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Composite Warming Effects on the Mechanical Properties of Bulk Fills
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Composite Warming Effects on Mechanical Properties of Bulk Fills

Clinical Relevance

Heating composite may significantly increase the mechanical strength of bulk fill composite resins.

Summary

New bulk-fill restorative composites have surged into the market. The purpose of this study was to assess the effect pre-warming had on the mechanical properties (flexural strength/modulus, Knoop hardness [KH], polymerization shrinkage), of 5 new bulk-fill composites (Aura Bulk Fill, SDI, Bayswater, Victoria, AU; Fill-Up!, Coltene; Filtek One, 3M ESPE St Paul, MN, USA; SDR Flow+, Dentsply Sirona, ;Tetric Evo Ceram Bulk Fill, Ivoclar Vivadent;) to an incremental nanocomposite (Filtek Supreme Ultra, 3M ESPE). Flexural strength/modulus was determined accomplished according to ISO 4049, composite specimens were created both from room temperature (22C) [RT] and pre-warmed (52C) [PW] composites, stored for 24hrs and tested to failure on a universal testing machine. For Knoop hardness testing, RT and PW composites were placed in cylinder molds, light cured, stored for 24 hrs, and KH performed on irradiated top and non-irradiated bottom. Polymerization shrinkage utilized a video-imaging device to record the shrinkage of RT and PW composites after light curing. Statistical Analysis: Kruskal-Wallis with Dunns Post-hoc ($\alpha=0.05$)

Warmed composite showed significantly greater flexural strength/modulus, and hardness for all materials. Irrespective of temperature, the incremental fill composite demonstrated greater flexural strength/modulus, micro-hardness ratio compared to bulk-fill composites. Knoop hardness ratio using bottom-to-top hardness ratio was less than 80% for bulk fill composites; materials were tested at manufacture advised depth of cure.

Intro

Composite resin is a restorative material designed to replicate the esthetics and function of natural tooth. With the high esthetic demand on dentistry, composite resin has become the restorative material of choice for most patients. The basic formulation of composite resin is methacrylate monomeric mixtures, inorganic fillers coated with a silane coupling agent, and a photo initiator system.

There has been several attempts to improve the basic formula of the composite resins and most of the changes have been related to the inorganic filler. Strides have been made with the reduction of inorganic filler partial size and material type that has led to composite resin with improved wear resistance, and polishability¹.

Although improvement of inorganic filler particles has led to composite resins with improved mechanical properties. Changes in the organic matrix has led to concerns for the degree of conversion of monomer to polymer, and the amount of polymerization shrinkage, both which can decrease the clinical performance of the composite resin. The traditional 2mm depth incremental fill technique has been utilized to combat the stress caused by polymerization shrinkage and to ensure adequate polymerization. With each increment utilizing a light irradiation of 10-40 secs. In large posterior restorations this technique is very time consuming for the operator and patient. Longer procedure times increase risk for water contamination, and potential introduction of voids.

Clinical practitioners are opting for materials and techniques that decrease time enabling clinical efficiency. Bulk fill composites were introduced to with the claim depth of cure of 4-5mm, doubling the curing depths of the traditional incremental cure resin composites. Manufactures of bulk fill resin composites have claimed a reduction in restorative time, while still maintaining clinically acceptable degree of conversion, polymerization shrinkage, and mechanical properties. Bulk fill composites have advanced technology that help facilitate the increased mechanical properties to include, increased transparency, addition-fragmentation monomer (AFM), additional photoinitiators and improved refractive indices of the monomer and filler materials for greater depth of cure. The Draenert study revealed the material class of bulk-fill RBCs revealed similar flexural strength values when compared to the class of nanohybrid and microhybrid RBCs, and significantly higher values when compared to the class of flowable RBCs.²

Past studies have shown that there is benefit in increasing the flow rate of composite resins and degree of conversion by increasing the temperature of the composite before placement, and light cure.² The increase in temperature of a resin

composite is expected to exhibit enhanced flow due to the higher thermal energy that allows molecular motion, resulting in greater flow.³

Limited research has been published evaluating the effects of increasing the temperature of the bulk fill resin composite has on the mechanical properties. The objective of this study is to assess the effect prewarming has on the mechanical properties of bulk-fill composite resins.

Materials & Methods

The restorative materials used in this study were comprised of 5 bulk fill composite resin products, and one incremental fill composite resin (listed in Table 1). All tests were evaluated at room temperature (22C) and composite resin warmed (52C) for 10 minutes using a composite warmer (Therma-Flo, Vita, Racine, WI, USA).

Flexural Strength

Twelve flexural strength/modulus specimens were fabricated using standardized (2x2x25mm), stainless-steel molds (Sabri Dental Enterprises, Downers Grove, IL, USA). Materials were placed into respective molds on a mylar-strip-covered glass slab with a second mylar strip placed on top of the filled mold. A glass microscope slide was then placed with digital pressure to form a uniformly flat surface with composite resin materials were polymerized using a light-emitting-diode (LED) visible-light-curing (VLC) unit 1200 +- mW/cm² (Bluephase G2, Ivoclar-Vivadent, Amherst, NY, USA) according to manufacturer's specifications in an overlapping fashion on both sides. The performance of the VLC unit was periodically assessed using a laser power meter (Bluephase Meter II, Ivoclar-Vivadent, Amherst, NY, USA). Specimens were then removed from their respective molds and refined as needed using 320 grit removing any flash material from the edges. Completed specimens were stored in 0.1M Phosphate Buffered Solution (37°C/98%RH) until time of testing. Testing occurred 24 hours after fabrication. Specimens were tested on a three point bend apparatus mounted on a universal testing machine (Criterion 43, MTS Corporation, Eden Prairie, MN, USA). Specimens were stressed using a cross head speed of 1.0mm/min until failure with the maximum force recorded. Flexure strength results was determined using the flexural strength formula. Modulus of Elasticity was determined by the slope of the linear portion of the flexure strength stress-strain curve. Mean results were determined and recorded.

Micro Hardness

Knoop hardness testing of the irradiated top and non-irradiated bottom surfaces on 4mm diameter specimens fabricated with heights based on manufactures advertised depth of cure (2mm,4mm,5mm, 6mm). Test were performed on composite specimens (room temperature/warmed) after 24hrs storage. Five Knoop Hardness (KH) indentations were made in an alternating fashion over the surface with >0.5 millimeter spacing between indentations using a pyramidal Knoop indenter (MicroMet 6040, Buehler Manufacturing, Inc., Lake Bluff, IL, USA) using a 300-gram load and a dwell time of 15 seconds. The length of the indentations were directly measured at 50X magnification. The mean of the five measurements was calculated as the representative mean hardness for each surface (top and bottom) per sample. A total of three specimens were tested for each material.

Polymerization Shrinkage

Twelve specimens of each composite both from room temperature and pre-heated were placed on a pedestal in a video-imaging device (AcuVol, Bisco, Schaumburg, IL, USA) at a distance of 10cm. The video camera digitized and analyzed the images with the provided image-processing software. The specimens were polymerized according to manufactures specifications using aforementioned light-curing unit. Polymerization shrinkage was recorded continuously for 10 minutes after the light initiation. The mean percent shrinkage and standard deviation was calculated for each restorative materials.

Statistical Analysis:

Twelve specimens (n=12) were created per group for flexural strength/modulus and volumetric polymerization shrinkage. Three specimens (n=3) were created per group for Knoop hardness testing.

Data was analyzed with a Krushkal-Wallis and Dunns Post-hoc test to determine the effect heating composite has on each individual property (alpha=0.05) using statistical software (IBM SPSS, version 24, Chicago IL, USA).

Results

Table 1 – Flexural Strength/Modulus and Volumetric Shrinkage

Materials	Temp	Flexural Strength - MPa (SD)	Flexural Modulus - Gpa (SD)	Volumetric Shrinkage - % (SD)
aura bulk fill	22°C	77.066 (5.412) A	5.701(0.259)A	2.063 (1.890) AB
	52°C	81.810(7.021) AB	6.067 (0.521) AB	2.108 (0.389) BC
Fill-Up!	22°C	104.08 (10.070) C	5.872 (0.577) A	5.173 (0.417) D
Filtek One Bulk Fill	22°C	83.388 (9.701) AB	5.9128 (0.536) A	2.053 (0.250) A
	52°C	124.955 (9.840) D	7.474 (0.582) C	2.145 (0.124) BC
Filtek Supreme Ultra	22°C	120.73 (18.907) D	8.575 (0.440)	2.156 (0.151) AB
	52°C	139.541 (9.721)	9.708 (0.319)	2.274 (0.178) ABC
SDR Flow+ Bulk	22°C	69.529 (8.739)	3.133(0.362)	5.412 (0.327) D
	52°C	85.538 (8.080) B	3.987 (0.390)	5.006 (0.774) D
Tetric Evo Ceram Bulk Fill	22°C	86.581 (8.750) B	6.495(0.430) B	2.391 (0.328) BC
	52°C	105.881 (9.302) C	7.643 (0.328) C	2.683 (0.303) C

n=12; Kruskal-Wallis with step-wise comparison test (α=0.05); Uppercase letters indicate a statistical similar subsets by column

Table 2 – Knoop Hardness

Material	Knoop Hardness		Top			Bottom			Ratio
	Height	Temp	KH	SD	KH	SD	B/T		
aura Bulk Fill	2mm	22°C	38.3	I 3.2	38.5	CDE 2.9	1.0		
	2mm	52°C	40.3	EFG 3.0	39.3	CD 2.4	1.0		
	5mm	22°C	38.7	HI 3.0	21.1	J 1.0	0.5		
	5mm	52°C	40.2	GHI 4.7	22.3	J 4.9	0.5		
Fill-Up!	2mm	22°C	45.2	EFG 2.1	31.0	EFGH 4.4	0.7		
	4mm	22°C	46.1	EF 2.5	31.3	EFGH 4.2	0.7		
	6mm	22°C	45.5	EF 0.9	32.3	CDEFGH 5.3	0.7		
Filtek One Bulk Fill	2mm	22°C	52.7	D 2.3	52.3	B 5.2	1.0		
	2mm	52°C	48.1	DEF 3.3	46.5	B 4.4	1.0		
	4mm	22°C	56.0	EF 1.6	40.6	BCD 6.4	0.7		
	4mm	52°C	57.4	C 2.9	42.7	BC 1.8	0.7		
Filtek Supreme Ultra	2mm	22°C	76.2	A 2.1	66.7	A 3.9	0.9		
	2mm	52°C	73.1	A 1.7	67.7	A 1.5	0.9		
	4mm	22°C	74.1	A 2.8	36.4	CDEFG 4.9	0.5		
	4mm	52°C	66.0	B 4.9	29.6	FGHI 4.2	0.4		
SDR Flow+ Bulk	2mm	22°C	32.0	J 2.2	29.7	FGHI 3.2	0.9		
	2mm	52°C	30.1	K 1.6	29.3	HI 1.1	1.0		
	4mm	22°C	42.8	FGH 1.5	28.9	GHI 3.0	0.7		
	4mm	52°C	32.0	J 0.7	23.8	J 1.3	0.7		
Tetric Evo Ceram Bulk Fill	2mm	22°C	41.7	FGHI 5.1	36.5	CDEFG 5.2	0.9		
	2mm	52°C	45.8	EF 3.6	39.8	CD 1.5	0.9		
	4mm	22°C	52.4	DC 2.6	23.7	J 4.3	0.5		
	4mm	52°C	50.5	EF 2.8	31.7	DEFGH 1.3	0.6		

n=5, KH for each specimen was an average of 3 measurements; Kruskal-Wallis with step-wise comparison test (α=0.05); Uppercase letters indicate a statistical similar subsets by column; Lowercase letters indicate a statistical similar subsets by row

The results are summarized in Table 1 and 2. Filtek Supreme Ultra heated (52°C), and Filtek One Bulk Fill heated (52°C) had the greatest flexural strength 139.541 ± 9.621 MPa and 124.955 ± 9.840 MPa respectively. These values were significantly greater (p=0.05) than all other bulk fill composite resins. The flexural modulus of Filtek Supreme Ultra incremental cure composite heated (9.708 ± 0.319 GPa) and room temperature (8.575 ± 8.575 GPa) was significantly greater than all bulk fill composites. Filtek Supreme Ultra heated and room temperature, Filtek One room temperature, and Aura bulk fill room temperature had the lowest polymerization shrinkage. SDR Flow the lone flowable bulk fill composite resin had the highest polymerization shrinkage, but no statistical significance in change in polymerization shrinkage when SDR was warmed.

In summary, heating composite resins significantly increases flexural strength and flexural modulus of Filtek One, Filtek Supreme Ultra, SDR Flow Bulk, and Tetric Evo Ceram Bulk Fill, without statistical significant increase in polymerization shrinkage. Heating has no effect on the top and bottom knoop hardness ratios of the composite resins tested. For all bulk fill composite resins examined the top and bottom ratios at the manufactures advertised depth of cure have a reduction of 30-50% compared to ratios at 2mm depth of cure.

Discussion

The purpose of this study was to investigate the effects preheating has on the mechanical properties of bulk fill composite resins. The extent of polymerization directly affects the physical and mechanical properties of composite resins. Pre-heating appears to improve flexural strength and hardness, without statistically significant increase in polymerization shrinkage. Daronch et al study concluded pre-heating composite prior to photoactivation provides greater conversion.² Gupta further explains free radicals and developing polymer chain become more fluid as temperature increases which leads to more complete polymerization and enhanced cross linking.⁴

Polymerization shrinkage has been a restorative challenge for resin based composites. The results of this study revealed no difference in polymerization shrinkage between pre-heated and room temperature composite resins. Filtek One heated exhibited the greatest flexural strength of the bulk fill composites while still maintaining a negligible increase in polymerization shrinkage. Filtek One utilizes addition fragmentation monomer technology. This monomer allows the central group to fragment and re-polymerize to relieve shrinkage stress.⁵

Bulk fill manufacturers advertise increased depth of cure (4-6mm) while still maintaining superior clinical properties. Top and bottom hardness ratios for all bulk fill composite resins were 70% or less. Tetric Evo, and Aura Bulk Fill at the advertised depth of cure of 5mm and 4mm respectively have top and bottom ratios 0.5, which is statistically significant difference from the top and bottom ratios seen for those materials at the traditional 2mm depth of cure. Past authors⁶ suggest bottom and top surface hardness of the specimens should be at least 0.8 or 80%. Noticeable visual differences could be seen at the top and bottom of all bulk fill composite specimens tested at manufacturers advertised depth of cure.

Bulk fill composites can be classified into two groups of base bulk fill composites, and full-body bulk fill composite resins. The base bulk fill composites have low viscosity, lower filler content, and require a final capping layer⁶. The full body bulk fill composites have higher filler loads, more wear resistant and are more viscous. Tetric Evo Ceram Bulk fill, Filtek One, and Aura Bulk fill all report inorganic fill load of 74% or greater by weight. These products noticeably required increased operator effort to dispense from the composite capsule during fabrication of room temperature fabrication. Heating these bulk fill composites decreased the viscosity of these composites allowing ease of placement into specimen molds.

Tetric Evo Ceram utilizes 0.10% Ivocerin, which is advertised as a highly reactive photoinitiator that allows deeper depth of cure⁷, which was not seen in this study. The absorption range of Ivocerin is 390-450 nm, which is slightly lower than the absorption range of champhorquinone which is 410-500nm. This study indirectly measures depth of cure by measuring surface hardness ratios (ISO 4049). Similar to this study Yap et al found Tetric EvoCeram depth of cure to be below 4mm using the 10 sec polymerization time. AlQahtani et al reported that 80% bottom-to-top hardness threshold could only be obtained when Tetric EvoCeram Bulk Fill was over exposed to polymerization for 40 secs.⁶

Multiple studies have shown inconsistent degree of conversion results based on study parameters.⁶ Van Ende et al examined the parameters that affect depth of cure, and concluded total light energy, transparency of mold used and location of hardness test conducted on specimens had the most influence on hardness test on results. Depth of cure was greater in the center of the specimen compared to the periphery, and depth of cure was significantly higher ($p>0.05$) for specimens prepared with a PMMA mold than an aluminum mold.⁶ Degree of conversion was reduced with metal molds.⁶ Incomplete conversion may result in leaching of unreacted monomers in the oral environment, which act as plasticizers decreasing mechanical strength. Further research is needed into the degree of conversion of bulk fill composites at the advertised depth of cure.

Conclusions

Incremental fill composite resin Filtek Supreme Ultra had significantly greater flexural strength and modulus, top and bottom Knoop hardness without a significant increase in polymerization shrinkage in comparison to all bulk fill composite resins. Comparison of bulk fill composites showed increased in mechanical properties, flexural strength and modulus of elasticity, when preheated. Based on the properties tested, there may be significant advantage to pre-warming bulk fill composite resins.

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