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Comparison of degree of conversion (DC) and depth of cure (DoC) of bulk fill composites and Filtek Supreme Ultra as the conventional

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In Partial Fulfillment of the Requirements for the Degree of

Master of Science in Oral Biology

By

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DEDICATION

To my husband, Euntak Lee, thanks for your encouragement, support, and love throughout the past two years. I couldn't have done it without you.

The author hereby certifies that the use of any copyrighted material in the thesis manuscript entitled:

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Abstract

Comparison of degree of conversion (DC) and depth of cure (DoC) of bulk fill composites and Filtek Supreme Ultra as the conventional

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Introduction: Resin based composites go through a process of polymerization. Degree of conversion (DC) is the percentage of methacrylate carbon double bonds that are converted to single bonds during the curing reaction and is a reflection of the degree of polymerization. A high DC correlates to improved mechanical properties, chemical stability, and longevity of the restoration.[7] Recently, manufacturers have developed bulk fill resin composite that can be placed in 4mm or 5mm increments.

Objective: The aim of this study is to evaluate the degree of conversion (DC) and depth of cure (DoC) for bulk fill composites in comparison with a traditional incremental composite.

Methods: Five bulk fill composite brands; Surefil SDR+ (SDRFP), Tetric Evoceram (TECB), Filtek 1 Bulk Fill (F1B), Venus Bulk Fill (VB), Sonicfill 3 (SF3); and 1 conventional resin composite Filtek Supreme Ultra (FSU) were investigated. Sample heights were 3mm and 5mm. The specimens were light cured for 20 seconds with irradiance of 2031$\pm$ 5mW/cm^2. A Fourier transform infrared attenuated total reflection (FTIR-ATR) Perkin Elmer Spectrum 400 spectrometer was used to measure DC. Real-time recording of the infrared (IR) spectrum began immediately after curing to 15 minutes and 24 hours after curing. In addition, DoC in accordance to ISO 4049:2000 for each material was tested. Mean DC and mean DoC values were compared using repeated measures of analysis of variances (ANOVA) and comparison of all pair using Tukey and Kramer HSD.

Results: Mean DC for the six materials with 5mm curing height after 24 hours were 79.5% for TECB, 75.7% for VB, 69.2% for SDRFP, 65.8% for SF3, 51.8% for F1B, and 44.0% for FSU. Mean depth of cure were 5.059mm for VB, 4.624mm for SDRFP, 3.803mm for F1B, 3.450mm for TECB, 2.966mm for FSU, 2.684mm for SF3. TECB and VB had the highest DC. DoC, VB had the highest while the newest product SF3 had significantly the lower DoC.

Conclusion: According to the DC findings of the bulk fill composites; VB, TECB, and SDRFP yielded higher percentages. The DoC findings showed that VB performed better than the manufacturer claims as well as had the greatest DoC compared to all the other bulk fill materials.

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Background

The setting reaction of RBC involves a polymerization process in which methacrylate monomers will interact with one another combining to form long-chain polymers. The polymerization process is activated by visible light and is fast initially. After a majority of the monomers have been converted, polymerization slows down. Therefore, most of the polymerization will occur in the first few minutes.[1] Furthermore, the conventional composite resins require curing in 2mm increments to allow sufficient light penetration[2]. For the sake of convenience and time savings, manufacturers have developed bulk fill RBCs that can be placed in 4mm or 5mm increments. This increase in increment size is accomplished by adding additional photo-initiators and increasing translucency.[3] Because of the bulk placement, technique sensitivity decreases. More importantly, bulk placement can prevent void formation and contamination between layers, establishing a more dense and consistent filling[19].

On the contrary, there are concerns with bulk fill RBC such as depth of cure. Furthermore, the higher surface area of the contracting material can cause more polymerization shrinkage. Incremental placement of RBC allows less polymerization shrinkage. This also means with bulk fill RBC, there are higher bonded surfaces, increasing configuration factor leading to possible tooth or restoration fracture.[29] With these concerns, manufacturers claim that their bulk fill RBCs have improved depth of cure and reduced polymerization shrinkage in order to ensure layers up to 4 to 5mm[19]. This is accomplished by increasing translucency and photoinitiators, as well as particles with low elastic modulus[21]. There have been much research done on bulk fill RBCs and have shown preferred mechanical properties. Nevertheless, there is a need for more research to be done for a better understanding of bulk fill RBCs especially the new products available for use.

Kerr has recently developed a new bulk fill composite, Sonicfill 3. Due to its higher viscosity, Sonicfill bulk fill composites require a sonic hand piece for placement into the tooth[4], which delivers sonic energy at varying intensities[5]. When sonic energy is applied through the hand-piece, the viscosity drops (up to 87%), during the composite insertion[5]. The lowered viscosity permits plastic flow for better adaptation to the prepped walls of the tooth. When the sonic energy is no longer present, the composite returns to a more viscous, non-slumping state that allows for carving and contouring[5]. Sonicfill has a manufacturer's claim of 5mm depth of cure[6]. In addition, other claims are better wall adaptability and low shrinkage stress[6].

An accurate measurement of the degree of polymerization is the degree of conversion (DC). This is defined as the percentage of carbon-carbon double bonds (- C = C -) converted to single bonds (- C - C -) to form a polymeric resin.[13] A high DC augments strength, hardness, dimensional stability, chemical stability, color stability, and longevity of the restoration.[7] The continuation of DC over time is described by polymerization kinetics. This provides information on the process of polymerization in addition to DC.[8] There is an initial light cure phase which DC increases rapidly after applying the

irradiation source[26]. Although the remaining free monomers have decreased movement, polymerization continues up to or beyond 24 hours after light cure[27]. This process of post-cure polymerization is also known as the dark cure phase[27]. The presence of free radicals and catalysts will continue the process of polymerization[10]. Al Adhal et al, found that XPosterior and Beautiful- Bulk Flowable significantly increased in DC at 24 hours post cure, while the DC of Sonicfill did not increase after 30 minutes post-cure[11]. An acceptable DC for modern RBCs is 55-75%[22]. In addition, unreacted monomers is cytotoxic to the pulpal tissues, therefore a reduced amount of free monomers of RBCs will increase biocompatibility[25].

Spectroscopy is a branch of science in which light or other electromagnetic radiation is resolved into its component wavelengths to produce spectra[9]. This method to study materials is useful because matter will interact with electromagnetic radiation to absorb or emit certain frequencies of radiation which are characteristic of the sample of matter[9]. The complexity of a certain spectrum is found in most infrared spectra[9]. For this study, real time Fourier transform infrared attenuated total reflection (FTIR-ATR) spectrometer was used to measure and compare DC of the bulk fill composite materials and the conventional composite material. IR spectra were taken in range $\lambda \sim 2.5$ - 15.0µm, which corresponds to 4000-650 cm^{-1}.[9] Baseline peak height at 1637 cm^{-1} (aliphatic carbon double bonds) and 1608 cm^{-1}(aromatic carbon double bonds) was measured and obtained from the FTIR spectra[8] (Fig. 1). Below is an example of a spectrum.

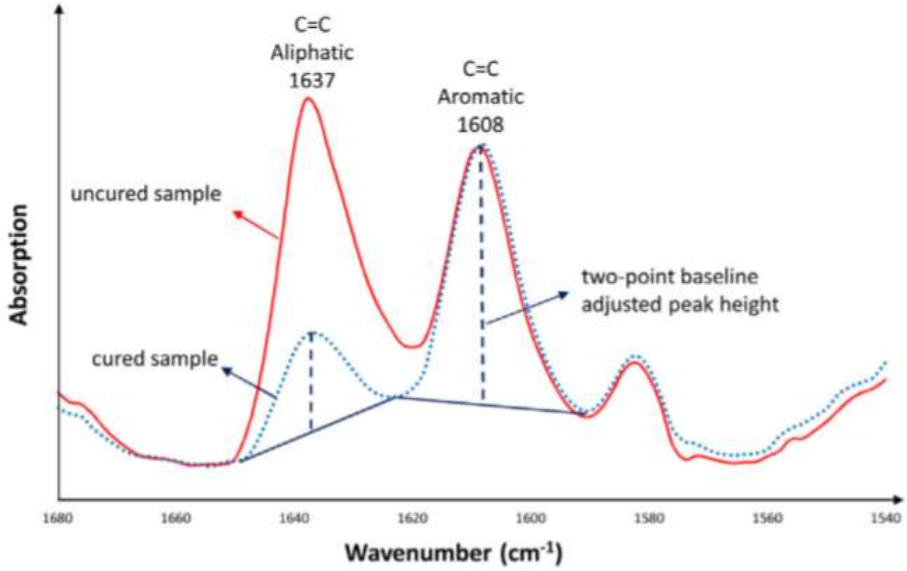

Fig. 1: FTIR spectra with the two-point baseline adjusted peak height at 1637 cm^{-1} and 1608 cm^{-1} for DC calculation[8].

In addition, depth of cure (DoC) was measured and compared to test the manufacturer claims of each product. DoC is the thickness of a light cured resin that can be converted from a monomer to a polymer when exposed to a light source under a specific set of conditions[13]. The greater the depth of the material is measured, the greater amount of light has gone through the RBC. Therefore, the higher depth of cure will reflect more polymerization.

With an increasing popularity of bulk fill resin composites due to its convenience, individual manufacturers are constantly evolving their products. Therefore, this research study demonstrated how well Sonicfill 3 performed compared to the other leading bulk-fill RBCs and the conventional RBC.

DC and DoC are critical measurements for RBCs. Therefore, inadequate polymerization of RBCs can diminish physical properties. Strength, hardness and solubility have been shown to be directly related to increased DC percentages as a result of the improvements of the bulk fill RBC brands[1].

The present study aims to evaluate DC over time and DoC among 5 bulk fill composites and 1 conventional composite. The null hypothesis were:

1.) There are no significant differences in DC and DoC amongst various commercial bulk fill resin based composite brands.
2.) There will be no significant difference in DC amongst various commercial bulk fill resin based composite brands at 24 hours post cure.

Methods and Materials

Degree of Conversion Testing

Five bulk fill composite brands including Sonicfill 3 and 1 conventional resin composite were evaluated. To measure the DC for each situation, a Fourier transform infrared attenuated total reflection (FTIR-ATR) Perkin Elmer Spectrum 400 spectrometer was used. Samples were made from polyvinylsiloxane (PVS) molds. Each PVS mold was 4mm in diameter and a height of 3mm or 5mm for each material. PVS mold was placed on the ATR crystal of the FTIR spectrometer and RBC material was placed into the mold. A mylar strip, then a microscope glass slide, and a metal disk to have a flat surface were placed on top of the mold. A force gauge that was part of the FTIR was placed over the metal disk to ensure the specimen was in full contact with the ATR crystal for an accurate scan. The uncured material was scanned by the FTIR. The microscope glass slide and the metal disk were removed. The specimen was light cured for 20 seconds with the 3M Paradigm curing ligh of exposure to $2031 \pm 5 mW/cm^2$ irradiances. Real-time recording of the infrared (IR) spectrum was set at 8 cm^{-1} resolutions wave number with 4 scan accumulation began immediately after curing, 20 seconds, 1 minute, 3 minutes, 5 minutes, 8 minutes, and 15 minutes after curing. Specimens were kept in room

temperature in a dark room to undergo dark curing for 24 hours, and 3 scans were taken at this time with the FTIR set at 8 cm^{-1} resolutions wave number with 16 scan accumulation. There were 2 sets for each height group. DC (%) was calculated by the following equation:

$$\text{Degree of Conversion (DC) \%} = [\ 1 - \frac{(1636\ cm^{-1}/1609 cm^{-1})}{(1636\ cm^{-1}/1609 cm^{-1})}\ ] \times 100$$

Wavenumber 1636 – Peak height of IR absorption of C=C of aliphatic bond stretch after curing

Wavenumber 1609 – Peak height of IR absorption of C=C of aromatic bond stretch after curing. [6,8]

Depth of Cure Testing

RBC specimens (n=3) were made for each bulk fill composite and the conventional resin composite according to the ISO-4049[14]. The height of each specimen was obtained by the manufacturer's recommendation of increment doubled plus 2[14]. For example, Sonicfill 3 has a manufacturer claim of 5mm, therefore 5mm doubled is 10mm plus 2 makes the specimen height 12mm. Once a proper stainless steel mold was selected and placed onto a Mylar strip, it was filled with the tested RBC material. After the material was condensed and the excess material was removed, the material was light cured with a 3M Paradigm curing light for 20 seconds exposure to 2031 ± 5 mW/cm^2 irradiances at the exit aperture of the stainless steel mold. The cured composite specimen was removed from the SS mold and the uncured material was scraped off with a plastic instrument. Each specimen height was measured and then divided by 2 to obtain the DoC. In addition, each specimen was labelled at the top part with the letter "T" and the bottom part with the letter "B". T and B of each specimen were scanned three times each with the FTIR set at 8 cm^{-1} resolutions wave number with 16 scan accumulation.

Statistics and Data Analysis:
Mean DC and mean DoC values were compared using repeated measures of analysis of variances (ANOVA) and comparison of all pair using Tukey and Kramer HSD. Therefore, data was analyzed with nonlinear regression and ANOVA/Tukey ($\alpha = 0.05$).

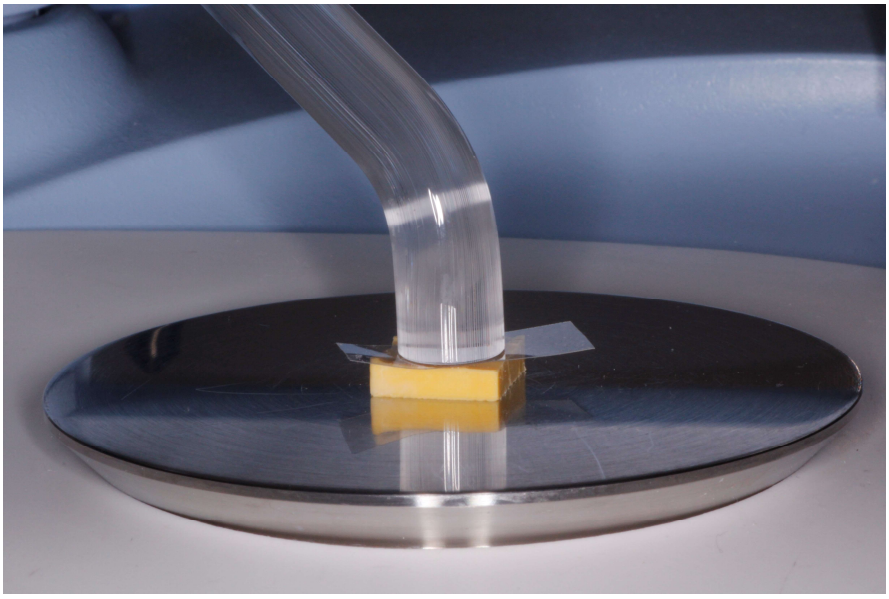

Figure 2. Light curing of specimen placed on top of the crystal of the FTIR.

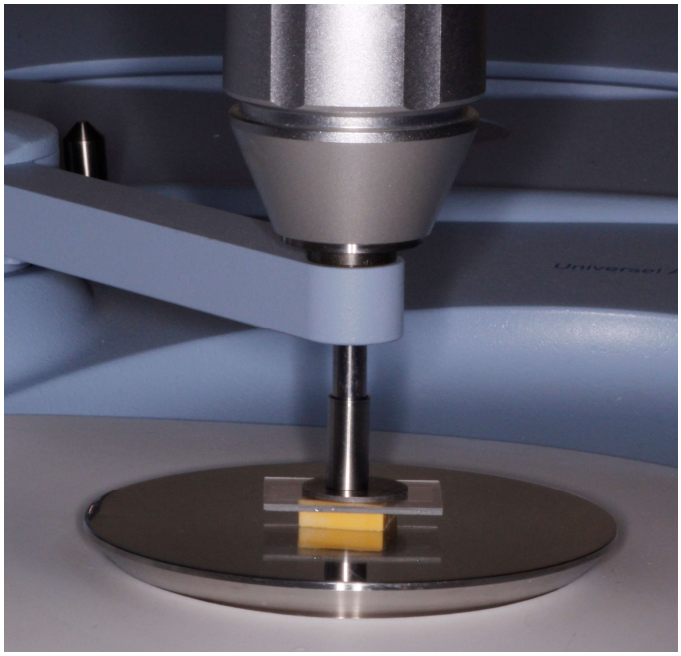

Figure 3. Uncured sample being scanned by the FTIR with the force gauge.

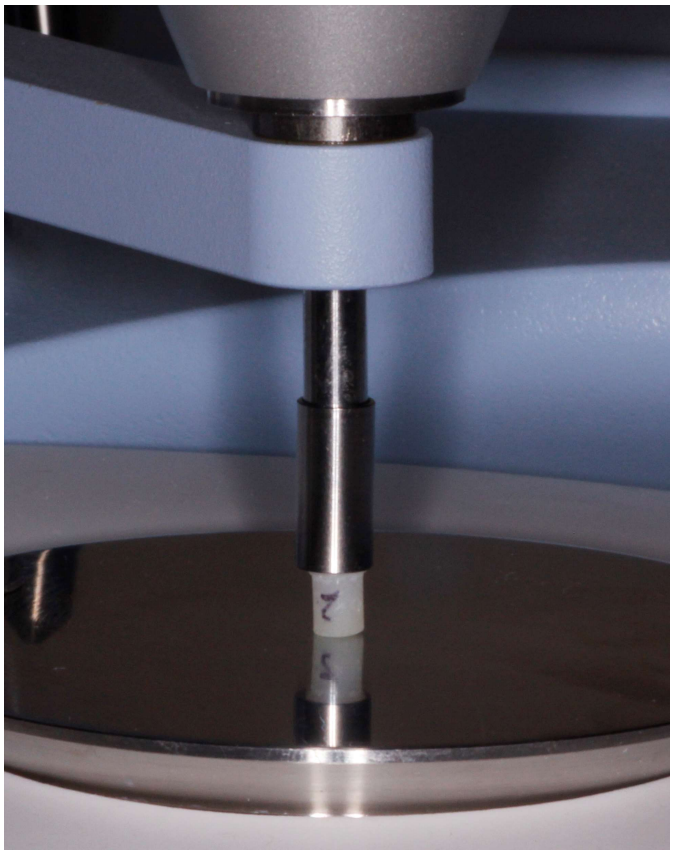

Figure 4. Sample being read at 24 hours.

Results

Figure 5 represents the DC of the specimens at 5mm height for 3 minutes time frame. It is plotted via 20 seconds of exposure to 2031 $\pm$ 5mW/cm^2 irradiances. The origin corresponds to the beginning of dark cure at which the light curing unit (LCU) is turned off. The time point, -20 seconds, represents LCU is turning on, and curing begins. As expected, the majority of the increase in DC was during the 20 seconds of the curing time. Venus Bulk Flow (VB) followed by Sonicfill 3 (SF3) demonstrated the higher DC at 5mm height. Surefil SDR+ (SDRFP), Filtek 1 Bulk Fill (F1B), and Tetric EvoCeram Bulk (TECB) were notably lower in DC. As expected, Filtek Supreme Ultra (FSU) has the lowest DC since it is the conventional composite that was used as the control. The DC at up to 3 minutes are shown in Figure 6 for 3mm height specimens. Among all bulk fill RBCs, Sonicfill 3 showed the highest DC. At 3mm of height, the other bulk fill RBCs displayed a lower DC pattern than Sonicfill 3. Filtek Supreme Ultra had DC pattern only slightly lower than the other bulk fill RBCs.

Figure 7 shows the DC at 24 hour post cure. The cylindrical height of each resin-composite sample for which the curing light travels through was either 3mm or 5mm thick. This defines the amount of DC that occured during dark curing. At 5mm, Tetric Evoceram Bulk Fill and Venus Bulk Flow demonstrated the most pronounced increase in DC at 24 hour post cure. Sonicfill 3 and Venus Bulk Flow had the highest DC at 3mm

height at 24 hour post cure. Each RBC had different percentages of DC at the 24 hour time point at 3mm and 5mm heights. The percent differences for each material for 3mm and 5mm DC are as follows: FSU 37.5%, F1B 34.4%, SF3 22.5%, SDRFP 11.1%, VB 9.4%, TECB 2.6%.

DoC of all the materials are presented in Figure 8. Venus Bulk Flow demonstrated the highest DoC of 5.059mm whereas Sonicfill 3 had the lowest DoC of 2.684mm. It was lower than Filtek Supreme Ultra which had a DoC of 2.966mm.

Statistical Analysis revealed a significant difference between certain RBCs tested. According to Figure 7, columns not connected by the same letters are significantly different for both 5mm and 3mm height analysis at the 24 hour post cure. For the depth of cure statistical analysis, all materials were significantly different according to Figure 8. The manufacturer claim for the DoC of Sonicfill 3 is 5mm, but this study showed the depth of cure of Sonicfill 3 is 2.684mm which equates to a 36-46% discrepancy between the manufacturer claim and the measured value.

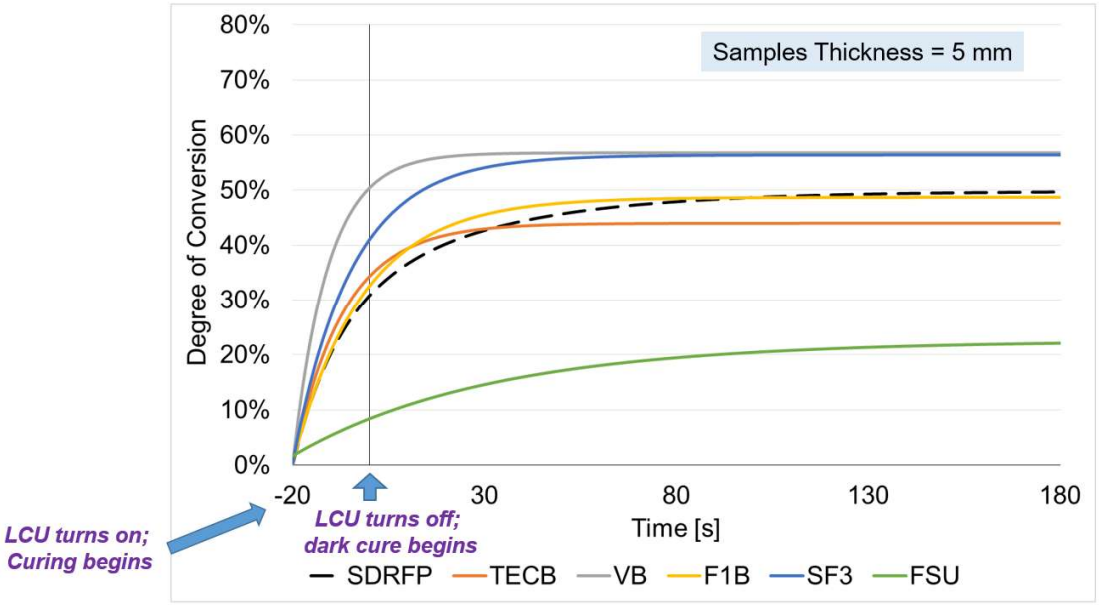

Figure 5. Degree of conversion as a function of time for various 5mm bulk-fill RBCs.

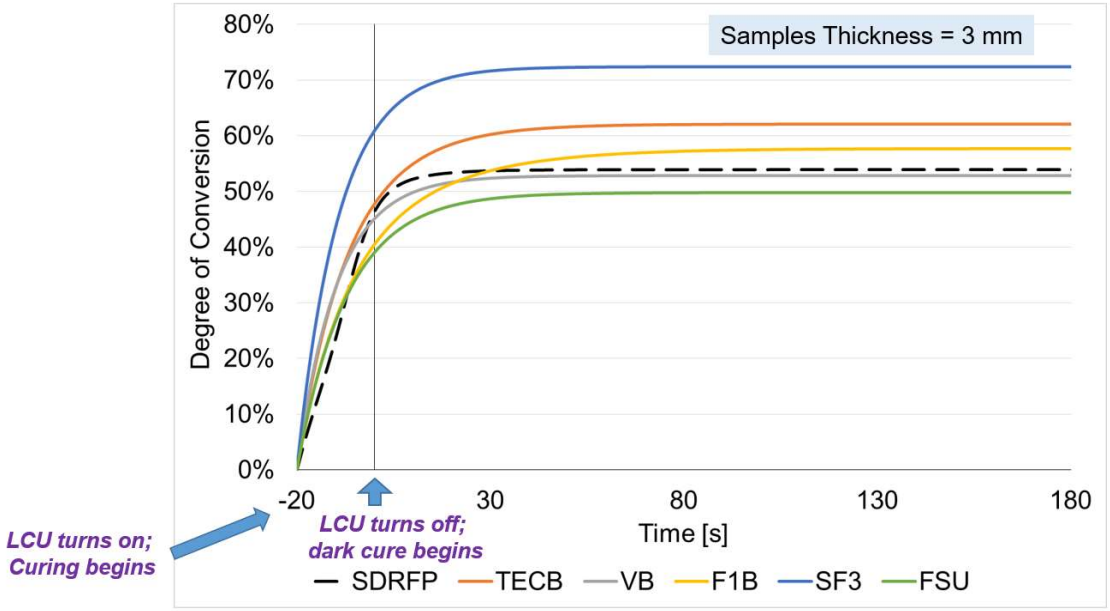

Figure 6. Degree of conversion as a function of time for various 3mm bulk-fill RBCs.

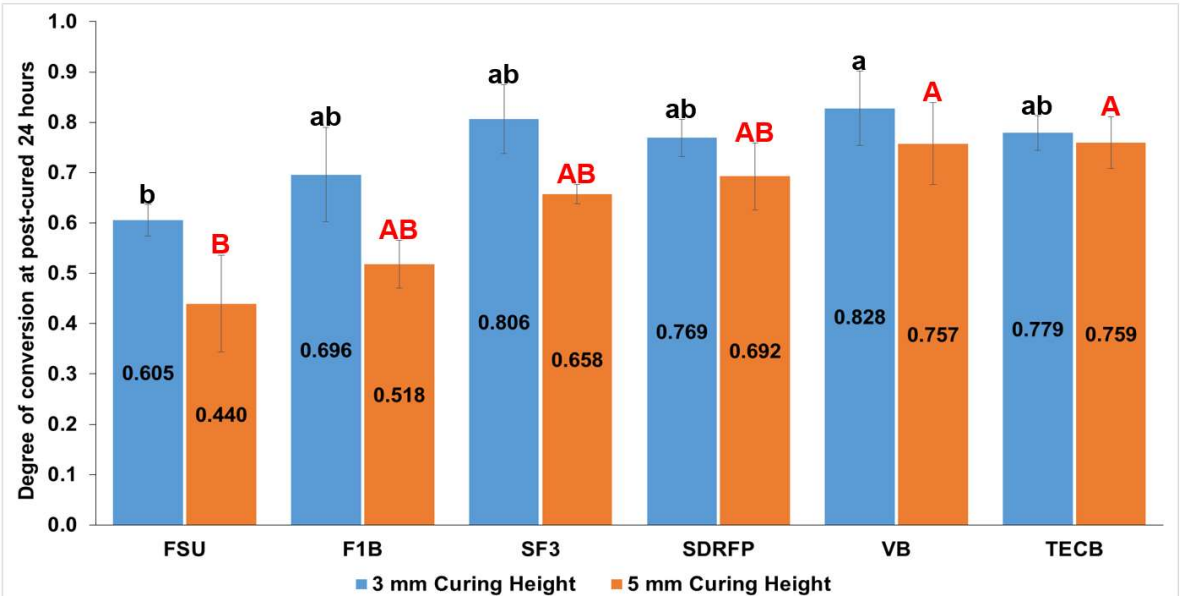

Figure 7. Degrees of conversion of various bulk-fill resin-based composites are plotted, after 24 hours of dark cure. The same case letters across column are not significantly different than each other ($p>0.05$).

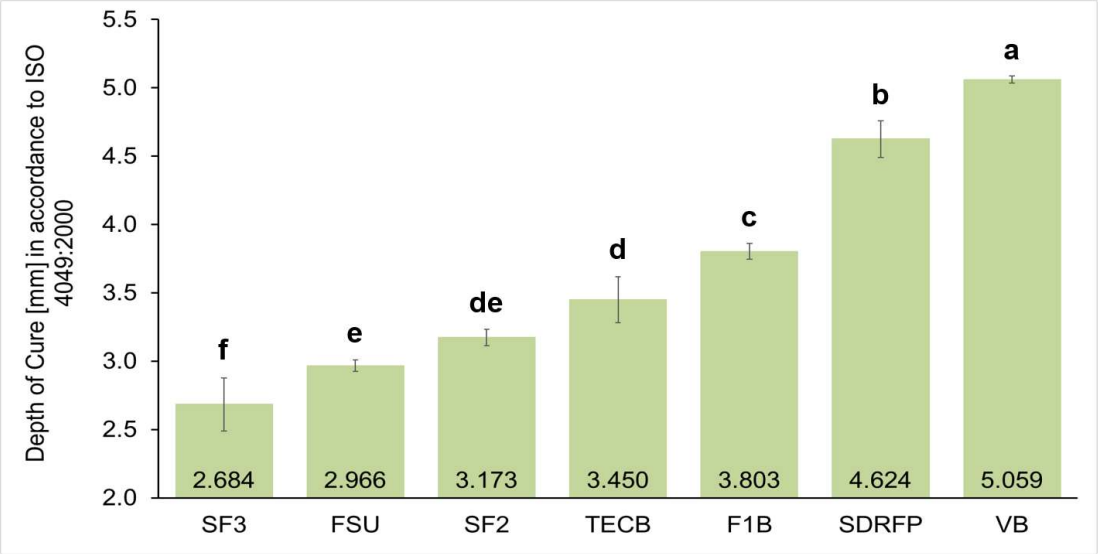

Figure 8. Depths of Cure of various bulk-fill resin-based composites using the ISO 4049:2000 method, via 20 s exposure to 2031 ± 5 mW/cm^2 irradiances, are plotted. The same case letters across columns are not significantly different than each other ($p > 0.05$).

Discussion

The present study was performed to determine DC and DoC of the selected bulk fill composites. Sonicfill 3, the most recently developed bulk fill material of the selected materials, and Filtek Supreme Ultra, the control conventional composite. Observations were made with the difference of DC and DoC according to RBC type and the varying bulk fill RBC brands that were tested. Within this study, our null hypothesis were rejected since:

1.) There were significant differences in DC and DoC amongst various commercial bulk fill resin based composite brands.
2.) There were significant differences in DC amongst various commercial bulk fill resin based composite brands at 24 hours post cure.

DC Immediately Post Cure and 24 hours Post Cure

For all of the bulk fill resin based composites, a steep uphill curve is seen during the 20 seconds of light curing. Once the dark cure begins, the linear curve flattens only changing in DC at minimal increments. However, a significant increase in DC have been seen from the 15minute time point post cure to the 24 hours post cure seen in most bulk fill RBCs and the conventional RBC. In addition, post cure polymerization is more amplified in specimens with initially lower DC. This allows for more movement in the unreacted monomer to form a polymer network.[18] Therefore for materials with lower DC immediately after light curing, 24 hours post cure yielded higher DC readings. This was apparent for Tetric EvoCeram Bulk Fill. At 5mm, the average DC immediately post cure was 35.9% and at 24 hour post cure DC was 75.9%. In addition, the manufacturer

recommendations for the incremental thickness is 4mm. However, contradicting the finding of a previous study, Venus Bulk Flow started with a high DC immediately after light cure (51.2%) and still had a high DC 24 hour post cure (75.7%), categorizing this material with the same group of TECB. Moreover, Filtek 1 Bulk Fill had an immediate post cure DC of 33.8% and a 24 hour post cure DC of 51.8%. Therefore, Filtek 1 Bulk Fill had a lower DC than the rest of the bulk fill resin based composite. Sonifill 3, which is the newest product tested, is in the same group as Filtek 1 Bulk fill. SF3 only had a slightly higher DC values for the immediately post cure and 24 hour post cure, 43.3% and 65.8% respectively.

According previous studies done by Daughtery et al and Par et al, TECB performed very well in their studies and yielded the highest 24 hour post cure. TECB is composed of about 61% volume filler and is composed of Bis-GMA and UDMA in resin matrix. Bis-GMA is a monomer with at least two carbon-carbon (C=C) double bonds to be able to build a three dimensional polymer network[11]. In addition, UDMA has an imino group (_NH_) which contributes to a continuation of polymization[11]. This is what we also see in VBF because of the presence of Bis-GMA and UDMA. Sonicfill 3 had an acceptable DC of 65.8% at 5mm height, and grouped into the middle category of DC results. Unlike TECB and VBF, Sonicfill 3 does not have UDMA, which may be the reason why its DC was not as high. Also Filtek 1 Bulk fill does not have Bis-GMA, which might explain why this material did not yield a higher DC. Another reason TECB had a high DC is the presence of photoinitiators. These particles allow for curing light attenuation and may influence the DC at depth[19].

DC at Various Depths 24 hours Post Cure

Testing DC at varying specimen heights were made to assess the light curing efficiency. A decrease of DC at increased RBC height was seen due to light attenuation[20]. For RBCs, light does not evenly distribute in composite and light also becomes bent due to resin matrix and volumetric filler. Therefore, specific particle shapes of the filler influenced by translucency and presence of photoinitiators will influence the amount of light reaching the depths of the RBC.[15] 5mm height specimen was chosen because that is the manufacturer recommendations for Sonicfill 3[4]. 3mm height specimen was selected to observe how effective the light cure is with decreased height compared to the 5mm height. This allowed us to see if 3mm was acceptable for the conventional composite because the manufacturer recommendation for Filtek Supreme Ultra is 2mm. Therefore it is expected that Filtek Supreme Ultra had the highest difference of DC for 3mm and 5mm. Filtek 1 Bulk and Sonic fill 3 had the highest difference of DC, 34.4% and 22.5% respectively for the bulk fill RBC type. These two materials had lower DC's 24 hours post cure. Filtek 1 bulk has zirconia filler which is known to be more radiopaque causing the light to have less light to penetrate into the bulk fill composite[19]. This explains why the DC at 3mm and 5mm has a high discrepancy. Sonicfill 3 does not have zirconia filler particles, however, still had a lower than expected DC and a high difference in DC for 3mm and 5mm height specimens. TECB had a DC difference of 2.6% which is in agreement of TECB having the highest DC at 5mm. Venus Bulk Flow also had a small difference in DC of 9.4%. This could be because of low filler content and very high

translucency[21]. According to a different study, they found that high filler amounts may interfere the mobility of reactive sites and cause a decrease in the DC[19].

Depth of Cure of Bulk Fill Composites

For some materials, the results were unexpected. Sonicfill 3 which claims to have a DoC of 5mm only had a DoC of 2.684mm. This is a strong contradiction to the manufacturer claim. However, Sonicfill 3 DC was 80.6% at 3mm and 65.8% at 5mm, which falls in the ideal range because RBC have a DC ranging from 55% to 75%[22]. A previous study done by Daughtery et al, had a similar finding where Sonicfill 2 had the lowest DoC and a high degree of polymerization which was the same measurement unit for our study[15]. The low depth of cure could be because of the use of the 20 second light curing as opposed to the manufacturer instructions for use. It is recommended to do a 10 second occlusal and an additional 10 second light cure on the lingual and buccal surfaces of the tooth[4]. In addition, a dense stainless steel mold was used to contain the uncured material which allowed for no addition light to penetrate the material other than the simulated occlusal surface. The same could be true for Filtek One Bulk Fill which had a DoC of 3.803mm as opposed to the manufacturer claim of 5mm increment depth. Likewise with Filtek One Bulk Fill, the manufacturer instructions for use is also 10 seconds occlusal, 10 seconds buccal, and 10 seconds lingual for proper curing instructions[23].

Tetric EvoCeram Bulk Fill has a manufacturer claim of 4mm and had a depth of cure of 3.45 which is not too far from the manufacturer claim, and the instructions for use is 10 seconds light cure[21]. Therefore this is very close to meeting the standards according to the ISO-4049, which states that depth of cure measurement should not be below 0.5mm from the manufacturer claim value[14]. Surefil SDR Flow Plus and Venus Bulk fill surpassed their manufacturer clam of 4mm with 4.624 and 5.059mm respectively. The mentioned study by Daughtery et al found Venus Bulk Fill to have the highest depth of cure[15]. This can be due to low filler content and high translucency contributing to high DC[21].

Conclusion

Within the scope of this research, this study concluded all of the tested bulk-fill RBC brands had a higher DC at 3mm than at 5mm at 24 hours. In addition, all materials had an increased DC at 24 hours compared to immediately after cured. All the tested bulk fill and conventional RBC materials had acceptable DC. All of the bulk fill RBC had a higher DC than the conventional RBC. However, at 3mm the DC for the conventional RBC was in the acceptable range. There were significant differences found with the DoC between the bulk fill RBC brands. There were limitations with the DoC methods. Therefore further studies should be done to replicate a test more similar to a clinical setting to measure the DoC.

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