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Dynamic Viscoelastic Characterization of Bulk-fill Resin-based Composites and Their Conventional Counterparts

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Clinical Relevance

The viscoelastic properties of bulk-fill resin-based composites (RBCs) were affected by aqueous solutions and are often inferior to their conventional counterparts. The clinical use of conventional RBCs are generally preferred over bulk-fill materials for high-stress-bearing situations.

SUMMARY

This study compared the viscoelastic properties of restorative and flowable bulk-fill resin-based composites (RBCs) with their conventional counterparts and evaluated the impact of aqueous solutions on viscoelastic properties. The materials examined 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) in addition to three flowable bulk-fill RBCs (Filtek Bulk-Fill Flowable, Tetric N Flow Bulk-Fill, and Beautifil Bulk-Fill Flowable). Beam-shaped specimens (12×2×2 mm) were fabricated using customized stainless-steel molds, finished, and measured. The specimens were randomly divided into four groups and conditioned in air (control), artificial saliva, 0.02 N citric acid, and 50% ethanol-water solution for seven days at 37°C. They were then subjected to dynamic mechanical analysis (n = 10) in flexure mode at 37°C with a frequency of 0.1 to 10 Hz. Storage modulus, loss modulus, and loss tangent data were subjected to statistical analysis using one-way analysis of variance/Tukey *post hoc* test at a significance level of $\alpha = 0.05$. Viscoelastic properties of the RBCs were found to be product and conditioning medium dependent. For most RBCs, exposure to aqueous solutions, particularly an ethanol-water solution, degraded viscoelastic properties. With the exception of Filtek Bulk-Fill Restorative, bulk-fill restor-

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ative and flowable RBCs generally had significantly lower storage and loss modulus than their conventional counterparts regardless of conditioning medium. Conventional RBCs are thus favored over their bulk-fill counterparts, particularly for high-stress-bearing areas.

INTRODUCTION

Resin-based composites (RBCs) are one of the most widely used materials in contemporary dental practice. They are utilized as direct fillings for all cavities classes as well as indirect restorations, core buildups, and luting cements. Due to depth of cure and polymerization shrinkage issues, conventional RBCs have to be placed in 2-mm increments using a variety of layering techniques for direct restorations.[1] However, these incremental techniques are rather time consuming and technically challenging and may result in voids or bond failure between layers.[2] Bulk-fill RBCs were developed to address the complexities posed by incremental techniques and to streamline restorative processes.[3] These bulk-fill RBCs are said to cure in 4- to 5-mm increments without intensifying polymerization shrinkage stress.[4] Methods employed by manufacturers to create bulk-fill RBCs include reduction of filler content, modification of filler type to increase light transmission, and use of more effective initiators and monomer systems that allow for stress relief during polymerization.[3,4] Based on viscosity, bulk-fill materials can be divided into restorative (high-viscosity) or flowable (low-viscosity) RBCs. Flowable RBCs with their lower filler content are indicated primarily as bases/liners. They generally have lower wear resistance and mechanical properties and require a 2-mm "cap" of conventional hybrid RBCs when used in occlusal areas.[3,5] In contrast, bulk-fill restorative materials are designed to withstand occlusal loading and may not require a "capping" layer of conventional materials.

As bulk-fill RBCs are indicated for restoring large and deep posterior cavities, it is imperative to consider their mechanical properties. To increase curing depths, many commercial bulk-fill products employ the use of larger filler particles to reduce filler content, leading to higher filler weights but lower filler volumes.[6] Light penetration is thought to be enhanced by decreasing the difference in refractive index between the fillers and matrix as well as the quantity of filler-matrix interfaces.[7] Notwithstanding the potential reduction in filler content, the mechanical properties of bulk-fill restoratives remain contentious with studies reporting both lower and similar mechanical properties when compared to conventional RBCs.[6-10] The variance in findings may be attributed to differences in polymerization initiators, resin composition, filler type, size, and content, all of which influence material viscosity. In addition, food-simulating liquids and aqueous solutions have also been shown to degrade the mechanical properties of RBCs.[9,11]

Although RBCs are viscoelastic in nature, they are usually investigated using destructive static flexure, compression, and tension tests that examine only the elastic element of the materials and provide "single-event" values for strength.[9-12] Dynamic mechanical analysis (DMA) allows for nondestructive characterization of both viscous and elastic components of materials and is particularly useful for evaluating RBCs.[13] DMA is also capable of simulating a wide range of cyclic masticatory and thermal stresses encountered intraorally.[14] The technique involves the application of small oscillatory/sinusoidal stress to materials and measuring the resultant strain response. The stress applied can be divided into viscous and elastic stress, depending on whether it is out of phase or in phase with strain, respectively. Good correlations were observed between static and dynamic testing of RBCs even with smaller flexural specimens.[15,16]

The objectives of this study were to compare the viscoelastic properties of restorative and flowable RBCs with their conventional counterparts and to determine the impact of aqueous solutions on viscoelastic properties. Correlations between the various viscoelastic properties were also performed. The null hypotheses were as follows: 1) viscoelastic properties of restorative/flowable bulk-fill RBCs are no different from their conventional counterparts, 2) aqueous solutions do not influence the viscoelasticity of bulk-fill restorative/flowable and conventional RBCs, and 3) no correlations exist between the various viscoelastic properties of RBCs.

METHODS AND MATERIALS

Specimen Preparation and Conditioning

The materials and specimen preparation had been described in our earlier work.[10] RBCs evaluated were comprised of three conventional RBCs (Filtek Z350 [FZ], Tetric N Ceram [TN], and Beautifil II [BT]), three bulk-fill restorative RBCs (Filtek Bulk-Fill Restorative [FB], Tetric N Ceram Bulk-Fill [TB], and Beautifil Bulk-Fill Restorative [BB]) along with three bulk-fill flowable RBCs (Filtek Bulk-Fill Flowable [FF], Tetric N Flow Bulk-Fill [TF], and

Table 1: *Technical Profiles and Manufacturers of the Materials Evaluated*

Manufacturer	Material (Abbreviation)	Type	Resin (Photoinitiator)	Filler	Filler Content % by Weight/ % by Volume	Lot No.
3M ESPE (St Paul, MN, USA), manufacturer A	Filtek Z350 (FZ)	Conventional	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	AUDMA, AFM DDDMAA UDMA (CQ)	Zirconia/silica cluster, ytterbium trifluoride	76.5/58.4	N693019
	Filtek Bulk-Fill Flowable (FF)	Bulk-fill flowable	Bis-GMA Bis-EMA UDMA (CQ, EDMAB)	Zirconia/silica, ytterbium trifuoride	64.5/42.5	N884479
Ivoclar Vivadent Inc (Amherst, NY, USA), manufacturer B	Tetric N Ceram (TN)	Conventional	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	Bis-GMA Bis-EMA UDMA (CQ, TPO, Ivocerin)	Barium glass filler, ytterbium fluoride and spherical mixed oxide	79/60	S38368
	Tetric N Flow Bulk-Fill (TF)	Bulk-fill flowable	Bis-GMA UDMA TEGDMA (CQ, TPO, Ivocerin)	Barium glass, ytterbium trifluoride, mixed oxide, silicon dioxide	64.9/NA	V49336
Shofu Inc (Kyoto, Japan), manufacturer C	Beautifil II (BT)	Conventional	Bis-GMA TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	83.3/68.8	031731
	Beautifil-Bulk Restorative (BB)	Bulk-fill restorative	Bis-GMA UDMA Bis-MPEPP TEGDMA (CQ)	S-PRG based on F-Br-Al-Si glass	87/74.5	051623
	Beautifil-Bulk Flowable (BF)	Bulk-fill flowable	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, fluoroboroaluminosiliciate; Bis-MPEPP, bisphenol-A polyethoxy-dimethacrylate.

Beautifil Bulk-Fill Flowable [BF]). The materials and their technical profiles are specified in Table 1. Briefly, 40 beam-shaped specimens (12×2×2 mm) of the different RBCs were fabricated with customized stainless-steel molds. The RBCs were light polymerized using an LED curing light (Demi Plus, Kerr Corp, Orange, CA, USA) with an output irradiance of 1330 mW/cm^2 in two overlapping irradiations of 10 seconds. Specimens were then finished using fine polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA), inspected for defects/parallelism, and measured with a digital caliper (Mitutoyo Corp, Kawasaki, Japan). The RBCs were then randomly divided into four groups (n=10) and stored in sealed containers with the following conditioning mediums for seven days at 37°C in an incubator (IN-460, Memmert, Schwabach, Germany): air, artificial saliva (SAGF),[17] 0.02 N citric acid, and 50% ethanol-water solution. Air served as the control medium, while artificial saliva replicated natural saliva. Citric acid and 50% ethanol mimicked certain fruits, candies, and beverages, including alcohol. The pH of the artificial saliva (6.8) and citric acid (2.6) was verified via a digital pH meter (pH2700, Eutech, Singapore).

Dynamic Mechanical Analysis

Following conditioning, the RBCs were subjected to dynamic flexural testing (DMA RSAG2, TA Instruments, New Castle, DE, USA) in the various conditioning mediums at 37°C. Details of the testing protocol have been described previously.[16] Specimens were loaded using a three-point bending fixture with a 10-mm support distance and loading frequency of 0.1 to 10 Hz to simulate the range from almost static to the upper limit of chewing rates.[18] However, the upper limit is extremely fast and exceeds the range expected for normal chewing. Loading frequency was progressively increased from 0.1 to 10 Hz in 15 steps with a delay time of one second and a delay cycle of 0.5 Hz. Storage modulus, loss modulus, and loss tangent data were subsequently calculated as follows:[16,19]

Storage modulus:

$$E' = (\sigma°/\varepsilon°)\cos\delta = (f_o/bk)\cos\delta$$

Loss modulus:

$$E'' = (\sigma°/\varepsilon°)\sin\delta = (f_o/bk)\sin\delta$$

Table 2: *Mean Storage Modulus (E′ [GPa]), Loss Modulus (E″ [GPa]), and Loss Tangent (×10^{-3}) Values for the Various Restorative Materials With Standard Deviations (in Parentheses)*

Medium/Material	Storage Modulus (GPa)				Loss Modulus (GPa)	
	Air	Artificial Saliva	Citric Acid	Ethanol	Air	Artificial Saliva
Filtek Z350 (FZ)	6.29 (0.29)	5.48 (0.56)	5.19 (0.42)	4.49 (0.48)	0.42 (0.04)	0.47 (0.06)
Filtek Bulk-Fill Restorative (FB)	6.20 (0.72)	6.10 (0.49)	5.86 (0.50)	6.04 (0.37)	0.43 (0.06)	0.46 (0.06)
Filtek Bulk-Fill Flowable (FF)	4.05 (0.35)	4.03 (0.41)	3.99 (0.18)	2.47 (0.21)	0.38 (0.05)	0.41 (0.05)
Tetric N Ceram (TN)	6.18 (0.57)	5.56 (0.53)	5.47 (0.50)	4.45 (0.49)	0.58 (0.08)	0.59 (0.08)
Tetric N Ceram Bulk-Fill (TB)	5.00 (0.37)	3.76 (0.40)	3.72 (0.48)	3.68 (0.18)	0.38 (0.04)	0.36 (0.05)
Tetric N Flow Bulk-Fill (TF)	5.20 (0.38)	4.60 (0.35)	4.57 (0.64)	4.53 (0.53)	0.45 (0.04)	0.44 (0.06)
Beautifil II (BT)	7.02 (0.58)	6.63 (0.81)	6.02 (0.35)	5.78 (0.68)	0.46 (0.07)	0.51 (0.08)
Beautifil-Bulk Restorative (BB)	5.79 (0.36)	5.51 (0.53)	5.04 (0.56)	4.61 (0.47)	0.38 (0.05)	0.43 (0.06)
Beautifil-Bulk Flowable (BF)	6.17 (0.55)	5.81 (0.57)	5.17 (0.65)	5.14 (0.54)	0.41 (0.05)	0.39 (0.06)

Loss tangent:

$$\text{Tan } \delta = E''/E'$$

where σ° is the maximum stress at the peak of the sine wave, ε° is the strain at the maximum stress, f_o is the force applied at the peak of the sine wave, b is the sample geometry term, and k is the sample displacement at the peak.

Sample geometry b for a three-point bending bar was calculated as follows:

$$4BH^3/L^3$$

where B is the width of the specimen (in mm), H is the height of the specimen (in mm), and L is the distance between the supports (in mm).

Statistical Analysis

Data were analyzed using the SPSS statistical program (version 12.0.1, SPSS Inc, Chicago, IL, USA). Parametric analysis was performed, as data were normally distributed based on the Shapiro-Wilk test. Two-way analysis of variance (ANOVA) was used to determine interactions between independent variables (materials and conditioning mediums) and dependent variables (storage modulus, loss modulus, and loss tangent). One way ANOVA followed by the *post hoc* Tukey test were used to assess intermedium and intermaterial differences. The latter was grouped by both manufacturer and material type (ie, conventional, bulk-fill restorative, and bulk-fill flowable). Correlation between viscoelastic properties was carried out with the Pearson correlation. All statistical analyses were performed at a significance level of $\alpha = 0.05$.

RESULTS

Mean storage modulus, loss modulus, and loss tangent for the RBCs after conditioning in the various aqueous solutions are shown in Table 2. Intermedium comparisons are shown in Table 3, while intermaterial evaluations are presented in Table 4 (grouped by manufacturer) and Table 5 (grouped by material type). Correlations between the various viscoelastic properties are displayed in Table 6. Mean storage modulus, loss modulus, and loss tangent ranged from 2.47 to 7.02 GPa, 0.33 to 0.64 GPa, and 66 to 143 × 10^{-3}, respectively, for the various material-medium combinations. Viscoelastic properties of the RBCs were found to be material and conditioning medium dependent.

With the exception of FB and FF, storage in air (control) frequently resulted in a significantly higher storage modulus than in aqueous solutions (Table 3). Exposure to the ethanol-water solution usually presented the lowest storage modulus. For the majority of RBCs, conditioning in the ethanol-water solution also resulted in a significantly higher loss modulus and loss tangent. Moreover, the lowest loss tangent was typically observed with the control group (air).

For manufacturer A, FB had a significantly greater storage modulus than FZ, which in turn was stiffer than FF in all mediums except air (Table 4). While no obvious trends were noted for loss modulus, FF had a significantly greater loss tangent than FB and FZ regardless of conditioning medium. For manufacturer B, TN had a significantly greater storage modulus, loss modulus, and loss tangent than TF and TB with the exception of storage in the ethanol-water solution. For manufacturer C, BT showed a significantly higher storage and loss

Table 2: *Mean Storage Modulus (E′ [GPa]), Loss Modulus (E″ [GPa]), and Loss Tangent (×10^{-3}) Values for the Various Restorative Materials With Standard Deviations (in Parentheses) (ext.)*

Medium/Material	Loss Modulus (GPa)		Loss Tangent (×10^{-3})			
	Citric Acid	Ethanol	Air	Artificial Saliva	Citric Acid	Ethanol
Filtek Z350 (FZ)	0.48 (0.06)	0.42 (0.05)	68 (5)	88 (9)	91 (10)	97 (3)
Filtek Bulk-Fill Restorative (FB)	0.51 (0.06)	0.64 (0.06)	69 (4)	84 (10)	85 (5)	106 (5)
Filtek Bulk-Fill Flowable (FF)	0.43 (0.04)	0.35 (0.02)	95 (6)	102 (5)	107 (7)	143 (11)
Tetric N Ceram (TN)	0.59 (0.07)	0.61 (0.08)	95 (6)	108 (8)	109 (5)	138 (7)
Tetric N Ceram Bulk-Fill (TB)	0.33 (0.04)	0.44 (0.05)	76 (5)	95 (7)	94 (10)	135 (9)
Tetric N Flow Bulk-Fill (TF)	0.41 (0.06)	0.55 (0.08)	88 (5)	97 (10)	92 (6)	121 (6)
Beautifil II (BT)	0.51 (0.08)	0.55 (0.07)	66 (6)	78 (6)	86 (10)	96 (5)
Beautifil-Bulk Restorative (BB)	0.48 (0.07)	0.42 (0.06)	66 (7)	73 (5)	101 (6)	93 (3)
Beautifil-Bulk Flowable (BF)	0.36 (0.05)	0.43 (0.04)	67 (5)	68 (3)	70 (3)	85 (4)

modulus than BF and BB apart from conditioning in citric acid. However, trends for loss tangent were mixed.

When conventional RBCs were compared (Table 5), BT had a significantly higher storage modulus than FZ and TN regardless of conditioning mediums. The loss modulus and tangent of TN was mostly significantly greater than the other two conventional RBCs. For bulk-fill restoratives, FB and BB had a significantly higher storage modulus than TB for all four mediums. No distinct trends were noted for loss modulus and tangent. For the flowable RBCs, BF generally had a significantly higher storage modulus than TF and FF. While no obvious patterns were observed for loss modulus, FF and TF had a significantly larger loss tangent than BF.

A significant and moderate correlation was observed between storage and loss modulus (r=0.496) as well as storage modulus and loss tangent (−0.560) (Table 6). The correlation between loss modulus and loss tangent, though significant, was weak (0.356).

Table 3: *Results of Statistical Analysis for Mean Values of Storage Modulus (E′), Loss Modulus (E″), and Loss Tangent (Tanδ) Based on Materials*[a]

Manufacturer	Material	Differences		
		Storage Modulus	Loss Modulus	Loss Tangent
A	FZ	A > S, C > E	Nonsignificant	E > S > A C > A
	FB	Nonsignificant	E > C > A E > S	E > C, S > A
	FF	A, S, C > E	C, S > E	E > C > A E > S
B	TN	A > C > E S > E	Nonsignificant	E > C, S > A
	TB	A > S, C, E	E > A, S, C	E > S, C > A
	TF	A > S, C, E	E > A, S, C	E > S > A E > C
C	BT	A > C, E S > E	E > A	E > C, S > A
	BB	A > C, E S > E	C > A	C > E > S > A
	BF	A > C, E	E > C	E > C, S, A

Abbreviations: FZ, Filtek Z350; A, air; S, saliva; C, citric acid; E, ethanol; FB, Filtek Bulk-Fill Restorative; FF, Filtek Bulk-Fill Flowable; TN, Tetric N Ceram; TB, Tetric N Ceram Bulk-Fill; TF, Tetric N Flow Bulk-Fill; BT, Beautifil II; BB, Beautifil-Bulk Restorative; BF, Beautifil-Bulk Flowable.
[a] *Results of one-way analysis of variance and* post hoc *Tukey test; > indicates statistical significance ($p<0.05$).*

Table 4: *Results of Statistical Analysis for Mean Values of Storage Modulus (E′), Loss Modulus (E″), and Loss Tangent (Tanδ) Grouped According to Manufacturers and Based on Conditioning Mediums*[a]

Properties	Manufacturer/Medium	Differences		
		Manufacturer A	Manufacturer B	Manufacturer C
Storage modulus	Air	FZ, FB > FF	TN > TF, TB	BT > BF, BB
	Artificial saliva	FB > FZ > FF	TN > TF > TB	BT > BF, BB
	Citric acid	FB > FZ > FF	TN > TF > TB	BT > BF, BB
	Ethanol 50%	FB > FZ > FF	TF, TN > TB	BT > BF, BB
Loss modulus	Air	Nonsignificant	TN > TF > TB	BT > BB
	Artificial saliva	Nonsignificant	TN > TF > TB	BT > BB, BF
	Citric acid	FB > FF	TN > TF > TB	BT, BB > BF
	Ethanol 50%	FB > FZ > FF	TN, TF > TB	BT > BF, BB
Loss tangent	Air	FF > FB, FZ	TN > TF > TB	Nonsignificant
	Artificial saliva	FF > FZ, FB	TN > TF, TB	BT > BF
	Citric acid	FF > FZ, FB	TN > TB, TF	BB > BT > BF
	Ethanol 50%	FF > FB > FZ	TN, TB > TF	BT, BB > BF

Abbreviations: FZ, Filtek Z350; FB, Filtek Bulk-Fill Restorative; FF, Filtek Bulk-Fill Flowable; TN, Tetric N Ceram; TF, Tetric N Flow Bulk-Fill; TB, Tetric N Ceram Bulk-Fill; BT, Beautifil II; BF, Beautifil-Bulk Flowable; BB, Beautifil-Bulk Restorative.
[a] *Results of one-way analysis of variance and* post hoc *Tukey test; > indicates statistical significance (*$p<0.05$*).*

DISCUSSION

This study compared the viscoelastic properties of restorative and flowables RBCs with their conventional counterparts and determined the impact of aqueous solutions on viscoelastic properties. As the viscoelastic properties of the RBCs were found to be material and conditioning medium dependent and significant correlations were observed between viscoelastic properties, all three null hypotheses were duly rejected. The "miniflexural" specimens employed for this study were approximately half the length recommended by the International Organization for Standardization (ISO) (25×2×2 mm).[16] Besides being more efficient to execute and clinically relevant, these miniflexural specimens also correlated well with the ISO flexural tests for both dynamic and static assessments.[16] DMA provided information on storage modulus, loss modulus, and loss tangent. The stiffness/rigidity and flow of the RBCs are characterized by storage and loss modulus, respectively.[14,20] For use in posterior teeth, RBCs with a high storage modulus and low loss modulus are advantageous to resist deformation from occlusal forces. Although a greater flow could possibly decrease or delay wear and fracture, loss modulus values were about 10 times smaller than storage modulus values and may not affect clinical performance greatly. Loss tangent is the ratio of loss modulus to storage modulus.[20] It represents the energy-dissipating capacity of the RBCs, with lower values indicating faster response to load and return to original form.[13]

Comparison Between Conditioning Mediums

In the present study, exposure to aqueous solutions degraded the viscoelastic properties of the RBCs with the exception of FB and FF. Findings corroborated previous studies pertaining to the effect of conditioning mediums/food-simulating liquids on viscoelastic properties of RBCs.[21,22] The lowest storage modulus and highest loss tangent were observed after conditioning in ethanol. This could be attributed to the high propensity of the RBC polymers to absorb ethanol-water solutions, resulting in matrix swelling and decreased mechanical properties.[23,24] The better performance of FB and FF may be attributed partly to the introduction of methacrylates that undergo free-radical addition fragmentation. This might enhance postcuring structural stability in addition to reducing polymerization stress.[25] For manufacturer C, no significant difference in storage modulus was observed between conditioning in citric acid and ethanol for all material types. All the products of manufacturer C were giomers based on prereacted glass ionomer (PRG) technology, where fluoride-containing glass is reacted with polyacid acids, freeze-dried, milled, silanized, and used as fillers.[2] Giomers have been found to be particularly prone to degradation by acids of low pH due to their glass ionomer constituent.[26]

Comparison Between Material Types

When material types were compared, bulk-fill restorative and flowable RBCs generally had a signif-

Table 5: *Results of Statistical Analysis for Mean Values of Storage Modulus (E′), Loss Modulus (E″), and Loss Tangent (Tanδ) Grouped According to Material Type and Based on Conditioning Mediums*[a]

Properties	Material Type/Medium	Differences		
		Conventional	Bulk-Fill Restorative	Bulk-Fill Flowable
Storage modulus	Air	BT > FZ, TN	FB, BB > TB	BF > TF > FF
	Artificial saliva	BT > TN, FZ	FB > BB > TB	BF > TF > FF
	Citric acid	BT > TN, FZ	FB > BB > TB	BF > FF
	Ethanol 50%	BT > FZ, TN	FB > BB > TB	BF > TF > FF
Loss modulus	Air	TN > BT, FZ	Nonsignificant	TF > FF
	Artificial saliva	TN > FZ	FB, BB > TB	Nonsignificant
	Citric acid	TN > FZ	FB, BB > TB	FF, TF > BF
	Ethanol 50%	TN, BT > FZ	FB > TB, BB	TF > BF > FF
Loss tangent	Air	TN > FZ, BT	TB > FB, BB	FF > TF > BF
	Artificial saliva	TN > FZ > BT	TB > FB > BB	FF, TF > BF
	Citric acid	TN > FZ, BT	BB, TB > FB	FF > TF > BF
	Ethanol 50%	TN > FZ, BT	TB > FB > BB	FF > TF > BF

Abbreviations: BT, Beautifil II; FZ, Filtek Z350; TN, Tetric N Ceram; FB, Filtek Bulk-Fill Restorative; BB, Beautifil-Bulk 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; > indicates statistical significance (*$p<0.05$*).*

icantly lower storage modulus than their conventional counterparts with the exception of FB. FB was significantly more rigid than both conventional (FZ) and bulk-fill flowable (FF) materials from manufacturer A. As the filler content by volume of FB was lower than FZ, improvements in mechanical properties can be attributed more to resin modifications. The bulk-fill restorative and flowable RBCs of manufacturers B and C were significantly less rigid than their conventional equivalents. Accordingly, their conventional RBCs should be used preferentially over bulk-fill materials, especially in high-stress–bearing areas. The storage modulus of the bulk-fill restoratives TB and BB were similar or even lower than their flowable equivalents TF and BF. This was unexpected, as the filler content of flowable RBCs is lower than restorative ones to reduce viscosity and enhance cavity adaptation. Previous studies based on static techniques found that bulk-fill RBCs have mechanical properties between conventional and flowable hybrid materials.[6-8] It is plausible that bulk-fill flowable RBCs are formulated similarly to flowable hybrid materials, resulting in

Table 6: *Pearson Correlations Between the Various Flexural Properties*

Properties	Storage Modulus	Loss Modulus	Loss Tangent
Storage modulus		0.496*	−0.560*
Loss modulus	0.496*		0.356*
Loss tangent	−0.560*	0.356*	

** Correlation significant at the 0.01 level (two-tailed).*

comparable mechanical properties. The depth of cure and degree of conversion of bulk-fill flowable RBCs are also typically greater than bulk-fill restorative materials due to their greater translucency.[27-29] The latter is achieved via decreasing light absorption by pigments and light refraction by matrix-filler interfaces (arising from lower filler content). Degree of conversion governs mechanical properties (with higher degree of conversion being associated with better mechanical properties) and explains in part the similar rigidity of bulk-fill restorative and flowable RBCs in the present study.[30]

Although no obvious trends were observed for manufacturer A, the two conventional RBCs for manufacturers B and C usually had significantly higher loss modulus and flowed more than their bulk-fill counterparts. As filler content of the conventional RBCs was higher or similar to the bulk-fill restoratives, observations can be accounted for only by variance in initiators and resin constituents. While TN is based on camphorquinone, both TF and TB incorporate an adjunct photoinitiator, Ivocerin, giving them a wider absorption range of 370 to 460 nm.[31] As initiator systems influence both the degree of conversion and cross-linking, it is not surprising that flow is reduced in both TF and TB.[32] For BF and BB, the supplementary copolymers added included urethane dimethacrylate (UDMA) and bisphenol-A polyethoxy-dimethacrylate. The lower flow of both giomers can be credited to UDMA, which is a rigid and viscous material, and possibly the use of PRG fillers.[33] As loss tangent values are dependent on both storage and loss modulus, trends are expected to vary, depending on viscoelastic changes after conditioning in the various mediums.

Comparison Between Products

When conventional materials were compared, BT was found to be significantly stiffer than the other two conventional materials regardless of conditioning medium. This may be due primarily to the higher volume filler loading of BT, including PRG fillers. Likewise, the greater loss modulus and tangent of TN can be accounted for by its relatively lower filler content. Among the bulk-fill restoratives, TB was significantly less rigid than FB and BB. As filler content was similar, the disparity could be due to differences in resin composition and the eclectic mix of fillers used in TB. The lower storage modulus of TB could also be contributed by the nonuse of a polywave curing light that is necessary for the photoactivation of Ivocerin. No obvious patterns were observed for loss modulus and tangent with ranking varying for different conditioning mediums. This reiterates the need for RBCs to be evaluated in different chemical environments instead of just air and/or distilled water, which is often practiced. When comparing the bulk-fill flowable materials, BF, in view of its high filler volume content, was significantly more rigid than TF and FF. As loss tangent is inversely proportion to rigidity, the significantly lower loss tangent of BF was anticipated in view of its higher storage modulus.

Correlations Between Viscoelastic Properties

As RBCs exhibit both viscous and elastic characteristics under stress, some degree of correlation is anticipated between storage and loss modulus. The correlation was moderately strong, supporting the need for routine viscoelastic characterization of RBCs in conjunction with static testing that evaluates only the elastic component of materials.[10-12] Significant correlations between storage/loss modulus and loss tangent were anticipated, as loss tangent is the ratio of loss modulus to storage modulus. As the correlation between storage modulus and loss tangent was substantially stronger than that between loss modulus and loss tangent, any variance in energy-dissipating capacity is due primarily to changes in rigidity. This corroborated previous work that reported loss tangent increases with increasing filler content.[34]

Limitations of the Present Study

Some of the limitations of this study had been discussed in our earlier work.[10] They included the need for longer-term conditioning in the various mediums and independent appraisal of rheological properties and filler content instead of reliance on manufacturers' data, which may not be comparable due to methodological differences. In addition, the evaluation of products from more manufacturers is warranted given the heterogeneity of products under the same material type or classification. With regard to DMA, future work could encompass variation of frequencies, temperatures, and displacement that simulate the range of forces and temperatures encountered intraorally. The glass transition temperature (T_g), which is the temperature at which RBCs undergo transition from a glassy to a rubbery state, can also be determined with DMA. This represents the point at which marked changes in physical properties occur and had been linked to curing light energy density and degree of monomer-to-polymer conversion.[35] T_g values may provide valuable insights toward explaining the observed viscoelastic responses. Viscoelastic properties should also be interrelated with the clinical performance of bulk-fill and conventional RBCs.

CONCLUSION

The following conclusions can be drawn within the limitations of this study:

- Viscoelastic properties of conventional, bulk-fill restorative, and flowable RBCs were product and conditioning medium dependent.
- For the majority of RBCs evaluated, conditioning in aqueous solutions, particularly an ethanol-water solution, degraded viscoelastic properties.
- Bulk-fill restorative and flowable RBCs generally had lower storage and loss modulus than their conventional equivalents with the exception of FB.
- Conventional and bulk-fill flowable giomers exhibited higher storage modulus and lower loss tangent than the other products.
- Conventional RBCs are generally preferred over their bulk-fill counterparts for high-stress–bearing restorations.

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Conflict of Interest

The authors of the 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.

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