The effect of different composite shades and curing distances on the depth of cure of the low shrink Filtek Silorane composite compared to Filtek Supreme XT and Z100

J MISTRY

MASTERS DEGREE IN DENTAL SCIENCE

THE EFFECT OF DIFFERENT COMPOSITE SHADES AND CURING DISTANCES ON THE DEPTH OF CURE OF THE LOW SHRINK FILTEK SILORANE COMPOSITE COMPARED TO FILTEK SUPREME XT AND Z100

by

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200818323

DISSERTATION

Submitted in fulfilment of the requirements for the degree of

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DECLARATION

I declare that **THE EFFECT OF DIFFERENT COMPOSITE SHADES AND CURING DISTANCES ON THE DEPTH OF CURE OF THE LOW SHRINK FILTEK SILORANE COMPOSITE COMPARED TO FILTEK SUPREME XT AND Z100** (dissertation) hereby submitted to the University of Limpopo, for the degree of Masters of Dental Science has not previously been submitted by me for a degree at this or any other university; that it is my work in design and in execution, and that all material contained herein has been duly acknowledged.

..... **.....**

J. Mistry (Dr) **Date**

Student number: 200818323

DEDICATION

With Gratitude and Love

To

Lord Krishna

(for His blessings and for guiding me with love through every sphere of my life)

To

My parents

Mr NR Mistry and Mrs P Mistry

(for laying a solid foundation for my life and for moulding me into the woman I am today)

To

My husband

Mr Balesh Devjee

(for having confidence in me and for being my pillar of strength)

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ABSTRACT

The importance and demand for aesthetics has resulted in an increase in tooth-coloured restorations in dental practice. Composite undergoes polymerization using a curing device, after which it gains its physical and aesthetic properties. An improperly cured composite restoration is weak and prone to discolouration and secondary decay. The aim of this study was to determine and compare the effect that composite shade and curing distance would have on the depth of cure (DOC) of composite Filtek Silorane, Filtek Supreme XT and Z100. The scrape and penetrometer techniques were used to determine the DOC of the 450 specimens prepared. The DOC decreased with an increase in the curing distance and the darker shade had a lower DOC than the lighter shade for each of the composites tested. Both the scrape and penetrometer techniques yielded similar results in the DOC. Z100 had the highest DOC followed by Supreme XT and lastly Silorane. Thus a lighter composite shade in posterior teeth (non-aesthetic zone) and in deep Class II cavities will ensure an optimal depth of cure. The curing distance should be minimal and as close as possible to the composite surface to achieve an optimal depth of cure.

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CHAPTER 1

INTRODUCTION

The demand for tooth-coloured restorations led to the development of composite resins. The initial resin systems introduced were the powder-liquid methylmethacrylate or acrylate polymer systems in 1937. These direct restorative materials were colour stable, insoluble in the oral cavity and were polishable. However, they underwent severe polymerization shrinkage; the co-efficient of thermal expansion (COTE) was high including a high degree of wear and they were prone to fracture. Thereafter improvements to this system resulted in the introduction of filler particles to the resin matrix. Dr Ray Bowen in 1962 developed 2, 2- bis [4(2-hydroxy-3-methacryloxy-propyloxy)-phenyl] propane (Bis-GMA) which is a larger molecule and less hydrophilic than methylmethacrylate. This solved the drawbacks experienced with methylmethacrylate because the improved resin system underwent less polymerization shrinkage and it was less soluble in oral fluids. Bis-GMA has additional advantages such as a decreased COTE, increased physical properties, improved colour stability, aesthetics and polishability (Sakaguchi and Powers, 2012).

The resin matrix may also comprise of urethane dimethacrylate (UDMA) as the resin backbone which was introduced by Foster and Walker in 1974. It has advantages over Bis-GMA such as colour stability, hydrophobicity and high viscosity. However, it undergoes more polymerization shrinkage than Bis-GMA (Kramer *et al*, 2008).

The main drawback with these resin systems has been polymerization shrinkage. Polymerization shrinkage causes microleakage, cuspal displacement and cracks in healthy tooth structure. Two solutions to reducing polymerization shrinkage, according to Weinmann, Thalacker and Guggenberger (2005), are the reduction of reactive sites per volume unit or the reduction of shrinkage using different types of resin monomers. 3M ESPE in recent years have developed Filtek Silorane which displays low shrinkage and high reactivity. According to 3M ESPE, it has been

developed to reduce the negative effects of polymerization shrinkage and polymerization stress. 3M ESPE claims that it has been shown to display lower shrinkage than all methacrylate composite resins (Weinmann *et al*, 2005; Garcia *et al*, 2006).

The ability to achieve a complete cure of the resin matrix will result in obtaining the favourable properties of light cured composites. If a resin is insufficiently cured, the restoration becomes weaker due to decreased monomer conversion, decreased hardness with an increase in marginal breakdown and wear, resulting in a weak bond to the tooth structure (Aguiar *et al*, 2005; Koupis *et al*, 2004 and 2006).

Therefore it is important to consider certain factors regarding the depth of cure achieved after light curing the composite. These factors are curing time, curing intensity, temperature, distance between curing light and the resin, the angle of the light, thickness of the resin, curing through tooth structure, the shade of resin, the type of filler, the amount of photo-initiator in the resin, the amount of heat generated by the curing unit and room temperature (Visible Light Curing 2002; Albers, 2000; Aguiar *et al*, 2005; Koupis *et al*, 2004 and 2006; de Araujo *et al*, 2008).

The composite shade affects the depth of cure that can be achieved and therefore in this study three shades of each composite were compared and this was done at curing distances of 0, 1, 2, 3 and 5mm.

The surface hardness of a cured composite is not an accurate indicator of an optimally cured composite as a sufficiently cured composite surface can be achieved with an inadequately functioning curing device. This results in uncured composite in the deeper parts of the restoration which remains concealed by the hardened top surface (Shortall, Harrington and Wilson, 1995).

According to the 2009 ISO standard, resin-based composites need to have a minimum depth of cure of 1.5mm after irradiation according to the manufacturer's instructions. They further define depth of cure as 50% of the length of the cured composite sample after the soft, uncured portion has been scraped away manually. The materials depth of cure is then compared with the 1.5mm requirement to determine if the material meets the ISO standard (Fan *et al* 2002; Aravamudhan *et al*, 2006).

Another instrument to measure the depth of cure directly is the penetrometer similar to the one suggested by Harrington and Wilson (1993). The needle connected to a weight is lifted and then lowered onto the middle of the uncured composite specimen. In this way the needle stops when the cured portion of the specimen is reached and the depth of cure is read directly from the digital indicator gauge. This method has advantages over the ISO 4049:2000 scrape test because it is not subjected to variable force applied by the operator as the weight applied to the needle is constant (Jandt *et al*, 2000).

The aim of this study was to determine and compare the effect that curing distance and composite shade would have on the depth of cure of methacrylate-based composites and silorane based composites. Secondly, to determine and compare if there is any difference in the depth of cure of methacylate based composites and silorane-based composites. Lastly, to compare the scrape and penetrometer techniques which were used to determine the depth of cure.

CHAPTER 2

LITERATURE REVIEW

Tooth coloured restorations have increasingly grown in demand during the last decade. During the 1970's, it was discovered that mercury vapour was released from amalgam during mastication which could be inhaled hence, the beginning of the aesthetic revolution. The fear of mercury toxicity has resulted in the replacement of many amalgam restorations with tooth-coloured restorations (Sadowsky, 2006). Composites have acquired a prominent position amongst direct restorative materials due to their good aesthetics and lower costs (Garcia *et al*, 2006; David *et al*, 2007).

They also conserve tooth structure as they bond to the tooth by adhesive methods rather than depending on a retentive cavity design, and can thus be utilized in a variety of therapeutic measures (Garcia *et al*, 2006).

Composite resin restorations are also currently used in 50% of all posterior direct restorations. Despite concerns regarding abrasion, marginal leakage, post-operative sensitivity and toxicity, its popularity is increasing. The aesthetic value is enhanced by its ability to mimic tooth colour and through the use of tints and opaquers, it is able to modify tooth colour (Sadowsky, 2006).

2.1 Composite Resin

Dental composites are composed of three chemically different materials namely, the organic matrix, the inorganic matrix and an organosilane or coupling agent to bond the filler to the organic resin. These constituents are responsible for the physical,

mechanical, aesthetic and clinical properties of composite resins (Sadowsky, 2006; Bhamra and Fleming, 2008; Garcia *et al*, 2006; Sakaguchi and Powers, 2012).

2.1.1 Resin Matrix

The organic matrix is composed of a system of mono-functional, di-functional or tri-functional monomers which form the backbone of composite resins. Most composite resin systems consist of 2, 2- bis [4(2-hydroxy-3-methacryloxy-propyloxy) -phenyl] propane (Bis-GMA) as the monomer or together with urethane dimethacrylate (UDMA). They both have reactive carbon double bonds at both ends that undergo polymerization due to the presence of initiators and accelerators in the composite resin. The lower mean molecular weight of these monomers results in an increased shrinkage of the cured composite resin.

To assist the manufacturing process of composite resins, low viscosity monomers are added to dilute the organic monomer matrix and are referred to as viscosity controllers. Monomers commonly used are bisphenol-A-dimethacrylate (Bis-DMA), ethylene glycol dimethacrylate (EGDMA), triethylene glycol dimethacrylate (TEGDMA), methyl methacrylate (MMA) or urethane dimethacrylate (UDMA) (McCabe and Walls, 2008; Garcia *et al*, 2006).

2.1.2 Filler Particles

The filler particles constitute the largest portion of a composite resin, in weight or volume. Its primary function is to reinforce the resin matrix, provide the correct degree of translucency and also to minimise shrinkage during polymerization. The inorganic matrix consists of a filler material which largely determines the mechanical and physical properties of a composite and therefore it is incorporated to improve these

properties. Including a high percentage of filler particles to the composite reduces the co-efficient of thermal expansion and polymerization shrinkage; it also provides radio-opacity and improves the handling and aesthetics (McCabe and Walls, 2008).

Traditionally filler particles have been obtained by grinding minerals such as quartz, glasses or sol-gel derived ceramics. Barium and zinc oxide are heavy metal oxides and are radiopaque making it visible on radiographs. Filler particles vary greatly in respect of the composition, morphology and dimensions, with the most common filler particle being used is silicon dioxide. Other filler particles such as boron silicates and lithium aluminium silicates may also form part of the filler material. More radiopaque elements such as barium, strontium, zinc, aluminium or zirconium are partially replacing quartz (Sakaguchi and Powers, 2012; Garcia *et al*, 2006).

2.1.3 Activator-initiator system

The purpose of the activator-initiator system is to initiate the polymerization of resin monomers and to transform the resin matrix with into a hardened substance during the polymerization process. This process of polymerization can be activated through chemical curing (self-curing), light activation and dual curing (a combination of self-curing and light activation). The most commonly used photo-initiator is camphorquinone which comprises 0.1-1.0% of the resin matrix (Craig and Powers, 2002; Sakaguchi and Powers, 2012).

2.1.3.1 Chemically activated resins

An organic amine (accelerator) reacts with an organic peroxide (initiator) at room temperature during chemical activation and initiates polymerization of resin monomers (Sakaguchi and Powers, 2012).

2.1.3.2 Light activated resins

In methacrylate composites, upon activation from blue light at wavelengths ranging from 400-700nm, free radicals are generated and the monomers are converted to polymer networks (resin matrix) (Sakaguchi and Powers, 2012).

2.1.4 Coupling Agents

The coupling agent, an organosilane, is added to the inorganic matrix in order to treat the surface of the filler particles before it is added to the resin matrix. The more commonly used coupling agent is 3-methacryloxypropyltrimethoxysilane (MPTS) in methacrylate-based composites, whereas 3-glycidoxypropyl-trimethoxysilane is found in the low-shrink silorane composite.

The coupling agent bonds the inorganic matrix to the organic matrix of the composite. It is a molecule with a silane group at one end which form an ionic bond and the methacrylate group at the opposing end forms a covalent bond with the resin. Hence the function of a coupling agent is to form a strong interfacial bridge that binds the resin matrix to the filler particles, to enhance the mechanical properties of the composite and to prevent dislodgement of the filler particles from the matrix during function (Garcia *et al*, 2006; Craig and Powers, 2002; Sakaguchi and Powers, 2012).

2.1.5 Pigments and optical modifiers

Pigments are inorganic oxides which are added in small quantities to composites to

provide a variety of shades that match natural tooth structure of which iron oxides are the most common. Various shades ranging from very light to yellow to gray are available in many composite products on the market today. To prevent colour changes due to oxidation processes, ultraviolet (UV) absorbers are added.

Another component found in composite are dyes or fluorescent pigments that absorb light in the ultraviolet range (340-370nm) and re-emits this light in the blue spectrum (420-470nm). They enhance the optical character of the composite and provide a natural looking composite restoration. This is achieved by producing a material which appears whiter by increasing the reflection of blue light in the electromagnetic spectrum (Sakaguchi and Powers, 2012).

2.1.6 Inhibitors and Stabilisers

Stabilisers or inhibitors (hydroquinone monomethyl ether) increase the storage life of composite resins prior to polymerization and additionally they provide chemical stability after polymerization. Absorbers such as 2-hydroxy-4-methoxybenzophenone absorb ultra-violet wavelength below 350nm and provide long term colour stability and negate the effects of UV light on the amine compounds in the initiator system thereby preventing medium to long term discolouration of the composite resin (Sakaguchi and Powers, 2012; Garcia *et al*, 2006).

2.1.7 Accelerators

Another constituent of a composite resin include an acceleration system which initiates the polymerization process. These may compose of organic amines which are aromatic and aliphatic. Such amines used in composites are dimethylaminoethylmethacrylate (DMAEM), ethyl-4-dimethylaminobenzoate

(EDMAB) or N,N-cyanoethyl-methylaniline (CEMA) (Garcia *et al*, 2006; Sakaguchi and Powers, 2012).

2.2 Types of Composites

Composites resins can be classified according to the type of filler particle, filler particle size, filler distribution, the type of resin matrix or the curing mechanism. Willem *et al* (1992) classified composite resins according to the size of the filler particle as densified composites, microfine composites, miscellaneous composites, traditional composites and fiber-reinforced composites. Lutz and Phillips (1983) classified composites according to their filler particle size, thereby dividing composite resins into macrofilled composites (particles from 0.1 to 100 μ), microfilled composites (average particle size of 0.04μ) and hybrid composites (fillers of various sizes) (Garcia *et al*, 2006).

Continuous changes in technology have brought about improvements in aesthetics, wear resistance and higher durability that closely resemble natural tooth structure. One such change is the introduction of nanotechnology which has brought about changes in the filler configuration of composite resins (Sakaguchi and Powers, 2012). Up until recently the emphasis has been on reduction of the filler particle size to produce composites with greater wear resistance and to facilitate easy handling. These properties were necessary for both anterior and posterior restorations (Ferracane, 2011).

Traditionally dimethacrylate monomers such as Bis-GMA, ethoxylated bisphenol-A-dimethacrylate (Bis-EMA) and UDMA, glass and silica dioxide fillers and a photo-initiator are the main components of composite resins (Perez *et al*, 2010; Ferracane, 2011).

Over the last decades, composite resins in restorative dentistry have progressed from Bowen's resin (Bis-GMA) with modifications to its polarity or viscosity. These changes were made either to the functionality or to the backbone of the resin, with the resultant development of triethylene glycol dimethacrylate (TEGDMA) and urethane dimethacrylate (UDMA) (Weinmann *et al*, 2005; Garcia *et al*, 2006; Ferracane, 2011).

New resin technology aims at reducing polymerization shrinkage and stress by making changes to the type, quantity and size of the filler particles or to improve the monomer chemistry (Perez *et al*, 2010).This development over the years of different resins has lead to improvements in physical strength, wear resistance and stability in the oral environment, with modern composites showing good physical resistance and beautiful aesthetics. However the drawbacks that remain are polymerization shrinkage and polymerization stress. These provide challenges such as reduced marginal integrity and post-operative sensitivity, with imperfect margins leading to marginal staining and resultant secondary caries (Weinmann *et al*, 2005; Garcia *et al*, 2006).

2.2.1 Macrofilled Composites

Macrofilled composites were the early composites on the market. Macrofilled composites consisted of large spherical or irregular shaped filler particles with an average particle size of 20-30 μm. These composites had a high compressive strength but were aesthetically unpleasing (very opaque) and difficult to polish to a smooth surface due to its large particle size (Ferrance, 2011; Sakaguchi and Powers, 2012; McCabe and Walls, 2008).

2.2.2 Microfilled Composites

Microfilled composites consist of an average particle size of 0.04 μm silica fillers with pre-polymerized resin which can occasionally be filled with colloidal silica. The total filler content is 32-50% by volume. These composites are used in Class III and Class V cavities where there is no or minimal stress bearing and where an aesthetic and highly polished restoration is required. Due to the lower filler loading they have a higher water absorption and higher thermal expansion when compared to microhybrid and nanocomposites. Polymerization shrinkage can also be higher depending on the quantity of pre-polymerized resin in the composite (Sakaguchi and Powers, 2012).

2.2.3 Hybrid Composites

Hydrid composites consist of a combination of two types of fillers. These are fine particles of sizes 2-4 μm and 5%-15% of microfine particles, made of silica with particles size 0.04-0.2 μm (Sakaguchi and Powers, 2012).

2.2.4 Microhybrid Composites

Fine particles of particle size 0.04-1μm are combined with microfine silica to be classified as a microhybrid composite. These fine particles are obtained by grinding glass such as borosilicate glass, lithium or barium aluminium silicate glass, strontium or zinc glass. Other materials such as quartz or ceramic materials may also be used due to its irregular shapes. Microhybrid composites may contain 60-70% filler by volume thus improving the handling properties of the composite. They show good stress bearing capabilities and wear resistance; however they lose their surface smoothness and result in dull and rough composite restorations (Sakaguchi and Powers, 2012).

2.2.5 Nanofilled Composites

Nanotechnology has introduced the production of functional materials and structures in the range of 1-100 nm by chemical and physical methods. Nanotechnology has changed the inorganic component of the composite resin by incorporating nanoparticles (approximately 25 nm) and nanoaggregates (approximately 75 nm) which consist of zirconium/silica or nanosilica particles coated with silane to bond to the resin.

This method provides a high filler load of up to 79.5% by voulme. This reduction in particle size (below wavelength of visible light 400-800 nm) provides the restoration with a highly translucent material with an improved finish and surface texture and thus it reduces the degradation of the restoration over time. Additionally, its improved mechanical properties allow its use in both anterior and posterior restorations. With a higher load, the polymerization shrinkage is reduced, creating less cuspal deflection and reducing microfissures at the enamel margins. This prevents marginal leakage, composite discolouration, penetration of bacteria and thereby reduces post-operative sensitivity.

The uniqueness of the nanofilled composite is that it possess the mechanical properties of a microhybrid composite and the smoothness and polishability of a microfill composite. In nanofilled composites the nanoclusters wear at the same rate during abrasion as the surrounding matrix resulting in a smoother surface over a longer time period than conventional composites. They provide improved and advanced optical properties to composites with a greater range of shades to assist the clinician in obtaining better aesthetics. They achieve this by scattering the blue light component in UV light and thus giving an opalescent effect and a life-like appearance to the tooth.

However, the drawback of the nano size particles are that they do not reflect light and therefore are combined with larger particles (around 1µm) to improve its optical properties and to provide a substrate (Sakaguchi and Powers, 2012; Garcia *et al*, 2006).

2.3 New Developments in Composites

Polymerization shrinkage causes micro-leakage, cuspal displacement and cracks in healthy tooth structure. Two solutions to reducing polymerization shrinkage, according to Weinmann *et al* (2005), are the reduction of reactive sites per monomer resin volume or the reduction of shrinkage using different types of resin that would expand rather than shrink. 3M ESPE in recent years have developed Filtek Silorane with a cationic ring opening monomer system which displays low shrinkage and high reactivity. According to 3M ESPE, this has been developed to reduce the negative effects of polymerization shrinkage and polymerization stress and 3M ESPE claims that it has been shown to display lower shrinkage than all methacrylate composite resins (Weinmann *et al*, 2005; Garcia *et al*, 2006).

2.3.1 Silorane

Silorane derives its name from the combination of its chemical building blocks siloxanes and oxiranes (known as epoxy). The low shrinkage and low polymerization stress of Silorane is generated by the cationic ring opening polymerization (Weinmann *et al*, 2005). The hydrophobic nature of siloxane ensures that the composite maintains its physical strength intra-orally over an extended period. In addition, it absorbs less exogenic stains than hydrophilic materials making it more aesthetic. The oxirane molecules are responsible for the cationic ring opening of Silorane during polymerization resulting in low shrinkage and low polymerization stress of Silorane (Weinmann *et al*, 2005; Sakaguchi and Powers, 2012; Zimmerli *et al*, 2010).

The initiating system of Silorane consists of camphorquinone, an iodonium salt and an electron donor. As with methacrylates, camphorquinone was chosen for Silorane as a photo-initiator, because it falls within the emission spectrum of the currently available curing systems (Weinmann *et al*, 2005; Zimmerli *et al*, 2010).

The filler particle of fine quartz provides its aesthetic and mechanical stability. The silane layer enhances the hydrophobic nature of the filler surface as well as enhancing and reinforcing the filler and resin interface. Clinically Silorane is a restorative composite with the lowest polymerization shrinkage and stress, and a high light stability. Mechanically it is comparable to properties found in methacrylate composites (Weinmann *et al*, 2005; Zimmerli *et al*, 2010).

2.4 Curing of Composites

For a composite resin restoration to achieve ideal physical and clinical properties, most of the monomer must be polymerized during light curing to achieve long term clinical success (Aguiar *et al*, 2005). The light is absorbed by the organic matrix and the differences in refractive indices between the matrix and filler particle provide the scattering effect (Perez *et al*, 2010). However according to Koupis *et al* only 35-75% of the monomer is converted to polymer, while the remainder is in the form of methacrylate groups or residual or unreacted monomer (Koupis *et al*, 2004).

2.4.1 Chemical curing of methacrylate composites

Previously composite resins required mixing of the base paste with the catalyst in chemically cured composites. The most common composite consisted of two pastes, each containing a combination of resin and filler. The one paste consists of an peroxide activator such as benzoyl peroxide, while the other paste consists of a tertiary amine activator such as *N*, *N*′ dimethyl-*p*-toluidine. Another system consisted

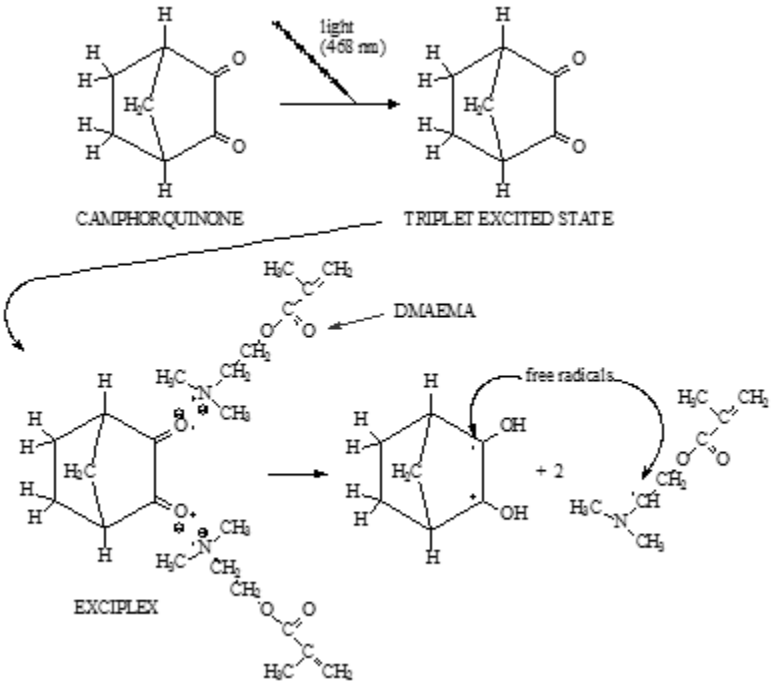
of a powder liquid system. In this system, the powder contained the filler particles and peroxide initiator, whilst the liquid contained the monomer and chemical activator. Other systems included the paste/liquid combination and encapsulated composite system. This had many drawbacks such as mixing proportions of the base and catalyst, colour variations and stability and also a time consuming mixing process (Garcia *et al*, 2006; McCabe and Walls, 2008).

2.4.2 Light curing of methacrylate composites

Most dental restorative resins consist of methacrylates which undergoes polymerization within a wavelength range of 450-470 nm due to activation of the camphorquinone-amine system which is accelerated by a tertiary amine such as an aromatic amine (Ferracane, 2011; Bhamra and Fleming, 2008). This polymerization reaction consists of three stages, namely initiation, propagation and termination (see next page). During the initiation stage, free radicals are formed which combine with monomers to form an active center monomer free radical. These centers combine with additional monomer molecules during the propagation stage to form growing polymer chains.

This process continues by forming larger polymer chains and resulting in a greater molecular weight until all the free radicals have reacted with the neutral monomers. The termination stage may happen in various ways. During the process of light curing, the monomers are converted to polymers by the C=C double bonds of the methacrylate groups polymerized to C-C single bonds. This reaction brings the monomers closer to one another in order to form chemical bonds resulting in more densely packed molecules (Krämer *et al*, 2008). As a result of this contraction, polymerization stress is induced within the resin and the extent is dependent on the type of resin matrix and filler particle load of the resin. Polymerization shrinkage can clinically compromise the bond between the tooth surface and resin with resultant microleakage, pulpal inflammation and caries (Bhamra and Fleming, 2008; Sakaguchi

and Powers, 2012).



Light activation of camphorquinone

J.C.S.Moraes, M.M.D.S. Sostena and Carlos Roberto Grandini (2011). The Glass Transition Temperature in Dental Composites, Metal, Ceramic and Polymeric Composites for Various Uses, John Cuppoletti (Ed.), ISBN: 978-953-307-353-8, InTech, Available from: http://www.intechopen.com/books/metal-ceramic-and-polymeric-composites-for-various-uses/the-glass-transition-temperature-in-dental-composites

Initiation: $R\bullet + H_2C = CHR \rightarrow I - H_2C - CHR\bullet$ (M; $MR\bullet$)

Propagation: $I - H_2C - CHR\bullet + H_2C = CHR \rightarrow I - H_2C - CHR - H_2C - CHR\bullet$ ($MR\bullet$; M; $MR_2\bullet$)

$....- I - H_2C - CHR - H_2C - CHR\bullet + H_2C = CHR \rightarrow ....- H_2C - CHR\bullet.$ ($MR_{n-1}\bullet$; M; $MR_n\bullet$)

Termination

Combination:

$$.... - H_2C - CHR\bullet + \bullet RHC - CH_2 - .... \rightarrow ....- H_2C - HRC - CHR - CH_2 - ....$$

Disproportionation:

$$.... - H_2C - CHR\bullet + \bullet RHC - CH_2 - .... \rightarrow .... - H_2C = CHR + RH_2C - CH_2 - ....$$

Transfer:

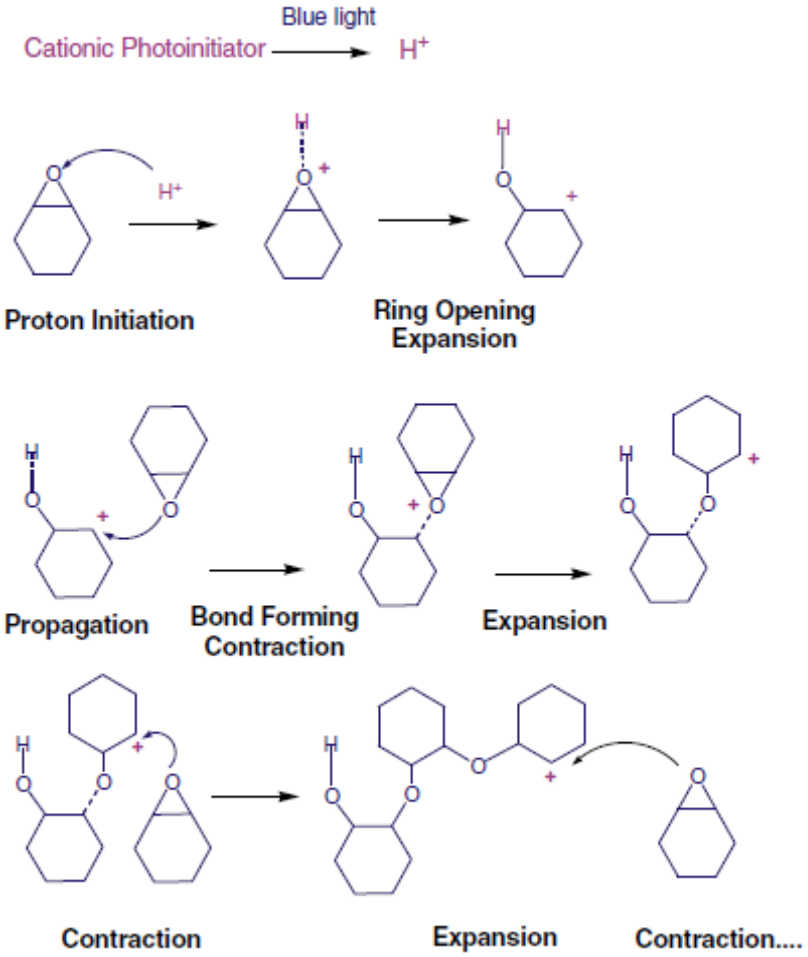
$$....- H_2C - CHR\bullet + H_2C = CHR \rightarrow .... - CH_2 - CHR + H_2C - CHR\bullet$$

Polymerization of methacrylate resins

J.C.S.Moraes, M.M.D.S. Sostena and Carlos Roberto Grandini (2011). The Glass Transition Temperature in Dental Composites, Metal, Ceramic and Polymeric Composites for Various Uses, John Cuppoletti (Ed.), ISBN: 978-953-307-353-8, InTech, Available from: http://www.intechopen.com/books/metal-ceramic-and-polymeric-composites-for-various-uses/the-glass-transition-temperature-in-dental-composites

2.4.3 Light curing of Silorane composites

Polymerization of silorane-based composites (see sketch below) is through cationic reaction processes. This is achieved by ring opening of the oxirane molecule which forms covalent single bonds with its adjacent molecule. The ring opening mechanism of oxirane increases the space occupied by the molecule within the matrix and thus reduces volumetric shrinkage when polymerized. This ensures lower volume shrinkage and polymerization stress (Sakaguchi and Powers, 2012).



Sakaguchi RL, Powers JM (2012), Restorative Materials- Composites and Polymers. Craig's Restorative Dental Materials, 13th Edition, Elsevier, Mosby.

2.5 Light Curing Systems

The increased usage and demand for dental composites has significantly increased the use of curing lights to polymerize composite resins. Curing systems range from Quartz-Tungsten-Halogen (QTH), Light emitting diode (LED), Plasma-Arc (PAC) or laser technology.

Curing lights are used to activate photo-initiators in photo-activated resin restorative materials to initiate polymerization. The most common photo-initiator being camphorquinone, with activity peaks between 470-480 nm. The light intensities and light sources differ amoung curing units, ranging from 300 to more than 1000milliwatts per square centimeter (Krämer *et al*, 2008).

With the introduction of high intensity halogen lights, LED and plasma arc lights, improvements in light technology and with it changes in resin polymerization have been brought about. The aim has been to decrease curing time with less heat production. Factors influencing the light source such as intensity and wavelength together with factors influencing the resin such as its composition and shade, all play a role in ensuring optimal polymerization of the resin (Aravamudhan *et al*, 2006).

2.5.1 Quartz-Tungsten-Halogen curing lights (QTH)

Halogen curing units emit light of a very wide range of wavelengths within the visible spectrum thus requiring filters to narrow this range to 370-550nm (David *et al*, 2007). Halogen bulbs have a shortcoming in that it generates light through heating of tungsten filaments and this heat causes degradation of the curing unit over time. Only a small percentage (<1%) of the energy is given off as light (Visible Light Curing, 2002; Jandt *et al*, 2000; de Araujo *et al*, 2008). The curing process is however increased by the heat generated by curing lights but it may lead to pulpitis and pulpal death in some cases. The limited lifespan of the bulb and degradation of the reflector and the filter over time are drawbacks of halogen curing units (Lindberg *et al*, 2005). This will result in decreased curing effectiveness with resultant poorly cured restorations with insufficient physical properties and a greater failure rate (Soh *et al*, 2003; Jandt *et al*, 2000).

2.5.2 Light emitting diode curing lights (LED)

In the past LED curing devices were not comparable to high power QTH curing devices. However, the current high power LEDs are in the same league or even superior to QTH curing devices due to its increased power output with intensities greater than 800mW/cm^2 (Schattenberg *et al*, 2008). LEDs were developed to overcome the problems associated with halogen curing units. Instead of utilizing heated filaments it makes use of doped semiconductors to generate light. LEDs do not generate energy in the form of heat and therefore suffer little degradation of the curing unit and thus minimal damage to the pulp. The spectral output of LEDs (400 to 500 nm) lies within the absorption spectrum of the photo-initiator camphorquinone, therefore no filters are required (Visible Light Curing, 2002; Jandt et al, 2000; David *et al*, 2007; Schattenberg *et al*, 2008; de Araujo *et al*, 2008; Soh *et al*, 2003; Lindberg *et al*, 2005, Price *et al*, 2004; Kurachi *et al*, 2001; Craig and Powers, 2002).

These high power LEDs are popular amoung clinicians as they save chair-side time. This is possible due to their higher intensities and thus allows the clinician to reduce the curing time. They have a higher life span, are light weight and portable thereby making LEDs a popular curing device in dentistry today (Wiggins *et al*, 2004).

2.5.3 Plasma-Arc curing lights (PAC)

Plasma-Arc lights are heated by current, therefore they give off light and heat, thus they require filters. The difference being that the light intensity given off by Plasma-Arc units is greater than for halogen-based units and thus resulting in a decreased curing time of up to 75% with a narrower wavelength range (Visible Light Curing, 2002; Deb and Sehmi, 2003). However, the rapid setting provided by the PAC curing device results in an increased pulpal temperature, short monomer chains and an inadequate pre-gel phase of the composite during polymerization. Thus this may lead to

increased water absorption, poor aesthetics and decreased longevity of the composite restoration.

In order to shorten the curing time, manufacturers had to increase the power output of curing devices, but this did not result in an improved depth of cure or higher conversion rate but rather caused a significant increase in temperature (Schattenberg *et al*, 2008).

2.5.4 Laser curing lights

These lights emit light at bandwidths of 454-466nm, 472-497nm and 514nm with minimal heat production. A drawback is the narrow size of the light guide which requires the operator to repeat curing cycles especially in cases where the restoration is wider than the curing tip and this increases the working time. Thus a halogen lamp with a larger curing tip and a lower intensity is able to cure a larger restoration in a shorter time than a laser curing device. However, laser curing devices with their small curing tips are ideal in interproximal boxes which are difficult to reach with larger curing tips (Albers, 2000).

2.6 Curing Techniques

The curing technique can be divided into two groups namely, the continuous cure and discontinuous cure techniques.

2.6.1 Continuous cure

For this group of curing techniques the light source provides a continuous light cure sequence which is uninterrupted (Albers, 2000).

2.6.1.1 Uniform continuous cure

This curing unit provides a constant intensity for a specific time period which is determined by the operator (HF Albers, 2000). This constant intensity creates polymerization stress and shrinkage at the resin-tooth interface resulting in marginal gaps and internal stress (Jain and Pershing, 2003).

2.6.1.2 Step cure (soft start)

Initially the composite is cured with a low intensity for a period of time; thereafter it is increased to a higher intensity for a set time period. This is done in order to reduce the polymerization stress by allowing the composite to flow in the gel state during the first phase of low intensity curing. However the reduction in polymerization shrinkage is minimal and it may yield a poorly polymerized composite due to the lower initial light intensity. This also results in an unevenly cured composite since the top layer is cured with a higher light intensity (Albers, 2000; Krämer *et al*, 2008; Hofmann *et al*, 2003).

2.6.1.3 Ramp cure

The light intensity of the curing light is gradually increased or ramped up during curing

of the composite. A low intensity is applied and it is increased gradually to a higher intensity over time. The composite is thus allowed to flow during polymerization and this reduces the polymerization stress. This is done with the intention of passing through each of the light intensities to ensure optimal polymerization. Some studies have shown longer and more stable polymer chains within composites cured in this manner (Anusavice, 2003; Albers, 2000; Jain and Pershing, 2003).

2.6.1.4 High energy pulse cure

Extremely high intensity (1000-2800 mW/cm^2) is applied for 10 seconds. This intensity is three to six times the normal intensity applied to composite during polymerization. This rapid application of very high intensity may result in a weaker composite restoration with shorter polymer chains. This rapid application of high energy may also reduce the diametral tensile strength and result in a brittle composite restoration (Albers, 2000). By increasing the light intensity, the curing time is reduced, thus consuming less time. This method increases the residual stress build-up as a result of insufficient stress relaxation (Anusavice, 2003).

2.6.2 Discontinuous cure (soft cure)

A low intensity or soft light initiates slow polymerization allowing the composite to flow from the free restoration surface to the tooth structure thus reducing polymerization stress at the margins and prevent marginal defects. During the next curing cycle the intensity is greatly increased to completely and optimally cure the composite (Albers, 2000).

2.6.2.1 Pulse delay cure

The restoration is initially under-cured at a low intensity (e.g. 100mW/cm^2 for 3 seconds or 20 seconds) and then paused. The clinician is then able to contour, mould and adjust the occlusion and thereafter completely cure the composite for a final cure with a higher intensity and longer curing time (e.g. 30 seconds). This allows for stress relaxation within the composite and decreases polymerization stress. This method has flaws in that it does not ensure sufficient polymerization at the deepest part of the cavity (Anusavice, 2003; Albers, 2000; Krämer *et al*, 2008).

2.7 Factors affecting the curing of light cured composites

The ability to achieve a complete cure of the resin matrix will result in obtaining the favourable properties of light cured composites. Inadequate polymerization of the composite can result in loss of biocompatibility, colour shifts, loss of retention, breakage and degradation, excessive wear, marginal breakdown, softness and a low depth of cure (Visible Light Curing 2002; Albers, 2000; David *et al*, 2007; Soh *et al*, 2003). If a resin is insufficiently cured, the restoration becomes weaker due to decreased monomer conversion, decreased hardness with an increase in marginal breakdown and wear, resulting in a weak bond to the tooth structure (Aguiar *et al*, 2005; Koupis *et al*, 2004 and 2006; Danesh *et al,* 2004).

Therefore it is important to consider certain factors when using and maintaining a light curing system. These are factors related to composition of the composite or related to the curing unit such as the amount of photo-initiator in the resin, the type of filler, the angle of the light, the shade of resin, the wavelength of the curing light, curing intensity, thickness of the resin, curing time, distance between curing light and the resin, temperature, the amount of heat generated by the curing unit and room temperature polymerization (Visible Light Curing 2002; Albers, 2000; Aguiar *et al*,

2005; Koupis *et al*, 2004 and 2006; de Araujo *et al*, 2008). Factors such as the design and size of the light guide, the condition of the bulb, filters, line voltage and battery power must also be considered (Aguiar *et al*, 2005; Aravamudhan *et al*, 2006).

2.7.1 Concentration of photo-initiator

The amount of photo-initiator varies with composites. Less photo-initiator than what is required is found in some products in an attempt to increase the operator's working time under the operatory light. With time the photo-initiator deteriorates and it varies between light-cured and self-cured composites. The photo-initiator must be in the correct concentration and undergo a reaction within a specific wavelength to achieve a strong and clinically competent restoration (Sakaguchi and Powers, 2012; HF Albers, 2000).

2.7.2 Type of filler particles

According to Garcia *et al* (2006) and Albers (2000), microfilled composites are poorly cured when compared to heavily filled composites. This is due to the increased light scattering in the presence of numerous and smaller sized filler particles. Whereas, this is not the case with microhybrid composites which have less particles and larger filler particles in comparison (Sakaguchi and Powers, 2012).

2.7.3 Angle of the curing light

As the angle at which a composite is cured, diverges from a 90° angle, the energy of the light is deflected away from the composite. This results in a reduced light penetration as can be seen in molar Class II cavities where light penetration is

blocked by the marginal ridge of the adjacent tooth (Albers, 2000).

2.7.4 Shade of composite

More opaque composite shades have higher concentrations of opacifying agents and pigments which cause increased light scattering and this result in a lower curing depth (Aguiar *et al*, 2005). To overcome this problem, increasing the curing time and placing smaller increments of composite are advisable (Garcia *et al*, 2006; Harrington and Wilson, 1993).

A much shallower and slower cure is achieved with darker composite shades than a lighter composite shade (Garcia *et al*, 2006; Harrington and Wilson, 1993). At a depth of 1mm a darker shade achieves only two thirds the depth of cure when compared to translucent shades. A brighter light may reduce the curing time of a dark composite shade (Albers, 2000).

2.7.5 Wavelength

To adequately cure an increment of composite, a wavelength of 400-500 nm is required (Garcia *et al*, 2006).

2.7.6 Light intensity

An optimal light intensity ensures optimal polymerization of a composite restoration and as such problems may occur if the minimum intensity (300mW/cm^2) is not achieved (Albers, 2000). As the light source moves away from the composite surface,

the light intensity decreases. Another factor that reduces the light intensity is the presence of filler particles which cause scattering of the light as it travels through the composite (Sakaguchi and Powers, 2012).

As the light passes through the composite, the light intensity is diminished which is due to light absorption and scattering which is caused by the composite particles. This is an important factor when considering the effectiveness of the composite cure at the bottom layers of the restoration. Hence the light intensity of the curing unit plays a major role in the effectiveness of the depth of cure and polymerization of a composite restoration (Soh *et al*, 2003).

Other factors such as degradation of the bulb drop in the voltage, wear and tear of the curing tip and degradation of the filters may over time reduce light intensity. Therefore curing units should be assessed regularly with the use of a radiometer to ensure adequate light transmission and intensity (Albers, 2000).

2.7.7 Composite increment

Clinicians should be knowledgeable about the maximum composite thickness to be used when packing a cavity as it can greatly limit composite curing. Placing very thick layers will result in an inadequate depth of cure and an insufficiently cured composite, especially the lower portion of a layer. This can negatively affect the mechanical and clinical properties of the composite restoration (Leprince *et al*, 2012; Albers, 2000).

2.7.8 Curing time

A standard curing time of 20 seconds will ensure a curing depth of 2-2.5mm of a light

composite shade (Krämer *et al*, 2008). By increasing the curing time to 40 seconds this will ensure sufficient curing of also the darker shades to a depth of 2.5mm. However, Krämer *et al* (2008) stated that increasing the curing time does not correlate with an increased DOC. The curing time of composites is affected by many factors such as the shade of the composite, the light intensity, the cavity depth, the thickness of the composite layer and whether there is a need to cure through tooth structure (Garcia *et al*, 2006; Sakaguchi and Powers, 2012).

2.7.9 Distance between the light source and composite

Ideally, if the curing tip is positioned at right angles to the composite surface and if there is a distance of less than 1mm between the curing tip and the composite this would ensure optimum polymerization of the composite (Garcia *et al*, 2006; Albers, 2000). The light intensity decreases rapidly as the curing distance is increased. To overcome this the curing time can be increased as in the case of Class II interproximal areas where the curing distance is greater than 1mm (Albers, 2000).

2.7.10 Temperature

At room temperature a composite will cure more quickly and effectively than at colder or warmer temperatures. Therefore they should only be used after being left at room temperature for one hour (Garcia *et al*, 2006; Albers, 2000).

2.8 Depth of Cure of Composite

The depth of cure and the effectiveness thereof are important factors to consider. The top surface hardness of a cured composite is not a true indication of the degree of

polymerization within the entire composite specimen. Two major factors influencing the depth of cure of resins are the exposure time and the curing distance. Other factors to consider are the resin shade, its translucency and the thickness of the resin layer to be cured (Schattenberg *et al*, 2008; Soh *et al*, 2003; Lindberg *et al*, 2005).

Clinically, it is difficult to control the distance between the curing tip and the resin surface because it depends on factors such as the extent of caries progression, the size of the cavity and the position of the cavity. If the curing distance is greater than 2mm, the light is dispersed and this results in ineffective polymerization, which can occur in deep class I cavities and in the proximal boxes of class II cavities which may extend as deep as 6.3mm (+/- 0.7). In this scenario the intensity and quantity of light that could reach the deepest resin layer may be significantly decreased, due to an increased curing distance. Multiple studies have shown that there is a reduction in light intensity as the curing distance is increased, for both tungsten-halogen and LED curing lights (Aravamudhan *et al*, 2006).

Inadequate polymerization of the resin at the tooth interface and exposure to the oral environment may lead to microleakage, marginal discolouration and secondary caries (Aguiar *et al*, 2005). A resin's mechanical properties and its dimensional stability are compromised if there are areas of partially polymerized or unpolymerized monomer. These residual monomers could be cytotoxic, they may leach and produce irritation, and or allergy reactions in patients, thereby compromising the materials biocompatibility. To overcome these unwanted effects, the resin should be adequately cured to an acceptable degree and appropriate depth (Koupis *et al*, 2004; Olivier *et al*, 2012).

According to the International Standard for Dentistry as stipulated in ISO 4049, the depth of cure, water sorption and water solubility as well as three-point flexure strength are the methods by which composite resin's performance are to be assessed (Bhamra and Fleming, 2008).

2.9 Techniques for measuring depth of cure

The depth of cure can be measured indirectly or directly. The scraping test, visual and surface hardness tests are indirect methods to determine the depth of cure. Direct methods such as infrared spectroscopy and laser Raman spectroscopy are costly and time consuming (Soh *et al*, 2003). Numerous methods have been employed to measure depth of cure which include surface hardness tests such as the Wallace indentation hardness test (Lindberg *et al*, 2005; Peutzfeldt, 1997; Van Dijken 2005; Schattenberg *et al*, 2008) and the Knoop hardness test (Polydorou *et al* 2008), with colour dyes, translucency changes, double bond conversion, nuclear magnetic resonance micro-imaging, tactile tests, penetration tests and scraping tests (Fan *et al*, 2002).

Koupis *et al* (2004) compared the scrape technique to a two-body wear test and the penetrometer to a hardness test based on indentation. The scrape technique is a simple, cost-effective and suitable method that dentists can employ in their day-to-day practice in comparing the curing depths of composites (Koupis *et al*, 2004). In another study done by Koupis *et al* in 2004, the researchers measured the hardness of their composite samples with the Knoop hardness test, using a Knoop diamond indenter and a 100g load applied to the sample surface for 30 seconds (Koupis *et al*, 2004 and 2006; de Araujo *et al*, 2008).

According to the 2009 ISO standard, resin based composites need to have a minimum depth of cure of 1.5mm after irradiation according to the manufacturer's instructions. They further define depth of cure as 50% of the length of the cured composite sample after the soft, uncured portion has been scraped away manually. The materials depth of cure is then compared with the 1.5mm requirement to determine if the material meets the ISO standard (Fan *et al* 2002; Aravamudhan *et al*, 2006).

An inadequately cured resin restoration degrades both physically and biologically, therefore the degree of conversion essentially determines the success of the restoration (Lindberg *et al*, 2005; Peutzfeldt, 1997; Van Dijken, 2005; Polydorou *et al*, 2008).

Polydorou *et al* (2008) and Leprince *et al* (2012) proposed that the depth of cure of composite resins is a function of filler size and composition, shade and translucency of the material, intensity of the light source and length of irradiation exposure, monomer composition and polymerization initiator's concentration.

2.9.1 Scrape technique

According to the International Standard ISO 4049, the depth of cure should be measured using the scrape technique. After curing of the composite from one direction, the uncured portion is scraped off and the remaining specimen is measured with a micrometer which is accurate to 0.1mm. This value is then halved and must be greater than 1.5mm (ISO 4049:2009).

2.9.2 Surface hardness technique

These tests are widely used and they provide results which are a good estimate of the actual depth of cure. An example of such a test is the Wallace indentation hardness tester. The hardness is measured at each 0.5mm depth starting at 0.5mm from the top surface of the specimen. The penetration of a Vickers diamond after application of a load of 1g for 15 seconds followed by a test load of 100g for 60 seconds is used to measure the depth of penetration. It therefore measures the degree of softness, thus the higher the Wallace hardness value the softer the material (Lindberg *et al*, 2005).

2.9.3 Spectroscopic techniques

Infra-red spectroscopy and laser Raman spectroscopy are alternate techniques used to measure the depth of cure. However, drawbacks for these techniques are that they are costly and time consuming (Lindberg *et al*, 2005).

2.9.4 Penetration technique

To directly measure the depth of cure a penetrometer designed by Harrington and Wilson (1993) can be utilized. It comprises of a free sliding housing which is in contact with the moveable part of a digital indicator gauge. The free sliding housing is mounted on a stand with a specimen location chamber at its base. This specimen location chamber ensures the correct positioning and placement of the resin specimen situated within the specimen mould. The digital indicator gauge can be zeroed at any position and when it is zeroed at the base plate of the stand, it will measure the thickness of any material between the base plate and the penetration needle. The penetration needle attached at the base of the free sliding housing is a stainless steel rod with a diameter of 0.5mm.

The indentation made by the needle is measured by the digital indicator gauge which can be zeroed at any position during the total travelling distance of 12mm. The reading obtained is accurate to 0.01mm. When the needle is in contact with the base at the beginning, it is at zero on the digital indicator gauge. Therefore when the penetration depth is measured it will provide a direct reading of the thickness of the hardened specimen. According to Harrington and Wilson the needle should always penetrate the centre of the specimen to provide an accurate reading of the depth of cure. This is a simple method as it provides a single reading for the depth of cure. A weight is applied to the instrument to provide a constant force for every reading (Harrington and Wilson, 1993).

Research Objectives

1. To determine and compare the effects of different curing distances on the depth of cure of the three composite resins, namely, Filtek Silorane, Filtek Supreme XT and Z100 (3M ESPE).
2. To determine and compare the effects that different shades have on the depth of cure of the three composite resins Filtek Silorane, Filtek Supreme XT and Z100 (3M ESPE).
3. To determine and compare the effectiveness of measuring the depth of cure using the scrape technique and the penetrometer technique.

Hypotheses

1. The depth of cure will not be reduced as the distance between the tip of the curing light and the composite increases.
2. The depth of cure will not be affected by the different shades.
3. The penetrometer will not provide more accurate information on the depth of cure than the scrape technique.
4. There will be no difference in the depth of cure between Filtek Silorane, Filtek Supreme XT and Z100.

CHAPTER 3
MATERIALS AND METHODS

3.1 Materials

3.1.1 Composite resins

Composite resins are tooth coloured restorative materials which are widely used in dentistry. They are aesthetically pleasing and thus provide the clinician with a very versatile material.

3.1.1.1 Filtek Silorane[1]

Filtek Silorane is a low shrink posterior restorative material which is classified as a microhybrid composite (Fig.1). Its chemical composition consists of siloxanes and oxiranes which provide the low shrinkage properties of this material. Filtek Silorane is a single opacity system with four radiopaque shades. Shades A2, B2 and C2 (these represent a light, medium and dark composite shade) were utilised for determining the depth of cure at the curing distances of 0, 1, 2, 3 and 5mm.

3.1.1.2 Filtek Supreme XT[1]

Filtek Supreme XT is an universal direct restorative, nanocomposite material (Fig.1).

[1]3M ESPE, Seefeld, Germany

[2]Hawe Neos Dental, CH-6934, Bioggio, Switzerland

[3]Demetron Research Corporation, Danbury, CT, USA

[4]NSK Manufacturers, Japan Micrometer MFC, Co. Ltd

It is a combination of non-agglomerated 20nm nanosilica filler and loosely bound agglomerated zirconia/silica nanoclusters. They consist of agglomerates of primary zirconia/ silica particles with size of 5-20nm fillers.

The cluster particle size range is 0.6 to 1.4 microns. Filtek Supreme XT is available in dentine, enamel, body and translucent opacities. Body shades A2B, B2B and C2B were utilised for determining the depth of cure at the curing distances of 0, 1, 2, 3 and 5mm. The body opacity shades were selected because they are radiopaque.

3.1.1.3 Z100[1]

Z100 is a radiopaque microfilled anterior and posterior restorative material with zirconia/silica filler system (Fig. 1). It is offered in twelve shades of which eight are Vita shades and four are speciality shades. Shades A2, B2 and C2 were utilised for determining the depth of cure at the curing distances of 0, 1, 2, 3 and 5mm.

3.1.2 Hawe Transparent Strips[2]

Hawe Transparent Strips (Fig. 2) were used to cover the uncured resin specimen applied to the central cavity of the specimen mould prior to curing the composite.

3.2 Apparatus

3.2.1 Light Emitting Diode (LED) curing light

The Elipar Freelight 2[1] (Fig. 3) was used to cure each specimen for 20 seconds as recommended by the manufacturer (3M ESPE) with a light intensity of 600mW/cm^2.

3.2.2 Radiometer

A Demetron radiometer[3] (Model 100) (Fig. 4) was utilised to monitor the light intensity of the LED curing light at regular intervals i.e. after curing 10 specimens.

3.2.3 Penetrometer

A revised design of the Harrington and Wilson penetrometer (Fig. 5) (Harrington and Wilson, 1993) was utilised in measuring and determining the depth of cure of the composite resin specimens. The penetrometer consists of four parts; a free sliding housing (Fig. 6), a specimen location chamber (Fig. 7), a digital indicator gauge (Fig. 8) and a penetration needle (Fig. 8). The free sliding housing is mounted on a stand with a specimen location chamber at its base. This specimen location chamber ensures the correct positioning and placement of the resin specimen situated within the specimen mould. The digital indicator gauge is also in contact with the free sliding housing.

The gauge can be zeroed at any position and when it is zeroed at the base plate of the stand, it will measure the thickness of any material between the base plate and the penetration needle. The penetration needle attached at the base of the free sliding housing is a stainless steel rod with a diameter of 0.5mm.

A weight (Fig. 9) was placed on the sliding house table to ensure that a constant force was applied by the penetration needle. The resultant force applied by the needle is 12.5N with a resultant stress of 62MPa.

3.2.4 Curing Alignment Device

3.2.4.1 Stainless Steel Alignment Tube

The stainless steel alignment tube with an internal diameter of 24mm, 21mm high and 2mm thick (Fig. 10), includes a stainless steel light guide alignment ring (24mm in diameter, 5mm thick with a central cavity of 7mm in diameter) (Fig. 11) and a stainless steel base plate (24mm in diameter and 5mm thick) (Fig. 12). The base plate was painted black to prevent light reflection during light curing of the composite specimens. The light guide alignment ring aligns the tip of the curing light with respect to the composite specimen within the specimen mould.

3.2.4.2 Stainless Steel Specimen Mould

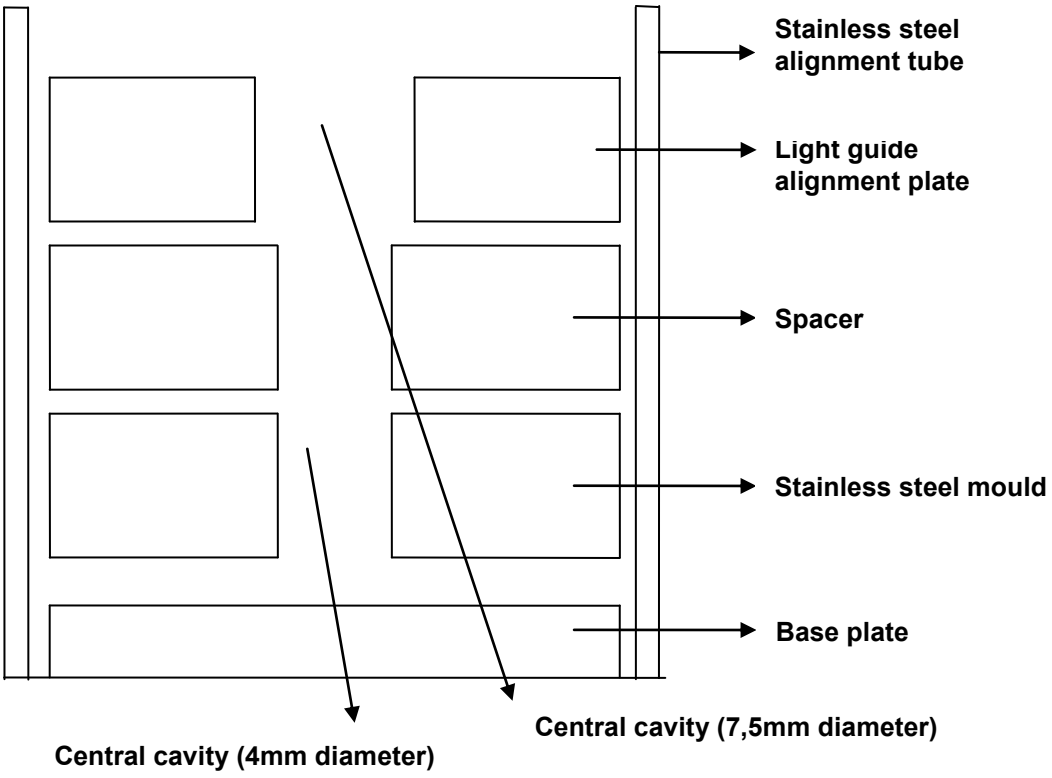
The specimen mould is a stainless steel plate which is 24mm in diameter with a central cylindrical cavity of 4mm in diameter (Fig. 13). The height of the mould used was 6mm for preparation of the composite specimens.

3.2.4.3 Stainless Steel Spacers

Stainless steel spacers (Fig. 14) of 1, 2, 3 and 5mm thick were utilised to produce accurate and reproducible curing distances.

3.2.4.4 Assembling of the Curing Alignment Device

The composite specimens were cured utilising the curing alignment device and its individual components. Sketch 1 illustrates the assembling of the curing alignment device. To cure the specimens at distances of 1, 2, 3 and 5mm, the respective spacers were utilised. Whereas, for the distance of 0mm, no spacer was utilised and the curing tip of the curing light was placed directly in contact with the composite specimen covered by a Hawe transparent strip.



Sketch 1: Curing alignment device

3.3 Experimental Method

3.3.1 Curing of the composite resin specimens at different distances

To determine the effect of the various curing distances (0, 1, 2, 3 and 5mm) and the different composite shades (A2, B2 and C2) on the depth of cure at 600mW/cm^2 for 20 seconds, a total of 450 specimens (10 specimens per test variable per composite resin) were prepared.

The composite specimens for the three different composites, Filtek Silorane, Filtek Supreme XT and Z100, were prepared by placing the composite within the central cavity of the mould and covered by Hawe strips on both ends of the mould.

The mould was placed in the alignment tube and light cured from the top with the Elipar Freelight 2 (Fig. 15) for 20 seconds at distances of 0, 1, 2, 3 and 5mm. The curing intensity of the curing light was measured after every ten specimens cured, using a Demetron radiometer.

3.3.2 Depth of cure measurements

3.3.2.1 Scrape technique

After the specimens were measured for the depth of cure by the penetrometer, the specimens were gently removed from the stainless steel mould and the uncured

portion was scraped away with a plastic knife using light finger pressure. The cured portion of the specimen was cleaned with a gauze and alcohol. Thereafter the depth of cure was determined by measuring the thickness of the hardened portion of the specimen using the NSK Micrometer[4] (Fig. 16). The readings were recorded in millimeters and then divided by two to obtain the depth of cure according to the ISO standards (ISO 4049:2009).

3.3.2.2 Penetrometer

The composite specimens were removed from the alignment tube and the Hawe strips were discarded. The specimen was placed beneath the penetrometer needle, in the specimen location chamber (Fig. 7), such that the mould was inverted and positioned with the needle pointing at the centre of the specimen. The needle penetrated the uncured composite for 10 seconds and a direct reading of the depth of cure was read off the digital gauge. The accuracy of the reading was to 0.01mm. The readings were divided by two and recorded as such, according to the ISO standards (ISO 4049:2009).

3.4 Statistical Analysis

All data was captured in an electronic database by the statistician. Data capturing was verified and validation checks were performed.

The statistical analyses of the depth of cure comprised of:

- An analysis of variance (ANOVA) for a factorial design with three factors, namely composites, curing distances and shades. Main effects and interactions were analyzed, followed by multiple comparisons (e.g. Bonferonni) as applicable.

- Correlating measurements by the scrape and the penetrometer technique. Mean scrape and penetrometer technique values in specific subgroups (e.g. for a specific composite or shade) were compared by two-sample t-tests if required.

All statistical procedures were performed on SAS®, Release 9.1.3, running under Microsoft® Windows® Vista® Business. Statistical tests were two-sided and p-values ≤ 0.05 were considered significant.

Fig. 1: Filtek Silorane, Filtek Supreme XT, Z100 Composite resins

Fig. 2: Hawe Transparent Strips

Fig. 3: Elipar Freelight 2, curing light

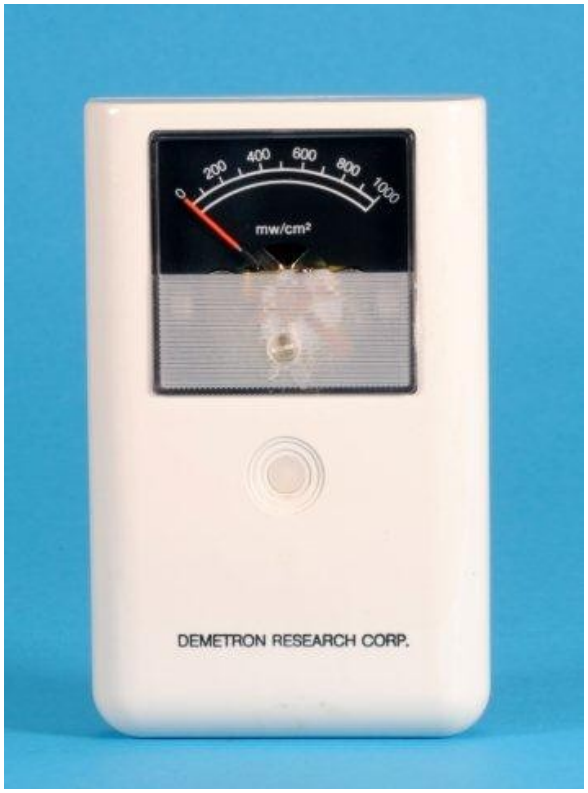


Fig. 4: Demetron Radiometer

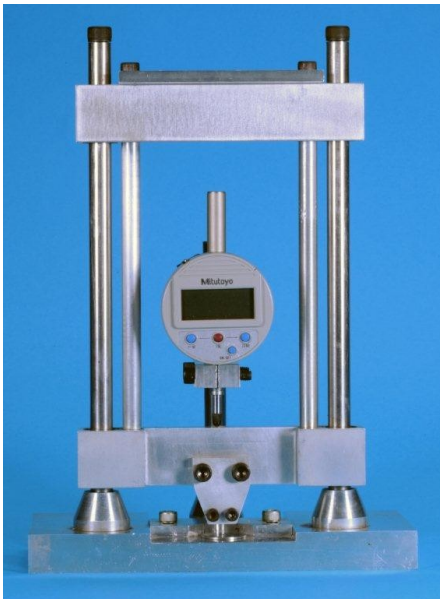


Fig. 5: Penetrometer

Fig. 6: Free sliding housing

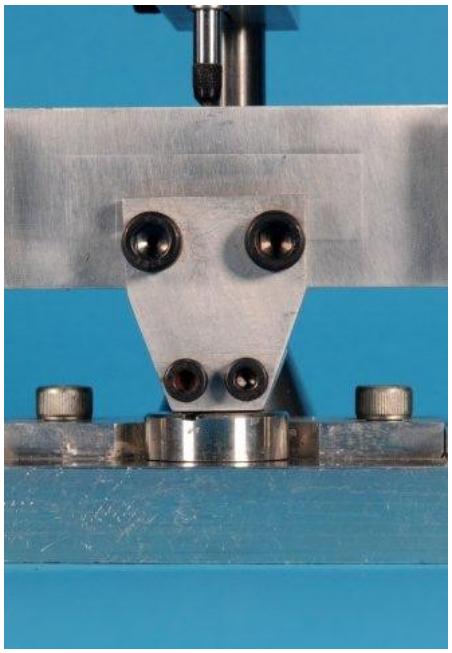


Fig. 7: Specimen location chamber

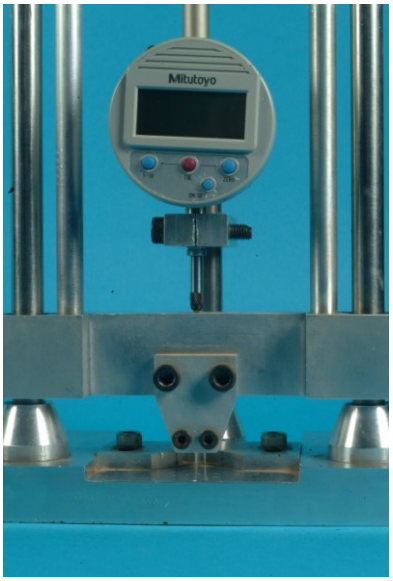


Fig. 8: Digital indicator gauge and penetration needle

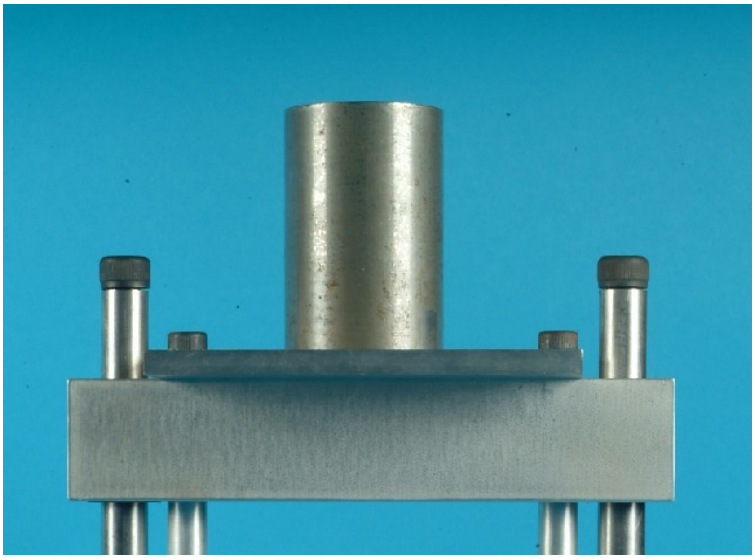


Fig. 9: Weight

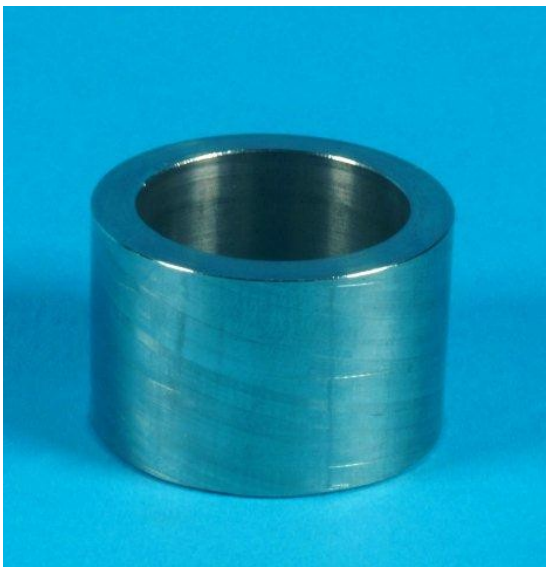


Fig. 10: Stainless steel alignment tube

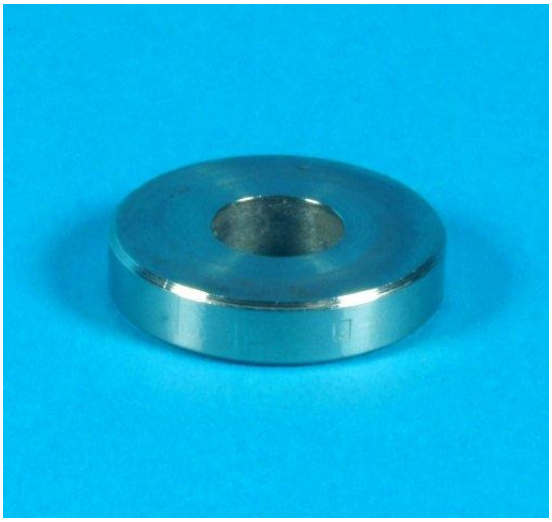


Fig. 11: Light guide alignment ring

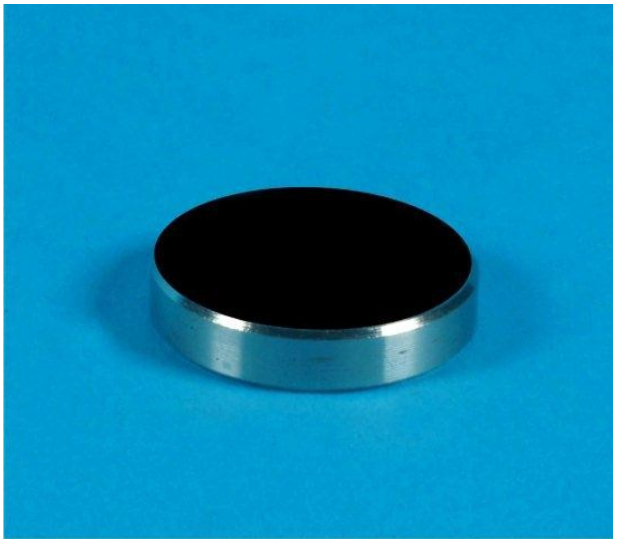


Fig. 12: Black painted stainless steel base plate

Fig. 13: Stainless steel specimen mould with central cylindrical cavity

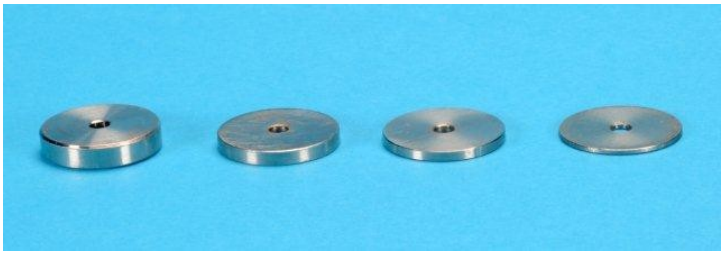


Fig. 14: Stainless steel spacers (1, 2, 3 and 5mm thick)

Fig. 15: Elipar Freelight 2 curing light in light guide alignment ring

Fig. 16: NSK digital micrometer with cured composite specimen

CHAPTER 4

RESULTS

The depth of cure (DOC) of Filtek Silorane, Filtek Supreme XT and Z100 were determined by light curing the specimens for 20 seconds at an intensity of 600mW/cm^2. The DOC of shades A2, B2 and C2 of each composite were measured at curing distances of 0, 1, 2, 3 and 5mm, using the scrape and the penetrometer techniques.

4.1 Results for the scrape technique

4.1.1 The effect of curing distance on the DOC of Silorane shades A2, B2 and C2

When comparing the DOC of Silorane shade A2 (Table 1, Graph 1) at curing distances 0, 1, 2, 3 and 5mm, the DOC decreased significantly as the curing distance increased ($p<0.05$). The same pattern was displayed for Silorane B2 and C2 at the various curing distances.

4.1.2 The effect of shade on the DOC of Silorane A2, B2 and C2 at curing distances of 0, 1, 2, 3 and 5mm

When comparing Silorane shades A2, B2 and C2 (Table 1, Graph 2) at 0mm there is a significant difference in DOC between the three shades ($p<0.0001$). Silorane B2 (2.23mm) has a higher DOC at 0mm than Silorane A2 (2.16mm) and both Silorane A2 and B2 have a higher DOC than C2 (2.00mm). At a curing distance of 1mm this

pattern continues ($p<0.0001$) with DOC of Silorane B2 being 2.09mm, Silorane A2 2.02mm and Silorane C2 1.90mm. However, at curing distances of 2mm ($p=0.0531$), 3mm ($p=0.0597$) and 5mm ($p=0.7919$) there was no significant difference in the DOC between Silorane A2 and B2. The general trend shown was that Silorane A2 and B2 display greater DOC than C2 at 0, 1, 2, 3 and 5mm ($p<0.0001$). As the curing distance increased, the DOC of Silorane A2 and B2 were similar whereas C2 was significantly lower than A2 and B2 at each curing distance.

4.1.3 The effect of curing distance on the DOC of Filtek Supreme XT shades A2, B2 and C2

With Supreme XT A2, the DOC decreased as the curing distance increased (Table 2, Graph 3) ($p<0.05$). However, there was no significant difference in the DOC ($p=0.4290$) at the 1mm (2.45mm) and 2mm (2.43mm) distances. Supreme XT B2 showed a significant decrease in DOC as the curing distance increased ($p<0.0001$). Supreme XT C2 displayed the same pattern as A2, there was also no significant difference in the DOC ($p=0.8041$) between 1mm (2.21mm) and 2mm (2.21mm).

4.1.4 The effect of shade on the DOC of Supreme XT A2, B2 and C2 at curing distances of 0, 1, 2, 3 and 5mm

Supreme XT A2 displayed a greater DOC than Supreme XT B2 ($p<0.05$) (Table 2, Graph 4), except at a curing distance of 1mm where A2 (2.45mm) and B2 (2.44mm) displayed a similar DOC ($p=0.6660$). Supreme XT A2 and B2 displayed a greater DOC than C2 at each curing distance ($p<0.0001$).

4.1.5 The effect of curing distance on the DOC of Z100 shades A2, B2 and C2

With Z100 A2 (Table 3, Graph 5) the DOC decreased significantly as the curing distance increased ($p<0.05$), except between 0mm (3.38mm) and 1mm (3.35mm) where there was no significant difference in the DOC ($p=0.2884$). Z100 B2 followed the similar trend as A2 except at curing distances of 1mm (3.35mm) and 2mm (3.35mm) where the DOC was the same ($p=1.0000$). For C2 it was found that at curing distances of 2mm (2.83mm) and 3mm (2.81mm) there was no significant difference in the curing depths ($p=0.5390$). For the remaining curing distances for C2 there was a significant decrease in the DOC as the curing distance increased ($p<0.05$).

4.1.6 The effect of shade on the DOC of Z100 A2, B2 and C2 at curing distances of 0, 1, 2, 3 and 5mm

When comparing Z100 A2 (Table 3, Graph 6) to B2 there was no significant difference in the DOC at 0mm ($p=0.0503$) where B2 (3.45mm) displayed a greater curing depth than A2 (3.38mm). At 1mm A2 and B2 had the same curing depth (3.35mm) ($p=0.9559$) and at 3mm there was no significant difference ($p=0.5014$) in the DOC of A2 (3.18mm) and B2 (3.16mm). Shade A2 and B2 displayed significantly greater curing depths than C2 ($p<0.05$) at each curing distance.

4.1.7 Comparing Silorane, Supreme XT and Z100

The DOC of Z100 A2 is greater than Supreme XT A2 and Silorane A2 ($p<0.0001$) at curing distances of 0, 1, 2, 3 and 5mm (Graph 7). Supreme XT A2 provided a greater curing depth than Silorane A2 at each of the curing distances ($p<0.0001$). This can be seen at a curing distance of 0mm where Z100 A2 has a DOC 23.37% higher than Supreme XT A2 and 36.09% higher than Silorane A2. Supreme XT A2 has a DOC 16.60% higher than Silorane A2 at 0mm.

The same pattern can be seen when comparing Silorane B2, Supreme XT B2 and Z100 B2 (Graph 8) ($p<0.0001$). At a curing distance of 0mm, Z100 B2 has a DOC 26.09% higher than Supreme XT B2 and 35.36% higher than Silorane B2. Supreme XT B2 has a DOC 12.55% higher than Silorane B2 at 0mm.

The same follows for Silorane C2, Supreme XT C2 and Z100 C2 (Graph 9) ($p<0.0001$). This can be seen at a curing distance of 0mm where Z100 C2 has a DOC of 25.55% higher than Supreme XT C2 and 37.69% higher than Silorane C2. Supreme XT C2 has a DOC 16.32% higher than Silorane C2 at 0mm.

Overall, all three shades of Z100 provided the highest curing depths at each curing distance of 0, 1, 2, 3 and 5mm, followed by Supreme XT and lastly Silorane (Graph 7, 8 and 9) ($p<0.0001$).

4.2 Results for the Penetrometer

4.2.1 The effect of curing distance on the DOC of Silorane shades A2, B2 and C2

Silorane A2, B2 and C2 (Table 4, Graph 10) displayed a decrease in the DOC as the curing distance increased from 0mm to 5mm ($p<0.05$).

4.2.2 The effect of shade on the DOC of Silorane A2, B2 and C2 at curing distances of 0, 1, 2, 3 and 5mm

At curing distances of 0, 1, 2 and 3mm (Table 4, Graph 11) there were no significant differences between the DOC of Silorane A2 compared to B2. At 0mm A2 provided a mean DOC of 2.10mm and B2 provided a mean DOC of 2.11mm ($p=0.6183$). At 1mm

A2 provided a mean DOC of 1.95mm and B2 provided a mean DOC of 1.96mm ($p=0.7031$). At 2mm the same pattern (A2=1.89mm and B2=1.89mm) followed with a resultant p-value of 0.8237 and at 3mm a p-value of 0.0058 was obtained. Silorane B2 and C2 display no significant difference ($p=0.1930$) in DOC at a curing distance of 5mm, where B2 has a mean DOC of 1.61 mm and C2 has a mean DOC of 1.58mm. As the curing distance increased A2 and B2 provide a greater DOC than C2 ($p<0.05$).

4.2.3 The effect of curing distance on the DOC of Filtek Supreme XT shades A2, B2 and C2

As the curing distance increased for A2, B2 and C2 (Table 5, Graph 12) the DOC decreased for each shade. However, for C2 at 1mm (2.21mm) and 2mm (2.19mm) there was no significant difference in the DOC ($p=0.3305$).

4.2.4 The effect of shade on the DOC of Supreme XT A2, B2 and C2 at 0, 1, 2, 3 and 5mm

Supreme XT A2 (Table 5, graph 13) displayed greater curing depths than B2 and C2 at each curing distance ($p<0.05$). B2 also displayed the same pattern, showing greater curing depths than C2 ($p<0.05$), at curing distances of 0, 1, 2, 3 and 5mm. Thus C2 displayed the lowest curing depth when compared to both A2 and B2.

4.2.5 The effect of curing distance on the DOC of Z100 shades A2, B2 and C2

For A2 (Table 6, Graph 14) there is no significant difference in the DOC at 0mm (3.35mm) and 1mm (3.33mm) with a p-value of 0.2905. As the curing distance increased from 1 to 5mm, the DOC decreased for A2 ($p<0.05$). For B2 the DOC decreased as the curing distance increased, except there was no significant

difference in the DOC at 1mm (3.33mm) and 2mm (3.32mm) (p=0.8466). At 2mm (2.81mm) and 3mm (2.80mm), C2 displayed no significant difference in the DOC (p=0.9045). For the remaining curing distances for C2, there was a significant difference in the DOC with each increase in the curing distance (p<0.05).

4.2.6 The effect of shade on the DOC of Z100 A2, B2 and C2 at 0, 1, 2, 3 and 5mm

At 0mm B2 (3.43mm) displayed a greater DOC than A2 (3.35mm) (p=0.0602). At 1mm B2 (3.33mm) and A2 (3.33mm) displayed the same DOC (p=0.8503) that was not significantly higher (Table 6, Graph 15). At 2mm and 3mm there were no significant differences in the DOC between A2 and B2 with p-values of 0.1423 and 0.6202 respectively. A2 and B2 displayed significantly greater curing depths than C2 at each curing distance (p<0.05).

4.2.7 Comparing Silorane, Supreme XT and Z100

The DOC of Z100 A2 at 0mm is greater than Supreme XT A2 by 23.28% and by 37.31% greater than Silorane A2 (Graph 16). Supreme XT A2 has a DOC 18.29% higher than Silorane A2 at 0mm. This pattern can be observed for curing distances of 1, 2, 3 and 5mm (p<0.0001), where Z100 provides the highest DOC, followed by Supreme XT and Silorane.

The same pattern can be observed for shade B when comparing Silorane B2, Supreme XT B2 and Z100 B2 (Graph 17) (p<0.0001). This can be seen at 0mm where Z100 B2 has a DOC 26.24% higher than Supreme XT B2 and 38.48% higher than Silorane B2. Supreme XT B2 has a DOC 16.60% higher than Silorane B2 at 0mm.

The same follows for Silorane C2, Supreme XT C2 and Z100 C2 (Graph 18) ($p<0.0001$). At a curing distance of 0mm Z100 C2 has a DOC 25.47% higher than Supreme XT C2 and 39.31% higher than Silorane C2. Supreme XT C2 has a DOC 18.57% higher than Silorane C2.

Overall, all three shades of Z100 provided the highest curing depths at each curing distance of 0, 1, 2, 3 and 5mm, followed by Supreme XT and lastly Silorane (Graph 16, 17 and 18) ($p<0.0001$).

4.3 Comparing the scrape and penetrometer techniques in determining the DOC

4.3.1 Silorane A2, B2 and C2

For all shades of Silorane, the scrape technique provided a significantly higher DOC than the penetrometer at curing distances of 0, 1, 2, 3 and 5mm ($p<0.05$) (Graph 19, 20 and 21). This can be seen at 0mm for A2, where the scrape technique (Table 1) had a 2.78 % higher DOC than the penetrometer (Table 4) ($p<0.0001$). For B2 at 0mm, the scrape technique had a 5.38% higher DOC than the penetrometer ($p<0.0001$) and the same for C2 with the scrape technique having a 3.5% higher DOC ($p<0.0001$).

4.3.2 Supreme XT A2, B2 and C2

For shade A2 at 0mm there was no significant difference ($p=0.0879$) (Graph 22) between the scrape technique (2.59mm) (Table 2) and the penetrometer (2.57mm) (Table 5). At 1mm however the scrape technique and the penetrometer recorded the

same DOC (2.45mm) (p=0.7518). At 2, 3 and 5mm the scrape technique provided a greater DOC than the penetrometer (p<0.05).

For shade B2 (graph 23) there was no significant difference in the DOC readings between the scrape technique and the penetrometer at 0mm (p=0.2890), 2mm (p=0.1611), 3mm (p=0.6758) and 5mm (0.0990) (Table 2 and 5). At 1mm the scrape technique had a higher DOC than the penetrometer (p=0.0092).

For shade C2 there was no significant difference in the DOC between the scrape technique and penetrometer (Graph 24) as seen at 0mm (p=0.0887), 1mm (p=0.2400), 3mm (p=0.9537) and 5mm (p=0.0861) (Table 2 and 5). At 2mm however the scrape technique recorded a higher DOC reading than the penetrometer (p=0.0006).

4.3.3 Z100 A2, B2 and C2

Z100 A2 (Graph 25) had a significantly higher DOC for the scrape technique at 0mm (p=0.0151) and at 1mm (p=0.0007). However the penetrometer and the scrape technique's DOC recorded no significant difference at 2mm (p=0.7369), 3mm (p=0.0811) and 5mm (p=0.0622) (Tables 3 and 6).

For shade B2 there was no significant difference in the DOC between the scrape technique and the penetrometer (Graph 26) at 1mm (p=0.1095) and 3mm (p=0.7459). However, the scrape technique recorded a significantly higher DOC at 0mm (p=0.0226), 2mm (p<0.0001) and 5mm (p=0.0119) than the penetrometer (Table 3 and 6).

For shade C2 (Graph 27), there was no significant difference in the DOC between the scrape technique and the penetrometer at 0mm (p=0.05), 2mm (p=0.2677), 3mm (p=0.3948) and 5mm (p=0.2100). At 1mm the scrape technique recorded a significantly higher DOC (p=0.0259) (Table 3 and 6).

Table 1: Scrape technique. Depth of cure (mm) of Filtek Silorane composite resin (shades A2, B2 and C2) cured through air (0,1, 2, 3 and 5 mm) using an Elipar Freelight 2 (LED) curing unit (600mW/cm^2, 20 seconds)

Spacer (mm)	0			1			2			3			5		
Shade of Resin	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2
Sample No															
1	2.11	2.18	2.02	2.10	2.12	1.92	1.87	2.03	1.83	1.91	1.90	1.69	1.84	1.63	1.64
2	2.12	2.18	2.00	2.05	2.11	1.92	1.85	1.98	1.83	1.90	1.93	1.75	1.74	1.69	1.64
3	2.15	2.25	1.99	1.94	2.13	1.88	2.02	2.01	1.86	1.89	1.88	1.71	1.73	1.80	1.65
4	2.15	2.27	1.94	1.96	2.10	1.90	1.97	2.01	1.85	1.93	1.88	1.74	1.75	1.75	1.62
5	2.19	2.26	2.04	2.03	2.08	1.92	2.02	1.98	1.85	1.93	1.88	1.71	1.71	1.73	1.60
6	2.15	2.32	2.01	1.99	2.08	1.93	2.03	1.99	1.89	1.88	1.85	1.72	1.73	1.77	1.59
7	2.18	2.22	2.00	2.02	2.02	1.94	2.00	2.06	1.86	1.91	1.82	1.79	1.72	1.79	1.65
8	2.18	2.20	1.96	2.07	2.11	1.92	1.96	2.01	1.87	1.85	1.85	1.72	1.69	1.70	1.62
9	2.19	2.25	2.05	2.01	2.09	1.87	2.04	2.02	1.85	1.94	1.95	1.74	1.74	1.77	1.65
10	2.18	2.23	2.00	2.02	2.09	1.85	1.99	2.03	1.86	1.93	1.80	1.75	1.79	1.76	1.68
Mean Depth of Cure	2.16	2.23	2.00	2.02	2.09	1.90	1.97	2.01	1.85	1.90	1.87	1.73	1.74	1.74	1.63
Maximum Depth	2.19	2.32	2.05	2.10	2.13	1.94	2.04	2.06	1.89	1.94	1.95	1.79	1.84	1.80	1.68
Minimum Depth	2.11	2.18	1.94	1.94	2.02	1.85	1.85	1.98	1.83	1.85	1.80	1.69	1.69	1.63	1.59

Table 2: Scrape technique. Depth of cure (mm) of Filtek Supreme XT composite resin (shades A2, B2 and C2) cured through air (0,1, 2, 3 and 5 mm) using an Elipar Freelight 2 (LED) curing unit (600mW/cm^2, 20 seconds)

Spacer (mm)	0			1			2			3			5		
Shade of Resin	A2B	B2B	C2B	A2B	B2B	C2B	A2B	B2B	C2B	A2B	B2B	C2B	A2B	B2B	C2B
Sample No															
1	2.61	2.64	2.39	2.32	2.40	2.20	2.50	2.40	2.17	2.23	2.17	2.08	2.10	2.12	1.99
2	2.54	2.51	2.41	2.45	2.39	2.27	2.46	2.36	2.12	2.25	2.23	2.04	2.20	2.11	1.98
3	2.61	2.57	2.40	2.44	2.48	2.13	2.44	2.34	2.26	2.22	2.22	2.10	2.18	2.10	2.02
4	2.63	2.57	2.39	2.42	2.43	2.17	2.45	2.33	2.21	2.21	2.18	2.13	2.09	2.15	1.96
5	2.55	2.52	2.40	2.54	2.36	2.28	2.44	2.37	2.24	2.21	2.17	2.01	2.16	2.15	2.03
6	2.66	2.55	2.40	2.43	2.48	2.21	2.41	2.29	2.17	2.28	2.19	2.11	2.13	2.15	1.99
7	2.58	2.55	2.39	2.49	2.46	2.21	2.45	2.35	2.24	2.28	2.26	2.10	2.19	2.16	2.00
8	2.60	2.55	2.30	2.48	2.47	2.08	2.40	2.34	2.22	2.27	2.19	2.11	2.29	2.14	2.02
9	2.52	2.51	2.40	2.45	2.47	2.29	2.42	2.43	2.24	2.32	2.19	2.05	2.24	2.08	2.01
10	2.57	2.50	2.40	2.49	2.46	2.29	2.38	2.37	2.22	2.22	2.27	2.11	2.24	2.08	1.95
Mean Depth of Cure	2.59	2.55	2.39	2.45	2.44	2.21	2.43	2.35	2.21	2.25	2.20	2.08	2.18	2.12	1.99
Maximum Depth	2.66	2.64	2.41	2.54	2.48	2.29	2.50	2.43	2.26	2.32	2.27	2.13	2.29	2.16	2.03
Minimum Depth	2.52	2.50	2.30	2.32	2.36	2.08	2.38	2.29	2.12	2.21	2.17	2.01	2.09	2.08	1.95

Table 3: Scrape technique. Depth of cure (mm) of Z100 composite resin (shades A2, B2 and C2) cured through air (0,1, 2, 3 and 5 mm) using an Elipar Freelight 2 (LED) curing unit (600mW/cm^2, 20 seconds)

Spacer (mm)	0			1			2			3			5		
Shade of Resin	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2
Sample No															
1	3.27	3.36	3.24	3.36	3.40	3.05	3.29	3.37	2.94	3.07	3.21	2.79	2.93	3.01	2.54
2	3.36	3.39	3.22	3.34	3.37	3.12	3.18	3.35	2.92	3.24	3.16	2.83	3.10	3.00	2.63
3	3.42	3.38	3.27	3.35	3.36	3.11	3.29	3.35	2.88	3.30	3.09	2.77	3.12	2.97	2.63
4	3.39	3.23	3.24	3.38	3.36	3.14	3.20	3.35	2.67	3.16	3.24	2.79	3.10	3.08	2.58
5	3.40	3.34	3.28	3.35	3.29	2.98	3.32	3.38	2.84	3.16	3.08	2.80	3.07	3.00	2.57
6	3.40	3.58	3.23	3.36	3.34	3.04	3.33	3.36	2.77	3.25	3.12	2.81	3.11	2.89	2.63
7	3.39	3.53^	3.16	3.35	3.36	2.99	3.34	3.36	2.79	3.14	3.19	2.85	3.09	3.00	2.65
8	3.40	3.60	3.17	3.35	3.33	3.01	3.34	3.34	2.78	3.06	3.11	2.84	3.14	2.97	2.60
9	3.40	3.56	3.21	3.31	3.36	2.98	3.25	3.33	2.88	3.14	3.15	2.84	3.16	2.95	2.66
10	3.36	3.55	3.15	3.37	3.36	3.10	3.29	3.35	2.83	3.26	3.24	2.84	3.13	2.97	2.56
Mean Depth of Cure	3.38	3.45	3.21	3.35	3.35	3.05	3.28	3.35	2.83	3.18	3.16	2.81	3.09	2.98	2.60
Maximum Depth	3.42	3.60	3.28	3.38	3.40	3.14	3.34	3.38	2.94	3.30	3.24	2.85	3.16	3.08	2.66
Minimum Depth	3.27	3.23	3.15	3.31	3.29	2.98	3.18	3.33	2.67	3.06	3.08	2.77	2.93	2.89	2.54

Table 4: Penetrometer technique. Depth of cure (mm) of Filtek Silorane composite resin (shades A2, B2 and C2) cured through air (0, 1, 2, 3 and 5 mm) using an Elipar Freelight 2 (LED) curing unit (600mW/cm^2, 20 seconds)

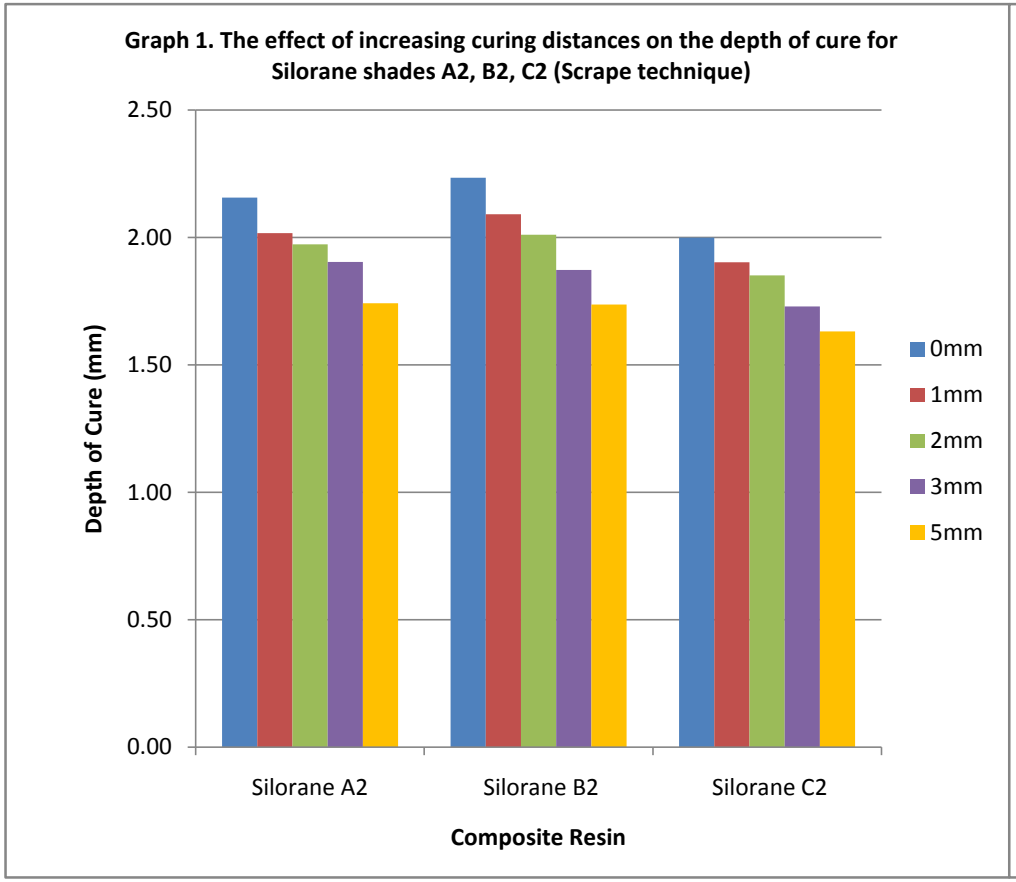
Spacer (mm)	0			1			2			3			5		
Shade of Resin	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2
Sample No															
1	2.05	2.05	1.93	2.04	1.95	1.88	1.81	1.89	1.80	1.82	1.88	1.61	1.80	1.45	1.61
2	2.06	2.10	1.96	1.99	2.01	1.85	1.67	1.90	1.82	1.85	1.84	1.67	1.68	1.60	1.50
3	2.09	2.10	1.90	1.91	2.04	1.82	1.90	1.93	1.80	1.79	1.80	1.65	1.73	1.67	1.60
4	2.11	2.12	1.86	1.92	1.94	1.84	1.96	1.84	1.81	1.86	1.77	1.63	1.70	1.65	1.58
5	2.11	2.14	1.98	2.00	1.96	1.83	2.01	1.87	1.82	1.85	1.78	1.66	1.66	1.46	1.54
6	2.08	2.13	1.91	1.94	1.97	1.84	1.97	1.92	1.82	1.83	1.75	1.65	1.65	1.66	1.57
7	2.12	2.11	1.94	1.95	1.86	1.89	1.91	1.93	1.78	1.83	1.71	1.73	1.63	1.65	1.58
8	2.12	2.10	1.93	2.03	1.99	1.84	1.88	1.86	1.77	1.78	1.73	1.66	1.62	1.68	1.61
9	2.14	2.16	1.97	1.85	1.96	1.73	1.91	1.89	1.78	1.84	1.76	1.70	1.69	1.65	1.60
10	2.15	2.08	1.91	1.94	1.96	1.83	1.94	1.86	1.76	1.90	1.63	1.73	1.73	1.70	1.61
Mean Depth of Cure	2.10	2.11	1.93	1.95	1.96	1.83	1.89	1.89	1.79	1.83	1.76	1.67	1.69	1.61	1.58
Maximum Depth	2.15	2.16	1.98	2.04	2.04	1.89	2.01	1.93	1.82	1.90	1.88	1.73	1.80	1.70	1.61
Minimum Depth	2.05	2.05	1.86	1.85	1.86	1.73	1.67	1.84	1.76	1.78	1.63	1.61	1.62	1.45	1.50

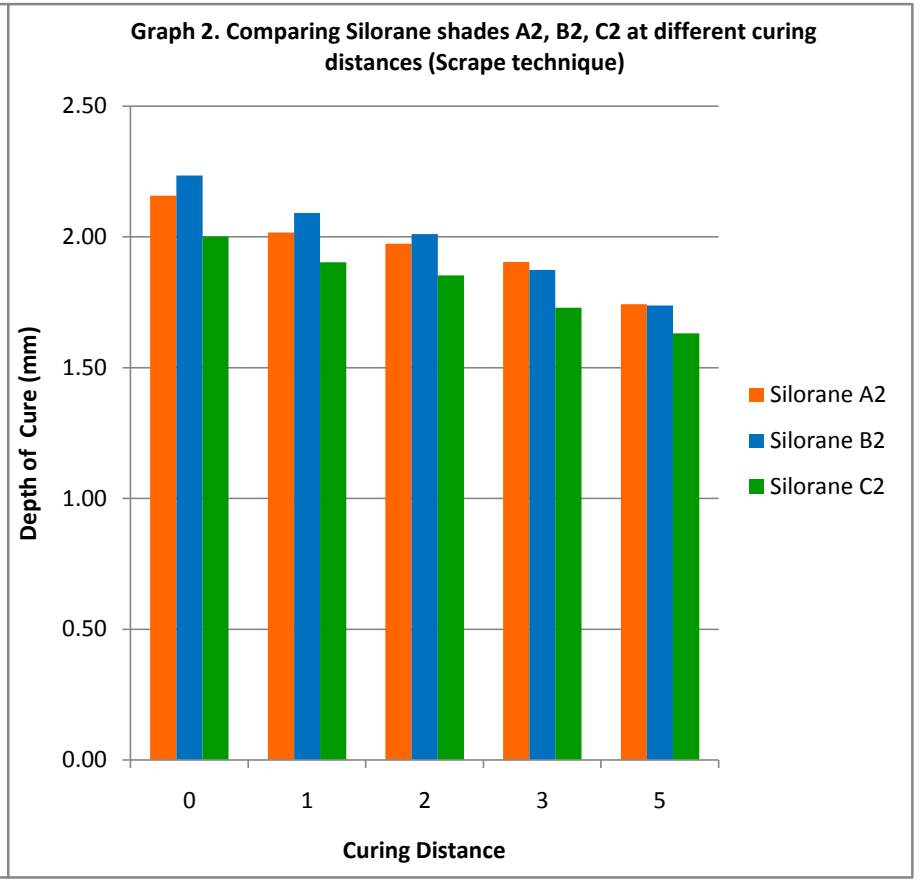
Table 5: Penetrometer technique. Depth of cure (mm) of Filtek Supreme XT composite resin (shades A2, B2 and C2) cured through air (0,1, 2, 3 and 5 mm) using an Elipar Freelight 2 (LED) curing unit (600mW/cm^2, 20 seconds)

Spacer (mm)	0			1			2			3			5		
Shade of Resin	A2B	B2B	C2B	A2B	B2B	C2B	A2B	B2B	C2B	A2B	B2B	C2B	A2B	B2B	C2B
Sample No															
1	2.57	2.58	2.39	2.36	2.43	2.22	2.47	2.36	2.14	2.21	2.16	2.08	2.11	2.05	1.96
2	2.57	2.57	2.39	2.50	2.39	2.25	2.42	2.41	2.12	2.23	2.18	2.07	2.15	2.10	1.96
3	2.59	2.56	2.41	2.44	2.40	2.14	2.39	2.33	2.22	2.23	2.23	2.11	2.15	2.11	1.99
4	2.59	2.54	2.39	2.41	2.35	2.16	2.38	2.32	2.20	2.21	2.18	2.09	2.11	2.02	1.97
5	2.56	2.51	2.41	2.51	2.32	2.25	2.44	2.38	2.21	2.20	2.19	2.05	2.12	2.14	2.03
6	2.60	2.55	2.35	2.37	2.37	2.20	2.39	2.29	2.15	2.25	2.20	2.07	2.12	2.12	2.01
7	2.53	2.53	2.37	2.50	2.42	2.21	2.39	2.33	2.24	2.26	2.25	2.10	2.16	2.15	1.99
8	2.60	2.51	2.31	2.52	2.38	2.09	2.33	2.30	2.19	2.25	2.21	2.10	2.20	2.09	2.01
9	2.50	2.49	2.37	2.39	2.45	2.30	2.40	2.37	2.22	2.25	2.20	2.03	2.19	2.13	1.98
10	2.59	2.52	2.37	2.49	2.45	2.26	2.38	2.33	2.20	2.22	2.22	2.13	2.20	2.05	1.94
Mean Depth of Cure	2.57	2.53	2.37	2.45	2.39	2.21	2.40	2.34	2.19	2.23	2.20	2.08	2.15	2.09	1.98
Maximum Depth	2.60	2.58	2.41	2.52	2.45	2.30	2.47	2.41	2.24	2.26	2.25	2.13	2.20	2.15	2.03
Minimum Depth	2.50	2.49	2.31	2.36	2.32	2.09	2.33	2.29	2.12	2.20	2.16	2.03	2.11	2.02	1.94

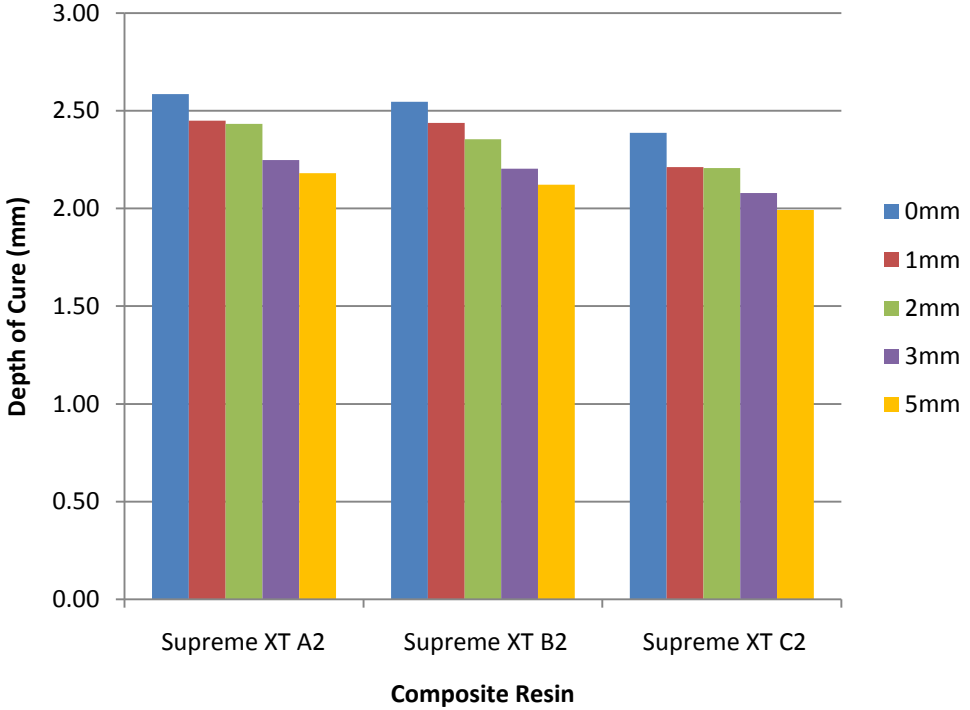
Table 6: Penetrometer technique. Depth of cure (mm) of Z100 composite resin (shades A2, B2 and C2) cured through air (0,1, 2, 3 and 5 mm) using an Elipar Freelight 2 (LED) curing unit (600mW/cm^2, 20 seconds)

Spacer (mm)	0			1			2			3			5		
Shade of Resin	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2	A2	B2	C2
Sample No															
1	3.31	3.33	3.23	3.35	3.40	3.08	3.26	3.35	2.76	3.06	3.16	2.84	2.95	3.00	2.56
2	3.34	3.36	3.20	3.31	3.33	3.08	3.24	3.32	2.93	3.25	3.19	2.81	3.07	2.96	2.60
3	3.39	3.34	3.26	3.33	3.32	3.04	3.26	3.32	2.88	3.27	3.18	2.76	3.06	2.96	2.63
4	3.36	3.23	3.22	3.35	3.27	3.10	3.24	3.30	2.65	3.16	3.24	2.76	3.00	3.02	2.54
5	3.38	3.30	3.18	3.33	3.35	2.98	3.31	3.34	2.82	3.14	3.10	2.82	3.07	2.99	2.53
6	3.35	3.50	3.24	3.34	3.30	3.03	3.31	3.33	2.74	3.20	3.12	2.75	3.09	2.90	2.59
7	3.36	3.52	3.11	3.32	3.34	2.99	3.30	3.32	2.79	3.14	3.12	2.85	3.06	2.93	2.64
8	3.37	3.60	3.19	3.34	3.31	2.99	3.34	3.32	2.79	3.08	3.11	2.85	3.15	2.86	2.61
9	3.37	3.57	3.11	3.32	3.34	2.96	3.32	3.30	2.92	3.13	3.12	2.79	3.16	2.89	2.63
10	3.34	3.54	3.12	3.35	3.35	3.05	3.30	3.34	2.81	3.26	3.22	2.80	3.11	2.97	2.59
Mean Depth of Cure	3.35	3.43	3.18	3.33	3.33	3.03	3.29	3.32	2.81	3.17	3.15	2.80	3.07	2.95	2.59
Maximum Depth	3.39	3.60	3.26	3.35	3.40	3.10	3.34	3.35	2.93	3.27	3.24	2.85	3.16	3.02	2.64
Minimum Depth	3.31	3.23	3.11	3.31	3.27	2.96	3.24	3.30	2.65	3.06	3.10	2.75	2.95	2.86	2.53

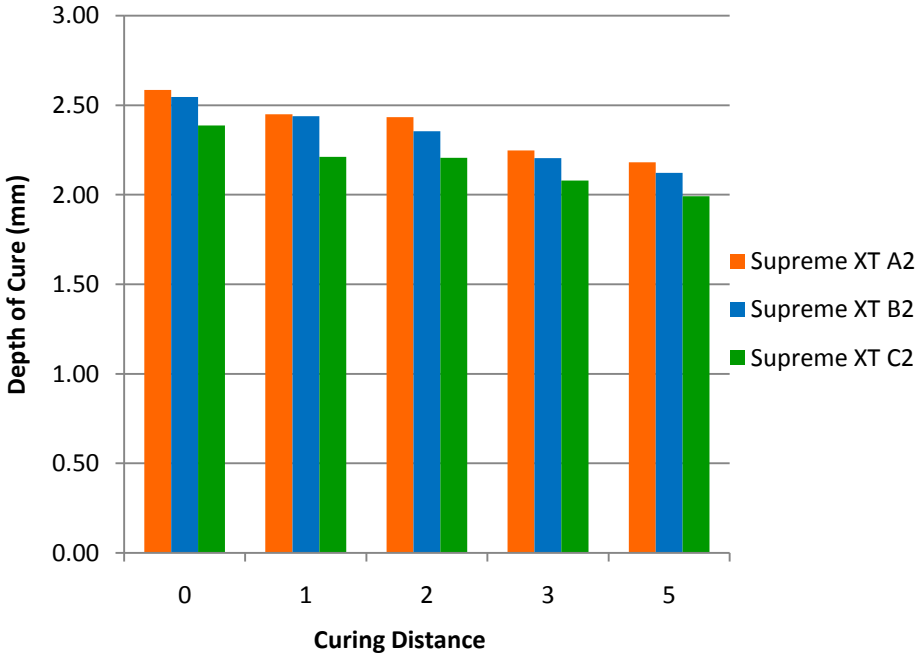

Graph 1. The effect of increasing curing distances on the depth of cure for Silorane shades A2, B2, C2 (Scrape technique)
2.50
2.00
1.50
1.00
0.50
0.00
Depth of Cure (mm)
Silorane A2
Silorane B2
Silorane C2
Composite Resin
0mm
1mm
2mm
3mm
5mm


Graph 2. Comparing Silorane shades A2, B2, C2 at different curing distances (Scrape technique)
2.50
2.00
1.50
1.00
0.50
0.00
Depth of Cure (mm)
0
1
2
3
5
Curing Distance
Silorane A2
Silorane B2
Silorane C2

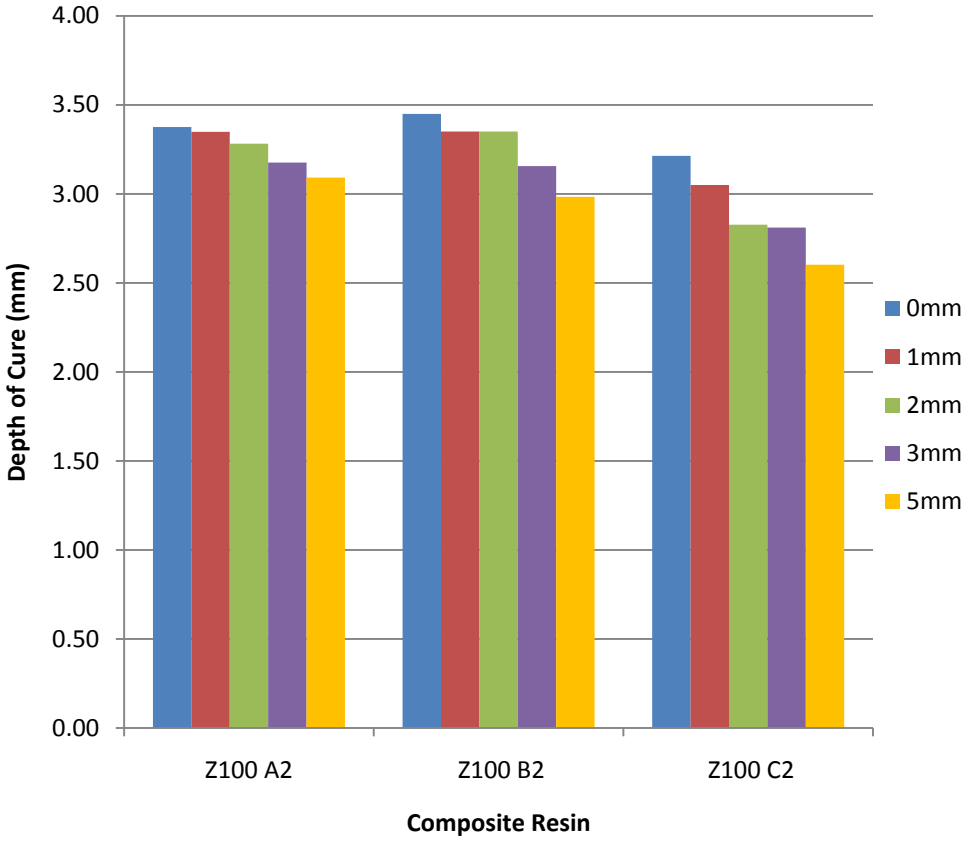
Graph 3. The effect of increasing curing distances on the depth of cure for Supreme XT shades A2, B2, C2 (Scrape technique)



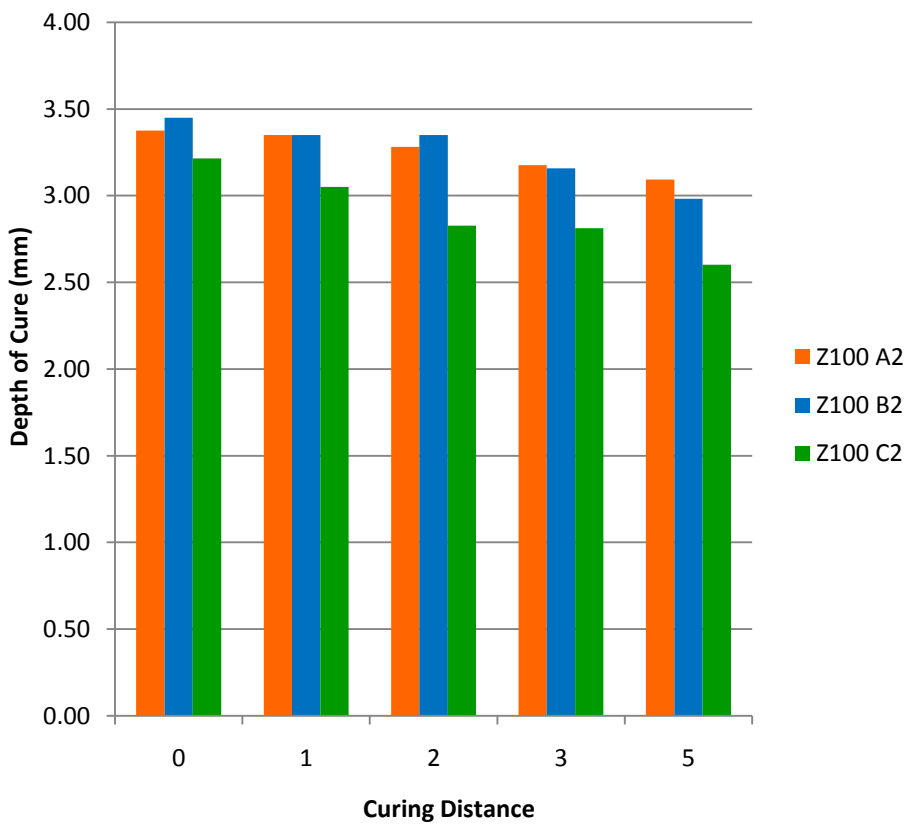
Graph 4. Comparing Supreme XT shades A2, B2, C2 at different curing distances (Scrape technique)



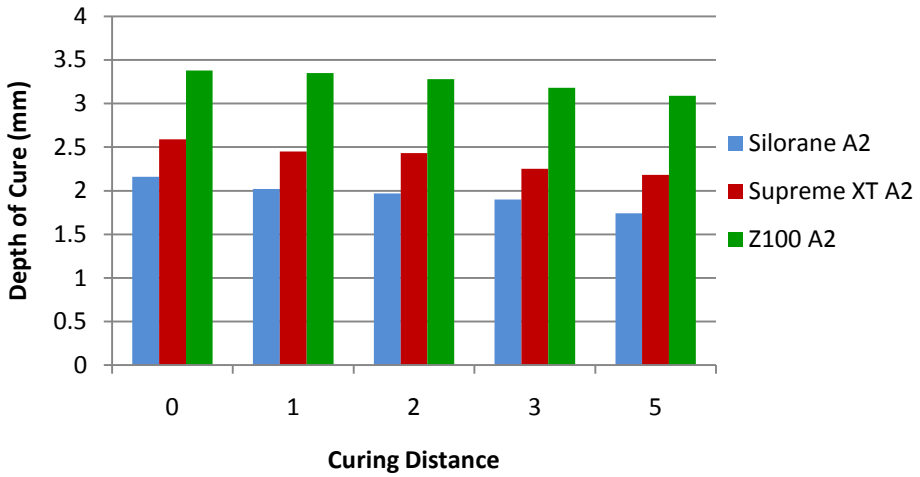
Graph 5. The effect of increasing curing distances on the depth of cure for Z100 shades A2, B2, C2 (Scrape technique)



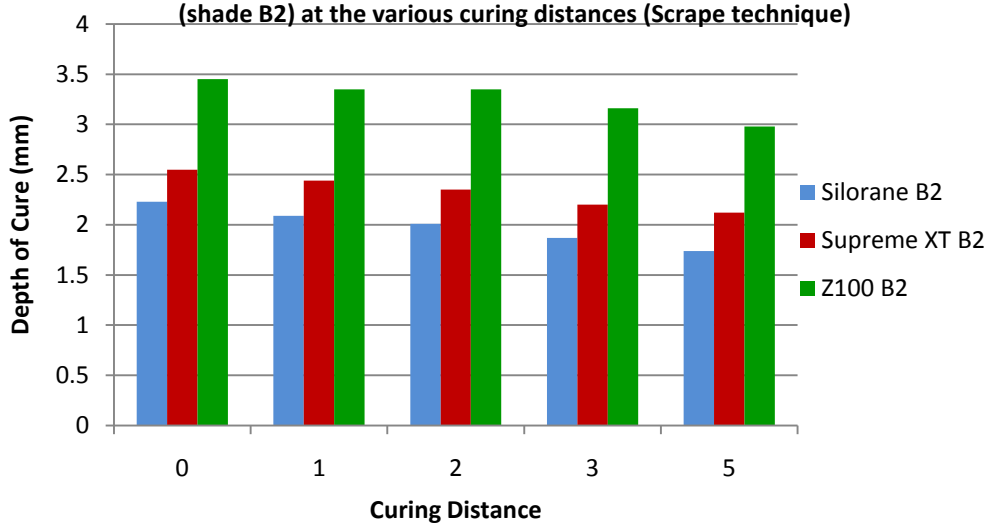
Graph 6. Comparing Z100 shades A2, B2, C2 at different curing distances (Scrape technique)



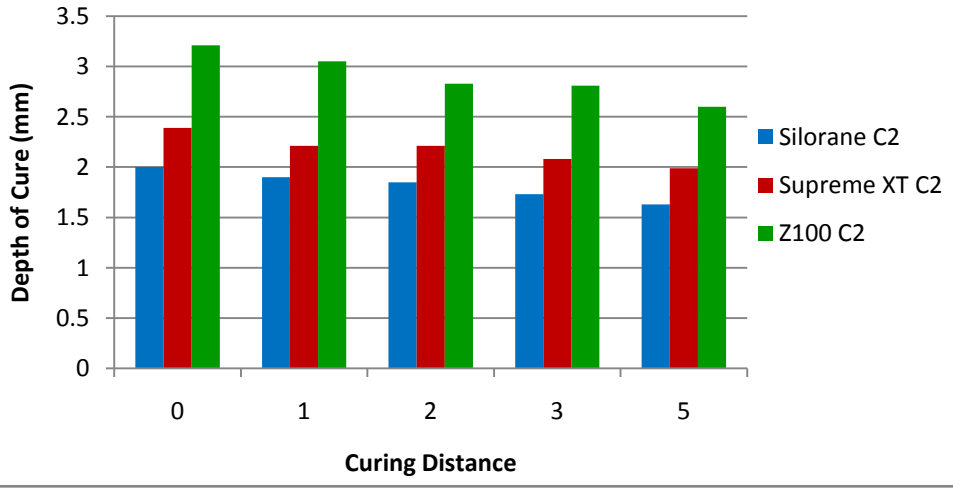
Graph 7. Comparing depth of cure of Silorane, Supreme XT and Z100 (shade A2) at the various curing distances (Scrape technique)



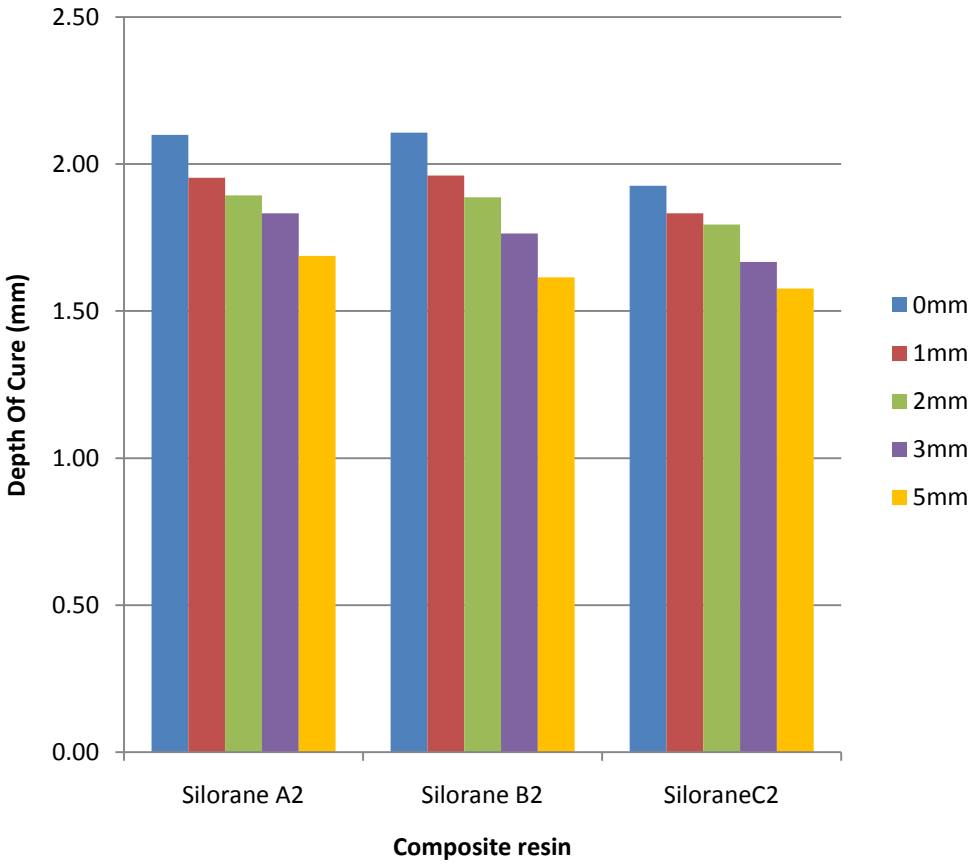
Graph 8. Comparing depth of cure of Silorane, Supreme XT and Z100 (shade B2) at the various curing distances (Scrape technique)



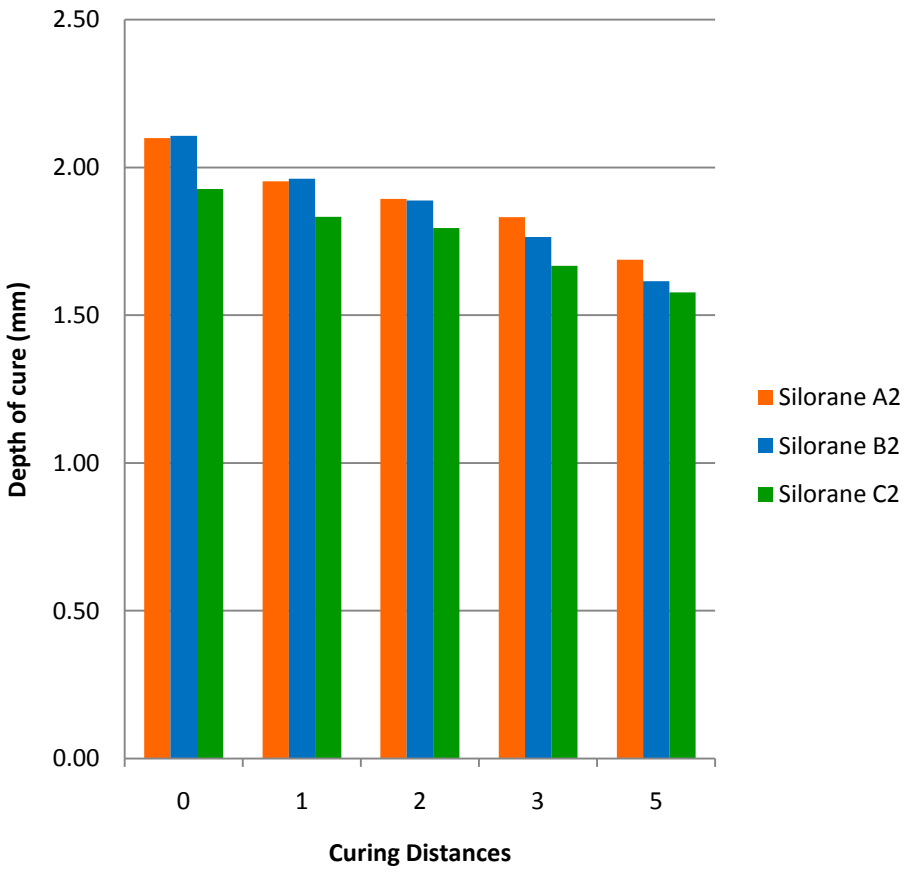
Graph 9. Comparing depth of cure of Silorane, Supreme XT and Z100 (shade B2) at the various curing distances (Scrape technique)



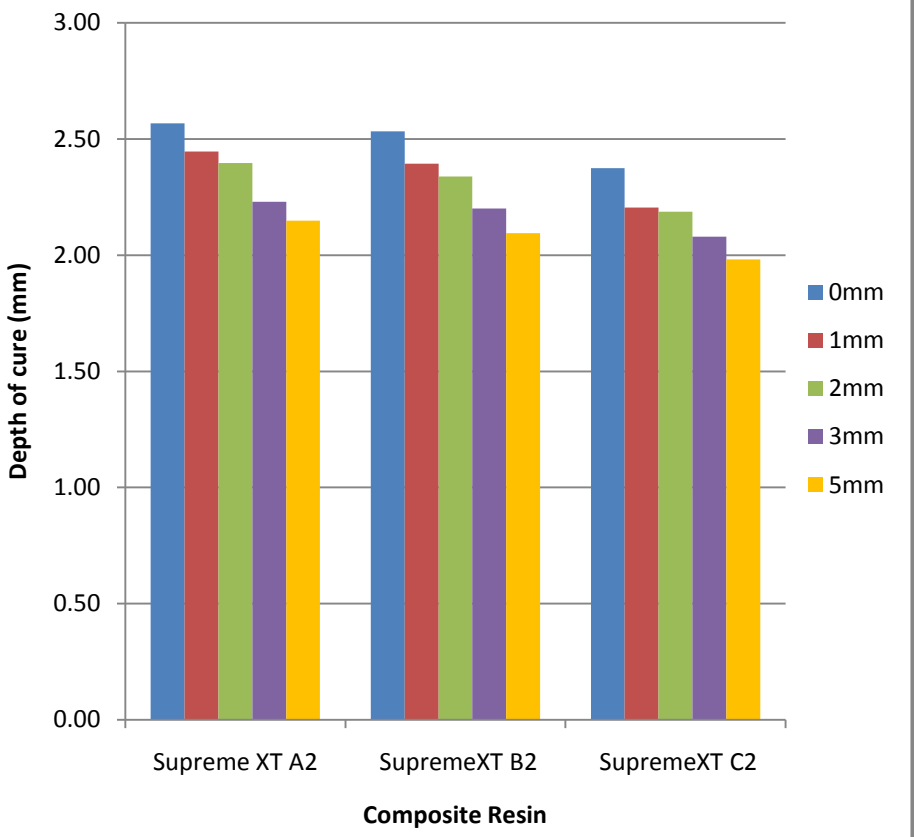
Graph 10. The effect of increasing curing distances on the depth of cure for Silorane shades A2, B2, C2 (Penetrometer)



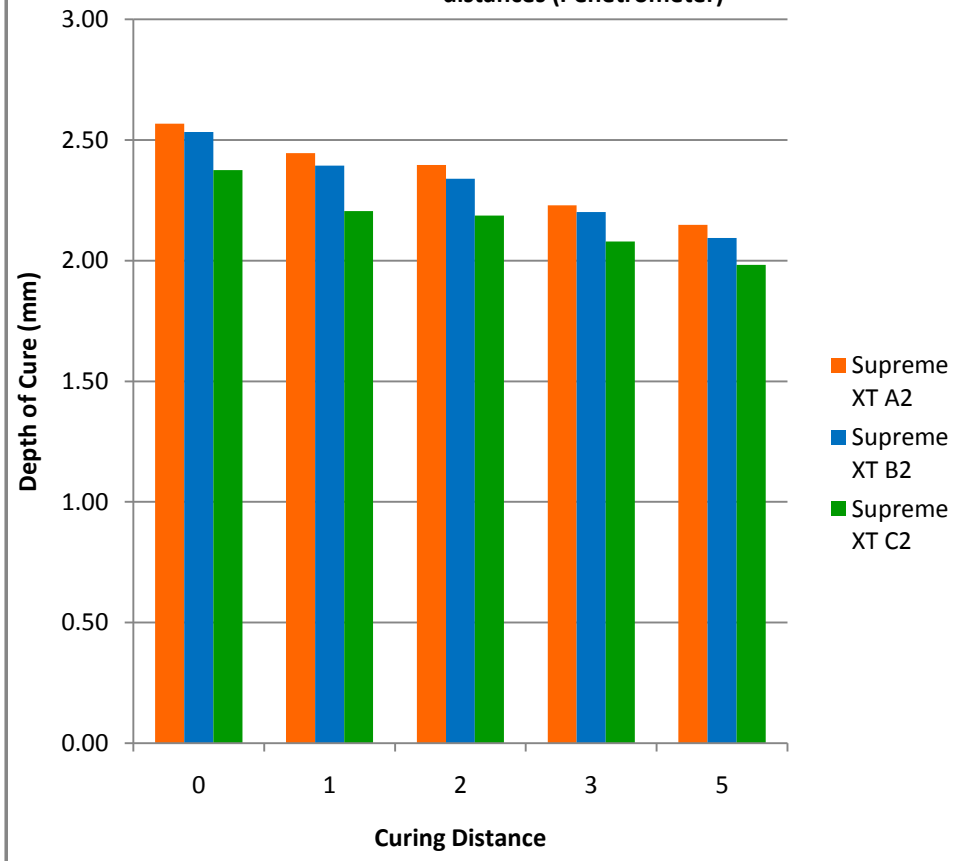
Graph 11. Comparing Silorane shades A2, B2, C2 at different curing distances (Penetrometer)



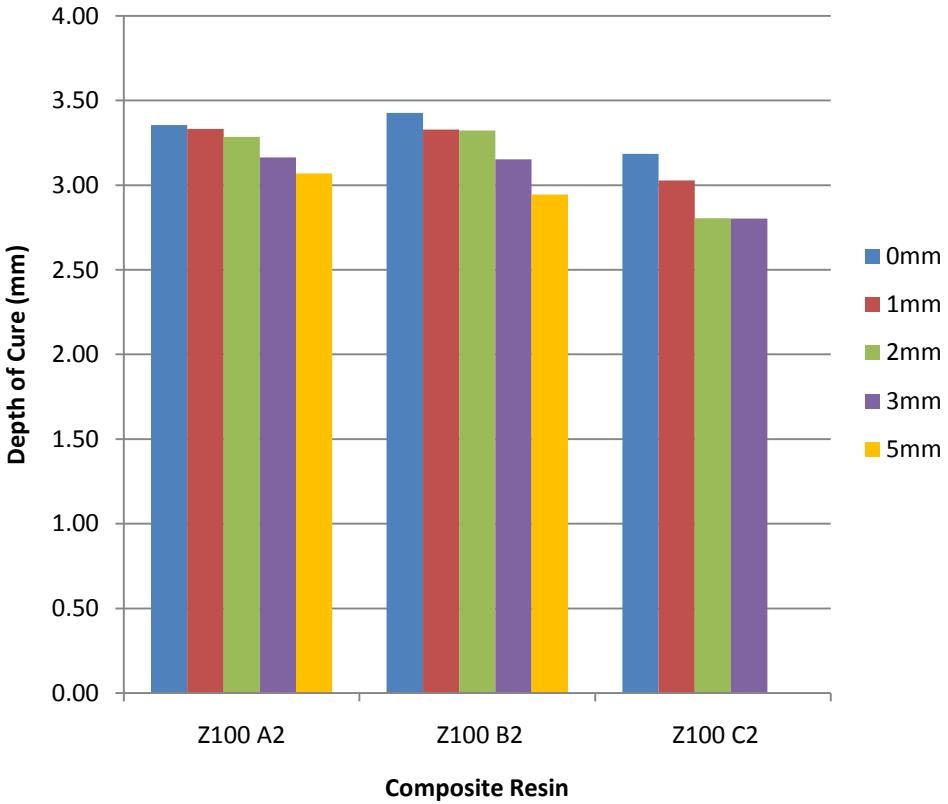
Graph 12. The effect of increasing curing distances on the depth of cure for Supreme XT shades A2, B2, C2 (Penetrometer)



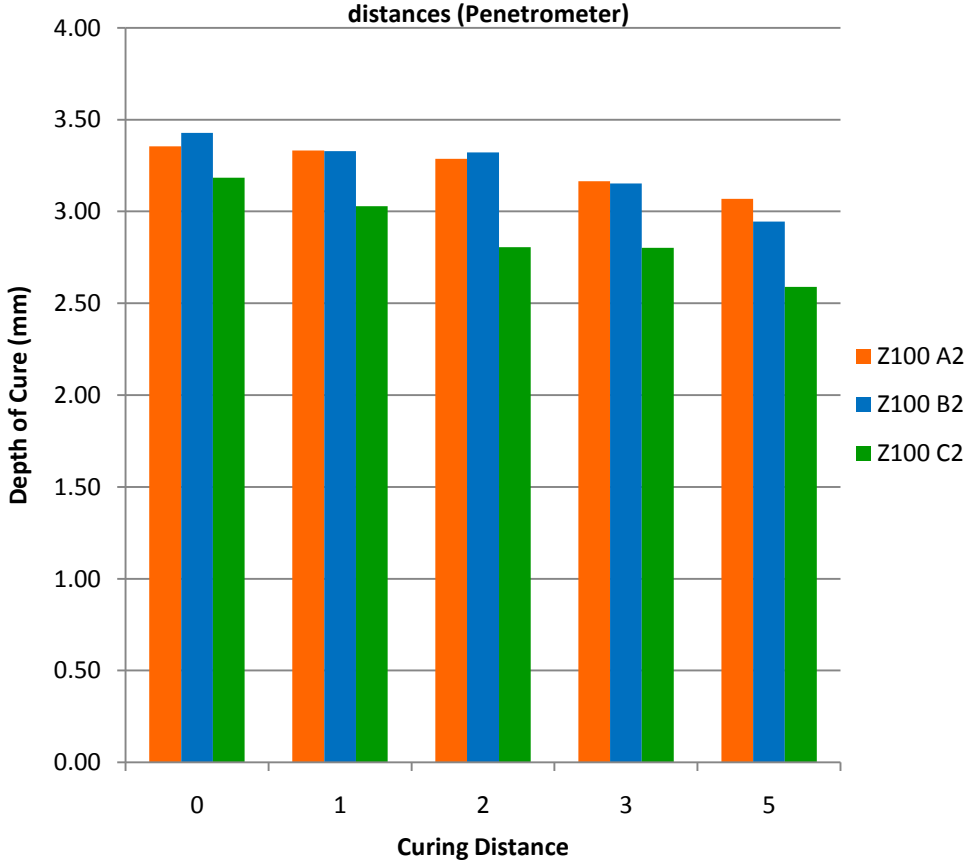
Graph 13. Comparing Supreme XT shades A2, B2, C2 at different curing distances (Penetrometer)



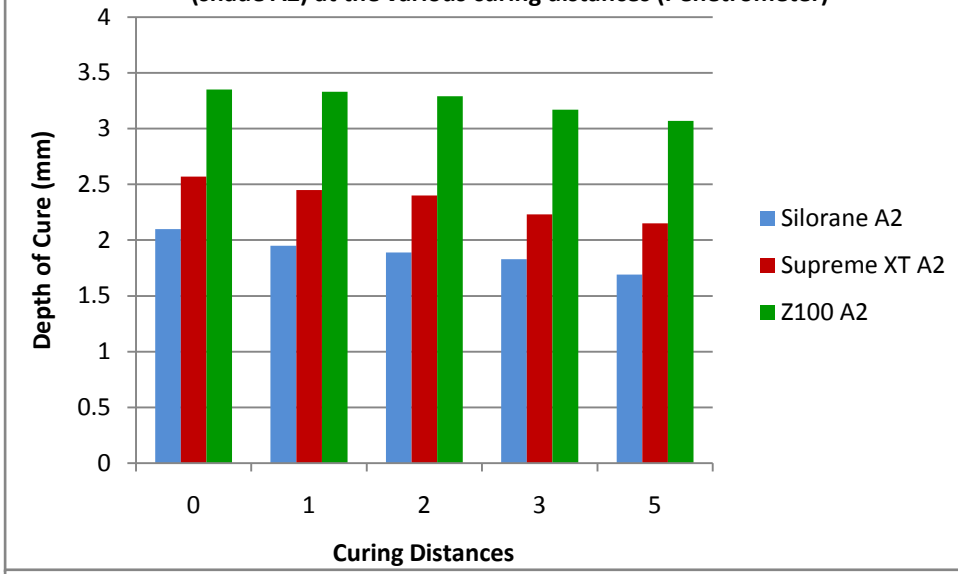
Graph 14. The effect of increasing curing distances on the depth of cure for Z100 shades A2, B2, C2 (Penetrometer)



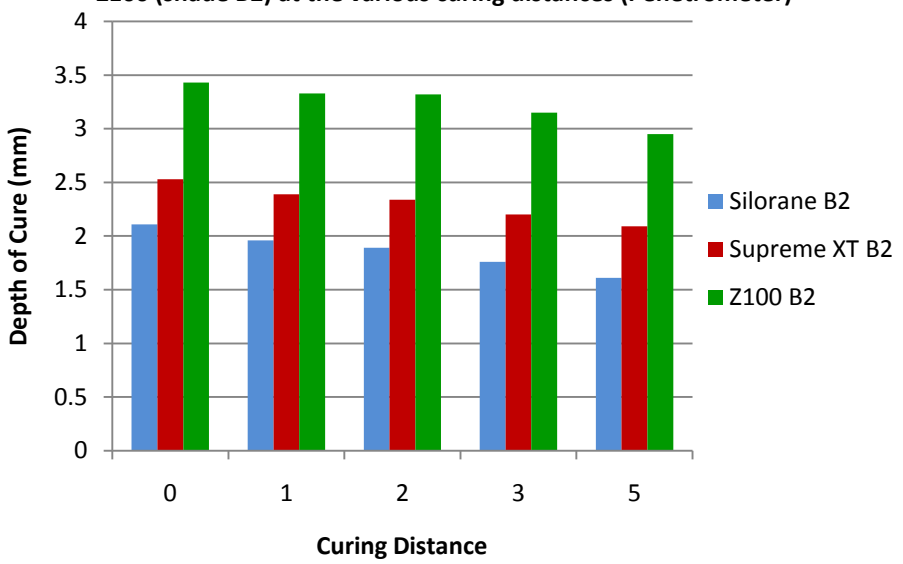
Graph 15. Comparing Z100 shades A2, B2, C2 at different curing distances (Penetrometer)



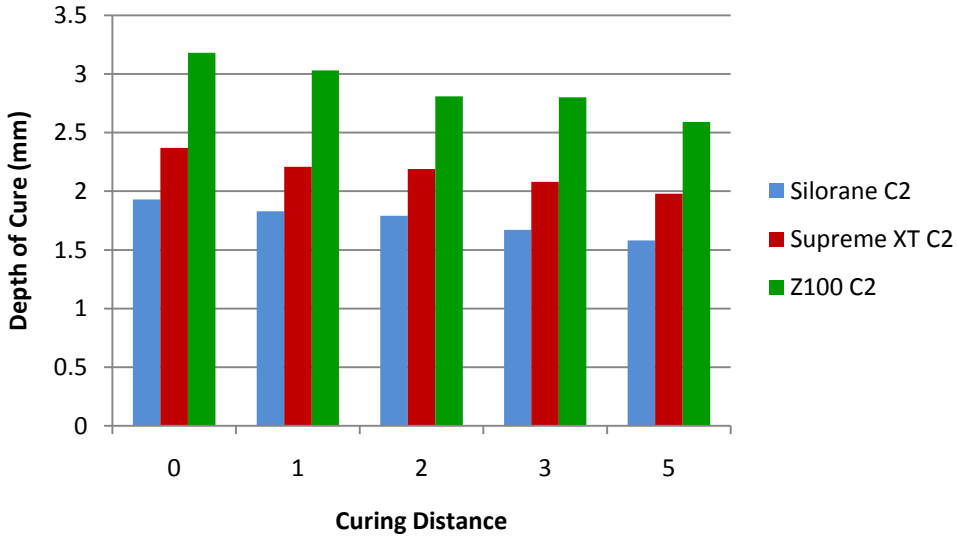
Graph 16. Comparing depth of cure of Silorane, Supreme XT and Z100 (shade A2) at the various curing distances (Penetrometer)

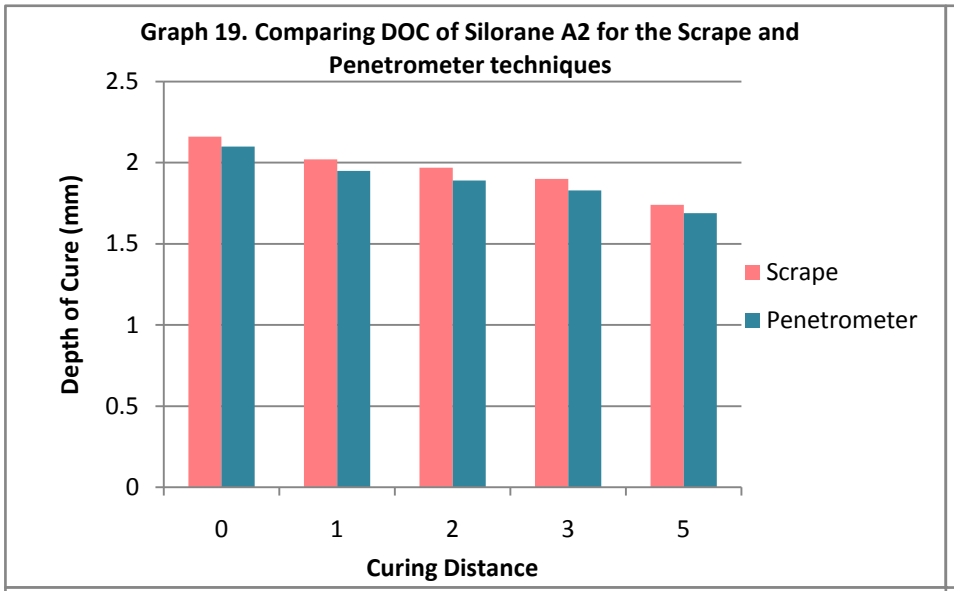


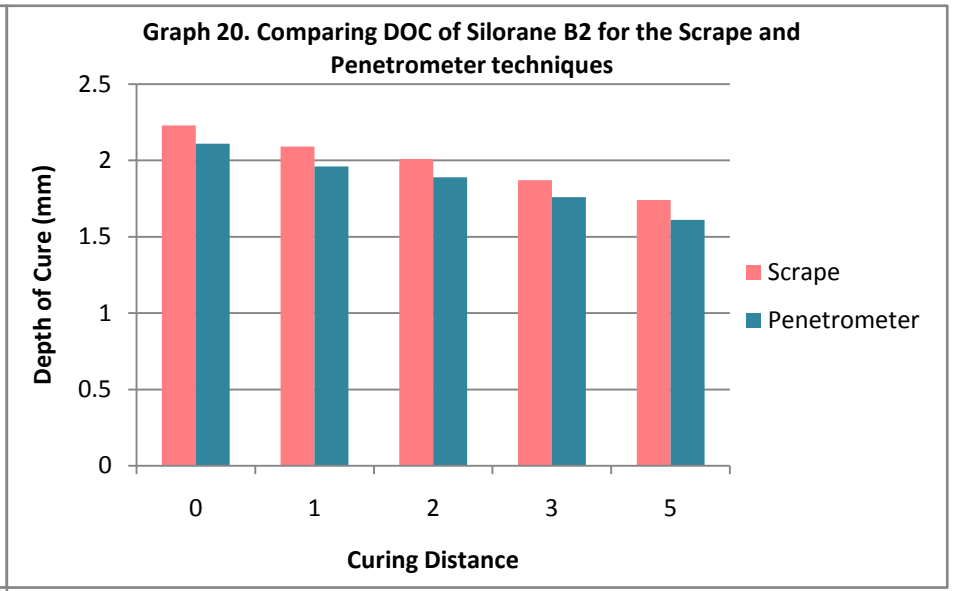
Graph 17. Comparing depth of cure of Silorane, Supreme XT and Z100 (shade B2) at the various curing distances (Penetrometer)

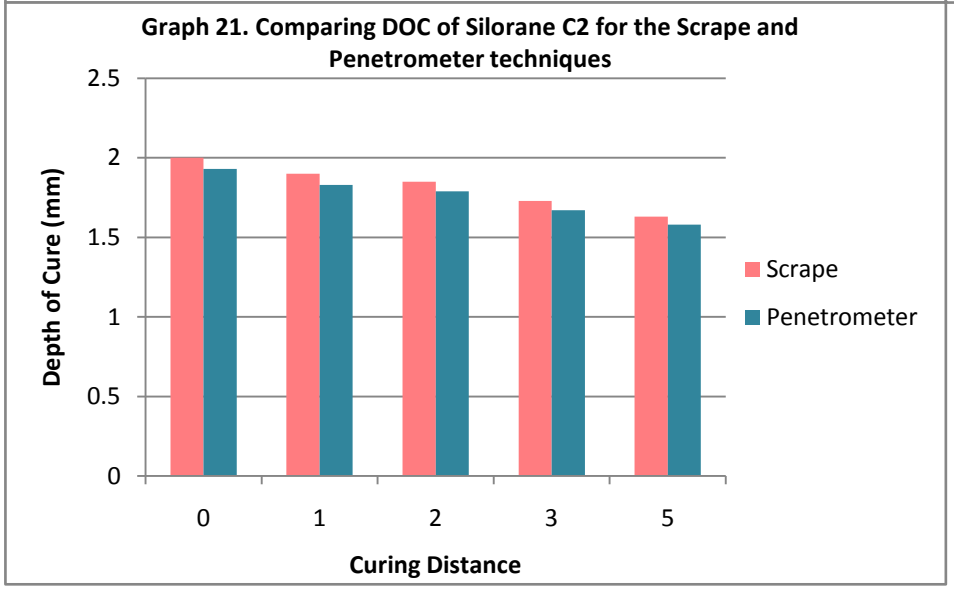


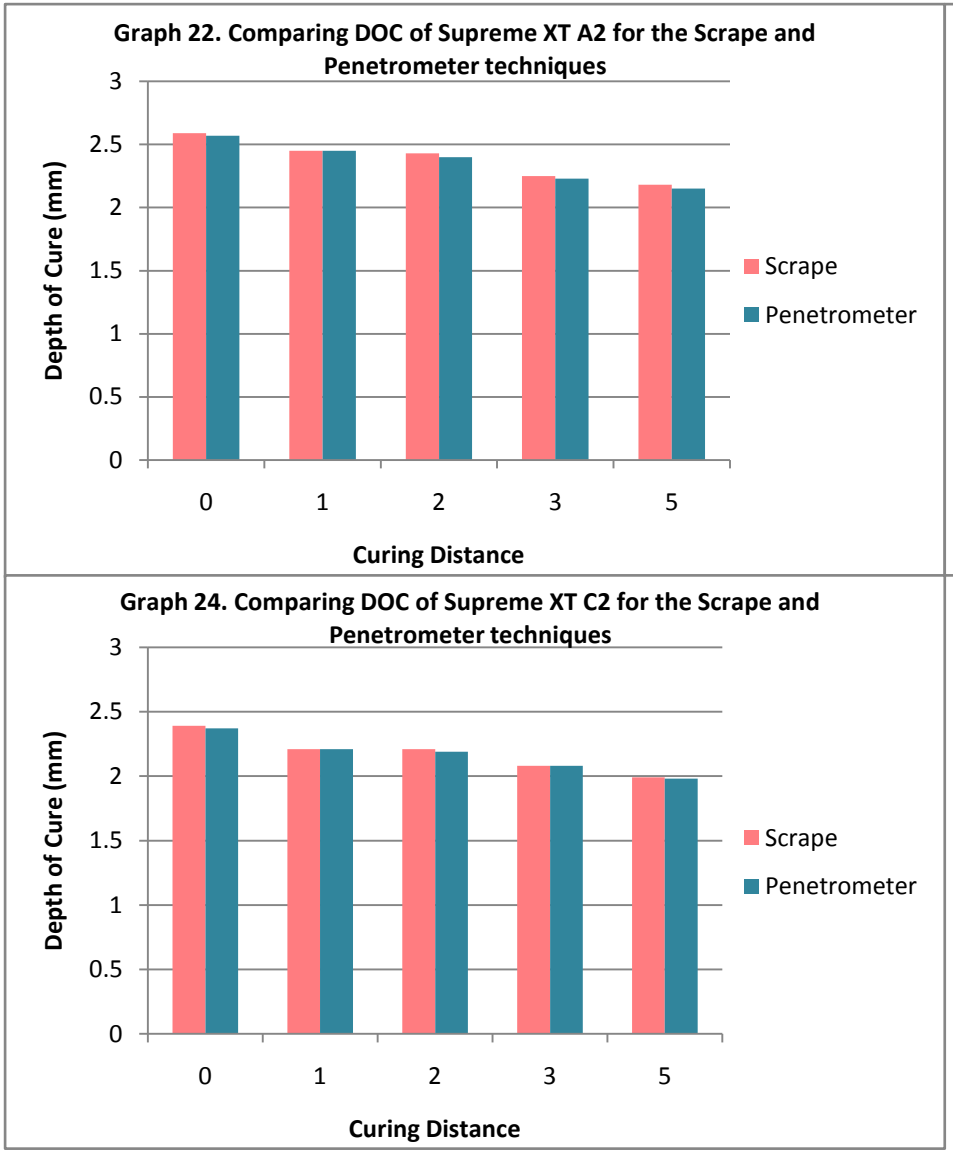
Graph 18. Comparing depth of cure of Silorane, Supreme XT and Z100 (shade C2) at the various curing distances (Penetrometer)

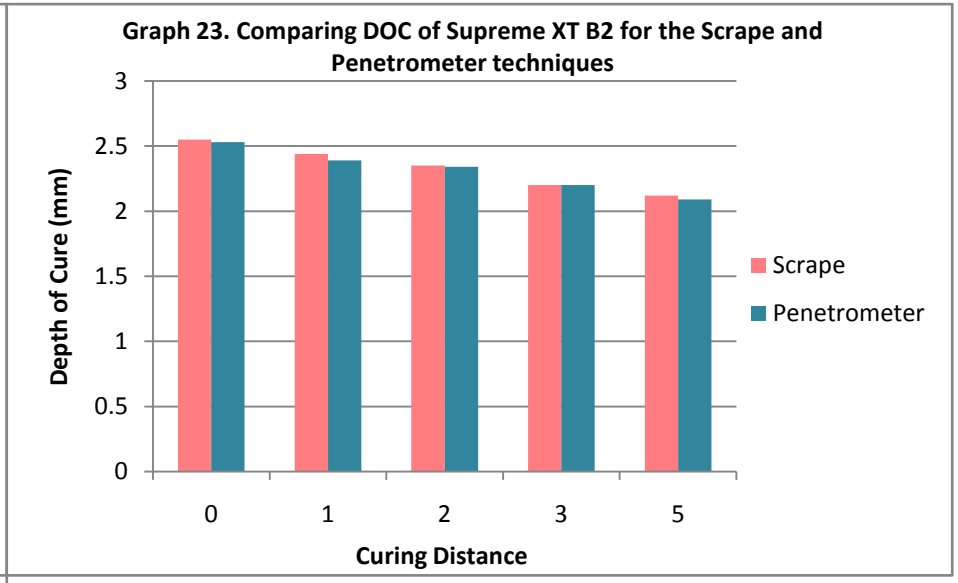



Graph 19. Comparing DOC of Silorane A2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer

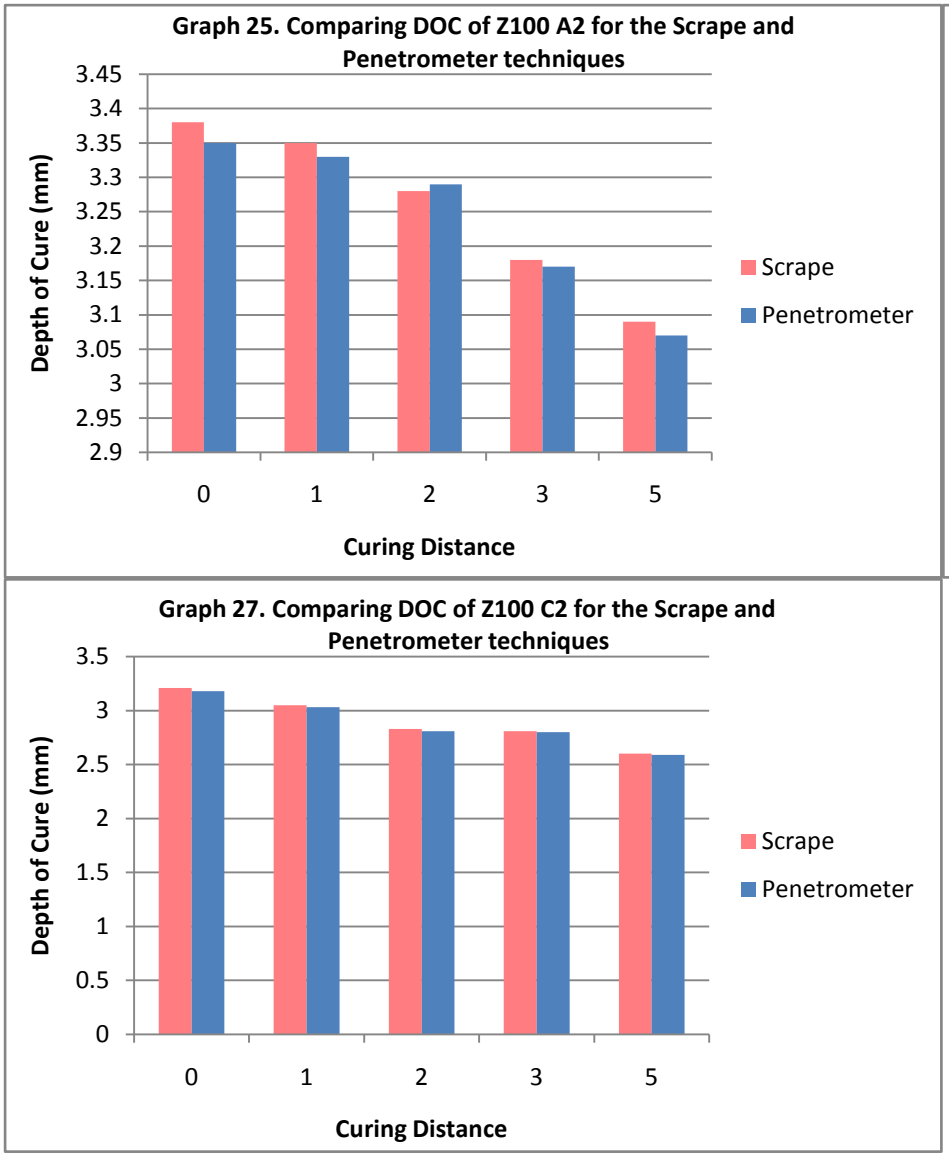

Graph 20. Comparing DOC of Silorane B2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer

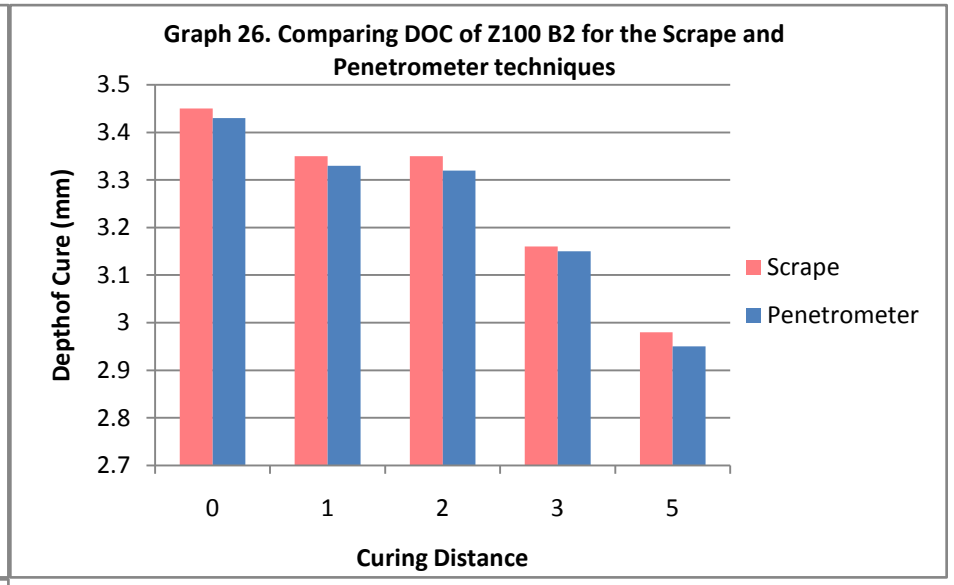

Graph 21. Comparing DOC of Silorane C2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer


Graph 22. Comparing DOC of Supreme XT A2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
3
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer


Graph 23. Comparing DOC of Supreme XT B2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
3
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer

Graph 24. Comparing DOC of Supreme XT C2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
3
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer


Graph 25. Comparing DOC of Z100 A2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
3.45
3.4
3.35
3.3
3.25
3.2
3.15
3.1
3.05
3
2.95
2.9
0
1
2
3
5
Curing Distance
Scrape
Penetrometer


Graph 26. Comparing DOC of Z100 B2 for the Scrape and Penetrometer techniques
Depthof Cure (mm)
3.5
3.4
3.3
3.2
3.1
3
2.9
2.8
2.7
0
1
2
3
5
Curing Distance
Scrape
Penetrometer

Graph 27. Comparing DOC of Z100 C2 for the Scrape and Penetrometer techniques
Depth of Cure (mm)
3.5
3
2.5
2
1.5
1
0.5
0
0
1
2
3
5
Curing Distance
Scrape
Penetrometer

CHAPTER 5

DISCUSSION

Filtek Silorane is a low shrinkage composite composed of siloxane and oxirane developed by 3M (ESPE). Siloxanes have been utilised in the industrial sector and it is well known for its hydrophobic properties and thus it has been incorporated into Silorane for this reason. Oxirane has been used in the automotive, aviation and in the production of sports equipment because of its strong physical properties. They are also known for their low shrinkage and endurance to physical and chemophysical forces. The inorganic filler is a combination of fine quartz particles and radiopaque yttrium fluoride. To ensure a proper interface between the resin and filler particles, the quartz surface was coated with a silane layer. Filtek Silorane was thus classified as a microhybrid composite due to its filler particles. Therefore the combination of siloxane and oxirane results in a biocompatible, low shrinkage and hydrophobic composite resin with a resin matrix that is very different from methacrylate-based composite resins on the market.

The other two composite resins utilised in this study are Filtek Supreme XT and Z100, both also developed by 3M (ESPE). They are methacrylate-based composites. Filtek Supreme XT is a nanocomposite and it has a BIS-GMA, BIS-EMA, UDMA and TEGDMA based resin system. Its filler composition is a combination of 20nm nanosilica filler which is non-agglomerated and loosely bound agglomerated zirconia/silica nanocluster. These agglomerated clusters consist of primary zirconia/silica which has particle sizes of 5-20 nm fillers. This nanotechnology provides Supreme XT with a high polishability comparable to a microfilled composite and the physical strength of a hybrid composite.

Z100 a hybrid composite consists of a BIS-GMA and TEGDMA resin matrix with synthetic zirconia/silica particles which compose the filler particles of the inorganic matrix. These filler particles have a broad particle size distribution ranging from 4

microns to 0.2-0.04 microns. These fine particles provide good handling and aesthetic properties to this material.

As defined by DeWald and Ferracane (1987) (as cited in Sobrinho *et al,* 2000), Koupis *et al* (2006), Krämer *et al* (2008) and Schattenberg *et al* (2008) the depth of cure of a visible light activated composite is a function of multiple factors. These factors include the filler composition, resin building blocks; the shade and translucency; the light intensity of the curing device and the curing time.

For this study shades A2, B2 and C2 were selected. These are all radiopaque shades so the composite opacity would not be a variable in this study and only the shade would be one of the chosen variables. The composite shade is an important factor influencing the depth of cure as shown in many studies (Schattenberg *et al*, 2008; de Araujo *et al*, 2008). To determine the effect that the composite shade would have on the depth of cure for Silorane, Supreme XT and Z100, shades A2, B2 and C2 were included in this study.

The other variable in this study is the curing distance, because the aim of the project was to study the effect that the curing distance would have on the depth of cure. Hence the curing distances of 0, 1, 2, 3 and 5mm were selected for the study. These variables were tested for each of the three composite resins that were utilised in this study.

Wiggins *et al* (2004) compared Elipar Freelight 2 (HP LED) to a 1st generation LED, conventional and high intensity halogen curing devices in their study. They compared the DOC of the composite specimen which was 6mm high and utilised the scrape technique as defined by ISO 4049:2000. They showed that Elipar Freelight 2 produced comparable and sometimes higher DOC readings when the composite was cured for 10 seconds whereas the other curing devices were used for 20 seconds. Price *et al* (2005) found differences amoung various LED curing devices and also in LED curing devices of the same model with regards to the light emission of these curing devices.

The Elipar Freelight 2 by 3M (ESPE) was chosen due to the increasing use and popularity of LED curing devices in dentistry.

The curing time recommended by the manufacturer 3M (ESPE) for Silorane was 20 seconds if an LED such as Elipar Freelight 2 was utilised (3M ESPE Elipar Freelight 2 product profile). For Supreme XT a 20 second curing time was recommended for body shades and for Z100 a 20 second curing time was recommended by 3M (ESPE) Elipar Freelight 2 technical product profile. Based on this, a curing time of 20 seconds was selected as this was the minimum curing time recommended. A 20 second curing time was also chosen as time is an important factor in dentistry today and many dentists would employ a 20 second curing time to save time and shorten the length of the procedure. Based on this the study aimed to determine what the effect of a 20 second curing time would have on the depth of cure of the composite materials in this study. As stated by Koupis *et al* (2006) and Olivier *et al* (2012), a composite which is not sufficiently cured will possess sub-optimal chemical and physical properties which will affect the wear resistance, strength and water absorption of the composite. In addition an under-cured composite will leak allergic and/or cytotoxic components into its surroundings.

The resin composition and the curing device are important factors that affect the polymerization of composite resins. Together with the resin composition and shade, other factors such as wavelength and light intensity are important for optimal polymerization. The total energy produced by the curing device as well as the curing time affect the mechanical properties of the composite (Aravamudhan *et al*, 2006). The composite cures most effectively when the wavelength produced by the curing device is between 450-500 nm. The absorption co-efficient of camphorquinone is between 460-480 nm, therefore the maximum light intensity produced by the curing device should be in this range. Therefore a high light intensity is not the only factor to consider achieving optimal curing of a composite; the correct wavelength must also be considered (van Noort, 2002). Many curing devices emit a wavelength in the range of 400-515nm. The Elipar Freelight 2 utilised in this study has a maximum emission of photons in the wavelength of approximately 465 nm which lies in the absorption peak

of camphorquinone which is the photo-initiator found in all three composites tested in this study.

There are many factors that may cause variation in the light intensity. These factors are ageing of the bulb, fluctuations in the line voltage, filter degradation and breakdown of the electrical components of the curing device. The light intensity of the curing device has a considerable effect on the DOC of composites. Due to light scattering within the composite during polymerization, the light intensity is a significant factor especially when the curing distance from the composite surface is increased. Shortall, Harrington and Wilson (1995) showed that radiometers should only be used as a method to assess and monitor the curing devices periodically to determine the need for repair and to measure the effectiveness of the device. They however should not be used to compare different curing devices. Elipar Freelight 2 (3M ESPE) is a high intensity LED curing device utilised in this study to cure all three composites for 20 seconds. According to the manufacturer (3M ESPE) the intensity of the Elipar Freelight 2 cannot be measured utilising a radiometer as it will not produce an accurate reading. The manufacturer recommends utilising the testing area on the charger base to determine the intensity with the curing device positioned in the charger base. The number of illuminated mini LED lights on the charger base will indicate the intensity. There are five of these lights on the charger base and the number of illuminated lights indicates the measured light intensity. When five lights are illuminated this indicates that the intensity of the curing device is 100%, subsequently if only four lights are illuminated then the intensity is now 80%. The lowest intensity is measured when only one light is illuminated to indicate 20% intensity (3M ESPE Elipar Freelight 2 product profile).

As the angle at which a composite is cured, diverges from a 90° angle, the energy of the light is deflected away from the composite. This results in a reduced light penetration as can be seen in molar Class II cavities where light penetration is blocked by the marginal ridge of the adjacent tooth. This affects the polymerization of the composite as well as the depth of cure (Albers, 2000). In this study, the light tip was positioned at a 90° angle to the surface of the composite specimen with the aid of the light guide alignment ring. This would ensure that the light would not be

deflected from the composite surface and that the light would reach the entire composite surface with equal intensity.

In order to measure the depth of cure the scrape technique recommended by the ISO Standard 4049:2009, was employed. The main advantage of this technique is that it is easy to perform and it does not require advanced equipment. It can be performed in any dental surgery in a short time period to determine the depth of cure of a composite resin.

The composite specimens were prepared utilising the curing alignment device and its individual components. Sketch 1 illustrates the assembling of the curing alignment device. To cure the specimens at distances of 1, 2, 3 and 5mm, the respective spacers were utilised. Whereas, for the distance of 0mm, no spacer was utilised and the curing tip of the curing light was placed directly in contact with the composite specimen covered by a Hawe strip.

To determine the effect of the various curing distance (0, 1, 2, 3 and 5mm) and the different composite shades (A2, B2 and C2) would have on the depth of cure at 600mW/cm^2 for 20 seconds, a total of 450 specimens (10 specimens per test variable) were prepared.

The composite specimens for the three different composites, Filtek Silorane, Filtek Supreme XT and Z100, were prepared by placing the composite within the central cavity of the mould. The composite was covered by the Hawe strips on both ends of the mould.

The mould was placed in the alignment tube and light cured from the top with the Elipar Freelight 2 (Fig. 15) for 20 seconds at distances of 0, 1, 2, 3 and 5mm. The

curing intensity of the curing light was measured after every ten specimens cured, using a Demetron radiometer.

As a comparison for the scrape technique, the revised version of the penetrometer developed by Harrington and Wilson (1995) was included in this study. The same specimens could be used for both the penetrometer and scrape technique. The revised penetrometer is similar to the one designed by Harrington and Wilson except that the needle attachment was replaced by an adjustable clamp and the sliding mechanism by a free moving ball bearing system. The indicator gauge provides a direct reading which is accurate to 0.01mm. This eliminates any form of variation such as operator pressure exerted to remove the uncured portion of the resin as is the case with the scrape technique. However, the penetrometer does not measure the quality of conversion of the cured composite.

The results of this study showed that when Silorane shades A2, B2 and C2 were cured at the various curing distances chosen in this study; it was found that A2 and B2 both had a significantly higher DOC than C2 when the scrape technique was utilised ($p<0.05$) (Table 1, Graph 2). At 0mm A2 had a DOC of 2.16mm whereas B2 had a DOC of 2.23mm and this trend continued for a curing distance of 1mm and 2mm. However at 3mm and 5mm curing distances, A2 and B2 were statistically similar ($p>0.05$). For Z100 shades A2 and B2 both had a significantly higher DOC than C2 when the scrape technique was utilised ($p<0.05$) (Table 3, Graph 6). At 0, 1 and 2mm, shade B2 had a significantly higher depth of cure than A2 (Table 3, Graph 6). An explanation for shade B2 having a higher DOC than A2 for both Silorane and Z100 at curing distances of 0, 1 and 2mm could not be provided in this study. For Supreme XT it was found that the DOC of A2 and B2 were higher than C2 at each of the curing distances (Table 2, Graph 4). For all three composite materials that were tested, the DOC was lower for the darker shade C2. However with shade A2 and B2 the differences were only significant at a curing distance of 5mm and not at lower curing distances.

Aguiar *et al* (2005) showed in their research conducted that composite shade is a significant factor that affects polymerization. Shade A1 had higher Knoop hardness number (KHN) values (top and bottom surfaces) than A3.5 and the lowest KHN values were obtained for C2. These composites were also cured at 2, 4 and 8mm curing distances. A2 again provided the highest KHN values when compared to A3.5 and C2, with the KHN values decreasing as the curing distance was increased for each of the shades tested. Koupis *et al* (2006) also found that Z100 provided a higher depth of cure than did the other visible light cured materials that they tested. They also found that Z100 provided a KHN value of 82.4 for shade A2 and a KHN value of 80.6 for shade A4. Jandt *et al* (2000) showed similar results, they found a higher DOC for shade A2 (2.67mm) when compared to A4 (2.19mm) when cured with a LED curing device ($p<0.05$). Thus, concluding that lighter composite shades provide a higher depth of cure than darker shades. The composite shade may influence the transmission co-efficient and this then influences the depth of cure of composites of different shades (Aguiar *et al*, 2005). The opacity in dark shades decreases the light transmission by hindering the path of light to penetrate to the bulk of the composite. Also different composites which have the same Vita shade may present with different colour values (Aguiar *et al*, 2005). This difference may also contribute to the varying results recorded in this study. As in the case of deep Class I and Class II cavities, Aguiar *et al* (2005) suggest using a lighter shade in the deepest parts of the cavity to ensure adequate polymerization. Thereafter the darker composite shade can be utilised for the final composite layer when the darker shade was selected to match the tooth colour.

As shown by Ferracane (1985) (as cited in Sobrinho *et al*, 2000), the depth of cure of a composite is directly dependent on the size of the filler particles that constitute the composite resin. If the size of the filler particles approaches the wavelength of the curing device, scattering of the curing light within the composite is increased. This results in less light being transmitted through the composite. This explains as to why large particle composites have higher depths of cure than small particle composites as the larger particle composites are less affected by light scattering (Sobrinho *et al*, 2000). This can explain the differences in the DOC of Silorane, Supreme XT and Z100 which were observed in this study because the average particle size of Silorane is smaller than that of Supreme XT followed by Z100. Z100 provided the highest DOC,

followed by Supreme XT and lastly Silorane. This pattern was also found for each of the shades tested (A2, B2 and C2) at 0, 1, 2, 3 and 5mm curing distances.

Methacrylate-based composites comprise of Bis-GMA, Bis-EMA or UDMA forming the resin matrix. Silorane's resin matrix is a combination of siloxane and oxirane. David *et al* (2007) showed that the presence of UDMA (found in Supreme XT) increases the composites reactivity to light in comparison to Bis-GMA. Bis-EMA is a longer molecule that forms a more flexible resin matrix than Bis-GMA which forms a more rigid resin matrix. Supreme XT has both UDMA and Bis-EMA whereas Z100 has a Bis-GMA resin matrix. This difference in the resin matrix systems may explain why Z100 and Supreme XT provided a higher DOC than Silorane which does not have any of these constituents in its resin matrix. However a similarity in all the 3M ESPE composites is that they all comprise of camphorquinone, tertiary amine and iodonium salt which constitute the initiator system.

The findings of Koupis *et al* (2006) show that Z100, a hybrid composite, produced the highest depth of cure and they correlated this to the fact that the co-efficient of light transmission is higher in hybrid composites than microfilled composites. This is due to the light scattering within the material which is dependent on the filler particle size as well as the composition and quantity of the filler particles. They also explained that the lower depth of cure in darker composite shades is due to the increased amount or type of pigment in darker composite shades, as the remainder of the components such as the particle size, filler particle type and quantity all remain constant for a particular brand of composite.

However, Jain and Pershing (2003) found that microhybrid composite resins produced the highest depth of cure, followed by condensable composites then hybrid composites and lastly flowable resin-based composites which produced the lowest depth of cure under the same curing parameters. They also attributed these differences in depth of cure to the size of the filler particles, where smaller filler particles cure to a lower depth than larger filler particles due to increased light scattering. The ratio of filler particles to

unfilled resin is another factor that Jain and Pershing (2003) noted for the differences in depth of cure. They found that the higher the filler load, the more difficult it is for the passage of light through the composite, thus hindering polymerization. Z100 has a filler loading of 66% by volume (3M ESPE Filtek Silorane product profile), Supreme XT has a filler loading of 59.5% by volume (3M ESPE Filtek Supreme XT product profile) and Silorane has a filler loading of 55% by volume (Kang *et al*, 2012; Lien and Vandewalle, 2010). This may explain why Supreme XT and Z100 have a higher depth of cure than Silorane and this was shown by Albers (2000) that the more heavily filled composites and composites with a larger particle size have a greater depth of cure.

In this study a pattern was found with all three composite materials that were tested. It was found that the curing distance negatively affects the polymerization of a composite. The DOC of Silorane, Supreme XT and Z100 decreased as the curing distance increased for each of the shades that were tested. For example, the DOC of Silorane shade A2 (Table 1, Graph 1) decreased from 2.16mm to 2.02m (1mm curing distance), then to 1.97mm (2mm curing distance), to 1.90mm (3mm curing distance) and to 1.74mm (5mm curing distance). As shown by Aguiar *et al* (2005) composite polymerization is dependent on the curing distance and the light intensity also decreases significantly as the curing distance increases. They showed that 1mm of air will decrease the light intensity of the curing device by 10%. However Krämer *et al* (2008) found that only curing distances of 6mm and greater produced considerable effects on the depth of cure and at a 12mm curing distance there was no significant curing of the composite irrespective of the type of curing device and the curing mode. Similar results were obtained by Aguiar *et al* (2005) where a curing distance of 8mm showed a significant decrease in composite hardness and therefore also reflecting on the DOC.

Schattenberg *et al*, 2008 and Krämer *et al*, 2008 found that LED curing devices sufficiently cured a 2mm composite increment when cured for 20 seconds. They cured three 2mm increments from a distance of 7mm and they concluded that the incremental layering and curing of each layer produced a post curing effect on the previous layers of the specimen. This post curing may positively influence

polymerization without having to increase the light intensity of the curing device. They also found that different composites require different curing times and this is dependent on the composite-curing device combination. As suggested by Aguiar *et al* (2005) that instead of increasing the curing time, the composite thickness should be reduced.

Clinically insufficient polymerization occurs in deep Class I and Class II cavities as a result of light scattering in air and thus energy dispersion due to the increased distance between the curing device tip and the first composite layer. This factor cannot be controlled as it is dependent on the extent of the carious defect, the size of the cavity and the location of the cavity (Aguiar *et al*, 2005). In a study conducted by Sobrinho *et al* (2000), they found that when the curing distance was increased it resulted in a decreased depth of cure for both Z100 and Silux Plus. They tested curing distances of 0, 6 and 12mm and they found that Z100 provided the higher depth of cure than Silux Plus at each curing distance they tested using the Knoop hardness test. Z100 had KHN values of 78.19 at 0mm, 71.12 at 6mm and 59.28 at 12mm, whereas Silux Plus had KHN values of 35.29 at 0mm, 36.83 at 6mm and 32.54 at 12mm. The KHN values of Silux Plus are not statistically significant for each increase in the curing distance. In another study conducted by Aravamudhan *et al* (2006) they found a decrease in depth of cure when the curing distance was increased from 0mm to 10mm when utilising the scrape technique. They also found that the light intensity of each of the curing lights that they tested, decreased as the curing distance was increased, however this rate of decline in intensity varies between curing devices. Freelight 2 showed a 80% loss of intensity when the curing distance was increased to 10mm whereas Smartlite only lost 64% intensity at 10mm.

In conjunction with the scrape technique the DOC of the three composites included in this study were also determined using the penetrometer technique in order to determine whether the two techniques would produce the same results. What was found is that the scrape and the penetrometer provided significantly similar results for each of the composites that were tested to compare the effect of the shade as well as the curing distance on the DOC. Similar results were found by Koupis *et al* (2004), where they found that the scrape technique and the penetrometer gave comparable

results for the resin-based materials that were tested. They cured their specimens at an intensity of 800mW/cm^2 for 40 seconds. Wiggins *et al* (2004) utilised the scrape technique as defined by ISO 4049:2000 to determine the DOC of their composite specimens. Jandt *et al* (2000) chose the revised version of the penetrometer to determine the DOC rather than the scrape technique as they felt that the penetrometer uses a reproducible force determined by the weight used rather than the subjective force applied during the scrape technique which is variable and operator dependent.

Although all the shades of Z100 and Supreme XT have a higher DOC than Silorane at each curing distance, there remains a place for Silorane in dentistry. According to Zimmerli et al (2010), Silorane has low polymerization shrinkage (<1%) which leads to a lower polymerization stress compared to methacrylate-based composites which have volumetric shrinkage in the range of 2-3%. Lower polymerization stress reduces the risk of post-operative sensitivity and cusp deflection. Also its colour stability was seven times longer than methacrylate-based composites and it displays low water absorption and water solubility. Thus Silorane overcame some of the challenges experienced with methacrylate-based composites in dentistry today (Zimmerli et al, 2010; Weinmann *et al,* 2005).

CHAPTER 6

CONCLUSIONS

This research study was conducted to determine the effects of curing distance and shade on the depth of cure on the silorane-based composite, Filtek Silorane and to compare it to the methacrylate-based composites, Filtek Supreme XT and Z100. The following conclusions were derived from this study:

1. The greater the curing distance, the lower the DOC for all three composites tested.

2. Shade A2 and B2 provided a DOC at each curing distance that were not significantly different. The darker shade C2 was significantly lower than A2 and B2 at each curing distance tested.

3. The DOC of the three composites were not significantly different at curing distances of 0, 1, 2 and 3mm, but at 5mm the DOC was significantly lower than the DOC at the shorter distances.

4. The darker the composite shade, the lower the DOC for all three composites tested.

5. The scrape and penetrometer techniques provided similar results in measuring the DOC for all the specimens tested.

6. Z100 provided the highest DOC followed by Filtek Supreme XT and lastly Filtek Silorane.

CHAPTER 7

RECOMMENDATIONS

From the results of this study, the following recommendations are provided:

1. To achieve an optimal DOC, the curing distance should be minimal and as close as possible to the composite surface.

2. The darkness of the shade should be taken into account when curing composite resins. Utilise a lighter composite shade in the deeper areas of the cavity in deep Class I and Class II cavities and thereafter utilise the darker shade as the final composite layer.

3. In posterior teeth (non-aesthetic zone) where the composite shade is not of aesthetic importance, rather use light composite shades to ensure an optimal DOC.

4. The scrape as well as the penetrometer techniques can be utilised to determine the DOC for composites.

5. The scrape technique requires less sophisticated equipment and can be used to measure the DOC easily in a dental surgery. Thus the scrape technique is an ideal tool to determine the DOC of composites.

6. For a curing time of 20 seconds the curing distance for Z100 and Filtek Supreme XT (all three shades) may be increased up to 5mm, however, the curing distance for Silorane should not be more than 1mm to ensure proper curing of a 1.5mm thick layer (ISO 4049:2009).

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