

In-Vitro Evaluation of effect of Pre-Heating on Degree of Conversion and Surface Micro-Hardness of Bulk-Fill Composite at Different Depths

Pre-Heating on Degree of Conversion and Surface Micro-Hardness of Bulk-Fill Composite

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ABSTRACT

Objective: This in-vitro study objective is to evaluate whether preheating improves degree of conversion and surface micro-hardness of the Filtek bulk-fill posterior composite.

Study Design: An in-vitro experimental laboratory study

Place and Duration of Study: This study was conducted at the Conservative Dental Sciences Department, College of Dentistry, Qassim University, KSA from 20 May 2026 to 30 June 2026.

Methods: This study was conducted to evaluate the effect of preheating temperature on the degree of conversion and Vickers microhardness of Filtek™ Bulk Fill Composite. Sixty specimens were prepared in 5mm diameter, 4mm depth molds, and assigned to 23°C, 37°C, or 54°C groups (n=20 each). Each group was divided for ATR-FTIR degree of conversion and Vicker's micro-hardness testing. Specimens were stored in distilled water at 37°C for 24 hours before analysis. The composite was cured for 30 seconds using LED light at 1200mW/cm². **Results:** Preheating significantly increased degree of conversion and micro-hardness with 54°C producing the highest values and 23°C the lowest. In all groups, the top surface showed higher values than the bottom surface, and the bottom to top hardness ratios remained clinically acceptable. Degree of conversion (DC) strongly and positively correlates with micro-hardness.

Conclusion: Preheating the tested bulk fill composite improved polymerization efficiency and surface hardness, particularly at 54°C, and may enhance performance in deeper restorations. The Data also showed a clear depth effect; top surface values exceeded bottom surface values across temperatures, suggesting that preheating improved but did not eliminate light attenuation through the 4mm increment. Because the correlation between conversion and hardness was strong, micro-hardness may serve as a practical indirect marker of polymerization in this material.

Clinical Relevance: Within the limits of this single material in-vitro study, preheating appears to be useful strategy for improving bulk fill composite polymerization especially when deeper restorations are planned with clinically relevant chairside implications for practice.

Key Words: Composite resins, Dental materials, Temperature, Micro-hardness, Spectroscopy, Fourier transform infrared

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INTRODUCTION

Resin-based composites (RBCs) have become the material of choice for direct posterior restorations due to their favorable esthetics and adhesive capabilities. ⁽¹⁾

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However, conventional incremental placement is technique-sensitive, causes polymerization shrinkage, and prolongs clinical time. ⁽²⁾ Many of these issues have been addressed by bulk-fill composites, allowing up to 4 mm placement with adequate depth of cure and reduced shrinkage stress. These materials are specifically engineered with modified monomer systems, increased translucency, and advanced photo initiators to enhance light penetration and polymerization efficiency. ⁽³⁾

One important factor influencing resin composites' mechanical, biological, and physical performance is the degree of conversion (DC), which is the percentage of monomer converted to polymer. Increased clinical lifespan, decreased solubility, and greater mechanical strength are all strongly correlated with higher DC. ⁽⁴⁾ Crucially, DC and microhardness are depth-dependent characteristics that frequently decrease from top surface

to deeper layers as a result of curing light attenuation, particularly in bulk-fill restorations.⁽⁵⁾

Preheating composite resins has gained attention as an adjunctive technique to optimize polymerization, reducing viscosity of the composite, enhances flowability, and improves adaptation to cavity walls. More importantly, it increases molecular mobility, facilitating greater monomer conversion during polymerization. Several recent in-vitro studies have demonstrated that preheating can enhance DC and, in some cases, improve micro-hardness.⁽⁶⁾ However, the effect appears to be material-dependent, with some studies reporting no significant changes in microhardness despite improvements in other mechanical properties.⁽⁷⁾⁽⁸⁾⁽⁹⁾

Despite growing interest, the literature presents conflicting evidence regarding the influence of preheating on bulk-fill composites, particularly when evaluated at different depths.⁽¹⁰⁾ Studies report that preheating may enhance polymerization and deep-layer hardness, but effects vary with composite formulation and testing conditions. Furthermore, existing studies either focus on surface properties alone or do not comprehensively correlate DC with microhardness across multiple depths within the same experimental design.^(11, 12)

Given the growing use of bulk-fill composites, the effect of preheating on degree of conversion and surface micro-hardness at different depths remains unclear. This gap is clinically relevant because inadequate polymerization in deeper layers may compromise restoration durability, leading to secondary caries, marginal breakdown, and restoration failure.

Thus, the purpose of this in-vitro experimental laboratory study was to assess how preheating affects bulk-fill composite resins' degree of conversion and surface micro-hardness at various depths. By correlating these two fundamental properties under controlled conditions, the study aims to provide better insights into whether preheating can be recommended as a reliable clinical strategy to enhance the performance and longevity of bulk-fill composite restorations.

METHODS

This study was conducted at the Conservative Dental Sciences Department, College of Dentistry, Qassim University, over a period of approximately two months, from 20th May to 30th JUNE 2026, following ethical approval from the Institutional Review Board (IRB) with approval number #26-21-4. The specimens were prepared using cylindrical silicon molds having a diameter of 5 mm and depth of 4 mm respectively. The bulk-fill composite was placed in a single increment (4 mm thickness) and cured for 30 seconds using LED

blue phase N LED light. (Ivoclar Vivadent, Schaan, Liechtenstein, 1200 mW/cm² light intensity). Three experimental groups (n=20) were assigned a total of 60 standardized specimens according to the preheating temperature. Group I was considered as control, with 23°C and no preheating. Group II was preheated to 37 °C (mild heating) and group III was preheated to 54°C (High preheating) using Calset Composite Warmer (Ad Dent Inc., Danbury, CT, USA). For Vicker micro-hardness testing and degree of conversion analysis, each group was further split into two subgroups (n=10). Before testing, each specimen was kept in distilled water at 37°C for the whole day.

An attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectrometer (IRPrestige-21, Shimadzu Corporation, Kyoto, Japan) was used to measure the degree of conversion. Comparing uncured and cured composite spectra. (Fig 1C) Absorbance peaks at 1638 cm⁻¹ (aliphatic C=C) and 1608 cm⁻¹ (aromatic C=C) were analyzed, and DC (%) was calculated using peak ratio changes before and after curing. Each specimen's top and bottom surfaces were measured.

Surface micro-hardness was assessed using Vickers microhardness tester (Shimadzu Corporation, Kyoto, Japan) under standardized loading conditions. Three indentations were made on both the top and bottom surfaces of each specimen, and the mean was calculated. The efficacy of polymerization at depth was then evaluated by calculating the bottom to top ratio.

Tuckey's post hoc test and one-way analysis of variance were used to analyze the data, with $p < 0.05$ was set as statistically significant.

RESULTS

All specimens' preparations and analysis using FTIR, followed the approval protocol, and no specimen was excluded from the analysis. (Fig 1A-C).

The mean degree of conversion values obtained from the FTIR analysis and Tukey's HSD Post-hoc Comparisons are presented in Table 1. Composite preheating significantly affected the degree of conversion ($p < 0.05$). The lowest degree of conversion was observed at 23°C, reflecting higher composite viscosity and reduced monomer mobility. For all groups, the top surface demonstrated significantly higher DC values than the corresponding bottom surface. Tukey's post hoc analysis revealed that specimens preheated to 54°C exhibited significantly high degree of conversion than those preheated 37°C and 23°C. Likewise, specimens at 37°C exhibit significantly higher degree of conversion than those at 23°C. All pair wise comparisons demonstrated statistically significant differences ($p < 0.001$).

Table No.1: Degree of Conversion (DC%) at the Top and Bottom Surfaces and Tukey's HSD Post-hoc Comparisons.

Temp (°C)	Top Surface *DC% (Mean ***SD) ±	Bottom Surface DC% (Mean ± SD)	Top–Bottom Diff	Pairwise Comparison	Top DC Mean Difference (95% ** CI)	Bottom DC Mean Difference (95% CI)	p-value
23°C	69.2 ± 2.5	60.6 ± 2.7	8.6	23°C vs 37°C	5.9 (3.6–8.2)	7.7 (5.3–10.1)	<0.001
37°C	75.1 ± 2.3	68.3 ± 2.4	6.8	23°C vs 54°C	12.1 (9.8–14.4)	16.7 (14.3–19.1)	<0.001
54°C	81.3 ± 2.1	77.3 ± 2.2	4.0	37°C vs 54°C	6.2 (3.9–8.5)	9.0 (6.6–11.4)	<0.001

*DC = Degree of Conversion, **CI = confidence Interval, ***SD = Standard deviation



Figure No. 1: Composite resin specimen preparation and degree of conversion (DC%) assessment. (1A) Customized silicone molds (5×4 mm); (1B) polymerized bulk-fill composite specimen removed from the mold; and (1C) FTIR-ATR analysis for DC% determination.

Table No.2: Vickers micro-hardness (VHN) and bottom to top hardness ratio.

Group	Top VHN	Bottom VHN	Bottom/Top VHN Ratio (%)
23 °C	58.4	46.8	80.1%
37 °C	63.7	54.2	85.1%
54 °C	69.9	63.8	91.3%

Table No.3: Correlation between degree of conversion and Vickers micro-hardness.

Variables	Correlation coefficient (r)	p-value
Top DC vs Top VHN	0.982	< 0.001
Bottom DC vs Bottom VHN	0.989	< 0.001

Surface micro-hardness results are summarized in Table 2. Preheating significantly influenced Vicker hardness values ($p < 0.05$). The group with the highest preheating temperature had the highest hardness ratings, whereas the room temperature group demonstrated the lowest values. Within each experimental group, the top surface exhibited significantly greater Vicker hardness than the bottom surface. The calculated bottom to top hardness ratio remained within the acceptable range for all groups.

The degree of conversion and Vickers micro-hardness showed a statistically significant positive link, according to Pearson's correlation analysis (Table 3).

Specimens with higher degrees of conversion generally exhibited greater surface micro-hardness, indicating a positive association between polymerization efficiency and mechanical performance.

DISCUSSION

Thermal energy reduces composite viscosity, increases segmental molecular mobility of the monomer network, and lowers the activation energy barrier for the free radical polymerization collectively enabling higher conversion rates of aliphatic C=C bonds during photocuring.

The observation that the 54°C group achieved significantly higher DC than both the 37°C and 23°C groups are consistent with several prior investigations. Keles and Tarim,⁽¹³⁾ studying Tetric Evo Ceram Bulk fill and Sonic fill2 preheated to 55°C reported enhanced DC and micro-hardness relative to room temperature controls, with the effects most pronounced at the bottom surface, a finding that parallels depth-dependent results. Similarly, Singha et al⁽¹⁴⁾ testing Filtek Bulk fill among other materials preheated to 68°C, documented a statistically significant increase in Vickers micro-hardness from 60 to 70HV, confirming the favorable correlation shown in this investigation between preheating temperature and mechanical characteristics observed in the present research. Prasanna et al,⁽¹⁵⁾ demonstrated using ATR-FTIR spectroscopy, that DC increased monotonically as preheating temperature was elevated, providing foundational thermodynamic

support for the temperature-response relationship, this study reported.

As photons scatter and are absorbed by the resin matrix and filler particles, irradiance diminishes with depth, reducing the number of photo-initiator radical events available to propagate the polymerization front. Preheating partially compensates for these phenomena, explaining why bottom surface DC improvement was proportionality greater in the preheated groups than at the top surface, a pattern also reported by Theobaldo et al, ⁽¹⁶⁾ for 4mm increment bulk fill specimens cured with both LED and QTH units.

The significant positive Pearson correlation between DC and Vickers micro-hardness cross experimental conditions supports the long-standing mechanistic proposition that surface hardness is a valid indirect indicator of polymerization efficiency in resin-based composites. As Prasanna et al ⁽¹⁵⁾ demonstrated across three nanohybrid composites, microhardness was systematically improved at higher preheating temperatures.

The bottom to top hardness ratio, which remained within clinically acceptable limits (≥ 80) in all groups in the present study, provides additional assurance of adequate depth of cure of 4mm increment used. Suhetia et al, ⁽⁸⁾ reported bottom to top ratios between 0.62 and 0.71 for several bulk-fill composites including Filtek Bulk-fill, values somewhat lower than those observed here. Not all studies report a uniformly positive effect of pre-heating on surface micro-hardness, and the divergence in the literature merits contextualization. Kahnamouei et al ⁽¹⁷⁾ evaluated X-tra fil and Opus Bulk-fill preheated to 68°C for 15 minutes, found no statistically significant difference in VH between preheated and non-preheated groups. ($p=0.719$), despite a significant increase in flexural strength. Perez et al ⁽¹⁸⁾ reported that in-fact preheating increased micro-hardness in Filtek One Bulk-fill and Aura bulk-fill but decreased it in Opus bulk-fill, underscoring the material dependence of the preheating response. Not all studies report a uniformly positive effect of pre-heating on surface micro-hardness, and the divergence in the literature merits contextualization. Kahnamouei et al ⁽¹⁷⁾ evaluating X-tra fil and Opus Bulk fill preheated to 68°C for 15 minutes, found no statistically significant difference in VH between preheated and non-preheated groups. ($p=0.719$), despite a significant increase in flexural strength.

From the clinical perspective, the temperatures investigated here 37°C and 54°C represent a clinically feasible range. Temperatures above 60°C risk premature polymerization during storage and potential pulpal irritation upon placement and have been associated in isolated reports with accelerated photo initiator degradation. Patussi et al ⁽¹⁹⁾ in a scoping review covering the breath of preheating research concluded that evidence favors preheating as a

technique to enhance polymerization efficiency and handling properties, while noting the heterogeneity that limits definitive clinical recommendations.

Limitation of the study

Several limitations of the present study should be acknowledged when interpreting these findings.

1. The use of single composite brand and a single curing protocol restrict the external validity of the results; performance across bulk fill formulations with varying filler contents monomer systems, and photo initiator packages cannot be inferred from the present data.
2. The specimen geometry (5mm diameter, 4mm depth) models are a simplified, idealized clinical increment without the geometric complexity, cavity walls or adhesive interface present in vivo.
3. No measures of polymerization shrinkage stress were obtained; which may partially off-set mechanical gains documented here.
4. Long term water sorption and the solubility assessments which degrade mechanical properties per se, were also beyond the scope of this investigation.

CONCLUSION

Preheating the bulk fill composite improved both degree of conversion and surface microhardness, with the best performance at 54°C. The top surface showed greater hardness than the bottom, while acceptable hardness ratios indicated adequate depth of cure and polymerization quality.

Authorship: The sole author designed the study, conducted and analyzed the research, interpreted the results, prepared the manuscript, and approved the final version, accepting full responsibility for its integrity.

Clinical Relevance

Scientific rationale of the study: Bulk fill composite exhibits reduced polymerization at greater depths, potentially compromising their mechanical properties and clinical longevity. Pre-heating has been proposed to enhance monomer conversion and material performance.

Principal findings: Pre-heating significantly improved the degree of conversion and surface micro-hardness at both top and bottom surfaces, with higher temperatures producing greater enhancements while maintaining adequate curing throughout the restoration.

Practical Implications: Pre-heating bulk-fill composite before placement may improve polymerization efficiency, optimize mechanical properties, and enhance the durability of deep posterior restorations, supporting its incorporation into routine restorative clinical practice where feasible.

Author's Contribution:

Concept & Design or acquisition of analysis or	Shahzad Ali Shah
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interpretation of data:	
Drafting or Revising Critically:	Shahzad Ali Shah
Final Approval of version:	The above author
Agreement to accountable for all aspects of work:	The above author

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