UNIVERSITY *of the*
WESTERN CAPE

Micro-hardness and Depth of Cure of Dental Bulk-fill Composites

UNIVERSITY *of the*
WESTERN CAPE

Dr. Hajer Abughufa

Student number: 3270137

Supervisor: Dr. Desi Moodley

Co. Supervisor: Dr. Naren Patel

October, 2015

A mini thesis submitted in partial fulfillment of the requirements

for the degree of Master of Science in Dental Sciences in

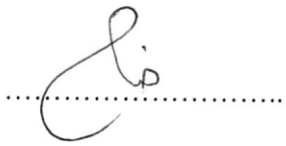
Restorative Dentistry at the Faculty of Dentistry

University of the Western Cape.

UNIVERSITY of the
WESTERN CAPE

DECLARATION

I hereby declare that, *"Micro-hardness and Depth of Cure of Dental Bulk-fill Composites"* is my own work, that it has not been submitted before for any degree or examination at any university, and that all the sources I have used or quoted have been indicated and acknowledged by complete references.



Hajer Abughufa

15th October, 2015

UNIVERSITY of the
WESTERN CAPE

ACKNOWLEDGMENTS

I would like to express my gratitude to my supervisor, Dr. Desi Moodley, an example of a wonderful person and a true professional, who I had great pleasure to work with. Throughout my thesis project he guided me and helped me make the most appropriate decisions.

My sincere gratitude also goes to my thesis co supervisor, Dr. Patel who's support, encouragement and insightful comments were fundamental for the completion of my work.

Special thanks go to Prof. Wilson the head of the department and Prof Osman Dean of faculty of dentistry for allowing me the opportunity to study at this prestigious institution.

Thanks also to Dr. Khan, who as program coordinator was always supportive and willing to help in this master program.

I would like to thank Dr. Adam for helping me initiate the idea of this research.

I am thankful to the staff of dental laboratory in Tygerberg Oral Health Center for their assistance during samples preparation procedure.

My gratitude is also due to all members of the Restorative Dentistry Department, faculty, staff and dental laboratory, who directly or indirectly helped me going through my master program and in finishing my thesis. Thanks for making my life easier and my time in Western Cape enjoyable.

UNIVERSITY of the
WESTERN CAPE

DEDICATION

I dedicate this to my parents, my supportive husband and my beautiful children for all their support, encouragement, patience and sacrifice to enable me to pursue my career.

ABSTRACT

Resin composite is one of the most commonly used materials in restorative dentistry. However, it has undergone continuous developments like changes in the fillers and initiators. One such improvement is the new bulk-fill composites which are materials intended for bulk placement up to 4mm. However, an optimum polymerization to the full depth of the restoration i.e. complete depth of cure is of utmost importance in order to obtain proper mechanical and physical properties of resin composites.

Aim: The aim of this study was to measure the surface hardness of the top and bottom surfaces of the composites and to determine the depth of cure of bulk-fill composites using two different types of light curing units.

Material and methods:

A total of 160 specimens were used in this study: four bulk-fill composite were used of which two were conventional viscosity bulk-fill composites namely, Tetric N Ceram (Ivoclar Vivadent) and SureFil bulk-fill composite (Densply Caulk) and two were low viscosity flowable bulk-fill composites namely, SDR flowable (Densply Caulk) and Filtek bulk-fill flowable restorative (3M ESPE). Two different curing light were used namely, LED (Elipar Freelight, 3M ESPE) at 1500 mW/cm^2 and a Quartz Tungsten Halogen (QTH) curing unit (Megalux CS, Megadenta, Germany) at 600 mW/cm^2. To evaluate micro-hardness, Vickers hardness at top and bottom of each sample was measured immediately after light curing and after 24 hours post curing using a Zwick micro-hardness machine load 300g/15 seconds. The mean hardness values obtained from the top and the bottom surface of each material were used to compare the micro-hardness of the various materials. The mean values obtained from the bottom surface were compared to the

UNIVERSITY of the
WESTERN CAPE

respective values of the top surface of each material (bottom/top ratio) and used to calculate the depth of cure.

Results: The micro-hardness test showed a significant difference between the four materials (ANOVA, $p<0.05$) immediately after curing and after 24 hours post curing. The material with the greatest micro-hardness was SureFil followed by Tetric N Ceram, Filtek bulk-fill flowable and SDR flowable respectively. The material with the greatest depth of cure was Filtek bulk-fill flowable followed by SDR flowable, Tetric N Ceram and SureFil.

When the curing lights were compared the Light Emitting Diode Curing Unit (LED) obtained significantly better depth of cure compared to Quartz Tungsten Halogen Light Curing Units. The LED curing light showed greater micro-hardness values than the QTH curing light except for Tetric N Ceram where the QTH curing showed more hardness values than the LED curing light. For all materials, the surface hardness and depth of cure values increased when tested 24hrs after light curing.

UNIVERSITY of the
WESTERN CAPE

Conclusion:

There was a difference in the micro-hardness values between the four materials where the conventional viscosity materials showed greater surface hardness values than the low viscosity materials but the depth of cure compared to the bulk-fill flowable LED curing lights showed higher hardness values than QTH curing light except for Tetric N Ceram. Depth of cure ratios were found to be lower than 0.80 for all composite types, however the flowable bulk-fill materials showed higher depth of cure than the conventional viscosity bulk-fills. In general LED curing light produced better hardness and depth of cure values than QTH curing light. The low micro-hardness values for the bulk-fill flowable composites and the inadequate polymerization raises a concern regarding placing these materials in bulk. In such cases, the flowable bulk-fills

should be protected with a conventional composite “covering or capping” especially in posterior teeth and in deeper cavities. Furthermore, bulk-fill composites should be used in layering incremental technique to ensure sufficient depth of cure.

Keywords:

Depth of cure

Micro-hardness

Surface hardness

Bulk-fill composite

Curing light

Table of Contents

DECLARATION ii
ACKNOWLEDGMENTS iii
DEDICATION iv
ABSTRACT v
Keywords: vii
List of figures x
List of tables xiv
Chapter 1 1
Literature Review 1
1.1. Introduction 1
1.2. History of Composite 2
1.3. Composition of Composite 2
1.4 Bulk- fill composites 9
1.5 Flowable composites 12
1.6 Activation of Dental Composites 13
1.7 Light curing units 16
1.8 Depth of curing 19
1.9 Micro-hardness 22
Chapter 2 25
Aims and Objectives 25
Chapter 3 26
Review of Materials Tested 26
Chapter 4 29
Materials and Methods 29
Chapter 5 42
Results 42
5.1 Data Analysis 42
5.2 Results 42
5.2.1 Micro-hardness measurements 43
5.2.2 Depth of cure measurements: 67
Chapter 6 77
Discussion 77
6.1 Introduction 77
6.2 Evaluation of micro-hardness 78
7.3 Evaluation of depth of cure (bottom/top ratio) 81
6.4 The efficiency of the LED and QTH curing light: 86
Chapter 7 89
Limitations and recommendations 89

UNIVERSITY of the
WESTERN CAPE

Chapter 8 91
Conclusion 91
Chapter 9 93
References 93

UNIVERSITY of the
WESTERN CAPE

List of figures

Figure1.	Schematic representation of the different properties used to evaluate photo polymerization efficiency	4
Figure2.	Schematic representation of the 3 steps of photo polymerization reaction	5
Figure4.	Chemical structures of photo-initiator systems	15
Figure5.	Synthesis of Ivocerin with 2 free radical formation	15
Figure6.	Tetric N Ceram (Ivoclar vivadent)	29
Figure7.	SureFil bulk-fill composite (Densply Caulk)	29
Figure8.	Filtek bulk-fill flowable restorative (3M ESPE)	29
Figure9.	SDR flowable (Densply Caulk)	30
Figure10.	LED (Elipar Freelight, 3 M ESPE)	31
Figure11.	QTH (Megalux CS Megadenta, Germany)	32
Figure12.	Glass slab with Mylar and Teflon mould	32
Figure13.	Packing the high viscosity bulk-fill composite	33
Figure14.	Bulk- fill specimen covered with Mylar strip	33
Figure15.	Packing the low viscosity bulk-fill composite	34
Figure16.	The bulk fill composite packed and covered with Mylar strip	34
Figure17.	Curing with LED curing light	35
Figure18.	Curing with QTH curing light	36
Figure19.	Marked top surface	36
Figure20.	Longitudinal pencil lines on the top surface	37
Figure21.	Longitudinal pencil lines on the bottom surface	37
Figure21.	Vickers hardness machine (Zwick)	38

UNIVERSITY of the WESTERN CAPE

Figure22.	Magnification of composite material	38
Figure23.	Vickers hardness indenter while indent is being placed	39
Figure24.	Specimens marked and stored in sterile containers	40
Figure25.	Comparison between the mean values of the micro-hardness (top surfaces) of the materials (immediate readings) with LED curing light	44
Figure26.	Comparison between the mean values of the micro-hardness (top surfaces) of the materials (immediate readings) with QTH curing light	45
Figure27.	Comparison of the mean values of micro-hardness between LED and QTH curing light top surface (immediate reading)	46
Figure28.	Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (immediate readings) with LED curing light	48
Figure29.	Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (immediate readings) with QTH curing light	49
Figure30.	Comparison of the mean values of micro-hardness between LED and QTH curing light bottom surface (immediate reading)	50
Figure31.	Comparison between the mean values of the micro-hardness (top surfaces) of the materials (after 24 hours readings) with LED curing light	52
Figure32.	Comparison between the mean values of the micro-hardness (top surfaces) of the materials (after 24 hours readings) with OTH curing light	53
Figure33.	Comparison of the mean values of micro-hardness between LED and QTH curing light top surface (after 24 hours reading)	54
Figure34.	Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (after 24 hours readings) with LED curing light	56

UNIVERSITY of the

Figure35. Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (after 24 hours readings) with QTH curing light 57

Figure36. Comparison of the mean values of micro-hardness between LED and QTH curing light bottom surface (after 24 hours reading) 58

Figure37. Comparison between the immediate and after 24 hours readings of the hardness values for LED 59

Figure38. Comparison between the immediate and after 24 hours readings of the hardness values for QTH (top surface) 60

Figure39. Comparison between the immediate and after 24 hours readings of the hardness values for LED (bottom surface) 61

Figure40. Comparison between the immediate and after 24 hours readings of the hardness values for QTH (bottom surface) 62

Figure41. Comparison between the micro-hardness of the top and bottom surface (immediate readings) with LED 63

Figure42. Comparison between the micro-hardness of the top and bottom surface (immediate readings) with QTH 64

Figure43. Comparison between the micro-hardness of the top and bottom surface (after 24 hours readings) with LED 65

Figure44. Comparison between the micro-hardness of the top and bottom surface (after 24 hours readings) with QTH 66

Figure45. Depth of cure values for the different materials with LED curing light 68

UNIVERSITY of the
WESTERN CAPE

Figure46. Depth of cure values for the different materials with QTH curing light (immediate readings) 69

Figure47. Comparison of depth of cure values between the two curing lights (immediate readings) 70

Figure48. Depth of cure values for the different materials with LED curing light (after 24 hours readings) 72

Figure49. Depth of cure values for the different materials with QTH curing light (after 24 hours readings) 73

Figure50. Comparison of depth of cure values between the two curing lights (after 24 hours readings) 74

Figure51. Comparison between the immediate and the 24hours readings for LED 75

Figure52. Comparison between the immediate and the 24hours readings for QTH 76

UNIVERSITY of the
WESTERN CAPE

List of tables

Table1.	Classification of composite according to matrix component	8
Table2.	Composition of test composites provided by manufacturer	28
Table3.	The curing time, thickness, and shade of the material used in the study	42
Table4.	Top surface micro-hardness comparison across groups (immediate readings)	43
Table5.	Bottom surface micro hardness comparison across groups (immediate readings)	47
Table6.	Top surface micro-hardness comparison across groups (after 24 hours readings)	51
Table7.	Bottom surface micro hardness comparison across groups (after 24 hours reading)	55
Table8.	Bottom to top ratio comparison across groups (immediate reading)	67
Table9.	Bottom to top ratio comparison across groups (after 24 hours readings)	71

UNIVERSITY of the WESTERN CAPE

Chapter 1

Literature Review

1.1. Introduction

Composite resin is a tooth coloured restorative material that is used frequently in dentistry because of the aesthetic needs of the patient. With improvements in the material it is now also used in the posterior teeth to restore the function and shape. Notable features of composite dental restorative materials are among others, handling characteristics, aesthetic appearance and clinical durability (Mikhail et al., 2013). However, a main disadvantage associated with dental composite use is polymerization shrinkage. The consequences of the polymerization shrinkage are crack formation in dentin and enamel, post-operative sensitivity, marginal discoloration, secondary caries (Tantbirojn et al., 2011) and inferior mechanical properties compared to tooth structures (Leprince et al., 2013).

UNIVERSITY of the
WESTERN CAPE

Several attempts have been made to improve the mechanical properties by altering the composition of the material. In the last decade, improvements have been made on the properties of dental composites including nanotechnology by reducing the filler particle size, increasing the filler volume to enhance wear resistance and polishing, and introducing the fillers that release fluoride (Finan et al., 2013). In addition, the development of the new silorane monomers and modified urethane monomers were introduced in order to reduce polymerization shrinkage (Finan et al., 2013). More recently, Bulk-fill composites have been introduced to the market were these materials are supposed to achieve a depth of cure of up to 4mm (Finan et al., 2013).

1.2. History of Composite

In order to minimize the disadvantages of the acrylic resins that replaced silicate cements, composite resins were introduced in the 1940s. Later in 1955, orthophosphoric acid was introduced by Buonocore to improve the adhesion of acrylic resins to the surface of the enamel (Garcia et al., 2006). The Bis-GMA (Bisphenol A-glycidyl methacrylate) monomer, a key advance in resin chemistry was developed in 1962 by Bowen to increase the physical properties of acrylic resins, as until then, their monomers only allowed linear chain polymers to be formed (Garcia et al., 2006). Prior to this, chemically cured composites that need mixing a base paste and a catalyst was used. Chemically cured composites were accompanied with some problems like the proportions of each component, as well as the mixing process and colour stability (Garcia et al., 2006).

UNIVERSITY of the
WESTERN CAPE

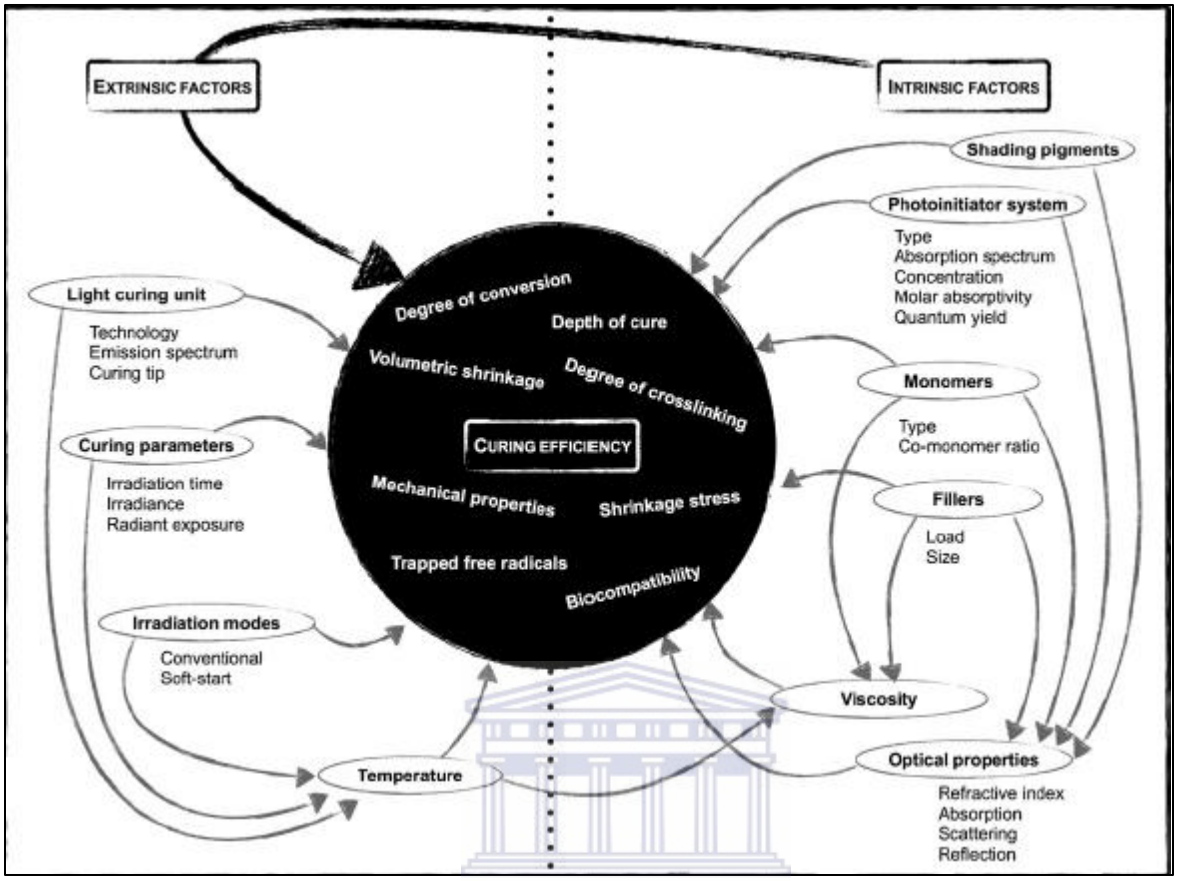
In an effort to reduce the problems related to the mixing process of chemically cured resin composite polymerization of composite material by electromagnetic radiation was introduced in 1971 (Garcia et al., 2006). The first light source used was ultraviolet (365nm) which was changed to visible light (427-491nm), which is still in use to date (Garcia et al., 2006)

1.3. Composition of Composite

The physical, mechanical and aesthetic properties of the composite depend on their structure (Garcia et al., 2006). One of the most important factors that affect the mechanical properties of dental composites is the composition of the composites itself and the depth of the curing (Mikhail et al., 2013). Ferracane, Pfeifer & Hilton (2014) stated that the chemistry of the resin monomers and the quality of the highly cross-linked network formed during the polymerization reaction greatly influences these properties. Dental composite is a mix of inorganic filler, an

organic resin matrix and coupling agent, which connect the filler with the matrix. The composite has the ability to convert from a soft state to the rigid state due to the chemical activity of the resin matrix, a process called polymerization (Van Noort, 2005), which makes it possible for this material to be used as a restorative material in dentistry. Leprince et al. (2013) reviewed the factors that may affect the success of the composite restoration (figure 1); one of which is photo polymerization efficiency of the resin composite. Photo polymerization of resin composite is a reaction in which free radicals will be generated by irradiation of a light-sensitive initiator and open the double bonds of methacrylate groups (Leprince et al., 2013). Photo polymerization can be described in three steps, initiation (formation of a free radical to start the polymerisation process), propagation (directed by the radical attack on methacrylic monomers leading to a larger molecule (chain growth) by preserving the free radical and termination (described by different mechanisms to stop the polymerization process forming a C-C double bond (figure 2) (Leprince et al., 2013). There are many factors that affect photo polymerization: extrinsic factors included, method of light curing, light spectrum, light guide tip positioning, and irradiation protocols and intrinsic factors like filler content including the percentage and the size, photoinitiator type, co-monomer composition and ratio (Leprince et al., 2013).

UNIVERSITY of the
WESTERN CAPE



UNIVERSITY of the
WESTERN CAPE

Figure 1: Schematic representation of the different properties used to evaluate photo polymerization efficiency, and of various extrinsic and intrinsic factors by which it is affected. Gray arrows indicate the influence of one factor on another or on the curing efficiency. The black arrow symbolizes the fact that the curing efficiency is not only governed by extrinsic parameters, but by intrinsic parameters as well, since differences in inherent material properties have a major influence on the way extrinsic factors affect the success of photo polymerization (Leprince et al., 2013).

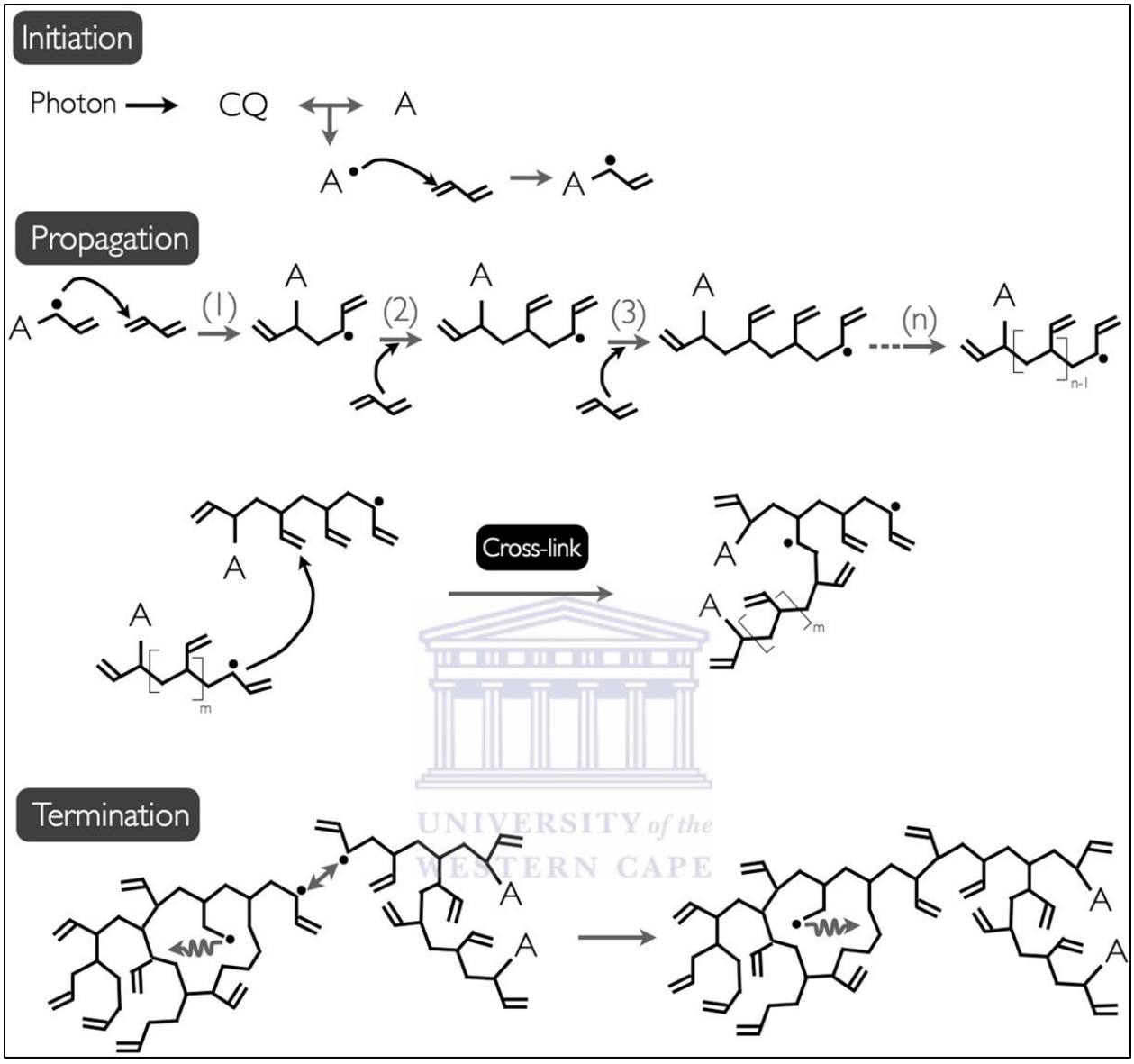


Figure 2: Schematic representation of the 3 steps of photo polymerization reaction (Leprince et al., 2013).

UNIVERSITY of the
WESTERN CAPE

1.3.1 Organic matrix

The organic matrix consists of the monomer, the most commonly used monomer is bis phenol A-glycidyl methacrylate (bis-GMA) which is mixed with other dimethacrylates, such as triethylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA) or other monomers in

order to lower the viscosity (Ferracane, 2011). An initiator - activator system which facilities the polymerization reaction by releasing free radicals is also mixed with the organic matrix. The resin matrix also contains photoinitiators like camphoroquinone the most commonly used photoinitiator which activated by using blue light of specific wavelength about 470nm to release free radicals in order to cure the composite. Pigments as well as stabilizers are also found within the organic matrix (Anusavice, 2012). Ferracane et al. (2014) reported that the physical properties of composite are influenced by the chemical composition of the monomers, the degree of polymerization, and the quality of the highly cross-linked network.

1.3.2 Filler system

The fillers are made of quartz, ceramic and or silica (Zimmerli et al., 2010). Adding fillers is one of the ways to improve the mechanical properties of the composite (Ravi et al., 2013). They can be inorganic or organic particles incorporated into the resin matrix to improve the compressive and tensile strength, abrasion resistance, modulus of elasticity, radiopacity and aesthetics (Ravi et al., 2013). It is also possible that adding these fillers may reduce polymerization shrinkage, water absorption of the resin, and the co-efficient of thermal expansion (Ravi et al., 2013). The resin composites used commonly these days contain 50 - 86 % by weight and 35 to 71 % by volume filler particle. The filler particles used have a big difference in their chemical composition, morphology and dimensions. The main filler is silicon dioxide boron silicates and lithium aluminum silicates (Ravi et al., 2013). Borges et al. (2013) reported in their study that flowable bulk-fill composites contain both spherical and irregularly shaped particles depending upon brand, in a broad distribution of sizes. The concept of not incorporating a mono modal formulation is that reducing the inter particle spacing, i.e., enhancing the wear resistance,

UNIVERSITY of the WESTERN CAPE

hardness and strength of the composite by minimizing the volume of the resin matrix through fitting smaller particles into spaces between larger particles.

With increasing filler content the linear expansion coefficient, water absorption and polymerization shrinkage are reduced. On the other hand, the modulus of elasticity the compressive, tensile strength, hardness, and wear resistance are generally increased with increasing filler content (Zimmerli et al., 2010).

Karabela & Sideridou (2011) concluded in their study that the physico-mechanical properties of the dental resin composite are affected by filler particles as well as their distribution. In a further study by Fortin and Vargas (2000) showed that the chemical composition of the filler particles influence the features of the composite. Similarly Ferracane et al. (2014) stated because the properties of the reinforcing fillers are much higher than that of the polymer resin matrix. The mechanical properties of dental composites are mainly determined by the filler component. The more highly filled materials are also more resistant to degradation in solvents, such as ethanol. One of the improvements that have been done on the composite is decreasing the particles size range from the conventional to nano hybrid. Lutz and Phillips in 1983 classified composite resins into macro filler composites (particles from 0.1 to 100 μ) micro filler composites (0.04 to 0.4μ particles) and hybrid composites (fillers of different sizes). This popular classification in term of filler particles is still valid (Garcia et al., 2006). However, Zimmerli et al. (2010) stated that this classification does not do justice to all the modern composites which are in use today as most of them are nanocomposites. So the author classifies the composite according to the matrix components (table 1). Recently, nano composites have been innovated, which contains nano particles (25nm) and nano aggregates (75 nm) (Garcia et al., 2006). As a result of the small size

UNIVERSITY of the
WESTERN CAPE

of the particles nano composites can achieve a better finishing, sufficient mechanical properties and decrease polymerization shrinkage (Garcia et al., 2006).

Matrix	**Chemical system**	**Group**
Conventional matrix	Pure methacrylate	Hybrid composite Nano composite
Inorganic matrix	Inorganic polycondensate	Ormocers
Acid modified methacrylate	Polar group	Compomers
Ring opening epoxide	Cationic polymerization	Silorane

Table 1: Classification of composite according to matrix component.

UNIVERSITY of the
WESTERN CAPE

In a recent study by Valente et al. (2013) reported on comparison of the properties of composites with two different filler distribution, one is submicron in average size (400nm with particles below 1μm) and the other one is micron (1000 nm average size but with all particles below 4 μm). The two groups had similar radiopacity, strength, and creep resistance. The material with submicron filler had higher hardness and gloss but a lower elastic modulus and reduced degree of conversion than the larger particle composite after toothbrush abrasion. Another recent study by Karabela & Sideridou (2011) evaluated the effect of the size of nanocomposites on depth of cure, showing depth of curing to be reduced by 4% as the particles was increased from 7 to 40 nm. Ferracane et al. (2014) stated that large particles lead to high surface roughness and reduced surface gloss. However, these particles enhance the strength of the composites.

1.3.3 Coupling agents

Incorporation of filler particles into the matrix resin increases the mechanical properties of the composite through the coupling agent. Coupling agents are used to adhere the inorganic filler to the organic resin matrix chemically. The most commonly used coupling agent is an organosilane such as gamma methacryloxy propyl trimethoxysilane (Ravi et al., 2013). Coupling agents transmit the stresses from the matrix to the filler particles (Anusavice, 2012). Filler content and filler size are important factors influencing the micro-hardness of the material as well as other factors like matrix-filler interactions (Manhart, 2000).

In conclusion, the concentration and the quality of the fillers, the polymer network forming monomers, and the coupling agent that links the fillers to the resin matrix to support stress transfer from the weaker polymer to the stronger filler all affect the properties of dental composites (Ferracane et al., 2014).

UNIVERSITY of the
WESTERN CAPE

1.4 Bulk- fill composites

According to Christensen (2012), the idea of bulk-fill composite is not new. It has been in and out of the market for the past two decades and was introduced as packable or condensable composites. The packable composites are stiffer and do not sticks easily to instruments like the conventional composites, which allow them to be packed into cavity preparations without slumping (Leinfelder, Bayne and Swift, 1999). These authors claimed that the packable composite might be an alternative to the conventional composite in terms of the convenience of the placement but there is no evidence that shows the clinical properties of the packable composites are better than the conventional composites. In a study by Cobb et al. (2000) the

physical properties of packable composites were shown to be better than the conventional composites. Burgess et al, (2002) reported that the large particles of the packable composites showed increased wear when compared with the conventional composites. Opposing results were shown by Manhart, Chen, and Hickel, (2001) were one of the packable composites had slightly higher flexural strength, modulus and fracture toughness when tested with a conventional composite. While another packable composite had significantly lower mechanical properties than three other conventional composites tested, suggesting that packable composites should not be used in bulk in deep cavities due to the inhomogeneous nature of the packable composite in terms of mechanical and physical properties. Recently, there have been more bulk-fill composites introduced to the market (Christensen, 2012).

Bulk- fill composites are new composite materials aimed to decrease the time taken to place the composite in the cavity by reducing the layers that have to be cured. They are also intended to minimize the shrinkage and the resulting stress by using the same exposure time and light intensity used for the regular composites (Finan et al., 2013). This is made possible by either a reduction in the filler content (Bulk-fill flowable composites), altering the filler matrix composition to improve the translucency of the material or by changing the photoinitiator system (Ferracane et al., 2014).

UNIVERSITY of the
WESTERN CAPE

There are several disadvantages associated with the layering technique in the conventional composite such as, bonding failure between the layers, contamination between composite layers, limitation to access in the small cavities leads to difficulty in placement, time consuming including placement of the composite in increments and curing it (Alrahlah et al., 2014). Saliva under rubber dam can also affect bond strengths.

Bulk-fill composites have been introduced to overcome these disadvantages. When compared with conventional composite filled in an oblique incremental layering technique, bulk-fill composite has shown reduced cuspal deflection. Also, in the evaluation of the marginal integrity bulk-fill composite performed well (Alrahlah et al., 2014).

Bulk-fill composites consist of ceramic fiber resin incorporated into the elongated filler network of about 100nm in length (Rao Kilaru, 2012). These materials have an increased depth of cure of up to 5 mm (Jackson, 2012). Bulk-fill composites are recommended for use in Class I, II, and VI restorations. They are mainly composed of light activated, dimethacrylate resins with a higher percentage of irregular (mixture of irregular particles and glass rods) or porous fillers (Fortin and Vargas, 2000). Filler loading in these composite resins varies from 60% to 80% by volume (Fortin and Vargas, 2000). The percentages of filler in the bulk-fill composites are high (Garcia, 2006). Christensen, (2012) reported the advantages of bulk-fill composites including fewer voids may be present as the composite in placed into the cavity as one piece, time saving since there is no need to place the composite in increments. Using the inter-locking particle technology is a main advantage for the bulk-fill composites where mixtures of different-sized filler particles are used. When these particles are packed together the larger particles mechanically interlock with the small particles (El-Nawawy et al., 2012). However, there are disadvantages for the bulk-fill; the shrinkage stress might be more when bulk-fill composites are used. The polymerization of these composites might be incomplete when the cavity is deep, making adequate contact areas more challenging unless adequate matrices are used (Christensen, 2012).

UNIVERSITY of the
WESTERN CAPE

In a study by El-Safty et al. (2012) comparing bulk-fill composites, conventional composites and flowable composites, the conventional composites had higher surface hardness and modulus of elasticity while the properties of bulk-fil composites was between the conventional and flowable

composites. In a recent study, (Leprince et al., 2014) the bulk-fill composites exhibited lower mechanical properties compared with the conventional composites. According to Ferracane et al. (2014), up until now, *in vitro* studies have shown that the mechanical properties of bulk-fill composites tend to be similar or lower than the conventional composites even when cured in 2mm thickness.

1.5 Flowable bulk fill composites

These are low viscosity composite resins that have been produced by lowering the percentage of inorganic filler about (20-25%) and increasing resin content when compared to conventional composites (Salerno et al., 2011). In flowable composites the resin is activated by incorporation of a photoactive group in a urethane-based methacrylate resin which leads to reduction in the shrinkage stress and polymerization rate. These resin composites are based on "stress decreasing resin" technology like in SDR flowable composite (Ilie & Hickel, 2011). Due to the low viscosity of the flowable composite, injection syringes are used to insert the composite in to the cavity, which is to make it easy to handle and decrease the risk for air entrapment and void inclusion (Salerno et al.., 2011). They have the ability to adapt to the cavity walls due to their wetting ability and low viscosity (Leprince et al., 2014). The first generation was introduced in 1990 and used as a liner or sealer, or to restore very small cavities because of the low modulus of elasticity. The latest generations of flowable composite have higher filler content and the mechanical properties have improved which allow them to be used as a bulk restoration (Salerno et al., 2011). However, the clinical reports do not show the expected success (Salerno et al., 2011). Leprince et al. (2014) stated that using flowable composite as a bulk restoration and subjected to high occlusal load is still questionable. Van Ende et al. (2013) stated that flowable composites still requires a conventional composite to be placed on top of the 4mm base. Bucuta

UNIVERSITY of the
WESTERN CAPE

& Ilie (2014) compared the micro-mechanical properties of bulk-fill composites and conventional composites and concluded that all composite materials showed adequate depth of cure when cured according to manufacturer's instructions but the flowable composites had the lowest mechanical properties.

1.6 Activation of Dental Composites

Initially, dental composites were cured chemically by mixing two pastes; one paste being the aromatic tertiary amine activator (N, N-dimethyl-p-toluidine) and the other paste is the benzoyl peroxide initiator. This method was accompanied with several drawbacks including uncontrolled working time, increased the finishing time and discoloration (Neeraj Malhotra & Mala, 2010). In 1970s, a new light activated curing system was introduced (UV light) with wavelength of 365 nm and the polymerization started only when the dental composite was activated by UV light. With this, the light splits the benzoin methyl ether into free radicals without the need for the tertiary amine activation. Therefore, one paste of composite was needed (Neeraj Malhotra & Mala, 2010). However there are some drawbacks related to the UV light including damaging the eye, soft tissue burns and the poor penetration through the tooth structure. Because of these negatives, the UV light was replaced by visible blue light activated system (Neeraj Malhotra & Mala, 2010). The most popular photo initiator (figure 3) in modern light composite curing system is a combination of a camphoroquinone (CQ) and an electron donor or co-initiator, which is generally different types of tertiary amines (Leprince et al., 2013). Camphoroquinone is sensitive to light with a wavelength, approximately 420 to 490 nm and is a source of free radicals for the curing process (Neeraj Malhotra & Mala, 2010). In addition to the standard light photoinitiator, camphorquinone, there are some other photoinitiators that absorb light at shorter wavelengths. CQ reactivity is further improved by the addition of an amine-reducing agent such

UNIVERSITY of the
WESTERN CAPE

as dimethylamino ethylmethacrylate (DMAEMA), ethyl-4-dimethylaminobenzoate (EDMAB), or N, N-cyanoethyl-methylaniline (CEMA). CQ and amine concentrations vary in commercial composites from 0.2 to 1.2 wt%. Another photoinitiator, 1- phenyl-1, 2-propanedione (PPD), which has an absorption peak near 410 nm, has also been suggested as an alternative (figure 3) (Uhl, Mills & Jandt., 2003). In bulk-fill composite, the photoinitiator system is still the same camphorquinone based as in the conventional composite except for Tetric N Ceram, which contains a new photoinitiator called Ivocerin (figure 4). Ivocerin is germanium based photoinitiotar which contributes to the increase in the depth of curing without affecting the optical properties of the composite (Moszner, 2013). Moszner et al. (2008) reported that the absorption spectrum of Ivocerin is very close to that of camphorquinone and due to its higher absorption of visible light Ivocerin has a higher photo-curing activity than camphorquinone. With ivocerin photo initiator at least two radicals that are able to start the polymerization are formed while only one in camphorquinone making it more efficient to initiate polymerization (Ilie & Stark, 2014).

UNIVERSITY of the
WESTERN CAPE

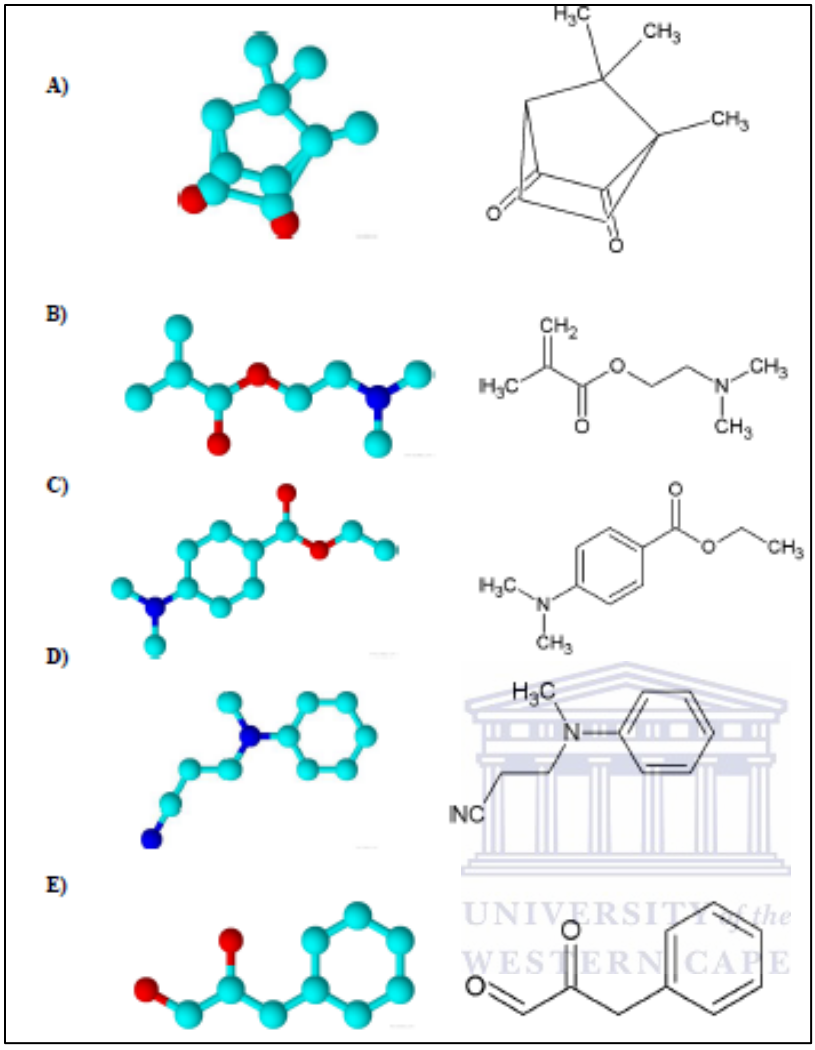


Figure 3: Chemical structures of photo-initiator systems, A. CQ; B. DMAEMA; C. EDMAB; D.CEMA; E. PPD (Alrahlah et al., 2014)

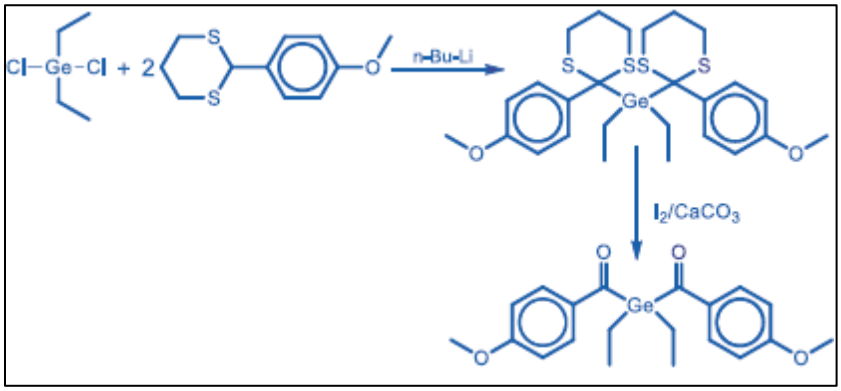


Figure 4: Synthesis of Ivocerin with 2 free radical formation (Moszner, 2013).

Leprince et al. (2013) reported that photoinitiotar concentration affects the efficiency of polymerization of composite and increase in the micro-hardness was shown when the concentration of the photoinitiator was increased. In contrast, Furuse, Mondelli, & Watts (2011) found that increasing the concentration of the photo initiator beyond the optimum value will result in reduction of degree of conversion and micro-hardness values due to the excessive absorption of the light in the superficial layers resulting in less transmission of the light to the deeper layers. Phenylpropanedione, mono- or bis-acylphosphine oxides, benzoyl germanium considered as alternative photoinitiator for camphorquinone either to improve the esthetic quality due to its yellowing effect or because of significantly increased molar absorptivity and thus, improved polymerization efficiency (Leprince et al., 2013).

1.7 Light curing units

UNIVERSITY of the
WESTERN CAPE

There are several light curing units available including Quartz Tungsten Halogen (QTH) light curing units, Plasma-arc lights and lights utilizing light emitting diodes (LED). However, the optimal light-curing unit for curing of composites has not yet been determined.

These light cure units (LCU) require the following important features to produce desirable polymerization: adequate light output, appropriate wavelength range of the light and exposure time (Knezevic et al., 2001). Furthermore, Boksman and Santos (2012) stated that the ideal light-curing units should have a broad emission spectrum, adequate light intensity, less drop-off energy with distance, various curing modes, enough duration for multiple curing cycles, durability, a big curing foot print, and fixed easily. The mechanical and the physical properties of dental composite are negatively affected by insufficient polymerization (Alrahlah et al., 2014). According to Mousavinasab and Meyers, (2009) the quality of curing light greatly influences the

properties of the light cured composite restoration. When the light transmitted through the composite resin the energy of emitted light decreases and this will lead to decrease in the degree of conversion of the composite, resulting in compromised physical properties and increase elution of the monomers (Flury et al., 2012).

1.7.1. Quartz Tungsten Halogen Light Curing Units

Due to a safety reason regarding the long term use of ultraviolet light, the halogen visible light curing unit (QTH) was introduced in 1980. The light contains a lamp with a tungsten filament in an inert gas and small amount of halogen. The tungsten is heated electrically to 2727°C, which creates light that is visible as well as infra-red radiation (Boksman& Santos, 2012). The composite absorbs this resulting in heat generation (Uhl et al., 2003). The heat generation which makes the bulb degrade fast is one of the negatives of the halogen light curing unit, therefore the life span of this light is limited about 100 hour and then the bulb must be replaced (Boksman & Santos., 2012). The fan cooling required to lessen the generated heat can be noisy and create a bio-burden trap. The light filtered to between 390 nm and 500 nm is capable of curing every composite, but the lights only use 9% of the total energy produced and the bulbs start to denature at between 30 to 50 hours and then they must be replaced (Boksman & Santos., 2012) . Further challenges are that narrow light tips emit a small curing footprint, which may require multiple curing cycles in large restorations (Boksman & Santos., 2012). The light intensity of this light is between 400-900mW/cm^2 (Kumar et al., 2012). One of the advantages of using the QTH is low cost technology.

UNIVERSITY of the
WESTERN CAPE

1.7.2. Light Emitting Diode Curing Units

There have been multiple generations of light emitting diode light curing units (LED LCUs) since its introduction in 2000 (Boksman & Santos., 2012). In this light curing unit the junction of doped gallium nitride semiconductors are exposed to electric current to generate the blue light. Due to the narrow emission spectrum of the first generations of LED LCUs they could only activate camphorquinone but not the other photo-initiators, thus could not fully polymerize some composite materials. However, the latest LED LCUs could cure most of the composite because they emit energy at the absorption spectrum for camphorquinone and the other photoinitaitors like phenylpropanedione as well (Boksman& Santos., 2012).

In comparison with QTH, LED curing units have narrower wavelength spectrum (440-490nm) which is sufficient to activate the camphoroquinone photo initiator, the diodes have long life span of more than 10,000 hours compared to 40-100 hours life time of (QTH) bulbs. The heat generation is less than the QTH and therefore most of the LED LCUs don't need a fan (Rueggeberg et al., 1996).

UNIVERSITY of the
WESTERN CAPE

In a study comparing the efficiency of different light curing units Price, Fahey & Felix, (2010) reported that some curing lights do not provide as much energy as recommended and produce softer composites. Rastelli et al. (2014) stated that Halogen LCU showed greater Vickers hardness values than LED LCU mainly because of the power density used.

In another study conducted by Mills, Jandt, & Ashworth, (1999) LED LCU with irradiance of 64% of halogen LCU achieved a significantly greater depth of cure. Yaman et al. (2011) found that the LED LCUs were to be more effective than the QTH LCUs regarding both curing depth and micro-hardness properties. These results are in contradiction to the result of the study by Uhl et al. (2003) which reported higher depth of curing in case of halogen LCU than LED LCU for

all materials tested and for the different curing times tested. According to Yazici et al. (2010) both curing units, QTH and LED produced acceptable clinical results and the LED LCUs are as effective as QTH LCUs when curing composites. In a further study Dunn & Bush, (2002) demonstrated that QTH LCUs produce harder surface for resin-based composite than LED LCUs regardless the type of the composite. Mousavinasab and Meyers, (2009) carried out a study comparing the efficiency of the light curing units on the depth of curing, they used ten different types of LED LCUs and two QTH LCUs and they found that a variety of LED light sources are as effective as the high intensity QTH lights on depth of curing. According to Choudhary & Suprabha, (2013) the type of composite influences the effectiveness of the LCUs, they stated that curing nano composite with QTH LCUs produce better surface hardness at both top and bottom than micro hybrid, and adequate depth of cure was obtained from both LCUs. In a study comparing the photointiators in hybrid composites, the photoinitiator that presented in one material responded differently to the light than the other materials (Thiab, 2012).

UNIVERSITY of the
WESTERN CAPE

1.8 Depth of curing

Leprince et al. (2013) defined the depth of cure as the maximum thickness of each cured composite layer. The depth of cure is the depth to which the light is able to harden the material (El-Nawawy et al., 2012). Inadequate depth of curing and insufficient monomer conversion depth is one of the problems associated with photo-polymerized resin composites (Lindberg et al., 2005). Penetration of visible light through the photo cured composite material determines the depth of curing (Alrahlah et al., 2014). Mousavinasab, (2011) stated that the amount of the light that reaches the photoinitiator limits the depth of curing and the intensity of this light decreases as it passes through the material. Rouhollahi, Mohammadibasir and Talim, (2012) showed that

the depth of cure decreased with the increase in thickness of the composite material which is similar to the study by Ceballos et al. (2009). One of the main challenges of successful composite restoration is to obtain sufficient depth of cure not only because the inadequate depth of cure will affect the mechanical properties but also to be sure that there will be no clinical problems will arise due to partially polymerized composite (El-Nawawy et al., 2012). In addition, insufficient curing may lead to release monomers which may affect the soft tissue (Moore et al., 2008).

1.8.1 Factors affecting depth of cure

There are several factors affecting the depth of cure including the type of resin composite, shade and translucency, increment thickness, distance from the tip of the light cure unit, post-irradiation, the wavelength of the curing light used for polymerization, intensity of the curing light and irradiation type (Martin, 1998 cited by Alrahlah et al., 2014). In a study by Atmadja & Bryant, (1990) the depth of cure was affected by composite composition rather than irradiance from light units. Light scattering is a very important factor for depth of cure that is related to filler particles size and it is maximized when the filler size is half of the wavelength of the light curing unit (Mousavinasab, 2011). The total energy of irradiation which is related to the exposure time will influence on depth of curing, thus on the mechanical properties of the dental composites (Lombardini et al., 2012). Although Ceballos et al. (2009) showed that an exposure time higher than 40 seconds is not effective. Halvorson et al. (2003) reported that the irradiance of the curing light, exposure time and composite type are significantly affecting the hardness and curing depth. Aguiar et al.(2007) point out that in deep cavities preparations it is very important to increase the curing time by at least three times to ensure adequate depth of curing.

UNIVERSITY of the

To minimize the polymerization shrinkage and to achieve the desirable depth of cure the incremental layering technique with 2mm thickness should be used (Alrahlah et al., 2014). However, there are disadvantages of using these incremental techniques, include contamination between composite layers, microleakage, placement difficulties especially in the small cavities and time consuming for placement of layers and curing them (Abbas et al., 2003). Bulk-fill composites have been introduced to overcome the aforesaid disadvantages. This allows packing composite in layers more than 2mm, generally 4mm thickness.

To investigate the adequate depth of curing there are direct and indirect methods. Infrared spectroscopy and laser Ramon are direct methods which are complex and very expensive. Micro-hardness, visual inspection and penetrometer technique are some of the indirect methods which are commonly used these days (Rouhollahi et al., 2012). Other techniques including measuring the hardness of the top and bottom specimen surfaces, the degree of conversion, optical microscopy, where there is a visual boundary between cured and uncured material (Alrahlah et al., 2014). In addition the International Organization for Standardization (ISO), in the second edition of ISO 4049 introduced the protocol for investigating the depth of curing for composites. In this method a cylindrical mould is used, the resin composite to be tested is filled in the mould, light cured, pushed out from the mould, and then the uncured composite at the bottom surface is removed by scraping it away using a spatula keeping the hard portion. The length of the remaining hard cured resin composite is measured. The absolute length is divided by total initial length and the latter value recorded the depth of curing (Flury et al., 2012). Leprince et al., (2013) stated that the recent data indicated that this method may overestimate the depth of cure due to the inability to distinguish between the changes of state of resin matrix from hard and soft composite occurring at the depth. Flury et al. (2012) compared the ISO 4049 method and Vickers

UNIVERSITY of the
WESTERN CAPE

hardness method to evaluate the depth of curing, and concluded that for bulk-fill materials the ISO 4049 method overestimated depth of cure compared to the determination by Vickers hardness method.

Also, Rastelli et al. (2014) pointed out that a hard top surface is not an indication of adequate depth of cure through the whole restoration. Choi et al. (2000) reported that no composite had adequate depth of cure when tested in increments greater than 2 mm thick and the polymerization contraction of the packable composites was similar to or higher than that of the conventional composites. Manhart et al. (2001) stated that they could only adequately cure packable composites ranging from 2.5-3.5 mm. However, in a comparative study by Cobb & MacGregor, (2000) investigated that the depth of cure of packable composites was a significantly greater than all other dental composites.

UNIVERSITY of the
WESTERN CAPE

1.9 Micro-hardness

The term hardness is difficult to define (Anusavice, 2012). The most accepted definition is "the resistance to indentation" and most hardness tests designed depending on this precept (Anusavice, 2012). Poskus, Placido & Cardoso, (2004) define the micro-hardness as the resistance to permanent deformation only caused by indentation after load. Micro-hardness of composites is affected by many factors such as: organic matrix composition, type and amount of filler particles and the size of the filler particles (Correr, 2005). Evaluation of micro-hardness is used widely as a test to assess the curing of the composites and the efficiency of light sources (Yaman et al., 2011). The surface micro-hardness of resin composites has been used to evaluate the efficiency of the light cure unit and to evaluate the extent of polymerization indirectly (Alrahlah et al., 2014). The micro-hardness of composites decreased with increasing depth of

composite (Rueggeberg et al., 1993). In a study about the effect of filler type, the light intensity, the duration of exposure and the thickness on the micro-hardness of composite resin it was claimed that at 1 mm depth, the order of influential factors were exposure duration, filler type and source intensity. At depths of 2 mm and more, the influences on cure were related only to light intensity and exposure time (Rueggeberg et al., 1996). Filler type and shade of the composite show less effect at these depths (Rueggeberg et al., 1996). The study by Coffey et al. (2004) showed that the top surfaces of resin composite materials show higher micro-hardness values compared to the bottom surfaces. Besides that there are some factors like the interactions of the matrix-filler which highly influence the micro-hardness and wear behavior of the materials (Manhart et al., 2000).

There are several types of surface micro-hardness tests including, Barcol, Brinell, Rockwell, Shore, Knoop, and Vickers. Rockwell and Shore are used to measure the hardness of rubber and plastic materials. Barcol and Brinell are employed to determine the hardness of the metal materials. Knoop and Vickers are classified as micro-hardness tests and both of them employ loads less than 9.8N. Because the resulting indentations are small and limited to a depth of less than 19μm, these two tests are able to measure micro-hardness in small regions of very thin objects. Knoop test is used to determine the hardness of both soft and hard materials because it minimizes the effect of plastic recovery while Vickers test is used to measure the hardness of the brittle materials (Anusavice, 2012). The surface micro-hardness tests are a widely used for the investigation of composite curing and the efficiency of light sources (Aguiar et al., 2007). The criteria of the bottom to the top hardness ratio from 0.80-0.90 have been used as a predictor for adequate depth of curing at a specific sample thickness. This criteria means that the ratio of

UNIVERSITY of the
WESTERN CAPE

bottom to top surface micro-hardness is 80% or more will indicate adequate curing (Rouhollahi et. al., 2012).

In a comparative study on placement techniques of composites, Poskus et al. (2004) stated that the results of the measurements for both tests were statistically similar and the conclusion of this study was that both Vickers hardness test and Knoop hardness test can be used to measure the depth of curing.

Most of the studies on depth of cure for conventional composite stated that there is adequate depth of cure at 2mm increments. Recently, the newer bulk-fill composites that were introduced suggest that they can be used in 4mm increments. The question now arises does 4mm increments provide sufficient depth of cure.

Therefore the aim of this study was to assess the depth of cure of four different bulk-fill materials, two of which are high viscosity bulk-fill composites and two are low viscosity bulk-fill composites.

UNIVERSITY of the
WESTERN CAPE

Chapter 2

Aims and Objectives

2.1 Aims:

The aim of this study was to determine the surface hardness and depth of cure of bulk-fill composites using two different types of light curing units.

2.2 Objectives:

1. To measure the surface micro-hardness of bulk-fill composite materials using two different types of light sources namely: Quartz Tungsten Halogen (QTH) and light emitting diode (LED) as determined using the Vickers hardness test.

2. To compare the effect of the two light curing units, i.e. Quartz Tungsten Halogen (QTH) vs. light emitting diode (LED) on the surface hardness of the bulk-fill composite materials.

3. To determine the depth of cure of bulk-fill composite materials based on the top and bottom surface micro-hardness using two different light sources namely: Quartz Tungsten Halogen (QTH) and light emitting diode (LED)

4. To compare the efficiency of the two light curing units on the depth of cure of bulk-fil composites.

UNIVERSITY of the
WESTERN CAPE

Chapter 3

Review of Materials Tested

3.1 Tetric N Ceram (Ivoclar Vivadent AG, Liechtenstein)

Tetric N Ceram is a nano hybrid bulk-fill composite specifically designed for the bulk-filling of posterior restorations using one increment. According to the manufacturer, the advanced chemical properties enable Tetric N Ceram to adapt better to cavity walls. Tetric N Ceram incorporates a new photoinitiator, Ivocerin. This is a germanium-based photoinitiotar, which increases the depth of curing without affecting the optical properties of the composite (Moszner, 2013). Ivocerin provides an efficient depth of cure of up to 4 mm and a shorter curing time of 10 seconds (Moszner, 2013). The other inclusions in this material are a light sensitive filter to prevent premature polymerization and shrinkage stress reliever.

UNIVERSITY of the
WESTERN CAPE

The new photoinitiotar, Ivocerin works in balance with other components such as camphorquinone and 2, 4, 6 trimethyl benzoyl diphenylphos- phine oxide, to obtain the desirable depth of curing as well as to increase the material's properties (Vogel, 2013). Tetric N Ceram Bulk-fill incorporates several different types of filler (barium aluminium silicate glass with two different particle sizes, an Isofiller, ytterbium fluoride and spherical mixed oxide in order to achieve the desired composite properties. Tetric N Ceram is similar to Tetric Evo Ceram, however, Tetric Evo Ceram contains slightly more filler (80% by wt, 60% by vol) than Tetric N Ceram (77% by wt, 55% by vol) (table2).

3.2 SureFil bulk-fill composite (Dentsply, USA)

This is a visible light activated, radiopaque restorative material designed to be used in posterior restorations of primary and permanent teeth as a bulk-fill. It is easy to manipulate, rapid, bulk placement and it can assist in creating a well-shaped proximal contact area. SureFil contains three differently sized fillers (midifiller, minifiller, microfiller). Table 2 shows the chemical composition provided by the manufacturer. It was the first bulk-fill composites on the market (Jackson, 2012).

3.3 SureFil SDR (Stress Decreasing Resin) Flowable (Dentsply, USA)

It is a flowable composite bulk-fill material (defined as a flowable composite with low filler content). The material is easy to sculpt and control which saves time during placement, bulk-fill fluoride-releasing material, it is designed to be used as a base for class I and II restorations and liner under direct restorative materials, Pit & Fissure sealant, conservative Class I restorations and Core build-up. It contains camphorquinone (CQ) as a photoinitiator. Table 2 shows the chemical composition provided by the manufacturer.

UNIVERSITY of the
WESTERN CAPE

3.4 Filtek Bulk-fill Flowable (3M ESPE, Germany)

This is a flowable restorative material designed for low shrinkage, low polymerization stress, and the low viscosity which provide a good adaptation. Table 2 shows the chemical composition provided by the manufacturer.

Property	Filtek Bulk-fill	SureFil	SDR	Tetric N Ceram
Filler level (wt%)	64.5%	77%	68%	75-77%
Filler level (vol%)	42.5%	58%	45%	53-55%
Filler type & particle size	Zirconia/silica, Ytterbium triflouride	Ba-B-F–Silicate,SiO_2 nanofiller	Barium and strontium alimino-flouro-silicate glasses	Barium aluminium silicate glass,Ytterbiumfluoride, spherical mixed oxides
Resin type	BIS-CMA, UDMA, BIS-EMA	UDMA	UDMA, DMA	Bis-GMA,UDMA, Bis-EMA
Compressive strength (MPa)	390	433	220	203
Flexural strength	154	125	126	135.16
Tensile strength	79	81		
Radiopacity		High		260
Shades	A3,B2,C2, universal	A,B,C	Universal	A1,A2,B1,B2
Fluoride release		Yes		

Table 2: Composition of test composites provided by manufacturer

UNIVERSITY of the WESTERN CAPE

Chapter 4

Materials and Methods

4.1 Materials

Four types of composite materials were used in this study, two bulk-fill composite materials namely, Tetric N Ceram (Ivoclar Vivadent) (figure 5), and SureFil bulk-fill composite (Densply Caulk) (figure 6), and two bulk-fill flowable materials namely Filtek bulk-fill flowable restorative (3M ESPE) (figure 7) and SDR flowable (Densply Caulk) (figure 8).

Figure 5: Tetric N Ceram (Ivoclar vivadent)

UNIVERSITY of the
WESTERN CAPE

Figure 6: SureFil bulk-fill composite (Densply Caulk)

Figure 7: Filtek bulk-fill flowable restorative (3M ESPE)

Figure 8: SDR flowable (Densply Caulk)

4.2 Methods

The methodology of the study included the following steps:

A. Calibration of the curing lights.

B. Preparation of the specimens.

C. Curing the specimens.

D. Calibration of Vickers hardness machine.

E. Vickers Indentation of specimens.

F. Measurement of the Vickers indents immediately.

G. Measurement of the Vickers indents after 24 hour.

H. Measurements of the depth of curing immediately and after 24 hours.

UNIVERSITY of the
WESTERN CAPE

A. Setting of the curing lights

Two types of curing lights were used in this study, LED light curing (Elipar Freelight, 3M ESPE) (Figure 9) and QTH blue curing light (Megalux CS Megadenta, Germany) (Figure 10). After complete charging, the LED and QTH curing units were tested for intensity using a Cure Rite visible curing light meter (Caulk, USA). The intensity was recorded >600mW/cm^2 for the QTH and, >1,500 mW/cm² for LED. The intensity was checked after every 10 specimens throughout the study for repeatability and reliability.

UNIVERSITY of the
WESTERN CAPE

Figure 9: LED (Elipar Freelight, 3 M ESPE)

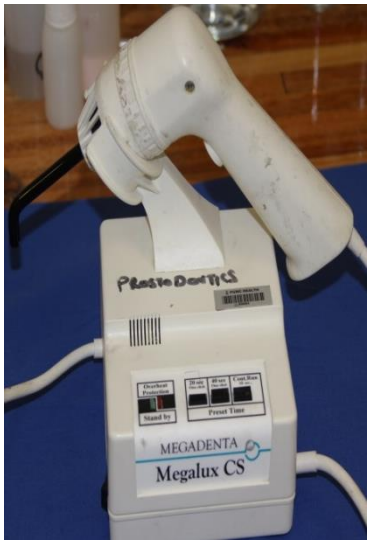


Figure 10: QTH (Megalux CS Megadenta, Germany)

B. Preparation of the specimens discs

A total of 160 specimens were used in this study; the specimens were divided into two sub groups. Each group was further divided into 4 groups, with each group having 20 specimens of each type of bulk-fill composite used in this study. A Teflon cylindrical mould with a central orifice (6mm in diameter, 4mm in thickness) was prepared as shown in figure 11. The cylindrical mould was placed on a cellulose acetate Mylar strip (3M ESPE, USA) resting on a transparent glass slab on a dark non-reflective surface (figure 12).

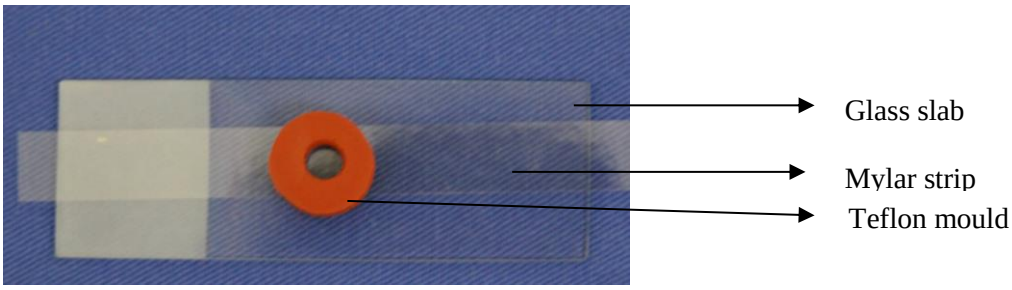


Figure 11: Glass slide with Mylar and Teflon mould

The mould was filled with the composite. The composite was dispensed directly from its container into the cavity of the mould using a flat plastic instrument (bulk-fill composite) (figures 13, 14) or injected from the syringes or capsules (flowable composites) as shown in Figure 15.

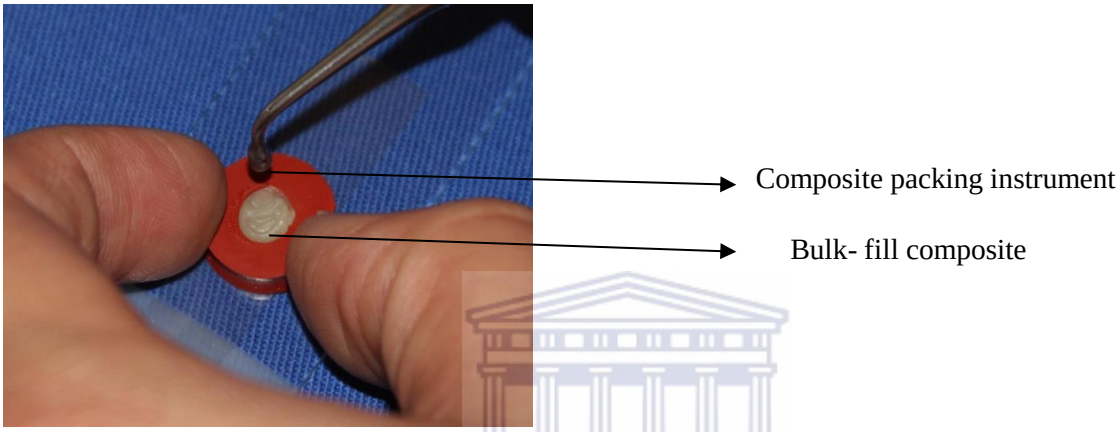


Figure 12: Packing the high viscosity bulk-fill composite

UNIVERSITY of the
WESTERN CAPE

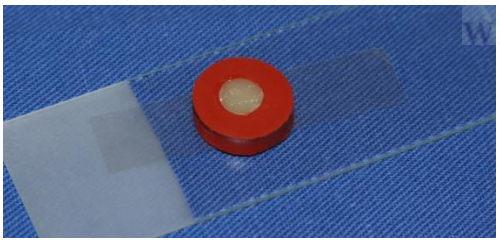


Figure 13: Bulk- fill specimen covered with Mylar strip

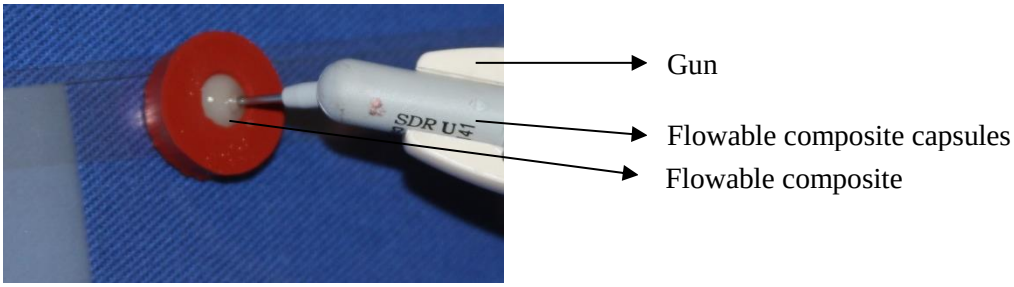
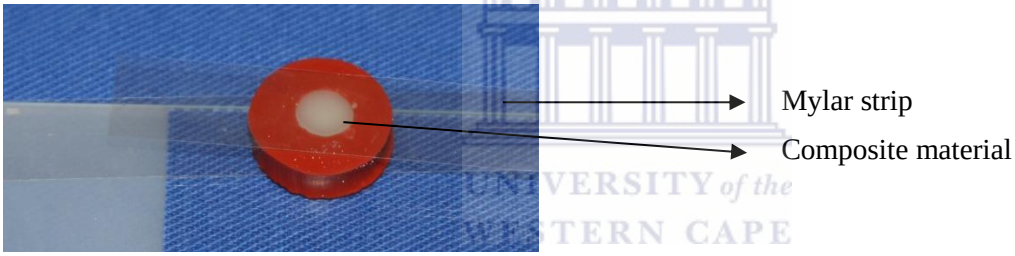


Figure 14: Packing the low viscosity bulk-fill composite

A transparent Mylar strip (3M ESPE, USA) was placed over the filling at the center of the mould (figure15). New and clean Mylar strips were used for each specimen.



UNIVERSITY of the
WESTERN CAPE

Figure 15: The bulk fill composite packed and covered with Mylar strip**.**

C. Curing the specimens:

The LED curing light (Elipar Freelight, 3M ESPE) at >1500mW/cm^2 was charged according to manufacturer's recommendations and placed back in their battery charger after each specimen was polymerized. QTH (Megalux Megadenta, Germany) at >600mW/cm^2 was used. The specimen mould, with the filling material at the center and covered with a Mylar strip was cured using one of the curing lights. According to Thome et al. (2007) the light intensity decreases with increasing distance from the light cure tip, hence, the light cure tip was kept in contact with the Mylar strip to standardize the procedure (Figures 16, 17). Curing time was done according to manufacturer's instructions (Table 3). Half the specimens were cured with the LED lights while the other half of the specimens were cured using the QTH curing light.

UNIVERSITY of the WESTERN CAPE

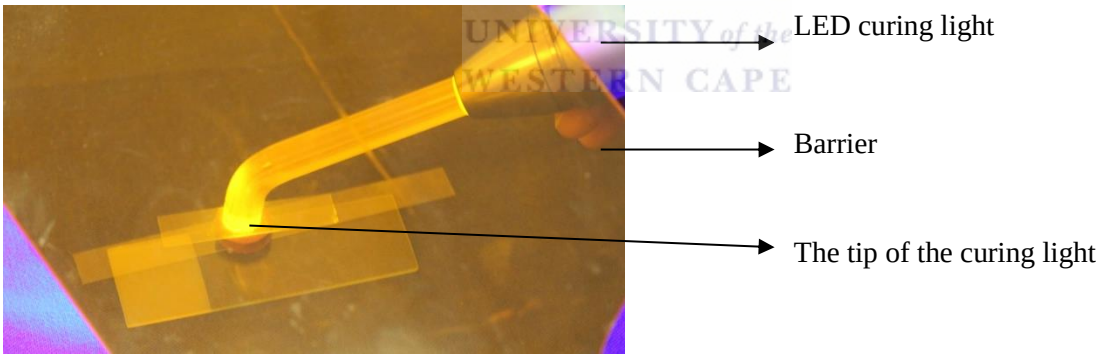


Figure 16: Curing with LED curing light

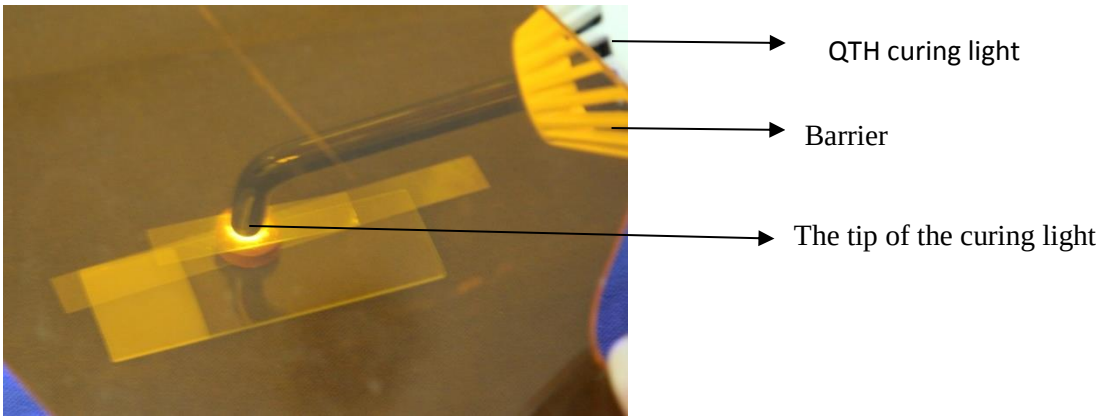


Figure 17: Curing with QTH curing light.

The top surface was identified with a permanent marker (Figure 18). The composite material was pushed out from the mould. All excess material was removed from the specimens. Then the top and the bottom surfaces were marked with three longitudinal pencil lines to indicate the position of the indentation (figures 19, 20).

UNIVERSITY of the
WESTERN CAPE

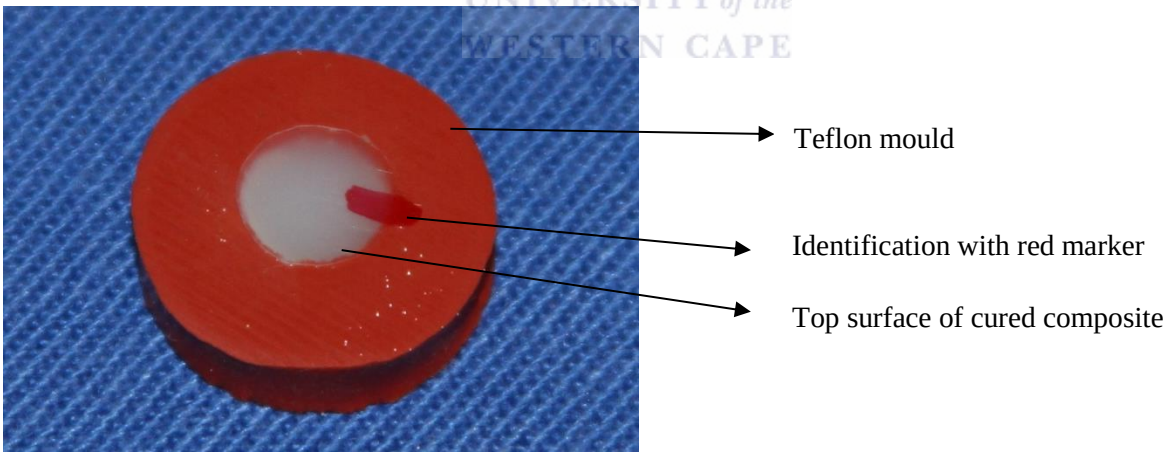


Figure 18: Marked top surface

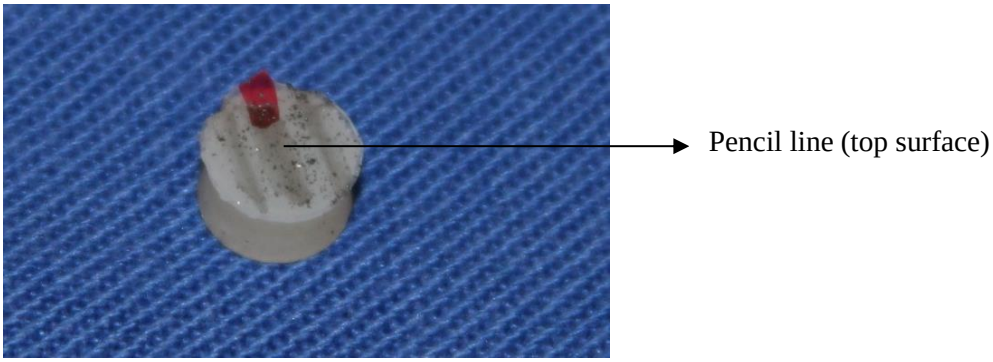


Figure 19: Longitudinal pencil lines on the top surface.

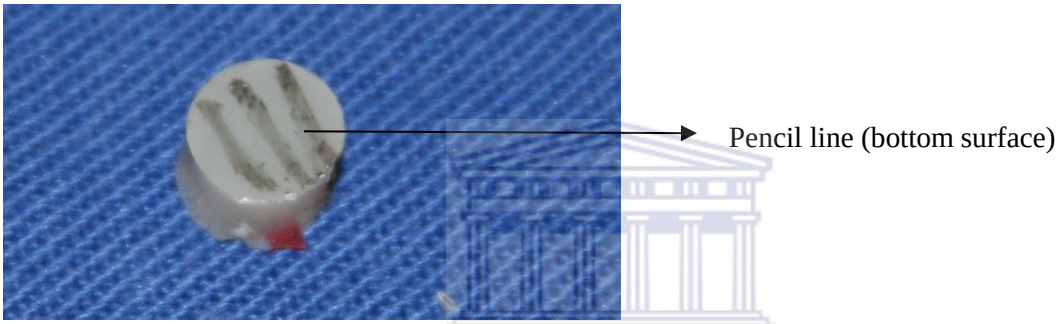


Figure 20: Longitudinal pencil lines on the bottom surface.

UNIVERSITY of the
WESTERN CAPE

D. Set up the Vickers hardness machine

The Vickers hardness machine (Zwick, Germany) (figure 21) was adjusted to a load of 300g for 15 seconds according to ISO 4049 (ADA, 2003). Each sample was placed on the stage of the machine and a magnification of 40X was used to adjust and bring into focus the center of the composite material in the disc to identify a smooth surface, devoid of voids or other irregularities (figure 22).

Figure 21: Vickers hardness machine (Zwick).

UNIVERSITY of the
WESTERN CAPE

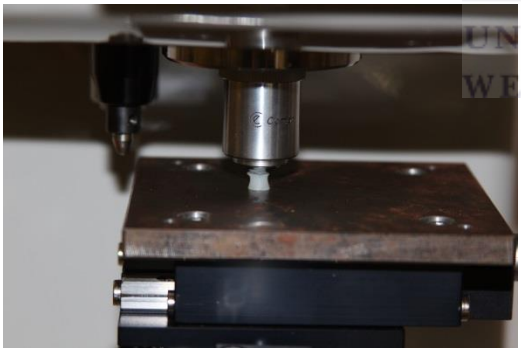
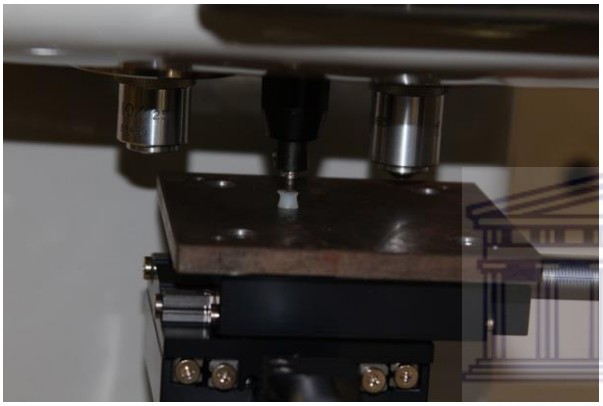


Figure 22: Magnification of composite material to bring surface of sample into focus to identify a smooth surface free of voids and other irregularities.

E. Vickers Indentation of specimens:

The machine was adjusted previously to a load of 300g for 15 seconds. The indenter is moved automatically to start the indentation. Three indentations were made on the top surface and three on the bottom surface of each specimen equidistant from each other. The mean values of the three indentations for the top and the bottom were evaluated for statistical analysis for each curing light.



UNIVERSITY of the
WESTERN CAPE

Figure 23: Vickers hardness indenter while indent is being placed.

F. Measurement of the Vickers indents immediately:

Three measurements were taken from the top and the bottom surfaces of the 4mm thick specimens immediately after curing and then the specimens were stored in dry and sterile containers (figure 24) in dark, dry incubator for 24 hours at 37 degrees Celsius.

Figure 24: Specimens marked and stored in sterile containers.

UNIVERSITY of the
WESTERN CAPE

G. Measurement of the Vickers indents after 24 hours

After 24 hours, every specimen was put on the stage of the Vickers hardness machine and the measurements were repeated for the top and the bottom surfaces of all thick specimens for each curing light.

H. Calculation of the depth of curing immediately and after 24 hours

Depth of cure was calculated immediately and after 24 hours for each specimen by calculating bottom to top ratio from the measured Vickers hardness mean values.

All specimens were treated according to manufacturer's instructions as shown in table 3.

The material	**Filtek bulk-fill flowable restorative (3M ESPE)**	**SDR flowable (Densply Caulk).**	**SureFil bulk-fill composite (Densply Caulk)**	**Tetric N ceram (Ivoclar vivadent)**
Number of specimens	40	40	40	40
The LED curing time	20 sec	20 sec	40 sec	10 sec
The (QTH) curing time	40 sec	20 sec	40 sec	20 sec
Thickness	4 mm	4 mm	4 mm	4 mm
Colour	A2	Universal	A	A2

UNIVERSITY of the WESTERN CAPE

Table 3: The curing time, thickness, and the shade of the material that were used in the study.

Chapter 5

Results

5.1 Data Analysis

The results were transferred to an Excel spreadsheet (Microsoft 2010, USA). The data was analysed statistically using one way ANOVA with p values less than 0.05 were taken to indicate statistical significance.

5.2 Results

A One Way analysis of variance (ANOVA) was carried out to investigate if statistically significant differences existed between the experimental groups at a significance level of $p<0.05$. The ANOVA test was used to compare the mean values of the micro-hardness of top surfaces, micro-hardness of bottom surfaces, depth of cure of the four restorative materials as well as to compare differences between the two curing lights with regards to its effects on the hardness of the top and bottom surfaces taken immediately after curing as well as 24 hours after curing.

UNIVERSITY of the
WESTERN CAPE

5.2.1 Micro-hardness measurements

5.2.1.1 Top surface: Measurement of hardness top surface (immediate reading)

The mean values, the standard deviation and the results of the ANOVA test for both curing lights are represented in Table 4.

Material	LED	SD	QTH	SD	P-value (LED vs QTH)
Filtek bulk-fill flowable	35.13	2.55	30.70	1.15	<0.0001
SDR bulk-fill flowable	28.35	1.62	26.61	1.30	=0.0007
SureFil bulk-fill composite	71.95	3.94	61.60	3.16	<0.0001
Tetric N Ceram bulk-fill	35.83	2.91	42.33	2.58	<0.0001

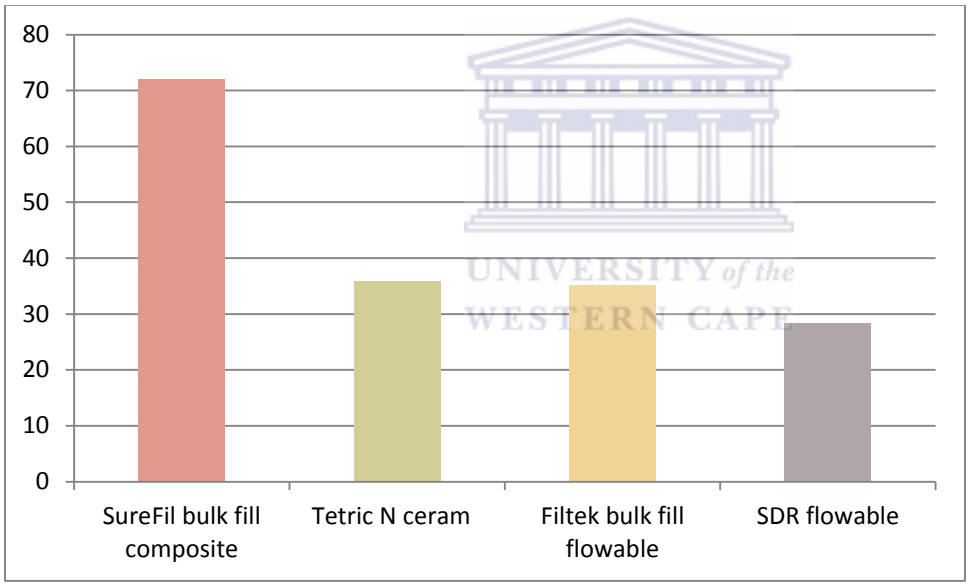
UNIVERSITY of the
WESTERN CAPE

Table 4: Top surface micro-hardness comparison across groups (immediate readings)

5.2.1.1.1 Micro-hardness measurements of the top surface with LED curing light (immediate readings)

There were statistically significant differences (ANOVA $p<0.05$) between the mean values with SureFil bulk-fill showing higher hardness values at the top surface and the SDR flowable showed the lowest values (Figure 25). However there was no significant difference between Tetric N Ceram and Filtek bulk-fill flowable ($p>0.05$). Of the two flowable, the Filtek flowable had higher micro-hardness values than SDR in the following order:

SureFil bulk-fill > Tetric N Ceram = Filtek bulk-fill flowable > SDR flowable.



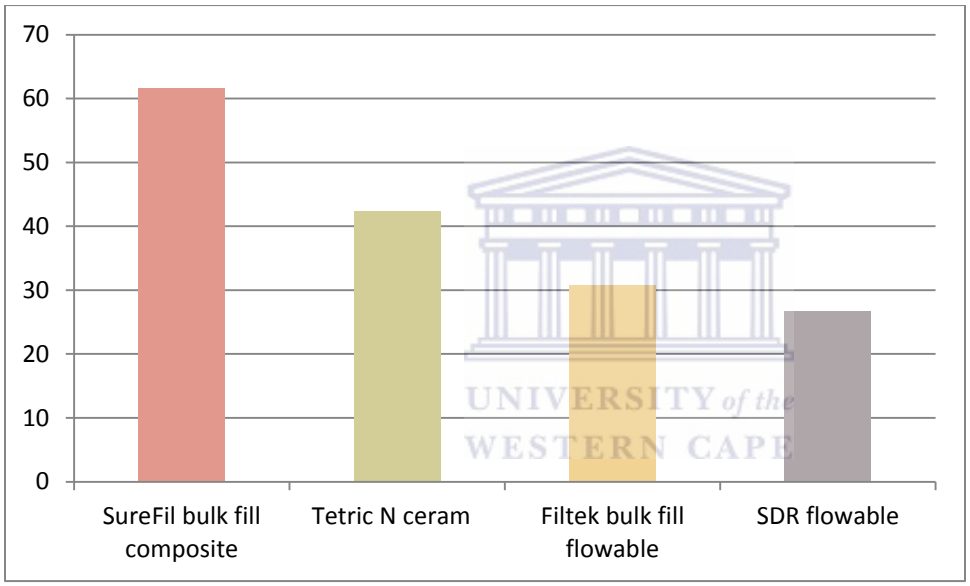
UNIVERSITY of the WESTERN CAPE

Figure 25: Comparison between the mean values of the micro-hardness (top surfaces) of the materials (immediate readings) with LED curing light.

5.2.1.1.2 Micro-hardness measurements of the top surface with QTH curing light (immediate readings)

There were statistical significant differences between the mean values (ANOVA $p<0.05$) with SureFil bulk-fill showing the greatest hardness on the top surface and the SDR flowable was the lowest (Figure 26) in the following order:

SureFil bulk-fill > Tetric N Ceram > Filtek bulk-fill flowable > SDR flowable.



UNIVERSITY of the WESTERN CAPE

Figure 26: Comparison between the mean values of the micro-hardness (top surfaces) of the materials (immediate readings) with QTH curing light.

5.2.1.1.3 Comparison of micro-hardness between LED & QTH top surface (immediate readings)

All materials showed a statistically significant difference ($p<0.05$) between the immediate readings taken by the LED curing light and QTH curing light except for SDR where there was no statistical difference between the LED and QTH. In general, LED showed higher hardness values than QTH except for Tetric N Ceram where the hardness values with QTH was more than the LED and the difference was significant, $p>0.05$ (Figure 27).

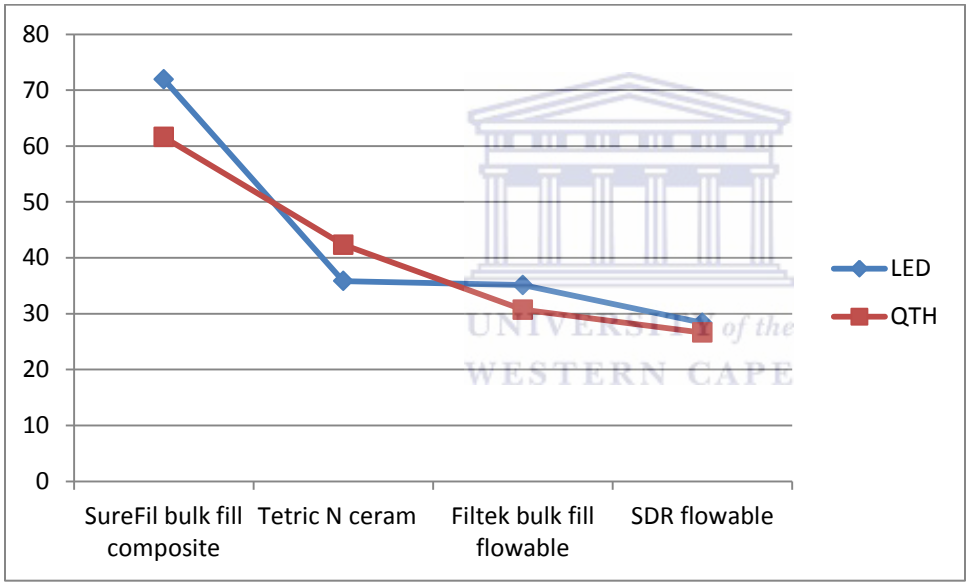


Figure 27: Comparison of the mean values of micro-hardness between LED and QTH curing light top surface (immediate reading).

5.2.1.2 Micro-hardness measurements of bottom surface (immediate reading)

Overall, there were statistically significant differences between the mean values of micro-hardness between the materials on the bottom surface ($p<0.05$). However, between the two curing lights for Tetric N Ceram, there was no significant difference between LED curing light and QTH curing light in micro-hardness values. The mean values, the standard deviation and the results of the ANOVA test were represented in (Table 5).

Material	LED	SD	QTH	SD	P-value(LED vs QTH)
Filtek bulk-fill flowable	20.20	1.73	14.67	1.68	<0.0001
SDR flowable	14.33	0.84	12.18	1.23	<0.0001
SureFil bulk-fill composite	34.56	4.01	23.78	9.01	<0.0001
Tetric N Ceram	17.66	1.89	17.33	1.91	=0.5837

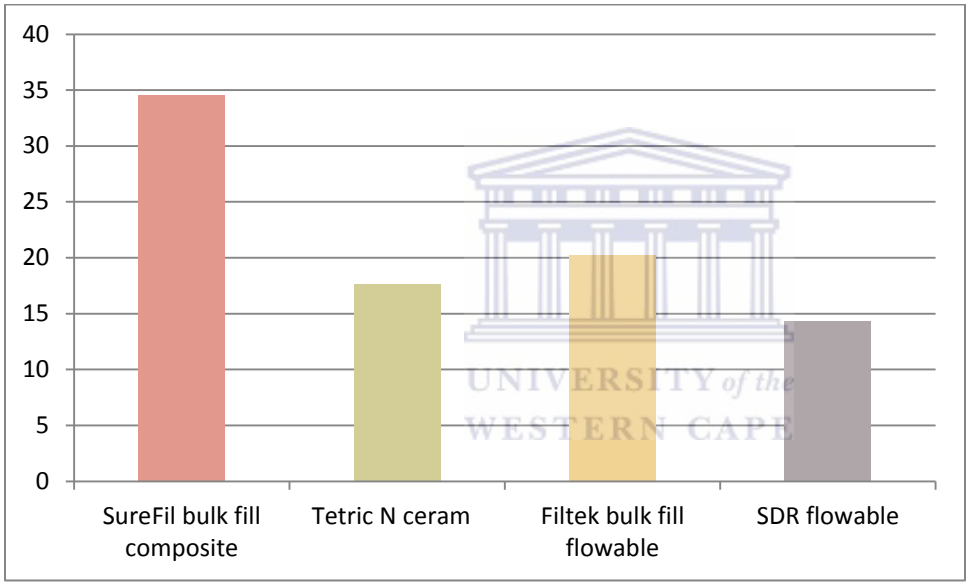
UNIVERSITY of the
WESTERN CAPE

Table 5: Bottom surface micro hardness comparison across groups (immediate readings)

5.2.1.2.1 Micro-hardness measurements of bottom surface with LED curing light (immediate readings)

There were statistically significant differences between the mean values $p<0.05$ (ANOVA). SureFil bulk-fill showed the greatest hardness values while SDR flowable showed the lowest hardness value (Figure 28) in the following order:

SureFil bulk-fill > Filtek bulk-fill flowable > Tetric N Ceram > SDR flowable.



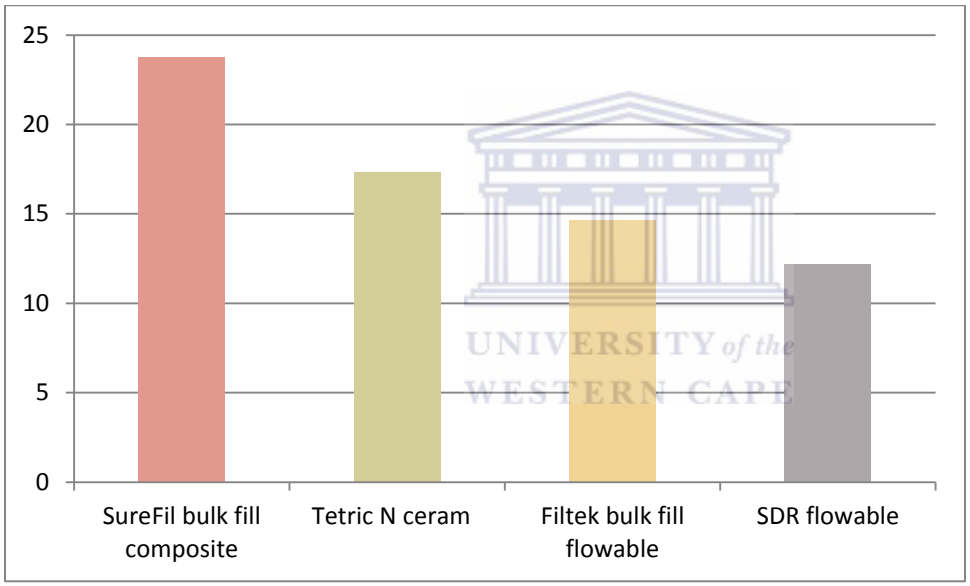
UNIVERSITY of the WESTERN CAPE

Figure 28: Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (immediate readings) with LED curing light.

5.2.1.2.2 Micro-hardness measurements of bottom surface with QTH curing light (immediate readings)

SureFil bulk-fill showed the greatest hardness values and SDR flowable showed the lowest hardness value as show below (Figure 29). However, there was no significance different between Filtek bulk-fill flowable, SDR flowable and Tetric N Ceram. The hardness values of the materials were in the following order:

SureFil bulk-fill > Tetric N Ceram= Filtek bulk-fill flowable= SDR flowable.

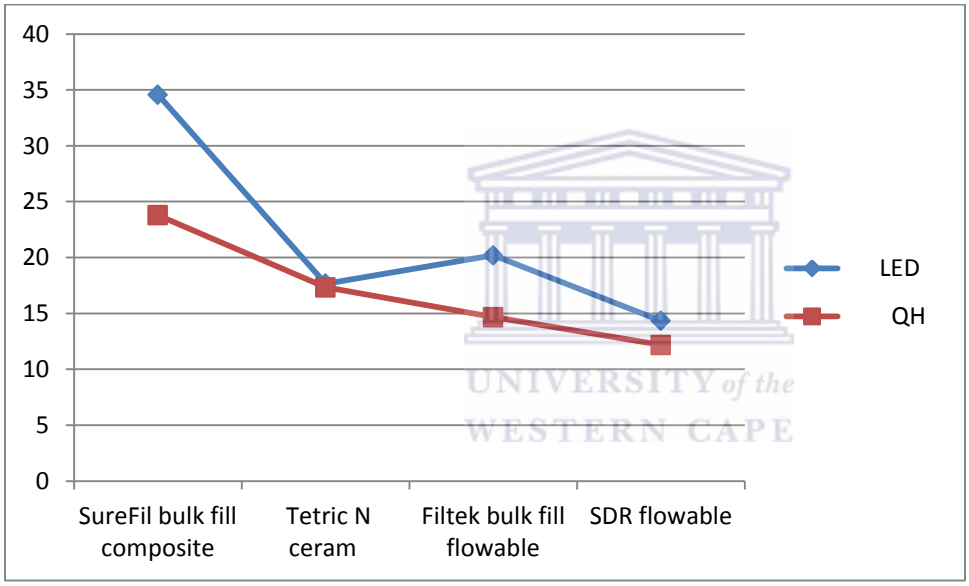


UNIVERSITY of the WESTERN CAPE

Figure 29: Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (immediate readings) with QTH curing light.

5.2.1.2.3 Comparison of micro-hardness between LED&QTH curing lights bottom surface (immediate readings)

There were statistically significance differences ($p<0.05$) between the immediate readings taken by the LED curing light and QTH curing light for the Filtek bulk-fill flowable, SDR flowable, and SureFil bulk-fill where the LED showed higher hardness values than QTH. However, there was no difference between the two curing lights for Tetric N Ceram (figure 30).



UNIVERSITY of the WESTERN CAPE

Figure 30: Comparison of the mean values of micro-hardness between LED and QTH curing light bottom surface (immediate reading).

5.2.1.3 Top surface: Measurement of micro-hardness of top surface (after 24h reading)

There were significant differences between the mean values. The mean values, the standard deviation and the results of the ANOVA test were represented in (Table 6).

Material	LED	SD	QTH	SD	p-value (LED vs QH)
Filtek bulk-fill flowable	39.76	3.45	33.12	1.75	<0.0001
SDR flowable	32.36	1.96	29.81	1.55	<0.0001
SureFil bulk-fill composite	79.15	3.78	76.23	3.68	=0.0181
Tetric N ceram	46.20	2.16	51.06	1.53	<0.0001

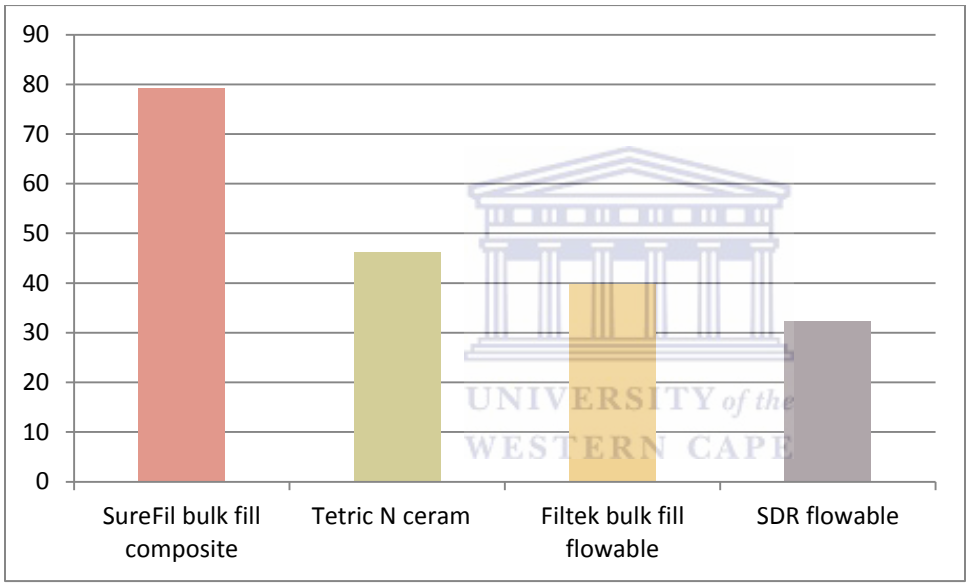
Table 6: Top surface micro-hardness comparison across groups (after 24 hours readings)

UNIVERSITY of the
WESTERN CAPE

5.2.1.3.1 24hr Readings: Micro-hardness measurements of top surface with LED curing light after 24hrs

There were significant differences between the mean values of micro-hardness measurements of top surface with LED curing light after 24 hour readings. SureFil bulk-fill showed the greatest hardness values and SDR flowable showed the lowest values (Figure 31) in the following order:

SureFil bulk-fill > Tetric N Ceram > Filtek bulk-fill flowable > SDR flowable



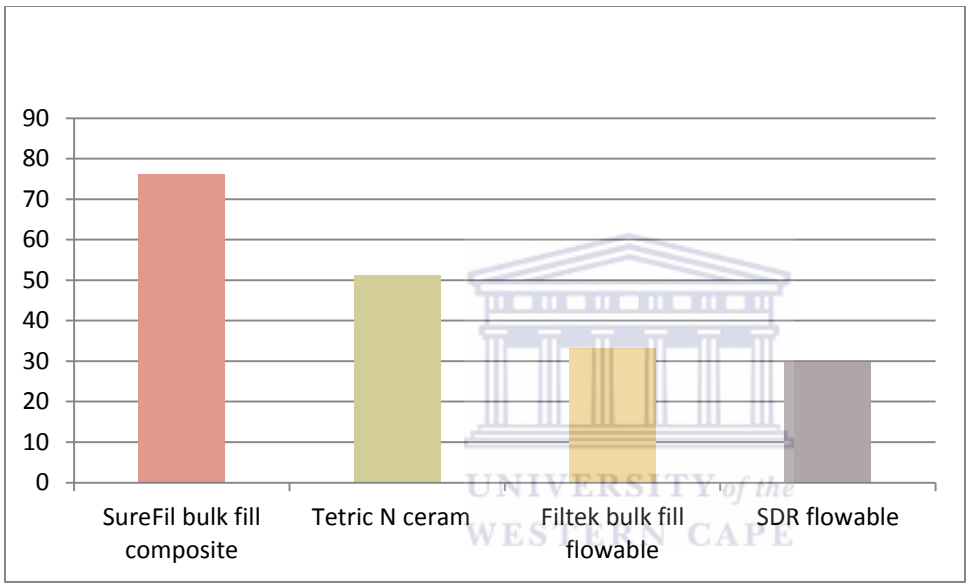
UNIVERSITY of the WESTERN CAPE

Figure 31: Comparison between the mean values of the micro-hardness (top surfaces) of the materials after 24 hours readings with LED curing light.

5.2.1.3.2. Micro-hardness measurements of top surface with QTH curing light after 24hrs

There were significant differences between the mean values of micro-hardness measurements of top surface with QTH curing light after 24 hour readings. SureFil bulk-fill showed the greatest hardness values and SDR flowable showed the lowest (Figure 32) in the following order:

SureFil bulk-fill > Tetric N Ceram > Filtek bulk-fill flowable> SDR flowable



UNIVERSITY of the WESTERN CAPE

Figure 32: Comparison between the mean values of the micro-hardness (top surfaces) of the materials after 24 hours readings with OTH curing light.

5.2.1.3.3 Comparison of micro-hardness between LED and QTH curing lights top surface after 24 hrs

The four materials showed statistically significant difference for the micro-hardness ($p<0.05$) after 24 hours with the LED curing light and QTH curing light. LED showed higher hardness values than QTH except Tetric N Ceram where the QTH showed higher hardness values than LED (Figure 33). This followed the same pattern observed when the immediate readings were taken.

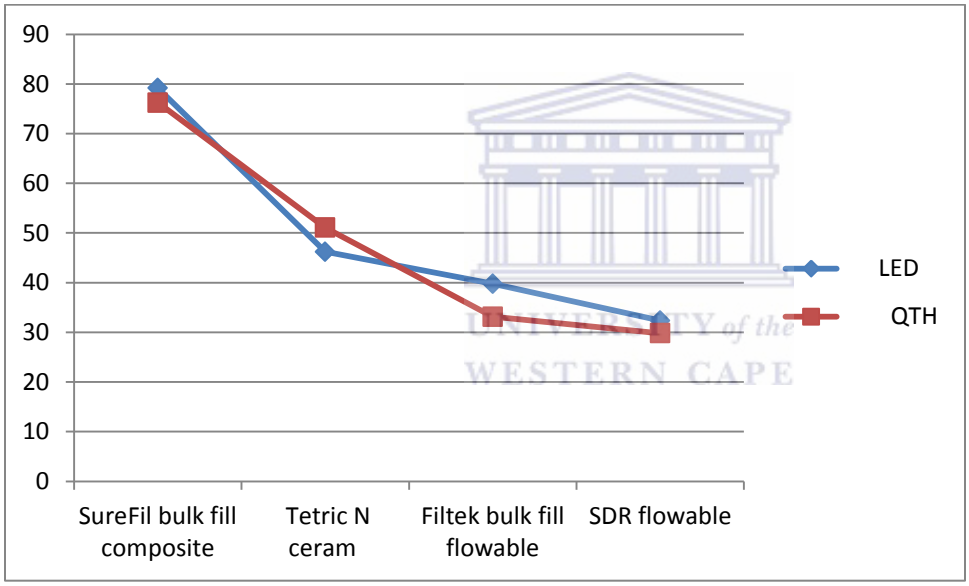


Figure 33: Comparison of the mean values of micro-hardness between LED and QTH curing light top surface after 24 hours reading.

UNIVERSITY of the WESTERN CAPE

6.2.1.4 Bottom surface: Micro-hardness measurements of bottom surface after 24h reading

There were significant differences between the mean values. The mean values, the standard deviation and the results of the ANOVA test were represented in (Table 7).

Material	LED	SD	QTH	SD	p-value (LED vs QH)
Filtek bulk-fill flowable	27.87	2.58	19.89	2.04	<0.0001
SDR flowable	22.30	1.73	17.80	2.04	<0.0001
SureFil bulk-fill composite	36.40	3.01	25.50	4.43	<0.0001
Tetric N Ceram	24.48	2.35	22.25	1.75	=0.0017

Table 7: Bottom surface micro hardness comparison across groups after 24 hours readings.

UNIVERSITY of the
WESTERN CAPE

5.2.1.4.1. Micro-hardness measurements of bottom surface with LED curing light after 24hrs

There were significant differences between the mean values. SureFil bulk-fill showed the greatest hardness values and SDR flowable showed the lowest (Figure 34) in the following order: SureFil bulk-fill > Filtek bulk-fill flowable > Tetric N Ceram > SDR flowable.

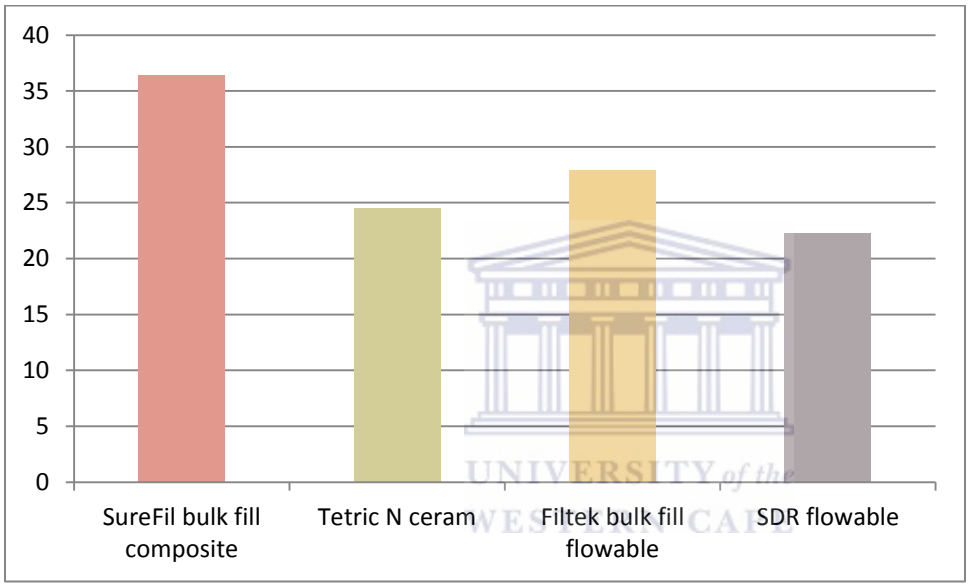
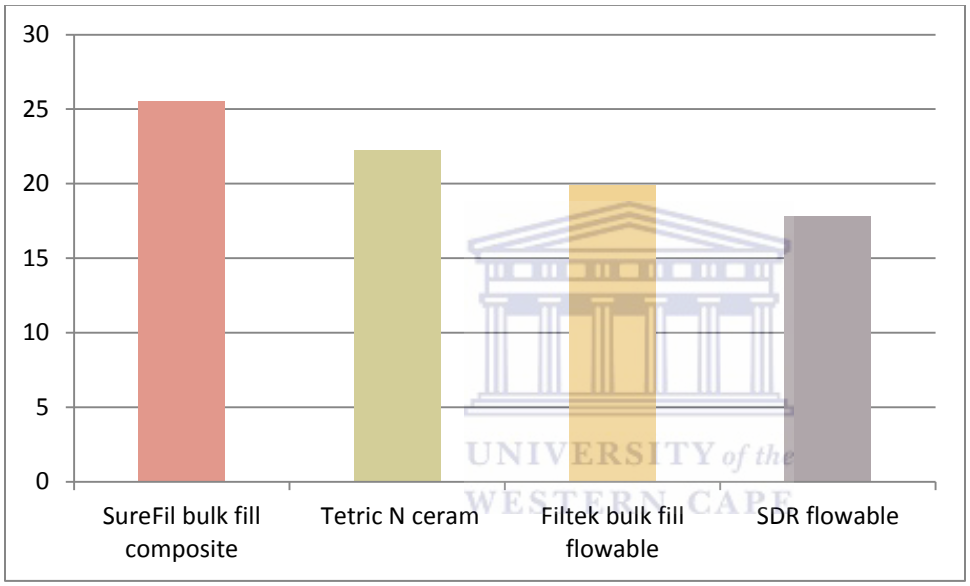


Figure 34: Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (after 24 hours readings) with LED curing light.

5.2.1.4.2 Micro-hardness measurements of bottom surface with QTH curing light after 24hrs

There were significant differences between the mean values. SureFil bulk-fill showed the highest hardness values and SDR flowable showed the lowest (Figure 35) in the following order:

SureFil bulk-fill > Tetric N Ceram > Filtek bulk-fill flowable > SDR flowable.

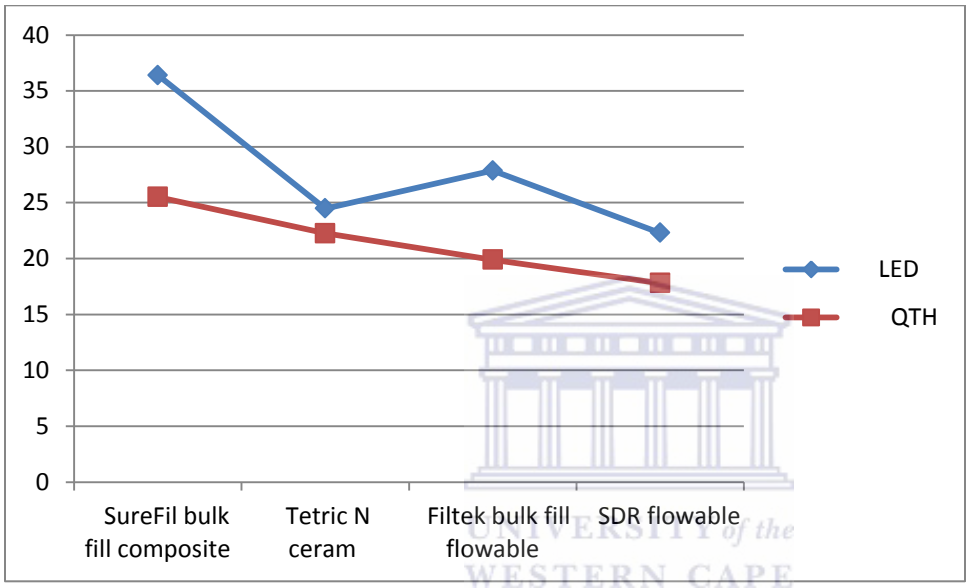


UNIVERSITY of the WESTERN CAPE

Figure 35: Comparison between the mean values of the micro-hardness (bottom surfaces) of the materials (after 24 hours readings) with QTH curing light.

5.2.1.4.3 Comparison between LED & QTH curing lights bottom surface after 24 hours

The four materials showed statistically significant difference ($p<0.05$) after 24 hours between the LED curing light and QTH curing light, where LED showed higher hardness values than QTH except for Tetric N Ceram where there was no significant difference between the LED and QTH curing light .



UNIVERSITY of the WESTERN CAPE

Figure 36: Comparison of the mean values of micro-hardness between LED and QTH curing light bottom surface after 24 hours reading.

5.2.1.5 Comparison between immediate vs. 24h readings for the micro-hardness - top surface

There were statistically significant differences in the hardness values of the top surfaces for both curing lights between the immediate readings and after 24 hours (Figure 37 & Figure 38).

5.2.1.5.1 Comparison of immediate and after 24 hours readings for the top surface with LED curing light

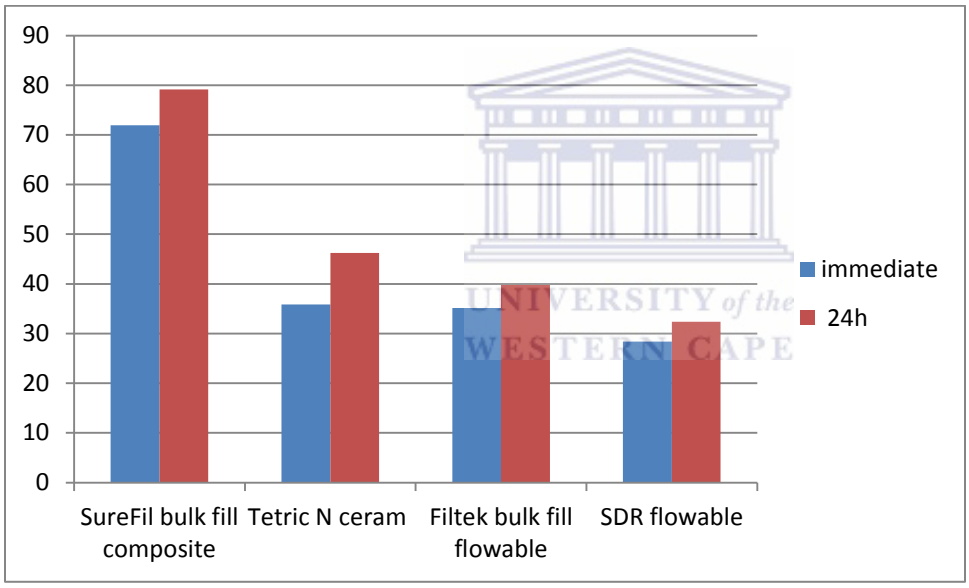


Figure 37: Comparison between the immediate and after 24 hours readings of the hardness values for LED.

UNIVERSITY of the WESTERN CAPE

6.2.1.5.2 Comparison of immediate and after 24 hours readings for the top surface with QTH curing light

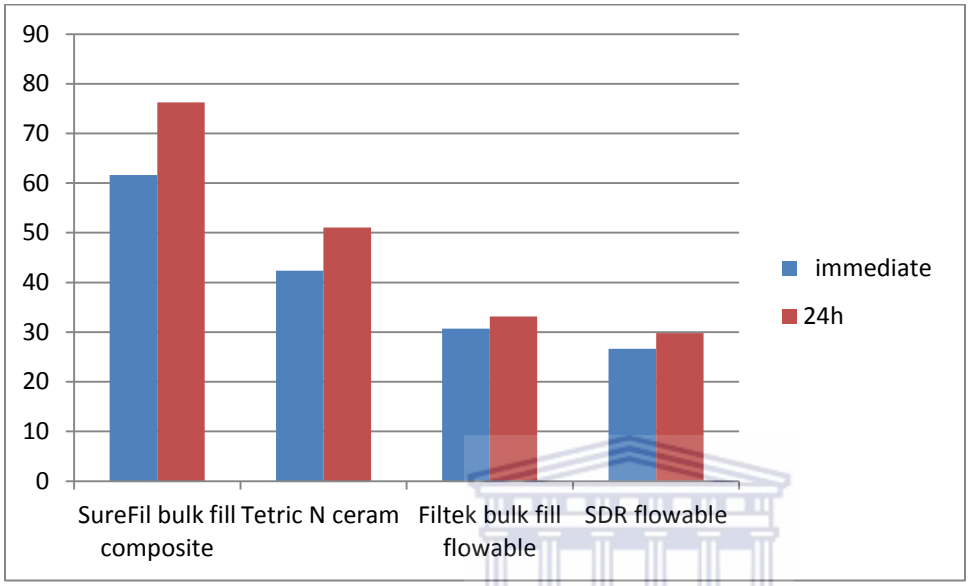


Figure 38: Comparison between the immediate and after 24 hours readings of the hardness values for QTH top surface.

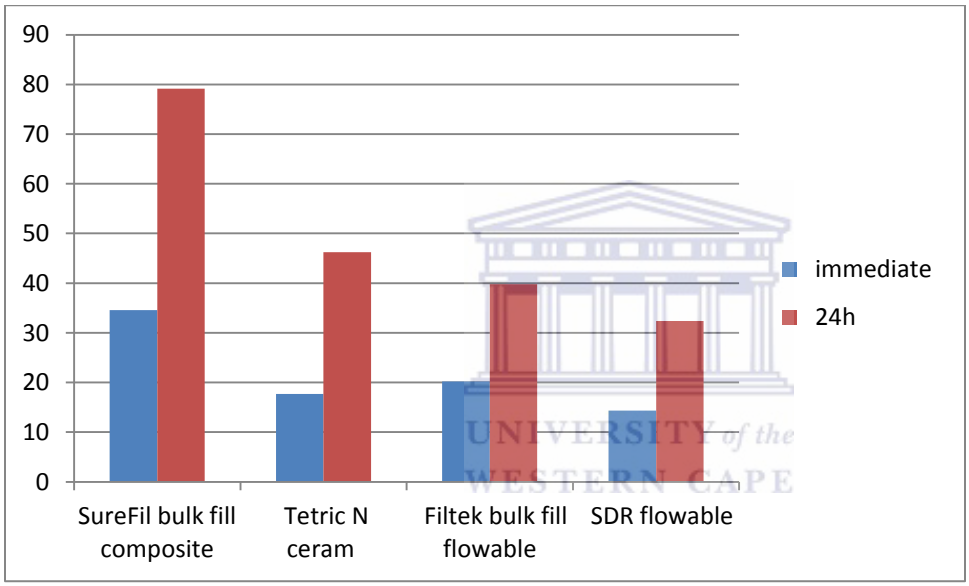
For all materials and for both curing lights the surface hardness of the materials increased after 24 hours on the top surface.

UNIVERSITY of the WESTERN CAPE

5.2.1.6 Immediate vs. 24h readings for the micro-hardness bottom surface

There were statistically significant differences in the hardness values of the bottom surfaces for both curing lights between the immediate readings and after 24 hours.

5.2.1.6.1 Comparison of immediate and after 24 hours readings for the bottom surface with LED curing light



UNIVERSITY of the WESTERN CAPE

Figure 39: Comparison between the immediate and after 24 hours readings of the hardness values for LED (bottom surface).

5.2.1.6.2 Comparison of immediate and after 24 hours readings for the bottom surface with QTH curing light

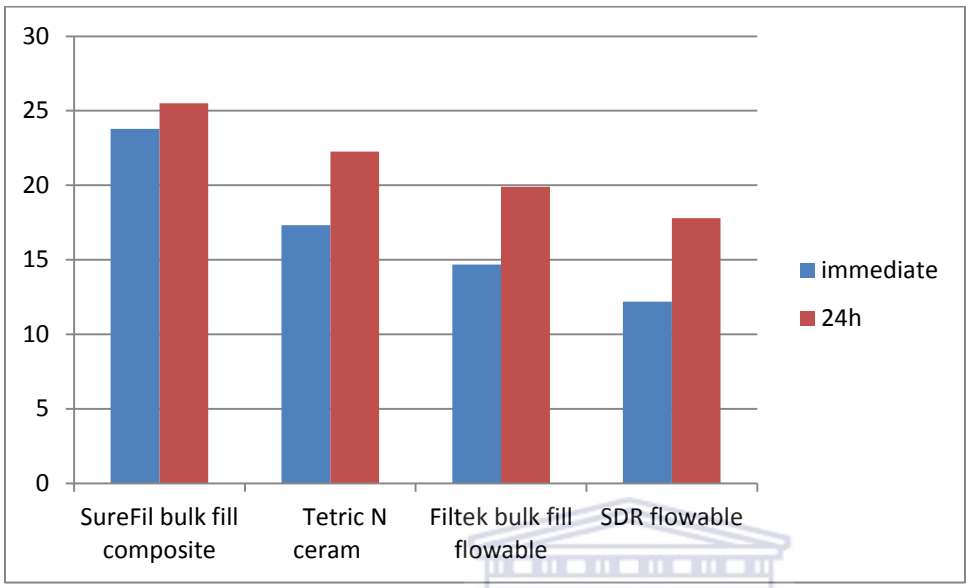


Figure 40: Comparison between the immediate and after 24 hours readings of the hardness values for QTH (bottom surface).

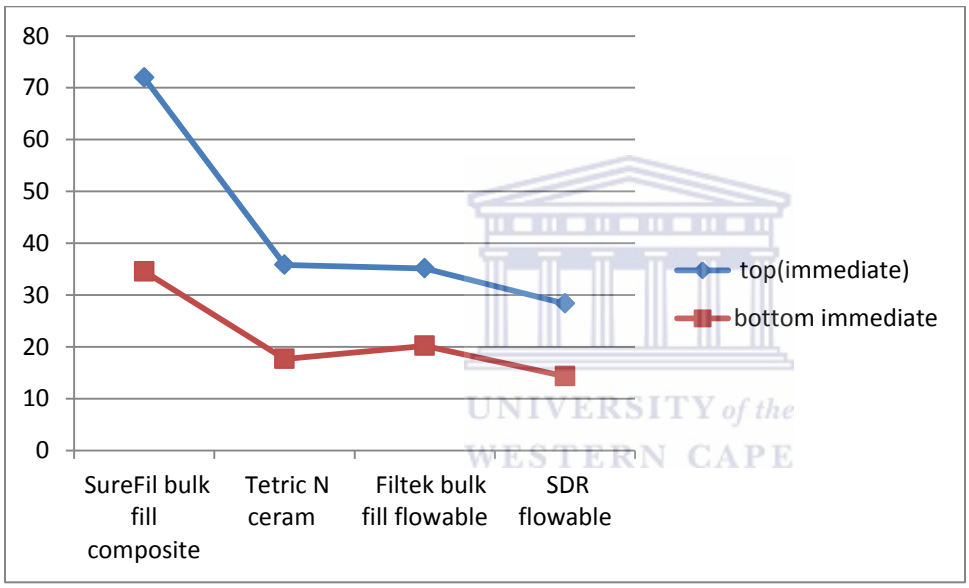
UNIVERSITY of the WESTERN CAPE

The hardness values of the bottom surface for all materials increased after 24hrs for both curing lights (figure 40).

5.2.1.7 Top vs bottom immediate readings for micro-hardness

When the top and bottom surfaces were compared there were statistically significant differences between the hardness values of the top and the bottom surface for both curing lights where the top surface showed the greatest values for both curing lights (figure 41, 42).

Top vs bottom immediate readings for micro-hardness with LED curing light



UNIVERSITY of the WESTERN CAPE

Figure 41: Comparison between the micro-hardness of the top and bottom surface (immediate readings) with LED.

Top vs bottom immediate readings for micro-hardness with QTH curing light

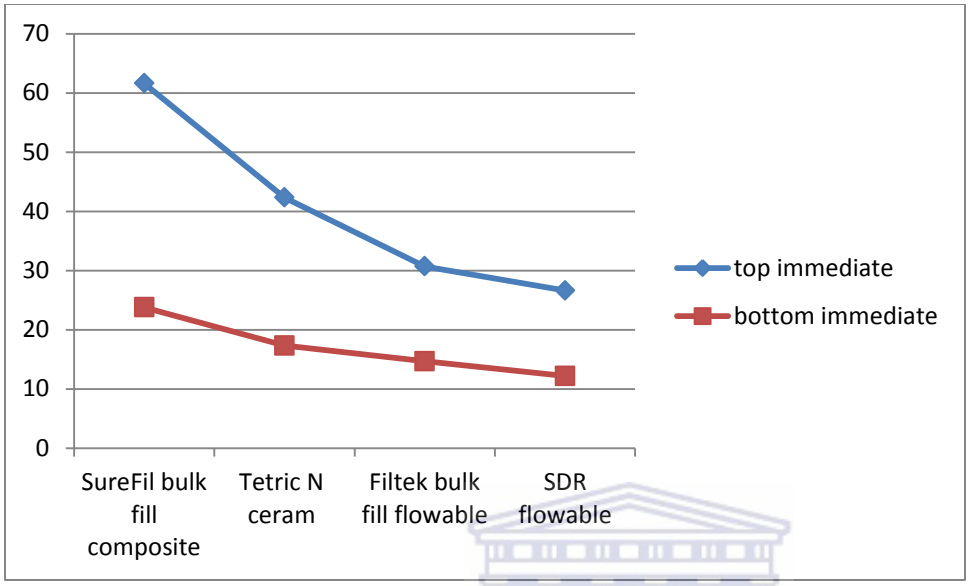


Figure 42: Comparison between the micro-hardness of the top and bottom surface (immediate readings) with QTH.

UNIVERSITY of the
WESTERN CAPE

5.2.1.8 Top vs bottom after 24 hours readings for micro-hardness

There were statistical significant differences between the values. The values obtained for the bottom surface were found to be lower than those of the top surface for both curing lights after 24hrs (figure43, 44).

Top vs bottom after 24 hours readings for micro-hardness with LED curing light

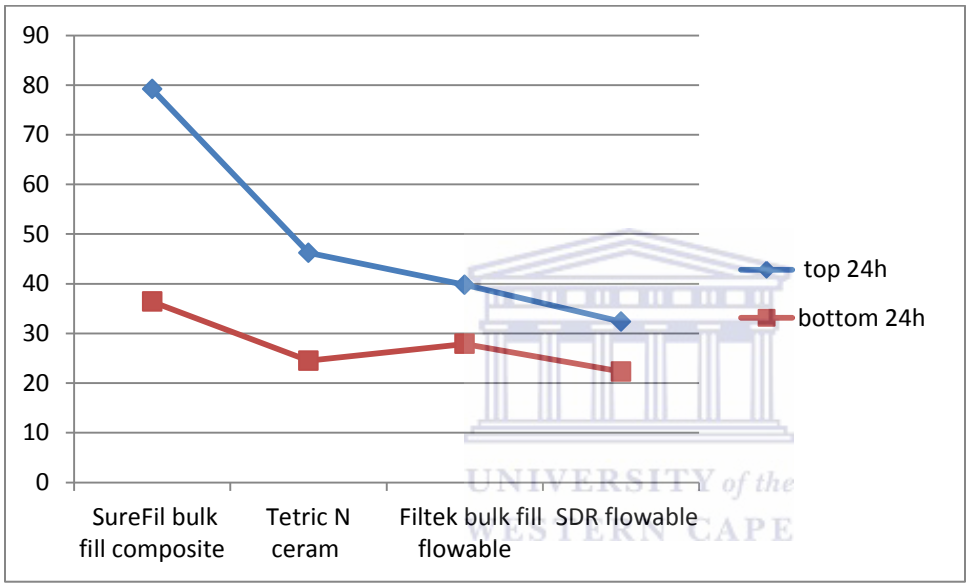


Figure 43: Comparison between the micro-hardness of the top and bottom surface (after 24 hours readings) with LED.

UNIVERSITY of the WESTERN CAPE

Top vs bottom after 24 hours readings for micro-hardness with QTH curing light

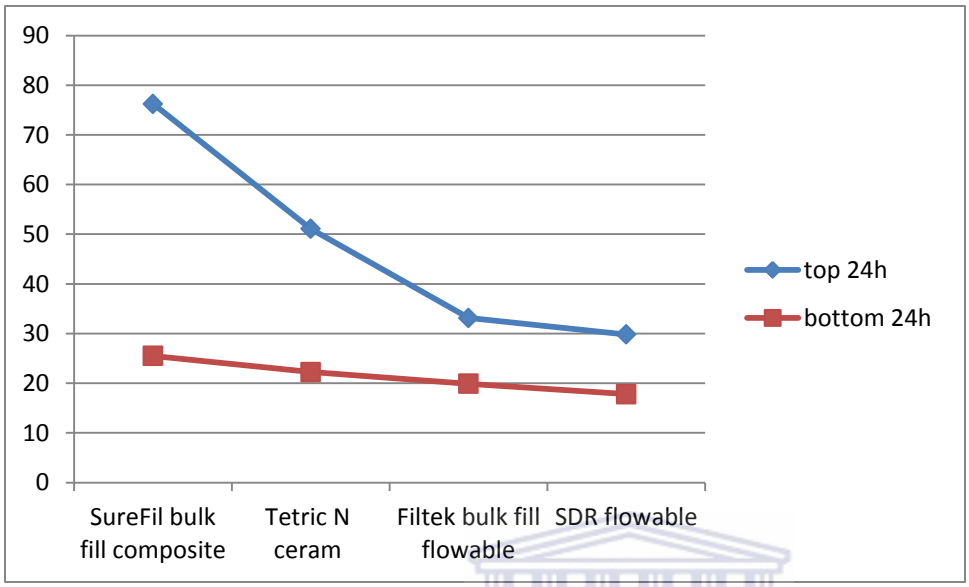


Figure 44: Comparison between the micro-hardness of the top and bottom surface (after 24 hours readings) with QTH.

UNIVERSITY of the
WESTERN CAPE

5.2.2 Depth of cure measurements:

The micro-hardness mean values obtained were used to calculate a bottom/top hardness ratio. A ratio above 80% has often been suggested as a minimum acceptable threshold value (Watts et al., 1984). It is clinically acceptable that the bottom surface can be about 80% of the upper hardness value as the minimum depth of cure.

5.2.2.1 Depth of cure- bottom to top ratio immediate readings

There were significant differences ($p<0.05$) between the mean values of depth of cure. The mean values of depth of cure, the standard deviation and the results of the ANOVA test were represented in (Table 8).

UNIVERSITY of the WESTERN CAPE

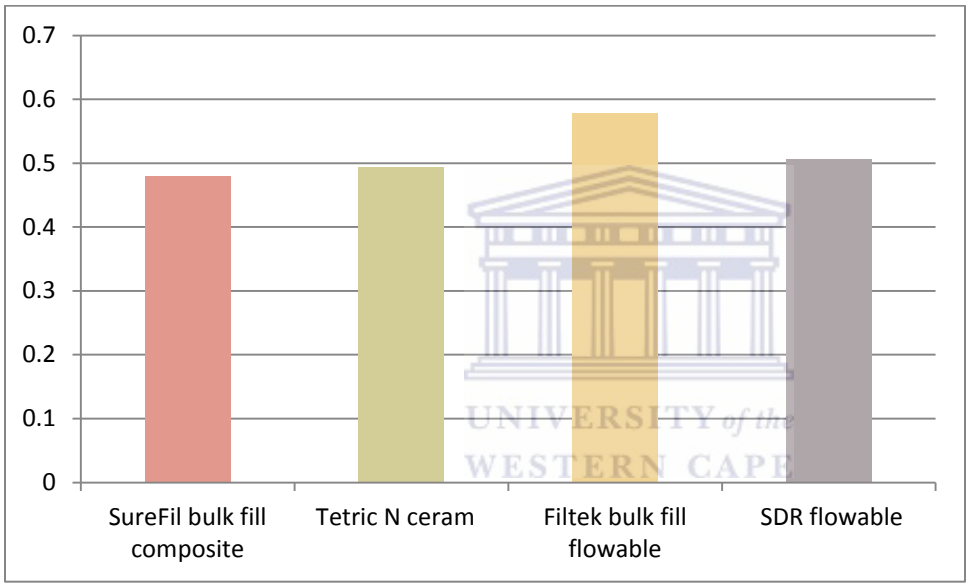
Material	LED	SD	QH	SD	P-value(LED vs QH)
Filtek bulk-fill flowable	0.57	0.06	0.47	0.05	<0.0001
SDR flowable	0.50	0.03	0.45	0.04	=0.0008
SureFil bulk-fill composite	0.46	0.04	0.33	0.15	= 0.0192
Tetric N Ceram	0.49	0.04	0.41	0.04	<0.0001

Table 8: Bottom to top ratio comparison across group's immediate readings.

5.2.2.1.1 Depth of cure measurements immediate readings with LED curing light

There was significant differences p-value < 0.0001 between the mean values when Filtek flowable was compared. Filtek bulk-fill flowable showed the highest depth of cure and SureFil bulk-fill showed the lowest (Figure 45). However there was no significant difference between SDR flowable, SureFil bulk-fill and Tetric N Ceram in the following order:

Filtek bulk-fill flowable > SDR flowable = Tetric N Ceram = SureFil bulk-fill.



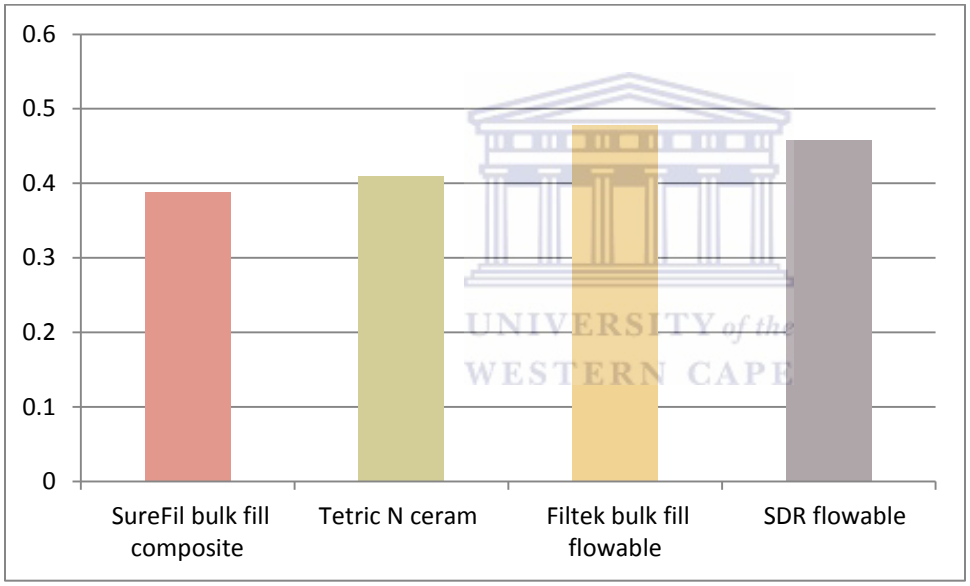
UNIVERSITY of the WESTERN CAPE

Figure 45: Depth of cure values for the different materials with LED curing light.

5.2.2.1.2 Depth of cure measurements immediate readings with QTH curing light

There was significant difference p-value = 0.0064 between the mean values for Filtek flowable compared to the other materials. Filtek bulk-fill showed the greatest depth of cure values and SureFil bulk-fill was the lowest (Figure 46). However, there was no significant difference between Filtek bulk-fill, SDR flowable, Tetric N Cearm in the following order:

Filtek bulk-fill flowable> SureFil bulk-fill; Filtek bulk-fill flowable = SDR flowable = Tetric N Ceram.



UNIVERSITY of the WESTERN CAPE

Figure 46: Depth of cure values for the different materials with QTH curing light (immediate readings).

5.2.2.1.3 Comparison of depth of cure immediate readings between LED &QTH curing light

There was statistically significant difference ($p<0.05$) between the LED curing light and QTH curing light immediate readings for depth of cure. Filtek bulk-fill flowable and Tetric N Ceram showed greatest values of depth of cure with LED than QTH. However, there was no significant difference between the two curing light in the depth of cure values with SDR flowable and the SureFil bulk-fill (figure 47).

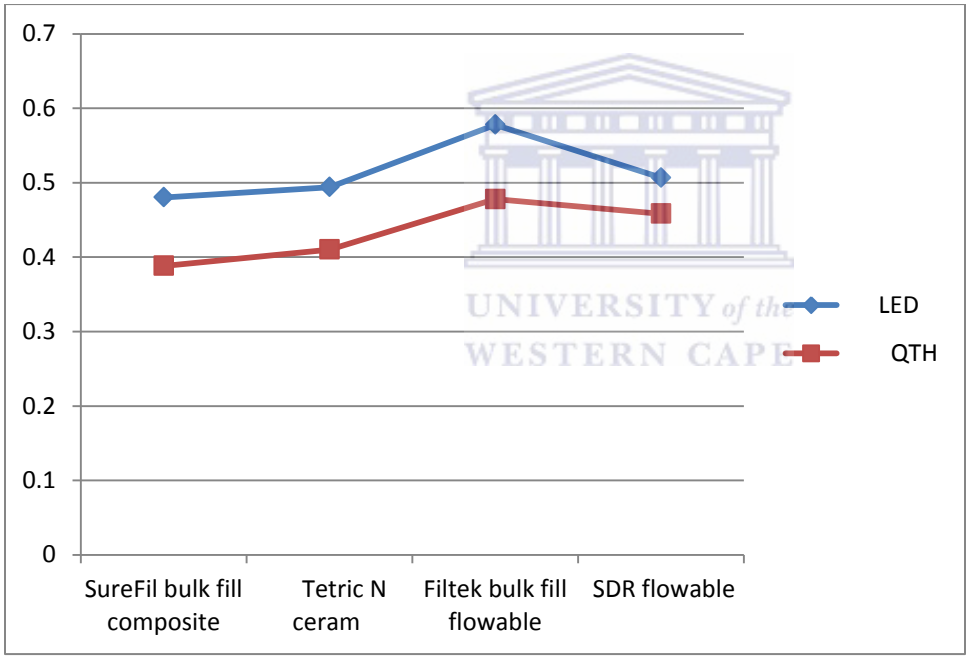


Figure 47: Comparison of depth of cure values between the two curing lights immediate readings.

UNIVERSITY of the WESTERN CAPE

5.2.2.2 Measurement of depth of cure after 24h reading

There were significant differences ($p<0.05$) between the mean values. The mean values, the standard deviation and the results of the ANOVA test were represented in (Table 9).

Material	LED	SD	QH	SD	P-value(LED vs QH)
Filtek bulk-fill flowable	0.70	0.07	0.59	0.04	<0.0001
SDR flowable	0.68	0.03	0.59	0.05	<0.0001
SureFil bulk-fill composite	0.48	3.01	0.38	0.06	<0.0001
Tetric N Ceram	0.53	0.04	0.43	0.03	<0.0017

Table 9: Bottom to top ratio comparison across groups (after 24 hours readings)

UNIVERSITY of the
WESTERN CAPE

5.2.2.2.1 Measurement of depth of cure after 24h with LED curing light

There was significant difference p-value <0.0001 between the mean values. Filtek bulk-fill and SDR flowable showed the highest depth of curing and SureFil bulk-fill showed the lowest values (Figure 48). However, there was no significant different between Filtek bulk-fill and SDR flowable in the following order:

Filtek bulk fill flowable = SDR flowable > Tetric N Ceram > SureFil bulk fill.

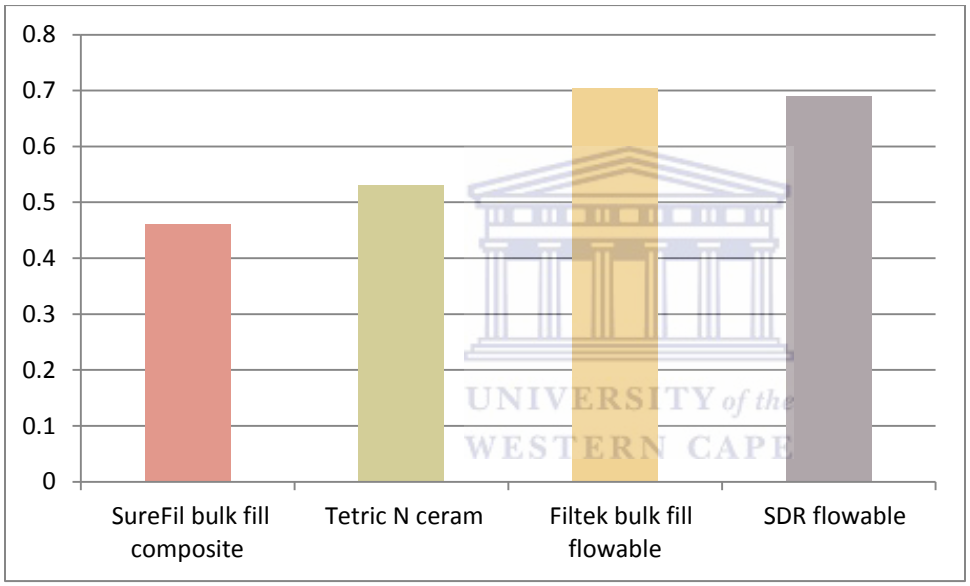


Figure 48: Depth of cure values for the different materials with LED curing light after 24 hours readings.

UNIVERSITY of the WESTERN CAPE

5.2.2.2.2 Measurement of depth of cure after 24h with QTH curing light

There was significant difference p-value <0.0001 between the mean values. Filtek bulk-fill and SDR flowable showed the greatest depth of cure and SureFil bulk-fill showed the lowest values (Figure 49). However, there was no significant different between Filtek bulk-fill and SDR flowable.

Filtek bulk-fill flowable = SDR flowable > SDR flowable > SureFil bulk-fill.

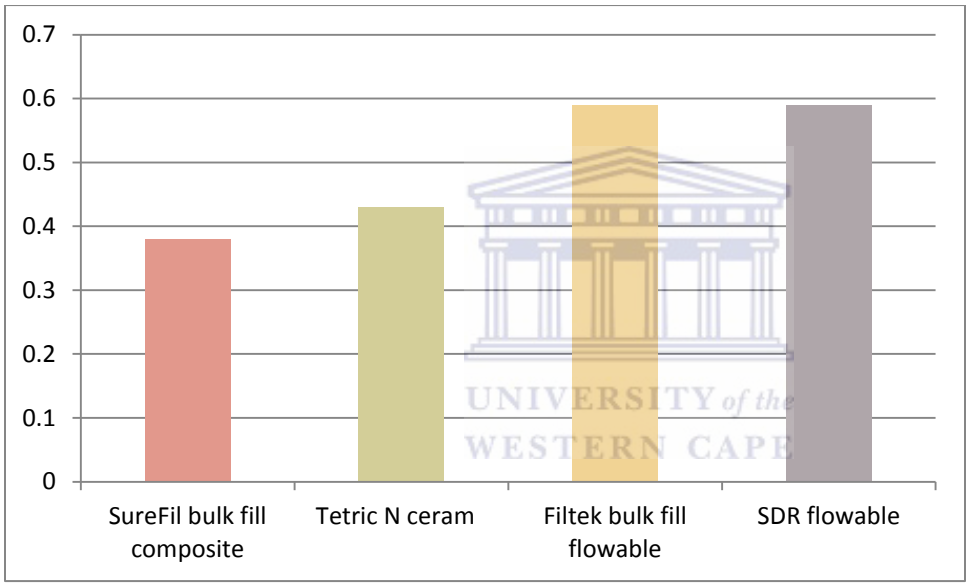
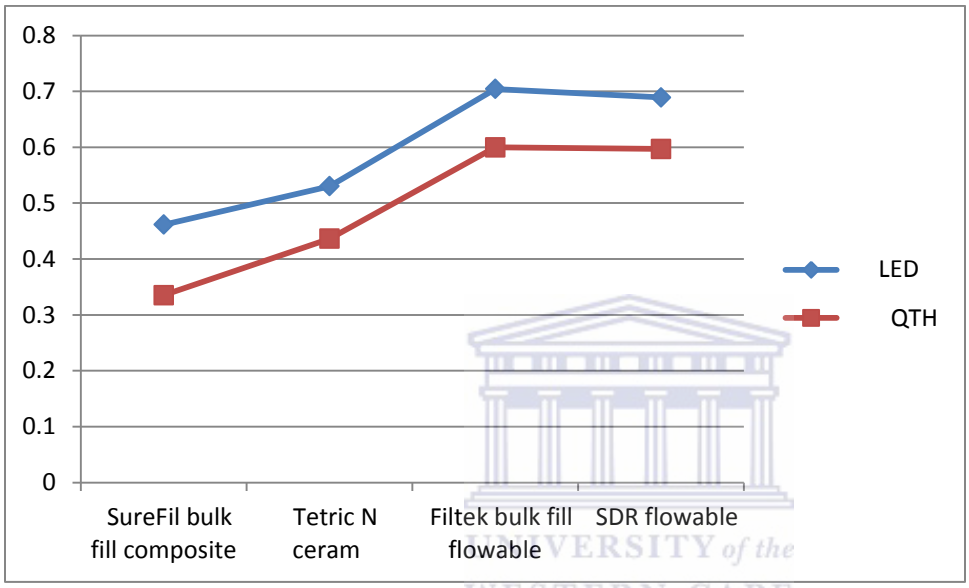


Figure 49: Depth of cure values for the different materials with QTH curing light after 24 hours readings.

UNIVERSITY of the WESTERN CAPE

5.2.2.2.3 Comparison of depth of cure after 24 hours between LED & QTH curing

All materials showed statistically significant differences ($p<0.05$) between the 24h readings taken by the LED curing light and QTH curing light, where LED showed higher depth of cure values than QTH**.**



UNIVERSITY of the WESTERN CAPE

Figure 50: Comparison of depth of cure values between the two curing lights (after 24 h readings).

5.2.2.3 Depth of cure - immediate vs 24hours

There were statistical significant differences between the values. The values obtained immediately were found to be lower than those of after 24 hours for both curing lights. However, there was no significant difference between the two readings for SureFil bulk-fill and Tetric N Ceram).

Comparison between the immediate and 24 hours readings of depth of cures With LED curing light

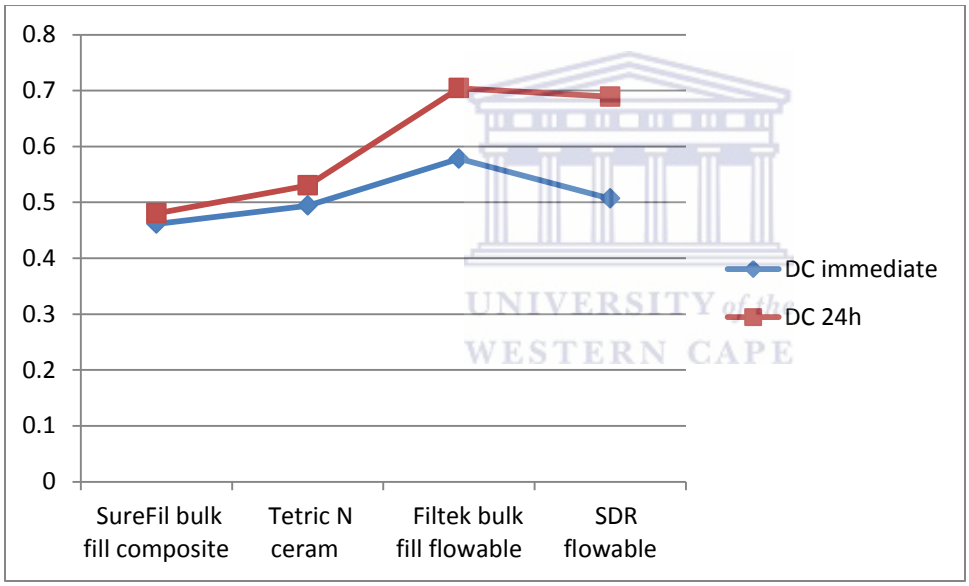


Figure 51: Comparison between the immediate and the 24h readings for LED

UNIVERSITY of the WESTERN CAPE

Comparison between the immediate and 24 hours readings of depth of cure With QTH curing light

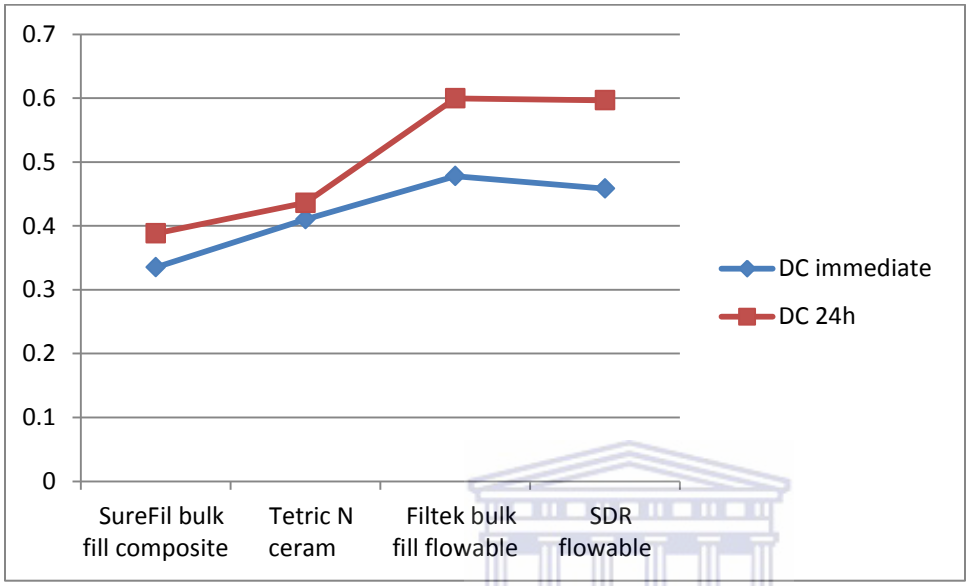


Figure 52: Comparison between the immediate and the 24h readings for QTH

UNIVERSITY of the
WESTERN CAPE

Chapter 6

Discussion

6.1 Introduction

Surface micro-hardness and depth of cure are considered to be important physical properties of resin composites and play a significant role in comparing and characterizing dental restorative materials. Micro-hardness is commonly used for evaluation of the depth of cure and thus, also the efficiency of the curing lights (Yaman et al., 2011). The possibility of insufficient monomer conversion and the limitations of depth of curing are one of the problems associated with photo-polymerized resin composites (Yaman et al., 2011). Depth of cure measurements has shown clear relevance to the clinical aspects of composite curing (Hubbezoglu et al., 2007). Evaluating the degree of conversion is considered to be a reliable method however; there is a high correlation between this method and the micro-hardness evaluation (Hubbezoglu et al., 2007). With the newer bulk-fill composites that are advocated to be placed in increments of 4mm may pose a problem with regards to depth of cure.

UNIVERSITY of the
WESTERN CAPE

Based on the viscosity of these newer bulk-fill composites, they can be further divided into two categories, namely low viscosity bulk-fill flowable composite and conventional viscosity bulk-fill material. The changes made in bulk-fill resin based composites to increase the depth of cure are basically represented by the fillers which are generally increased in size in all materials and with a decrease in load of fillers in the flowable bulk-fill composites (Ilie et al., 2013). Also, adding higher molecular weight monomers as in SDR, a modified UDMA, which is claimed to reduce polymerization shrinkage or new photo initiator, Ivocerin as in Tetric N Ceram attempted towards increasing the depth of cure.

6.2 Evaluation of micro-hardness

The micro-hardness achieved in the present study on the top surface of bulk-fill composite specimens was higher compared to the bottom surface in all experimental groups. This may be due to the reduction of light as it travels through the composite material (Halvorson, Erickson & Davidson, 2002) or may be due to light scatter through the filler particles.

On the top surface, SureFil bulk-fill composite and Tetric N Ceram showed significantly higher micro-hardness values than the other materials (Filtek bulk-fill, SDR) indicating that the conventional bulk-fill composites have a higher surface hardness than the flowable bulk-fill composites. SureFil bulk-fill had the highest value and SDR showed the lowest surface hardness values. This variation of micro-hardness values was expected as SureFil composite and Tetric N Ceram contain a high load of filler particles as shown in table 2. This result is similar to that of Ilie et al. (2011) where SDR exhibited the lowest Vickers hardness values. Similarly, another study raised a concern regarding the low micro-hardness values for some bulk-fill composite especially SDR and Filtek bulk-fill (Ilie et al., 2013). Leprince et al. (2014) concluded that some of bulk-fill composites like SDR and Filtek bulk-fill displayed very low Vickers micro-hardness values. Also, Flury et al. (2012) found that SDR showed low hardness values.

UNIVERSITY of the
WESTERN CAPE

Moreover, De Biasi et al. (2010) reported the low hardness values for SDR especially after storing in artificial saliva. Low micro-hardness values usually lead to poor wear resistance, which can compromise the strength of the material and lead to failure of the restoration (Moore et al., 2008). Thus, the manufacturer's recommend that these materials to be finished with a resin composite by adding a layer of conventional composites as a “capping material” because of the lower hardness values. Also Leprince et al. (2014) suggested that veneering the bulk-fill composite with regular composite is essential and should not only be limited for aesthetic

reasons. Leprince et al. (2010) stated that there is a linear correlation between the surface micro-hardness and the filler content and this correlation was further highlighted by the results of this study.

The efficiency of curing and the micro-hardness of resin composite cannot be evaluated by study the top surface only. The bottom surface micro-hardness is more affected by light intensity and thus the effectiveness of curing (Hubbezoglu et al., 2007). Based on this, micro-hardness of the bottom surface was also measured in this study.

Irrespective of the type of the light curing unit used in this study, the bottom surface showed lower micro-hardness values than the top surfaces for all materials tested. Surefil bulk-fill showed the highest micro-hardness values and SDR flowable showed the lowest. Flury et al. (2012) measured the Vickers hardness (VH) for different composite materials at different distances ranging from 0.5mm to 13mm and found that there was a gradual decrease in micro-hardness from the top toward the bottom and this decrease differed from one type of composite to another. Similarly, Ceballos et al. (2009) reported a decrease in micro-hardness values with increased thickness of the composite restoration. Leprince et al. (2014) reported that Tetric N Ceram bulk-fill presented Vickers hardness values similar to the conventional Tetric N Ceram (VH 50) which was similar to the micro-hardness value obtained for the same material in this study (VH 46.2 with LED and VH 51 with QTH).

Alrahlah et al. (2014) stated that after curing the polymerization process of resin composites continues at a slow rate and may reach a termination point at almost 24 hours. However, some studies show surface hardness increases up to 1 month following light curing (Alrahlah et al.,

UNIVERSITY of the
WESTERN CAPE

2014; Flury et al., 2012). In this study micro-hardness was evaluated immediately following light curing and after 24 hours to determine hardness after complete curing has taken place.

There was a difference between the immediate micro-hardness values and after 24 hours. The micro-hardness values on top and bottom surfaces increased after 24 hours for both curing lights indicating the polymerization continues even after the initial light curing process.

This difference in micro-hardness between the materials can be due to composition of the organic matrix, like differences in the density of the polymer network or low filler content (as in SDR and Filtek) or increased particle size, using other photo initiators, such as Ivocerin in Tetric N Ceram, or the greater percentage of filler by weight as in SureFil.

The shade of the resin composite may also affect the micro-hardness. According to Thome et al.(2007) resin with lighter shades exhibit higher micro-hardness values in comparison with the darker shades which require more exposure time than light shades and thinner increments in order to achieve higher hardness values. Ikeda, Murata and Sano, (2004) stated that the translucency of the resin composite also has an influence on the transmission of the light through the restoration thickness. In this study, shade A2 was used for Filtek bulk-fill, Tetric N Ceram and since A2 is not available for SureFil and SDR, shade A was used for SureFil and Universal shade was used for SDR. These shades were selected to standardize this study and to minimize the effects of colour on light polymerization.

Thome et al. (2007) stated that higher micro-hardness values were obtained when the tip of the light source was in contact with the surface of the specimen. Additionally, Caldas et al. (2003) reported that the ideal distance between the light tip and the specimen's surface is 0mm i.e.

UNIVERSITY of the
WESTERN CAPE

directly contacting the surface. In this laboratory study the light tip was in contact with the specimen's surface with only the universal strip separating the tip.

7.3 Evaluation of depth of cure (bottom/top ratio)

Ilie et al. (2013) define the depth of cure "as the thickness of a resin based composite that is adequately cured or the depth where hardness equals the surface value multiplied by an arbitrary ratio, usually 0.8". Leprince et al. (2013) proposed definition for depth of cure as "the depth at which the resin matrix switches from a glassy to a rubbery state". According to research carried out by Watts, Amer, & Combe, (1984) an acceptable curing depth is achieved if the bottom hardness corresponds to at least 80% of the top surface hardness. Zorzin et al. (2015) proposed that the optimum curing could be achieved only if there is no significant decrease of micro-hardness with increasing material depth.

The manufacturers for the materials used in this study claim that the depth of cure is 5mm for SureFil and 4 mm for the remaining bulk-fill composites. In order to standardize this study the thickness of all the specimens were kept at 4mm. The results of the present study showed that there is inadequate depth of cure for all bulk-fill materials tested when used at 4mm thickness.

The evaluation of depth of cure was done immediately and after 24 hours. The difference in depth of cure was observed for all bulk-fill composites for both the light curing units and this suggests variation in the extent of polymerization of these materials. Tetric N Ceram and SureFil bulk-fill showed the highest hardness values but showed the lowest depth of cure the values. For the bulk fill flowable, SDR and Filtek flowable bulk-fill showed higher values of depth of cure than the other materials but did not meet the standard of ISO 4049 of 80% for depth of cure.

UNIVERSITY of the
WESTERN CAPE

Sobrinho et al. (2000) stated that a reduction in hardness was obtained with increased depth of the composite. Also, Tsai, Meyers and Walsh, (2004) reported that the micro-hardness of the composite will reduce with increasing depth of resin as was found in this study as well.

The results of this study is similar to that of Garcia et al. (2014) where the bottom/top ratio was below 70% at 4mm depth for SDR as well as for the other bulk-fill materials that was used in their study and they reported that using bulk-fill composite greater than 3mm depth should be questioned. These findings have been corroborated by Soygun et al. (2013) who evaluated the same product, as well as other bulk-fill composites and failed to achieve the 80% of depth of cure. Finan et al. (2013) evaluated the depth of cure indirectly by the biaxial flexure strength and found the measurement was significantly lower than 4mm for the two bulk-fill flowable composites, which included SDR.

When the depth of cure from the micro-hardness values for SDR was calculated, it was shown to be inadequate (De Biasi et al., 2010)

UNIVERSITY of the
WESTERN CAPE

Another calculation was done for the micro-hardness values in a study by Giuliano et al. (2013) and it was found that the bottom/top ratio of Filtek bulk-fill, SDR, and Tetric N Ceram did not achieve the standard depth of cure as stated by the guidelines of ISO 4049. Leprince et al. (2014) reported that the values for the degree of conversion for SDR and Filtek bulk-fill were low and not in the range of the control. The lower value of degree of conversion indicates not enough depth of curing which was confirmed by the results in this current study.

Filtek Bulk-fill flowable contains additional zirconia fillers which are said to improve mechanical properties. However, due to its high refractive index zirconia is also said to reduce the transmittance of light in the restorative materials thus may affect the depth of cure (Guo et al., 2012).

Although Tetric N Ceram contains translucent filler and matrix which allows the light to penetrate through the material, it contains the new photo initiator, Ivocerin which can absorb the light in the region between 400 and 450 nm which is considered as being high (Jang, Park & Hwang, 2015). Tetric N Ceram did not demonstrate enough depth of cure in this study. Also, Jang et al. (2015) stated that Tetric N Ceram failed to reach the 80% bottom/top ratio. Similarly, Flury et al. 2012 demonstrated the same results for the Tetric Evo Ceram which did not meet the 80% bottom to top ratio. Garoushi et al. (2013) confirmed that the maximum depth that Tetric Evo Ceram cured to was 2.3mm, which does not meet the manufacture's claim. Similar results were demonstrated by Benetti et al. (2015) regarding the Tetric Evo Ceram and the inability to achieve sufficient depth of cure.

SureFil bulk-fill showed the lowest values of depth of cure which does not meet the standard guidelines of ISO 4049. This can be explained by the high percentage of the filler which may prevent the light from penetrating through the restoration. This was confirmed by Arikawa et al. (2007), who stated that increased filler content and irregular filler shape will lead to decrease in the light transmittance in the composite, thus decrease the depth of cure.

UNIVERSITY of the
WESTERN CAPE

The possibility of inadequately curing these materials to greater than 4mm thickness was confirmed by micro-hardness scraping methods (ISO 4049) for SDR in studies by Czasch & Ilie, (2013), Filtek bulk-fill in a study by Zorzin et al. (2015) and Tetric N Ceram in a study by Ilie, KeBler & Durner, (2013) and by measurement of degree of conversion for SDR in a study by Finan et al. (2013). Furthermore, Moore et al. (2008) demonstrated that the use of ISO 4049 scraping method to assess the quality of cure in depth might lead to an overestimation of the depth of cure. Similarly, Garcia et al. (2014) study showed that the scraping method of ISO 4049 significantly overestimated depth of cure values. Hence, in this study the comparison of top to

bottom surface hardness values were used as this may more accurately represent depth of cure compared to the scraping method.

These variations in the depth of cure between bulk-fill resin composites may also be attributed to a high percentage of the wavelengths being absorbed near the top surface of the resin composite and not used to stimulate co-initiators at greater depths or because light scattering at particle interfaces and the difference in the ability of the photo initiators and any pigments to absorb the light (Garcia et al., 2014). The pigments in the resin composite have an effect on depth of cure because the pigments are opaque particles which will limit the light penetration into the restoration and decrease the degree of polymerization at greater depths (Garcia et al., 2014). Leprince et al. (2013) stated that adequate curing of light cured composite depends on the initiator receiving sufficient energy at correct wavelengths. In addition, the filler size and content in dental composites may affect light penetration and it has a direct relationship with depth of cure (Garcia et al., 2014).

UNIVERSITY of the
WESTERN CAPE

The polymerization reaction of the resin composite is dependent on deep light penetration to ensure adequate mechanical properties. There may be some barriers that prevent this penetration including scattering and absorption of the light by the restorative material attenuating its potential to cure. The photo initiators also have an effect on penetration of the light as they act as a filter to specific wavelengths (Ferracane, Alex, & Margeas, 2014). All of these factors may explain the variation in depth of cure between the bulk-fill composite and conventional composites and these variations have been reported specifically regarding bulk-fill composites (Leprince et al., 2014).

In order to increase the depth of cure manufacturers have followed different strategies including reducing the filler amount as in Filtek Bulk-fill flowable, increasing the filler size as in SDR and

SureFil, introducing a new photo-initiator Ivocerin as in Tetric N Ceram, or enhancing the translucency to allow deeper penetration of sufficient photons of light for activating the photo initiator system (Bucuta & Ilie, 2014). Moszner, (2013) reported that to increase the thickness of depth of cure all the factors that have an effect on depth of cure must be considered including the shade of the material, the photo initiator, translucency, exposure time and light intensity.

According to Bucuta & Ilie (2014) the light energy passing the composite restoration is one of the most important factors that the depth of cure is dependent upon. The depth of cure is dependent on the composite translucency, which can be increased by matching the refractive indices of fillers and matrix. A sufficient depth of cure will be achieved only when the light energy measured at the bottom of the specimen is more than 0.7 J/cm^2. In another study by Ilie& Stark, (2014) confirmed that to be able to achieve appropriate depth of cure, an energy density of at least 23.51 J/cm^2 is recommended for the high viscosity bulk-fill composite. Furthermore, to achieve the manufacturer's claims regarding depth of cure of 4 to 5mm the majority of the studies suggested that the light energy should be approximately 20 J/cm^2 when applied very close to the surface of the material and increasing curing time by 50% should be considered when the distance increased from 2 or 3mm to affect adequate bulk curing (Ferracane et al., 2014).

UNIVERSITY of the
WESTERN CAPE

It is very rare that the manufacturers and the suppliers of the materials provide a basic recommendation about depth of cure and light intensities but usually they only provide the light exposure time. It is very important for the clinician to be aware of the depth of cure at specific activation times and light intensities that can help in planning placement technique that will ensure adequate cure of the bulk of the restoration (Moore et al., 2008). Since it has been shown that some residual monomers can elute even from a well polymerized resin it can be assumed

that more substances would be released from poorly polymerized resin at the bottom of the restoration (Moore et al., 2008). These substances can harm the soft tissue; promote allergic reactions as well as stimulating bacterial growth (Sideridou & Achilias, 2005).

6.4 The efficiency of the LED and QTH curing light:

Ilie & Stark (2014) demonstrated that the depth of cure is the most sensitive parameter to describe the efficiency of curing. Halvorson, Erickson & Davidson (2002) reported that there is correlation between the amount of energy delivered from the curing unit to a composite material and the resultant polymerization and physical properties. Also, Haenel et al. (2015) stated that the irradiance from the curing light has an influence on the surface hardness as well as on the depth of cure.

Since the introduction of the LED curing units there has been an increased interest in comparing their ability to cure dental composites to that of QTH (Alpoz et al., 2008). One of the aims of this study was to compare the effect of LED curing light and QTH curing light on the micro-hardness and the depth of cure of bulk-fill composites tested immediately and 24 hours after curing.

UNIVERSITY of the
WESTERN CAPE

A. The micro hardness of LED vs QTH

LED curing light showed higher micro-hardness values than the QTH curing units for all the materials except Tetric N Ceram. These results were similar to the findings reported by Oglah (2011) who concluded that the surface micro-hardness for LED curing light is higher as it can cure composite resin more efficiently than QTH curing light. Similarly, Agrawal et al. (2014) showed that the micro-hardness and depth of cure values were greater with LED light than with QTH. Additionally, Shamsi & Alaghehmand, (2015) confirmed that LED curing light had greater micro-hardness values for resin composites than QTH curing light.

Tetric N Ceram was the only material that presented a significantly lower hardness on top and bottom surface when cured with LED unit than with the QTH. This may be explained by differences in LED and QTH ability to excite the photo initiators, Ivocerin present in this material. These results are in agreement with Sabatini (2013) results, which demonstrated significantly higher hardness values when polymerized with QTH for Tetric Evo Ceram than LED curing light. Cefaly et al. (2005) confirmed that the hardness of these composites obtained with LED curing light was not as hard as when the material is cured with QTH curing light. The LED curing light did not achieve the same micro-hardness as QTH did in curing these composites that has a combination of photo initiators rather than camphorquinone only. Alpoz et al. (2008) concluded that the superiority of LED units over QTH curing light is still questionable when different dental composite materials are polymerized. In addition, LED curing technology may not be compatible with some dental composites. The photoinitiator systems found in some composites need to be adjusted to the spectra of the curing light (Yaman et al., 2011).

UNIVERSITY of the
WESTERN CAPE

B. Depth of cure:

The depth of cure is dependent on the properties of curing lights. The intensity of the curing light depends on various factors (light guide, power of the battery and condition of the bulb). The total energy irradiation determines the mechanical properties of the composite (Lombardini et al., 2012). LED curing light demonstrated greater values of depth of cure than QTH for all the material used in this study. However, there was no significant difference in depth of cure values immediately for SDR flowable and SureFil bulk-fill. Yaman et al. (2011) confirmed the same results for conventional composites with depth of cure and micro-hardness values where LED curing light was found to be more successful than QTH.

Kumar et al. (2012) reported different results where curing with QTH curing light produced better depth of cure than LED curing lights. Effectiveness of LED curing light as compared to QTH is dependent on the type of product as well as the type of composite resin (Choudhary & Suprabha, 2013).

Theoretically, irrespective of the type of the curing unit, the degree of polymerization should be equal when the same radiant exposure is delivered. The information about the radiant exposure or the amount of energy required ensuring sufficient polymerization is always neglected by manufactures. This information should be provided in their product instructions as it is of great clinical relevance. Alternatively polymerization time is provided by the manufactures and terms like high-intensity light or standard light are used to describe the type of the curing light resulting in unclear estimation of the energy requirements (Sabatini, 2013).

UNIVERSITY of the
WESTERN CAPE

Chapter 7

Limitations and recommendations

7.1 **Limitations of the study:**

- This study was done under ideal laboratory conditions where the curing light was in direct contact with the restoration, which may not be possible to apply on the tooth as the anatomy of the tooth plays a role in affecting the amount of light entering the restoration. A further clinical study may be needed to corroborate the results of this study.
- The storage condition of the specimens in this study may differ from the clinical situation were the material was used dry in this laboratory study.
- This study used only one shade. Using a darker shade may need to be tested as well as these results obtained from one shade limits the conclusions and thus cannot be generalized.

UNIVERSITY of the
WESTERN CAPE

7.2 **Recommendations:**

- In the current study the efficiency of the curing light was evaluated by micro-hardness test, a further study that uses infrared spectroscopic analysis to determine the actual degree of conversion may add value to our knowledge on depth of cure.
- The effect of extended curing exposure times on the hardness and depth of cure distribution requires further study. Also, measuring the intensity of the light at the bottom of the restoration may provide some information as to the amount of light being absorbed within the material itself.

- The effect of extended curing time and the resultant increase in temperature or heat generation from the curing lights on the pulp tissue in bulk placement may also need to be studied.

Chapter 8

Conclusion

This was an *in vitro* study investigating the micro-hardness and the depth of cure of bulk-fill composites and based on the results the null hypothesis was rejected.

Overall, while the low viscosity bulk-fill flowable composites showed better depth of cure compared to the conventional viscosity bulk-fill composites, their surface hardness was lower than the conventional viscosity bulk-fill composites.

Within the limitation of this study it can be concluded that:

1. SureFil bulk-fill composite showed the highest hardness values amongst the materials tested.
2. Bulk-fill low viscosity flowable composites showed the higher depth of cure compared to the conventional bulk-fill composites. Filtek flowable showed the best depth of cure however, there was no significant difference between the two flowable bulk-fill materials.
3. Filtek and SDR low viscosity bulk-fill flowable composites showed an acceptable depth of cure in comparison with the other bulk-fill materials i.e. high viscosity bulk-fill composite.
4. None of the materials achieved the standard depth of cure of 80% bottom to top ratio.
5. With regards to effect of curing light on depth of cure: LED and QTH mean values were significantly different where LED showed better hardness and depth of cure for all materials except for Tetric N Ceram.

UNIVERSITY of the
WESTERN CAPE

6. With regards to effect of curing light on micro-hardness: LED showed better results than QTH except for Tetric N Ceram where QTH showed higher hardness values than LED.
7. The hardness and depth of cure values for all materials increased after 24hrs.

Clinical relevance: The low micro-hardness values for the flowable composites and the inadequate polymerization raises a concern regarding placement of these materials in bulk and their effect on the oral environment. In such cases, the flowable bulk-fills should be protected with a conventional composite covering or "capping material" especially in posterior teeth and in deeper cavities bulk-fill composites should be used in layering incremental technique to ensure sufficient depth of cure.

UNIVERSITY of the
WESTERN CAPE

Chapter 9

References

Abbas, G., Fleming, G., Harrington, E., Shortall, A. & Burke, F. 2003, "Cuspal movement and microleakage in premolar teeth restored with a packable composite cured in bulk or in increments", *Journal of dentistry*, vol. 31, no. 6, pp. 437-444.

ADA. American Dental Association. 2003. "Resin Based Composite". *J Am Dent Assoc*, 4,510-513.

Agrawal, A., Manwar, N., Hegde, S., Chandak, M., Ikhar, A. & Patel, A. 2014, "Comparative evaluation of led and halogen light curing on polymerization of silorane based posterior composite resin: An in vitro study", *Universal Research Journal of Dentistry*, vol. 4, no. 3, pp. 167.

Atmadja, G. & Bryant, R.W. 1990, "Some factors influencing the depth of cure of visible light-activated composite resins", *Australian Dental Journal*, vol. 35, no. 3, pp. 213-218.

Aguiar, F., Braceiro, A., Lima, D., Ambrosano, G. & Lovadino, J. 2007, "Effect of light curing modes and light curing time on the microhardness of a hybrid composite resin", *J Contemp Dent Pract*, vol. 8, no. 6, pp. 1-8.

Alrahlah, A., Silikas, N. & Watts, D. 2014, "Post-cure depth of cure of bulk fill dental resin-composites", *Dental Materials*, vol. 30, no. 2, pp. 149-154.

Alpoz, A.R., Ertugrul, F., Cogulu, D., Ak, A.T., Tanoglu, M. & Kaya, E. 2008, "Effects of light curing method and exposure time on mechanical properties of resin based dental materials", *European journal of dentistry*, vol. 2, no. 1, pp. 37-42.

Anusavice, K.J., Shen, C. & Rawls, H.R. 2012, *Phillips' science of dental materials*, Elsevier Health Sciences.

UNIVERSITY of the WESTERN CAPE

Arikawa, H., Kanie, T., Fujii, K., Takahashi, H. & Ban, S. 2007, "Effect of filler properties in composite resins on light transmittance characteristics and color", *Dental materials journal,* vol. 26, no. 1, pp. 38-44.

Benetti, A.R., Havndrup-Pedersen, C., Honoré, D., Pedersen, M.K. & Pallesen, U. 2015, "Bulk-fill resin composites: polymerization contraction, depth of cure, and gap formation", *Operative dentistry,* vol. 40, no. 2, pp. 190-200.

Boksman, L. & Santos Jr, G.C. 2012, "Principles of light-curing", *Inside Dentistry,* vol. 8, no. 3, pp. 94-97.

Borges, B.C.D., Bezerra, G.V.G., Mesquita, J.d.A., Silva, Talles Rodrigo Santiago Freitas, Alves-Júnior, C., Pinheiro, Isauremi Vieira de Assunção, Braz, R. & Montes, Marcos Antônio Japiassú Resende 2013, "Filler morphology of resin-based low-viscosity materials and surface properties after several photoactivation times", *Acta Odontologica Scandinavica,* vol. 71, no. 1, pp. 215-222.

Bucuta, S. & Ilie, N. 2014, "Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites", *Clinical oral investigations,* vol. 18, no. 8, pp. 1991-2000.

Burgess, J., Walker, R. & Davidson, J. 2002, "Posterior resin-based composite: review of the literature", *Pediatric dentistry,* vol. 24, no. 5, pp. 465-479.

Caldas, D., Almeida, J., Correr-Sobrinho, L., Sinhoreti, M. & Consani, S. 2003, "Influence of curing tip distance on resin composite Knoop hardness number, using three different light curing units", *Operative Dentistry-University Of Washington-,* vol. 28, no. 3, pp. 315-320.

Ceballos, L., Fuentes, M.V., Tafalla, H., Martínez, Á., Flores, J. & Rodríguez, J. 2009, "Curing effectiveness of resin composites at different exposure times using LED and halogen units", *Med Oral Patol Oral Cir Bucal,* vol. 14, no. 1, pp. E51-6.

Cefaly, D.F.G., Ferrarezi, Giovano Augusto de Oliveira, Tapety, C.M.C., Lauris, J.R.P. & Navarro, Maria Fidela de Lima 2005, "Microhardness of resin-based materials polymerized with LED and halogen curing units", *Brazilian dental journal,* vol. 16, no. 2, pp. 98-102.

UNIVERSITY of the
WESTERN CAPE

Choi, K.K., Ferracane, J.L., Hilton, T.J. & Charlton, D. 2000, "Properties of packable dental composites", *Journal of Esthetic and Restorative Dentistry,* vol. 12, no. 4, pp. 216-226.

Choudhary, S. & Suprabha, B. 2013, "Effectiveness of light emitting diode and halogen light curing units for curing microhybrid and nanocomposites", *Journal of conservative dentistry: JCD,* vol. 16, no. 3, pp. 233.

Christensen, G. 2012, "Advantages and challenges of bulk-fill resins", *Clinician's Report,* vol. 5, no. 1, pp. 1-2.

Cobb, D.S., MacGregor, K.M., VARGAS, M.A. & Denehy, G.E. 2000, "The physical properties of packable and conventional posterior resin-based composites: a comparison", *The Journal of the American Dental Association,* vol. 131, no. 11, pp. 1610-1615.

Coffey, O., Ray, N.J., Lynch, C.D., Burke, F.M. & Hannigan, A. 2004, "In vitro study of surface microhardness of a resin composite exposed to a quartz-halogen lamp", *Quintessence international (Berlin, Germany : 1985),* vol. 35, no. 10, pp. 795-800.

Correr, A.B., Sinhoreti, M.A.C., Correr Sobrinho, L., Tango, R.N., Schneider, L.F.J. & Consani, S. 2005, "Effect of the increase of energy density on Knoop hardness of dental composites light-cured by conventional QTH, LED and xenon plasma arc", *Brazilian dental journal,* vol. 16, no. 3, pp. 218-224.

Czasch, P. & Ilie, N. 2013, "In vitro comparison of mechanical properties and degree of cure of bulk fill composites", *Clinical oral investigations,* vol. 17, no. 1, pp. 227-235.

Dunn, W.J. & Bush, A.C. 2002, "A comparison of polymerization by light-emitting diode and halogen-based light-curing units", *The Journal of the American Dental Association,* vol. 133, no. 3, pp. 335-341.

De Biasi, M., Calvi, R., Sossi, D., Maglione, M. & Angerame, D. 2010, "Microhardness of a new flowable composite liner for posterior restorations", *dental materials,* vol. 26, pp. e25.

El-Nawawy, M., Koraitim, L., Abouelatta, O. & Hegazi, H. 2012, "Depth of Cure and Microhardness of Nanofilled, Packable and Hybrid Dental Composite Resins", *American Journal of Biomedical Engineering,* vol. 2, no. 6, pp. 241-250.

UNIVERSITY of the WESTERN CAPE

El-Safty, S., Akhtar, R., Silikas, N. & Watts, D. 2012, "Nanomechanical properties of dental resin-composites", *Dental Materials,* vol. 28, no. 12, pp. 1292-1300

Ferracane, J.L. 2011, "Resin composite—state of the art", *Dental materials,* vol. 27, no. 1, pp. 29-38.

Ferracane, J.L., Pfeifer, C.S. & Hilton, T.J. 2014, "Microstructural Features of Current Resin Composite Materials", *Current Oral Health Reports,* vol. 1, no. 4, pp. 205-212.

Finan, L., Palin, W.M., Moskwa, N., McGinley, E.L. & Fleming, G.J. 2013, "The influence of irradiation potential on the degree of conversion and mechanical properties of two bulk-fill flowable RBC base materials", *Dental Materials,* vol. 29, no. 8, pp. 906-912.

Flury, S., Hayoz, S., Peutzfeldt, A., Hüsler, J. & Lussi, A. 2012, "Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials?", *Dental Materials,* vol. 28, no. 5, pp. 521-528.

Fortin, D. & Vargas, M.A. 2000, "The spectrum of composites: new techniques and materials", *The Journal of the American Dental Association,* vol. 131, pp. 26S-30S.

Furuse, A.Y., Mondelli, J. & Watts, D.C. 2011, "Network structures of Bis-GMA/TEGDMA resins differ in DC, shrinkage-strain, hardness and optical properties as a function of reducing agent", *dental materials,* vol. 27, no. 5, pp. 497-506.

García, A.H., Lozano, M.A.M., Vila, J.C., Escribano, A.B. & Galve, P.F. 2006, "Composite resins. A review of the materials and clinical indications", *Med Oral Patol Oral Cir Bucal,* vol. 11, no. 2, pp. E215-220.

Garcia, D., Yaman, P., Dennison, J. & Neiva, G. 2014, "Polymerization shrinkage and depth of cure of bulk fill flowable composite resins", *Operative dentistry,* vol. 39, no. 4, pp. 441-448.

Garoushi, S., Säilynoja, E., Vallittu, P.K. & Lassila, L. 2013, "Physical properties and depth of cure of a new short fiber reinforced composite", *Dental Materials,* vol. 29, no. 8, pp. 835-841.

UNIVERSITY of the
WESTERN CAPE

Giuliano, C., Manzon, E., Alovisi, M., Pasqualini, D., Cadenaro, M., Breschi, L., Berutti, E. & Scotti, N. 2013, "Micro-hardness and contraction stress evaluation of bulk-fill resins composites", *Dental Materials*, no. 29, pp. e41-e42.

Guo, G., Fan, Y., Zhang, J., Hagan, J.L. & Xu, X. 2012, "Novel dental composites reinforced with zirconia–silica ceramic nanofibers", *Dental Materials,* vol. 28, no. 4, pp. 360-368.

Haenel, T., Hausnerová, B., Steinhaus, J., Price, R.B., Sullivan, B. & Moeginger, B. 2015, "Effect of the irradiance distribution from light curing units on the local micro-hardness of the surface of dental resins", *Dental Materials,* vol. 31, no. 2, pp. 93-104.

Halvorson, R., Erickson, R. & Davidson, C. 2003, "An energy conversion relationship predictive of conversion profiles and depth of cure for resin-based composite", *Operative Dentistry-University Of Washington-,* vol. 28, no. 3, pp. 307-314.

Halvorson, R.H., Erickson, R.L. & Davidson, C.L. 2002, "Energy dependent polymerization of resin-based composite", *Dental Materials*, vol. 18, no. 6, pp. 463-469.

Hubbezoglu, I., Bolayir, G., Dogan, O.M., Dogan, A., ÖZER, A. & Bek, B. 2007 "Microhardness evaluation of resin composites polymerized by three different light sources", *Dental materials journal,* vol. 26, no. 6, pp. 845-853.

Ikeda, T., Murata, Y. & Sano, H. 2004, "Translucency of opaque-shade resin composites", *American Journal of Dentistry*, vol. 17, no. 2, pp. 127-130.

Ilie, N., Bucuta, S. & Draenert, M. 2013, "Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance", *Operative dentistry,* vol. 38, no. 6, pp. 618-625.

Ilie, N. & Hickel, R. 2011, "Resin composite restorative materials", *Australian Dental Journal,* vol. 56, no. s1, pp. 59-66.

Ilie, N., Keßler, A. & Durner, J. 2013, "Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites", *Journal of dentistry,* vol. 41, no. 8, pp. 695-702.

UNIVERSITY of the WESTERN CAPE

Ilie, N. & Stark, K. 2014, "Curing behaviour of high-viscosity bulk-fill composites", *Journal of dentistry*, vol. 42, no. 8, pp. 977-985.

Jackson, R.D. 2012, "New posterior composite materials improving placement efficiency", *Compendium of continuing education in dentistry (Jamesburg, N.J.: 1995)*, vol. 33, no. 4, pp. 292-293.

Jang, J., Park, S. & Hwang, I. 2015, "Polymerization Shrinkage and Depth of Cure of Bulk-Fill Resin Composites and Highly Filled Flowable Resin", *Operative dentistry*, vol. 40, no. 2, pp. 172-180

Karabela, M.M. & Sideridou, I.D. 2011, "Synthesis and study of properties of dental resin composites with different nanosilica particles size", *Dental Materials*, vol. 27, no. 8, pp. 825-835.

Knežević, A., Tarle, Z., Meniga, A., Šutalo, J., Pichler, G. & Ristić, M. 2001, "Degree of conversion and temperature rise during polymerization of composite resin samples with blue diodes", *Journal of oral rehabilitation*, vol. 28, no. 6, pp. 586-591.

Kumar, C., Gururaj, M., Paul, J., Krishnaprasada, L. & Divyashree, R. 2012, "A comparative evaluation of curing depth and compressive strength of dental composite cured with halogen light curing unit and blue light emitting diode: an in vitro study", *J Contemp Dent Pract*, vol. 13, pp. 834-837.

Leinfelder, K.F., Bayne, S.C. & Swift Jr, E.J. 1999, "Packable composites: overview and technical considerations", *Journal of Esthetic and Restorative Dentistry*, vol. 11, no. 5, pp. 234.

Leprince, J.G., Palin, W.M., Hadis, M.A., Devaux, J. & Leloup, G. 2013, "Progress in dimethacrylate-based dental composite technology and curing efficiency", *Dental Materials*, vol. 29, no. 2, pp. 139-156.

Leprince, J.G., Palin, W.M., Vanacker, J., Sabbagh, J., Devaux, J. & Leloup, G. 2014, "Physico-mechanical characteristics of commercially available bulk-fill composites", *Journal of dentistry*, vol. 42, no. 8, pp. 993-1000.

UNIVERSITY of the WESTERN CAPE

Leprince, J., Palin, W., Mullier, T., Devaux, J., Vreven, J. & Leloup, G. 2010, "Investigating filler morphology and mechanical properties of new low-shrinkage resin composite types", *Journal of oral rehabilitation*, vol. 37, no. 5, pp. 364-376.

Lindberg, A., Peutzfeldt, A. & van Dijken, J.W. 2005, "Effect of power density of curing unit, exposure duration, and light guide distance on composite depth of cure", *Clinical oral investigations*, vol. 9, no. 2, pp. 71-76.

Lombardini, M., Chiesa, M., Scribante, A., Colombo, M. & Poggio, C. 2012, "Influence of polymerization time and depth of cure of resin composites determined by Vickers hardness", *Dental research journal*, vol. 9, no. 6, pp. 735.

Manhart, J., Kunzelmann, K., Chen, H. & Hickel, R. 2000, "Mechanical properties and wear behavior of light-cured packable composite resins", *Dental Materials*, vol. 16, no. 1, pp. 33-40.

Manhart, J., Chen, H.Y. & Hickel, R. 2001, "The suitability of packable resin-based composites for posterior restorations", *The Journal of the American Dental Association*, vol. 132, no. 5, pp. 639-645.

Mikhail, S.S., Schricker, S.R., Azer, S.S., Brantley, W.A. & Johnston, W.M. 2013, "Optical characteristics of contemporary dental composite resin materials", *Journal of dentistry*, vol. 41, no. 9, pp. 771-778.

Mills, R., Jandt, K. & Ashworth, S. 1999, "Restorative Dentistry: Dental composite depth of cure with halogen and blue light emitting diode technology", *British dental journal*, vol. 186, no. 8, pp. 388-391.

Moore, B., Platt, J., Borges, G., Chu, T.G. & Katsilieri, I. 2008, "Depth of cure of dental resin composites: ISO 4049 depth and microhardness of types of materials and shades", *Operative dentistry*, vol. 33, no. 4, pp. 408-412.

Moszner, N., Fischer, U.K., Ganster, B., Liska, R. & Rheinberger, V. 2008, "Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials", *dental materials*, vol. 24, no. 7, pp. 901-907.

UNIVERSITY of the WESTERN CAPE

Mousavinasab, S.M. 2011, *Effects of Filler Content on Mechanical and Optical Properties of Dental Composite Resin*, INTECH Open Access Publisher.

Mousavinasab, S.M. & Meyers, I. 2009, "Curing efficacy of light emitting diodes of dental curing units", *Journal of dental research, dental clinics, dental prospects*, vol. 3, no. 1, pp. 11.

Moszner, N., 2013. State of the art Photopolymerization in dentistry. *Research and Developmental IvoclarVivadent*, 19, pp*4*-10.

Neeraj Malhotra, M. & Kundabala Mala, M. 2010, "Light-curing considerations for Resin-based composite materials: A review Part I", *Compendium*, vol. 31, no. 7.

Oglah ,F,S. 2011, Surface microhardness and depth of cure of composite resin cured with LED and Halogen units, *MDJ*,vol.8, no1,pp.13-18.

Poskus, L.T., Placido, E. & Cardoso, P.E.C. 2004, "Influence of placement techniques on Vickers and Knoop hardness of class II composite resin restorations", *Dental Materials*, vol. 20, no. 8, pp. 726-732.

Price, R., Fahey, J. & Felix, C. 2010, "Knoop microhardness mapping used to compare the efficacy of LED, QTH and PAC curing lights", *Operative dentistry*, vol. 35, no. 1, pp. 58-68.

Rouhollahi, M., Mohammadibasir, M. & Talim, S. 2012, "Comparative depth of cure among two light-cured core build-up composites by surface Vickers hardness", *Journal of dentistry (Tehran, Iran)*, vol. 9, no. 3, pp. 255.

Rueggeberg, F.A., Caughman, W.F., Curtis, J.W.,Jr & Davis, H.C. 1993, "Factors affecting cure at depths within light-activated resin composites", *American Journal of Dentistry*, vol. 6, no. 2, pp. 91-95.

Rueggeberg, F, A.**,** Twiggs, S, W., Caughman, W, F., Khajotia, S., 1996. Lifetime intensity profiles of 11 light-curing units, *JDent Res*, 75,380.

Ravi ,R.K., Alla,R.K., Shammas,M., Devarhubli A., 2013. Dental Composites - A Versatile Restorative Material: An Overview, *Indian Journal of Dental Sciences*, vol. 5, no.5,pp11-115.

UNIVERSITY of the WESTERN CAPE

Rao kilaru, k., Hinduja, D., Kumar ,S. & Rao, N. 2012, Comparative evaluation of compressive strength, Vickers hardness and modulus of elasticity of hybrid and packable (condensable) posterior composites: An in vitro study. *Annals and essences of dentistry*, vol. 5, no. 2, pp. 9-16.

Rastelli A,N,S, Alessandra Nara, Navarro, R.S., Saad, J.R.C., de Andrade, M.F. & Bagnato, V.S. 2014, "Effect of different light-curing techniques on hardness of a microhybrid dental composite resin", *Brazilian Dental Science,* vol. 17, no. 1, pp. 45-53.

Sabatini, C. 2013, "Comparative study of surface microhardness of methacrylate-based composite resins polymerized with light-emitting diodes and halogen", *European journal of dentistry,* vol. 7, no. 3, pp. 327.

Salerno, M., Derchi, G., Thorat, S., Ceseracciu, L., Ruffilli, R. & Barone, A.C. 2011, "Surface morphology and mechanical properties of new-generation flowable resin composites for dental restoration", *Dental Materials,* vol. 27, no. 12, pp. 1221-1228.

Sobrinho, L.C., De Lima, A., Consani, S., Sinhoreti, M. & Knowles, J. 2000, "Influence of curing tip distance on composite Knoop hardness values", *Braz Dent J,* vol. 11, no. 1, pp. 11-17

Sideridou, I.D. & Achilias, D.S. 2005, "Elution study of unreacted Bis-GMA, TEGDMA, UDMA, and Bis-EMA from light-cured dental resins and resin composites using HPLC", *Journal of Biomedical Materials Research Part B: Applied Biomaterials,* vol. 74, no. 1, pp. 617-626.

Shamsi, N. & Alaghehmand, H. 2015, "A comparative study of the effects of QTH and LED light curing units on the surface hardness of colored compomer and Hybrid composites", *Caspian Journal of Dental Research,* vol. 4, no. 1, pp. 21-27.

Soygun, K., Ünal, M., Özer, A., Gülnahar, E. & Bolayır, G. 2014, "Farklı Akışkan Bulk Fill Kompozitlerin Mikrosertliklerinin Araştırılması", *Cumhuriyet Dental Journal,* vol. 17, no. 1, pp. 64-69.

Tantbirojn, D., Pfeifer, C.S., Braga, R.R. & Versluis, A. 2011, "Do low-shrink composites reduce polymerization shrinkage effects?", *Journal of dental research,* vol. 90, no. 5, pp. 596-601.

UNIVERSITY of the WESTERN CAPE

Thiab, S.S. 2012. Influence of Light Curing Method and Curing Time on the Surface Hardness and Degree of Cure in Composite Resins. *Journal of Babylon University*, vol. 20, no 2, pp. 779-790.

Thomé, T., Steagall Jr, W., Tachibana, A., Braga, S.R.M. & Turbino, M.L. 2007, "Influence of the distance of the curing light source and composite shade on hardness of two composites", *Journal of Applied Oral Science,* vol. 15, no. 6, pp. 486-491.

Uhl, A., Mills, R.W. & Jandt, K.D. 2003, "Photoinitiator dependent composite depth of cure and Knoop hardness with halogen and LED light curing units", *Biomaterials,* vol. 24, no. 10, pp. 1787-1795.

Valente, L.L., Peralta, S.L., Ogliari, F.A., Cavalcante, L.M. & Moraes, R.R. 2013, "Comparative evaluation of dental resin composites based on micron-and submicron-sized monomodal glass filler particles", *Dental Materials,* vol. 29, no. 11, pp. 1182-1187.

Van Ende, A., De Munck, J., Van Landuyt, K.L., Poitevin, A., Peumans, M. & Van Meerbeek, B. 2013, "Bulk-filling of high C-factor posterior cavities: effect on adhesion to cavity-bottom dentin", *Dental materials,* vol. 29, no. 3, pp. 269-277.

Van Noort, R. 2005, *Introduction to dental materials,* Mosby, UK.

Vogel, K., 2013. From TetricEvoCeram to TetricEvoCeram Bulk Fill. *Research and Developmental IvoclarVivadent, 19, 16*-18.

Watts, D.C., Amer, O. & Combe, E.C. 1984, "Characteristics of visible-light-activated composite systems", *British dental journal,* vol. 156, no. 6, pp. 209-215.

Yaman, B.C., Efes, B.G., Dorter, C., Gomec, Y., Erdilek, D. & Buyukgokcesu, S. 2011, "The effects of halogen and light-emitting diode light curing on the depth of cure and surface microhardness of composite resins", *Journal of conservative dentistry : JCD,* vol. 14, no. 2, pp. 136-139.

Yazici, A.R., Celik, C., Ozgunaltay, G. & Dayangac, B. 2010, "The effects of different light-curing units on the clinical performance of nanofilled composite resin restorations in non-carious cervical lesions: 3-year follow-up", *The journal of adhesive dentistry,* vol. 12, no. 3, pp. 231-236.

UNIVERSITY of the
WESTERN CAPE

Zimmerli, B., Strub, M., Jeger, F., Stadler, O. & Lussi, A. 2010, "Composite materials: composition, properties and clinical applications. A literature review", *Schweizer Monatsschrift fur Zahnmedizin = Revue mensuelle suisse d'odonto-stomatologie = Rivista mensile svizzera di odontologia e stomatologia / SSO*, vol. 120, no. 11, pp. 972-986.

Zorzin, J., Maier, E., Harre, S., Fey, T., Belli, R., Lohbauer, U., Petschelt, A. & Taschner, M. 2015, "Bulk-fill resin composites: Polymerization properties and extended light curing", *Dental Materials*, vol. 31, no. 3, pp. 293-301.

UNIVERSITY of the
WESTERN CAPE