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Effect of Thermocycling on Biaxial Flexural Strength of CAD/CAM, Bulk Fill, and Conventional Resin Composite Materials

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Clinical Relevance

Biaxial flexural strength of tested materials vary with composition and thermocycling significantly decreases their strength. Future clinical studies should evaluate if recommendations based on cavity size and tooth location improve long-term performance.

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SUMMARY

New resin-based restorative materials have been developed, such as computer-aided design/computer-aided manufacturing (CAD/CAM) and bulk-fill composites, as an alternative to traditional layering techniques. This study evaluated the biaxial flexural strength (BFS) before and after thermocycling of five different resin composites: one hybrid resin/ceramic CAD/CAM indirect material, Lava Ultimate CAD-CAM Restorative (LU, 3M Oral Care); a conventional composite, Filtek Z350 XT (Z350, 3M Oral Care); two bulk-fill composites, Tetric N-Ceram Bulk Fill (TBF, Ivoclar Vivadent) and Filtek Bulk Fill (FBF, 3M Oral Care); and one bulk-fill flow resin composite, Filtek Bulk Fill Flow (FBFF, 3M Oral Care). Three hundred disc-shaped specimens (6.5 mm

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in diameter and 0.5 mm thick) were fabricated and divided into five groups (n=30 for each composite and condition). The BFS test was performed in a universal testing machine at a crosshead speed of 0.5 mm/min immediately (i, 24 hours) and after thermocycling (a, 500 thermal cycles of 5°C to 55°C with a 30-second dwell time). The Weibull modulus (*m*) and characteristic stress (η) were calculated, and a contour plot was used (*m* vs η) to detect differences between groups (95% two-sided confidence intervals). Significantly higher characteristic stress was observed for LUi (286.6 MPa) and Z350i (248.8 MPa) compared to the bulk-fill groups (FBFi=187.9 MPa, FBFFi=175.9 MPa, TBFi=149.9 MPa), with no differences between LUi and Z350i. Thermocycling significantly decreased the characteristic stress of all groups with the highest values observed for LUa (186.7 MPa) and Z350a (188.9 MPa) and the lowest for FBFFa (90.3 MPa). Intermediate values were observed for FBFa (151.6 MPa) and TBFa (122.8 MPa). The Weibull modulus decreased only for FBFa compared to FBFi. Composition and thermocycling significantly influenced the biaxial flexural strength of resin composite materials.

INTRODUCTION

Although excellent esthetic results can be obtained with direct composite resin restorations, this material still presents disadvantages, such as polymerization shrinkage stress and dentin adhesion, which may lead to marginal infiltration, postoperative sensitivity, and a decrease in dentin bond strength.[1,2] To meet the recommendations of the Minamata Convention, recent developments in composite resins include different compositions to minimize polymerization contraction stress and different fabrication methods, including milling from computer-aided design/computer-aided manufacturing (CAD/CAM) resin composite blocks or additive manufacturing, such as 3D printing.[3-5]

Despite reported high survival rates of composite resin restorations when compared to amalgam, even in wide restorations,[6] there is still a need for improvement of this material because it undergoes loss of mechanical properties and esthetics as a function of fatigue, aging at body temperature, and a moist environment.[7] The cause of discoloration, polish loss, opacity increase, and loss of adhesion, as well as the appearance of fractures, is multifactorial but highly influenced by internal chemical changes in the material that occur due to degradation, which can be simulated by artificial aging methods.[8]

The hydrothermal degradation of resin composites is mediated by sorption and solubility, phenomena that produce chemical changes with deleterious effects on the mechanical properties of polymeric materials.[9] The diffusion of solvents into the polymer network leads to a volumetric expansion due to the separation of polymeric chains and depends on the composition and microstructure of the materials.[10] Swelling caused by absorption of aqueous solvents is accompanied by a loss of non-reacted components,[11] erosion of the filler-matrix interface, and plasticization with a reduction in hardness, stiffness, wear resistance, and flexural strength,[9,12] which may compromise long-term clinical results of composite resin materials.

Although there are several laboratory tests available for dental composite resins, they present a challenge in simulating clinical performance. However, they can guide understanding the effects of changes in composition or processing on material properties. In flexural strength testing, flexural forces are generated to simulate clinical situations where materials need to withstand flexing, especially in the posterior region. Although not confirmed clinically, high flexural strength is desired for these materials that might experience cracking under occlusal stress.[13]

Also, because of the heterogeneous composition of resin composites and the dissimilar coefficient of thermal expansion of their components, thermal stresses are easily generated in the material and may be exacerbated by thermal cycling.[14] Thus, thermocycling has been widely accepted in the literature as a method to promote the degradation of the mechanical properties[15] and the bond strength of resin composite materials.[16-18]

A promising approach to overcome the difficulties of sensitive and time-consuming restorative procedures using the incremental technique include low-shrinkage composite resins that allow the clinician to apply layers up to 5 mm thick.[18] Bulk placement of resin composites has been reported to provide more compact fillings, preventing void contamination between composite layers.[17,19]

Another alternative to direct restoration techniques is milling restorations from composite blocks in CAD/CAM systems. The main advantages include production of homogeneous blocks with a high degree of conversion, reduced equipment wear

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Table 1: *Manufacturers, Composition, and Batch Numbers of the Tested Materials*

Brand Name	Type	Manufacturer (Lot Number)	Monomers	Filler Type	Filler Volume (%)	Batch
Lava Ultimate (LU)	Resin/ceramic hybrid material for CAD/CAM	3M Oral Care (St Paul, MN, USA)	UDMA	Silica, zirconia, nanoparticle clusters	80	550835
Filtek Z350 XT (Z550XT)	Nanoparticle	3M Oral Care	UDMA, bis-GMA, bis-EMA, TEGDMA	Silica, zirconia, clusters, zirconia/silica aggregated particles	78.5	1524600312
Filtek Bulk Fill (FBF)	Bulk fill	3M Oral Care	Bis-GMA, bis-EMA, UDMA, TEGDMA, Procrylat resins	Zirconia/silica, ytterbium trifluoride	76.5	1522200095
Tetric N-Ceram Bulk Fill (TBF)	Bulk fill	Ivoclar Vivadent (Schaan, Liechtenstein)	Bis-GMA, UDMA	Ba-Al-Si glass, prepolymerized filler (monomer, glass filler, and ytterbium fluoride), pherical mixed oxide	61% of inorganic fillers and 17% prepolimerized “isofillers”	T40644
Filtek Bulk Fill Flowable Composite (FBBF)	Flowable bulk fill	3M Oral Care	Bis-GMA, UDMA, bis-EMA, Procrylat resins	Zirconia/silica, ytterbium trifluoride	64.5	1526900194

Abbreviations: UDMA, urethane dimethacrylate; bis-GMA, bisphenol-A glycidyl methacrylate; bis-EMA, ethoxylated Bisphenol A glycidyl methacrylate ; TEGDMA, triethylene glycol dimethacrylate.

(CAD/CAM burs), reduced cost of fabrication, and ease of adjustments and repair.[20-22] The ceramic particles embedded in the organic matrix can be of various compositions and, due to the high-filling percent content, these materials have been included in the American Dental Association’s Code of Dental Procedures and Nomenclatures as ceramic/porcelain. They are classified elsewhere as hybrid or ceramic-like materials (resin-matrix ceramics) due to the presence of an organic matrix, which excludes them from ceramics as known in materials science.[23] Most of them have been indicated for inlays, onlays, veneers, and crowns.

Because of the presence of an organic matrix, resin-based composite materials are subject to aging, which can decrease mechanical properties and clinical longevity. Thus, in the present study, we evaluated the biaxial flexural strength (BFS) and the effect of aging by thermocycling on a group of resin-based restorative materials, including conventional composites, CAD/CAM, and bulk-fill composite resins. The hypotheses were 1) that composition and manufacturing method would influence the characteristic stress or Weibull modulus of composite resins and 2) that the characteristic stress of composite resins would decrease after aging regardless of composition or fabrication/layering method.

METHODS AND MATERIALS

Specimen Preparation

Three hundred composite resin disc-shaped specimens (6.5 mm in diameter and 0.5 mm thick)[24] were fabricated using a Teflon mold and divided into five groups (n=60 for each composite) as follows: 1) resin nanoceramic - Lava Ultimate CAD-CAM Restorative (LU, 3M Oral Care, 3M, St Paul, MN, USA), 2) resin nanoceramic - Filtek Z350 XT (Z350, 3M Oral Care), 3) bulk fill composite - Tetric N-Ceram Bulk Fill (TBF, Ivoclar Vivadent, Schaan, Liechtenstein), 4) bulk-fill composite - Filtek Bulk Fill (FBF, 3M Oral Care), and 5) bulk-fill flow resin composite - Filtek Bulk Fill Flow (FBFF, 3M Oral Care). Details regarding manufacturers, composition, and batch of the composites used are detailed in Table 1. Half of the samples (n=30) for each group were tested for BFS immediately (i, 24 hours) after fabrication and the other half (n=30) after thermocycling (a).

For the resin nanoceramic, bulk-fill, and bulk-fill flow composite resins, a single increment of composite material was confined in the Teflon mold between two opposing polyester strips. Light curing was performed for 20 seconds on both sides with an LED unit (Valo, Ultradent, South Jordan, UT, USA) having an irradiance of 1000 mW/cm^2. The specimens were then removed from the mold and excesses were eliminated with a scalpel blade (Surgical Scalpel Blade No. 15, Swann-Morton, Sheffield, England). The CAD-CAM Lava Ultimate blocks were milled into 6.5-mm-diameter cylinders using Cerec (Dentsply Sirona Inc, Benshein, Germany) and then sliced using a slow-speed diamond saw (Extec Corp, Enfield, CT, USA) in a precision water-cooled machine (Isomet Low Speed Saw,

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Table 2: *Results of Biaxial Flexural Strength Test*[a]

Group	Immediate		Aged	
	Characteristic Stress, MPa (95% CB)	Weibull Modulus (*m*)	Characteristic Stress, MPa (95% CB)	Weibull Modulus (*m*)
Lava Ultimate (LU)	286.6 (307.3-267.2) Aa	5.1 (6.7-3.9) Aa	186.7 (195.5-178.3) Ba	7.6 (10-5.81) Aa
Filtek Z350 XT (Z350)	248.8 (266.5-232.2) Aa	5.2 (6.8-4) Aa	188.9 (188.9-178.3) Ba	8.1 (10.6-6.2) Aa
Filtek Bulk Fill (FBF)	187.9 (194.9-181.1) Ab	9.9 (12.9-7.6) Ab	151.6 (169.2-135.9) Bb	3.1 (4.1-2.4) Bb
Tetric N-Ceram Bulk Fill (TBF)	149.9 (157.5-142.8) Ac	6.7 (8.6-5.3) Aab	122.8 (127.8-118.0) Bc	9.0 (11.7-7.0) Aa
Filtek Bulk Fill Flow (FBBF)	175.9 (192.5-160.8) Ab	4.2 (5.3-3.2) Aa	90.3 (103.2-79.0) Bd	2.5 (3.4-1.9) Ab

[a] *Uppercase letters show differences between rows (comparing immediate and aged groups), and lowercase letters show differences between columns (comparing materials). Values in parentheses represent 95% upper and lower confidence bounds.*

Buehler, Lake Bluff, IL, USA) to obtain 60 disc-shaped specimens approximately 0.5 mm thick. All specimens were manually polished under water cooling with 800-, 1200-, and 2000-grit silicone carbide paper (Carbimet Paper Discs, Buehler), and their dimensions were measured using a digital caliper (Mitutoyo, Takatsu-ku, Kanagawa, Japan). Although clinical protocols may be different, the standardization of the polishing protocol during laboratory testing allowed for comparison of the characteristic stress and Weibull modulus between all groups.

The specimens were dark stored dry at 37°C for 24 hours (i). One-half of the specimens for each composite resin (n=30) were subjected to thermocycling of 5°C to 55°C with a 30-second dwell time at each temperature, and 500 thermal cycles (a).

Biaxial Flexural Strength

A piston-on-ring device was used for the biaxial flexural test, following methodology described by Rueggeberg and others.[24] Each disc was placed into a custom-made jig with 1-mm circumferential support, and the load was applied at the center of the specimen using a flat piston with a 0.5-mm diameter. The device was positioned in a universal testing machine (Kratos, São Paulo, Brazil), and specimens were tested at a crosshead speed of 0.5 mm/min until failure. The maximum load was recorded for each specimen, and the following formulas were used to calculate biaxial flexural strength:

$$S = -0.2387P(X - Y)/d^2$$

$$X = (1 + v)\ln(r_2/r_3)^2 + ([1 - v]/2)(r_2/r_3)^2$$

$$Y = (1 + v)(1 + \ln[r_1/r_3]^2) + (1 - v)(r_1/r_3)^2$$

where S = biaxial flexural strength (MPa), P = fracture load (N), d = disc specimen thickness at fracture site (0.5 mm), v = Poisson ratio (0.25), r_1 = radius of support circle (2.75 mm), r_2 = radius of loaded area (0.25 mm), and r_3 = radius of the specimen (3.25 mm).

The fractured specimens were examined with an Axio Zoom V16 Stereo Zoom Microscope (Carl Zeiss, Oberkochen, Germany).

Statistical Analysis

Strength data (MPa) were analyzed using the Weibull distribution as an alternative to the normal distribution due to its ability to evaluate the properties, characteristic stress (η), and Weibull modulus (*m*) of the materials, and a contour plot was used (*m* vs η) to detect differences between groups (95%, two-sided confidence intervals).

RESULTS

Table 2 shows the results for the characteristic stress (MPa) and the Weibull modulus (*m*) for the immediate and aged groups with respective upper and lower 95% confidence bounds. Significantly higher characteristic stress was observed for LUi (286.57 MPa) and Z350i (248.76 MPa) than all bulk-fill composites (FBFi=187.88 MPa, FBFFi=175.93 MPa, TBFi=149.93 MPa), with no differences between LUi and Z350i. TBFi presented the lowest characteristic stress (149.93 MPa) among all immediately tested groups.

The characteristic stress of all groups significantly decreased after thermocycling. Among aged groups, the highest values were observed for LUa (186.71 MPa) and Z350a (188.94 MPa) and the lowest for FBFFa (90.34 MPa). Intermediate values were observed for FBFa (151.6 MPa) and TBFa (122.77 MPa). The Weibull modulus was not significantly

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different for the same material tested immediately and after aging, except for FBFa, which showed a significantly lower Weibull modulus after aging (m=3.12).

The information regarding characteristic stress vs the Weibull modulus is instructively presented in a contour plot (Figure 1A), where differences between groups (95%, two-sided confidence intervals) are detected when an overlap does not exist between the contours. When plotting the probability of survival as a function of characteristic stress (Figure 1B), a clear trend in decrease of survival was observed for all aged groups. Also, the same differences between groups observed in the contour plot regarding characteristic stress were observed for probability of survival between groups.

Fractographic analyses showed different fracture patterns for the LU and Z350 groups when compared with bulk-fill and bulk-fill flow resin composites. LU and Z350 composites fractured in several pieces (three to five), while bulk fill composites fractured in fewer pieces (two or three). Fractographic analysis showed that regardless of aging or group, fractures always initiated on the tensile side, leaving marks such as hackles which depicted the direction of crack propagation, and a surface flaw as the fracture origin (Figure 2).

DISCUSSION

Composite resins have been widely used as a more esthetic, mercury-free alternative for amalgam restorations. Although clinical research data have shown high survival rates for resin composite restorations, secondary caries and bulk fractures are still the main reasons for clinical failure.[25] Both issues are commonly associated with sensitive restorative techniques, polymerization shrinkage, and hydrothermal degradation.[9,26]

In order to overcome such problems, efforts in polymer chemistry engineering have led to the development of bulk-fill resin composite materials with reduced volumetric shrinkage and increased depth of cure that allows the clinician to place increments up to 4 or 5 mm thick.[27] Considering that degree of conversion and material composition are determinants of the mechanical properties of resin composites, modifications in their composition to tailor polymerization shrinkage are challenging when fracture strength needs to be maintained or improved.

The enhanced translucency of bulk-fill resin composites allows greater light transmission and deeper polymerization in greater increments. Further, modification in the composition of resin composites, such as the addition of stress-relieving monomers, different and more reactive photoinitiators, and the incorporation of specific types of fillers, such as prepolymerized particles and glass fibers, allows modulation of the polymerization reaction.[28] Nevertheless, the introduction of prepolymerized particles as inorganic fillers may result in a decrease in mechanical properties due to a relatively low ceramic particle volume.

Among tested groups, TBF presented the lowest ceramic filler content (approximately 61% [vol] and 17% for prepolymerized polymer fillers or "isofillers"), which may explain its lowest characteristic stress among all immediately tested groups. The results of BFS testing showed a direct correlation between inorganic content and BFS for all immediately tested materials, which led us to accept our first hypothesis that composition and manufacture method would influence the characteristic stress and Weibull modulus of composite resins. Two composites containing a high content of ceramic particles (zirconia and silica), regardless of fabrication method (layered Z350 or milled LU), presented the highest characteristic stress among all groups. With lower filler content, intermediate characteristic stress values were observed for FBF and FBFF with no statistical difference between them. In contrast, a significantly higher Weibull modulus was observed for FBF than FBFF, likely due to filler volume differences (76.5% for FBF and 64.5% for FBFF) that eventually led to a higher flaw population in the flowable bulk-fill composite.

Among a great variety of factors, mechanical properties of composite resins depend on their composition and manufacture. Highly inorganic filled composite resins have been associated with improved flexural strength[29] and Young's modulus,[30] while a greater organic content has been commonly associated with increased hydrothermal degradation and greater polymerization shrinkage. In addition, defects and porosities may work as stress raisers, frequently associated with failure origin, as shown in our fractographic analysis. Within this context, CAD/CAM resin-based composite materials have been developed as an alternative to conventional layering of composite resins for the manufacture of indirect restorations through the machining of homogeneous CAD/CAM blocks. Although higher characteristic stress and Weibull modulus were expected for CAD/CAM blocks, no differences were observed between layered and

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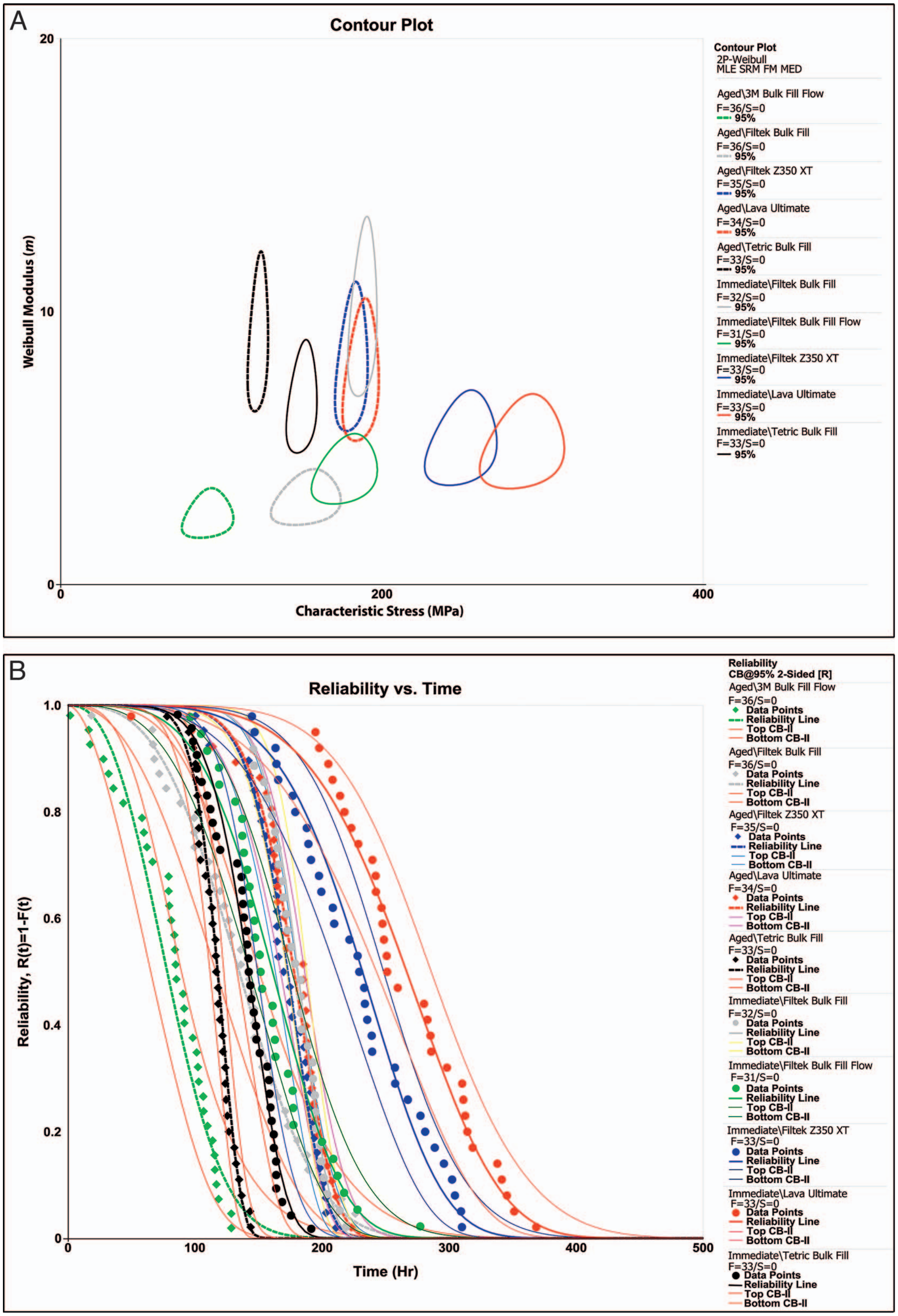

Figure 1. *(A): Contour plot of characteristic strength (MPa) vs Weibull modulus (m). Dotted contour lines indicate aged samples. (B): Probability of survival vs characteristic strength shows a significant decrease in survival for all aged composites. Lowest survival was observed for FBFFa, intermediate values for FBFa and TBFa, and significantly higher values for LUa and Z350a (non-overlap between upper and lower confidence intervals shown by the red lines).*

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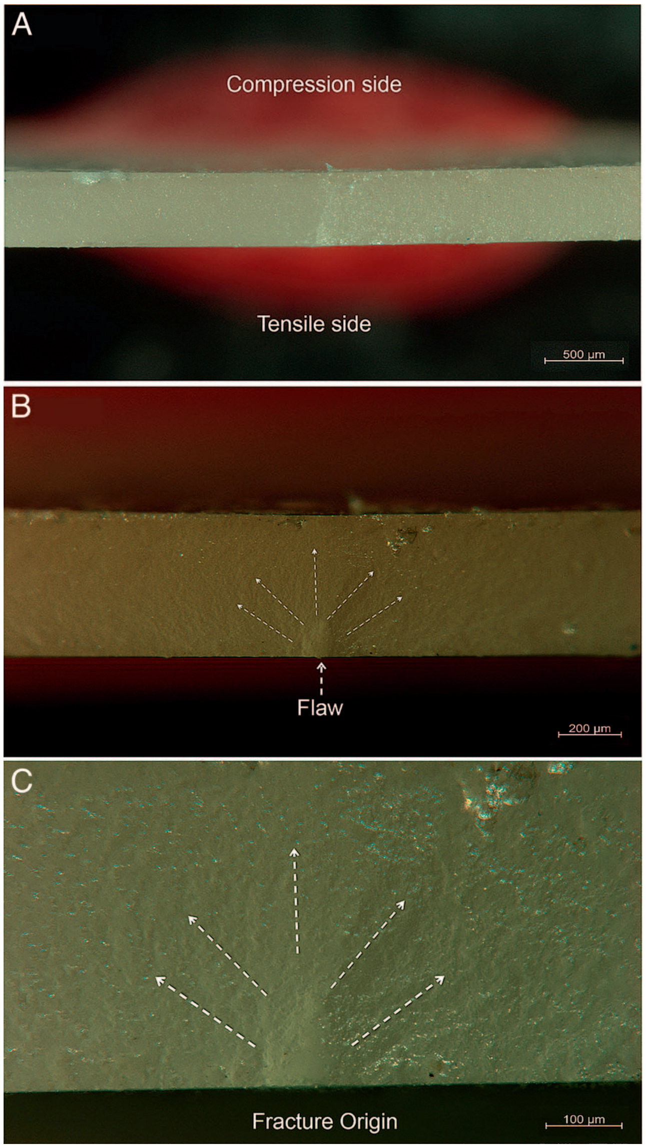

Figure 2. *(A): Representative sample fractured in biaxial flexural strength showing compressive and tensile sides under low magnification. (B): Higher magnification at the bottom center of the specimen shows hackles that allow trace back to the failure origin shown in higher magnification (C).*

milled resin "nanoceramics" tested immediately. Also, thermocycling reduced these parameters in the two composite resins similarly, suggesting that flaw population differences were not great enough to affect their characteristic stress and Weibull modulus. Thus, our second hypothesis was also accepted.

The present study used 500 thermal cycles, an amount that has been suggested to represent approximately six months of *in vivo* usage.[16,31] Other authors consider that 10 thermal cycles represent one day of use.[32] However, the correlation between the number of thermal cycles and clinical service is still debated with limited conclusive evidence between the *in vitro* simulation and *in vivo* aging of resin composites.[16] Regardless of the protocol used, thermocycling protocols have been reported to be efficient in promoting degradation of the mechanical properties of resin composites[15,33] as observed in this study.

Among several options for flexural testing described in the materials literature, a piston-on-ring test was used in the present study, as have others,[24,28] to determine the biaxial flexural strength. The main advantage of this test consists in the concentration of the maximum tensile stresses in the central loading area where the failure is initiated, leading to an accurate measurement of the load necessary to fracture the specimen (further plotted in strength) and the elimination of spurious edge failures frequently seen in three-point flexure testing.[34]

The Weibull distribution was selected for the statistical analysis due to its ability to evaluate the properties, characteristic stress, and Weibull modulus. Mean strength values associated with standard deviations have been frequently used to report composite resin strength data;[34] however, the lack of homogeneity in the distribution of the flaws from specimen to specimen may result in failures at lower stresses.[35]

The Weibull modulus (m) measures the variability of the results, where higher m values indicate greater structural reliability (higher homogeneity), whereas lower m values reflect more flaws in the structure (lower homogeneity) and thus a decrease in reliability. The clinical translation is that a resin composite with a higher Weibull modulus denotes a more homogeneous distribution of the flaws and a more reliable clinical performance (failure in a narrower range of stress). When BFS values are evaluated as characteristic stress, the data indicate the load at which 63.2% of the specimens of each group would fail.[36,37]

Success of composite resin restorations is multifactorial and several variables are involved.[38] There are clinical-, patient-, professional-, and material-related factors that may influence the survival of dental restorations.[39] Understanding of flexural strength ranking is a starting point in material selection, along with clinical indications and patient factors.[38] Clinical studies are warranted to determine the survival of different restorative materials under controlled scenarios.

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CONCLUSIONS

Composition and thermocycling significantly influenced the BFS of resin composite materials. Future clinical studies should evaluate if specific recommendations based on cavity size and tooth location should be made to improve their long-term performance.

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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.

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