Evaluation of Various Properties of "Bulk EZ Prototype" – Final Report

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Objective

This protocol was designed to provide basic information on the physical properties of a new prototype material developed by Zest Dental Solutions, further known in this proposal as "Bulk EZ Prototype." The material was compared against other manufacturer's products.

Materials

Bulk EZ Prototype and two competitive products, Sonicfill 3 and SureFil SDR Flow +, were supplied by Zest Dental Solutions. Zest also provided the dispensing systems for the materials.

Methods:

The properties tested includes the following:
1. Polymerization contraction stress (Bioman)
2. Flexure strength/modulus
3. Fracture toughness
4. Depth of cure – tooth model
5. Maintenance of surface polish during toothbrush abrasion

Note: As the new material is a dual cure material, like its predecessor, the manufacturer has asked that the material be allowed to self-cure for 200 seconds prior to light-curing. This method was followed in all of the study protocols.

1. Polymerization Contraction Stress:

The Bioman stress measurement device has been described previously in detail (Watts et al., 2003). The system is based on a cantilever load-cell (500 kg) fitted with a rigid integral clamp. The compliant end of the cantilever holds a circular steel rod (5 mm diameter × 22 mm long) vertically and perpendicular to the load-cell axis. The counter-face consists of a removable rigid glass plate that is held rigidly relative to the base-plate in a special clamp during measurement.

Test Method: The lower end of the steel rod is abraded on silicon carbide paper and treated with Z-Prime (Bisco), and in contrast to the original methodology described by Watts, the surface of the glass-plate opposing the steel-rod is only treated with silane (3M/ESPE Dental Products, St Paul, MN 55144, USA)) (instead of sandblasting + silanation). The composite is then introduced between the plate and vertical rod to form an uncured specimen-disk of 5 mm diameter and 0.8 mm thickness (which represents a bonded to non-bonded surface area, i.e. C-factor, of ~3). The composite specimen is irradiated through its thickness dimension from below by the LCU (Demi, Kerr) for 20 s (8 mm guide; actual irradiance reaching the specimen = 1200 mW/cm^2), as measured with the CheckUp device (BlueLight Analytics). *Note: The light application wa delayed until 200 seconds for the Danville product to allow time for the self-cure reaction.* The registered load (in Newton, N) is divided by the disk area to obtain the stress values (MPa). Note: we did not multiply the raw stress data by a "correction factor" of 4 in order to relate the present data to a low compliance system, such as a human tooth cusp. as was done in previous published studies with the Bioman device (Watts and Satterthwaite, 2008). The presented results are raw values.

Measurements were performed during 10 min after the photoactivation procedure at ~22°C (n = 5). After each evaluation, the Bioman clamps were removed and the set resin sample/glass-plate/metal piston was removed and carefully observed to verify if signs of any debonding was present. It is important to mention that detachments are rare (none were observed in this study). The results for the different composites were compared using ANOVA and Tukey's multiple comparison test (α=0.05).

Results: A typical plot of stress measured vs. time is shown in Figure 1.

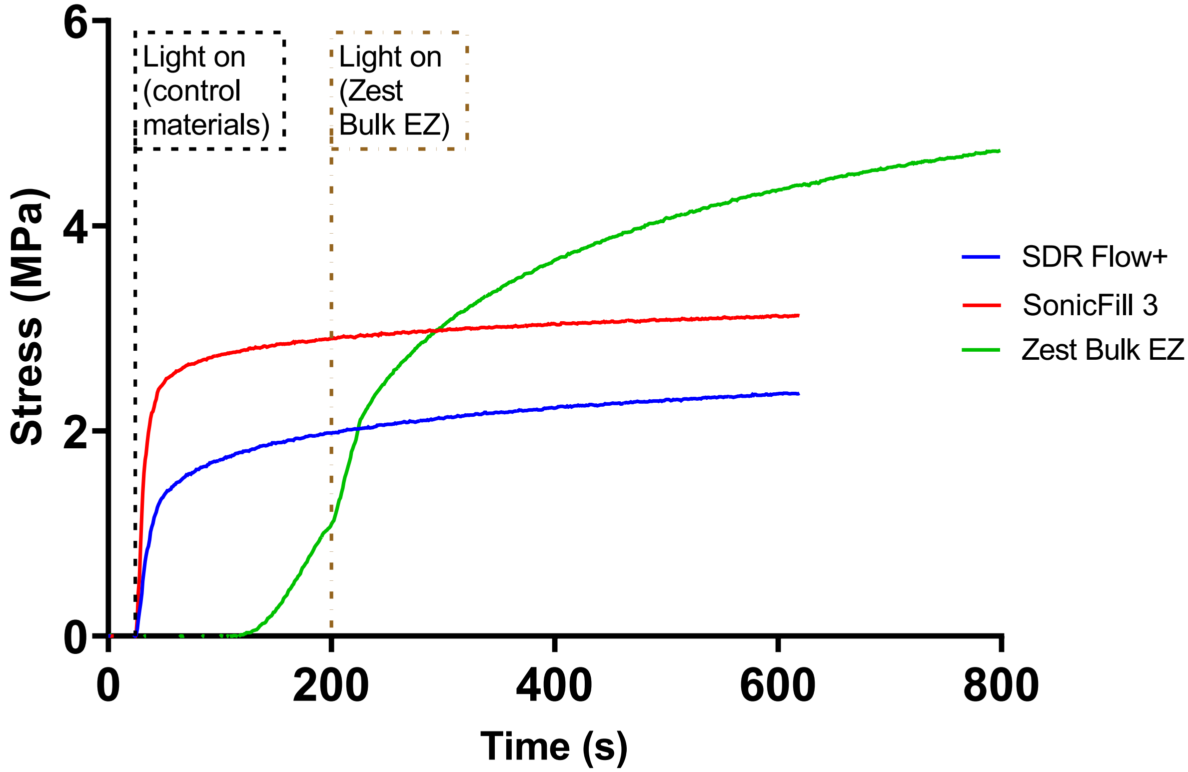

Figure 1. Average polymerization stress vs time plotted for all groups. SDR Flow+ and SonicFill3 were irradiated at time = 20 s, and Zest Bulk EZ was irradiated at time = 200 s to allow time for self-cure reaction.

Note that the Zest material had delayed stress acquisition due to the delay in applying the light. However, it is obvious from this plot that by the time the light was applied, setting due to the self-cure reaction had begun in the material based on the initial rise in recorded stress from about 120 to 200 seconds.

Overall, the Zest material had developed significantly higher stress at 10 minutes after light activation than the other two products (Figure 2). Note also that the stress for SonicFill 3 was significantly higher than for SDR Flow+. These results are consistent with previous studies in our lab on previous formulations of these two other commercial products, though the stress values recorded here are higher than for earlier formulations tested on this same device. Note that the relationship of these three materials is identical to that obtained for the original study completed for Bulk EZ in 2016.

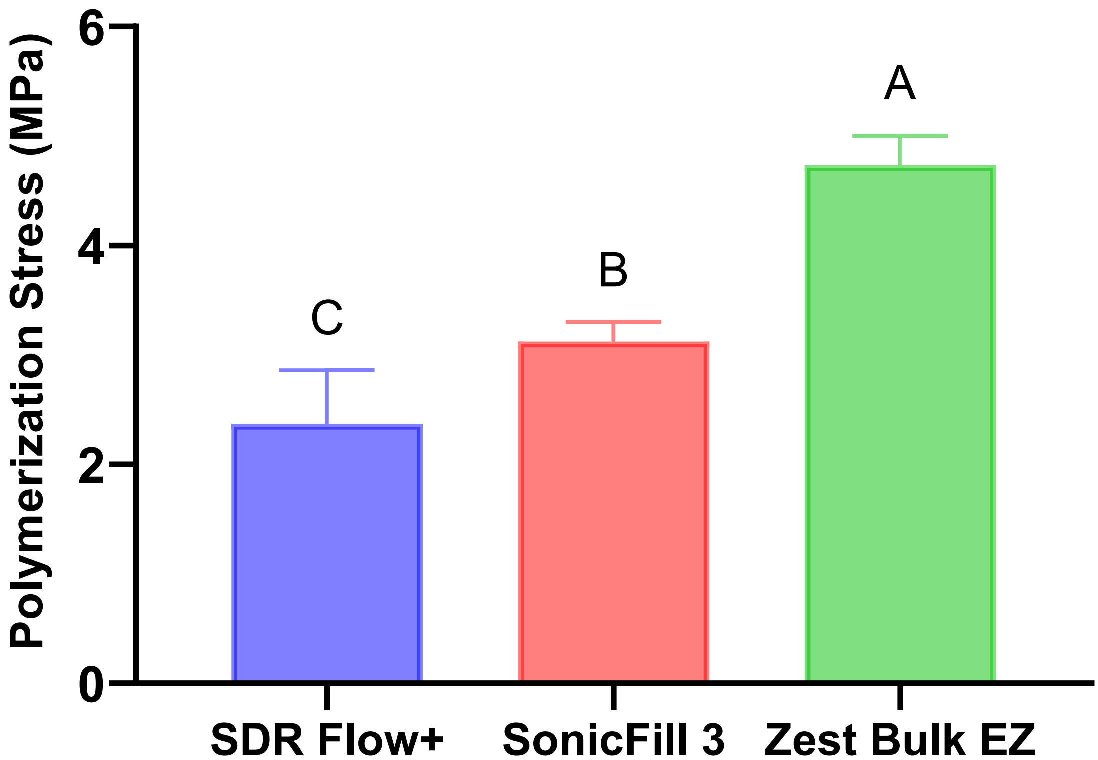

Figure 2. Final polymerization stress (MPa) at time = 600 s after irradiation. Results were compared using ANOVA/Tukey's multiple comparison test (α = 0.05).

2. Flexural Strength/Modulus:

Flexure properties are determined in three point bending, following ISO4049.

Test method: Beam-shaped specimens (n=10) were made in two part steel rods with dimensions of 2 mm x 2 mm x 25 mm. The composites were light-cured from the top and the bottom with a Demi light, 9 mm tip, with stepped exposures of 20 seconds to cover the entire specimen. *Note: The light application was delayed until 200 seconds for the Danville product to allow time for the self-cure reaction*. The specimens were stored in water at 37°C for 24 hours and then tested in 3-point bending (20 mm span) on a universal testing machine at a cross-head speed of 0.25 mm/min. The flexure strength was determined using the maximum load. Any evidence of plastic deformation was noted, but not seen for these materials. The flexure modulus was determined from the initial slope of the force-deflection curve. The protocols followed ISO 4049 for dental restoratives. Ten specimens were tested for each material. Data is presented as mean (with standard deviation). The results for the different composites were compared using ANOVA and Tukey's multiple comparison test (α=0.05).

Results: The flexure strength results are plotted in Figure 3. Note that Sonicfill had numerically the highest strength, but this was not significantly different than SDR Flow+. Zest material was equivalent to SDR Flow+, but statistically lower than Soncifill 3. The relationship between the three materials is essentially identical to the results from the initial study in 2016, and the flexure strength values are also similar. This Zest material is a bit lower than its predecessor, which was almost exactly 100 MPa.

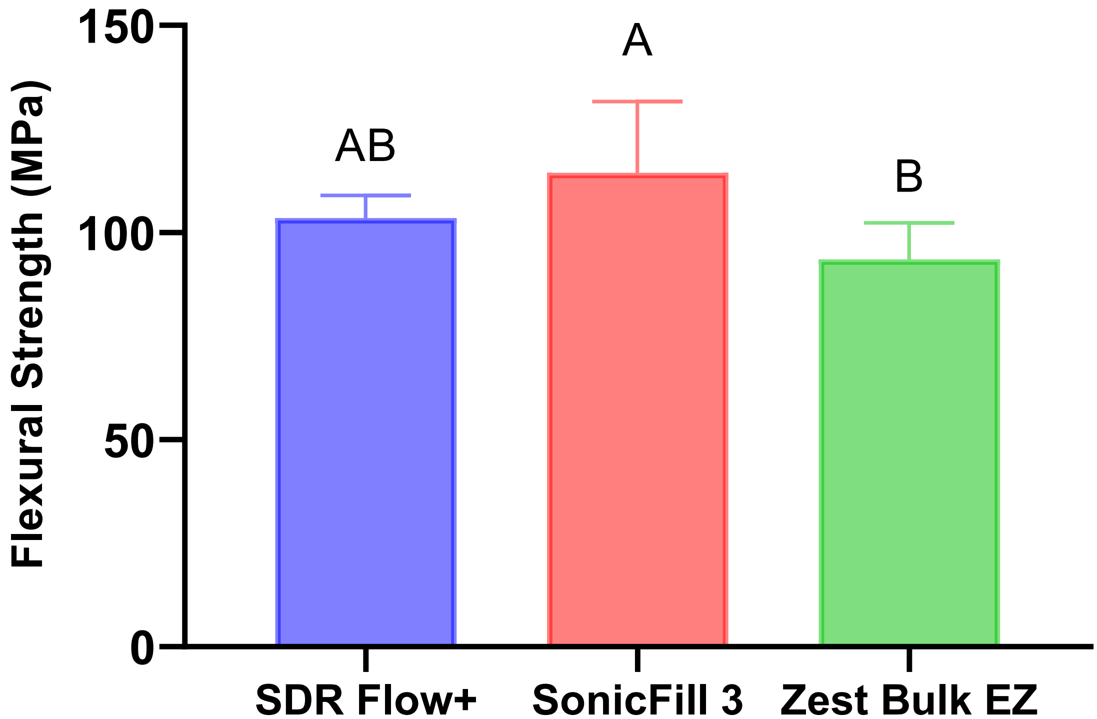

Figure 3. Flexural strength (MPa) for all groups. Results were compared using ANOVA/Tukey's multiple comparison test ($\alpha = 0.05$).

The flexure modulus results are presented in Figure 4. The Sonicfill 3 material had the highest modulus, followed by SDR Flow+, which was significantly higher than Zest. This is the same relationship as for the previous study, with the exception that there was no difference between Surefull SDR and Bulk EZ in that study. Note that these values for flexure modulus were slightly lower than those from the previous study, but within approximately 10%.

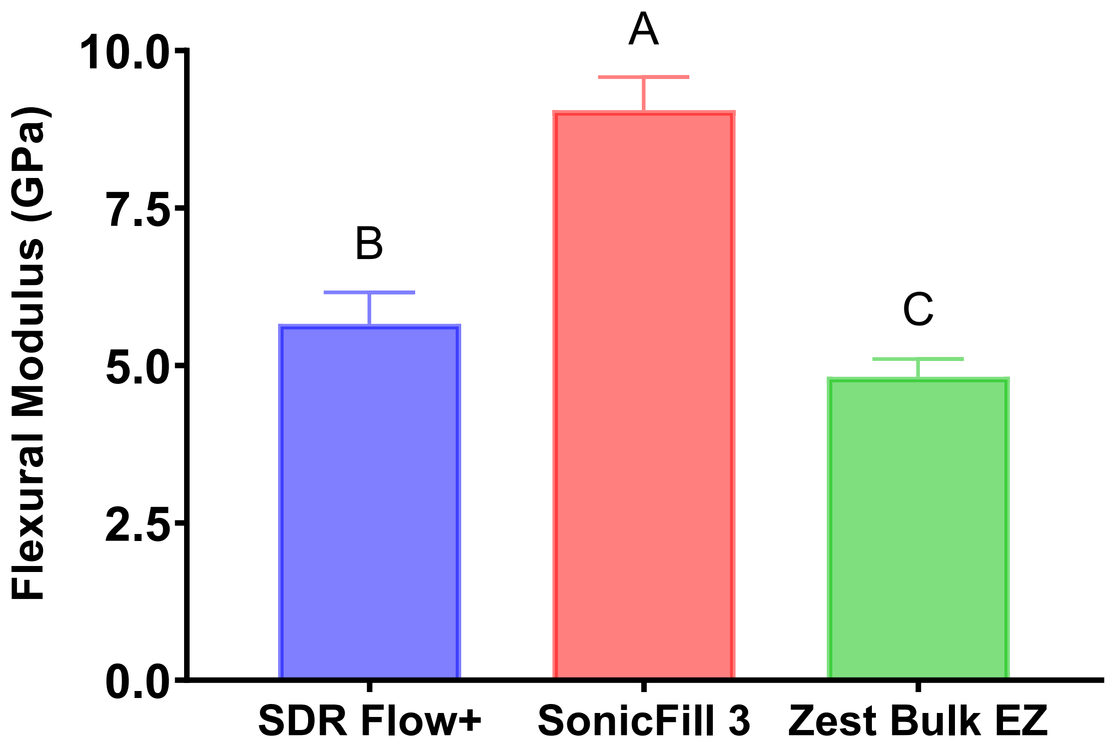

Figure 4. Flexural modulus (GPa) for all groups. Results were compared using ANOVA/Tukey's multiple comparison test (α = 0.05).

3. Fracture Toughness:

Fracture toughness was tested on single-edge notched beams in three-point bending following ASTM E-399.

Test method: Specimens (n=10) were made in two-part stainless steel molds with dimensions 2.5 mm x 5 mm x 25 mm with a razor blade notch at the mid-span producing an a/w = 0.5. Specimens were light-cured from the top and bottom as described for the flexure strength test, with stepped exposures to cover the entire specimen. *Note: The light application was delayed until 200 seconds for the Danville product to allow time for the self-cure reaction.* The specimens were stored in water at 37°C for 24 hours and then tested in 3-point bending (20 mm span) on a universal testing machine at a cross-head speed of 0.254 mm/min. The fracture toughness was determined using the maximum load (unless there is evidence of plastic deformation - there was not). The protocol followed ASTM E399 and has been used for years in our laboratory (Ferracane et al., 1987). Ten specimens of each composite were tested. Data is presented as mean (with standard deviation). The results for the different composites were compared using ANOVA and Tukey's multiple comparison test ($p<0.05$).

Results: The fracture toughness is plotted in Figure 5 and shows no difference between SDR Flow+ and Sonicfill 3, but both were significantly higher than the Zest material. The actual difference was less than 10%. Note: three specimens for the Zest material gave odd behavior and broke prematurely. These specimens were replaced with three new ones, and this can be seen in the spreadsheet with all of the individual results. It is not clear what happened at this point.

Note that these fracture toughness values compared favorably numerically with the results obtained in 2016. The only difference is that in the previous study, Surefill SDR had higher toughness than both Bulk EZ and SonicFill 2, which were the same.

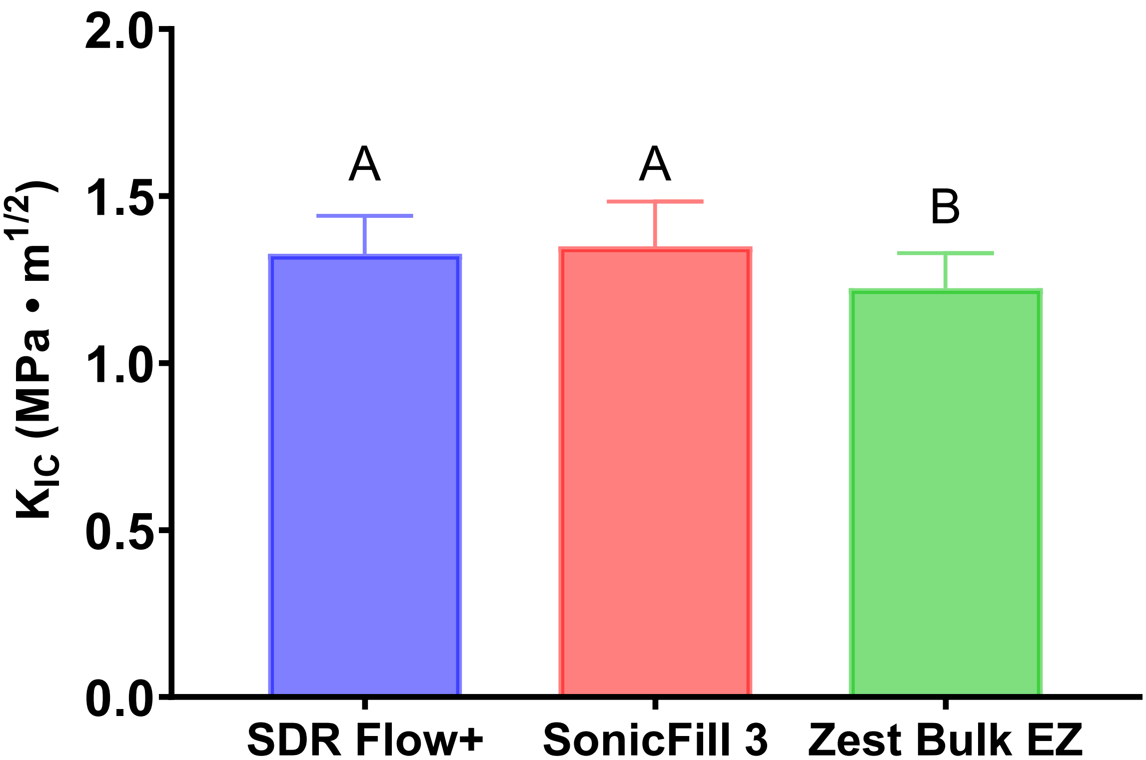

Figure 5. Fracture toughness (MPa · $m^{1/2}$) for all groups. Results were compared using ANOVA/Tukey's multiple comparison test ($\alpha = 0.05$).

4. Depth of Cure – Near IR Method Mapping Method

Rectangular specimens (5 mm × 2 mm) of 5 mm in thickness are produced by inserting the different composites in custom-made polyvinyl siloxane molds, sandwiched between glass slides. The light source (Demi) was positioned directly above the surface of the specimen to ensure uniform light distribution at the surface. *Note: The light application was delayed until 200 seconds for the Danville product to allow time for the self-cure reaction.*

After curing, the specimens (n=5) were embedded in epoxy resin (Buehler, Lake Bluff, IL, USA) and sectioned along the long axis using a high-speed diamond saw to produce slices with 300 µm thickness. This procedure was carried out under copious water cooling to avoid potentially increased conversion due to heating. The slices were then stabilized on glass slides with silly putty, and placed on the automated stage of the IR-coupled microscope (Continuum, ThermoScientific, Madison, WI, USA. Near-IR was used to obtain spectra at 500 µm intervals from the surface, with 3 measurements per depth, totaling 30 points in each map. Degree of

Conversion at each point was assessed by recording the decrease in area of the methacrylate absorption peak at 6165 cm^{-1}. Degree of conversion (DC, in %) was calculated with the following equation:

$$DC = \left(1 - \left(\frac{vinyl\ peak\ area\ for\ the\ polymer}{vinyl\ peak\ area\ for\ the\ monomer}\right)\right) \times 100$$

The three points at each depth were averaged, and the means used to build a 2D conversion map as a function of depth. The results for the different composites were compared using ANOVA and Tukey's multiple comparison test ($p<0.05$).

Results: Figure 6 shows the heat map of degree of conversion vs. depth for the three materials. The graph shows the obvious consistent conversion for the Zest material at all depths, due to the combination of light and self-curing. Also, the degree of conversion was highest for the Zest material as shown by the yellow color throughout. The map also reveals that Sonicfill 3 had a greater decline in conversion with depth than the other two materials.

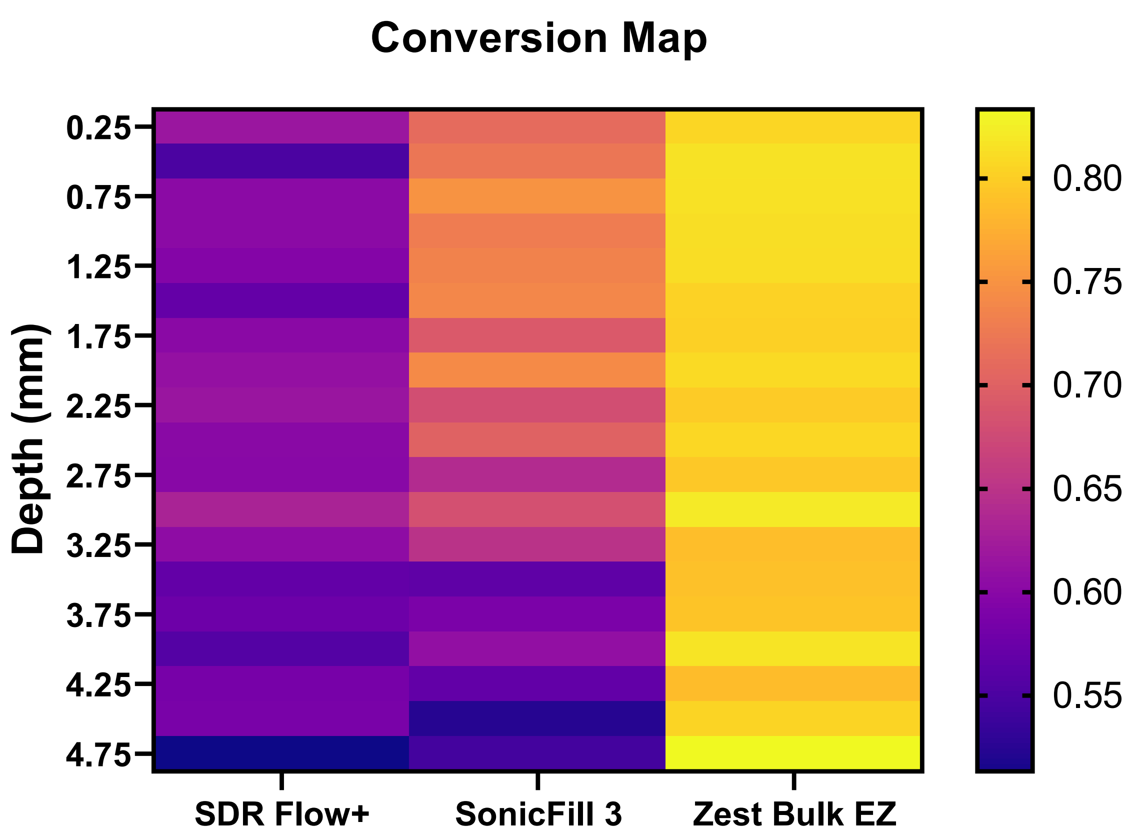

Figure 6. 2D conversion map for all groups. The map for each respective material shows the average conversion of 3 specimens, measured at 3 discrete locations for each depth.

Figure 7 shows the conversion as a function of depth based on the normalized values for each material, i.e. maximum recorded conversion is 100% for each material. The plot shows that each material was able to produce at least 80% of maximum cure to a depth of at least 4 mm, though SDR Flow+ and Zest remained above the 80% threshold even down to 5 mm.

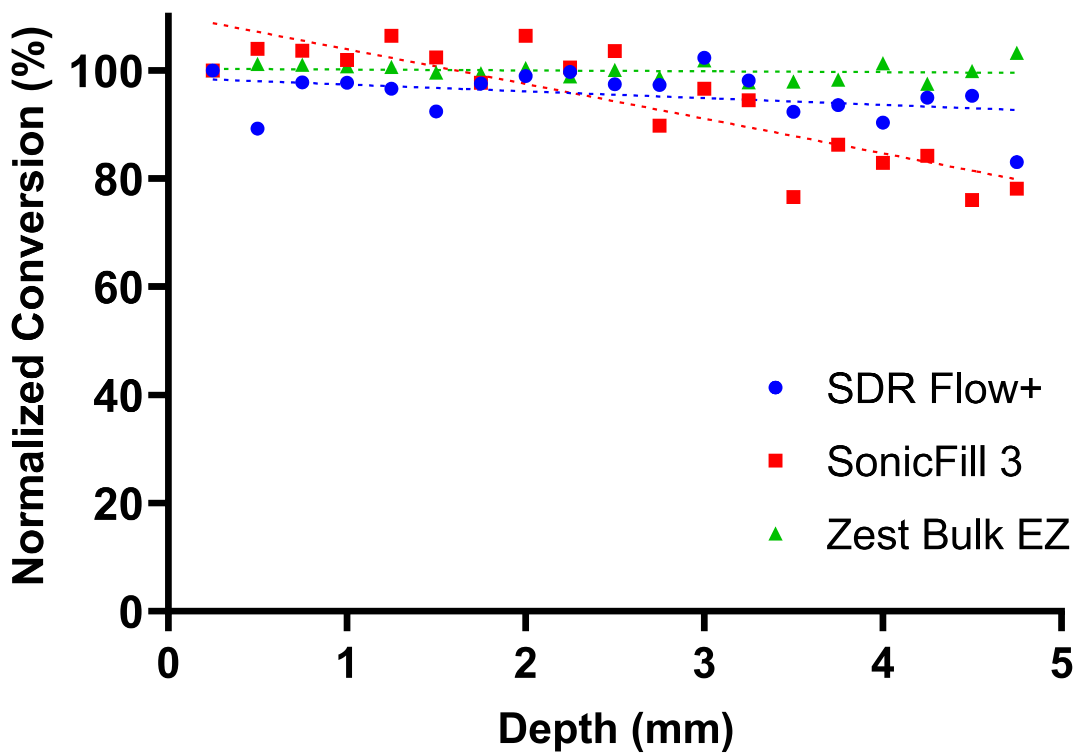

Figure 7. Normalized conversion (i.e. Percent change in conversion relative to the conversion at the top of the specimen) versus depth for all groups.

5. Maintenance of Surface Polish – Toothbrush Abrasion

Rectangular-shaped composite specimens (W=5.0 mm, L=12 mm, 2.5-mm thick), the half-bars from the fracture toughness tests, were used.

The specimens had been abraded by one pass on #600 silicon carbide paper to produce a standard surface. The surface was then wet finished/polished using metallurgical polishing papers, from 1000 grit Silicon carbide, and proceeding to 2400 grit and finally to 4000 grit. After each step, the gloss was measured, as described below. The goal was to achieve maximum gloss. Once the procedure was established for each material, it was followed for each specimen.

Gloss Evaluation:

Gloss was measured using a small-area glossmeter (Novo-Curve, Rhopoint Instrumentation, East Sussex, UK), with a square measurement area of 2 × 2 mm and 60° geometry. Gloss measurements are expressed in gloss units (GU). The specimen was aligned on the gloss meter with a custom jig to ensure that the measurements were made in the same place each time.

Toothbrush abrasion:

Composites specimens were mounted onto an acrylic disk mounted onto a dc gearmotor. The disk was turned at a frequency of 1 Hz, dipping the specimens in a slurry of commercial toothpaste

(Crest Pro Health, Proctor and Gamble) in water prior to each contact with a soft bristle toothbrush. The toothpaste was mixed with water in the ratio of approximately 1 part toothpaste to 3 parts water. The specimens contact the toothbrush once each cycle, and the toothbrush is held against the specimen at a load of approximately 100 g to ensure contact. The specimen being rotated through the slurry on each cycle provides fresh abrasive for the brushing episode for each cycle.

While a person might brush their teeth for two minutes, twice per day, it is likely that each tooth surface is only experiencing the brushing for a fraction of this time. As an outside estimate (i.e. “worst case”), each tooth may be brushed for 1 minute per day. Based on an estimated brushing stroke in the oral cavity of 4 strokes per second, 1 minute of brushing would produce 240 strokes. Therefore, we will equate 240 seconds, or 4 minutes in the toothbrushing machine, with normal brushing exposure for a restoration per day.

The gloss was measured for each specimen after 1 episode of 90 minutes (i.e. equivalent to 22 days), at 180 minutes (i.e. equivalent to 45 days), and a final episode at 360 minutes (90 days total). Therefore, there were four measurement periods (baseline = maximum gloss, 90, 180, and 360 minutes) at which gloss was assessed. After each brushing cycle the specimens were rinsed, measured, and then brushed again. The final gloss values are compared among the composites with one-way Analysis of Variance (variable = composite), followed by post-hoc multiple comparison testing (Tukey’s) at a significance level of 0.05.

Results: Figure 8 below shows that the maximum gloss achieved using this method was very similar for the three materials: 76.8 for SDR Flow+, 80.2 for Sonicfill 3, and 84.0 for the Zest material. Statistically, there was a difference at baseline between the Zest material and SDR Flow+, with Zest having higher gloss. There was no difference between Zest and Sonicfill 3, or between Sonicfill 3 and SDR Flow+.

The gloss for each composite was reduced with toothbrushing, but there were differences between the materials.

After 90 minutes of brushing, the gloss of Zest (70.2) was greater than SDR Flow+ (52.7), but statistically equivalent to Sonicfill 3 (63.5). Sonicfill 3 was higher than SDR Flow+.

After 180 minutes of brushing, the gloss of Zest (68.1) had hardly declined, and was greater than SDR Flow+ (46.3) and Sonicfill 3 (52.3). There was no longer a difference between Sonicfill 3 and SDR Flow+.

After 360 minutes of brushing, the gloss of Zest (59.9) had declined, but it was still greater than SDR Flow+ (37.3), which was greater than Sonicfill 3 (25.5).

Thus, after 6 hours of tooth brushing, the Zest material maintained 71% of its initial gloss, which SDR Flow+ maintained 48.6% of its initial gloss, and Sonicfill 3 maintained only 31.7% of its initial gloss. The curves show a very linear loss of gloss for the Sonicfill 3 composite, while the Zest material and SDR Flow+ saw the majority of the loss in gloss during the initial 90 minute brushing, and then tended to stabilize somewhat.

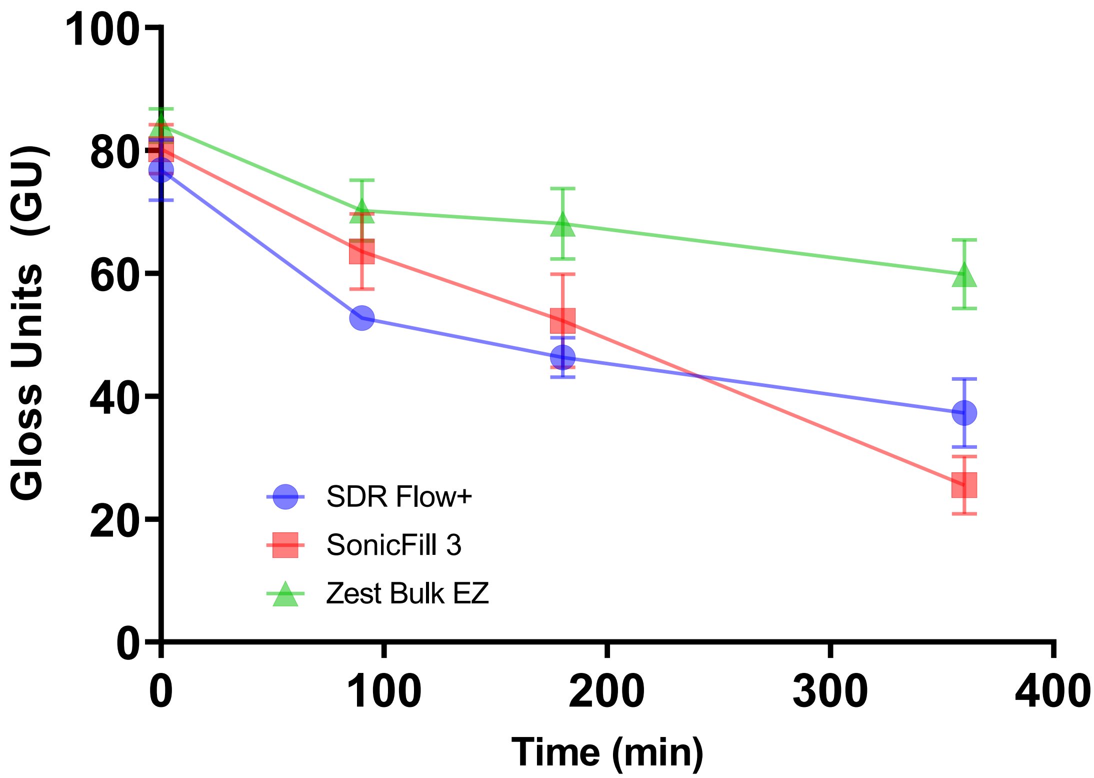

Figure 8. Gloss reduction during toothbrushing for all composite groups. Measurements were taken at time = 0, 90, 180 and 360 minutes. Each specimen was measured 3 times on the gloss meter at each time point, and 5 specimens were tested for each composite.