the manufacturer, as they clearly disclose the alternative photoinitiator used in their composite formulation. The PW LCU used in this study (Bluephase Style, Ivoclar Vivadent) is the recommended one compatible with the photoinitiator used in the TetricEvoCeram composite family (Ivocerin®). BF composites have unique composition to enhance polymerization properties, including addition of alternative photoinitiators. The absorption spectrum of Ivocerin® falls within the blue-violet (in PW LCUs) and also in the blue (in MW LCUs) light range (Ilie et al., 2013; Ilie & Stark, 2014), thus leading to the conclusion that TBF can be effectively cured by both LCU (MW and PW) up to 4mm with minimum of 5.88 J/cm^2 energy (Ilie & Stark, 2014). Such enhanced optical properties were observed in curing profiles of BF composites (Li et al.; 2015).

The failure mode analysis showed higher percentage of adhesive and mixed failures for BF composites, which corresponds to the most common failure pattern in μTBS studies. In contrast, conventional RBC failure mode analysis presented higher percentage of cohesive failures in composite (up to 75%), which is a point for deeper consideration. As per optical and SEM observations (Figure 3.2.B), most of these cohesive failures within composite occurred close to adhesive interface, with a distinct layer of remaining conventional resin composite adhered to the bonding agent and dentin. The high percentage of these failures in this particular material (conventional RBC) is of interest, since it cannot be attributed to

material flaws and/or imperfections that could result in areas of stress concentration. As briefly discussed above, the findings can be related to what Wakasa et al., 1995 had observed, and it could be ascribed to the predominance of poorly cured composite near the interface for conventional RBCs, since they are not recommended to be used in a BF technique, with 4 mm increment.

Our results for the DOC support the findings from μTBS test since the average DOC values for T group was 2mm and even as low as 1.98mm for the 10 seconds curing time with MW LCU (Table3.6) which will be discussed further in the DOC section. In addition, these results are supported by previous investigations on polymerization of RBCs with 2mm increments to allow sufficient curing (Abbas et al., 2003, Ma'an et al., 2008). Even though this study did not assess the DC or DOC of bonded samples, the observations on the DOC results of the present study could be a potential explanation for the high percentages of cohesive in composite failures for conventional RBCs.

4.2 Depth of cure (DOC)

The second null hypothesis of this study was that the type of resin composite and curing time would not affect the DOC of the resin composites tested for different LCUs. Two-way ANOVA demonstrated that the main effects (time and material) had a significant effect on DOC for both, MW and PW LCUs, in pre-MEK and post-MEK immersion samples (Tables 3.6 to 3.9). Thus, the second null hypothesis can be rejected. A consistent pattern of DOC for MW and PW LCUs as well as for pre- (Tables 3.6 and 3.7) and post-MEK immersion (Tables 3.8 and 3.9) was observed. Overall, an increased curing time always led to an increased DOC for all composites for both LCUs. Additionally, in regard to the comparisons among materials, BF composites consistently showed significantly greater DOC

for both curing times (10 and 20 seconds) and both LUCs (MW and PW). These findings were similar for pre- and post-MEK immersion measurements, which correspond to what was observed by Menees et al., 2015, where no significant differences in DOC were found with the use of a MW or a PW LCUs. In contrast, previous studies that questioned the impact of the material's composition and LCUs (Santini et al., 2012), found significant improvement on the DC and micro hardness when a PW LCU was used in TPO containing resin composite, as Tetric EvoCeram, in comparison to a MW LCU. It is important to consider, however, that the methods used in those studies are completely diverse from what was used in this study.

Hardness tests, especially Vickers (VHN) and Knoop indentations, are widely used to indirectly assess the material's DC (Rueggeberg et al., 2000; Flury et al., 2014; Bouschlicher & Rueggeberg, 2004). Alrahlah, Silikas & Watts, 2014 study used Vickers hardness profiles (VHN) for testing the DOC of some BF materials. The results overall showed acceptable curing depths and VHN values for BF composites, mostly attributed to the increased translucency and enhanced photoinitiator systems.

The DOC method, proposed by ISO 4049 in 1988, consists of the mechanical removal of uncured resin composite light cured inside a capsule, followed by the measurement of the remaining length divided by 2. As indicated by Rueggeberg et al., 2009, the simple scraping method correlate very well with those of much more sophisticated flexural strength tests, being the main reason to use the method in this study. However, it is important to highlight that the method was proposed in 1988, using microfill RBCs and the first visible LCU available, which do not necessarily correspond to what is now available. It is important to acknowledge that RBCs chemistry and LCU photo dynamics has changed in

the past 30 years (Leprince et al., 2011). As recently described by Flury et al., 2012, the scrape method can overestimate DOC for BF composites when compared to DOC determined by hardness profiles. However, the scrape method is a valid tool for direct comparison among materials, LCUs, and curing times. As previously reported, the results observed in our study are consistent with what has been reported in the literature. Even though the extended curing time did not reflect greater bond strengths for the present study, it resulted in greater DOC. Other studies investigating polymerization properties of BF or conventional RBCs found similar results. They observed that BF composites obtained sufficient polymerization properties at 4 mm depth, and increased curing time improved polymerization properties for both BF and conventional RBCs (Zorzin et al., 2015; Rueggeber et al., 2009).

Lastly, the third null hypothesis, that there is no difference in the scrape test values before and after MEK immersion, can be rejected. The paired t-test performed for all samples showed significant increase in the composite remove after MEK immersion, regardless of curing time, LCU, and resin composite tested. This is clear and valuable information in regard to what we are measuring in the traditional ISO 4049 scrape tests. As previously reported, MEK or other solvents have been used to dissolve poorly cured composite (Al Sunbul, Silikas & Watts, 2016), thus avoiding the overestimation of DOC for resin-based composites. To our knowledge, this is the first investigation where the use of solvents was incorporated into the traditional ISO 4049 scrape test method, however it still requires further investigation to determine the ideal solvent and immersion time, and their correlation with DC and hardness. From this investigation, we were able to demonstrate that 2 hours MEK immersion was capable of dissolving under cured resin composite, which had a significant

impact the final DOC of composite cylinders.

Overall, our results are in line with previous observations in the literature, which demonstrated higher DOC for BF composites compared to conventional RBCs (Li et al., 2015, Salgado et al., 2015, Alrahlah, Silikas & Watts, 2014).

Ultimately, a direct relationship between bond strength to dentin and DOC was not perceived, as higher μTBS values did not correspond to higher DOC. However, The DOC results support the findings from μTBS and failure modes. It was evident that the average DOC data was shorter than 4mm increment compared to μTBS tested resins. This would explain the presence of cohesive in composite failures close to the interface due to presence of poorly polymerized composite observed in the T group, which is originally recommended to be applied in 2mm increments. The DOC data here shows limited DOC to 2mm regardless of the curing mode for T resins especially post-MEK immersion for MW resins cured at 10 seconds. At greater than 3 mm DOC, poorly polymerized composite led to cohesive failure in composite for BF materials tested at 4 mm increment with μTBS. This would question the quality of the bonded resin to pulpal floor of cavities at or greater than 4mm depth.

However, further research can address this research question more specifically, by isolating only one resin composite restorative material to assess the impact of curing protocols on the μTBS to dentin. An alternative study design to understand this potential relationship would be helpful to the efficiency in clinical application of the materials and curing protocols.

Chapter 5: Conclusions

Within the limitation of this study we can conclude that:

1. Curing modes did not result in differences in μTBS to dentin, with the exception of lower bond strengths on TBF composite cured with PW for 20 seconds.
2. The DOC was overall greater for both BF composites evaluated, regardless of curing time and LCU.
3. The extended curing time (20 seconds) resulted in a significant increase in the DOC for all composites in both LCUs.
4. MEK immersion can significantly decrease the final DOC measurement for all resin composites.

Chapter 6: Future Directions

Suggestions for future research for BF composites include the following:

- Long term randomized clinical trials on the use of BF composites for primary and permanent teeth.
- More investigations in the effects of curing time and energy on the DOC and bond strength of BF composites.
- Clinical comparative studies for conventional RBCs and BF composites used in primary teeth with different curing protocols.

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