

Influence of Irradiation Time on Volumetric Shrinkage and Flexural Properties of Flowable Resins

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The purpose of this study was to evaluate the influence of irradiation time on volumetric change and flexural properties of flowable resins. Four commercially available flowable resins were employed. For volumetric shrinkage measurement, resin pastes were inserted into a mold (2 mm in height, 4 mm in diameter) and put into a water-filled dilatometer. This was followed by light irradiation for 10, 20, or 30 seconds at 600 mW/cm². Volumetric shrinkage of the specimens was then determined from the change in the height of water meniscus, and the percentage volumetric change thereof was calculated. For flexural strength measurement, resin pastes were filled into a stainless steel mold (25×2×2 mm), and the middle one-third of the specimen was first irradiated. The remaining two-thirds were irradiated under the same irradiation conditions as volumetric shrinkage measurement. After 24-hour storage in 37°C water, three-point flexural tests were performed with a span length of 20 mm at a crosshead speed of 1.0 mm/min. One-way ANOVA followed by Tukey's HSD test were used for statistical analysis. For all materials tested except Estelite Flow Quick, both volumetric shrinkage and flexural strength increased with longer light irradiation time. Results of this study indicated that both volumetric shrinkage and flexural properties were influenced by light irradiation time and resin composite type.

Keywords: Flowable resin, Volumetric shrinkage, Flexural strength

INTRODUCTION

Resin composites are widely used in dentistry for various purposes. In the wake of the increased use of resin composite formulations in dentistry, scientific interest in the polymerization reaction of these materials has likewise increased^{1,2}. Polymerization of light-cured resin composites is influenced by a wide range of factors — such as light absorption and dispersion within the resin composite, shade and opacity of the composite material, filler type and filler load, concentration of the photoinitiator, power density delivered by the curing unit, and irradiation time.

Upon polymerization, resin composites undergo volumetric shrinkage. The latter phenomenon then generates elevated stress at the tooth-restoration interface that can result in marginal gap formation. Consequently, marginal gaps will negatively influence the longevity of a resin composite restoration^{3,4}. In light of the adverse effects that accompany volumetric shrinkage, it is therefore important that dental practitioners have a good knowledge of the polymerization characteristics of their choice of resin composite products before their clinical use.

Flowable resin composites are useful as restorative materials in that they can be applied by means of a small-gage dispenser. They are especially useful for cavities that are difficult to access⁵⁻⁷. With special attention and emphasis on the flow characteristics of these composites, manufacturers have developed improved formulations to meet the clinical require-

ments for better functionality. To obtain these specific flow characteristics, the filler content of the flowable composites are relatively low compared with those of hybrid resin composites⁸. Therefore, these materials might exhibit more volumetric shrinkage and lower mechanical properties than hybrid type resin composites that have higher filler loading.

As for the polymerization of visible light-cured resin composites, it is initiated by a photoinitiator activated by visible light at approximately 470 nm wavelength⁹. Regarding the critical factors that influence the thoroughness of polymerization, they are namely the intensity of incident radiation in the appropriate wavelength range and the length of irradiation time. In the same vein, it has been reported that both the power density of the curing light and the light-attenuating characteristics of the material influence the depth of cure¹⁰. To avoid lower mechanical properties, irradiation times longer than those recommended by the manufacturers have been advocated. However, clear and concrete specifications for irradiation times in such situations are not known.

In view of the many concerns that were unanswered pertaining to volumetric shrinkage and irradiation time, the objectives of this study were to test the hypotheses that (1) volumetric shrinkage and (2) flexural strength of flowable resin composites would not differ significantly with different irradiation times.

MATERIALS AND METHODS

Light-curing resins and curing unit

As listed in Table 1, the flowable resins employed in this study were Clearfil Flow FX (Kuraray Medical, Tokyo, Japan), Estelite Flow Quick (Tokuyama Dental, Tokyo, Japan), Filtek Flow (3M ESPE, St. Paul, MN, USA), and UniFil Lo Flo Plus (GC Corp., Tokyo, Japan). As for the curing unit, Optilux 501 (SDS Kerr, Danbury, CT, USA), of which the power density was adjusted to 600 mW/cm² as measured with a dental radiometer (Model 100, SDS Kerr), was used. Light guide diameter was 13 mm.

Volumetric shrinkage measurement

The test apparatus consisted of a water-filled dilatometer with a capillary tube of uniform 0.5 mm diameter and a length of approximately 130 mm. It was attached to a 25 cm³, brass-bottom density bottle by means of a ground glass joint¹¹⁾. A diagram of the dilatometer is shown in Fig. 1. The density bottle was filled with distilled water. During the test, liquid temperature was maintained by placing the density bottle on a thermostatically controlled plate.

Resin pastes were placed into a Teflon mold with 4.0 mm diameter and 2.0 mm height, and covered by a glass plate (Matsunami Glass, Tokyo, Japan) of 0.5 mm thickness. Light tip end of the curing unit was placed in a hole of the glass lid, and light irradiation of the specimen was done for 10, 20, or 30 seconds. Change in height of water meniscus was recorded using a CCD camera (DS-505, Nikon Corp., Tokyo, Japan) every five seconds, and magnified by a VCR (CT-1450, Hitachi Corp., Tokyo, Japan) from the start of light irradiation. Measurement accuracy of water meniscus was 0.2 mm.

Volumetric shrinkage (ΔV) of the specimens was calculated from the change in height of water meniscus (Δh) by the equation $\Delta V = 0.25\pi \cdot \Delta h \cdot d^2$, where "d" is the diameter of the capillary tube. Percentage volu-

metric change was given by the equation $\Delta V/V \times 100$, where V is the original specimen volume. Five measurements were conducted, and the average volumetric shrinkage thereof was obtained.

Flexural strength measurement

Flexural properties of the composites were tested according to ISO 4049 specification¹²⁾. Resin composites were filled into a stainless steel split mold with dimensions of 25×2×2 mm, positioned on a glass slide with a white filter backing during irradiation. Exit window of the curing unit was placed against the glass plate at the center of the specimen and exposed to light irradiation for 10, 20, or 30 seconds. Following which, the exit window was moved to the section next to the center overlapping the previous section. Light irradiation was performed by sequentially curing at three overlapping points on each upper and lower side until the entire sample surface was irradiated as recommended by the ISO 4049 specification. The specimens were then stored in the dark for 24 hours in water at 37°C before the mechanical test.

Eight specimens for each material were prepared and tested. The tests were performed with a universal machine (Instron Type 4204, Instron Corp., Canton, MA, USA) at a crosshead speed of 1.0 mm/min until specimen fracture. Maximum loads applied to the specimens were recorded, and the flexural strength (σ_f) in MPa was calculated as follows:

$$\sigma_f = 3W \cdot l / 2b \cdot d^2$$

where W is the maximum load, l is the distance between the supports (=20.0 mm), b is the width, and d is the depth of the specimen. In addition, elastic modulus in GPa was determined from the stress-strain curve using a computer software (Bluehill 2 Ver. 2.5, Instron Corp.) linked to the testing machine.

Table 1 Resin composites used in this study

Core foundation resin	Components	Filler content	Lot No.	Manufacturer
Clearfil Flow FX	Bis-GMA, TEGDMA, filler	61.3wt%	00002B	Kuraray Medical, Tokyo, Japan
Estelite Flow Quick	Bis-GMA, TEGDMA, filler	68.4wt%	P6	Tokuyama Dental, Tokyo, Japan
Filtek Flow	Bis-GMA, TEGDMA, dimethacrylate, filler	64.9wt%	8300A3	3M ESPE, St. Paul, MN, USA
UniFil Lo Flo Plus	UDMA, filler	42.5wt%	0406241	GC Corp., Tokyo, Japan

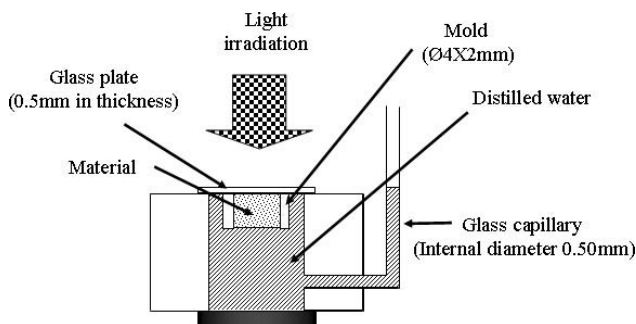


Fig. 1 Volumetric shrinkage measurement system used in this study.

Statistical analysis

The mean and standard deviation of the obtained data were calculated and tested for homogeneity of variance using Bartlett's test. Since the variances were found to be homogeneous, the data were subjected to one-way ANOVA followed by Tukey's HSD test. Significance level was set at 0.05 to make comparisons among the different irradiation times for each of the four products using a computer statistical package (Sigma Stat Ver. 3.1, SPSS Inc., Chicago, IL, USA).

RESULTS

Figure 2 shows the results of volumetric change, for a duration of 180 seconds, for each material irradiated for 30 seconds at a power density of 600 mW/cm². Volumetric shrinkage began soon after the start of light irradiation and continued after the end of light irradiation. Average volumetric shrinkage of the flow-

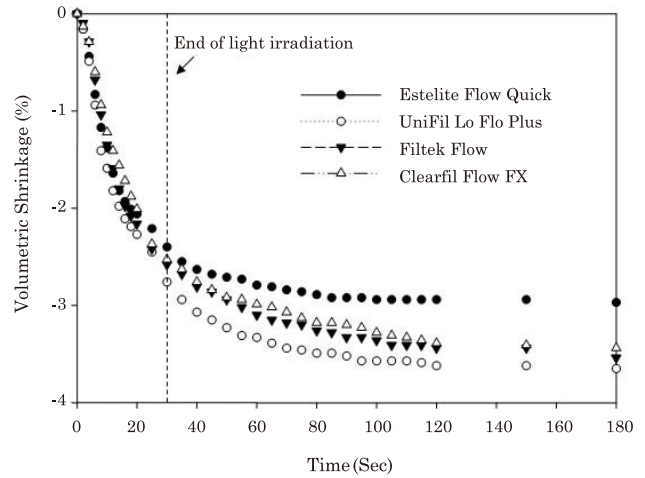


Fig. 2 Volumetric shrinkage of each material irradiated under the condition of 30-sec light irradiation with power density at 600 mW/cm².

Table 2 Effects of light irradiation time on volumetric shrinkage of resin composites

Composite	Light irradiation time (sec)	Shrinkage at the end of light irradiation (%)	Shrinkage at 180 sec (%)
Clearfil Flow FX	10	1.59 (0.07)	3.26 (0.08)
	20	2.01 (0.10)	3.44 (0.08)
	30	2.50 (0.09)	3.57 (0.10)
Estelite Flow Quick	10	1.43 (0.05)	2.92 (0.08)
	20	2.06 (0.07)	2.97 (0.08)
	30	2.46 (0.08)	2.99 (0.09)
Filtek Flow	10	1.33 (0.04)	3.27 (0.09)
	20	2.16 (0.06)	3.54 (0.10)
	30	2.40 (0.07)	3.67 (0.12)
UniFil Lo Flo Plus	10	1.61 (0.06)	3.31 (0.16)
	20	2.27 (0.08)	3.65 (0.12)
	30	2.34 (0.10)	3.71 (0.14)

Values in parentheses indicate standard deviations (n=5).

Values connected by lines indicate no significant differences among the different light irradiation times (p>0.05).

Table 3 Effects of light irradiation time on flexural properties of resin composites

Composite	Light irradiation time (sec)	Flexural strength (MPa)	Elastic modulus (GPa)
Clearfil Flow FX	10	103.9 (8.4)	5.8 (0.7)
	20	119.6 (9.3)	6.6 (0.8)
	30	122.6 (9.5)	6.8 (0.8)
Estelite Flow Quick	10	133.8 (8.9)	7.5 (0.8)
	20	134.1 (9.9)	7.5 (0.7)
	30	135.1 (9.6)	7.6 (0.9)
Filtek Flow	10	101.7 (9.6)	5.6 (0.7)
	20	120.4 (8.4)	6.5 (0.6)
	30	122.2 (9.0)	6.6 (0.7)
UniFil Lo Flo Plus	10	80.1 (8.9)	3.9 (0.8)
	20	90.3 (9.6)	4.5 (0.8)
	30	90.5 (8.5)	4.7 (0.7)

Values in parentheses indicate standard deviations (n=8).

Values connected by lines indicate no significant differences among the different light irradiation times (p>0.05).

able resin pastes after 180 seconds ranged from 2.99 to 3.71%.

Table 2 shows the effects of light irradiation time on the total volumetric shrinkage of flowable resins. For all materials tested except for Estelite Flow Quick, volumetric shrinkage increased with longer light irradiation time and the influence of this variable was significant.

Table 3 shows the means and standard deviations of flexural strength and elastic modulus. Under the conditions of 30-second light irradiation at a power density of 600 mW/cm², results of flexural strength and elastic modulus ranged from 90.5 to 135.1 MPa and from 4.7 to 7.6 GPa, respectively. Except for Estelite Flow Quick, there were significant increases in flexural strength and elastic modulus for specimens irradiated for 20 and 30 seconds, as compared with those irradiated for 10 seconds. However, there were no significant differences between those irradiated for 20 and 30 seconds.

DISCUSSION

For the evaluation of volumetric change of resin composites, the method used has a significant influence on the measured values. The modified dilatometer¹³⁻¹⁵⁾ used in this study eliminated the significant attenuation of the curing light by the density bottle. This was possible because specimens were light-irradiated through a cover glass of merely 0.5 mm thickness, and the reduction of power density through the cover glass was estimated to be very small. In clinical situations, resin composite materials are stored at room temperature (23°C) before being placed in the oral environment (37°C). As such, measurements in this study were conducted at 23°C room temperature. In addition, the dilatometer was set on a thermo-controlled plate to maintain this temperature during measurement. The latter temperature control precautionary measure was necessary as temperature discrepancy would cause volumetric expansion in resin composite materials. Although the latter situation inevitably arises in clinical practice, maintaining a constant-temperature environment for the dilatometer during the test was critical to measurement accuracy¹⁶⁾.

Volumetric shrinkage began soon after the start of light exposure and continued even after the end of light exposure (Fig. 2). The shrinkage noted after removal of the light source might be partially attributed to the postpolymerization reaction of residual monomers. Shrinkage was caused by the polymerization reaction, whereby van der Waals distances were changed to covalent bond distances. The degree of shrinkage was dictated by the number of covalent bonds formed, which was the extent of the polymerization reaction¹⁷⁾.

Water sorption and subsequent swelling of a restorative material due to its hydrophilic nature, such as resin-modified glass ionomer cement, might contribute to compensating for volumetric shrinkage¹⁸⁾. A reason for volumetric change is a swelling of the material by increased absorption of water. With resin composites, volumetric changes observed suggested that immersion in water might not negate part of the volumetric shrinkage. This was because water uptake by resin composites requires hours or days to reach saturation¹⁹⁾. Moreover, water sorption is reduced by the use of more hydrophobic monomers like Bis-GMA, which was used in the resin composites in this study. The delayed nature of water sorption thus minimized its effect on volumetric shrinkage measured in this study.

From the results of this study, null hypothesis (1) was not supported. Volumetric shrinkage of the flowable resins used in this study was influenced by the light irradiation time of the resin pastes, except for Estelite Flow Quick. Volumetric shrinkage of a resin paste depends on factors such as filler loading, filler type, and filler size. Other factors that affect shrinkage are the monomer and polymerization initiator systems because they determine the polymer structure of the material²⁰⁾. Most light-cured materials use camphorquinone as a photoinitiator²¹⁾. To initiate polymerization, an adequate intensity of visible light at wavelengths around 470 nm is required^{22,23)}. Light of the appropriate wavelength is absorbed by the photoinitiator, which then reacts, in its excited state, with an amine-reducing agent to produce reactive free radicals^{22,23)}. Working hand-in-hand, the transmission of light through a light-cured material and the composition of the photoinitiator both influence the mechanical properties of the resin composite material²³⁾. As for the intensity of light passing through the material, it is controlled by the absorption and scattering of the latter's components²⁴⁻²⁶⁾.

The polymerization of resins is invariably accompanied by the volumetric shrinkage of the cured material. Shrinkage is associated with the polymerization reaction in a complex way. Reduction in shrinkage strain could be attributed to reduced network connectivity, or to an increased propensity for flow of the material²⁷⁾. Throughout the entire polymerization process, plastic deformation or flow of the composite resin may occur to a limited extent. Reduced irradiation time may result in storage modulus development at a slow enough rate to allow for flow and dissipation of stress, while maintaining a sufficient bond to tooth structure²⁸⁾. As the setting process proceeds, shrinkage and flow decrease gradually as storage modulus increases. With a shorter irradiation time, flowable resins restrain the stress relief much more by not allowing enough flow to reduce internal stress. Restriction of the flow capacity by the configu-

ration of the restoration also enhances contraction stress²⁹. In other words, marginal adaptation could be enhanced by optimal rheological effects. In particular, these beneficial effects of flow are being utilized when shorter irradiation times are employed with most flowable composites.

An irradiation unit with high power density is recommended based on mechanical properties^{30,31}. On the other hand, the possible negative influence of longer irradiation time on stress development must also be considered. As polymerization shrinkage increases with longer irradiation time, adverse effects on the marginal adaptation of resin fillings may occur. In the present study, prolonged light irradiation resulted in an increase in volumetric shrinkage (Table 2). This increase in shrinkage suggested that further polymerization occurred with increased irradiation time. Longer light irradiation times than those quoted by the manufacturers have been advocated to avoid portions of poor polymerization in the material. On the other hand, a shorter light irradiation time might retain some flow capacity within the polymerized specimen as a result of its lower mechanical properties. In light of these pros and cons of longer and shorter light irradiation times, irradiation conditions appropriate to light-cured materials should be suggested and specified.

In the present study, the flexural strength test was chosen to assess whether differences in mechanical properties were present in flowable resins when cured with different irradiation times. A three-point flexural test typically utilizes rectangular bars subjected to flexural loading, thereby providing a state of pure tensile stress at the point of the specimen opposite the load. The relative ease of predictably concentrating this tensile stress may be the key reason why this test is used more frequently than diametral tensile measurement. On the other hand, it should be highlighted that the dimensions of three-point flexural strength specimens are not representative of the clinical situation. This laboratory condition might lead to inconsistencies in the polymerization of the composite specimens.

No significant differences in volumetric shrinkage and flexural properties with different irradiation times were found for Estelite Flow Quick. Estelite Flow Quick employed a novel photopolymerization initiator system including a radical amplifier. It has been reported that the radical amplifier had higher polymerization activity than conventional polymerization initiators³². The flowable resins used in this study were cured by a free radical polymerization reaction, and a photoinitiator such as camphorquinone (CQ) was employed. CQ required a co-initiator for an effective polymerization process to occur, and a tertiary amine photoreductant was employed. The tertiary amine interacted

with an activated triplet state of CQ to form an intermediate excited complex, followed by producing reactive radicals for polymerization. To improve polymerization, an accelerator such as the radical amplifier was incorporated with the initiator system of Estelite Flow Quick. As a result, the irradiation time for this resin was reduced by 30%, as compared with those of the original light-cured resins.

For the other flowable resins used in the current study, the irradiation time of 10 seconds tended to show less polymerization shrinkage and lower flexural properties than the irradiation times of 20 and 30 seconds. This result provided some support for shorter irradiation times as a means to reduce polymerization shrinkage and the associated internal stresses³³. For flowable resins, an increased flow capacity might provide more contraction stress relaxation — thereby reducing the frequency of marginal debonding and associated microleakage. On the other hand, an increased irradiation time is recommended to achieve adequate mechanical properties of flowable resins. Taken together, the success of a flowable resin restoration indeed depends on many other critical factors related to the clinical performance of the restoration.

CONCLUSIONS

The results of this *in vitro* study indicated that the volumetric shrinkage and flexural properties of flowable resins were affected by light irradiation time and resin composite type. With longer light irradiation, higher volumetric shrinkage and flexural properties were recorded except for a flowable resin which included a radical amplifier.

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