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ORIGINAL RESEARCH

Degree of Conversion of Bulk Fill Composites at Different Depths

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ABSTRACT

Aim: The aim of this study was to evaluate the degree of monomer conversion of bulk fill composites at different depths.

Materials and methods: The bulk fill products tested were SDR™, SonicFill™ System, Venus® Bulk Fill, and Tetric EvoCeram® Bulk Fill. In addition, this study tested conventional composites that have been indicated for use in association with bulk fill composites (Venus® and Tetric EvoCeram®), as well as the Ceram X™ conventional composite. To analyze the degree of conversion (DC), specimens (n = 10) consisting of four individual portions through the depths of cure (1, 2, 3, and 4 mm) were created. These samples were ground, pressed with potassium bromine, and analyzed using Fourier-transform infrared (FT-IR) spectroscopy. Two-way analysis of variance (ANOVA) and Bonferroni *post hoc* methods were applied to the results ($p < 0.05$).

Results: Venus® Bulk Fill exhibited the best results at all of the depths tested. None of the bulk fill composites considered herein exhibited any significant differences between the depths. Conventional composites were found to have significantly lower DCs at greater depths.

Conclusion: The results show that when depth of cure is being considered, bulk fill composites may be used at thicknesses of up to 4 mm.

Clinical significance: Bulk fill composites are indicated to be inserted in increments of up to 4 mm. In this sense, it is fundamental to determine DC in these depths to ensure obtaining adequate mechanical properties.

Keywords: Composite resin, Degree of conversion, Fourier-transform infrared spectroscopy.

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INTRODUCTION

Since the introduction of the acid conditioning technique by Buonocore in 1955 and as a result of the advancements obtained with composite resins, new perspectives have formed regarding cosmetic and functional dental restorations, as well as the preservation of dental tissue.[1] In this context, efforts have been focused on developing composites with less shrinkage and less shrinkage stress. In addition to load quantity, changes in monomer structure and in polymerization reaction dynamics are the most promising approaches.[2,3]

Composites mainly consist of mineral loads dispersed in an organic resin matrix and an initiator system.[4] The resin matrix consists of a multifunctional long-chain monomer which, when polymerized, results in a three-dimensional network of crossed bonds known as polymers. The bisphenol A glycidyl methacrylate monomer (Bis-GMA) is the most commonly used monomer in the development of dental composite resins. It consists of one long, rigid molecule with double carbon–carbon bonds at both ends.[5] The extent of the reaction in which the monomers form a polymer is understood as the DC, which corresponds to the measurement of the percent of double carbon—carbon bonds in the polymerization process.[5]

Since the introduction of photoactivated composite resins, curing quality and its consequences have become a significant focus for researchers. The adequate polymerization of resin materials is a fundamental factor in the development of good physical properties and improved clinical performance in cases of restoration.[6,7] The DC is a codeterminant of the properties of restorative resins.[8] Numerous studies have found that many of the physical and chemical properties of composite resins, including hardness, wear, compressive resistance, flexural strength, dimensional stability, solubility, and the extent of discoloration and degradation reactions, all depend on the

degree of polymerization and the organic components involved.[9,10] Other studies have also found that the release of unreacted monomers that remain in the material[11,12] may stimulate the growth of bacteria around the restoration site[13,14] and cause allergic reactions in some patients.[15]

The existing options for controlling shrinkage stress include the incremental technique, in which the professional fills the cavity with 2-mm composite increments and photopolymerizes them individually.[16] Meanwhile, the bulk fill insertion technique involves the insertion of large volumes of the material (4 mm thick). Two important factors associated with the incremental insertion technique are the chance of trapping voids forming between the increments and the clinical time required for the technique to be performed. In the case of class II caries, many increments are necessary and must be polymerized individually, which results in a considerable amount of insertion and polymerization time. Thus, there is substantial market pressure to develop materials and techniques to reduce the clinical time required for the procedure.[17] Even when these factors are considered, the incremental technique may be useful for ensuring adequate polymerization of the material in deeper regions.[18]

Many different materials have been released in recent years to reduce the clinical time required to perform direct adhesive restorations of posterior teeth.[3,19] According to their manufacturers, these materials share the property of allowing light to pass through even when greater thicknesses are used (4–5 mm) without negatively affecting polymerization shrinkage, adaptation to the cavity walls, or the DC. It is clear that, in addition to the DC of a given material, its depth of cure must also be determined.

Inside of these factors, this study sought to evaluate the degree of monomer conversion of bulk fill composites at different depths.

MATERIALS AND METHODS

This study tested the materials as described in Table 1.

For each material, 10 cylindrical specimens (4 mm in diameter × 4 mm in height) were created using the appropriate matrix. Each sample was divided into four 1-mm-thick discs representing the respective polymerization depths (1–4 mm) starting from the top of the sample (surface facing the light). The samples were created from the method as shown in Figure 1. Four 1-mm-thick metal matrices, each with a central orifice 4 mm in diameter, were positioned on a metal base so that each could be filled with the composite and each base could have an individual polyester slide. After the matrices were completely filled with the material, another polyester slide was placed on the top surface and was itself topped with a glass coverslip. After slight pressure to allow for the excess composite to drain off and the surface material to become homogenized, the tip of the light curing (Bluephase, Ivoclar Vivadent, Schaan, Germany) was positioned, and the composite was photoactivated for 20 seconds.

Table 1: Experimental groups

Groups	*Material*	*Manufacturer*
I	SDR™	DENTSPLY DeTrey GmbH, Konstanz, Baden-Württemberg, Germany
II	SonicFill™ System	Kerr Corporation, Orange, California, USA
III	Venus® Bulk Fill	Heraeus Kulzer GmbH, Hanau, Germany
IV	Venus®	Heraeus Kulzer GmbH, Hanau, Hesse, Germany
V	Tetric EvoCeram® Bulk Fill	Ivoclar Vivadent AG, Schaan, Liechtenstein, Germany
VI	Tetric EvoCeram®	Ivoclar Vivadent AG, Schaan, Liechtenstein, Germany
VII	Ceram.X™	DENTSPLY DeTrey GmbH, Konstanz, Baden-Württemberg, Germany

After the photopolymerization, the samples were kept in dry storage in an incubator kept at 37°C. They were uniquely identified containers in which they were

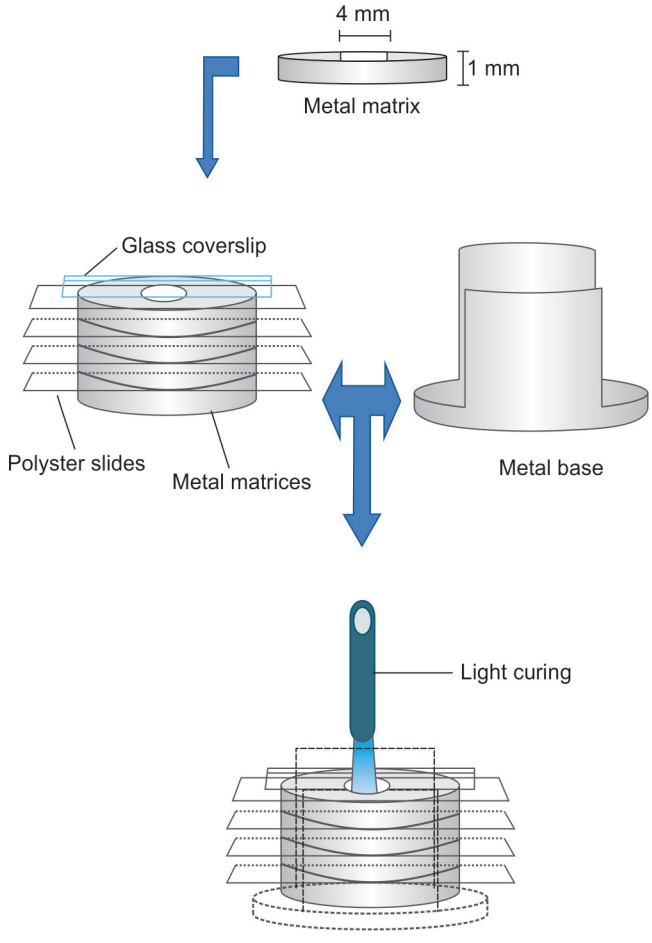

Fig. 1: Representation of the method used to create sample molds

protected from light. The samples were created after 24 hours, and they were ground into a fine powder in an agate mortar and pestle. Next, 5 mg of the ground material was mixed with 100 mg of potassium bromide, or KBr (Merck KGaA, Darmstadt, Germany). After homogenization, the powder obtained was pressed under 8 tons load for 1 minute to obtain the pastille, which was evaluated using FT-IR spectrometry.[20] The pastille samples were 13 mm in diameter and 1 mm thick. Five uncured resin samples were created, and a small portion of the resin was mixed with 100 mg of KBr for subsequent evaluation.

To obtain spectra in the IR range, the FT-IR spectrometry technique was used (Nexus-470) equipped with a triglycine sulfate detector in the 4000–300 cm^{-1} range using the diffuse reflectance accessory at a resolution of 4 cm^{-1} coupled with a server microcomputer to collect 32 scans. The spectra were obtained using the transmission technique and by monitoring the peaks in absorbance.

After the absorbance peaks were obtained, the percentage of unformed double carbon–carbon bonds (%C=C) was determined based on the rate of absorbance intensity between the C=C bonds at 1.637 cm^{-1} and those at 1.610 cm^{-1} before and after polymerization. The results were analyzed using two-way ANOVA and Bonferroni test at a 5% level of significance.

RESULTS

The DC results, as well as the comparisons between the groups are presented in Table 2.

DISCUSSION

When it comes to composites, knowledge on the DC is fundamental for determining their biocompatibility and mechanical properties.[21] Properties, such as the modulus of elasticity, hardness, and solubility are directly associated with DC.[7,22] The current study sought to determine the DC of three different composites at different depths of cure. The results clearly show that the composites recommended for bulk fill insertion (SDR, SonicFill, Venus Bulk Fill, and Tetric EvoCeram Bulk Fill) did not differ when the DC was compared at different depths. The same result was not produced by the conventional composites. These results show that the bulk fill composites used in this study have the capacity to enable adequate polymerization, even when the material is inserted at a large thickness. Venus Bulk Fill exhibited the highest DC values at all of the depths tested. While a higher DC is ideal for restorative composites, other properties of the materials were not evaluated. Therefore, it is not possible to conclude that the material with the highest DC necessarily exhibits the best mechanical properties.

All of the conventional composites used in this study exhibited some type of significant compromise to the DC when the thickness was increased. It is important to note that, even at a thickness of 2 mm, the Venus composite exhibited a significant decrease in the DC relative to the 1-mm thickness. This phenomenon also occurred in the case of the Ceram.X™ at 3 mm and in the case of Tetric EvoCeram at 4 mm. It is clear that the conclusion, i.e., the composites must be polymerized in increments with a maximum thickness of 2 mm, is not true for all of the materials, at least when a time of 20 seconds was used. It is evident that the light source is a determining factor for obtaining an adequate DC. In this study, the light-emitting diode curing light device was used at an intensity of 1200 mW/cm^2. This value is considered adequate for light curing dental composites.

It should be clarified that there are differences in the compositions of the different materials used in this study. Furthermore, the increased concentration of monomers

Table 2: Results of the DC (%) (± standard deviation) for the different resin materials at different depths (1–4 mm)

	1 mm	*2 mm*	*3 mm*	*4 mm*
SDR™	41.12 Ca	52.58 Ba	47.81 Ba	50.40 Ba
	(± 20.37)	(± 12.84)	(± 13.02)	(± 12.68)
SonicFill™ System	42.69 Ca	31.89 Ca	40.41 Ba	37.06 CDa
	(± 11.19)	(± 11.88)	(± 11.50)	(± 17.37)
Venus®	73.51 Aa	69.97 Aa	66.41 Aa	67.58 Aa
Bulk Fill	(± 7.58)	(± 6.49)	(± 6.25)	(± 6.32)
Venus®	57.73 Ba	43.31 BCb	26.88 Cc	7.51 Ed
	(± 5.18)	(± 20.61)	(± 24.58)	(± 23.44)
Tetric	49.08 BCa	51.56 Ba	45.13 Ba	42.44 BCa
EvoCeram® Bulk Fill	(± 3.59)	(± 5.02)	(± 4.18)	(± 10.23)
Tetric	53.04 BCa	51.04 Ba	43.06 Ba	28.25 Db
EvoCeram®	(± 4.74)	(± 5.67)	(± 3.84)	(± 4.39)
Ceram.X™	50.93 BCa	47.26 Ba	40.72 Bab	29.75 CDb
	(± 8.66)	(± 8.02)	(± 4.62)	(± 7.86)

The same lowercase letters on a given row represent statistical similarity; the same uppercase letters in a given column represent statistical similarity (two-way ANOVA and Bonferroni; $p < 0.05$)

and diluents is known to artificially increase the DC without improving the mechanical properties of the material. The SDR Bulk Fill composite has a polymerization modulator to reduce polymerization shrinkage stress. In theory, at least, this modulator is able to optimize the flexibility of the material.

Other studies have demonstrated adequate polymerization of bulk fill composites. In 2013, Czasch and Ilie[20] evaluated different bulk fill composites and found adequate mechanical properties and DCs at a thickness of 4 mm and at a photopolymerization time of 20 seconds, similar to results which were found with 2-mm increments and at a photopolymerization time of 40 seconds. Ilie et al[23] also found that bulk fill composites are adequately polymerized at 4-mm increments, an opinion shared by Finan et al[24] and El-Damanhoury and Platt.[25]

Bulk fill composites may be adequately polymerized at greater thicknesses, but the differences both in the chemical composition of the resin matrix and in the type of load particle significantly affect the DC and the mechanical properties of the materials. In the SDR composite, the organic matrix contains a urethane dimethacrylate (UDMA) with incorporated photoactive groups meant to control polymerization kinetics.[26] Meanwhile, the Tetric EvoCeram Bulk Fill has both a camphorquinone-amine initiator and Ivocerin, an intensifier capable of polymerizing the material at greater depths by absorbing light at a higher wave length range.[27] The study performed by Alrahlah et al[27] found that SonicFill and Tetric EvoCeram Bulk Fill exhibited greater depth of cure than Venus Bulk Fill. In the current study, the exact opposite was found: Venus Bulk Fill provided better results than the other two materials at a depth of 4 mm. It is important to note that, in the study by Alrahlah et al,[27] the methodology was based on an evaluation of microhardness to indirectly determine the DC. Once again, we question the methodology: When different products are considered, those with the highest microhardness do not necessarily provide the highest DC. This association is particularly important when the same material is evaluated at different depths.

The composites cured using monomers with high molecular weights, such as Bis-GMA and UDMA, have been found to have a considerable number of remaining double bonds.[28] The limited conversion that occurs in many network polymers is the result of the restricted mobility of radical chain ends, pendant methacrylate, and monomers imposed at a high crosslink density.[29] Photoactivated dental composites typically reach a DC ranging from 43 to 75%, a value which largely depends on composite composition, irradiation intensity, and exposure time. More specifically, the factors that affect the DC are the resin matrix, the diluents concentration, concentrations of photo indicators and amine, tone and translucency of the material, format and quantity of the inorganic particles, and refractive indices of the matrix and of the inorganic particles.

The differences in the DCs of the different materials may be attributed to variations in the chemical compositions of the resin matrices. The main features of monomers affecting the DC are the initial viscosity and the flexibility of its crystalline structure.[21] The SDR and Venus Bulk Fill composites have considerable quantities of UDMA, a viscous monomer that enables the polymerization reaction to be prolonged, thus increasing the degree of monomer conversion.[21] One of the reasons for the DC being maintained at greater bulk fill composite thicknesses is likely the translucency of these products, which itself is the result of a lower filler content and a larger particle size.[26]

Infrared spectroscopy offers direct approximation when evaluating the depth of cure of the photoactive composites by directly determining the quantity of monomers converted during the curing process. This technique determines the DC based on the amount of radiation absorbed in the IR frequency range based on the molecular vibrations of the functional groups contained in the polymer chains. The use of this method, when combined with the practice of measuring the mechanical properties as a way to indirectly evaluate the DCs of these materials, has proven to be effective in characterizing the behavior of these materials in dental work.

While the substantial benefits of reducing clinical time using bulk fill composites are widely known, it is also important to know whether the use of greater thicknesses results in undesirable consequences for margin quality and stress on the dental structures. Some authors have demonstrated adequate margin sealing in restorations performed using bulk fill composites, even after thermomechanical loading cycles.[17,30]

CONCLUSION

Bulk fill composites are adequately cured at depths of up to 4 mm. Further research should be performed to determine whether stresses are placed on these materials, on the adhesive interface, or on the adjacent dental structures.

Bulk fill composites are indicated to be inserted in increments of up to 4 mm. In this sense, it is fundamental to determine the DC in these depths to ensure the obtaining of adequate mechanical properties.

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