

Degree of conversion and polymerization shrinkage of low shrinkage bulk fill resin composites

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ABSTRACT

Background: The effectiveness of each resin composite material composition in enabling homogeneous monomer conversion and reducing polymerization shrinkage is an important issue. Thus; this study aimed to evaluate the degree of conversion (DC) and polymerization shrinkage (PS) of low shrinkage bulk-fill composites in 4mm thickness. **Materials and Method:** Four bulk fill (Tetric EvoCeram Bulk Fill, Sonic Fill, XTra Fill and Venus Bulk Fill) and one conventional (Filtek Z350 XT) resin composites were tested in this study. Teflon molds of 4mm depth were used to prepare resin composite specimens. All tested resin composites were packed in bulk then light cured for the recommended time by their manufacturers. DC% was determined by FT-IR spectroscopy; PS was determined using the strain gauge method. Data were subjected to One-way ANOVA/Tukey's test ($\alpha = 0.05$). **Results:** DC % results were as follow; Venus Bulk Fill > XTra Fill > Sonic Fill > Tetric EvoCeram Bulk Fill > Filtek Z350 XT with a significant difference between each other's at $p \leq 0.001$. For PS %, significant difference resulted between tested groups at $p \leq 0.001$. Where Venus Bulk Fill < XTra Fill = Sonic Fill < Tetric EvoCeram Bulk Fill < Filtek Z350 XT. **Conclusions:** Low-shrinkage resin monomers seem to be beneficial for reducing PS with enhancement in the DC. Increased filler loading in bulk fill composites had positive effect on reducing PS, while its effect on the DC was material dependent.

Keywords: Degree of conversion, low Shrinkage, Bulk fill, Resin, polymerization.

Introduction

Resin based composites (RBCs) restorations are the most widely demanded restorations in dental practice due to its esthetic properties. In spite of the continuous improvements in resin composite over the last decades, it still suffers from its shrinkage during polymerization and the accompanied stresses plus being a technique sensitive restoration with prolonged application time. The incremental application technique is a common protocol for application of conventional light activated composites, which is done by applying increments no thicker than 2mm to ensure a homogeneous degree of conversion throughout the material thickness plus being a way to reduce polymerization stress (Gonçalves *et al.*, 2018 and Nagi *et al.*, 2015).

Aiming to decrease the clinical application time of resin composite, the so-called "bulk fill" composites (BFCs) were introduced in the market. Manufacturers of BFCs claimed that, these materials allow uniform polymerization of increments up to 4 to 5 mm thickness, with low polymerization shrinkage (PS) than conventional RBCs (Hirata *et al.*, 2015).

Different attempts were followed by BFCs manufacturers by optimize the polymerization and lower the volumetric polymerization shrinkage such as utilizing; stress reliever, polymerization modulator (Hirata *et al.*, 2015), or by variations in the filler size [XTra Fill (VOCO)] and filler content [Tetric EvoCeram Bulk Fill (Ivoclar Vivadent)] and change in the chemistry of their resin matrix (Gonçalves *et al.*, 2018 ; Nagi *et al.*, 2015 and Hirata *et al.*, 2015) e.g; introducing low shrinkage monomers (e.g; EBPADMA) [Sonic Fill (Kerr), and Venus Bulk Fill (Heraeus Kulzer)].

Results of studies that have been carried out with this new category of materials are contradicting. Some authors found similar or higher degree of conversion, and lower shrinkage of bulk fill materials at 4 mm thickness (Tsujiyama *et al.*, 2016 and Par *et al.*, 2015). On the other hand; others revealed

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significant decrease of conversion in bulk fill composites at 4 mm thickness, (Gonçalves *et al.*, 2018 and Benetti *et al.*, 2015) or higher volumetric shrinkage (Son *et al.*, 2017 and El-Korashy, 2010) than that of conventional composites. Thus, the clinical implications of the use of these new materials seem unclear.

Based on this knowledge, the purpose of the present study was to evaluate the degree of conversion and polymerization shrinkage of different bulk fill composites compared to conventional resin composite, in order to assess the effectiveness of each manufacturer approach in enabling homogeneous monomer conversion and reducing polymerization shrinkage. The null hypothesis was that the different bulk fill and conventional resin composites tested in this study present similar performance in terms of degree of conversion, and polymerization shrinkage.

Materials and Method

Selected materials:

Five commercial resin composites; one conventional and four bulk fill resin composite materials were selected for this study. Materials name, manufacturers and their composition, time/irradiance are presented in Table 1.

Table 1: Materials name, manufacturers, composition and their time/ irradiance.

Composite type	Material/ Manufacturer	Organic matrix	Filler content	Tim/ Irradiance
Conventional composites	Filtek Z350 XT / 3M ESPE, St Paul, USA	Bis-GMA, Bis-EMA, UDMA, TEGDMA	72.5 wt%	20-sec/ (>1000mW/cm ²)
Bulk-fill composites	Tetric EvoCeram Bulk Fill / Ivoclar Vivadent AG, Schaan, Liechtenstein, Germany	Bis-GMA, Bis-EMA, UDMA	79-81wt%	10-sec / (>1000mW/cm ²)
	Sonic Fill / Kerr Corporation, Orange, California, USA	Bis-GMA, TEGDMA, EBPADMA	83.5 wt%	20-sec/ (>1000mW/cm ²)
	XTra Fill / VOCO, Cuxhaven, Germany	Bis-GMA, UDMA, TEGDMA	86wt% 70.1vol%	10-sec / (>1000mW/cm ²)
	Venus Bulk Fill /Heraeus Kulzer GmbH, Hanau, Germany	EBPADMA, UDMA	65 wt%, 38vol%	20-sec/ (>1000mW/cm ²)
Bis-GMA , bisphenol-glycidyl methacrylate; Bis-EMA , ethoxylated methacrylate; UDMA , urethane dimethacrylate; TEGDMA , triethylene glycol dimethacrylate; EBPADMA , ethoxylated bisphenol A dimethacrylates				

Degree of conversion (DC) test

Specimens preparation for the degree of conversion testing:

Fifty specimens were prepared for the degree of conversion test. They were divided into five groups (n=10/group) representing the tested resin composites. The 10 specimens of each group were divided as follow: five uncured specimens to serve as control and five experimental cured resin composite specimens.

For preparing the 25 experimental cured resin composite specimens (n=5/group); sectional Teflon molds of 6 mm diameter and 4 mm thickness were used. The molds were first mounted on the top of a microscope slide and a Mylar strip, and then the mold was filled in bulk with one of the tested resin composites using a plastic spatula. Only, Sonic Fill resin composite; was inserted into the molds with a hand piece that produced ultrasonic waves according to the manufacturer's instructions. The top side of the mold was covered with a second Mylar strip to prevent oxygen inhibition. A glass microscope slide with a load of 1 kg was applied for 30 seconds to ensure consistent packing of the specimens (Nagi *et al.*, 2015). The load and the microscope slide were then removed. The specimens were light cured from the top surface only using LED Elipar S10 light curing unit (Elipar S10, 3M

ESPE Dental Products, St. Paul, MN, USA) with an output of $>1000 \text{ mW/cm}^2$ for the recommended time as mentioned by the manufacturer of each tested resin composite (Table 1).

The light curing tip was kept centered and in direct contact with the second Mylar strip. The power density of light curing unit was assessed using a hand held radiometer (Curing Radiometer, Demetron, Danbury, CT, USA). After light curing, the cylindrical specimens were pushed out of the mold and excess resin composite material was removed with a plastic spatula Nagi *et al.*, 2015).

Specimens were stored in light proof containers before the tests were conducted, in complete darkness at 37°C for 24 hours to prevent ambient light from causing additional post light curing polymerization (Hirata *et al.*, 2015).

Degree of Conversion (DC) testing:

FT- IR spectra of the uncured and cured tested resin composite materials were obtained using 24 scans at 4cm^{-1} in the absorbance mode (Jasco FT - IR 6400, Japan). The FT- IR spectroscopy was completed using a potassium bromide pellet technique.

Twenty-five uncured specimens ($n=5/\text{group}$) were prepared for FT-IR testing to be used as control specimens for the five resin composites tested in this study.

For preparation of the control specimens; 2mg of each of the uncured resin composites were blended with 7mg of Ispectropic grade (IR) potassium bromide powder in a specimen holder; then it was pressed into a transparent disc (1mm thickness) under heavy pressure for one minute,, using a pellet maker kit (KBr Product - A- Press, International Crystal Labs, Garfield, NJ, USA), before it was subjected to FT- IR analysis (Hirata *et al.*, 2015).

For the previously prepared experimental cured resin composite specimens; FT- IR spectra were obtained after 24 hours in dark storage at 37°C . Each specimen was completely crushed and ground into fine powder using a mortar and a pestle. Subsequently, 2mg of each resin composite powder were blended with 7mg of Ispectropic grade (IR) potassium bromide in a specimen holder, and then it was pressed into a transparent disc using a pellet maker kit (Par *et al.*, 2015). The specimen holder was transferred to the spectrometer and a spectrum was obtained using the same parameters as for the uncured resin composites specimens.

For calculating the DC %, the percentage of unreacted carbon-carbon double bonds (% C=C) was determined from the ratio of absorbance peak areas of aliphatic carbon-carbon double bonds C=C (peak at 1638 cm^{-1}) against aromatic component (peak at 1608 cm^{-1}) which was used as an internal standard before and after curing. The underlying peak area was calculated for each peak, using a standard baseline technique with the aid of a computer software program provided with the spectrometer (Spectra Manager Version 2). The degree of conversion was determined according to the formula (El-Korashy, 2010):

$$\%DC = 1 - (\text{aliphatic C=C}/\text{aromatic C=C}) \text{ of polymer} \times 100 / (\text{aliphatic C=C}/\text{aromatic C=C}) \text{ of monomer.}$$

Polymerization shrinkage test

A total of fifty specimens were prepared for the polymerization volumetric shrinkage test. Specimens were divided into five groups ($n=10/\text{group}$) representing the tested resin composites.

The test setup included a white Teflon frame 4 mm diameter and 4 mm height that was used to circumscribe the resin composite specimens. The Teflon frame was chosen so as not to adhere to the resin composite, thus allowing its free shrinkage. A glass slide served as a base for the setup. A foil electrical resistance strain gauge (Strain Gauges, Kyowa Electronic Instruments Co, LTD, Tokyo, Japan, Lot #Y4003S) was placed onto the flat glass surface at the center of the mold. The gauge was 1 mm in length and had an electric resistance of 120W and a gauge factor of $2.09 \pm 1.0\%$ (Benetti *et al.*, 2015 and El-Korashy, 2010).

Resin composite restorative material was placed in the cavity of the Teflon frame in a single increment using a plastic spatula, with the strain gauge centralized in place. Only, Sonic Fill resin composite; was inserted into the molds with a hand piece that produced ultrasonic waves according to the manufacturer's instructions. Care was taken to ensure complete filling of the frame followed by placement of a Mylar polyester strip, then the excess composite material was extruded using pressure applied through a second glass slide that was then removed (Cramer *et al.*, 2011). The foil strain gauge was connected to a strain-monitoring device (Strain-Meter PCD-300A Kyowa-Electronic Instruments

Co, LTD, Tokyo, Japan) initially balanced at zero. The resin composite was cured using LED curing unit (Elipar S10, 3M ESPE Dental Products, St. Paul, MN, USA) with an output of >1000 mW/cm² for the recommended time of each material by the manufacturers (Table 1). Strain measurements were recorded during curing and 5 minutes following light irradiation at room temperature (25± 1°C) (Par *et al.*, 2015 and El-Korashy, 2010).

Statistical analysis

Data presented as mean and standard deviation (SD). Data explored from normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. Data showed a parametric distribution, so One-Way ANOVA was used to study the effect of different materials followed by Tukey's HSD post-hoc test for pairwise comparison. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Coporation, NY, USA) version 25 for windows.

Results

Table 2 shows results of the degree of conversion percentage (DC %) and polymerization shrinkage %.

Degree of conversion % overlapped with line chart showing the polymerization shrinkage % for different tested materials was presented in Figure 1.

Table 2: One-way ANOVA results for degree of conversion% and polymerization shrinkage %.

	Filtek Z350 XT		Tetric EvoCeram Bulk Fill		SonicFill		XTra Fill		Venus Bulk Fill		p-value
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Degree of Conversion %	14.76 ^c	0.05	41.06 ^d	0.09	63.04 ^c	0.05	64.20 ^b	0.07	86.10 ^a	0.12	≤0.001*
Polymerization shrinkage %	2.12 ^a	0.08	1.95 ^b	0.03	1.24 ^c	0.05	1.17 ^c	0.09	0.39 ^d	0.02	≤0.001*

Same letter within each row is not significant. *= Significant

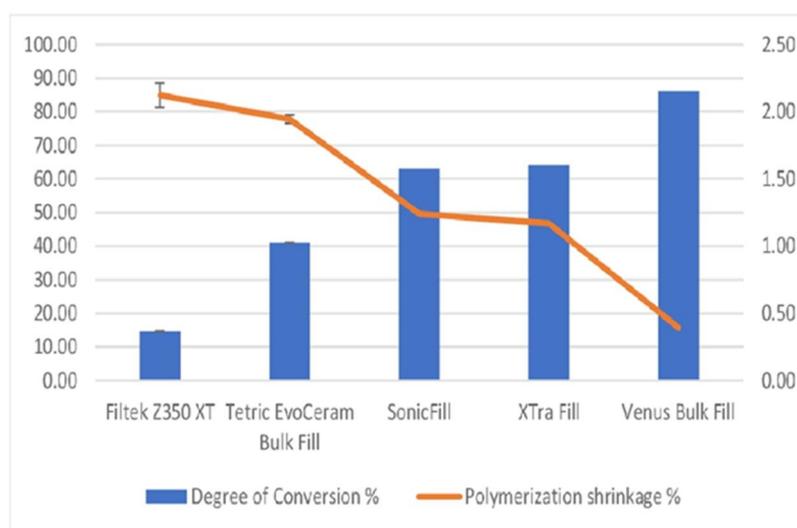


Fig. 1: Degree of conversion % overlapped with line chart showing the polymerization shrinkage % for different tested materials.

Discussion

The setting process of resin composite has a major effect on its mechanical and biological properties (Cramer *et al.*, 2011). Filler loading, shape and size, resin matrix composition, photoinitiator concentration and the polymerization conditions are all factors influencing resin polymerization (Leprince *et al.*, 2013). Since polymerization conditions, such as layer thickness, intensity of the curing unit and exposure times (according to each manufacturer instruction), were standardized in this study,

differences in the DC value of conventional and bulk fill RBCs could be explained by the different composition of the materials, mainly to difference in the chemistry of their resin matrix and the filler loading.

Results of this study revealed that the conventional resin composite Filtek Z350 XT showed the least degree of conversion % compared to all tested bulk fill resin composites. These results were expected as the conventional resin composite was placed in bulk and tested at 4mm thickness, while it is recommended by its manufacturer to be placed in increments not more than 2mm thickness to ensure homogeneous degree of conversion throughout the material thickness plus being a way to reduce polymerization stress (Gonçalves *et al.*, 2018)

It seems that bulk fill composites manufacturers had followed different strategies to increase the depth of cure in these resin composites, which in turn affect the degree of monomer conversion into polymer. Which can explain the high degree of conversion at 4 mm observed for bulk fill materials when compared to conventional composites.

The Venus Bulk Fill resin, revealed the highest degree of conversion, compared to all tested bulk fill materials at this study at 4mm thickness. This could be explained by its high translucency (Kim *et al.*, 2015) and lower filler content (Cramer *et al.*, 2011) leading to more light transmission and constant conversion from the top to the 4mm depth. More over the chemical compositions of the resin matrices play an important role in the degree of conversion of this bulk fill resin composite. The initial viscosity and the flexibility of the monomers crystalline structure are the main features affecting the DC (Miletic *et al.*, 2017).

Venus Bulk Fill composites have large amount of UDMA and EBPADMA, which are low viscous monomers that allow the prolonging of the polymerization reaction so increase the degree of monomer conversion (Alshali *et al.*, 2013). Our results are in agreement with other studies that found higher conversion of Venus Bulk Fill compared to other bulk fill resin composites both in superficial layers and at 4 mm depth (Zorzin *et al.*, 2015 and Par *et al.*, 2015).

For XTra Fill resin composite, the manufacturer increased the filler size. Consequently, the specific surface between fillers and organic matrix is lowered, thus reducing light scattering. XTra Fill has high translucency despite of its high filler loading, which is related to the increase filler size as mentioned before and by the improved refractive indices of the filler particles and the resin matrix (Primus *et al.*, 2002).

On the other hand; Sonic Fill (sonic activated bulk fill system) contains special modifiers of photoinitiators and uses refractive index matching in the composite material, aiming to increase the depth of cure and the degree of conversion in depth up to 5mm.

Sonic fill revealed significant decrease in the degree of conversion compared to (Venus Bulk Fill and XTra Fill resin). This might be due to that it has low translucency which was confirmed by previous studies compared to other bulk fill materials (Roggendorf *et al.*, 2011 and Ilie *et al.*, 2009). Low translucency affects the light transmission with negative effect on the depth of conversion (Howard *et al.*, 2010).

The superiority of Venus Bulk Fill, Xtra Fill and Sonic Fill compared to Tetric EvoCeram bulk fill regarding DC% was related to their resin matrix composition which contains low viscosity monomers TEGDMA, UDMA (XTra Fill and Venus Bulk Fill), and EBPADMA (Venus Bulk fill and Sonic Fill) these low viscous monomers increased the flow and decreased the viscosity of the resin matrix and thus increasing DC%, as these monomers are more flexible and flowable monomers than Bis- GMA and Bis- EMA present in Tetric EvoCeram Bulk Fill (Arahalah *et al.*, 2014).

The manufacturer of Tetric EvoCeram Bulk Fill added an additional photo-initiator (Ivocerin) in the resin composite formula, aiming to act as a polymerization booster offering greater reactivity to curing light compared to champhorquinone at depth up to 4mm. Despite this it revealed the least degree of conversion compared to the other tested bulk-fill resin composites. Tetric EvoCeram Bulk Fill is a nanohybrid composite contains high nano-filler plus pre-polymerized resin fillers loading (Nagi *et al.*, 2015)

. Pre-polymerized fillers are fillers embedded in resin, polymerized and milled to obtain a desired particle size. It is well known that light intensity decreases as it passes through the material. Both intensity of the light source and attenuating power of the material influence the degree of conversion (Zorzin *et al.*, 2015). Pre-polymerized fillers, plus the high filler loading (79-81wt%) of Tetric EvoCeram Bulk Fill might strongly influence the intensity of the incident light, limiting the depth

of cure.

On the contrary; a study performed by (Alrahlah *et al.*, 2014). Found that Sonic Fill and Tetric EvoCeram Bulk Fill showed greater depth of cure than Venus Bulk Fill at a depth of 4 mm. this conflict might be due to the different methodologies applied. Alrahlah *et al.*, 2014 applied a methodology based on an evaluation of micro-hardness to indirectly determine the DC, and it is not necessarily for a material with the highest micro-hardness to provide the highest DC.

On the other hand; results of the polymerization shrinkage were very interesting as it was unexpected. Although Filtek Z350 XT revealed the lowest degree of conversion, it presented a high volumetric polymerization shrinkage, while Venus Bulk Fill showed the highest degrees of conversion, but it had the lowest volumetric polymerization shrinkage. It is well known and commonly reported in the literature, that monomer conversion is directly proportional to volumetric shrinkage (Kaisarly *et al.*, 2016).

For the conventional resin composite Filtek Z350 XT, the high concentration of double bonds of TEGDMA, used as diluent monomer, may have increased its shrinkage. Greater volumetric shrinkage of the Filtek Z350 XT composite compared to several bulk fill composites was observed by (Kim *et al.*, 2015). Move over all tested bulk- fill resins in this study presented lower shrinkage than the conventional Filtek Z350 XT, which was in agreement with other studies (Kim *et al.*, 2015 and Jang *et al.*, 2015).

Many factors affect the volumetric polymerization shrinkage of resin composite. The filler content, flow and rate of modulus of the resin are the major factors (Chung *et al.*, 1990). The least shrinkage was presented for Venus bulk fill, this was in consistent with (Lee *et al.* 2008 and Rosatto *et al.*, 2015); which showed that the consistency of the composite is a crucial aspect for determining the axial shrinkage. The low shrinkage of Venus bulk fill could be explained by its very low viscoelastic properties in comparison to the other tested composites (Papadogiannis *et al.*, 2015), that would increase the radial shrinkage and subsequently reduce the axial shrinkage.

More over Venus Bulk Fill resin matrix is based on the low shrinkage [ethoxylated bisphenol A dimethacrylates monomer (EBPADMA)], which are more hydrophobic analogs of Bis-GMA, and reportedly exhibit higher DC and lower polymerization shrinkage than the standard Bis-GMA/TEGDMA resin systems (Jang *et al.*, 2015). These characteristics are mainly related to the more flexible structure, lower viscosity, higher molecular mass and lower content of diluent monomer needed for EBPADMA, compared to Bis-GMA monomer (Jang *et al.*, 2015).

Results of our study revealed that both XTra Fill and Sonic Fill followed Venus Bulk Fill in the polymerization shrinkage percent with insignificant difference between them. This might be related to the high filler content of XTra Fill (86wt %), thus reducing the amount of organic matrix so in turn reducing shrinkage (Chung *et al.* 1990). On the other hand, reduced polymerization shrinkage mechanism in Sonic Fill system was related to its low shrinkage (EBPADMA) resin polymers properties and its high filler content (83.5%). More over the sonic energy application lead to activation of rheological modifiers in the material's matrix, which drop the material viscosity up to 87%. This drop in the viscosity increases particle mobility in the early stages of polymerization. This increased mobility delays gelation of the material, and also enables greater stress relief via internal flow prior to gelation (Tiba *et al.*, 2013 and Benetti *et al.*, 2015)

Although Tetric EvoCeram Bulk Fill resin composite revealed the highest polymerization shrinkage compared to the other tested bulk fill materials, it showed lower shrinkage than the conventional resin composite. This could be explained due to its pre-polymerized filler particles functionalized with silane, that seems to have relatively low elastic modulus (~110 GPa), causing it to act like a microscopic spring, attenuating the forces of shrinkage stress (Nagi *et al.*, 2015).

Conclusion

Under the limitations of this in vitro study, the following conclusions could be made:

- Utilizing low-shrinkage resin monomers (e.g; EBPADMA) in bulk fill resin composites seem to be beneficial for reducing polymerization volumetric shrinkage with enhancement in the degree of conversion.
- Increased filler loading in bulk fill composites had positive effect on reducing polymerization volumetric shrinkage, while its effect on the degree of conversion was material dependent.

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