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## Effect of temperature on composite polymerization stress and degree of conversion

Fernanda C. Calheiros<sup>a</sup>, Márcia Daronch<sup>b</sup>, Frederick A. Rueggeberg<sup>c</sup>, Roberto R. Braga<sup>d,\*</sup>

<sup>a</sup> University of Ibirapuera, São Paulo, Brazil

<sup>b</sup> Department of Pediatric Dentistry, NY University, USA

<sup>c</sup> Dental Materials Section, Department of Oral Rehabilitation, Georgia Regents University, Augusta, GA, USA

<sup>d</sup> Department of Biomaterials and Oral Biology, School of Dentistry, University of São Paulo, São Paulo, SP, Brazil

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### ABSTRACT

**Objective.** To test the following hypotheses: (1) degree of conversion (DC) and polymerization stress (PS) increase with composite temperature (2) reduced light-exposure applied to pre-heated composites produces similar conversion as room temperature with decreased PS.

**Methods.** Composite specimens (diameter: 5 mm, height: 2 mm) were tested isothermally at 22 °C (control), 40 °C, and 60 °C using light-exposures of 5 or 20 s (control). DC was accessed 5 min after light initiation by FTIR at the specimen bottom surface. Maximum and final PS were determined, also isothermally, for 5 min on a universal testing machine. Non-isothermal stress was also measured with composite maintained at 22 °C or 60 °C, and irradiated for 20 s at 30 °C. Data were analyzed using two-way ANOVA/Tukey and Student's t-test ( $\alpha = 5\%$ ).

**Results.** Both DC and isothermal maximum stress increased with temperature ( $p < 0.001$ ) and exposure duration ( $p < 0.001$ ). Isothermal maximum/final stress (MPa) were  $3.4 \pm 2.0b/3.4 \pm 2.0a$  (22 °C),  $3.7 \pm 1.5b/3.6 \pm 1.4A$  (40 °C) and  $5.1 \pm 2.0a/4.0 \pm 1.6A$  (60 °C). Conversion values (%) were  $39.2 \pm 7.1c$  (22 °C),  $50.0 \pm 5.4b$  (40 °C) and  $58.5 \pm 5.7a$  (60 °C). The reduction of light exposure duration (from 20 s to 5 s) with pre-heated composite yielded the same or significantly higher conversion (%) than control (22 °C, 20 s/control:  $45.4 \pm 1.8b$ , 40 °C, 5 s s:  $45.1 \pm 0.5b$ , 60 °C, 5 s s:  $53.7 \pm 2.7a$ ,  $p < 0.01$ ). Non-Isothermal conditions showed significantly higher stress for 60 °C than 22 °C (in MPa, maximum:  $4.7 \pm 0.5$  and  $3.7 \pm 0.4$ , final:  $4.6 \pm 0.6$  and  $3.6 \pm 0.4$ , respectively).

**Clinical significance:** Increasing composite temperature allows for reduced exposure duration and lower polymerization stress (both maximum and final) while maintaining or increasing degree of conversion.

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\* Corresponding author at: Department of Biomaterials and Oral Biology, School of Dentistry, University of São Paulo. Av. Professor Lineu Prestes, 2227, Cidade Universitária, 05508-000, São Paulo, SP, Brazil. Tel.: +55 11 3091 7840.

E-mail address: [rbraga@usp.br](mailto:rbraga@usp.br) (R.R. Braga).

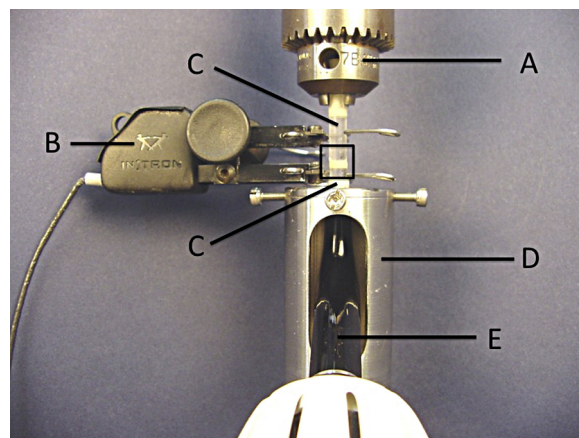
## 1. Introduction

Temperature plays an important role in the polymerization process of multifunctional monomers used in photo-activated, dimethacrylate-based dental restorative materials [1-5]. Increased polymerization temperature enhances radical and monomer mobility, resulting in higher conversion because of lowered system viscosity [3,6-9]. From a clinical standpoint, pre-heating was shown to greatly increase composite flow [10-13]. No pulpal damage is expected with pre-heating, as the difference in *in vitro* intrapulpal temperature was less than 1 °C when using composite pre-heated to 60 °C compared to composite tested at room temperature [14]. However, little information is available on the effect of preheated composites on polymerization stress.

Previous *in vitro* studies on isothermal polymerization indicated a significant increase in degree of conversion, as well as an increase in polymerization rate and conversion at maximum cure rate, when composite curing occurred above room temperature [4,5]. Moreover, pre-heated composite reached high conversion values using relatively low radiant exposures, allowing a reduction in exposure duration up 75% compared to composites polymerized at room temperature [4]. However, it has been shown that increased polymerization rate and conversion increase polymerization stress development [15,16]. Therefore, in theory, the use of pre-heated composites may result in increased stress, unless the reduction in viscosity due to heating would allow for increased viscous flow and chain relaxation, offsetting the effects of the higher conversion and reaction rate. Also, secondary transitions involving comparatively small changes in modulus as a result of temperature increase, such as side-group motions [17], could also contribute to stress relaxation.

Though information on isothermal polymerization stress is important, it does not necessarily represent what occurs clinically. With clinically available pre-heating devices, the temperature drop once composite is removed from the heating device is likely to result in lower conversion compared to that found when composite is cured isothermally at elevated temperatures [5,18]. *In vivo* tooth temperature during a restorative procedure is approximately 30 °C and a composite material originally pre-heated to 60 °C placed into a tooth preparation attained only 38 °C at the time of photo-activation [19]. Therefore, it is also important to evaluate the effect of pre-heating dental composites on polymerization stress development in a clinically realistic scenario, where composite temperature is elevated in the heating device and then lowers when in contact with the prepared tooth.

The purpose of the present study was to evaluate the influence of composite temperature and light-exposure duration on polymerization stress and degree of conversion of a commercial composite photocured either isothermally or non-isothermally, according to a clinically representative temperature scheme. Since conversion is influenced by kinetic parameters such as temperature and also radiant exposure, and polymerization stress is dependent on conversion, polymerization stress is expected to be affected by both temperature and exposure duration. The working hypotheses were (1) for a given exposure duration, increase in composite



**Fig. 1 – Polymerization stress test configuration. A: clamp connected to the load cell; B: Extensometer; C: Glass rods; D: Metallic fixture for light-curing end tip (E). Box: resin composite specimen. (Note: all items above were contained within a temperature-controlled chamber).**

temperature will increase polymerization stress and degree of conversion and (2) for a given composite temperature, reduction in exposure duration will decrease polymerization stress and degree of conversion values.

## 2. Materials and methods

### 2.1. Polymerization stress testing

One of the ends of two 5-mm diameter glass rods, one 13-mm and the other 28-mm in length, were ground flat with SiC paper and sandblasted with alumina (250 μm). The roughened surfaces were silanated (Dentsply Ind. e Com. Ltda., Rio de Janeiro, Brazil), coated with two layers of bonding agent (Scotchbond Multi-Purpose Plus, 3M ESPE, St. Paul, MN, USA) light-cured for 30 s. The glass rods were attached to opposite fixtures of a universal testing machine (model 5565, Instron, Canton, MA, USA) by their non-treated surfaces. The long rod was attached to the actuator, while the short rod was attached to a stainless steel fixture connected to the lower clamp of the machine. This fixture had a slot allowing contact of the light tip with the glass rod (Fig. 1). A nanohybrid composite (Esthet X, shade A2, lot#0302054, Dentsply/Caulk, Milford, DE, USA) was placed between the treated glass surfaces (height = 2 mm, C-factor = 1.25, volume = 39.3 mm<sup>3</sup>).

Relevant portions of the testing assembly were maintained inside a temperature-controlled chamber (model 3119-405, Instron). During isothermal testing, composite compules (unidose) were also kept inside the chamber prior to testing in order to stabilize the material's temperature. The chamber temperature was kept at 22 ± 2 °C (room temperature, control), 40 ± 2 °C, or 60 ± 2 °C. The latter value is similar to that used in a commercial heating device (Calset™, AdDent Inc. Danbury, CT, USA). For the non-isothermal conditions, the temperature-controlled chamber was set to 30 °C, while composite compules were kept either at room temperature

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