THE PHOTOCURABILITY OF A NEW BULK-FILL COMPOSITE RESIN: X-TRAFIL

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A THESIS

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LIST OF ABBREVIATIONS

KHN- Knoop hardness number

D4- 4mm thickness

S- sample

LED- light emitting diode

T – top surface hardness measurement

B – bottom surface hardness measurement

Max - maximum

LCU - light curing unit

mm - milimeters

DC – depth of cure

DC4- depth of cure at 4mm

DoC- degree of conversion

FTIR- Fourier transformer infrared spectrometer

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ABSTRACT

Objective: To evaluate photocurability of a 4mm sample of a bulk-fill composite resin, X-traFil, using the Knoop hardness test after curing 10 seconds at a distance of 0 mm.

Materials and Methods: X-traFil, Voco composite material was examined. Ten specimens were prepared in a delrin mold 8mm diameter × 4mm long and polymerized at one end with the Mini light emitting diode (LED) light curing system (Aceton North America). The 4mm thick samples were subjected to three top and three bottom surface microhardness measurements utilizing the Knoop hardness tester (Leco, LM 300 AT, St Joseph, MI) under a load of 100 grams for 15 seconds. For each sample a bottom to top Knoop hardness surface ratio was determined and a value of at least 80% was used to indicate the acceptable depth of cure.

Results: The bottom to top surface Knoop microhardness ratio mean was 79.40 ± 1.5%. The bottom to top surface KHN hardness ratio reflecting the relative degree of cure showed an acceptable hardness (cure) at a depth of 4mm at 10 seconds for X-traFil**.**

Conclusion: Within the limitations of the study, it is acceptable that the new bulk-fill resin X-traFil achieved a 4mm curing depth.

INTRODUCTION

For the past twenty years, the use of composite resin restorations has been on the rise and in many geographical markets they have replaced amalgam restorations. [1]. Composite restorations are preferred by most patients due to their esthetic appeal [2]. However, composite resin restorations are more technique sensitive and difficult to manipulate, requiring more time to place compared to amalgam restorations costing provider time and money [3]. Therefore, dental product manufacturers have attempted to improve the characteristics and qualities of composites to make them more ideal esthetic restorative materials possessing strong fundamental restorative characteristics.

Curing time and depth of cure are important factors in light-cured composite resins. Placement and curing of resins in small increments, approximately 2mm or less, is advised in order to assure complete polymerization [4]. Most frequently, the curing reaction in composite restorative materials involves visible-light-initiated photopolymerization of dimethylacrylate monomers to form a highly cross-linked polymerizable resin, filler and the filler-resin interface. It has been found that inadequate polymerization reduces the physical properties of the resin due to the decreased filler–resin interface [5]. The filler has several roles, including enhancing the modulus of elasticity, radiopacity and coefficient of thermal expansion.

With polymerization comes polymerization-induced shrinkage, ranging from 2.44 – 6.79 %, the conversion of the monomer into a polymer leads to a bulk contraction. The reduction of volume is usually denoted as polymerization shrinkage [6,7]. Polymerization shrinkage is considered the most significant problem with composite

restorative materials and is the primary contributor to stress at the interface between the composite and cavity wall and weaken that bond leading to adhesion failure or microleakage. This resulting gap may vary from 1.67 to 5.68 % of the total volume of the restoration and may be filled with oral fluids containing toxins of bacteria, which can lead to postoperative sensitivity and recurrent caries [1, 8, 9, 17].

Polymerization shrinkage stress is influenced by the restorative technique, the modulus of resin elasticity, polymerization rate and the ratio of bonded to unbonded surfaces known as the “C-factor” or configuration factor [17]. Placing composite resin in smaller increments and curing each increment independently can reduce the net effect of polymerization shrinkage by relaxing polymerization induced stress which is why most manufacturers recommend small 2mm increments of composite at a time [10, 13]. Therefore, polymerization shrinkage is a very important factor when selecting restorative materials in clinics.

Some researchers have theorized alternative light curing methods may reduce shrinkage stress such as using soft-start and pulse-delay methods that slow down the polymerization reaction by emitting various rates of curing light intensities causing less stress at the restoration-cavity wall interface [8]. However, this problem has not yet been solved.

In addition to polymerization, the degree of conversion (the concentration of unreacted carbon double bonds in the resin when it is cured) of visible light-activated composite resins is vital to the success of these materials [5]. A high degree of conversion (DoC) is important in obtaining good mechanical properties and biocompatibility. This inherent property is directly correlated to the total irradiance

reaching the material, dependent on the light curing unit (LCU) and the distance between the curing tip and the composite resin [1].

Various curing lights belonging to different generations are available commercially for clinical use: Quartz tungsten halogen lamps (400 nm to 800 nm); Light-emitting diode units (450 nm to 490 nm); Plasma-arch lamps (370 nm to 500nm); and Argon-ion lasers (454 nm to 514 nm) each with varying ranges of “functions” [8, 11].

This study used an LED which is a much more efficient way of converting an electric current into light than compared to other curing light units and emits a specific narrow wave length within 400 nm to 500nm.

The light intensity and exposure of time also influence the depth of cure. The wavelength of light, the irradiance and the scatter of light within the restoration dictate the depth of light penetration through a composite restoration [10].

Generally, the tip of the light source is held within 1-2mm of the surface of the composite with a standard exposure time of 20 seconds and a resin depth of approximately 2mm [14]. A longer exposure time of the composite resin to the light source will increase the degree of conversion. The physical properties of a composite can be hampered if the material is not converted or polymerized thoroughly.

Several other factors can influence the depth of cure of a resin material including type of photo-initiator, type of methacrylate monomers, shade of resin, etc. The most common photoinitiator in dental materials is camphoquinone which has a peak activity around 470 nm. Incoming photons generated by the curing light are absorbed by a photoinitiator which, when activated, enables the formation of free radicals and thus trigger the polymerization reaction. Therefore, knowledge of the absorbance spectrum

of the material’s photoinitiator chemistry is critical to ensure the proper LCU is used which will function in the same wavelength for effective polymerization of the resin.

Methacrylate based monomers constitute the light cure component of composite resin material. They include 2, 2-bis [4-(2-hydroxy-3-methacrylyloxy-propoxy) phenyl] propane (Bis-GMA), urethane dimethacrylate (UDMA), and triethylene glycol dimethacrylate (TEGMA).

The incorporation of pigments to alter the shade of the resin reduces the ability of light to penetrate the material [12]. Therefore, an increased curing time of 40 seconds is recommended for darker composite resin shades or more opaque materials [5, 11, 12].

Once the resin is cured, the degree of conversion of the external surfaces of a light-cured composite resin can be assessed quite easily; however, it is the degree of conversion of the internal surfaces of the resin that cannot be assumed or easily evaluated [5]. Therefore, hardness of the external surface of the composite is not an indicator of the internal extent of polymerization [13].

Hardness can be evaluated indirectly or directly. Depth of cure is often assessed indirectly by measuring the hardness of a composite resin material at specified depths. Higher hardness values correlate with a more extensive polymerization [15]. The International Standards Organization (ISO) Standard No. 4049 criteria for the evaluation of hardness may tend to overestimate the degree of polymerization [5]. This ISO is a scraping technique which is an indirect method of assessing the depth of cure and is considered the standard for measurement of depth of cure.

According to this ISO, the resin composite to be tested is filled in a tube-shaped mold, light cured, pushed out of the mold and the uncured resin composite material is then removed ("scraped away") with a spatula leaving a hard cylindrical specimen. The absolute length of this hard specimen is measured and divided by two. The resulting value is recorded as the depth of cure and defines the incremental thickness. The rationale for the division is that not all the hardened specimen is actually cured. Depth of cure can also be defined as 50 percent of the remaining length of the composite after the uncured portion has been scraped off [21]. Several studies have found that the scraping method can result in exaggerated depths of cure values compared to those values attained through the hardness test [15]. Ferracane, et al found that although the scraping technique is relatively easy to perform, often resulting in an overestimate of adequate depth of polymerization [4].

The Knoop Hardness (KHN) or microhardness test is another indirect method in which the depth of cure is calculated. This test method is suitable for thin plastics and designed so that varying loads maybe applied to the indenting instrument. The resulting indentation area, therefore, varies according to the load applied and nature of the tested material. The advantage of this method is that materials with a great hardness can be tested simply by varying the test load [13]. The Knoop hardness test was utilized in the study to measure the top and bottom surfaces for hardness, calculating a ratio of bottom surface hardness to top surface hardness. Typically, a value of .80 and .85 has been used as this arbitrary minimum value. Therefore, a composite's bottom surface should be at least 80 percent as hard as the maximum hardness for that material [5, 16].

The depth of cure can also be evaluated by direct methods, such as infrared spectroscopy. The Fourier transform infrared (FTIR) spectrometer measures the extent of polymerization in photocurable polymers based upon the degree of carbon double bond conversion to carbon single bond, which is used as an indicator for the extent of reaction. The percentage of unreacted carbon - carbon double bonds (% C=C) is determined from the absorption intensities of carbon - carbon double bond. These values are represented as graph values by the computer, which is connected to the FTIR machine. The degree of conversion is determined by subtracting the percentage of carbon carbon double bond from 100%. In general, increasing degree of polymerization correlates with higher melting temperature and higher mechanical strength.

SIGNIFICANCE

Since the 1980's dentists have been in search of a tooth-colored amalgam replacement. Ideally, it would be a color-stable composite restoration that could be easily placed using a bulk-fill technique with a short curing time. The restoration would have minimal polymerization shrinkage with no microleakage or fracture concerns. This has not yet occurred due to the properties that exist in today's composites leading to polymerization shrinkage stress during the curing process and a limited depth of cure for composite materials. The effects of shrinkage is greater on larger increments of composites, and if the curing light cannot adequately reach deeper surfaces of the restoration, the uncured portion of material will affect the bonding of the material to tooth

structure and therefore affect the quality and longevity of the restoration. The recommended placement of composites in smaller increments assures adequate polymerization and limited shrinkage stress [4, 8, 18].

Since the development of composite resins several improvements in their chemical composition have occurred, leading to a large category of materials [27]. Recently, a new category of resin composites-called bulk fill – was introduced (X-traFil, Voco, GmbH, Cuxhaven, Germany) as bulk fill material. The particularity of the new material is stated to be the option to place it in 4mm thick bulks instead of the current incremental placement technique, without negatively affecting polymerization shrinkage or degree of conversion. Moreover, the manufacturer states that the polymerization shrinkage of the material is even lower when compared to conventional composite resin [19,29] Thus, problems related to polymerization shrinkage like gap formation causing secondary caries due to bacteria colonization, pulp irritation, post-operative, or cusp deflection when the "C" factor is high could be minimized. The idea of placing a material as bulk, saving time as well as improving material handling, is of great interest.

Now, manufacturers of "bulk-fill" materials claim an incremental curing depth of 4mm and lower polymerization shrinkage from the addition of the "polymerization modulator", a chemical moiety embedded in the center of the polymerization resin backbone of the X-traFil monomer, to lower polymerization shrinkage. The modulator has a high molecular weight. Due to conformational flexibility around the centered modulator impart, the modulator is supposed to increase flexibility and relax the polymerized network structure of the composite resin without harming degree of conversion [19,28], allowing the material to expand rather than contract. According to

Voco, the German manufacture, in terms of flexure strength, water uptake and biocompatibility, X-traFil performed analogously to conventional resin composite. Additionally, it has a low shrinkage of 1.7%, greater wear resistance because it is 87% filled by weight and has a curing depth in layers of up to 4mm in 10 seconds when using light curing units (LCU) rated at 800 mW/cm^2 or higher without losing stability [19].

PURPOSE

The purpose of this in vitro study is to evaluate the photocurability of a bulk-fill composite resin, X-traFil using the Knoop hardness test.

SPECIFIC AIM

This study will test the photocurability of X-traFil in 4mm increments at 10 seconds with a light intensity of 1250 mW/cm^2.

MATERIALS AND METHODS

Material:

X-traFil (Voco, GmbH, Cuxhaven, Germany; Universal shade) contains a resin mixture of 2,2-bis[4-(2-hydroxy-3-methacrylyloxy-propoxy)phenyl]propane(Bis-GMA) the most common molecule in modern resin composite material with high cross linking

and lower shrinkage; urethane dimethacrylate (UDMA) tends to lend color stability, hydrophobicity, high viscosity and tensile strength; triethylene glycol dimethacrylate (TEGMA) with 70.1% by volume and 86.0% by weight of bariumboroaluminiumsilicate filler [19, 28].

Experimental Design:

A basic piece of dental equipment that has made resin composite possible is the dental curing light. The curing light must deliver both sufficient energy and light at the correct irradiance level to produce an acceptably cured restoration. The manufacturer recommended that a 4mm increment of material should be irradiated > 800 mW/cm2 for 10 seconds. The Photocurability of X-traFil was evaluated after using curing light power with a density of 1250 mW/cm^2 in order to represent a typical irradiance level of light curing units that are available and commonly purchased today. The Mini LED LCU (Aceton North America) was used in this study. The Mini LED curing light with an 8-mm diameter curing tip achieves a broad emission spectrum of 420nm to 480nm which covers the range of photoinitiator at 470nm and includes a high intensity of 1250 mW/cm^2. Before each cure, the proficiency of the LCUs intensity was measured using a radiometer (LED Radiometer, SDS/Kerr, Orange, CA). The light-guide tip was placed on the radiometer, when in use the exact amount of light generated is measured. According to the manufacturer, the Mini LCU is suitable for all initiators in a relative wide spectrum of light wavelength (www. photo acetongroup.com). The light source was placed on the top surface of each at a centered distance of 0 millimeters (mm) before curing. The bulk fill composite resin was tested at 10 seconds. The depth of cure

property was evaluated under the surface microhardness test.

Surface microhardness test:

The specimens were prepared in an 8 millimeter inner diameter and 10mm outer diameter custom- made delrin split ring mold at a height of 4mm (Figure 1). The plastic ring mold was assembled by placing the two inner diameter semicircular split ring molds into the outer diameter ring forming a circular opening, and then the completely assembled mold was placed on top of a tofflemire mylar matrix on a glass microscope slide. The mold was filled with the bulk fill composite resin, in accordance with the manufacturer's instructions, taking care to extrude any bubbles. Then, slightly overfilled the mold and put a second tofflemire mylar strip on top, followed by the second microscope slide. Pressed the mold and strips of film between the glasses to displace any excess material. Removed the microscope slide covering the upper strip and gently placed the head of the LCU against the strip of film [20]. Only the top surface of the specimen was irradiated at 1250 mW/cm^2 for 10 seconds while keeping the curing light tip centered in contact with the second plastic strip. In this way, 10 specimens of composite resin were prepared (n=10).

The samples were removed from the molds and the top surfaces marked to distinguish from the bottom surfaces. The samples were tested immediately to avoid further exposure to ambient light after polymerization. The next test material was not prepared until the previous material hardness test had been completed. The bottom and top surface hardness of each specimen was evaluated at the 4mm depth utilizing a Knoop Hardness tester (Figure 2) under a load of 100 grams for 15 seconds dwelling

time (Figure 3). The Knoop microhardness was calculated according to the following equation [23]:

$HK = 1.451\frac{F}{d^2}$, where F is the test force (N), and d is the length of longer diagonal (mm)

For each sample, three KHN readings were recorded for the irradiated top and non- irradiated bottom. Three locations tested to compensate for inherent irregularities in the cured material. Then for each specimen, the mean value and corresponding standard deviation of the KHN were measured. Also, bottom to top KHN percentage was determined and the value of 80% was used to indicate the depth of cure. (Table1).

RESULTS

Of the 10 tested specimens, the top surface Knoop microhardness ranged from 41.00 – 48.60 KHN versus bottom surface microhardness range of 30.10-39.60 KHN. Each specimen's bottom to top surface microhardness ratio was between 73.32 – 81.48 KHN % with an overall average of 79.40 KHN±1.5%, which is insignificant from the standard 80%, a negligible 0.75% value difference, (Figure 4). The results reflect the relative and acceptable curing at a depth of 4mm for X-traFil after 10 seconds.

Table 1. X-traFil Data Collection Sheet

Depth	Sample	T/B	Run	KHN	Surface mean			
D4	S1	T	1	43.8				
D4	S1	T	2	41.3	42.06666667	D4.S1.T		
D4	S1	T	3	41.1			79.16006	D4.S1 ratio
D4	S1	B	1	33.2				
D4	S1	B	2	31.1	33.3	D4.S1.B		
D4	S1	B	3	35.6				
D4	S2	T	1	47.4				
D4	S2	T	2	47.4	45.33333333	D4.S2.T		
D4	S2	T	3	41.2			77.42647	D4.S2 ratio
D4	S2	B	1	36.8				
D4	S2	B	2	34.6	35.1	D4.S2.B		
D4	S2	B	3	33.9				
D4	S3	T	1	43.4				
D4	S3	T	2	43.9	44.23333333	D4.S3.T		
D4	S3	T	3	45.4			77.7694	D4.S3 ratio
D4	S3	B	1	37.4				
D4	S3	B	2	30.9	34.4	D4.S3.B		
D4	S3	B	3	34.9				
D4	S4	T	1	42.9				
D4	S4	T	2	44.5	43.56666667	D4.S4.T		
D4	S4	T	3	43.3			78.19434	D4.S4 ratio
D4	S4	B	1	32.5				
D4	S4	B	2	30.1	34.06666667	D4.S4.B		
D4	S4	B	3	39.6				
D4	S5	T	1	42.9				
D4	S5	T	2	41.3	43.03333333	D4.S5.T		
D4	S5	T	3	44.9			82.18435	D4.S5 ratio
D4	S5	B	1	33.7				
D4	S5	B	2	34.3	35.36666667	D4.S5.B		
D4	S5	B	3	38.1				
D4	S6	T	1	46				
D4	S6	T	2	42.4	45.2	D4.S6.T		
D4	S6	T	3	47.2			79.79351	D4.S6 ratio
D4	S6	B	1	37.5				
D4	S6	B	2	37.3	36.06666667	D4.S6.B		
D4	S6	B	3	33.4				

Depth	Sample	T/B	Run	KHN	Surface mean			
D4	**S7**	**T**	**2**	45.3	44.73333333	D4.S7.T		
D4	**S7**	**T**	**3**	43.9			76.22951	D4.S7 ratio
D4	**S7**	**B**	**1**	32.1				
D4	**S7**	**B**	**2**	34.6	34.1	D4.S7.B		
D4	**S7**	**B**	**3**	35.6				
D4	**S8**	**T**	**1**	41.1				
D4	**S8**	**T**	**2**	48.6	45.36666667	D4.S8.T		
D4	**S8**	**T**	**3**	46.4			80.74945	D4.S8 ratio
D4	**S8**	**B**	**1**	36.8				
D4	**S8**	**B**	**2**	36.4	36.63333333	D4.S8.B		
D4	**S8**	**B**	**3**	36.7				
D4	**S9**	**T**	**1**	43.2				
D4	**S9**	**T**	**2**	42.4	42.66666667	D4.S9.T		
D4	**S9**	**T**	**3**	42.4			79.92188	D4.S9 ratio
D4	**S9**	**B**	**1**	37.5				
D4	**S9**	**B**	**2**	33.3	34.1	D4.S9.B		
D4	**S9**	**B**	**3**	31.5				
D4	**S10**	**T**	**1**	45				
D4	**S10**	**T**	**2**	43.4	44.7	D4.S10.T		
D4	**S10**	**T**	**3**	45.7			79.71663	D4.S10 ratio
D4	**S10**	**B**	**1**	36.8				
D4	**S10**	**B**	**2**	33.3	35.63333333	D4.S10.B		
D4	**S10**	**B**	**3**	36.8				

D4 Ratio Mean 79.4010456

Figure 1: Custom-made Delrin molds. Pictured from left to right is inner split-ring mold, outer split-ring mold and assembled apparatus with test material and fabricated sample.

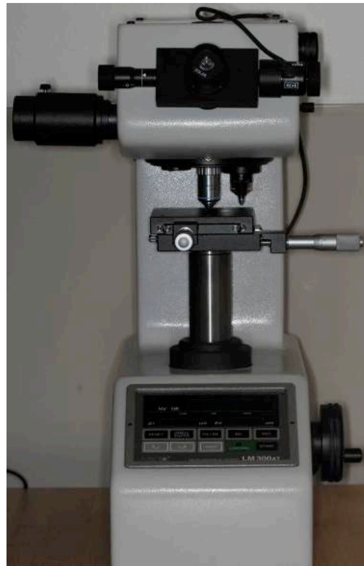

Figure 2: Surface hardness measurements were taken utilizing the Knoop hardness tester**.**

Figure 3: Samples were subjected to a load of 100 grams for 15 seconds by the Knoop hardness indenter.

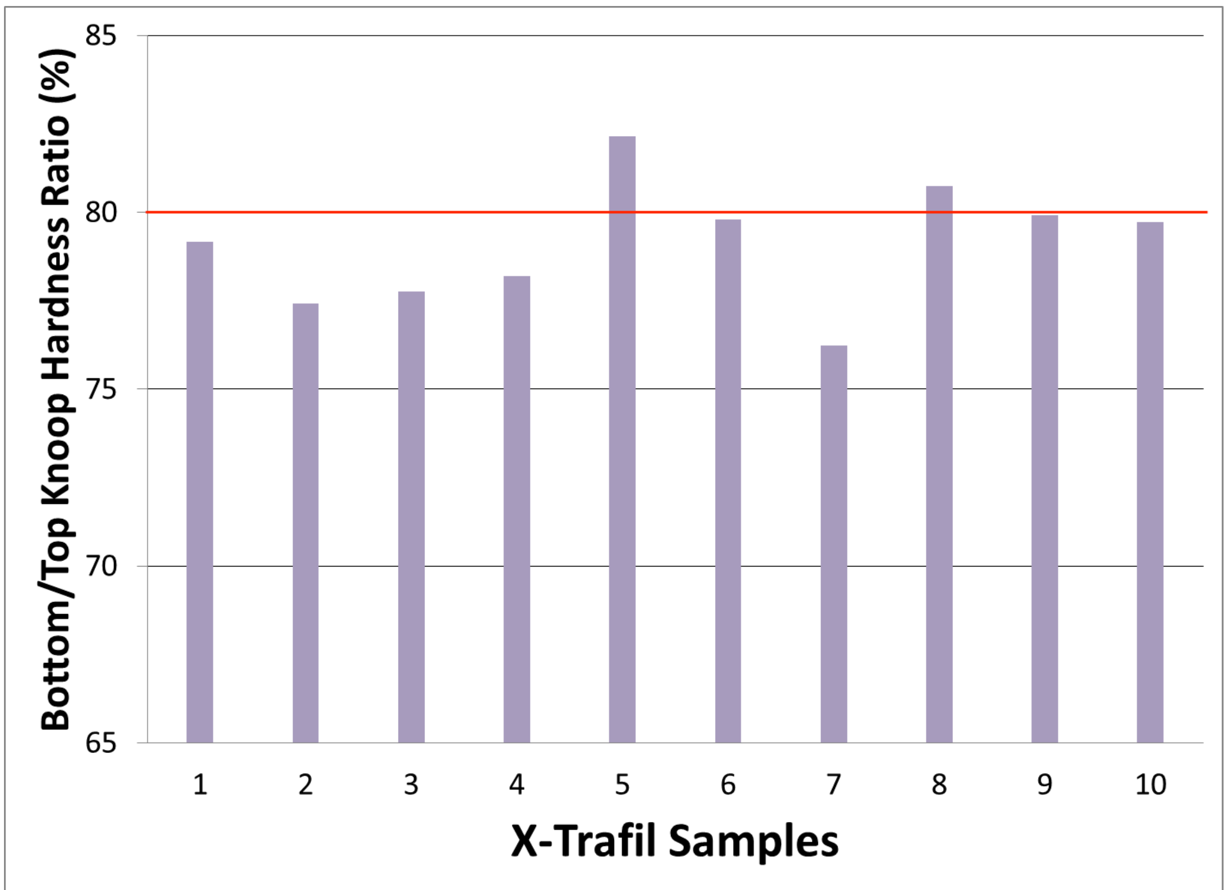

Figure 4: Bottom to top Knoop hardness ratios. Average 79.40 ± 1.5%; 80% acceptable cure.

DISCUSSION

The degree of polymerization plays an important role in the mechanical and physical properties of resin [5]. There are direct and indirect methods for measuring the depth of cure. Infrared spectroscopy is a direct method and microhardness and scraping are indirect methods [5, 16]. Depth of cure can also be defined as 50 percent of the remaining length of the composite resin after the uncured portion has been scraped off [22]. The depth of cure is often assessed indirectly by measuring the hardness of a composite resin material at specified depths. Higher hardness values correlate with a more extensive polymerization [1]. Direct methods, like infrared spectroscopy, are more complex and expensive; however, microhardness test are the most popular method because the other methods tend to overestimate the curing depth [15, 22]. The Knoop microhardness test has been shown to be one of the best methods for testing the hardness of resin composite, and a good correlation between degree of conversion and the Knoop microhardness has been reported [16,22]. The bottom to top hardness ratios ranging from 80% - 85% have been used as criteria for adequate degree of conversion at a specific sample thickness. It means that the bottom to top surface microhardness ratio of 80% or more is adequate curing. The current study reached bottom to top surface microhardness ratio of 79.4 ±1.5%, which is insignificant from 80% (a negligible 0.75% value difference).

Multiple factors might contribute to the test results in this study. In microhardness test (Knoop), the amount of load has a significant effect on microhardness measurements. The most common load is 100-500 grams. The indenter with a higher

load penetrates deeper into the composite, therefore measures a greater hardness [23] because optimum cure is achieved and therefore hardness is often reached slightly below the surface layer. A study by Yoldaz showed that a dwell time of 15 seconds could be accepted as an actual time of load application for the dental composite [24]. Therefore, in the study, the load of 100 grams and the dwell time of 15 seconds were chosen.

Curing light irradiance, exposure time and composition are variables significantly affecting hardness and curing depth [25]. In the present study, the manufacturer's instructions of 10 seconds of exposure time at 800 mW/cm^2 or higher was used to achieve adequate curing depth. Curing light intensity also varies slightly with time, and maintaining exactly the same light intensity for each curing site is almost impossible. The Yazici study determined that the bottom Knoop hardness number of a composite cured with a light emitting diode (LED) curing unit is greater than halogen curing unit [26]. The curing light used was a LED based curing unit. The distance between the curing light tip and the composite was 0 millimeters for all samples.

In addition, the test material has high filler loading of 86% by weight which means less resin in the composites. During microhardness measurement, it was noted that some particle sizes were larger than the indent, resulting in a significant false reading for extreme high microhardness and marked as unacceptable. Careful selection of indent location may also present some difficulty in this study.

Although 10 seconds cure of 4mm material reached acceptable levels, not all samples obtained 80%. Curing for 20 seconds would allow time for the residual material to polymerize further. In the present study, the hardness of X-traFil was acceptable for

the depth of cure at 4mm.

CONCLUSION

The depth of cure and microhardness of cured composites are directly related. Based on this study, X-traFil achieved a reasonable 4 mm depth of cure at 10 sec when light power density is higher than 800 mW/cm^2. It concludes that the X-traFil can be placed in bulk and cured for a clinically acceptable restoration.

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