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Post-curing in dental resin-based composites

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ABSTRACT

Objective. To determine the post-curing in six commercial contemporary resin-based composites (RBCs) using axial shrinkage, the degree of conversion, and Vickers hardness.
Methods. Five Bulk Fill and one conventional RBCs from three companies were selected with a wide range of filler volume content. The axial shrinkage of samples that were 1.00 mm thick by 9–10 mm diameter was measured using a modified bonded disk method over a time between 15 h and 19 h at temperatures of 26 °C and 34 °C (mouth temperature). The degree of conversion (DC) was collected continuously for 10 min using mid-infrared spectroscopy in the attenuated total reflectance geometry. Vickers hardness was measured at 1 h post-irradiation using a load of 300 gf. For all three tests, the samples were irradiated at five exposure times, 20, 5, 3, 1.5 and 1 s with a light curing unit radiant exitance of 1.1 W/cm^2. Three samples (n = 3) were used for each experimental condition.
Results. After light exposure, the axial shrinkage and degree of conversion exhibited a functional time dependence that was proportional to the logarithm of time. This suggests an out-of-equilibrium polymer composite glass that is transitioning to thermal equilibrium. At a sufficiently long time and among the RBCs investigated, the shrinkage related physical aging rate was found to vary between 1.34 and 2.00 μm/log(t). The rate was a function of the filler content. Furthermore, 15 h after light exposure, the post-curing shrinkage was estimated to be an additional 22.5% relative to the shrinkage at 100 s for one RBC at T = 34 °C. The hardness in the photo-cured RBC was varied by using different light exposure times. The first two experimental techniques show that the higher the initial DC 10 min after light exposure, the smaller is the post-curing shrinkage related and DC related physical aging rates. A direct correlation was observed between the shrinkage related and the DC related physical aging rates.
Significance. Post-curing shrinkage should be evaluated for longer than 1 h. The post-curing shrinkage 15 h after light exposure in dental RBCs can be appreciable. The long-term development of built-in stress within the tooth wall structure may shorten the restoration's lifespan.

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1. Introduction

A paradigm shift in the field of dentistry occurred with the introduction of photo-cured resin-based composites (RBCs) as an alternative to amalgam in dental restorations [1–3]. The main driving force is that the restored teeth can look like a natural decay free tooth. Due to the fundamental nature of the resin used in RBCs, after photo-curing, polymerization-induced shrinkage strain develops with time. This produces shrinkage-induced stress within the cavity walls of the teeth [4]. There is evidence that such stresses may result in enamel cracking and tooth post-operative pain [5], margin debonding [6], secondary caries [7], and premature failure of the restoration [8]. Dental manufacturers have taken different approaches to relieve the stress developed both during and after photo-curing by altering the monomers [9,10], or by incorporating pre-polymerized filler particles [11] in RBCs.

After the light curing unit (LCU) is turned off, one important contribution to shrinkage has been attributed to the continued cross-linking as the unterminated radicals continue to react, albeit rather slowly. This process is sometimes referred as the densification of the cured RBC with time [12] because it leads to an increased RBC density and elastic modulus. Several studies have been carried out in cured RBCs to investigate how the degree of conversion [13–17], glass transition temperature [18], axial shrinkage [19], density and mechanical properties [20–22] vary with time for up to a month after light exposure. It has been reported that during the post-curing period, the elastic modulus [21], ion viscosity [21], and degree of conversion (DC) [23] were found to be proportional to the logarithm of time, but no explanation was given for this unique time dependence. Post-curing was investigated in experimental photo-cured resins using six experimental techniques [20]. An overall explanation of the results was offered using the free volume fraction model to explain the properties of out-of-equilibrium amorphous polymer glasses [24–27]. In this model, an out-of-equilibrium amorphous polymer glass can have an excess of free volume in the form of a large number of sub-nanometer-sized voids dispersed throughout the polymer glass. Due to the thermal diffusion of these excess voids out to the surface, the polymer continues to shrink with time. As indicated in Fig. 2 of Ref. [27], after a relatively short time, the decrease in sample thickness can be predicted to be proportional to the logarithm of time [log(t)]. The observation of the log(t) dependence for the volume shrinkage of an out-of-equilibrium RBC in the glass state provides a direct evidence for this model. In the above study [20], the sample axial shrinkage was measured continuously over a period of 10 h. The axial shrinkage varied in a non-monotonic manner with time and, as a result, was inconclusive toward testing the applicability of the free volume fraction model to photo-cured methacrylate-based resins. In another study [19], the axial shrinkage on RBCs was measured continuously over a period of 1 h; this time period was too short to observe the log(t) dependence.

The free volume fraction model has been studied extensively in the field of polymer glasses and composites [27–31]. The process for the out-of-equilibrium polymer glass to reach thermal equilibrium is commonly referred to as physical aging. In this work, the constant of proportionality between the glass parameter such as axial shrinkage (or DC) and the logarithm of time is called the axial shrinkage (or DC) related physical aging rate [28].

To optimize dental RBC blends and photo-curing conditions with the goal of minimizing post-curing shrinkage, it is vital to understand the underlying mechanisms governing post-curing. Furthermore, it is also important to quantify the post-curing shrinkage in RBCs at times longer than 1 h. In this study, the post-curing of six commercial contemporary RBCs was investigated using axial shrinkage and degree of conversion as a function of time for different light exposure times, and hardness measured one hour after light exposure for different light exposure times. The research hypotheses were:

(1) at a sufficiently long time, the axial shrinkage is not proportional to the logarithm of time,
(2) the axial shrinkage related physical aging rate, α_S, does not vary with the light exposure time,
(3) at a sufficiently long time, the degree of conversion is not proportional to the logarithm of time,
(4) the degree of conversion related physical aging rate, α_{DC}, does not vary with the light exposure time,
(5) α_S and α_{DC} do not vary with the degree of conversion,
(6) α_{DC} is not correlated with α_S, and
(7) α_S and α_{DC} are not correlated with the hardness.

2. Materials and methods

2.1. Materials

Six commercial contemporary RBCs were used in this study. They consisted of five Bulk Fill and one conventional dental RBCs. The RBC compositions as described by the manufacturers are reported in Table 1. Two low (≤45 vol.%) filler content (3M Filtek Bulk Fill Flowable Restorative A2 shade and DENTSPLY Surefil SDR flow Posterior Bulk Fill A3 shade), and four high (>58 vol.%) filler content (3M Filtek Bulk Fill Posterior Restorative A2 shade, Filtek One Bulk Fill Restorative A2 shade, and Z100 Restorative A2 shade, and Voco GmbH X-tra fil Universal shade) were used to study the effect of time and filler volume fraction on the shrinkage and degree of conversion. Temperatures of 26 °C (representing a warm room and the minimum after washing and drying a cavity) and 34 °C (representing a maximum intraoral temperature inside a cavity) [32,33].

2.2. Sample preparation and photo-curing conditions

For all three experimental techniques used, the sample geometry and sample dimensions, and photo-curing conditions were the same. The substrate was a 25.4 mm diameter by 3 mm thick quartz disk. One surface was lightly sandblasted and then silanized using a ceramic primer (RelyX Ceramic Primer, 3M, St Paul, MN, USA). A 1.00 mm thick by 9–10 mm diameter RBC sample was inserted at the center of the substrate. For the axial shrinkage measurements, a 100 μm thick glass coverslip disk was placed on top of the sample. The coverslip was supported along its edge by a 1.00 mm thick brass

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