



# Effects of preheating on degree of conversion, flexural strength and flexural modulus of bulk-fill resin composites

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**Purpose:** The aim of this study was to investigate the effects of preheating on the degree of conversion (DC), flexural strength (FS) and flexural modulus (FM) of bulk-fill resin composites with varying viscosities and filler contents.

**Methods:** Four bulk-fill resin composites - EverX Posterior (EXP), EverX Flow Bulk (EXF), Filtek One Bulk Fill (ONE), SDR Plus Flow (SDR) - and a conventional composite - Filtek Z250 (Z250) - as a control group were evaluated. Each composite was divided into three subgroups: [1] Room temperature (20±1°C), [2] preheated to 39°C, and [3] preheated to 55°C. DC, FS and FM results of the specimens were evaluated. Statistical analysis included one-way ANOVA and post hoc Tukey tests (p<0.05).

**Results:** At room temperature, Z250 exhibited the lowest DC (41.32%), significantly lower than EXP (52.11%) and other bulk fill composites. FS values for Z250 (145.68 MPa), EXF (152.19 MPa) and EXP (157.24 MPa) at room temperature were comparable but higher than for ONE (100.70 MPa) and SDR (114.95 MPa). Preheating increased the DC and FS of Z250 and EXP but not of EXF, ONE or SDR.

**Conclusion:** Preheating is recommended to enhance the properties of viscous, fiber-reinforced (EXP) and conventional hybrid (Z250) composites for stress-bearing restorations. In contrast, it offers no significant mechanical advantage for the flowable bulk-fill composites tested (EXF, SDR, ONE), underscoring that the benefit of preheating is highly material-dependent.

**Keywords:** Bulk-fill; degree of conversion; flexural modulus; flexural strength; preheating.

## Introduction

Resin composites are among the most widely used restorative materials in dentistry. With the technological developments in the polymers and fillers it contains, resin composites with diverse properties that can be preferred for different clinical situations have been developed (1). Among these innovations, ‘bulk-fill’ resin composites have been developed, offering the significant clinical advantage

of being placed in thicker layers than conventional resin composites, which is particularly beneficial in large posterior cavities (2).

Low and high viscosity bulk-fill resin composites generally have different polymerisation initiator system designed to provide higher transparency and better deep polymerisation compared to conventional resin composites. These properties allow them to be placed in a single layer with a

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thickness of 4 mm or even 5 mm without the need for incremental layering, while still achieving a sufficient degree of conversion (DC) to ensure high physical and mechanical properties (3). DC of the C=C double bonds significantly influences critical properties such as physical, mechanical and biocompatibility of resin composites, with higher DC generally leading to improved mechanical properties (4). Many bulk-fill resin composites have been investigated for various parameters such as DC, polymerisation stress, mechanical properties or microleakage. Some studies suggest that bulk-fill composites are similar to conventional resin composites in terms of physical and mechanical properties (5-7). However, some other studies have found that bulk-fill resin composites used in 4 mm layers have reduced physical and mechanical properties compared to conventional composites used in 2 mm layers (8-10).

The physical and mechanical properties of resin composites are directly related to the composition of the material (11,12). In 2013, a short fiber-reinforced composite (everX Posterior; GC, Tokyo, Japan) was introduced to replace lost dentin with a material with similar mechanical properties (13). This composite, which can be used in 4mm layers, has very good mechanical properties in the bulk-fill resin composite group (14,15). Due to the high viscosity of everX Posterior, the manufacturer launched a low viscosity form in 2019 (everX Flow Bulk; GC, Tokyo, Japan) (16).

Preheating resin composites prior to their placement in the cavity is a clinically utilized technique to potentially enhance their handling and properties (17). The primary clinical advantage is the increased fluidity of the material, which facilitates easier placement and improved adaptation to the prepared cavity walls (18,19). Furthermore, a fundamental benefit of preheating is its ability to increase the degree of conversion (DC) of the resin matrix (20). This enhanced polymerization efficacy, in turn, contributes to the improvement of the material's mechanical properties (21). However, studies have also demonstrated that these beneficial effects are not universal and may vary depending on the specific technique employed and the particular composition of the resin composite (22).

Tooth or restoration fractures occurring in teeth restored with resin composites are the most important factors affecting the success of the treatment (23,24). The material properties related to fractures, such as crack propagation resistance, deformation under occlusal load, and marginal degradation, are typically evaluated by determining the basic material parameters of flexural strength (FS), flexural modulus (FM) and fracture toughness (25). The term "flexural strength" is defined as the failure stress of a material, as measured in bending. In contrast, the term

"flexural modulus" is defined as the stiffness of a material, as measured in bending (26).

While the effects of preheating on conventional resin composites have been investigated, there is a lack of evidence regarding its influence on the newer generation of short fiber-reinforced bulk-fill composites (EXP and EXF). Therefore, this study aimed to evaluate the effects of preheating at two different temperatures (39°C and 55°C) on the DC, FS, and FM of a range of bulk-fill composites with varying viscosities and filler contents, with a specific focus on addressing this gap in the literature.

The null hypotheses were as follows:

1. There is no difference in the DC, FS and FM between different resin composites.
2. The preheating process does not affect the DC, FS and FM of resin composites.

## Materials and Methods

This in vitro study was conducted following the 2021 PRILE guidelines (27). In this study, four different bulk-fill resin composites: EverX Posterior (GC Corporation, Tokyo, Japan), Everx Flow Bulk (GC Corporation, Tokyo, Japan), 3M Filtek One Bulk Fill (3M ESPE, St. Paul, MN, USA), SDR Plus Flow (Dentsply, Milford, DE, USA) and a conventional composite as a control group: Z250 (3M ESPE, St. Paul, MN, USA) were evaluated. The properties of all composites tested are shown in Table 1.

The sample size for the study was determined a priori using a power analysis. The analyses were performed using the G\*Power 3.1 software, based on the effect sizes reported in the study by Sarosi et al. (4). For a one-way ANOVA test, the significance level ( $\alpha$ ) was set at 0.05 and the statistical power ( $1-\beta$ ) at 0.80. For the DC, a very high effect size (Cohen's  $f \approx 0.8$ ) was used and 5 samples per experimental group (5 materials  $\times$  3 temperatures = 15 groups) were deemed sufficient. For the flexural tests, a medium effect size ( $f \approx 0.4$ ) was considered, leading to a requirement of 10 samples per group.

### Degree of Conversion

A stainless steel mold with a diameter of 10 mm and a thickness of 2 mm was used to prepare the specimens. A subgroup of each material (n=5) was prepared from resin composites that were stored at room temperature (20±1°C) and no preheating was applied. The other two subgroups were produced from resin composites that were preheated for 10 minutes in a preheating unit (Ena Heat Composite Heating Conditioner, Micrium, Genova, Italy) at T1 (39°C) and T2 (55°C) modes. The resin composites placed in the mold were covered with a mylar

**Table 1.** Resin composites tested in the study

Material	Manufacturer	Composition	Lot Number
3M Filtek Z250 (Z250) Microhybrid Resin Composite	3M ESPE, St. Paul, MN, USA	Resin Matrix: BIS-GMA, UDMA, BIS-EMA Inorganic Filler: zirconia-silica, particul size range of 0.01 – 3.5 µm Filler Load (w/V%): 77.5/60	10504771
everX Posterior (EXP) Short Fiber-Reinforced Composite	GC Tokyo, Japan	Resin Matrix: Bis-GMA, PMMA, TEGDMA Inorganic Filler: Short E-glass fiber filler, barium glass Filler Load (w/V%): 74.2/53.6	2309201
everX Flow (EXF) Flowable Short Fiber-Reinforced Composite	GC Tokyo, Japan	Resin Matrix: Bis-EMA, TEGDMA, UDMA Inorganic Filler: Short E-glass fiber filler, barium glass Filler Load (w/V%): 70/46	2312091
3M Filtek One Bulk Fill (ONE) High Viscosity Bulk Fill Resin Composite	3M ESPE, St. Paul, MN, USA	Resin Matrix: AUDMA, Dimethacrylate AFM, UDMA, DDMA Inorganic Filler: 20 nm silica particles, 4-11 nm zirconia particles, 100 nm ytterbium trifluoride, zirconia, and silica nanocluster Filler Load (w/V%): 76.5/58.4	10639667
SDR Plus Flow (SDR) Bulk Fill Flowable Resin Composite	Dentsply, Milford, DE, USA	Resin Matrix: UDMA, di-methacrylate resin, di-functional diluents Inorganic Filler: barium and strontium alumino-fluoro-silicate glasses, photoinitiators, colorants Filler Load (w/V%): 68/45	2305000193

Bis-GMA: bisphenol A glycidyl methacrylate; Bis-EMA: ethoxylated bisphenol A-dimethacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; PMMA: polymethylmethacrylate; AFM: addition-fragmentation monomer; AUDMA: aromatic urethane dimethacrylate; DDMA: 1,12-dodecane dimethacrylate; w: weight; V: volume.

strip and polymerised with an LED unit (3M Elipar, 1200 mW/cm<sup>2</sup>) for 20 seconds. The prepared specimens were stored in distilled water at 37°C for 24 hours. The DC was measured using Fourier Transform Infrared Spectroscopy (FTIR). The normalised absorbance of the functional group of the uncured ( $C=C_{\text{uncured}}/C=O_{\text{uncured}}$ ) and cured ( $C=C_{\text{cured}}/C=O_{\text{cured}}$ ) specimens was used to calculate the DC according to the formula:

$$DC = (1 - (C=C_{\text{cured}}/C=O_{\text{cured}}) / (C=C_{\text{uncured}}/C=O_{\text{uncured}})) \times 100$$

### Flexural Strength and Flexural Modulus

For the three-point bending test, a stainless steel mold with dimensions (25x2x2 mm) recommended by the ISO 4049/2009 specification was used to prepare the specimens. All composites were divided into 3 subgroups (n=10), as were the specimens prepared for the DC test. The composites placed in the mold were covered with a glass to obtain a smooth surface and excess composite was removed. The specimens were polymerised for 20 seconds at 3 points from both ends and the centre of the specimen using an LED unit with the tip in perpendicular contact with the glass surface. The back surface was then polymerised for 20 seconds at 3 points as on the front surface.

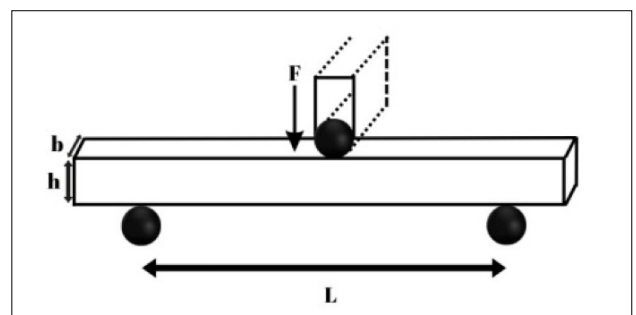
The dimensions of the specimens were measured using a digital caliper (Series 500 Caliper, Mitutoyo America Corp, Aurora, IL, USA) and any dimensional discrepancies were corrected using 400, 600 and 1200 grit SiC papers

until the desired values ( $\pm 0.01$  mm) were achieved. The prepared specimens were stored at 37°C for 24 hours. A three-point bending test (Fig. 1) was then performed using a 2 mm diameter cylindrical tip, a speed of 0.5 mm/min and a gap of 20 mm using a computer-controlled universal testing machine (DL 2000; Instron Universal Testing Machine). The breaking load (N) and deflection (mm) of the specimens were recorded. The FS in megapascals (MPa) and FM in gigapascals (GPa) were calculated using the following equations:

$$FS = 3FL/2bh^2$$

$$FM = (F/d) \cdot (L^3/4bh^3) \cdot (10^{-3})$$

\*F: Force (N), L: Distance between supports (mm), b: Specimen width (mm), h: Specimen height (mm), d: Amount of bending (mm)



**Fig. 1.** Three-point bending test.

## Statistical Analysis

SPSS (version 24.0; IBM, Armonk, NY, USA) was used for statistical analysis. The normality of the variables was analyzed with the Shapiro-Wilk test. As the data were normally distributed in all groups, one-way analysis of variance (ANOVA) and post-hoc Tukey tests were performed for all data, with  $p < 0.05$  deemed significant.

## Results

Table 2 shows the DC of all composites at all temperature groups. When the groups applied at room temperature were evaluated independently, Z250 (41.32%) had the lowest DC. Although the DC of EXP (52.11%) at room temperature was significantly higher than that of Z250, it was significantly lower than that of all other bulk-fill

composites. Preheating significantly increased the DC of Z250 and EXP, but had no significant effect on EXF, ONE and SDR.

FS values of all groups are presented in Table 3. When the groups applied at room temperature were evaluated independently, no difference was observed between Z250 (145.68 MPa), EXF (152.19 MPa) and EXP (157.24 MPa) in terms of FS. However, these composites exhibited significantly higher FS than ONE (100.70 MPa) and SDR (114.95 MPa). When the composites were evaluated individually, it was found that the preheating process did not affect the FS of EXF, ONE and SDR, but caused a significant increase in the FS of Z250 and EXP.

FM values of all groups are shown in Table 4. SDR showed significantly lower FM than all other composites

**Table 2.** Mean values of DC (%)

Material	Room Temperature	T1(39°C)	T2 (55°C)
Z250	41.32 (3.04) <sup>Aa</sup>	53.58 (3.41) <sup>Ab</sup>	60.62 (3.16) <sup>Ac</sup>
EXF	62.64 (3.74) <sup>Ba</sup>	60.67 (3.30) <sup>ABa</sup>	60.35 (3.44) <sup>Aa</sup>
EXP	52.11 (3.03) <sup>Ca</sup>	60.43 (3.85) <sup>ABb</sup>	64.86 (3.84) <sup>Ab</sup>
ONE	60.79 (3.90) <sup>Ba</sup>	59.62 (3.46) <sup>ABa</sup>	58.02 (3.22) <sup>Aa</sup>
SDR	63.87 (3.31) <sup>Ba</sup>	62.87 (4.07) <sup>Ba</sup>	61.54 (3.40) <sup>Aa</sup>

\*The same uppercase letters mean there is no statistically significant difference between the groups in same column. The same lowercase letters means that there is no significant difference between the groups in same line.

**Table 3.** Mean values of FS (MPa)

Material	Room Temperature	T1(39°C)	T2 (55°C)
Z250	145.68 (2.41) <sup>Aa</sup>	168.70 (3.73) <sup>ABb</sup>	151.11 (4.17) <sup>Aab</sup>
EXF	152.19 (5.64) <sup>Aa</sup>	146.60 (4.65) <sup>ACa</sup>	129.57 (2.32) <sup>Ba</sup>
EXP	157.24 (4.53) <sup>Aa</sup>	183.35 (1.36) <sup>Bb</sup>	170.00 (1.58) <sup>Aab</sup>
ONE	100.70 (5.72) <sup>Ba</sup>	122.07 (4.94) <sup>CDa</sup>	108.35 (5.55) <sup>Ba</sup>
SDR	114.95 (4.61) <sup>Ba</sup>	120.39 (2.10) <sup>Da</sup>	107.93 (5.28) <sup>Ba</sup>

\*The same uppercase letters mean there is no statistically significant difference between the groups in same column. The same lowercase letters means that there is no significant difference between the groups in same line.

**Table 4.** Mean values of FM (GPa)

Material	Room Temperature	T1(39°C)	T2 (55°C)
Z250	9.37 (0.28) <sup>Aa</sup>	10.70 (0.51) <sup>ABa</sup>	9.36 (0.27) <sup>Aa</sup>
EXF	9.09 (0.27) <sup>ACa</sup>	8.76 (0.45) <sup>ACa</sup>	8.62 (0.22) <sup>Aa</sup>
EXP	12.46 (0.17) <sup>Ba</sup>	12.95 (0.32) <sup>Ba</sup>	12.91 (0.15) <sup>Ba</sup>
ONE	7.71 (0.19) <sup>Ca</sup>	7.24 (0.34) <sup>Cab</sup>	6.19 (0.16) <sup>Cb</sup>
SDR	3.70 (0.06) <sup>Da</sup>	3.56 (0.15) <sup>Dab</sup>	2.87 (0.07) <sup>Db</sup>

\*The same uppercase letters mean there is no statistically significant difference between the groups in same column. The same lowercase letters means that there is no significant difference between the groups in same line.

at all temperature applications. However, EXP showed the highest average FM value for all temperature treatments. Preheating did not make a statistically significant difference to the FM of Z250, EXF and EXP. For ONE and SDR composites it was observed that preheating at 55°C significantly reduced the FM compared to room temperature application.

## Discussion

In the present study, the effect of preheating at different temperatures on the DC, FS and FM of four different bulk-fill resin composites, which were introduced to the market for use in high stress-bearing areas and to increase the fracture resistance of restorations, was investigated. The study revealed that there were significant differences between the tested composites in terms of their DC, FS and FM. In addition, the preheating process was found to have a significant effect on the DC, FS and FM values of some composites. Therefore, both of our null hypotheses were rejected.

In the current literature, while preheating of dental resins has been applied within a broad range of 30-69°C (28), concerns exist regarding the use of very high temperatures due to the potential for pulpal damage from temperature increases exceeding 5.5°C (29). However, experimental data suggests that these concerns may be mitigated in a clinically applicable context. For instance, it has been demonstrated that placing a composite resin preheated to 60°C increased pulp temperature by only 0.8°C, whereas light curing for 15 seconds caused a more significant increase of 4.5-5°C (30). Based on this literature and common practice in previous in-vitro studies, we therefore selected the preheating temperatures of 39°C and 55°C for this investigation.

The process of polymerisation has a significant effect on the mechanical and biological properties of resin composites (1). In addition to intrinsic factors such as co-monomer composition and ratio, filler content, photoinitiator type and concentration, there are also extrinsic factors such as light spectrum, irradiation protocols, temperature and light guide tip positioning that affect the DC of light-cured resin composites (31). DC can be obtained by direct (FTIR, FTIR-ATR and FTIR-Raman spectroscopy) and indirect methods (microhardness, depth of cure, differential scanning calorimetry, differential thermal calorimetry) (32). In the current study, the DC of the resin composites tested was evaluated by FTIR spectroscopy.

When the specimens prepared at room temperature were examined, the highest DC was observed in SDR, which has the lowest inorganic filler content. It was also observed that the DC decreased with increasing filler content

in the other composites except ONE. It is an expected result that increasing the filler content decreases the DC as it is accepted that increasing the amount of filler particles is an obstacle to polymer chain propagation (33). The exception of ONE to this rule is thought to be related to the AFM (addition-fragmentation monomer) in its organic content. These results obtained from our study are also similar to the literature (34).

Preheating of resin composites increases the system temperature, which reduces viscosity and improves molecular mobility, increases the collision frequency of reactive radicals, and delaying diffusion-controlled propagation, thereby improving final DC (20). As expected, in this study, preheating increased the DC of viscous composites with high filler content such as Z250 and EXP. However, it did not increase the DC of EXF, SDR and ONE, on the contrary, it caused a minimal decrease. Similar to our study, Lempel et al. (35) showed that preheating may have a positive effect on the DC in viscous composites, while it may have a negative effect in less viscous composites. The authors stated that this may be due to differences in the monomer content of the composites and a faster cooling process in composites with less filler. During cooling, the polymer formation has excessive heat loss. This deprives the system of the energy required for polymer chain propagation. The gel phase interval may be reduced, autoacceleration occurs, leading to premature vitrification and a reduction in the DC (36).

The FS and FM values of resin composites are considered to be critical indicators of the material's resistance to fracture under normal masticatory conditions (25,26,37). Although different tests can be used to measure these values, the three-point bending test is recommended as the gold standard. In this study, to measure the FS and FM of resin composites, specimens were prepared to the dimensions (25x2x2 mm) specified in ISO 4049/2009 (38) and three-point bending tests were performed. The same method has been preferred in many previous studies (13,39,40).

All composites tested in our study exhibited FS values above the 80 MPa threshold specified in ISO 4049/2009 for polymer-based restorative materials used in posterior restorations at all temperature applications (37). Among the bulk-fill composites, the highest FS values at room temperature were observed in the EXP and EXF groups containing short E-cam fibers. These composites showed significantly higher FS values than other bulk-fill composites (ONE and SDR) that did not contain fibers. These results are in parallel with similar studies in the literature (12,41). According to the authors, EXP and EXF allow stress to be transferred from the matrix to the fibers thanks

to the fibers they contain, thus providing effective reinforcement. In addition, these randomly oriented fibers also act as crack stoppers and strengthen the material (39). Therefore, it is possible that the high FS value exhibited by EXP and EXF may be related to the short E-glass fiber content.

Although EXF had similar FS values to EXP at room temperature, it had a significantly lower FM than EXP. This is interesting because it means that EXF, with a lower FM and similar FS, can be subjected to a higher load before failure. However, it was also found that lower FM creates less stress during polymerisation (41,42). In addition, EXF has the advantage of being easier to manipulate and place, especially in the posterior region of the oral cavity, due to its low viscosity. The same situation was observed between the ONE and SDR groups. Although SDR, with its lower viscosity, had similar FS values, it had a significantly lower FM than ONE.

Many studies have been carried out to investigate the effect of preheating on the physical and mechanical properties of resin composites. However, the results of these studies are not always consistent (28). The success of the technique depends on many variables such as material formulation, type of organic matrix, inorganic fillers, heating time and temperature, and light activation technique (43). Therefore, there is a lack of evidence regarding the effect of preheating on the physical and mechanical properties of resin composites. However, there is no study in the literature that investigates the effect of preheating on the flexural properties of short fiber-reinforced resin composites such as EXP and EXF. Therefore, in the present study, the effect of preheating at two different temperatures (39°C and 55°C) on the FS and FM values of 5 different resin composites, including short fiber-reinforced resin composites, was investigated.

Among the tested resin composites, the groups of Z250 and EXP with 39°C preheating treatment showed significantly higher FS values than those applied at room temperature. Although the FS value of the 55°C preheated groups of the same composites was higher than those applied at room temperature, this difference was not significant. These results are similar to the studies reporting that preheating increases the FS values of Z250 (44,45). In the present study, preheating increased the FS of Z250 and EXP, but had no effect on FM. Therefore, it can be said that preheating has a positive effect on the flexural properties of these composites.

In contrast to the Z250 and EXP groups, the EXF, ONE and SDR groups did not show any significant difference in the FS after preheating at the both temperatures. However, ONE and SDR showed significantly lower FM after

preheating at 55°C. Similar studies have reported that preheating has a positive effect on the FS of some composites, while making no significant difference to others (46,47). This discrepancy may be due to variations in resin composite formulations, such as monomer composition and filler content.

Although a strength of this study was that it evaluated popular bulk resin composites on the market, one of its major limitations was that only certain resin composites were tested. There are many resin composites on the market with different chemical contents and mechanical properties. Therefore, the results of the study cannot be generalized to all resin composites. Another limitation of the study is that the standard specimens prepared according to ISO standards in this study cannot exactly simulate the clinical scenario. Therefore, it is recommended that this in vitro study be supported by clinical studies.

## Conclusion

This in vitro study demonstrates that the clinical benefit of preheating is highly material-dependent. Preheating is recommended for viscous, fiber-reinforced (EverX Posterior) and conventional hybrid (Filtek Z250) composites to enhance their mechanical properties in stress-bearing restorations. In contrast, preheating provided no significant mechanical advantage for the flowable bulk-fill composites tested (EverX Flow Bulk, SDR Plus Flow, Filtek One Bulk Fill). For clinical practice, 39°C emerges as the prudent temperature choice, balancing efficacy with a safer physiological profile compared to higher temperatures.

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