Depth of Cure and Microhardness of a New Composite vs. Bulk-fill Composites

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ABSTRACT

Aim: To compare the depth of cure of new composite with bulk fill resin composites through using Vickers hardness profiles (VHN).

Materials and Methods: New composite and four bulk fill composite materials were examined: Tetric N Ceram®Bulk Fill, X-tra base, Compon, Filtek Posterior Bulk Fill, Sonic FillTM. Eight specimens of each material type were prepared in teflon molds which contained a slot of dimensions (8 mm × 4 mm × 2 mm), and a top plate. The molds were irradiated from one end. All specimens were stored at 37°C for 24 h, before measurement. The Vickers hardness was measured as a function of depth of material, at 1 mm intervals. Data were analyzed by one-way ANOVA using Turkey post hoc tests (p=0.05).

Results: The maximum VHN ranged from 46.7 to 68.1, whilst the 80% of max. VHN ranged from 37.4 to 54.5. The depth corresponding to 80% of maximum VHN ranged from 5.5 to 7 mm. This was taken as the depth-of cure.

Discussion: In this study, VHN, 80% VHN and depth profile properties of different Bulk Fill resin composites were measured and compared with the new composite by the determination of their VHN/depth profiles. There were statistically significant differences between different bulk fill composite materials in terms of max. VHN and depth of cure, corresponding to 80% of max. VHN. Depth of cure is related to the clinical success of restoration. When a more extensive polymerization and crosslinking occurs, greater VHN results are obtained. According to the manufacturer's claims bulk fill composites can be applied up to 4 mm, or even 5 mm in one time. In general, adequate polymerization along the recommended depth in bulk fill composites is critical to the long-term stability of restorations. According to the results of our research, the manufacturers' claim was approved for these materials.

Conclusion: New composite can be cured to an acceptable post-cure depth as Bulk fill resin composites, according to the manufacturers' claims. X-tra base had the greatest depth of cure among the composites examined.

Key words: Bulk fill composites, Microhardness, Depth of cure

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INTRODUCTION

Resin based composite have recently been used more often as posterior restorations instead of amalgam [1]. Since the day when composites entered the dentistry in the 1960's they have shown various developments [2]. The mechanical, physical [3], biological [4], and esthetical properties of composites are being improved day by day according to needs of dentists and research results. Despite these improvements, dentists are experiencing many problems in the placement and irradiation stages of the composite [1].

One of the most important problems is the inadequate polymerization due to the fact that the light does not reach certain depths for various reasons [5] insufficient polymerization causes degradation of the composite resin, side effects due to non-polymerized monomers [6], and low physical properties [7]. Especially in large cavities to prevent inadequate polymerization, the composites are applied in 2 mm layers for the standard composites in order to achieve sufficient hardness and avoid polymerization shrinkage [8].

A new type of resin based composites have been introduced to overcome both these problems and to provide convenience to dentists [9]. "Bulk fill Resin composites" Bulk fill Resin composites have been shown to overcome circumstances such as incorporating voids [10], contamination

between layers [11], and ease of application in deep cavities. It has also been proved that problems like cuspal deflection and marginal integrity are better than standard composites [12,13]. According to the manufacturer's claims bulk fill composites can be applied up to 4 mm, or even 5 mm in one time [14]. Bulk-fill composites contain low amount of enlarged filler [15].

Camphorquione is generally used In Bulk-fill Resin Based composites [16], for photo-initiation but Tetric EvoCeram Bulk fill contains an additional initiator called "Ivocerin". Ivocerin has higher photo curing activity than CQ due to it's higher absorption of visible light [17]. Bulk-fill composites were first launched to the market in flowable form. This flowable form of Bulkfill composites – Venus Bulk Fill, SDR and Filltek Bulk Fill - are needed an additional capping layer. This capping layer has using for aesthetical adjustments [15].

There is not only flowable form of Bulkfill Composites exist, also high-viscosity resin Based Bulk fill composites are available in market (Tetric EvoCeram Bulk Fill, X-tra fill and Sonic Fill) [15]. One of the high-viscosity composite is also sonic activated (Sonic Fill). It is applied like a flowable composite with sonic wave and can be carved and contoured like other high-viscosity resin based composites.

Nova Compo N contains ULS (Ultra Low Shrinkage) dimethacrylate monomer. The ULS monomer enhances high percent elongation and toughness to improve the durability of restorations. ULS monomer also has higher degree of conversion than traditional UDMA, Bis-GMA monomers. Nova Compo N consists of varying particle diameters (hybrid type) of fillers. These are prepolymerized fillers, Micro inorganic St-glass fillers and Silica nano fillers and nano ytterbium trifluoride.

The depth of cure can determine with several methods.

1. For dental composites ISO 4049 advocates scraping of the unset material after irradiation and measuring the length of the set specimen [18].

2. With measuring the hardness or conversion degree of the the top and bottom specimen surfaces [19].

3. Visual border between cured and uncured material is determined by Optical Microscopy [20].

4. Surface microhardness of resin composite [21].

The degree of polymerization of resin composites can be evaluated indirectly by surface microhardness. Researchers have been also used surface microhardness of resin composites for efficiency of the light cure unit. As the depth increases, the conversion degree decreases because of proceeded light rays passing through the resin composites will be reduced [21]. In this study, for measuring the depth of cure of different bulk fill resin composites were used a surface microhardness profile.

The purpose of this study was to evaluate the depth of cure of four different commonly used bulk fill resin composites and one new experimental composite. This evaluation includes the parameters

1. The maximum Vickers microhardness,

2. 80% of the maximum Vickers microhardness, and (iii) the depth corresponding to 80% of the maximum Vickers hardness.

By the null hypotheses there would be no differences between materials, both in maximum Vickers hardness and in the depth of cure that corresponding to 80% of maximum Vickers hardness for bulk fill materials.

MATERIALS AND METHODS

Five dental-composites (Tetric-N Cream Bulkfill, Ivoclar, Liechtenstein; X-trabase bulk fill, Voco, Germany; Filtek Bulkfill, 3 M, USA; Sonic Fill, Kerr, USA; Nova Compo-N, İmicryl, Turkey) were evaluated in this study (Table1). Eight specimens of each resin composite (n=8) were prepared for surface microhardness profile measurements in teflon molds. These teflon molds with two pieces contained a slot of dimensions (8 mm × 4 mm × 2 mm). The half mold was overfilled with composite than the other half of the mold subsequently pressed into position and a Mylar strip was placed on top of the material. This phase followed by the scraping of the excess material from the bottom of the mold. The two part of the mold was held together with key lock mechanism. The molds were irradiated from one end. Each specimen was photo-polymerized for 20 s using a visible light cure unit with a tip diameter 10 mm (VALO, USA) under the standard curing mode output wavelength range

390–480 nm; output irradiance was 1800 mW/cm^2). A calibrated radiometer system (MARC, BlueLight Analytics Inc, Halifax, NS, Canada) was used to verify the irradiance at each use of the light cure unit. All specimens were stored dry at 37°C for 24 h prior to measurement. The top of the mold and the Mylar strip were removed and the Vickers hardness number (VHN) was measured as a function of depth of material at 1 mm intervals. All specimens were examined by a microhardness instrument (Mitotoyo Japan). A fixed load of 300 g was applied for 15 s. Data were calculated as hardness numbers and accordingly plotted as hardness versus depth profiles.

STATISTICAL ANALYSIS

In each of the 5 groups; max.VHN values obtained at the defined distances, then VHN at 80% of max. VHN, depth at 80% of max. VHN values were calculated. Statistical analyses were done using statistical software (SPSS ver.20, IBM Corp, Armonk, NY, USA) at (p=0.05) significance level. The Kolmogorov–Smirnov Test indicated that all VHN data for the materials was normally distributed. All data were analyzed using one-way ANOVA, and post hoc Tukey tests for multiple comparisons of the following parameters:

1. Max.VHN.

2. VHN at 80% of max.VHN.

3. Depth at 80% of max.VHN (dependent variable) between different materials (independent variables) presented in Table 2.

The relationship between VHN and filler content was evaluated by linear regression.

RESULTS

The acronym-codes of composites are given in Table 1. The max. VHN, VHN at 80% of max. VHN and depth at 80% of max. VHN of each bulk fill resin composite examined are presented in Table 2. The maximum VHN ranged from 46.7 to 68.1 presented in Figure 1, whilst the 80% of max. VHN ranged from 37.4 to 54.5. According to Manufacturer's claim of all composites in 5mm depth ranges from 80-90% presented in Figure 2.

The depth corresponding to 80% of max.VHN ranged from 5 to 7 mm. This was taken as the depth-of-cure (DoC). One-way ANOVA showed statistically significant differences between materials for all parameters measured. SF exhibited the highest max.VHN with significant differences (p<0.05), whilst TNF the lowest (p ≤ 0.05). TNF, XB and CN showed no significant differences (p>0.05). Furthermore, FBF showed statistically significant differences with TNF (p<0.05). The depth obtained, for all materials, corresponding to 80% of the max. VHN met each matched manufacturer's claim. XB (p>0.05) had the greatest depth of cure (7 mm). SF showed no significant differences with TNF and XB. SF showed significant differences in depth of cure with TNF, XB, FBF and CN. Linear regression confirmed a positive correlation between max. VHN and depth of cure (r^2=0.94) (Figure 1).

Table 1: Material studied.

Materials	Code	Type	Manufacturer increment thickness (mm)	Matrix	Filler % (wt)	Manufacturer
Tetric N Ceram	TNF	Universal	4	Bis-GMA, dma dimethacrylate	80 17 (prepolimer)	Ivoclar Vivadent AG,Schaan, Liechtenstein
X-tra base Bulk fill	XB	Universal	4	Aliphatic dimethacrylate, bis-EMA, UDMA, EBADMA	75	Voco GmbH Cuxhaven, Germany
Filtek Bulk fill	FBF	Universal	4	Bis-GMA, BisEMA, UDMA	64.5	3M ESPE GmbH, Seefeld, Germany
Sonic fill	SF	Universal	5	Bis-GMA, TEGDMA, Bis-EMA, SIMA	83.5	Kerr Corp, Orange, USA
Compo-N	CN		4	Dimethacrylate ULS	78 18-22 (prepolimer)	Imicryl Konya, Turkey

Table 2: Mean (standard deviation) of max.VHN, VHN at 80% of max.VHN and depth at 80% of of max.VHN and depth at 80% of max.VHN for the examined bulk fill composites.

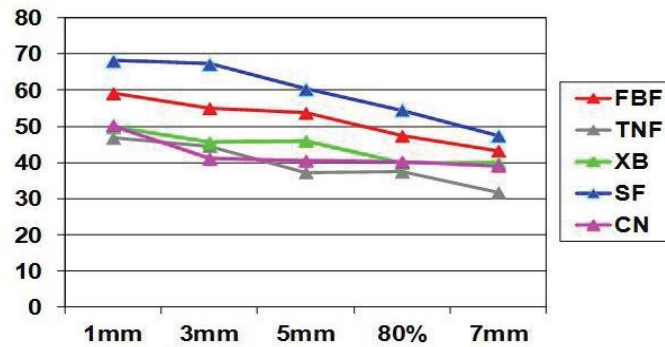| Materials	Max.VHN Mean(SD)	VHN at 80% of max.VHN Mean (SD)	Depth at 80% of max.VHN (mm) Mean(SD)
FBF	59.1875 (ab)	47.34	6 mm
SF	38.1875(a)	54.54	6 mm
XB	49.7875 (b)	39.83	7 mm
TNF	46.7625 (b)	37.4	5 mm
CN	50.2625 (b)	40.2	5.5 mm

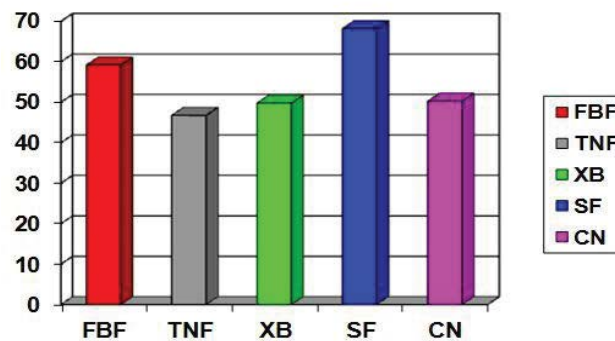Figure 1: Depth at 80% of max. VHN for bulk fill composites.

Figure 2: Maximum VHN of bulk fill composites.

DISCUSSION

In this study, VHN, 80% VHN and depth profile properties of different Bulk Fill resin composites were measured and compared with the new composite by the determination of their VHN/depth profiles. There were statistically significant differences between different bulk fill composite materials in terms of max.VHN and depth of cure, corresponding to 80% of max. VHN. Thus the null hypothesis was rejected. According to the results of our research, the manufacturers' claim was approved for these materials.

In previous research; as the thickness of the applied dental composite layer increased, a decrease in the degree of conversion was observed and a decrease in microhardness was observed. In conventional dental composites it is reported that the stratification should not exceed 2 mm and in another study, to achieve an even and high degree of polymerization, the applied dental composite sheets are ideal for a thickness of 2 mm. However According to the manufacturer's claims bulk fill composites can be applied up to 4 mm, or even 5 mm in one time [14]. For this reason, in our study 8 × 4 × 2 mm samples were prepared for bulk fill composites and new composite material in order to calculate polymerization depth.

Polymerization of resin composites proceeds slowly after curing and can reach a termination point of about 24 h moreover hardness may increases about up to 1 month [22]. Some factors reduce the light penetration correspondingly the degree of conversion. The factors are light scaterring at particle interfaces and light absorbance of photoinitiators and some pigments [23]. Camphorquinone (CQ) is a widely used photoinitiator for resin composites, absorbing light from 450 to 490 nm [16]. Some resin composites involves another photo-initiators; for example TNF. TNF involves a new photo-initiator system (IvocerinTM–a dibenzoyl germanium compound). This absorbs visible light over a wider range of wavelengths from 370 to 460 nm. With suitable formulations, reactivity is enhanced and greater depths of cure can be achieved. In addition, the power of the light source, the quality of the light source, the distance between the light end and the composite surface, the layer thickness of the applied composite, the color of the composite and the composition of the organo-inorganic structure in the composite vary depending on the composition [24]. Light intensity decreases as the amount of distance between the light gun tip and restoration increases. It is recommended that light guns have

a distance of 1 mm between the ends and the restoration [25]. As the distance between them increases, the light intensity decreases much more proportionally. This loss is reduced by 7% at a distance of 2mm, reaching up to 25% at a distance of 4 mm [26]. Therefore, in this study we irradiated a microscope slide thickness from a distance of 1 mm. Valo (Ultradent, USA) was used as light source as a wide spectrum LED light device designed to polymerize all light-curing products between 390–480 nm wavelength range; output irradiance was 1800 mw/cm^2.

The inorganic structure of the composites as well as the organic structure is very important in the depth of cure. The polymerization shrinkage is reduced when the inorganic filler ratio is high in the dental composite. Thus, a better composite of mechanical and physical properties is obtained [27]. In the studies performed with different brand composites, depth of cure, degree of conversion and physical properties of composites differences are explained by the organic matrix and inorganic filler diversity in the composite content [28]. Therefore, the most important aim of composite manufacturers is to increase the ratio of inorganic fillers in dental composites. There is a positive correlation between physical properties such as inorganic filler content and surface hardness in dental composites [29]. It was noted that when the inorganic filler ratio in the dental composites is increased or the filler shapes used are irregular, the light transmission decreases. The high particle size of the inorganic fillers reduces the transmission of visible light to deep layers. As a result, this causes a decrease in depth of cure [30].

SF has the highest filler content and it is applied with a special sonic method. Sonic energy has increased the flow with a special hand tool, which makes it easier to distribute the composite. The organic structure of SF includes ethoxylated bisphenol-A-dimethacrylate, Bisphenol-A-bis-(2-hydroxy-3-methacryloxypropyl) ether, trietylglycolylmethacrylate, 3-trimethoxysilylpropyl methacrylate. As filler there are SiO_2, glass, oxide and various chemicals. It contains 83.5 wt%, 69 wt% inorganic filler.

The refractive index of barium glass particles used in dental composites is higher than UDMA. This condition reduces the light transmittance of the composite, which leads to a reduction in the translucency of the composite. In addition, the refractive index of strontium glass particles is compatible with UDMA, which increases the light transmission [31]. In order to obtain a better depth of cure, refractive indices are targeted to improve the translucency of the material with using the compatible inorganic filler and organic matrix [32]. CN involves ULS monomer in its organic matrix. This monomer is claimed to diminish polymerisation shrinkage by manufacturer. CN's inorganic matrix was claimed to have a deeper irradiation depth and high mechanical properties due to prepolymer filler micro inorganic St-glass fillers and silica nanofiller and nano ytterbium trifluoride content. The high depth of cure of this composite can be explained by the content of glass-based fillers and modified organic matrix contents.

Optical microscopy and scrapping methods may give higher results than the hardness method as they include some prepolimerized material [20,32]. FTIR is less sensitive than hardness assessment in detecting small changes when the network is crosslinked [32].

DoC is very important for clinical success. DoC is significantly linked with the values of mechanical properties, biocompatibility and color stability. More efficient polymerization and more cross-linking connection means higher hardness results.

There is no statistically significant difference between XB, TNF and CN were observed in terms of maximum VHN between bulk fill materials. There was no significant difference between FBF, SF and CN in terms to DoC There is no significant difference between surface microhardness of FBF, XB, TNF and CN.

In the present study, SF showed significant differences in depth of cure with TNF, XB, FBF and CN. Similar results were reported using similar methodology. In those studies, SF and TNF had the greatest VHN. The good DoC may be due to refractive index matching between the resin and filler, which enhances light transmission. The highest DoC was observed in XB and the lowest in TNF. Benetti et al. [32] observed that the depth of cure of XB was higher than SF and TNF in their study. According to our in vitro study, newly produced BulkFill composite CN, showed clinically acceptable depth of cure and microhardness as other BulkFill composites.

In terms of DoC, this composite promising for restoration of deep cavities.

CONCLUSION

Within the limits of this in-vitro study all investigated Bulk fill composite resins had sufficient polymerization properties at 4 mm increment thickness, in agreement with the manufacturers' claims. The depth of cure of tested material (Compo N) is similar to other bulk fill composites; because it maintains the existing surface microhardness up to 5.5 mm. The materials used in this study are promising composite materials because they are useful in clinics for adhesive filling of deep cavities when time consuming incremental techniques are not possible due to low patients compliance.

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