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THE EFFECT OF DEPTH-OF-CURE ON THE FLEXURAL STRENGTH OF BULK FILL COMPOSITES

A manuscript

Presented to the Faculty of the Advanced Education in General Dentistry, Two-Year Program,

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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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MAY 2021

DENTAL

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Abstract

THE EFFECT OF DEPTH-OF-CURE ON THE FLEXURAL STRENGTH OF DENTAL BULK-FILL COMPOSITES

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Introduction: New bulk-fill composites claim to have a depth of cure (DOC) of 4-5 mm. Placing large single layered composites increases clinician speed and decreases technique sensitivity. To date, there are still many questions regarding how porosity and DOC in restorations can influence their mechanical strength.

Objective: To investigate how porosity and DOC can influence the flexural strength and flexural modulus of two popular resin-based composite (RBC) systems. To nondestructively compare the porosity between a conventional, nanofilled RBC and a bulk-fill RBC using micro-tomographic analysis.

Methods: Two RBC systems tested: Filtek Supreme Ultra (FSU) and Filtek One Bulk (F1B). Ten specimens per system (n = 100), having their 2mm width and 24mm length jig held constant, were made with varying thicknesses (2mm, 3mm, 4mm, 5mm, and 6mm). For each specimen, its top rectangular surface was equally divided into three segments, whereby they were cured simultaneously, receiving a 20s exposure of 1241 ± 5 mW/cm^2 irradiance per segment. Then, all specimens underwent 24 hours of dark cure in 37ºC water bath. Specimen density were measured using Archimedes method. Next, flexural strength were tested using a universal testing machine. Data was analyzed by one-way ANOVA and post hoc Tukey's test (α=0.05).

Results: F1B exhibited significantly higher flexural strength than FSU for both, the 2 mm and 4 mm curing thicknesses. Both RBC systems showed a proportional downward trend between flexural modulus and specimen thickness. Statistically, there was no significant difference in porosity between incremental and bulk placements of FSU. F1B specimens fabricated via bulk placement demonstrated statistically higher diametral tensile strengths and moduli than those specimens made from incremental technique.

Conclusion:
Flexural strength of F1B decreases as material thickness increased with the largest decline in flexural strength observed at 6 mm thickness. The DOC measurements at which FSU and F1B exhibited the highest flexural strength and flexural modulus were 2 mm and 4 mm respectively.

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LIST OF ABBREVIATIONS

RBC-----------Resin Based Composite
FSU----------- Filtek Supreme Ultra
F1B----------- Filtek One Bulk
DOC---------- Depth of Cure
SS------------- Stainless steel

Background

The purpose of a dental restoration is to replace the functional, esthetic, and biologic properties of healthy tooth structure.[1] Amalgam, being used for more than 150 years, has proven itself as a reliable means in restoring tooth structure. Nevertheless, amalgam, with its obvious esthetic short comings, is slowly being replaced by composite as the restorative material of choice in the United States and abroad.[2] Compared to amalgam, resin based composites are esthetically superior and more conservative and lacks the most controversial ingredient – mercury. Numerous organizations including the World Health Organization (WHO) affirm that mercury in dental amalgam poses no known systemic health concerns. However, the continued controversy surrounding it as a health and environmental hazard has resulted in the WHO calling for a global amalgam "phase down" program.[3,4,5,7] This has influenced dental schools such as NYU college of Dentistry and South California School of dentistry to restrict amalgam use. In addition to dental schools, countries such as Norway, Sweden, Denmark, Japan, and Finland have also placed bans or restrictions against amalgam.[3,5,6] The United States appears to be on the same amalgam-free trajectory as these other countries. In November 2013, the United States joined in with other countries and signed the Minamata Convention on Mercury, which calls for a phase down of dental fillings using mercury amalgam.[8] Amalgam's shortcomings, combined with its controversy, have fueled intensive research to improve other restorative materials such as composites.

Unfortunately, like amalgams, composites also have problems, which have kept them from being an ideal restorative material. For example, placement of a composite restoration is both technique sensitive and time consuming, requiring more finesse and attention to detail than amalgam placement.[9] Most conventional RBC are placed and cured in 2 mm increments. Larger increments can result inadequate curing, which can lead to bulk fractures, marginal breakdown, or recurrent caries. Layering has brought additional challenges such as the probability of incorporating porosities between layers, the need to light cure each successive layer, and an increase in overall restoration's shrinkage stress.[11,12,13]

Filtek Supreme Ultra (FSU) is the most used conventional RBC in military dental treatment facilities. FSU, though a break-through when first introduced, is marketed to be placed in increments of no more than 2mm thick.[14]

To address some of the above-mentioned limitations of conventional RBC, manufacturers have developed bulk-fill RBC systems that allow placement of 4 to 5mm increments with a full DOC. Placing these larger single layered composites has the potential to increase clinician speed and decreases technique sensitivity.[15] To achieve this, manufactures have employed a variety of strategies. Such strategies include increasing composite transparency, reducing monomer percentage through the addition of inorganic fillers, and utilizing multiple different filler particles, prepolymerized filler particles, and low-shrinking monomers.[16]

Other clinicians have been leery to change to a bulk-fill system, citing a lack of long-term clinical data available.[17] Campos et al (2014) concluded that there are no significant differences in marginal integrity, or microleakage between bulk-fill RBCs or conventional RBCs.[18] The American Dental Association (ADA) reported that the laboratory performance of the bulk-fill RBCs was comparable or better than the conventional RBCs.[19]

The body of clinical evidence is also steadily growing. A one-year clinical evaluation concluded that bulk-fill RBCs showed similar clinical performance when compared to conventional RBCs.[20] A recently published 6-year randomized controlled clinical study compared a bulk-fill and conventional RBC in posterior restorations and concluded there was no significant difference in survivability.[21] Furthermore, a 10-year randomized control trial comparing another bulk-fill RBC against a conventional RBC also showed no significant difference in survivability between the two materials. Both materials exhibited a high clinical effectiveness at the 10-year follow-up.[22] The present body of literature seems to validate that bulk-fill materials are a suitable treatment modality for posterior restorations.

Prior research has examined flexural strength of bulk-fill RBCs and has shown that bulk-fill composites having superior or equivalent flexural strengths as conventional RBCs.[28,29] To the best of the author's knowledge, no studies have examined how flexural strength or flexural modulus is affected when the composite samples are made greater than 2 mm in depths. The objective of this study was to investigate how porosity and DOC can influence the flexural strength and flexural modulus of two popular resin-based composite (RBC) systems. To nondestructively compare the porosity between a conventional, nanofilled RBC and a bulk-fill RBC using micro-tomographic analysis.

HYPOTHESIS:

The null hypothesis was that DOC and porosity do not affect the flexural strength or flexural modulus of bulk-fill and conventional resin-based composites.

Materials and Methods

<u>Measurements of flexural strength and flexural modulus:</u>

The flexural strength and flexural modulus were determined using a three-point bending test. All samples were prepared in an orange lit room that lessened any unwarranted polymerizing effect of white ambient light on uncured composites. Five different rectangular stainless steel (SS) molds, having dimensions of 2x2x25, 2x3x25, 2x4x25, 2x5x25, and 2x6x25 mm^3, were used to prepare a total of 100 specimens (See Figure 1).

To fabricate a rectangular specimen, a SS mold with one of the aforementioned dimensions was selected; it was then lightly lubricated with petroleum jelly (Vaseline, Unilever, London, United Kingdom) and placed on a plastic strip-covered glass slide. Next, a RBC was injected into the SS mold until it was filled. Then the top surface of the mold was covered with a second plastic strip and glass slide. A custom jig, consisted of three non-overlapping windows, was used to align three light guides for the delivery of adequate irradiance to the top surface. The three windows were linearly positioned such that they divided the $2X25mm^2$ rectangular surface into three equal segments – each having equivalent irradiance area with one another. Using three light curing units of the same brand (1241 ± $5mW/Cm^2$, Elipar DeepCure-S, 3M ESPE, St Paul, MN, USA), with one light guide per window, the three equally divided top rectangular surface was irradiated simultaneously with a duration of 20s light exposure, (See Figure 2). The specimens were then removed from the mold and polished with silicon carbide paper (grit size P1200/4000, Buehler, Lake Bluff, IL, USA) (See Figure 3). Afterwards, they were stored in distilled water at an intraoral temperature of 37ºC for 24 hours (See Figure 4).

These aforementioned steps were repeated to fabricate a total of 50 samples per each of the two different RBC brands: Filtek Supreme Ultra (FSU) and Filtek One Bulk (F1B), (See Figure 5). A "gold standard" integrating sphere fitted with a photo-spectrometer was used to verify the irradiance output and consistency of the three light curing units.

Subsequently, each specimen was placed on a 3-point bending test apparatus which was constructed with a 20 mm span length between the supporting rods (See Figure 6). A central load was applied with a head diameter of 2mm, and a crosshead speed of 0.5mm/min using a universal testing machine (Instron, Norwood, MA, USA). The flexural strength was calculated using the equation:

$$\sigma = \frac{3Fl}{2bd^2}$$

Where F is the loading force at the fracture point, l is the length of the support span (20 mm), b is the width, and d is the depth. Measurements were made using an electronic digital caliper (SPI 13-610-1, Swiss Precision Instruments, Melville, NY, USA) (See Figure 7). Flexural modulus will be determined from the slope of the linear region of the load-deflection curve using analytical software (Instron, Norwood, MA, USA).

Statistical Analysis:

Data was analyzed by one-way ANOVA and post hoc Tukey's test (p=0.05). A Non-linear regression was used to fit the correlation between thickness and flexural strength and a linear regression used to correlate thickness verses modulus. An analysis of variance was performed with p<0.0001.

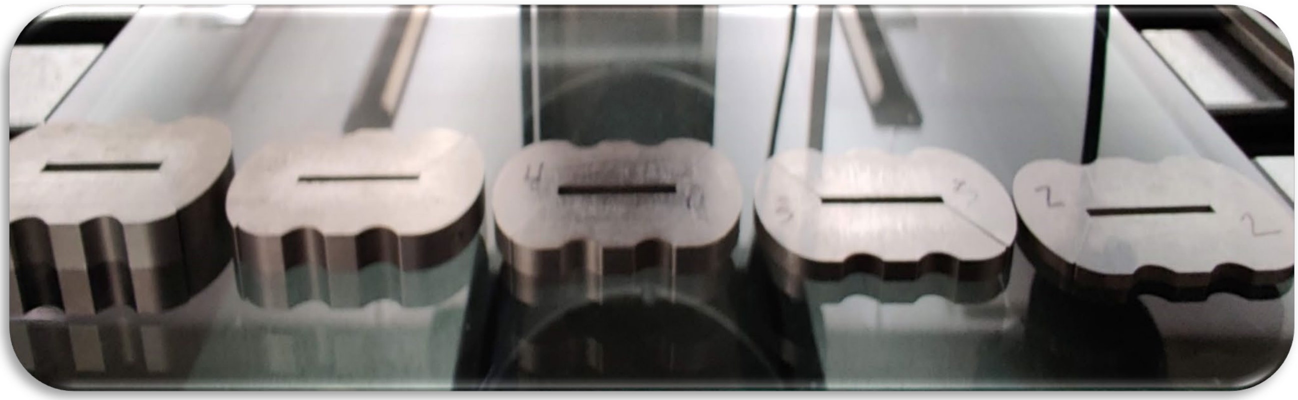

Figure 1. Flexural strength jigs used to make composite samples varying only in depth. From right to left; 2mm, 3mm, 4mm, 5mm, and 6mm jigs.

Figure 2. Specimen prepared in an orange ambient lighted room and photopolymerized with the Elipar DeepCure-S, 3M ESPE.

Figure 3. Specimen ground with silicon carbide paper to remove protruding edges.

Figure 4. Specimens stored in distilled water at room temperature prior to flexural testing

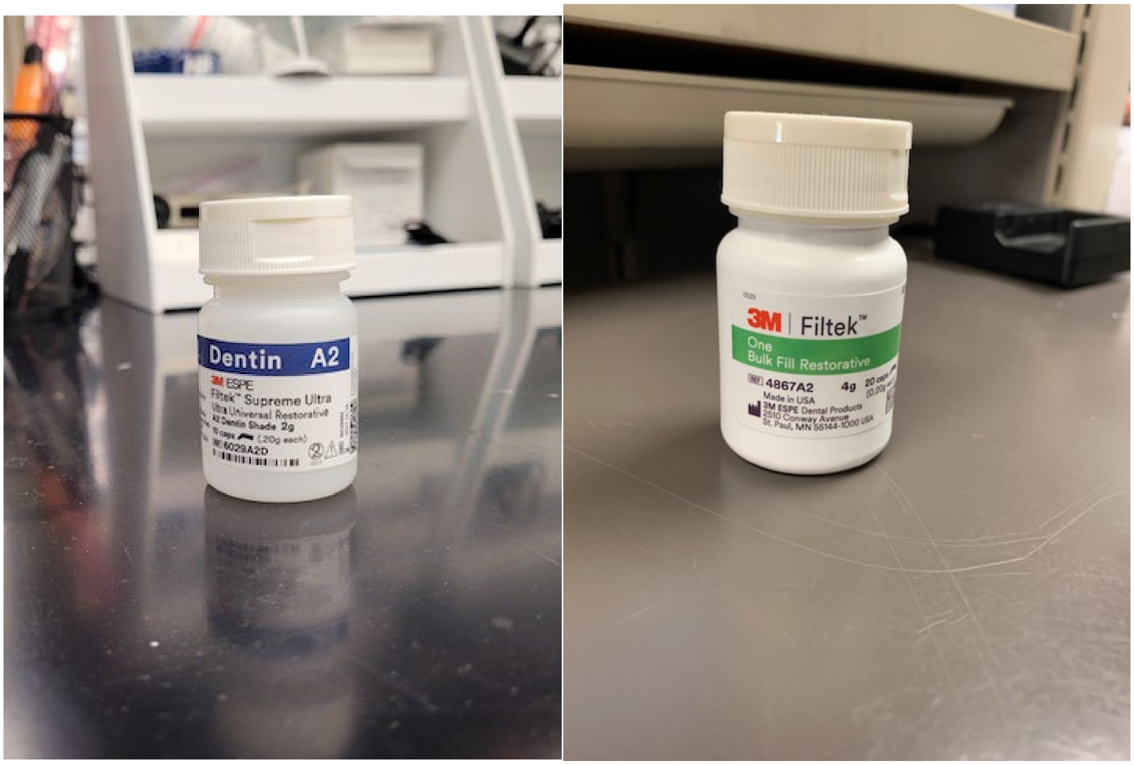

Figure 5. Two RBCs tested from left to right: FSU- Filtek Supreme Ultra and F1B- Filtek One Bulk Fill

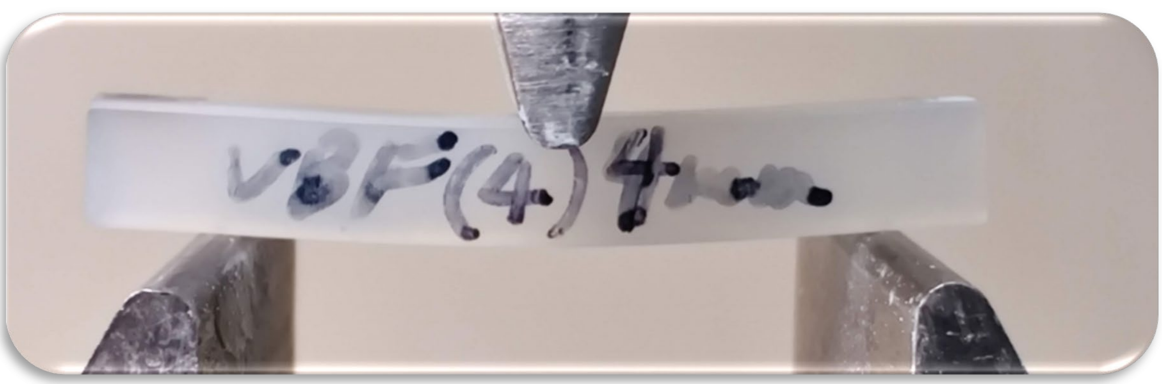

Figure 6. Specimen subjected to flexural strength testing using the MTS Systems Corp Alliance RT/5 testing machine

Figure 7. Digital calipers used to measure variations in sample depth after sanding edges.

Flexural strength

Figure 1: Flexural strengths of F1B and FSU plotted against specimen thicknesses, keeping length and width constant. The same letters across columns are not significantly different than each other (p > 0.05).

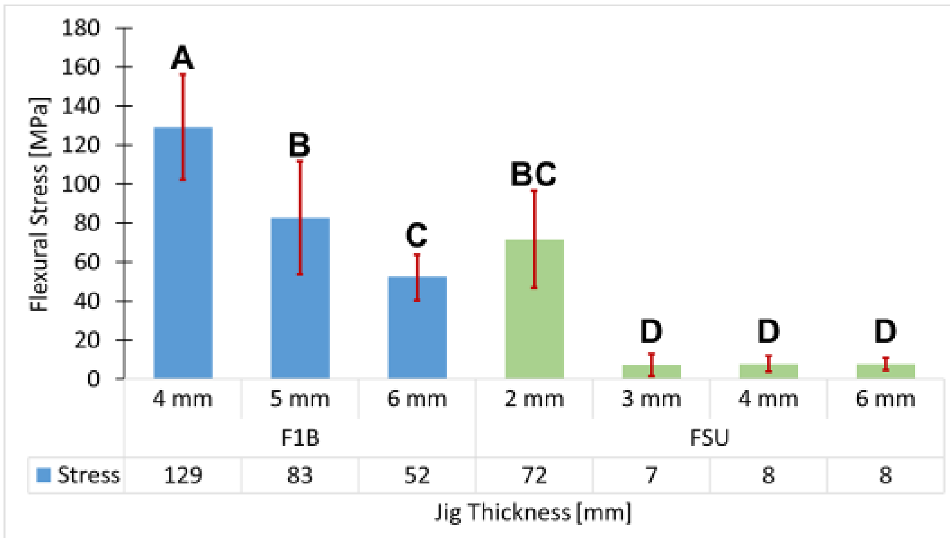

Figure 8. Flexural Strength as a function of specimen thickness.

Flexural modulus

Figure 2: Flexural moduli of F1B and FSU plotted against specimen thicknesses, keeping length and width constant. The same letters across columns are not significantly different than each other (p > 0.05).

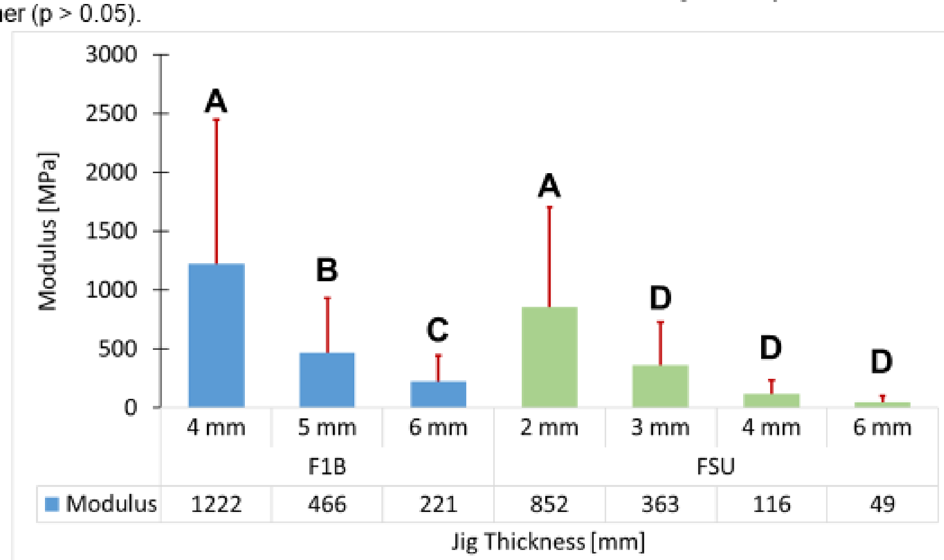

Figure 9. Flexural modulus as a function of specimen thickness.

Diametral tensile & modulus

Figure 4: Although there were no statistical porosity differences between incremental and bulk placements using the same resin-based composite, F1B, specimens fabricated via bulk placement demonstrated statistically higher diametral tensile strengths and moduli than those specimens made from incremental technique.

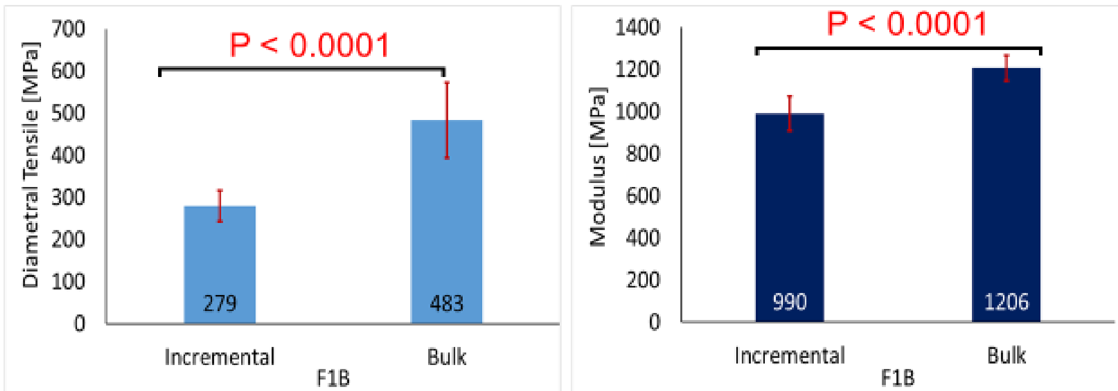

Figure 10. Diametral tensile and flexural modulus as a function of density.

Results

F1B exhibited significantly higher flexural strength than FSU for both, the 2 mm and 4 mm curing thicknesses. Both RBC systems showed a proportional downward trend between decreasing flexural modulus and increasing specimen thickness. There were no significant statistical porosity differences between incremental and bulk placements using the same resin-based composite. F1B specimens fabricated via bulk placement demonstrated statistically higher diametral tensile strengths and moduli than those specimens made from incremental technique.

Discussion

This study evaluated the flexural strength and flexural modulus of two RBC systems with specimens of various depths. Results were compared against a conventional RBC.

These findings matter because it can lead to success or failure of the material once placed inside the prepared tooth. Upon placement and curing of the material, the implications can lead to fracturing of the material or leakage of the material if not cured properly. These results influence clinical treatment by revealing the importance of these factors when placing technique sensitive material in bulk or incremental layers. It can make a difference of long-term success or short-term failure. Practical actions recommended are to follow the manufacturer's recommendations for placement and curing times to reduce porosities and concerns with flexural strength and moduli.

In general, flexural strength of the RBCs decreased as material thickness increased, with the largest decline in flexural strength observed at >4mm of DOC for F1B and >2mm of DOC for FSU (See Figure 8). Most materials perform optimally when placed in smaller increments. Nevertheless, as Figure 8 also demonstrates, the increasing thickness of the prepared sample beyond manufacturer's recommendations leads to a precipitous drop in flexural strength for both RBC's. After 4mm composite increments in F1B, a DOC can support 5mm but will be at a 35% decrease in flexural strength. FSU 2mm is recommended by manufacturer, but leads to a decrease in flexural strength >3mm at 90% per additional 1mm. The lowest recorded values were seen with FSU 3mm, 4mm, and 6mm samples with a mean of 10 MPa. F1B had a steadily decrease in flexural strength between 4mm and 6mm samples. FSU also had a vast decrease in flexural strength between 2mm and 3mm samples. Flexural strength values in the 5mm and 2mm sample depths were similar for F1B and FSU. Stress measured at newton per square millimeter (N/mm2) as force per unit area.

Both RBCs tested showed an inversely proportional trend between flexural stiffness and specimen thickness (See Figure 9). F1B had a high flexural modulus value approximating 1222 MPa in the 4mm sample range and a 6mm depth sample having a drastic reduction to 221 MPa. FSU had lower flexural modulus value approximating 852 MPa in the 2mm sample range and a 6mm depth sample having a drastic reduction to 49 MPa.

Generally, all samples tested showed 4mm samples being the densest (See Figure 10). F1B incremental and bulk RBCs exhibited a linearly proportional relationship between their porosity differences at 4mm for 20 seconds. However, between their tensile strength (compression breaking material on its side) and modulus, F1B bulk demonstrated statistically higher diametrical tensile strength and modulus than incremental.

Based on the data of this study, the null hypothesis was rejected. Since the results indicate that porosity may not be significantly different with the specimens tested, the flexural strength and modulus were affected by DOC when incrementally increased.

Some of the limitations and challenges when conducting this study were limited sample size and material available. With more material and time available, a larger specimen collection could've been tested for flexural strength, flexural modulus, diametral tensile, and porosity. Future studies should be performed with a larger sample size and more RBC systems to see if the outcome would be different.

See table 1 and table 2 for a breakdown of some of the main differences and similarities between the tested composite systems to include shade types.

Table 1: Technical Profiles of the RBCs Evaluated in the Study- Photointiators/Shades/DOC						
Materials	**Abbreviation**	**Photoinitiator**	**Shade**	**DOC claim**	**Manufacture**	**Product Launch**
Filtek Supreme Ultra (control)	FSU	Camphorquinone/Amine	A2	2mm	3M	2013
Filtek One Bulk	F1B	Camphorquinone/Amine	A2	5mm	3M	2017

Table 2: Technical Profiles of the RBCs Evaluated in the Study- Densities						
Abbreviation	**Composition (Resin)**	**Composition (Filler)**	**Filler Content (wt%/vol%)**	**Filler size (µm)**	**Volumetric Shrinkage**	**Uncured Density**
FSU	Bis-GMA, UDMA, TEGDMA, Bis-EMA(6), PEGDMA	Zirconia/silica clusters and non-aggregated silica and zirconia particles. (see Filler type)	78.5%/63.3%	4-20 nm zircona and silica filler (non-aggregated and aggregated) Average cluster size 0.6 to 10 microns	2%	1.9g/cm3
F1B	AFA, AUDMA, UDMA and 1,12-dodecane-DMA	Zirconia/silica clusters and non-aggregated silica and zirconia particles. Ytterbium trifluoride filler.	76.5%/58.5%	4-20 nm zircona and silica filler (non-aggregated and aggregated) Unknown average cluster size.	1.80%	1.9g/cm3

Abbreviation: U, Universal; DOC, Depth of Cure; Bis-GMA, bisphenol A glycidyl dimethacrylate; Bis-MPEPP, 2,2-bis[(4-methacryloxy polyethoxy)phenyl)propane; EBPADMA, ethoxylated bisphenol A dimethacrylate; NA, not available; S-PRG: surface pre-reacted glass ionomer; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-EMA(6), Bisphenl A polyethylene glycol diether dimethacrylate, EBADMA: ethoxylated Bis-GMA
Unless otherwise stated, all information listed is from manufacturer data and SDS sheets.
*NA for SF3- Data used from original SF
** NA for SDR+- Data used for original SDR flow
***Garcia, D., et al. "Polymerization shrinkage and depth of cure of bulk fill flowable composite resins." Operative dentistry 39.4 (2014): 441-448.

Conclusion

Although there was no statistical porosity differences between incremental and bulk placements using the same resin-based composites, F1B specimens fabricated via bulk placement demonstrated statistically higher diametral tensile strengths and moduli than specimens made from incremental technique.

Flexural strength of F1B decreases as material thickness increases with the largest decline observed >4mm thickness. FSU flexural strength decreases drastically as material thickness increases with the largest decline observed >2mm thickness.

F1B fill claims up to 5mm can increase productivity, time, and less flexural stress on the tooth. The F1B specimen of 5mm tested better than the FSU conventional specimens 2mm, 3mm, 4mm, and 6mm on the flexural strength. Bulk fill composite resin up to 5mm is less flexural stress and modulus on a tooth if curing is done correctly and can withstand higher MPa than conventional FSU resin. Of the 2 RBC systems tested, F1B exhibited the highest flexural strength and flexural modulus with porosity being statistically no different than FSU. The DOC however decreases when beyond the manufactured instructions of 2mm for FSU and 4mm for F1B, subsequently affecting the flexural strength and moduli.

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