

Comparison of Flexural Properties of Bulk-fill Restorative/Flowable Composites and Their Conventional Counterparts

AH Eweis • AU Yap • NA Yahya

Clinical Relevance

Bulk-fill restorative resin-based composites (RBCs), though stiffer than their flowable and conventional counterparts, were mostly weaker. Bulk-fill restorative RBCs should thus be used with caution in areas of high flexural stresses and an overlying final layer of conventional composite may be still be prudent.

SUMMARY

The objectives of the study were to compare the flexural modulus and strength of restorative and flowable bulk-fill resin-based composites (RBCs) to their conventional counterparts and to determine the effects of conditioning environment on their flexural

properties. The materials evaluated included three conventional RBCs (Filtek Z350, Tetric N Ceram, and Beautifil II), three restorative bulk-fill RBCs (Filtek Bulk-Fill Restorative, Tetric N Ceram Bulk-Fill, and Beautifil Bulk-fill Restorative), as well as three flowable bulk-fill RBCs (Filtek Bulk-Fill Flowable, Tetric N Flow Bulk-Fill, and Beautifil Bulk-Fill Flowable). Specimens were fabricated using customized stainless-steel molds, finished, measured, and randomly divided into four groups. The various RBCs were conditioned in the following mediums (n=10) for seven days at 37°C: air, artificial saliva (SAGF), 0.02 N citric acid, and 50% ethanol-water solution. After conditioning, the specimens were rinsed, blotted dry, measured, and subjected to flexural testing using a universal testing machine. Data were subjected to statistical analysis using analysis of variance and the Tukey test at a significance level of $\alpha = 0.05$. Significant differences in flexural properties were observed between materials and conditioning mediums. Bulk-fill restorative RBCs exhibited higher flexural modulus than their bulk-fill flowable

Ahmed H Eweis, MDS, MFDS RCSEd, BDS, postgraduate, Department of Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia

Adrian U-Jin Yap, PhD, MSc, BDS, Grad Dip Psychotherapy, adjunct professor, Department of Dentistry, Ng Teng Fong General Hospital, National University Health System, Singapore; Department of Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia; and Faculty of Dentistry, National University of Singapore, Singapore

*Noor A Yahya, MDentSci, BDS, DipTrans, senior lecturer, Department of Restorative Dentistry, Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia

*Corresponding author: Department of Restorative Dentistry, Faculty of Dentistry, University of Malaya, 50603, Kuala Lumpur, Malaysia; E-mail: nazlin@um.edu.my

DOI: <http://doi.org/10.2341/18-133-L>

and conventional counterparts. With the exception of Filtek Bulk-Fill Flowable, bulk-fill flowable RBCs had significantly higher flexural strength than bulk-fill restorative and conventional RBCs. Flexural properties were highest when RBCs were conditioned in air and generally the lowest after exposure to ethanol.

INTRODUCTION

With advances in materials science and clinical techniques, the indications of resin-based composites (RBCs) have expanded to include large posterior stress-bearing restorations that were traditionally restored using amalgam.^{1,2} Nonetheless, the posterior RBC restorations are still technically challenging to perform due to the incremental layering technique and depth-of-cure issues.^{3,4} The incremental layering technique is time consuming and might lead to void formation between composite layers. Bulk-fill RBCs were developed to address the previously mentioned problems associated with conventional materials. They can be placed and cured in layers of up to 4 to 5 mm in thickness.⁵ The greater depth of cure is achieved by means of novel resins, modified initiator systems, polymerization boosters, unique fillers, and filler control.^{4,6,7} Moreover, special modulators and light-sensitive fillers are incorporated into some products to provide expanded working time by acting as a protective shield against operator and ambient lights. Bulk-fill RBCs come in either restorative (sculptable/packable) or flowable forms.⁵ Typically, flowable bulk-fill RBCs, with their lower filler content, are used as liners or bases in large class I/II restorations where they are placed and bulk cured in 4-mm increments and “capped” occlusally with more highly filled RBCs.^{2,8} On the other hand, restorative bulk-fill RBCs are more highly filled and can be used in stress-bearing situations without the need for another overlying final layer.⁹

The mechanical properties of bulk-fill RBCs have been the subject of some debate. While some authors have reported lower mechanical properties than conventional highly filled RBCs, others have reported values that are close to conventional materials.^{5,10,11} Prior data on curing efficiency had also been ambivalent, with some reporting depths of cure of more than 4 mm and others describing insufficient curing at 4-mm layers.¹²⁻¹⁶ The differences in mechanical properties as well as depth of cure may be attributed to variances in resin compositions, material translucency, viscosity, filler type, and content.⁸

Until now, a limited number of studies have investigated the flexural properties of bulk-fill

RBCs, and few, if any, have assessed the performance of bulk-fill giomers or prereacted glass (PRG) composites. Giomers are based on PRG technology where acid-reactive fluoride containing glass is prereacted with polyacids in the presence of water, freeze-dried, milled, silanized, ground, and used as fillers.⁴ PRG technology has been incorporated into many Shofu products, ranging from restoratives to bonding agents. Besides being biocompatible and having antiplaque formation properties, giomers can also release and recharge fluoride.^{17,18}

Physical properties of RBCs are well known to be affected by their surrounding chemical environment. Different chemicals from food substances can cause softening and dissolution of matrices of RBCs as well as filler damage, debonding, and leaching, resulting in decreased restoration durability and longevity.^{19,20} The use of dietary solvents permits the accelerated assessment of dental RBCs in short periods of time together with an appraisal of chemical affinity and the elution process.²¹ The chemical environment and food substances have been found to affect the viscoelastic properties of bulk-fill RBCs as well.²²

The objectives of the study were to compare the flexural modulus and strength of restorative and flowable bulk-fill RBCs to their conventional materials and to determine the effects of conditioning environment on the flexural properties of bulk-fill composites. The null hypotheses were as follows: 1) there are no differences in flexural modulus and strength between restorative and flowable bulk-fill RBCs as well as their conventional counterparts, and 2) the flexural properties of bulk-fill restorative and flowable RBCs are not affected by their conditioning environment.

METHODS AND MATERIALS

Materials and Specimen Preparation

Table 1 shows the materials evaluated and their technical profiles. They included three conventional RBCs (Filtek Z350 [FZ], Tetric N Ceram [TN], and Beautifil II [BT]), three restorative bulk-fill RBCs (Filtek Bulk-Fill Restorative [FB], Tetric N Ceram Bulk-Fill [TB], and Beautifil Bulk-fill Restorative [BB]), as well as three flowable bulk-fill RBCs (Filtek Bulk-Fill Flowable [FF], Tetric N Flow Bulk-Fill [TF], and Beautifil Bulk-Fill Flowable [BF]).

The International Organization for Standardization (ISO) recommends the use of specimens that are 25 × 2 × 2 mm for flexural testing.²³ Such specimens are challenging to fabricate without flaws and necessitate the use of multiple overlapping irradiation because of the comparably smaller

Table 1: Technical Profiles and Manufacturers of the Materials Evaluated

Manufacturer	Material (Abbreviation)	Type	Resin (Photoinitiator)	Filler	Filler Content % by weight/% by Volume	Lot Number
3M ESPE (St Paul, MN, USA) [A]	Filtek Z350 (FZ)	Conventional nanohybrid composite	Bis-GMA Bis-EMA UDMA TEGDMA (CQ)	Zirconia/silica cluster and silica nanoparticle	78.5/63.3	N771467
	Filtek Bulk-Fill Restorative (FB)	Bulk-fill restorative composite	AUDMA AFM DDDMA UDMA (CQ)	Zirconia/silica cluster, ytterbium trifluoride	76.5/58.4	N693019
	Filtek Bulk-Fill Flowable (FF)	Flowable bulk-fill composite	Bis-GMA Bis-EMA UDMA (CQ, EDMAB)	Zirconia/silica, ytterbium trifluoride	64.5/42.5	N884479
Ivoclar Vivadent Inc (Amherst, NY, USA) [B]	Tetric N Ceram (TN)	Conventional microhybrid composite	Bis-GMA Bis-EMA UDMA (CQ)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide, prepolymers	81.2/57	V35260
	Tetric N Ceram Bulk-Fill (TB)	Bulk-fill restorative composite	Bis-GMA Bis-EMA UDMA (CQ, TPO, Ivocerin)	Barium glass filler, ytterbium fluoride, spherical mixed oxide	79/60	S38368
	Tetric N Flow Bulk-Fill (TF)	Flowable bulk-fill composite	Bis-GMA UDMA TEGDMA (Ivocerin)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide	64.9/NA	V49336
Shofu Inc (Kyoto, Japan) [C]	Beautifil II (BT)	Conventional giomer	Bis-GMA TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	83.3/68.8	31731
	Beautifil-Bulk Restorative (BB)	Bulk-fill restorative giomer	Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	87/74.5	51623
	Beautifil-Bulk Flowable (BF)	Flowable bulk-fill giomer	Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	73/60	101615

Abbreviations: Bis-GMA, bisphenol-A glycidyl methacrylate; Bis-EMA, ethoxylated bisphenol-A-glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; CQ, camphorquinone; AUDMA, aromatic urethane dimethacrylate; AFM, addition-fragmentation monomers; DDDMA, 1,12-dodecanediol dimethacrylate; EDMAB, ethyl 4-dimethyl aminobenzoate; TPO, 2,4,6-trimethylbenzoyl diphenylphosphine oxide; NA, not available; S-PRG, surface-modified prereacted glass; F-Br-Al-Si, fluoroboroaluminosilicate; Bis-MPEPP, bisphenol-A polyethoxy-dimethacrylate.

light exit windows of curing tips.²⁴ Moreover, these ISO specimens are not clinically relevant since the mesio-distal widths of molars are around 11 mm, and the cervico-incisal length of central incisors usually do not exceed 13 mm.²⁵ The miniflexural test, employing 12-mm specimens, was selected due to their significant correlation to the ISO flexural test, clinical relevance, and better efficiency.^{26,27} Forty beam-shaped test specimens (12×2×2 mm) of each of the various RBCs were fabricated using customized stainless-steel molds. The conventional

and bulk-fill RBCs were placed in a single increment, while the flowable materials were injected into the molds. Excess material was removed by compressing the molds between two Mylar strips with glass slides. The top surface of the specimens were light polymerized through the glass slide with two overlapping irradiations of 10 seconds each using a calibrated LED curing light (Demi Plus, Kerr, Orange, CA, USA) with an output irradiance of 1330 mW/cm² and wavelength range of 450 to 470 nm. The glass slides were removed, and the

specimens were light cured for another 10 seconds. The Mylar strips were subsequently discarded, and the composite beams were removed from their molds. Any minor material excess, or “fins,” was gently removed by fine polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA). The composite specimens were visually examined for the presence of voids, and any defective specimens were replaced. The final dimensions of the specimens and the parallelism between their opposite surfaces were verified with a digital caliper (Mitutoyo Corp, Kawasaki, Japan).

Conditioning Mediums and Time

Specimens of the various materials were randomly divided into four groups of 10 ($n=10$) and conditioned in the following mediums for seven days at 37°C: air (control), artificial saliva (SAGF),²⁸ 0.02 N citric acid, and 50% ethanol-water solution. Containers used to house the various solutions were sealed to minimize evaporation and stored in air within an incubator (IN-450, Memmert, Schwabach, Germany). The pH of the artificial saliva was verified via a digital pH meter (pH2700, Eutech, Singapore) and adjusted to 6.8 with diluted hydrochloric acid (where needed) to resemble natural saliva pH when it is released from the salivary ducts.²¹

Flexural Testing

After the seven-day conditioning period, the composite specimens were loaded until fracture using a universal testing machine (Shimadzu Corp, Kyoto, Japan) with a load cell of 5 KN and crosshead speed of 0.5 mm/min. Flexural strength, σ , in megapascals (MPa), was calculated using the following equation:

$$\sigma = \frac{3PL}{2BH^2}$$

where P is the maximum load exerted on the specimen in newtons, L is the distance between the supports in millimeters (10 mm), B is the width of the specimen in millimeters, and H is the height of the specimen in millimeters.

Flexural modulus, E' , in MPa, was calculated using the following equation:

$$E' = \left(\frac{F}{D}\right) \left(\frac{L^3}{4BH^3}\right)$$

where F/D is the slope, in newtons per millimeter, measured in the straight-line portion of the load-

deflection graph; L, B, and H are defined in the flexural strength equation. Flexural modulus was subsequently converted to gigapascals (GPa).

Statistical Analysis

The SPSS statistical program (version 12.0.1, SPSS Inc, Chicago, IL, USA) was used to analyze the data obtained. Normality testing was done using the Shapiro-Wilk test, and parametric data analysis was permissible, as data were normally distributed. Homogeneity of variance was assessed using the Levene test, and equal variances were assumed. The interactions between the independent variables (materials and conditioning mediums) and each of the dependent variables (flexural modulus and flexural strength) were evaluated using two-way analysis of variance (ANOVA). One-way ANOVA, followed by Tukey *post hoc* tests, was used to determine intermedium and intermaterial differences for flexural modulus and strength for the same material type from different manufacturers as well as for different material types from the same manufacturer. All statistical analyses were carried out at significance level of $\alpha = 0.05$.

RESULTS

Mean flexural modulus and strength for the various RBCs after conditioning in the different mediums are reflected in Table 2. Figures 1 and 2 compare the mean flexural modulus and strength between mediums for each material. Statistical comparisons of mean flexural properties between RBCs when grouped by manufacturers and material type after conditioning in the various mediums are summarized in Tables 3 and 4, respectively. Two-way ANOVA presented significant interactions ($p < 0.001$) between materials and mediums for both flexural modulus and strength.

Comparison Between Material Types by Manufacturers

Manufacturer (A) RBCs—With the exception of air, FB had the highest flexural modulus for all mediums. FZ showed the highest flexural modulus in air. For all mediums, FF had significantly lower flexural modulus than FZ and FB. The highest flexural strength was observed with FB for all mediums. FZ had the lowest flexural strength for all mediums aside from air, where FF was the lowest. There was, however, no significant difference between the three different manufacturer (A) RBCs when conditioned in air.

Table 2: Mean Flexural Modulus (GPa) and Flexural Strength (MPa) of the Various Resin-Based Composites (Standard Deviations in Parentheses)

Medium/Material (Abbreviation)	Flexural Modulus (GPa)				Flexural Strength (MPa)			
	Air	Artificial Saliva	Citric Acid	Ethanol	Air	Artificial Saliva	Citric Acid	Ethanol
Filtek Z350 (FZ)	8.23 (0.89)	6.58 (0.76)	6.38 (0.78)	6.89 (0.94)	135.20 (17.08)	91.71 (10.10)	89.03 (11.84)	62.50 (7.56)
Filtek Bulk-Fill Restorative (FB)	8.04 (1.11)	7.64 (1.07)	8.00 (1.05)	7.25 (0.99)	144.00 (19.32)	122.39 (16.63)	115.26 (10.51)	120.94 (12.75)
Filtek Bulk-Fill Flowable (FF)	3.59 (0.28)	3.52 (0.39)	3.06 (0.17)	1.91 (0.15)	127.89 (7.19)	101.09 (12.43)	105.13 (11.86)	66.56 (5.33)
Tetric N Ceram (TN)	5.22 (0.32)	4.62 (0.28)	4.01 (0.23)	2.94 (0.18)	109.84 (10.34)	86.08 (12.10)	89.53 (7.05)	60.25 (4.45)
Tetric N Ceram Bulk-Fill (TB)	6.51 (0.81)	5.72 (0.49)	4.86 (0.56)	3.33 (0.43)	106.85 (6.80)	99.17 (8.89)	93.20 (8.23)	55.77 (4.78)
Tetric N Flow Bulk-Fill (TF)	4.56 (0.29)	3.89 (0.29)	4.05 (0.39)	3.10 (0.33)	119.73 (11.72)	106.92 (7.74)	98.06 (10.01)	82.56 (5.04)
Beautifil II (BT)	6.66 (0.93)	6.51 (0.55)	6.43 (0.47)	5.82 (0.34)	110.36 (13.40)	79.50 (8.79)	86.94 (9.71)	68.26 (8.02)
Beautifil-Bulk Restorative (BB)	8.19 (1.12)	7.34 (0.92)	5.93 (0.84)	6.80 (0.72)	117.53 (10.22)	86.60 (3.57)	87.23 (8.06)	85.76 (6.86)
Beautifil-Bulk Flowable (BF)	5.56 (0.48)	5.51 (0.42)	5.21 (0.48)	4.47 (0.38)	113.91 (11.89)	105.56 (12.01)	100.07 (11.54)	99.81 (11.98)

Manufacturer (B) RBCs—TB had the highest flexural modulus for all mediums. TF showed the lowest flexural modulus when conditioned in air and artificial saliva, whereas TN presented the lowest modulus in citric acid and ethanol. The highest flexural strength was observed with TF for all mediums. TB showed the lowest flexural strength

when conditioned in air and ethanol, whereas TN was the lowest in artificial saliva and citric acid. Nonetheless, no significant difference in strength was observed between the three different manufacturer (B) RBCs when conditioned in citric acid.

Manufacturer (C) RBCs—Besides citric acid, BB had the highest flexural modulus in all mediums. BT

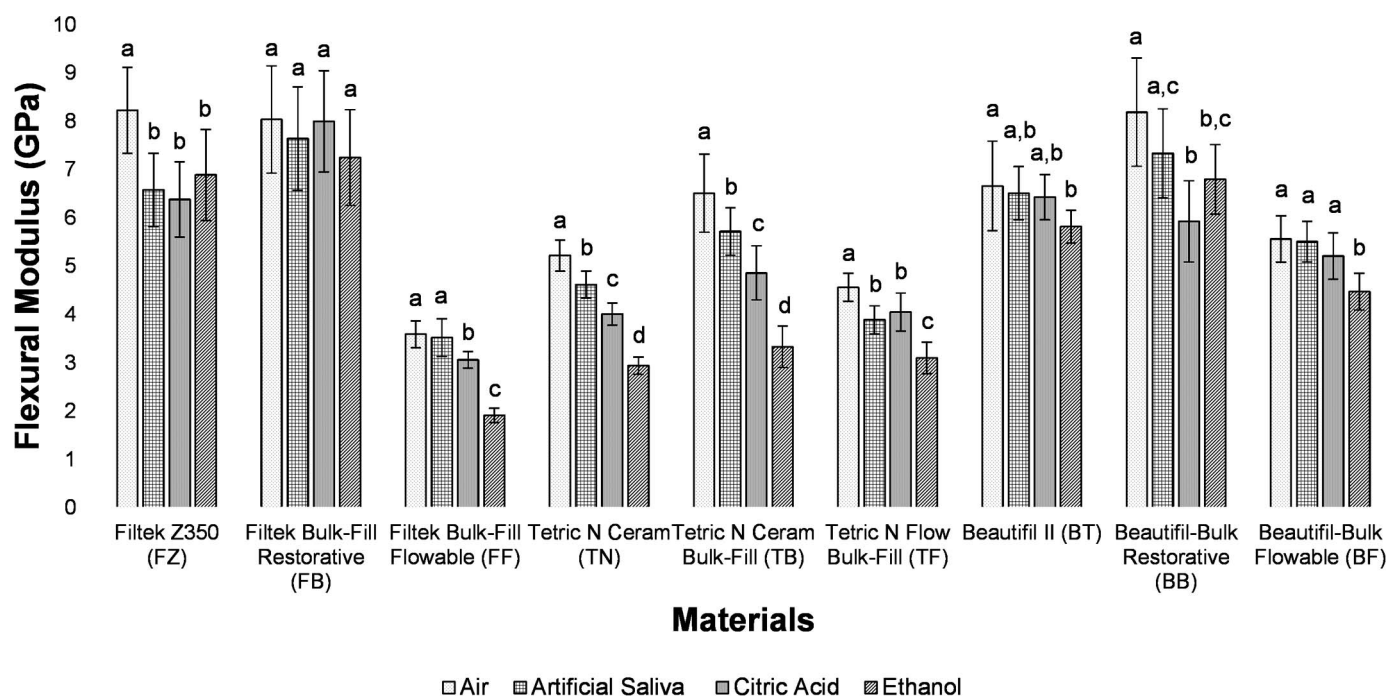


Figure 1. Mean flexural modulus values (GPa) for the different materials after storage in the different mediums. Results of one-way analysis of variance and post hoc Tukey test ($p < 0.05$). Same letters above the bars indicate no statistical significance between different mediums for each material.

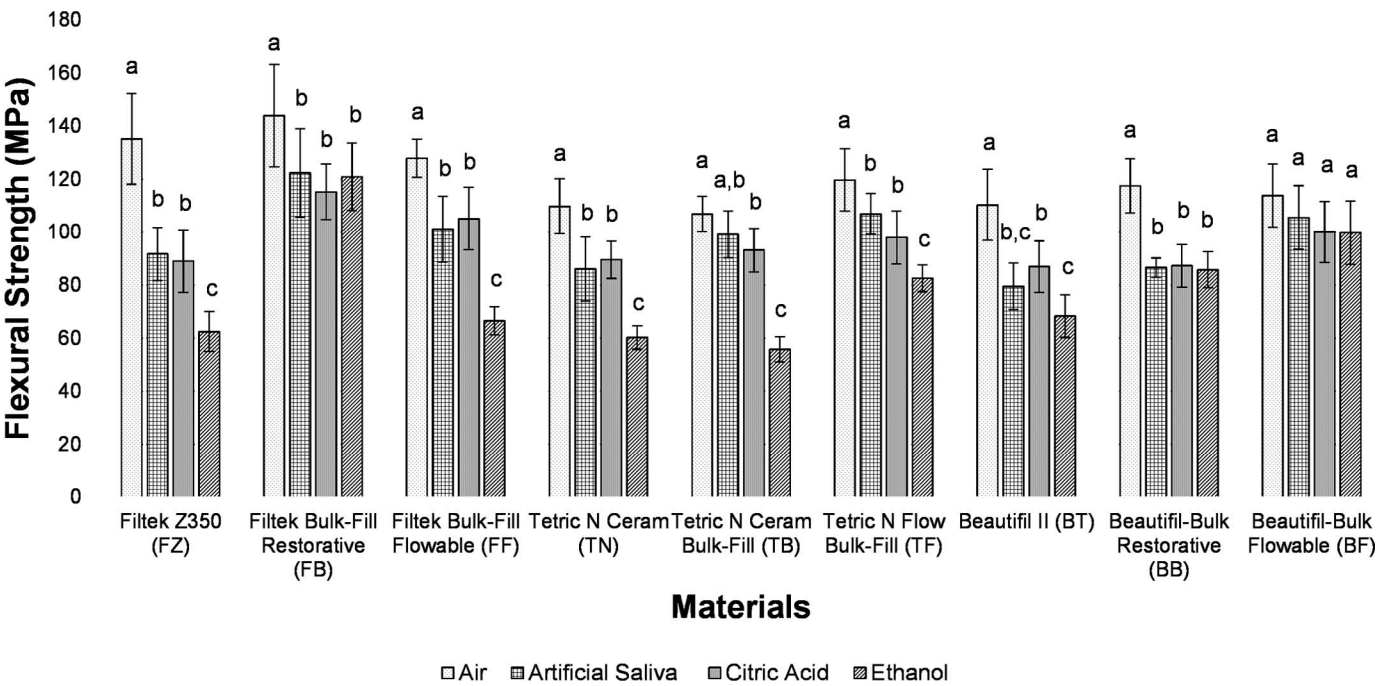


Figure 2. Mean flexural strength values (MPa) for the different materials after storage in the different mediums. Results of one-way analysis of variance and post hoc Tukey test (p<0.05). Same letters above the bars indicate no statistical significance between different mediums for each material.

showed the highest flexural modulus in citric acid. Regardless of mediums, BF had significantly lower modulus than BT and BB. With the exemption of air, BF had significantly higher flexural strength than the other two giomers. BB showed the highest flexural strength in air. However, no significant difference in flexural strength was observed between the three different manufacturer (C) RBCs when conditioned in air. BT had the lowest flexural strength for all mediums.

Comparison Between Different Manufacturers for Each RBC Type

Conventional RBCs—With the exception of citric acid, FZ had the highest flexural modulus for all mediums. BT presented the highest flexural modulus in citric acid. TN had significantly lower flexural modulus than FZ and BT for all mediums. FZ had the highest flexural strength when conditioned in air and artificial saliva, whereas TN and BT displayed the highest flexural strength when conditioned in citric

Table 3: Comparison of Mean Flexural Modulus and Strength Between Resin-Based Composites Grouped by Manufacturer After Conditioning in the Various Mediums ^a				
Properties	Medium	Differences		
		A	B	C
Flexural modulus	Air	FZ, FB > FF	TB > TN > TF	BB > BT > BF
	Artificial saliva	FB > FZ > FF	TB > TN > TF	BB > BT > BF
	Citric acid	FB > FZ > FF	TB > TF, TN	BT, BB > BF
	Ethanol 50%	FB, FZ > FF	TB > TN	BB > BT > BF
Flexural strength	Air	Nonsignificant	TF > TB	Nonsignificant
	Artificial saliva	FB > FF, FZ	TF, TB > TN	BF > BB, BT
	Citric acid	FB, FF > FZ	Nonsignificant	BF > BB, BT
	Ethanol 50%	FB > FF, FZ	TF > TN, TB	BF > BB > BT
Abbreviations: FZ, Filtek Z350; FB, Filtek Bulk-Fill Restorative; FF, Filtek Bulk-Fill Flowable; TB, Tetric N Ceram Bulk-Fill; TN, Tetric N Ceram; TF, Tetric N Flow Bulk-Fill; BB, Beautifil-Bulk Restorative; BT, Beautifil II; BF, Beautifil-Bulk Flowable.				
^a Results of one-way analysis of variance and post hoc Tukey test (p<0.05); > indicates statistical significance.				

Table 4: Comparison of Mean Flexural Modulus and Strength Between Resin-Based Composites Grouped by Material Type After Conditioning in the Various Mediums^a

Properties	Medium	Differences		
		Conventional	Bulk-Fill Restorative	Bulk-Fill Flowable
Flexural modulus	Air	FZ > BT > TN	BB, FB > TB	BF > TF > FF
	Artificial saliva	FZ, BT > TN	FB, BB > TB	BF > TF, FF
	Citric acid	BT, FZ > TN	FB > BB > TB	BF > TF > FF
	Ethanol 50%	FZ > BT > TN	FB, BB > TB	BF > TF > FF
Flexural strength	Air	FZ > BT, TN	FB > BB, TB	FF > BF
	Artificial saliva	FZ > BT	FB > TB > BB	Nonsignificant
	Citric acid	Nonsignificant	FB > TB, BB	Nonsignificant
	Ethanol 50%	BT > TN	FB > BB > TB	BF > TF > FF

Abbreviations: FZ, Filtek Z350; BT, Beautifil II; TN, Tetric N Ceram; BB, Beautifil-Bulk Restorative; FB, Filtek Bulk-Fill Restorative; TB, Tetric N Ceram Bulk-Fill; BF, Beautifil-Bulk Flowable; TF, Tetric N Flow Bulk-Fill; FF, Filtek Bulk-Fill Flowable.

^a Results of one-way analysis of variance and post hoc Tukey test ($p < 0.05$); > indicates statistical significance.

acid and ethanol, respectively. TN showed the lowest flexural strength when conditioned in air and ethanol, while BT had the lowest strength in artificial saliva and citric acid. No significant difference in strength was observed between the three conventional materials when conditioned in citric acid.

Bulk-Fill Restorative RBCs—With the exception of air, FB had the highest flexural modulus. BB showed the highest flexural modulus in air. TB had significantly lower flexural modulus than FB and BB for all mediums. FB had significantly higher flexural strength than BB and TB for all mediums. TB showed the lowest flexural strength when conditioned in air and ethanol, whereas BB had the lowest strength in artificial saliva and citric acid.

Bulk-Fill Flowable RBCs—BF had significantly higher flexural modulus than TF and FF for all mediums. FF showed the lowest flexural modulus for all mediums. However, FF presented the highest flexural strength when conditioned in air and citric acid, whereas TF and BF had the highest flexural strength when conditioned in artificial saliva and ethanol, respectively. On the other hand, FF had the lowest flexural strength when conditioned in artificial saliva and ethanol, while TF and BF showed the lowest flexural strength when conditioned in citric acid and air, respectively. However, there was no significant difference between the three different bulk-fill flowable materials when conditioned in artificial saliva and citric acid.

Comparison Between Conditioning Mediums

Manufacturer (A) RBCs—Conditioning in air showed the highest flexural modulus and strength for all manufacturer (A) materials. The lowest flexural modulus and strength were generally ob-

served after conditioning in ethanol. Conditioning in citric acid showed the lowest flexural modulus for FZ and the lowest flexural strength for FB. No significant difference in flexural modulus was observed between mediums for FB. For FZ, conditioning in air resulted in significantly higher flexural modulus than all other mediums, whereas no significant difference was observed between ethanol, artificial saliva, and citric acid. Flexural modulus of FF after conditioning in air and artificial saliva was significantly higher than in citric acid, which in turn was significantly higher than in ethanol. For all manufacturer (A) RBCs, no significant difference in flexural strength was noted between conditioning in artificial saliva and citric acid. When conditioned in artificial saliva and citric acid, FZ and FF showed significantly higher flexural strength than in ethanol.

Manufacturer (B) RBCs—Conditioning in air again resulted in the highest flexural modulus and strength. The lowest flexural modulus and strength were obtained after conditioning in ethanol. For TN and TB, significant differences in flexural modulus was noted between the four mediums. For TF, conditioning in air showed significantly higher flexural modulus than in saliva and citric acid, which in turn was greater than in ethanol. There was no significant difference in flexural strength between artificial saliva and citric acid for all manufacturer (B) RBCs. When conditioned in these mediums, all materials showed significantly higher flexural strength than ethanol.

Manufacturer (C) RBCs—Conditioning in air showed the highest flexural modulus and strength for all manufacturer (C) RBCs. The lowest flexural modulus and strength were observed after conditioning in ethanol except for BB, which had the

lowest flexural modulus after conditioning in citric acid. For BB, conditioning in citric acid resulted in significantly lower flexural modulus than in air and artificial saliva. Flexural modulus after conditioning in air was significantly higher than in ethanol for BT, whereas for BF, it was significantly lower than all other mediums when conditioned in ethanol. There was no significant difference in flexural strength between artificial saliva and citric acid for all giomers. For BT, conditioning in ethanol resulted in significantly lower flexural strength than citric acid. No significant difference in flexural strength was observed between all mediums for BF.

DISCUSSION

The current study investigated the differences in the flexural properties between bulk-fill and conventional RBCs and the effect of the conditioning environment on the different materials. As flexural properties were material and conditioning medium dependent, both the null hypotheses were rejected. The different RBCs were immersed continually in the various conditioning mediums for seven days at 37°C before flexural testing was performed. This conditioning period might appear considerably long since restorations come into contact with foods and liquids infrequently and for short durations intraorally. Continuous exposure may, however, take place when chemicals are absorbed by food particles or calculus at the margins or grooves of restorations.^{29,30}

Flexural testing is widely used in characterizing RBCs since it determines both flexural modulus and strength.²³ Flexural modulus describes the stiffness of RBCs, whereas flexural strength represents the maximum stress that RBCs can be subjected to prior to failure. Flexural testing yields complex tensions arising from the combination of compression, shear, and tensile stresses.²⁴ The variation between the flexural properties of various RBCs is useful in the different clinical situations.^{31,32} For example, in class I, II, III, and IV cavities, RBCs with high flexural properties are usually selected to minimize fracture or deformation under the high occlusal forces, while in class V cavities, RBCs having low flexural modulus are preferred, as they can flex with the teeth during function and parafunction, which in turn reduces the stresses at the adhesive interface and decreases the chances of debonding.^{31,33}

Bulk-fill restorative RBCs were generally stiffer than their bulk-fill flowable and conventional counterparts. This may be attributed to the similar or higher filler content of the bulk-fill restoratives in comparison to the other RBCs. Results corroborated

those of El-Safty and others,³⁴ who reported a significant positive correlation between modulus and filler loading. FZ had a higher filler content than FB, which explains the higher flexural modulus of FZ in air. Weak intraoral acids, such as citric acid, have been reported to degrade the inorganic fillers in RBCs.³⁰ This might explain why BB, with its relatively higher inorganic and prereacted glass ionomer filler content, was somewhat more susceptible to modulus degradation than BT. The flexural modulus of the bulk-fill flowable RBCs were mostly significantly lower than their bulk-fill restorative and conventional counterparts. This was consistent with the work of Jung and others.³⁵ These authors suggested that bulk-fill flowable RBCs, with their lower modulus, may not provide an effective buffer to occlusal stress and recommended that they be capped with conventional materials. In high-stress-bearing areas, RBCs of higher stiffness are required to prevent restoration deformation, which could accelerate marginal and restoration failures. In addition to modulus, other physical properties of RBCs, including strength, fracture, and wear resistance, must also be considered for stress-bearing situations. However, with their greater flexibility, bulk-fill flowable RBCs are preferred over bulk-fill restorative and conventional materials in deep class V cavities, as they appear to offer better marginal adaptation.³⁶

For materials from manufacturer (A), the bulk-fill restorative had higher flexural strength than the other RBCs. However, for manufacturer (B) and (C) materials, bulk-fill flowable RBCs were generally stronger than their bulk-fill restorative and conventional counterparts despite their relatively lower filler loading. These results contradicted those of Tomaszewska and others³⁵ and Jung and others,³⁷ which reported that bulk-fill flowable RBCs have lower mechanical properties when compared to either highly filled nanohybrid or bulk-fill restorative RBCs. The variance in results may be attributed in part to the differences in bulk-fill flowable RBCs evaluated and experimental designs. Commercial flowable composites have a wide range of filler loading from 52% to 68% weight.³⁸ The higher flexural strength of the bulk-fill flowable RBCs evaluated in the present study when compared to bulk-fill restorative and conventional materials could be attributed to their relatively high filler loading, resiliency, and ability to withstand higher stress prior to fracture (Table 1).

When comparing different products, TN and TB from manufacturer (B) and FF from manufacturer (A) had significantly lower modulus than the other conventional, bulk-fill restorative and bulk-fill flow-

able RBCs regardless of conditioning mediums. The significantly lower modulus of these RBCs may be attributed to their relatively lower filler loading, disparities in fillers, and resin matrices used. As for flexural strength, no obvious trends were observed when conventional and bulk-fill flowable RBCs were compared. Differences between products varied with conditioning mediums, highlighting the importance of conducting flexural testing with different dietary solvents. Variances can again be attributed to differences in filler and resin content/type employed. For bulk-fill restorative materials, FB from manufacturer (A) was significantly stronger than both TN and BB from manufacturers (B) and (C), respectively. FB contains two novel monomers: a high-molecular-weight aromatic dimethacrylate and addition fragmentation monomers that act to decrease the polymerization shrinkage stress.³⁹ According to the manufacturer, aromatic dimethacrylate decreases the reactive groups in the resin, controlling the volumetric shrinkage and the rigidity of the final polymeric matrix, whereas addition fragmentation monomers contain a third reactive site that cleaves through a fragmentation process. This in turn helps provide a relaxation mechanism of the network being developed, leading to stress relief. The fragments can still react with each other or with other reactive sites ensuring that the physical properties of the material are preserved. This might play a role in developing shorter and stiffer polymeric chains, leading to a more rigid and stronger bulk-fill RBC.⁴⁰

When storage environments were compared, conditioning in air presented the highest flexural modulus and strength for all RBCs regardless of their type or manufacturer. Conditioning in air does not result in the leaching out of silica and filler particles that occurs with storage in aqueous mediums.²⁹ The lowest flexural modulus was observed when the RBCs were conditioned in ethanol with the exception of FZ and BB, which showed the lowest flexural modulus after storage in citric acid. Other than FB, which was weakest after storage in citric acid, the lowest flexural strength was also observed with conditioning in ethanol for all RBCs. The effect of citric acid on RBCs has already been elaborated on. Ethanol is known to soften the resin matrix of RBCs by removing unreacted monomers and linear polymers from the polymeric structure.^{24,29} With resin dissolution, filler exposure and dislodgement may ensure weakening the RBCs. The dietary habits of patients should thus be considered during material selection to enhance the clinical longevity of composite restorations.

The current study can be improved in some areas. To begin with, the conditioning period could be extended to determine the longer-term effects of conditioning environment on flexural properties.^{21,41} Static flexural testing that was carried out in the present study cannot provide insights into material structure, as dental RBCs are viscoelastic in nature and exhibit both viscous and elastic characteristics when undergoing deformation. Dynamic testing with dynamic mechanical analysis can be performed to better assess the viscoelastic properties of the RBCs.⁴² Dynamic mechanical analysis can be carried out using various frequencies, temperatures, and displacements that resemble the variations of forces and temperatures in the oral cavity. Moreover, unlike static testing, dynamic testing enables retesting of specimens over extended time periods, as it is nondestructive.^{22,43} As flowable RBCs are not a homogeneous group of materials, appraisal of their rheological properties and correlating this to their flexural properties may be beneficial.

CONCLUSIONS

Within the limitations of this study, the following can be concluded:

- Flexural properties of bulk-fill restorative, bulk-fill flowable, and conventional RBCs were both material and conditioning medium dependent.
- Bulk-fill restorative RBCs were generally stiffer than their bulk-fill flowable and conventional counterparts.
- With the exception of FF, bulk-fill flowable RBCs were stronger than their bulk-fill restorative and conventional counterparts.
- While no patterns were observed for flexural strength, manufacturer (B) bulk-fill and conventional RBCs were less rigid than comparable products from manufacturers (A) and (C).
- Conditioning in air resulted in the highest flexural properties, while exposure to ethanol generally presented the lowest.

Acknowledgements

This work was supported by University of Malaya special grant (BKS010-2017). The authors would like to thank 3M ESPE and Shofu Asia for their material support.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 12 December 2018)

REFERENCES

- Ferracane JL (2011) Resin composite—State of the art *Dental Materials* **27**(1) 29-38.
- Fugolin APP & Pfeifer CS (2017) New resins for dental composites *Journal of Dental Research* **96**(10) 1085-1091.
- Versluis A, Douglas WH, Cross M, & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75**(3) 871-878.
- Yap AU, Pandya M, & Toh WS (2016) Depth of cure of contemporary bulk-fill resin-based composites *Dental Materials Journal* **35**(3) 503-510.
- Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, & Leloup G (2014) Physico-mechanical characteristics of commercially available bulk-fill composites *Journal of Dentistry* **42**(8) 993-1000.
- Fleming GJP, Awan M, Cooper PR, & Sloan AJ (2008) The potential of a resin-composite to be cured to a 4 mm depth *Dental Materials* **24**(4) 522-529.
- Lassila LVJ, Nagas E, Vallittu PK, & Garoushi S (2012) Translucency of flowable bulk-filling composites of various thicknesses *Chinese Journal of Dental Research* **15**(1) 31-35.
- Jang JH, Park SH, & Hwang IN (2015) Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin *Operative Dentistry* **40**(2) 172-180.
- Hirata R, Kabbach W, De Andrade OS, Bonfante EA, Giannini M, & Coelho PG (2015) Bulk fill composites: An anatomic sculpting technique *Journal of Esthetic and Restorative Dentistry* **27**(6) 335-343.
- Ilie N, Bucuta S, & Draenert M (2013) Bulk-fill resin-based composites: An in vitro assessment of their mechanical performance *Operative Dentistry* **38**(6) 618-625.
- El Gezawi M, Kaisarly D, Al-Saleh H, ArRejaie A, Al-Harbi F, & Kunzelmann K (2016) Degradation potential of bulk versus incrementally applied and indirect composites: Color, microhardness, and surface deterioration *Operative Dentistry* **41**(6) 195-208.
- Bucuta S & Ilie N (2014) Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites *Clinical Oral Investigations* **18**(8) 1991-2000.
- Garcia D, Yaman P, Dennison J, & Neiva GF (2014) Polymerization shrinkage and depth of cure of bulk fill flowable composite resins *Operative Dentistry* **39**(4) 441-448.
- Czasch P & Ilie N (2013) In vitro comparison of mechanical properties and degree of cure of bulk fill composites *Clinical Oral Investigations* **17**(1) 227-235.
- Alrahlah A, Silikas N, & Watts DC (2014) Post-cure depth of cure of bulk fill dental resin-composites *Dental Materials* **30**(2) 149-154.
- Ilie N, Kessler A, & Durner J (2013) Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites *Journal of Dentistry* **41**(8) 695-702.
- Itota T, Carrick TE, Yoshiyama M, & McCabe JF (2004) Fluoride release and recharge in giomer, compomer and resin composite *Dental Materials* **20**(9) 789-795.
- Saku S, Kotake H, Scougall-Vilchis RJ, Ohashi S, Hotta M, Horiuchi S, Hamada K, Asaoka K, Tanaka E, & Yamamoto K (2010) Antibacterial activity of composite resin with glass-ionomer filler particles *Dental Materials Journal* **29**(2) 193-198.
- Wu W, Toth EE, Moffa JF, & Ellison JA (1984) Subsurface damage layer of in vivo worn dental composite restorations *Journal of Dental Research* **63**(5) 675-680.
- Drummond JL (2008) Degradation, fatigue, and failure of resin dental composite materials *Journal of Dental Research* **87**(8) 710-719.
- Vouvoudi EC & Sideridou ID (2012) Dynamic mechanical properties of dental nanofilled light-cured resin composites: Effect of food-simulating liquids *Journal of the Mechanical Behavior of Biomedical Materials* **10** 87-96.
- Eweis AH, Yap AU-J, & Yahya NA (2017) Dynamic analysis of bulk-fill composites: Effect of food-simulating liquids *Journal of the Mechanical Behavior of Biomedical Materials* **74C** 183-188.
- International Organization for Standardization (2009) ISO 4049 Dentistry polymer-based filling, restorative and luting materials *Geneve: International Organization for Standardization* 15-18.
- dos Santos SG, Moyses MR, Alcantara CEP, Ribeiro JCR, & Ribeiro JGR (2012) Flexural strength of a composite resin light cured with different exposure modes and immersed in ethanol or distilled water media *Journal of Conservative Dentistry* **15**(4) 333-336.
- Wheeler RC (1965) *A Textbook of Dental Anatomy and Physiology* WB Saunders, Philadelphia PA.
- Yap AU & Teoh SH (2003) Comparison of flexural properties of composite restoratives using the iso and mini-flexural tests *Journal of Oral Rehabilitation* **30**(2) 171-177.
- Yap AU, Eweis AH, & Yahya NA (2018) Dynamic and static flexural appraisal of resin-based composites: Comparison of the ISO and mini-flexural tests *Operative Dentistry* **43**(5) e223-e231.
- Gal JY, Fovet Y, & Adib-Yadzi M (2001) About a synthetic saliva for in vitro studies *Talanta* **53**(6) 1103-1115.
- Yap AU, Tan DTT, Goh BKC, Kuah HG, & Goh M (2000) Effect of food-simulating liquids on the flexural strength of composite and polyacid-modified composite restoratives *Operative Dentistry* **25**(3) 202-208.
- Akova T, Ozkomur A, & Uysal H (2006) Effect of food-simulating liquids on the mechanical properties of provisional restorative materials *Dental Materials* **22**(12) 1130-1134.

31. Yap AU, Chandra S, Chung S, & Lim C (2002) Changes in flexural properties of composite restoratives after aging in water *Operative Dentistry* **27**(5) 468-474.
32. Rodrigues SAJ, Zanchi CH, Carvalho RV, & Demarco FF (2007) Flexural strength and modulus of elasticity of different types of resin-based composites *Brazilian Oral Research* **21**(1) 16-21.
33. Pontes LF, Alves EB, Alves BP, Ballester RY, Dias CGBT, & Silva CM (2013) Mechanical properties of nanofilled and microhybrid composites cured by different light polymerization modes *General Dentistry* **61**(3) 30-33.
34. El-Safty S, Akhtar R, Silikas N, & Watts DC (2012) Nanomechanical properties of dental resin-composites *Dental Materials* **28**(12) 1292-1300.
35. Jung J & Park S (2017) Comparison of polymerization shrinkage, physical properties, and marginal adaptation of flowable and restorative bulk fill resin-based composites *Operative Dentistry* **42**(4) 375-386.
36. Szesz A, Parreiras S, Martini E, Reis A, & Loguercio A (2017) Effect of flowable composites on the clinical performance of non-carious cervical lesions: A systematic review and meta-analysis *Journal of Dentistry* **65** 11-21.
37. Tomaszewska IM, Kearns JO, Ilie N, & Fleming GJP (2015) Bulk fill restoratives: To cap or not to cap—That is the question? *Journal of Dentistry* **43**(3) 309-316.
38. Moszner N & Salz U (2001) New developments of polymeric dental composites *Progress in Polymer Science* **26**(4) 535-576.
39. Kalliecharan D, Gernscheid W, Price RB, Stansbury J, & Labrie D (2016) Shrinkage stress kinetics of bulk fill resin-based composites at tooth temperature and long time *Dental Materials* **32**(11) 1322-1331.
40. Braga RR, Ballester RY, & Ferracane JL (2005) Factors involved in the development of polymerization shrinkage stress in resin-composites: A systematic review *Dental Materials* **21**(10) 962-970.
41. Mesquita RV, Axmann D, & Geis-Gerstorfer J (2006) Dynamic visco-elastic properties of dental composite resins *Dental Materials* **22**(3) 258-267.
42. Jacobsen PH & Darr AH (1997) Static and dynamic moduli of composite restorative materials *Journal of Oral Rehabilitation* **24**(4) 265-273.
43. Mesquita RV & Geis-Gerstorfer J (2008) Influence of temperature on the visco-elastic properties of direct and indirect dental composite resins *Dental Materials* **24**(5) 623-632.