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# The effect of resin composite pre-heating on monomer conversion and polymerization shrinkage

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ABSTRACT

*Objectives.* To determine monomer conversion and polymerization shrinkage of a resin composite after different pre-heating procedures and storage intervals.

*Methods.* For a commercial resin-based composite the immediate (5 min) and final (24 h) degree of conversion was measured on top and bottom surfaces utilizing FTIR spectroscopy. Composite pre-heating temperatures were selected between 10 and 68 °C. Polymerization shrinkage was measured according to Archimedes' principles of buoyancy after 5 min at respective pre-heating temperatures and after 24 h dark and wet storage at 37 °C. Intra-cavity temperature development was monitored using a K-type thermocouple.

*Results.* No significant increase in immediate as well as in final degree of conversion were measured from composite pre-heating at 68 °C compared to 54 and 39 °C. Linear correlations were detected immediately after photo-polymerization and on the top surface after 24 h storage. Polymerization shrinkage as a function of pre-heating temperatures exhibited a linear correlation after 5 min, but no statistically different behavior after 24 h.

*Significance.* Pre-heating of resin composites does not increase degree of conversion over time. It can be clinically beneficial, due to a superior marginal adaptation. This advantageous effect of reduced material paste viscosity has to be clinically addressed, since temperature rapidly drops to the physiological level upon removal from the pre-heating device.

## 1. Introduction

Directly placed composite resins serve as standard materials in restorative and esthetic dentistry. Their chemistry and properties have been extensively investigated in the relevant dental literature [1,2]. The clinical success of a composite restoration is closely related to the material characteristics like polymerization shrinkage, degree of conversion and mechanical properties [3–5]. Handling characteristics like paste viscosity, packability, stickiness and polishability play a critical role, as well [6]. To receive a perfectly sealed, long-lasting restoration, material adaptation to the cavity walls is of importance [7].

Recently, pre-heating resin composites with appropriate devices have been advocated as a method to reduce paste viscosity, improve marginal adaptation and monomer conversion, and to shorten curing times [8]. Elevated temperatures have been shown to strongly influence composite conversion and mechanical properties, but on the other side to increase the possibility of pulp necrosis [9,10]. Light-curing of methacrylate monomers results in a highly crosslinked structure, with residual unsaturation in the form of pen-

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dant methacrylate groups, due to steric hindrance [3,11]. The degree of conversion, defined as the percentage of reacted C═C bonds, affects several parameters including mechanical properties, solubility, dimensional stability, color change and biocompatibility of resin composites [4,5,12]. In diffusion controlled resin networks, mobility of monomers and polymerization reactivity is increased at elevated temperatures; moreover, under these conditions, the autodeceleration stage of the polymerization reaction can be delayed. Both these contribute to increased monomer conversion [9,13,14]. Indeed, significantly increased monomer conversion after external composite heating at 54 °C compared to room temperature has been reported [15]. Nevertheless, it has been observed, that the higher the degree of conversion in resin composites, the higher is the polymerization shrinkage [4]. In addition, since resin composites exhibit a six to eight times greater thermal expansion than the surrounding tooth structures [16], polymerization contraction along with thermal contraction might create high interfacial stresses in pre-heated composites upon thermal equilibrium, with detrimental effects on marginal adaptation, integrity and seal [17,18].

Commercially available pre-heating devices are operating at a temperature range of 54–68 °C, which is questioned regarding pulp compatibility in deep cavities. Nevertheless, only a 0.8 °C temperature increase was found after placement of a 60 °C heated composite, but a 5 °C increase upon 20 s light-curing [10].

The aim of the present study was to assess the monomer conversion, polymerization shrinkage and intracavity temperature profiles of a resin composite after different pre-heating procedures and storage intervals. The null-hypothesis tested was that pre-heating of a resin composite results in improved immediate and 24 h monomer conversion over time, without affecting polymerization shrinkage.

## 2. Materials and methods

### 2.1. Material

The visible light-curing dental restorative Tetric® EvoCeram (Lot: H35663, A3 shade, Ivoclar, Vivadent, Schaan, Liechtenstein) was used in this study. The material is based on a bisphenol glycidyl dimethacrylate (BisGMA)/urethane dimethacrylate (UDMA)/triethylene glycol dimethacrylate (TEGDMA) resin matrix, with camphoroquinone as photoinitiator and 82.5 mass% filler content (48.5% inorganic and 34% pre-polymerized fillers). The material was used in syringes and stored at the respective pre-heating temperatures for 30 min. Light-curing was performed using a quartz tungsten halogen unit (Elipar® TriLight, 3 M/ESPE, Seefeld, Germany, 800 mW/cm$^2$ emitted light intensity) operated in standard mode.

### 2.2. Dentin temperature profile

Human dentin discs with a thickness of 2 mm were prepared from the coronal sections of freshly extracted human molars. A cylindrical cavity was drilled into the top disc while a K-type thermocouple was embedded in the bottom disc. The discs were frictionally laminated according to Fig. 1 and stored at 37 °C in 100% relative humidity for a minimum of 30 min. The resin composite specimens were temperature controlled at 10 ± 2, 23 ± 2, 39 ± 2, and 68 ± 2 °C employing a refrigerator or a dry-heat oven and immediately placed in the dentin cavity at 37 °C. After a 60 s period, to simulate intraoral handling, the fillings were light-cured for 20 s. Temperature was measured at the bottom of the cavity or at a 2 mm distance (an additional specimen series for the 68 °C pre-heated group) by inserting a third sandwich dentin disc. A software controlled temperature datalogger (Voltcraft, Hirschau, Germany) with an accuracy of ±0.3% served to monitor dentin temperature. Mean temperature profiles were calculated from three measurements of each composite temperature group.

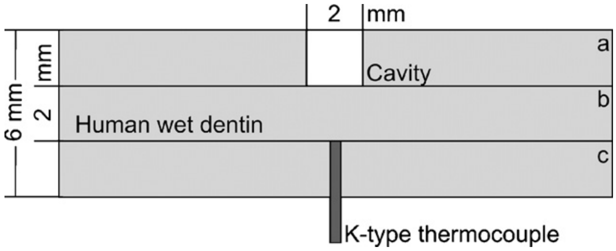

**Fig. 1 – Temperature measurement principles.**

### 2.3. Degree of conversion

Materials and molds were stored and polymerized at temperatures of 10 ± 2, 23 ± 2, 39 ± 2, 54 ± 2 and 68 ± 2 °C. Rectangular composite specimens (6 mm × 4 mm × 2 mm, $n = 5$) were produced by light-curing from only one direction in order to prepare specimens for surface and in-depth measurements. Specimens were subsequently transferred to a dry-heat oven and stored at 37 °C for 5 min or 24 h prior to DC measurements.

Degree of conversion of the specimens was measured by Fourier transform infrared micromultiple internal reflectance spectroscopy (FTIR). An FTIR spectrometer (Spectrum GX, Perkin-Elmer, Beaconsfield, Bacon, UK) was used, equipped with a micromultiple internal reflectance cell operated under the following conditions: 4000–400 cm$^{-1}$ range, 4 cm$^{-1}$ resolution, 50 scans coaddition, 45° para KRS-5 minicrystal (10 mm × 5 mm × 1 mm) of seven internal reflections. Spectra were acquired from top and bottom surfaces. The degree of conversion (% DC) on the tested surfaces was calculated by the two frequency technique using the net peak absorbance areas of the aliphatic C═C stretching vibrations at 1638 cm$^{-1}$ as analytical frequency and the aromatic C···C stretching vibrations at 1608 cm$^{-1}$ as reference frequency according to the equation:

$$\%\,\mathrm{DC} = \left(1 - \left(\frac{A_{\mathrm{M}}(\mathrm{C}\cdots\mathrm{C}) \cdot A_{\mathrm{P}}(\mathrm{C}=\mathrm{C})}{A_{\mathrm{M}}(\mathrm{C}=\mathrm{C}) \cdot A_{\mathrm{P}}(\mathrm{C}\cdots\mathrm{C})}\right)\right) \times 100 \qquad (1)$$

where $A_{\mathrm{M}}$ and $A_{\mathrm{P}}$ represent the net peak absorbance height ratios of the uncured and cured material, respectively.

Statistical analysis was performed employing three-way ANOVA to assess interactions between the independent factors site (top/bottom), storage time (5 min/24 h), and pre-heating temperatures on the % DC. Student–Newman–Keuls