# EFFECT OF INITIATOR AND INHIBITOR CONCENTRATION ON THE POLYMERIZATION SHRINKAGE KINETICS OF EXPERIMENTAL DENTAL COMPOSITES

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**Abstract**

*We prepared experimental dental composites with different curing rates by varying the concentration of photo-initiator (Camphorquinone) and inhibitor (Butylatedhydroxytoluene). During photo-activated curing, the polymerization shrinkage, maximum shrinkage rate and polymerization shrinkage stress of the composites were measured using a laboratory made strain-stress analyzer for 10 min. The polymerization shrinkage stress of the composites ranged 2.97 - 7.91 MPa, which increased with increasing initiator concentration. Increased inhibitor concentration reduced shrinkage stress and curing rate.*

## 1. Introduction

Dental composites are widely used to restore teeth owing to their superior esthetic properties. However, a major drawback of dental composite is polymerization shrinkage [1,2]. Polymerization shrinkage in a confined cavity generates polymerization shrinkage stress at the tooth-composite restoration interface. This would in turn lead to marginal fracture, de-bonding, microleakage, cuspal deflection, and enamel microcrack, resulting in the penetration of bacteria, secondary caries, pulpal inflammation, and post-operative hypersensitivity [3].

Numerous studies have been carried out in an attempt to reduce the polymerization shrinkage stress of dental composites. The polymerization shrinkage kinetics of light cured composites

can be influenced by photo-polymerization methods [4,5], composition and ratio of monomers [6], and type and concentration of initiator and catalyst [7,8].

At the optimum concentration of initiator and catalyst, the maximum degree of polymerization and mechanical properties can be attainable; however, the polymerization shrinkage stress would be maximized simultaneously. It has been reported that inhibitor concentration can be modified to reduce the polymerization shrinkage stress of experimental composites without compromising mechanical properties [9].

The purpose of this study was to investigate the effect of the concentration of initiator and inhibitor on the polymerization shrinkage kinetics such as polymerization shrinkage, polymerization rate and polymerization shrinkage stress of experimental dental composites.

## 2. Materials and Methods

### 2.1. Preparation of experimental composites

Bis-GMA (2,2-bis-[4-(methacryloxy-2-hydroxy-propoxy)-phenyl]-propane, Aldrichi, Steinheim, Germany) and TEGDMA (Triethylene glycol dimethacrylate, Aldrichi) resin monomers were mixed at the ratio of 6:4. Photo-initiator (Camphorquinone, Aldrichi) and catalyst (Ethyl 4-dimethylaminobenzoate, Aldrichi) were added to the resin matrix at concentrations of 0.1, 0.5, 1.0 and 2.0 wt%. An inhibitor (Butylatedhydroxytoluene, BHT, Aldrichi) was varied at concentrations of 0.05, 0.1, 0.5 and 1.0 wt%. The formulated resin matrices were filled 80% by weight with 0.7 μm Ba-glass filler (Schott, Mainz, Germany) to prepare 7 experimental dental composites (Table 1).

| Composite | Initiator (wt%) | Catalyst (wt%) | Inhibitor (wt%) |
|-----------|-----------------|----------------|-----------------|
| Exp1 | 0.1 | 0.1 | 0.1 |
| Exp2 | 0.5 | 0.5 | 0.05 |
| Exp3 | 0.5 | 0.5 | 0.1 |
| Exp4 | 0.5 | 0.5 | 0.5 |
| Exp5 | 0.5 | 0.5 | 1.0 |
| Exp6 | 1.0 | 1.0 | 0.1 |
| Exp7 | 2.0 | 2.0 | 0.1 |

**Table 1.** The concentration of initiator, catalyst and inhibitor in 7 experimental composites.

*2.2. Measurement of polymerization shrinkage*

The axial polymerization shrinkage was determined using an LVDT (linear variable differential transformer) displacement sensor (Fig.1a). A certain amount of experimental composite was placed between a slide glass and a cover glass (Fig.1b). The thickness of the sandwiched specimen was 0.5 mm. The specimen was photo-cured with a curing light (VIP Junior, 470 nm, 600 mW/cm$^2$, Bisco, Inc., IL, USA) for 40 s. The axial polymerization shrinkage was recorded for 10 min. After completion of polymerization, the cured specimen thickness was measured using a micrometer. The polymerization shrinkage% was given by $\Delta h/(\Delta h+h) \times 100$, where h is the specimen thickness after curing, $\Delta h$ is the axial shrinkage measured by the LVDT. The maximum shrinkage rate and peak shrinkage rate time were determined from the polymerization shrinkage per unit time ($d$shrinkage%/$d$t).

*2.3. Measurement of polymerization shrinkage stress*

An instrument was made to measure the polymerization shrinkage stress of composites during photo-polymerization (Fig.1c). The instrument composed of a voice coil motor (MGV52-20-0.5, Akribis systems, Singapore), a linear encoder and a servo amplifier with feedback mechanism. When the curing light was turned on, the polymerization shrinkage of composite was detected by the linear encoder via a slide glass, which in turn generates feedback current to the voice coil actuator by the servo-amp to maintain the initial specimen length. The feedback current is proportional to the shrinkage stress. Polymerization shrinkage stress was recorded for 10 min. In the experiments 2.2 – 2.3, the measurements were repeated 5 times for each composite. The data were analyzed with one-way ANOVA at the significant level of 0.05% followed by Duncan post-hoc test.
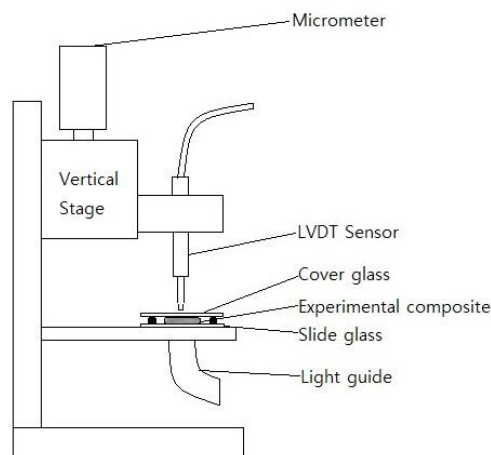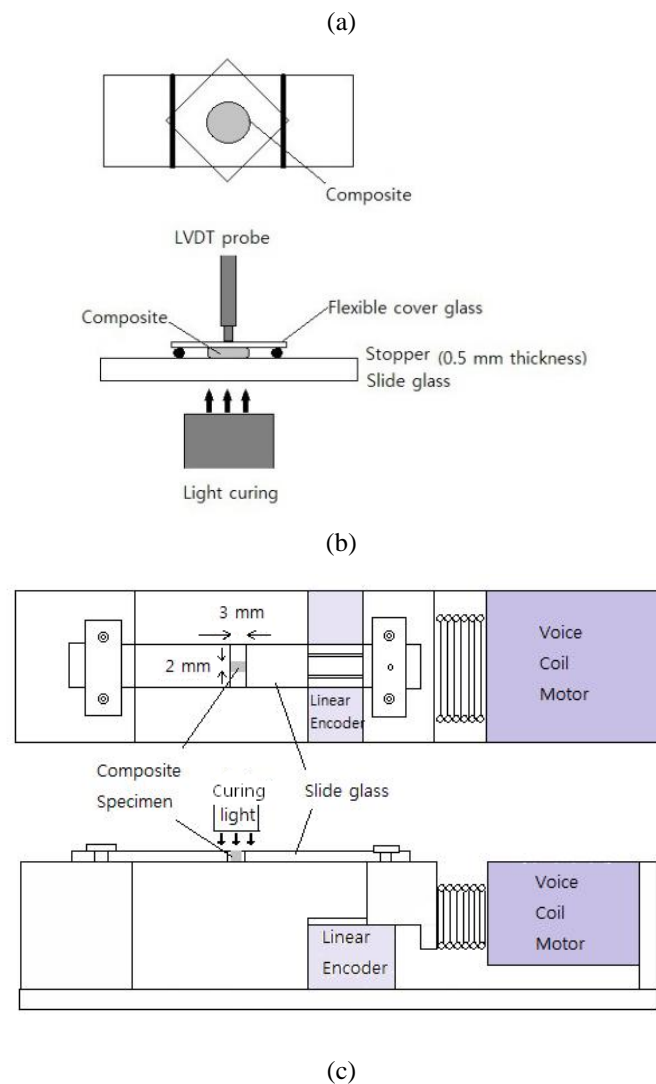
**Figure 1.** (a) The structure of an instrument for measuring axial shrinkage. (b) Specimen preparation and experimental set-up for the measurement of axial shrinkage using an LVDT probe. (c) The instrument for measuring polymerization shrinkage stress using a voice coil motor with feedback mechanism.

## 3. Results

Polymerization shrinkage (%), maximum shrinkage rate (%/s) and peak shrinkage rate time (s) with varying concentrations of initiator and inhibitor are presented in Table 2. At a fixed inhibitor concentration of 0.1 wt%, polymerization shrinkage increased with increasing initiator concentration. When the initiator concentration was set at 0.5 wt%, there was no statistically significant difference in polymerization shrinkage with increasing inhibitor concentration. The maximum rate of shrinkage increased with increasing initiator concentration, while being decreased with increasing inhibitor concentration. The peak shrinkage rate time decreased with increasing initiator concentration, while being delayed

with increasing inhibitor concentration. Polymerization shrinkage stress increased with increasing initiator concentration; however, it decreased with increasing inhibitor concentration.

| Composite | Shrinkage (%) | Maximum Shrinkage Rate (%/s) | Peak Time (s) | Shrinkage Stress (MPa) |
|---|---|---|---|---|
| **Initiator Conc. (Inhibitor: 0.1%)** | | | | |
| 0.1% (Exp1) | 2.65 (0.03) [a] | 0.21 (0.01) [a] | 2.84 (0.45) [c] | 2.97 (0.01) [a] |
| 0.5% (Exp3) | 3.17 (0.07) [b] | 0.65 (0.07) [b] | 2.17 (0.14) [b] | 6.05 (0.29) [b] |
| 1.0% (Exp6) | 3.33 (0.03) [b] | 0.80 (0.05) [c] | 1.67 (0.06) [a] | 7.08 (0.26) [c] |
| 2.0% (Exp7) | 3.59 (0.18) [c] | 0.87 (0.06) [c] | 1.65 (0.06) [a] | 7.91 (0.35) [d] |
| **Inhibitor Conc. (Initiator: 0.5%)** | | | | |
| 0.05% (Exp2) | 3.12 (0.09) [A] | 0.65 (0.03) [C] | 2.03 (0.16) [A] | 6.58 (0.36) [A] |
| 0.1% (Exp3) | 3.17 (0.07) [A] | 0.65 (0.07) [C] | 2.17 (0.14) [A] | 6.05 (0.29) [AB] |
| 0.5% (Exp4) | 3.16 (0.08) [A] | 0.54 (0.04) [B] | 2.41 (0.10) [B] | 5.77 (0.35) [AB] |
| 1.0% (Exp5) | 3.09 (0.08) [A] | 0.41 (0.05) [A] | 2.44 (0.08) [B] | 5.68 (0.62) [B] |

\* Same superscript number means that there is no statistical difference (p>0.05).

**Table 2**. The shrinkage (%), maximum shrinkage rate (%/s), peak time (s) and shrinkage stress (MPa) of experimental composites with varying initiator and inhibitor concentration.

## 4. Conclusions

Within the limitation of this study, the polymerization shrinkage stress of the experimental dental composites, ranging 2.97 - 7.91 MPa, could be reduced by increasing inhibitor concentration.

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