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Effect of Preheating and Fatiguing on Mechanical Properties of Bulk-fill and Conventional Composite Resin

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Clinical Relevance

Bulk-fill composite resins may have comparable mechanical properties to conventional composite resin. Preheating does not reduce the mechanical properties of composite resins.

SUMMARY

Statement of Problem: Bulk-fill composite resins are increasingly used for direct restorations. Preheating high-viscosity versions of these composites has been advocated to increase flowability and adaptability. It is not known what changes preheating may cause on the mechanical properties of these composite resins. Moreover, the mechanical properties of these composites after mastication simulation is lacking.

Purpose: The purpose of this study was to evaluate the effect of fatiguing and preheating on the mechanical properties of bulk-fill composite resin in comparison to its conventional counterpart.

Methods and Materials: One hundred eighty specimens of Filtek One Bulk Fill Restorative (FOBR; Bulk-Fill, 3M ESPE) and Filtek Supreme Ultra (FSU; Conventional, 3M ESPE) were prepared for each of the following tests: fracture toughness (International Organization for Standardization, ISO 6872), diametral tensile strength (No. 27 of ANSI/ADA), flexural strength, and elastic modulus (ISO Standard 4049). Specimens in the preheated group were heated to 68°C for 10 minutes and in the fatiguing group were cyclically loaded and thermocycled for 600,000 cycles and then tested. Two-/one-way analysis of variance followed by Tukey Honest Significant Difference (HSD) *post hoc* test was used to analyze data for statistical significance (α=0.05).

Results: Preheating and fatiguing had a significant effect on the properties of both FSU and FOBR. Fracture toughness increased for FOBR specimens when preheated and decreased when fatigued (*p*=0.016). FOBR had higher fracture toughness value than FSU. Diametral tensile strength decreased significantly after fatiguing for FSU (*p*=0.0001). FOBR had a lower diametral tensile strength baseline value compared with FSU (*p*=0.004). Fatiguing

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significantly reduced the flexural strength of both FSU and FOBR (p=0.011). Preheating had no effect on the flexural strength of either FSU or FOBR. Preheating and fatiguing significantly decreased the elastic modulus of both composite resins equally (p>0.05).

Conclusions: Preheating and fatiguing influenced the mechanical properties of composite resins. Both composites displayed similar mechanical properties. Preheating did not yield a major negative effect on their mechanical properties; the clinical implications are yet to be determined.

INTRODUCTION

Composite resin was introduced to the dental world by Rafael Bowen in 1957.[1] Composite resins have changed the way dentistry is practiced and have become one of the most important dental materials. Approximately 261 million composite resin restorations are placed around the world each year.[2] However, polymerization shrinkage and its resultant shrinkage stress is one of the major shortcomings of conventional composite resin.[3] The resultant polymerization shrinkage stress manifests clinically with several clinical complications such as cusp deflection, micro-cracking and fracture of enamel margins, microleakage, debonding, postoperative sensitivity, and pulpal irritation.[4-13]

To reduce shrinkage stresses, an incremental technique for composite resin placement was introduced. This technique has insured proper light polymerization of the resin composite and reduced the polymerization shrinkage stress.[14,15] However, incremental placement of conventional composite resin is a technique sensitive procedure that requires clinical skills and special instruments, and it is also time consuming. As a result, less technique-sensitive and more efficient approaches for composite resin placement were required.

Manufacturers realized desirability of a material that is simply and rapidly placed with reduced polymerization shrinkage. Their efforts resulted in the introduction of bulk-fill composite resins, a wide range of materials that can be placed and photopolymerized in a single layer of 4-8 mm thickness. Several changes in the chemistry of monomers, particle size, and shape were required to allow those materials to be used in bulk. Bulk-fill composite resins can be classified into low-viscosity and high-viscosity materials. Low-viscosity bulk-fills are meant to serve as dentin replacement and therefore need to be capped with conventional materials due to their poor physical and mechanical properties.[16] High-viscosity bulk-fill materials on the other hand have good physical and mechanical properties that allow them to restore an entire cavity without the need for a capping layer.[17,18]

Laboratory testing of the mechanical properties of composite resin materials is a common method for determining their properties. The clinical relevancy of those tests is established to some extent.[19] Mechanical properties testing can help identify materials with a high likelihood of premature failure due to fracture and their wear characteristics. Fatiguing of composite resins prior to mechanical testing by cyclic loading and simultaneous thermocycling is highly recommended to increase the clinical relevance of the results.[20] Chewing simulation is one of the methods that can be used to fatigue specimens at a reasonable cost and time.

There are multiple protocols and techniques that dentists use when they place composite resins in their practice. Preheating composite resin is a relatively common technique that is thought to increase flowability and reduce film thickness.[21] Preheating composite resin may maximize polymerization, reduce shrinkage forces, and increase surface hardness.[22-24] However, this effect on mechanical properties, wear, and clinical performance has yet to be investigated.

The purpose of the present study was to test the effect of fatiguing and preheating on the mechanical properties of a high-viscosity bulk-fill composite resin and compare it to its conventional counterpart. The null hypotheses were that fatiguing and preheating yield no significant effect on the mechanical properties of the tested materials. Also, there is no significant difference in the mechanical properties between high-viscosity bulk-fill and conventional composite resin.

METHODS AND MATERIALS

Two composite resin types were used in this study (Table 1): Filtek One Bulk Fill Restorative (FOBFR; 3M ESPE, St Paul, MN, USA) and Filtek Supreme Ultra (FSU; 3M ESPE).

Specimen Distribution and Group Descriptions

Specimens (N=180) were prepared and distributed into six groups (n=10) for each test performed: group 1, FOBR baseline; group 2, FSU baseline; group 3,

Table 1: *Composite Resins Used in This Study*

Product	Type	Manufacturer	Matrix Composition	Filler Type	Filler Size (nm)	Filler Load (weight %)
Filtek Supreme Ultra, A2	Conventional nanofilled composite resin	3M ESPE	Bis-GMA, UDMA, TEGDMA, bis-EMA	Ytterbium trifluoride, nonaggregated silica, nonaggregated zirconia, zirconia/silica clusters	4-20	78.5
Filtek One Bulk Fill Restorative,A2	Bulk-fill nanofilled composite resin	3M ESPE	AFM, AUDMA, UDMA, and DDDMA	Ytterbium trifluoride, nonaggregated silica, nonaggregated zirconia, zirconia/silica clusters	4-20	76.5

Abbreviations: AFM, addition fragmentation monomer; AUDMA, aromatic urethane dimethacrylate; bis-EMA, Ethoxylated bisphenol-A dimethacrylate; bis-GMA, bisphenol-A glycidyldimethacrylate; DDDMA, 1, 12-Dodecanediol dimethacrylate; TEGDMA, Triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate.

FOBR preheated; group 4, FSU preheated; group 5: FOBR fatigued; group 6, FSU fatigued.

Specimen preparation and photo-polymerization were done according to manufacturer's instructions at room temperature (23±1°C), except for groups 3 and 4 that were preheated to a temperature of (68±1°C) for 10 minutes before photo-polymerization by using a composite warmer (HeatSync, Bioclear, Seattle, WA, USA).

Specimens of groups 5 and 6 were subjected to fatiguing. This was achieved by cyclic loading the specimens under 50 N load with a steatite antagonist using a chewing simulator (CS-4.8, SD Mechatronik, Feldkirchen-Westerham, Germany) for 600,000 cycles at 1.4 Hz. Thermocycling was simultaneously performed using distilled water at 5°C and 55°C with a 30-second dwell time. Specimens were inspected for premature failure every 12 hours. Each chamber was supplied with a sensor that indicated if a specimen were to fracture at a specific cycle number. Specimens were tested for their mechanical properties.

Fracture Toughness

The single edge V-notched beam (SEVNB) method (ISO 6872)[25] was used to measure the fracture toughness of FOBFR and FSU composite resins. Sixty beam-shaped specimens 21.0 ± 0.1 mm in length, a rectangular cross section of 4.0 ± 0.1 mm in depth, and 3.0 ± 0.1 mm in thickness were prepared for each of the composite resins. Polyvinylsiloxane impression material (PVS) molds were created to the exact dimensions for easy removal after photo-polymerization. Composite resin material was injected into the mold preventing the entrapment of air bubbles. A transparent ethylene film and glass slide were placed with slight pressure over the mold to confine the material and minimize exposure to oxygen from the atmosphere during photo-polymerization. Each specimen was photo-polymerized according to the manufacturer's recommended time of exposure using a visible light curing unit (Elipar DeepCure-S, 3M ESPE, St. Paul, MN USA) with a useable wavelength range of 420-490 nm and mean light irradiance of 1168 mW/cm^2. The wavelength and irradiance of the curing unit were calibrated and confirmed using the MARC Light Collector (BlueLight Analytics, Halifax, Canada). After completion of photo-polymerization, the specimens were examined and any containing voids/defects were excluded from testing. A #15 blade was used to remove excess composite resin from the edges, and a 600-grit silicon-carbide abrasive paper (MicroCut, Buehler, Lake Bluff, IL, USA) was used for final smoothing. Specimens were then stored in deionized water at 37°C for 24 hours. The width (b) and thickness (w) of each specimen were recorded prior to testing using a digital micrometer capable of measurements to ±1 μm accuracy (QuantuMike Micrometer, Mitutoyo Corporation, Kawasaki, Japan). A notch depth of approximately 0.5 mm was cut into the bar specimen using a 150 μm-thick diamond blade. A razor blade impregnated with diamond polishing paste (3.5 μm, Kent Supplies, New York, NY, USA) was positioned in the notch, and a light force (5-10 N) was applied using a gentle back and forth horizontal motion while maintaining a constant pressure. The depths of the V-shaped notches were measured and confirmed from both sides with a calibrated microscope. An acceptable notch depth measured between 0.8 and 1.2 mm. For groups 5 and 6, the V-shaped notch was made after the fatiguing process.

Fracture toughness testing was performed using a four-point bending fixture. The 3-mm-width face with the V-notch was placed down on the testing fixture (tensile side). Specimens were loaded on an Instron Universal Testing Machine (Instron 4411, Instron, Norwood, MA, USA) with a crosshead speed of 0.5 mm/min until fracture.

The peak fracture load was recorded to three significant figures, and the fracture toughness [K_{IC} (MPa$\times$m$^{1/2}$)] was determined according to the following formula:

$$K_{IC} = F/bw^{½} * L/w * 3\alpha^{½}/2(1-\alpha)^{3/2} * Y;$$

$$Y = 1.9887 - (1.326 * \alpha) - (3.49 - 0.68 * \alpha + 1.35 * \alpha^2) * (\alpha) * (1-\alpha)/(1+\alpha)^2$$

where K_{IC} is the fracture toughness (MPa$\times$m$^{1/2}$); α = average V-notch depth of group; F = fracture load; b = width of specimen; w = thickness of specimen; L = distance between support beams; and Y is the stress intensity shape factor.

Diametral Tensile Strength

The diametral tensile strength (DTS) of FOBFR and FSU was determined under specification (No. 27 of ANSI/ADA, 1993).[26] Sixty cylindrical shaped specimens (6.0±0.1 mm diameter and 3.0±0.1 mm height) were prepared for each composite resin material according to a similar methodology previously described. Specimens were immersed in water at 37°C for 24 hours prior to testing. The cylindrical-shaped specimens were positioned on their side between two compression plate fixtures (Instron 4411, SINTECH, MTS System Corporation). Specimens were loaded at a crosshead speed of 0.5 mm/min until fracture. For groups 5 and 6, specimens were fatigued as described previously prior to loading. The peak load was recorded, and the DTS was determined according to the following formula:

$$DTS = 2F/\pi dt,$$

where F = maximum force applied; d = diameter of specimen; and t = thickness of specimen.

Flexural Strength and Young's Elastic Modulus

The flexural strength of FOBFR and FSU was determined by a three-point bending test according to ISO Standard 4049.[27] Sixty specimens (2.0±0.1 mm thickness, 2.0±0.1 mm width, and 25.0±0.1 mm length) were prepared for each composite resin material according to the previously described methodology. Testing was performed using the three-point bending fixture (Instron 4411, SINTECH, MTS System Corporation) with a crosshead speed of 0.5 mm/min. For groups 5 and 6, specimens were fatigued as described previously prior to loading. Flexural strength was determined according to the following formula:

$$\alpha = 3FL/2wt,$$

where F = maximum force applied; L = distance between support beams; w = width of specimen; and t = thickness of specimen.

The elastic modulus (Young's modulus) was determined using Test Works software (SINTECH, MTS System Corporation, Shakopee, MN, USA). The software required the dimensions of each specimen prior to starting the test. After fracture, the software yielded an elastic modulus based on the dimensions and flexibility of the specimen.

Statistical analysis was performed using analysis of variance (ANOVA) and Tukey's multiple comparison tests for pairwise comparisons at $p<0.05$ and 95% CIs to test significant differences in fracture toughness, flexural strength, elastic modulus, and DTS. The Statistical Package for the Social Sciences (SPSS) version 23.0 (SPSS/IBM, Armonk, NY, USA) was used for statistical analysis.

RESULTS

Fracture Toughness

Mean and SD values of fracture toughness for each group are summarized in Table 2. Preheating and fatiguing had no significant effect on the fracture toughness value of FSU ($p>0.05$). For FOBR, preheating and fatiguing had a significant effect on the fracture toughness value ($p=0.016$). Preheating increased fracture toughness, whereas fatiguing decreased fracture toughness (Figure 1). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that FOBR has significantly better fracture toughness values for all conditions ($p<0.05$).

DTS

Mean and SD values of DTS for each group are summarized in Table 3. In the FSU groups, preheating had no significant effect on the DTS ($p>0.05$), whereas fatiguing had a significant effect on DTS ($p=0.0001$; Figure 2). For FOBR, preheating and fatiguing had no significant effect on the DTS ($p>0.05$). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that FSU had a significantly higher DTS value for baseline only ($p=0.004$).

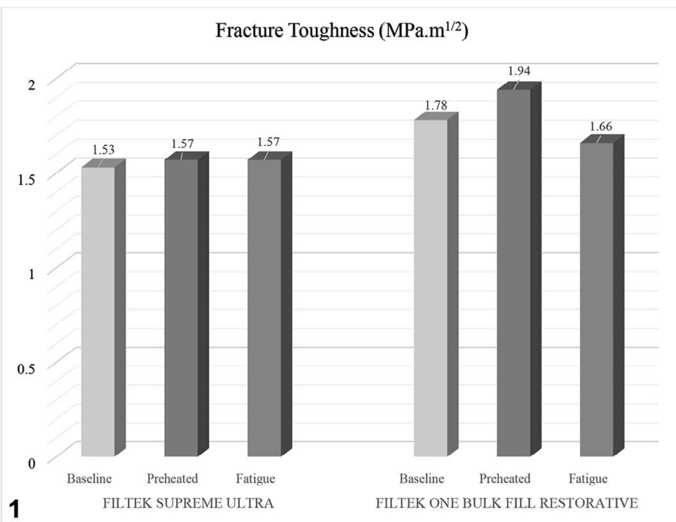
Table 2: *Mean and SD of Fracture Toughness (MPa × $m^{1/2}$) of Both Composite Resin Types*[a]

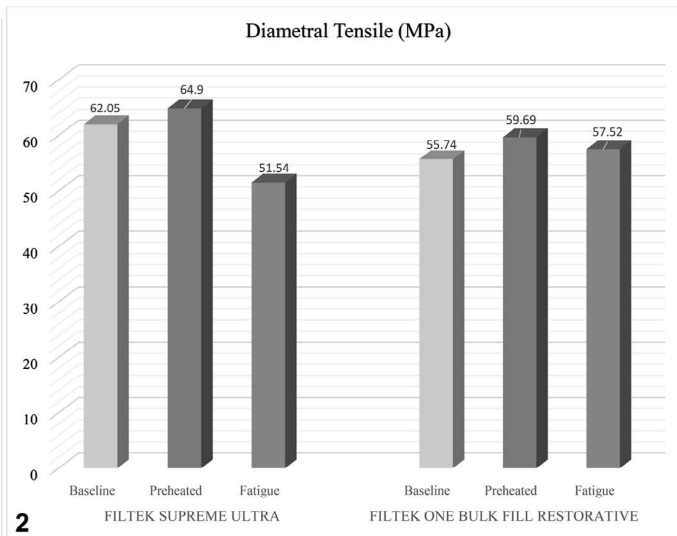
Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	1.53 ± 0.21	1.57 ± 0.13	1.57 ± 0.11
Filtek One Bulk Fill Restorative	1.78 ± 0.13 A	1.94 ± 0.16 Ab	1.66 ± 0.07 Aa

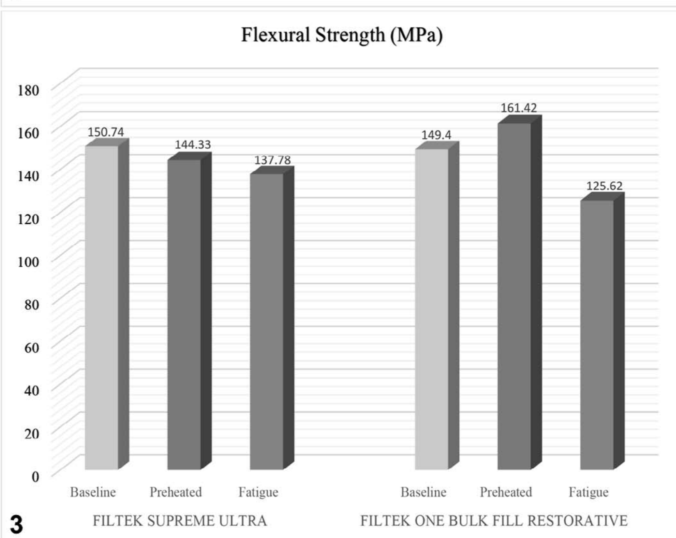
[a] *Uppercase letters indicate statistical significance between types of composite resin (*p>*0.05). Lowercase letters indicate statistical significance within the same composite resin (*p>*0.05).*

Flexural Strength

Mean and SD values of flexural strength for each group are summarized in Table 4. For FSU and FOBR, preheating had no significant effect on flexural strength ($p>0.05$), whereas fatiguing had a significant effect on flexural strength ($p=0.011$; Figure 3). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that FSU had significantly higher flexural strength in the fatigued group ($p<0.05$), whereas FOBR had significantly higher flexural strength in the preheated group ($p=0.045$).







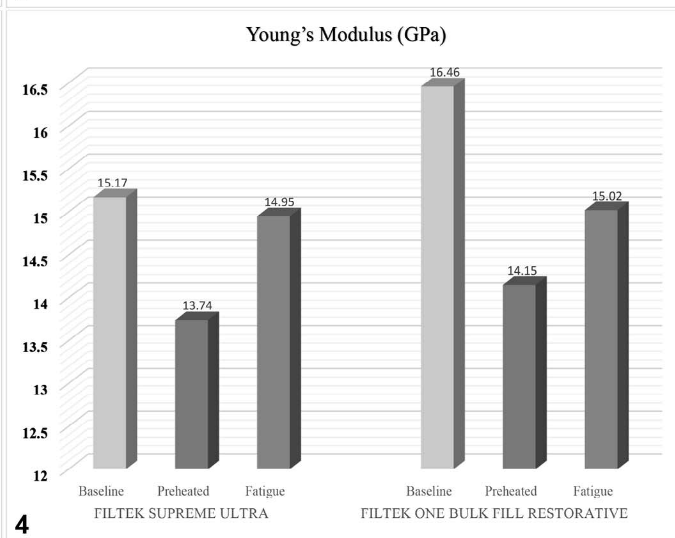


Figure 1. *Mean of fracture toughness of each composite resin type.*
Figure 2. *Mean of diametral tensile strength each composite resin type.*
Figure 3. *Mean of flexural strength each composite resin type.*
Figure 4. *Mean of Young's modulus each composite resin type.*

Table 3: *Mean and SD of Diametral Tensile Strength (MPa) of Both Composite Resin Types*[a]

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	62.05 ± 5.06 A	64.90 ± 7.74	51.54 ± 7.80 a
Filtek One Bulk Fill Restorative	55.74 ± 3.34	59.69 ± 6.70	57.52 ± 5.08

[a] *Uppercase letters indicate statistical significance between types of composite resin* (p>0.05). *Lowercase letters indicate statistical significance within the same composite resin* (p>0.05).

Young's Elastic Modulus

Mean and SD values of Young's elastic modulus for each group are summarized in Table 5. Regarding FSU, preheating and fatiguing had a significant effect on Young's modulus ($p<0.05$). For FOBR, preheating and fatiguing had a significant effect on Young's modulus ($p<0.05$; Figure 4). A pairwise comparison between FSU and FOBR for baseline, preheated, and fatigued showed that there is no significant difference in Young's elastic modulus between the two composite resin types ($p>0.05$).

DISCUSSION

The effect of preheating and fatiguing on the mechanical properties of two composite resins, bulk-fill and conventional, was tested in this *in vitro* study. The first null hypothesis was rejected because both preheating and fatiguing yielded a statistically significant difference on some of the mechanical properties of the two tested composite resins. The second null hypothesis was also rejected because there was a statistically significant difference in the mechanical properties between bulk-fill and conventional composite resins. Some differences were found at the baseline and others were found in the preheating and fatiguing groups.

Preheating is a common practice to increase flowability and reduce stickiness of the composite resins. Preheating is especially useful for composite resins with a high percentage of inorganic filler particles that are highly viscous. It has been shown that preheating these composites ensures better adaptation to cavity walls.[28] The two composites that were chosen for this study are highly filled and viscous. The FOBR has 76.5 wt% filler load, whereas FSU has 78.5 wt% filler load. FSU served as a control, and it can be used for the same applications that are indicated for FOBR.

Composite resin restorations may fail over time due to the accumulation of damage produced by cyclic forces (fatiguing).[19] Therefore, laboratory fatiguing of composite resins prior to testing is a valid technique to increase the clinical relevancy of the generated results.[20] This can be done by using simulators that cyclically load specimens with simultaneous thermocycling, in an effort to emulate the intraoral challenge. Hence, both materials were fatigued for 600,000 cycles, which is equivalent to 2.5 years of clinical performance.[19] Loading parameters included a 0.5-mm indentation with steatite indenter, vertical movement, 1.4 Hz, and a load of 49 N. These parameters were selected after multiple pilot tests to determine the best parameters. All samples survived the fatiguing challenge in this study.

Fracture toughness is considered the most important mechanical property in determining resistance to fracture because almost all materials contain flaws.[29] In this study, preheating increased the fracture toughness of both FOBR and FSU, with fracture toughness values of FOBR significantly higher. A possible explanation for this may be due to the different monomer of FOBR (Table 1) that may enhance a better degree of conversion after preheating. Daronch and others[23] clearly showed that preheating significantly enhanced monomer to polymer conversion of composite resin due to increasing mobility of the monomer and filler particles. The enhanced polymerization yielded an increase in the fracture toughness. Fatiguing, on the other hand, had no significant effect on the fracture toughness of FSU, but it decreased the fracture toughness of FOBR. Fatiguing with thermocycling may have led to filler particle loss and surface changes that

Table 4: *Mean and SD of Flexural Strength (MPa) of Both Composite Resin Types*[a]

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	150.74 ± 11.52	144.33 ± 7.00	137.78 ± 7.27 aA
Filtek One Bulk Fill Restorative	149.40 ± 13.66	161.42 ± 4.40 A	125.62 ± 16.28 a

[a] *Uppercase letters indicate statistical significance between types of composite resin* (p>0.05). *Lowercase letters indicate statistical significance within the same composite resin* (p>0.05).

Table 5: *Mean and SD of Elastic Modulus (GPa) of Both Composite Resin Types[a]*

Composite Resin	Baseline	Preheated	Fatigued
Filtek Supreme Ultra	15.17 ± 0.73	13.74 ± 1.35 b	14.95 ± 0.60 a
Filtek One Bulk Fill Restorative	16.46 ± 1.43	14.15 ± 2.03 b	15.02 ± 1.05 a

[a] *Uppercase letters indicate statistical significance between types of composite resin ($p>0.05$). Lowercase letters indicate statistical significance within the same composite resin ($p>0.05$).*

decreased fracture toughness. Those changes can be due to chemical breakdown by hydrolysis, stress-induced effects, and chemical composition changes by leaching or loss of strength due to corrosion.[30] Because fracture toughness is a function of microstructure, differences in chemical composition may explain why fatiguing decreased the fracture toughness of FOBR but not that of FSU. Baseline fracture toughness values of both composite types were comparable to other studies.[31,32] Tiba and others[33] investigated the fracture toughness of a range of bulk-fill composite resin materials ranging from 0.8 to 1.7 MPa $\times$ m$^{1/2}$. To the authors' knowledge, no published study has tested the effect of preheating and fatiguing on this property.

Many clinical failures of composite resin restorations are related to tensile stress. The DTS test, which is an indirect method to assess tensile strength, was performed in this study. Baseline DTS of FSU was significantly higher than that of FOBR. Schliebe and others[34] had a similar result in their study where the conventional composite resin had higher DTS than its bulk-fill counterpart. Preheating increased the DTS for both composite resins, but it was not statistically significant. This finding was similar to what was found by Nada and others.[35] This can be attributed to the enhanced polymerization of preheated composite resins. Fatiguing had a significant effect reducing the DTS of FSU (51.54 MPa). No other published studies were found that tested the effect of fatiguing on DTS.

Flexural strength and elastic modulus are two important qualities of composite resin materials. For the material to withstand masticatory forces, it should offer sufficient flexural strength to allow the material to resist fracture. The elastic modulus determines the stiffness of the material. The evaluated composites in all groups had adequate flexural strength according to ISO 4049-2009, which requires a value of at least 80 MPa. The flexural strength of both composites was not affected by preheating but was decreased by fatiguing. The results of this study are in agreement with the findings of Uctasli and others, who concluded that preheating had no effect on flexural strength of composite resins.[36] Although the clinical significance of this is unknown, fatiguing these composite resins seems to affect their mechanical properties. The literature that describes the effect of fatiguing is scarce, but it can be speculated that the microstructure is affected when those materials are fatigued. There was no difference in elastic modulus between the two composites (baseline). Preheating and fatiguing significantly reduced the elastic modulus of both FSU and FOBR. However, the clinical significance of this reduction is unknown, and data cannot be extrapolated to clinical outcomes without highlighting deficiencies in this method, such as flaw distribution and structural reliability of the material.[37,38] Nonetheless, this method is recommended by ISO 4049 for polymer-based materials and is applied for comparative purposes.

A limitation of this study is that only one brand of composite resin material was tested. Thus, the results of study cannot be extrapolated to other brands of composite resin. It is well known that not all brands are similar. Different brands of composite resin offer a wide range of materials with various chemical formulations that may result in different mechanical properties. Another limitation of the study is that the specimens used in this study are flat following ISO standards and do not have anatomical geometry that simulates the clinical scenario. Also, testing at different temperatures, and its effect on the mechanical properties, was not investigated and is encouraged for future research. This laboratory study does not substitute the need for well-conducted randomized controlled clinical trials.

Within the limitations of this laboratory study, the following conclusions were drawn:

- preheating had no major negative effect on the mechanical properties of composite resins;
- laboratory fatiguing yields useful information by emulating the intraoral challenge and predicting the effect of that on the properties of composite resins;

- conventional and bulk-fill composite resins have minimal difference in their mechanical properties; and
- the clinical implication of changes in the mechanical properties yielded by preheating is yet to be determined.

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Conflict of Interest

The authors of this manuscript 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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