

## 각기 다르게 분류되는 methacrylate 기반의 복합레진 중합 평가\*

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Investigation of polymerization of methacrylate-based composite resins those belong to different categories\*

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### ABSTRACT

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The present study investigated the polymerization of composite resins which belong to different categories. Each four different composite resins (low shrinkage, nanohybrid, microhybrid, and flowable resins) from three different manufacturers were chosen and light cured. Through the study, polymerization shrinkage and flexural properties were evaluated. Regardless of product and manufacturer, low shrinkage and flowable resins showed the lowest (7.7-12.3  $\mu\text{m}$ ) and highest (26.2-35.4  $\mu\text{m}$ ) polymerization shrinkage, respectively. The shrinkage difference between nanohybrid and microhybrid products was low. The flexural properties (strength and modulus) from products of different categories and from products of the same manufacturer had no consistency. All these values showed inconsistently changing pattern after immersion in distilled water. The tested composite resins belong to different categories but products of the same manufacturer and composite resins belong to the same category but products of different manufacturers had inconsistently different flexural properties.

KEY WORDS: Composite resin; Polymerization shrinkage; Flexural properties

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### INTRODUCTION

Since the introduction of Bis-GMA by Bowen in 1960s, countless methacrylate-based composite resins have been introduced in dentistry for the restoration of damaged teeth. To serve as a reliable and durable restorative material, composite resins are needed to satisfy two aspects: functional and aesthetic. To be compatible with host teeth, composite resins are needed to have high strength, modulus, and surface hardness, low water sorption, solubility, and thermal

expansion (Ferracane, 2011; Drummond, 2008). Also, due to the increased interest in the aesthetics of teeth, color matching through diverse color options and color stability are important (Tsubone et al., 2012, Lee et al., 2010). According to reviewing of many studies, one of the main reasons attributable to the restoration fracture and secondary caries is connected to the polymerization shrinkage that occurred during and after light curing (Kemp-Scholte & Davidson, 1988; Lai & Johnson, 1993; Davidson & Feilzer, 1997). Since polymerization shrinkage is the result of the reduction of molecular spacing in the resin matrix due to the change of governing force between molecules, it is inevitable in the monomer-based composite resins. To overcome this problem, increase of filler content has been suggested

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to reduce monomers content those are responsible for the shrinkage. Inclusion of nanofillers enhances translucency by increasing the optical path length of incident light through the reduction of light scattering with nanofillers those are shorter than the wavelength of the incident light, but such increase of filler content may reversely decrease mechanical properties and hinder easy handling due to reduced resin matrix (Ferracane, 1995; Kim et al., 2007). Introduction of a new monomer system using a ring-opening polymerization mechanism may challenge its use over the free radical-mediated conventional composite resins in addition to its first impact of low shrinkage modality (Weinmann et al., 2005; Ilie & Hickel, 2006). The oxirane and siloxane molecules allow the silorane-based composite resins to have low shrinkage, water sorption, and solubility features.

Composite resins available in dentistry can be differently classified basically by the filler system such as filler size and filler loading (Ferracane, 2011). Many

categories can be made according to the ways of classification, but based on the products produced by the same manufacturer, only several categories are possible. The purpose of the present study was to evaluate the polymerization shrinkage and flexural properties of the composite resins which can be categorized into four ways according to function and filler size: low shrinkage, nanohybrid, microhybrid, and flowable resins.

## MATERIALS AND METHODS

### Specimens and light-curing units

For the study, four different composite resins (all A3 shade) from three different manufacturers were used. Products were chosen based on their characteristics such as low shrinkage, nanohybrid, microhybrid, and flowable by the manufacturers' claim. Details of the

**Table 1.** Materials tested in the present study

Code	Composition	Filler type	Filler content vol%/wt% <sup>1</sup> /wt% <sup>2</sup>	Company
AP	Bis-EMA, Bis-GMA, TEGDMA	Glass frit, amorphous silica	74/88/85.8	Bisco Inc., Schaumburg, IL, USA
AE	Bis-EMA, Bis-GMA	Ba-B-F-Al-silicate glass, fumed SiO <sub>2</sub>	54/73/65.2	
AB	Bis-EMA, TEGDMA	Glass filler, amorphous silica	55/76/71.0	
AF	Bis-EMA, TEGDMA	Barium glass, glass filler	42/60/56.2	
CP	Bis-GMA, TEGDMA	Glass ceramics, alumina filler	82/62/58.1	Kuraray, Tokyo, Japan
CE	Bis-GMA, hydrophobic dimethacrylate	Prepolymerized (PPF) filler, barium glass filler	66/78/58.2	
CX	Bis-GMA, TEGDMA	Barium glass, silica, SiO <sub>2</sub>	70/86/83.2	
CF	TEGDMA	Barium glass, colloidal silica	62/81/75.9	
FL	Silorane	Silanized quartz, yttrium fluoride	55/76/75.6	3M ESPE, St. Paul, MN, USA
ZX	Bis-EMA, Bis-GMA, TEGDMA, UDMA	Non-aggregated silica, zirconia/silica, nanoclusters	63.3/78.5/74.7	
Z2	Bis-EMA, Bis-GMA, TEGDMA, UDMA	Zirconia/silica	60/84.5/78.0	
ZF	Bis-GMA, TEGDMA	Zirconia/silica cluster fillers	55/65/61.0	

wt%<sup>1</sup>: weight percent provided by the manufacturers

wt%<sup>2</sup>: weight percent determined by ash method by the present authors

AP: Aelite LS Posterior; AE: Aelite Aesthetic Enamel; AB: Aelite All Purpose Body; AF: AeliteFlo

CP: Clearfil Majesty Posterior; CE: Clearfil Majesty Esthetic; CX: Clearfil AP-X; CF: Clearfil Flow

FL: Filtek LS; ZX: Filtek Z350XT; Z2: Filtek Z250; ZF: Filtek Z350 Flow

Low shrinkage: AP, CP, and FL

Nanocomposites: AE, CE, and Z3

Microhybrid: AB, CX, and Z2

Flowable: AF, CF, and ZF

tested products were summarized in Table 1.

For the light curing, a QTH [Optilux 501, Kerr, Danbury, CT, USA] and a LED [L.E.Demetron, Kerr, Danbury, CT, USA] light-curing units were used as light sources. The output light intensity of QTH and LED was approximately 900 mW/cm<sup>2</sup> as measured using the built-in radiometer.

### Filler weight

The filler weight (wt%) of each resin product was determined using standard ash method in addition to the nominal weight which was provided by the manufacturers (Kim et al., 2002). The weight (approximately 50 mg) of each resin specimen was measured using an analytical balance after light curing for easy handling. Then the specimen was heated in an electric furnace at 650°C for 30 min to burn out the organic matrix. The wt% was determined by dividing the weight of original specimen to that of the remaining specimen after heating in furnace.

### Polymerization shrinkage

Polymerization shrinkage ( $\mu\text{m}$ ) of the specimens during and after the light-curing process was measured (n=5 for each product) using a linometer (RB 404, R&B Inc., Daejeon, Korea). To produce cylindrical specimens (diameter: 4 mm, thickness: 2 mm), composite resin was filled into a disc-type mold (inner diameter: 4 mm, thickness: 2 mm), and then removed carefully using a putty stick. To ease removal, the inner wall of the mold was thinly coated with resin separator. In the case of flowable resin, since resin can flow downward, placing resin over the aluminum disc was performed as quickly as possible after repeated exercise. The removed specimen was then placed at the centre of an aluminum disc (the specimen stage of the measurement system) and its top surface was covered with a glass slide. The end of the light guide was placed in contact with the glass slide. Before light curing, the initial position of the aluminum disc was set to zero. Each specimen was exposed to light from the light-curing unit for 40 s and polymerization

shrinkage was measured for 130 s using a shrinkage sensor placed beneath the aluminum disc. The resolution and measurement range of the shrinkage sensor were 0.1  $\mu\text{m}$  and 100  $\mu\text{m}$ , respectively.

### Flexural properties

A three-point bending test was performed to determine the flexural properties [flexural strength (FS) and modulus (FM)]. To make specimens, a metal mold (25×2×2 mm) was filled with resin according to the ISO 4049 guidelines (ISO 4049). After filling the mold, both top and bottom surfaces were covered with glass slides to make a flat surface. The specimen was irradiated for 40 s using a light-curing unit. Since the specimen was much wider (25 mm) than the tip size (7 mm), five light exposures were performed on each side by overlapping the curing light. After light curing (n=14), seven specimens were removed from the mold and aged for 24 h in a 37°C dry and dark chamber. The other specimens were then immersed in distilled water and kept in a 37°C dry and dark chamber for 2 weeks. After aging or immersion, the specimens were loaded to a universal test machine (Instron 3345, Grove City, PA, USA) at a crosshead speed of 1 mm/min. FS ( $\sigma_f$  in MPa) was obtained using the following formula

$$\sigma_f = 3DP/(2WH^2)$$

where D is the distance between the supports (20 mm), P is the maximum failure load (N), W is the width (2 mm), and H is the height (2 mm) of the tested specimen. FM (E in GPa) was obtained using the following formula

$$E = (P/D) \cdot (D^3/(4WH^3))$$

where P/D is the slope in the linear portion of the load-displacement curve.

### Statistical analysis

The results of each test were analyzed using ANOVA for resin products. A post-hoc Tukey test was followed

for a multiple-comparison, T-test was performed to find the statistical difference between light-curing units. All tests were analyzed at  $p < 0.05$ .

## RESULTS

The filler weight determined by ash method is shown in Table 1. The nominal wt% (provided by the manufacturers) of the tested products ranged from 60 to 92%, on the other hand, wt% determined by ash method ranged from 56.2 to 85.8%. Depending on product, the difference of both values ranged from 0.5 to 36.8%.

**Table 2.** Polymerization shrinkage ( $\mu\text{m}$ ) of the tested specimens

Code	LED <sup>1</sup>	QTH <sup>1</sup>
AP	10.0 $\pm$ 0.4 <sup>a</sup>	7.7 $\pm$ 0.9 <sup>a</sup>
AE	24.7 $\pm$ 0.4 <sup>b</sup>	24.3 $\pm$ 1.6 <sup>b</sup>
AB	21.0 $\pm$ 0.6 <sup>c</sup>	20.8 $\pm$ 0.4 <sup>c</sup>
AF	35.1 $\pm$ 1.8 <sup>d</sup>	35.4 $\pm$ 1.3 <sup>d</sup>
CP	12.1 $\pm$ 0.2 <sup>ae</sup>	12.3 $\pm$ 0.2 <sup>ef</sup>
CE	12.9 $\pm$ 0.4 <sup>eg</sup>	11.3 $\pm$ 0.4 <sup>fg</sup>
CX	13.5 $\pm$ 0.5 <sup>eg</sup>	12.8 $\pm$ 0.6 <sup>ef</sup>
CF	26.2 $\pm$ 1.8 <sup>b</sup>	26.5 $\pm$ 1.0 <sup>h</sup>
FL	10.1 $\pm$ 0.5 <sup>a</sup>	9.1 $\pm$ 0.5 <sup>ag</sup>
ZX	15.5 $\pm$ 0.5 <sup>g</sup>	14.7 $\pm$ 0.6 <sup>e</sup>
Z2	13.6 $\pm$ 0.3 <sup>eg</sup>	13.7 $\pm$ 0.7 <sup>e</sup>
ZF	30.0 $\pm$ 1.1 <sup>h</sup>	30.1 $\pm$ 0.9 <sup>i</sup>
p-value	<0.001	<0.001

\* Statistically significant difference on resin product is shown by superscript letters<sup>a, b, ...</sup>. Same letters or numbers (according to the T-test) are not significantly different ( $p > 0.05$ ).

Table 2 shows the polymerization shrinkage of the specimens for different light-curing units. Regardless of product and manufacturer, low shrinkage and flowable resins showed the lowest (7.7-12.3  $\mu\text{m}$ ) and highest (26.2-35.4  $\mu\text{m}$ ) values, respectively. Nanohybrid and microhybrid products showed less difference in their values, and two microhybrid products (AB and Z2) showed slightly lower polymerization shrinkage than that of their corresponding nanohybrid products (AE and ZX). Shrinkage difference by the different light-curing units was statistically insignificant ( $p > 0.05$ ).

Table 3 shows the flexural properties (FS and FM)

of the specimens before and after immersion test. Before immersion, FS and FM ranged 99.2-188.6 MPa and 5.24-20.87 GPa, respectively, depending on product and light-curing unit. After immersion, these values changed to 82.9-176.7 MPa and 3.89-21.80 GPa. Most products showed decreased FS and FM values after immersion (approximately 0.2-44.7% for FS and 0.5-32.7% for FM depending on product and light-curing unit). FS change was statistically significant ( $p < 0.05$ ), whereas FM change was statistically insignificant ( $p > 0.05$ ).

## DISCUSSION

The tested products in the present study can be differently classified based on the claimed characteristics by the manufacturers: low shrinkage, nanohybrid, microhybrid, and flowable resins. The basis of these classifications is the restoration function, filler type, and flowability of the tested products.

Low shrinkage nature is important in the dental restorative materials to minimize unwanted clinical problems such as marginal leakage, restoration fracture, postoperative sensitivity, and recurrence of secondary caries (Kemp-Scholte & Davidson, 1988; Lai & Johnson, 1993; Davidson & Feilzer, 1997). To overcome shrinkage problems, so far, there were two main approaches: increasing filler content and adopting new monomer system. AP and CP were claimed to contain much higher filler content than the other products. To increase the filler content, recently nanofillers are routinely contained in the dental composite resins. In the case of FL, it showed much lower filler content than AP and CP, but, contains completely different monomer system. Unlike most free radical-mediated methacrylate-based composite resins, FL adopts silorane-based ring-opening polymerization system. Silorane is a hybrid compound of siloxane and oxirane molecules, oxirane molecules in silorane achieve low polymerization shrinkage through a cationic ring-opening mechanism (Tilbrook et al., 2000; Eick et al., 2002). In the case of AP and FL, they certainly have less shrinkage values than the other products (7.7-10.1  $\mu\text{m}$  vs 13.6-35.4  $\mu\text{m}$ )

**Table 3.** Flexural properties of the tested specimens before and after immersion at 37 °C distilled water for 2 weeks

	Flexural properties (Before)		Flexural properties (After)		
	Strength (FS, MPa) <sup>1</sup>	Modulus (FM, GPa) <sup>1</sup>	Strength (FS, MPa) <sup>2</sup>	Modulus (FM, GPa) <sup>1</sup>	
LED	AP	127.3 ± 8.0 <sup>a</sup>	14.33 ± 0.85 <sup>ab</sup>	95.2 ± 6.2 <sup>abc</sup>	13.81 ± 0.68 <sup>a</sup>
	AE	112.1 ± 12.0 <sup>ab</sup>	9.22 ± 0.62 <sup>cd</sup>	85.9 ± 9.2 <sup>a</sup>	8.59 ± 0.15 <sup>b</sup>
	AB	149.5 ± 10.7 <sup>cd</sup>	10.62 ± 0.60 <sup>c</sup>	115.6 ± 10.4 <sup>cd</sup>	10.31 ± 0.31 <sup>c</sup>
	AF	131.2 ± 8.8 <sup>abc</sup>	5.24 ± 0.14 <sup>e</sup>	95.7 ± 7.5 <sup>abc</sup>	3.89 ± 0.08 <sup>d</sup>
	CP	114.6 ± 7.0 <sup>ab</sup>	7.90 ± 0.43 <sup>df</sup>	82.9 ± 4.7 <sup>ab</sup>	7.76 ± 0.15 <sup>b</sup>
	CE	99.2 ± 7.0 <sup>b</sup>	9.36 ± 0.35 <sup>cf</sup>	102.4 ± 7.6 <sup>abc</sup>	7.40 ± 0.21 <sup>b</sup>
	CX	185.7 ± 12.1 <sup>e</sup>	20.49 ± 1.21 <sup>g</sup>	164.8 ± 9.8 <sup>e</sup>	20.35 ± 1.50 <sup>e</sup>
	CF	172.9 ± 11.7 <sup>ef</sup>	10.89 ± 0.84 <sup>c</sup>	172.6 ± 15.4 <sup>e</sup>	12.40 ± 0.20 <sup>f</sup>
	FL	132.5 ± 11.2 <sup>ac</sup>	12.78 ± 0.89 <sup>a</sup>	128.5 ± 3.5 <sup>d</sup>	12.20 ± 0.98 <sup>fg</sup>
	ZX	164.0 ± 8.7 <sup>de</sup>	15.25 ± 0.21 <sup>b</sup>	90.6 ± 9.5 <sup>abf</sup>	11.02 ± 0.56 <sup>cg</sup>
	Z2	162.9 ± 13.4 <sup>df</sup>	15.66 ± 1.60 <sup>b</sup>	135.2 ± 11.8 <sup>d</sup>	13.32 ± 0.38 <sup>af</sup>
	ZF	167.9 ± 6.1 <sup>de</sup>	8.61 ± 0.28 <sup>df</sup>	114.5 ± 11.9 <sup>cd</sup>	5.89 ± 0.11 <sup>g</sup>
QTH	AP	138.2 ± 6.6 <sup>ab</sup>	20.47 ± 1.67 <sup>a</sup>	123.7 ± 7.4 <sup>ab</sup>	21.80 ± 1.65 <sup>a</sup>
	AE	107.2 ± 6.9 <sup>c</sup>	9.06 ± 0.83 <sup>bc</sup>	101.4 ± 4.6 <sup>ac</sup>	8.79 ± 0.72 <sup>b</sup>
	AB	135.5 ± 8.0 <sup>ad</sup>	10.79 ± 0.56 <sup>b</sup>	101.8 ± 11.1 <sup>ac</sup>	10.20 ± 0.46 <sup>bc</sup>
	AF	141.4 ± 3.1 <sup>ab</sup>	5.81 ± 0.26 <sup>d</sup>	99.2 ± 10.5 <sup>ac</sup>	3.91 ± 0.23 <sup>d</sup>
	CP	106.2 ± 7.8 <sup>c</sup>	9.98 ± 0.44 <sup>bc</sup>	90.5 ± 12.4 <sup>cd</sup>	9.56 ± 0.53 <sup>b</sup>
	CE	103.2 ± 9.8 <sup>c</sup>	10.34 ± 0.57 <sup>bc</sup>	85.4 ± 8.9 <sup>ce</sup>	9.19 ± 0.26 <sup>b</sup>
	CX	180.8 ± 14.6 <sup>ef</sup>	20.87 ± 1.45 <sup>a</sup>	176.7 ± 18.0 <sup>f</sup>	19.35 ± 1.50 <sup>e</sup>
	CF	168.3 ± 17.7 <sup>efg</sup>	10.47 ± 0.82 <sup>b</sup>	162.4 ± 16.6 <sup>f</sup>	11.51 ± 0.58 <sup>cf</sup>
	FL	125.4 ± 14.7 <sup>ac</sup>	12.77 ± 0.55 <sup>e</sup>	128.5 ± 22.8 <sup>ab</sup>	12.71 ± 0.85 <sup>f</sup>
	ZX	153.5 ± 11.9 <sup>bdg</sup>	14.74 ± 0.86 <sup>f</sup>	110.7 ± 8.8 <sup>adeg</sup>	12.20 ± 0.82 <sup>f</sup>
	Z2	188.6 ± 13.2 <sup>e</sup>	16.37 ± 0.99 <sup>f</sup>	134.0 ± 15.4 <sup>bg</sup>	13.16 ± 0.72 <sup>f</sup>
	ZF	162.1 ± 12.8 <sup>bf</sup>	8.42 ± 0.71 <sup>c</sup>	115.4 ± 4.6 <sup>adg</sup>	5.97 ± 0.16 <sup>g</sup>

\* Statistically significant difference on resin product is shown by superscript letters<sup>a,b</sup>. Same letters or numbers (according to the T-test) are not significantly different ( $p < 0.05$ ).

within the same manufacturer, but, CP has statistically similar shrinkage values compared to the other products (CE and CX) within the same manufacturer. High shrinkage in flowable resins (AF, CF, and ZF) is mainly due to low filler content to increase the content of diluents such as TEGDMA even though CF contains exceptionally high content of fillers as a flowable resin. The filler content of nanohybrid products was lower than that of microhybrid products even though nanofillers were claimed to be included in their resin matrix. However, the resultant shrinkage difference between them is low (11.3-24.7  $\mu\text{m}$  vs 12.8-21.0  $\mu\text{m}$ ).

Furthermore, the shrinkage pattern was not consistent in these two classes. According to the linear fit, there was an inverse linear correlation between the filler content and polymerization shrinkage ( $R=0.5-0.72$  depending on filler content and light-curing unit), so containing high content of fillers may produce less shrinkage if it is methacrylate-based composite resin.

The flexural properties (FS and FM) are the measure of material resistance against transverse stress. FS values of products of different categories had no consistent pattern among products of different manufacturers. In the case of Bisco and Kuraray products, microhybrid

and nanohybrid products (AB and CX; AE and CE) showed the highest and the lowest FS, respectively. On the other hand, in the case of 3M, except low shrinkage product (FL), there was no significant difference in FS among products. A linear correlation with filler content was not over 0.56 according to the linear fit, indicates other unknown factors those affect the resultant values. One probable factor that affects the result is the inclusion of internal defects during manufacturing or specimen preparation process (Zeng et al., 1996; Della Bona et al., 2003). Such formed defects become the origin of fracture when there is external stress. The FS values evaluated are similar to those of many other methacrylate-based composite resins (Ilie & Hickel, 2009). Modulus is the measure of material's stiffness. In the load-displacement curve of the bending test, modulus is the slope of the curve in the elastic range, so high modulus implies a great stiffness (Ferracane, 2001). FM of the tested products has a linear correlation ( $R=0.87$  and  $0.88$  for DE and OP, respectively) with the filler content (wt% determined by ash method). The FM values obtained are lower than that of dentin (17-25 GPa), so those products can compatible with the underlying dentin (Kinney et al., 2003; Xu et al., 1998). After immersion in distilled water, most products tested showed decrease of FS and FM values even though such decrease had no consistent dependence on product category.

## CONCLUSION

Within the limitations of the present study, the following conclusions could be reached:

1. Regardless of product and manufacturer, low shrinkage and flowable resins showed the lowest (7.7-12.3  $\mu\text{m}$ ) and highest (26.2-35.4  $\mu\text{m}$ ) polymerization shrinkage, respectively. The effect by light-curing units is insignificant ( $p>0.05$ ).
2. In most cases tested, composite resins produced by the same manufacturer but belong to different categories have inconsistently different flexural properties. Also, composite resins those belong to the

same category but products of different manufacturers showed inconsistently different flexural properties as well.

3. Accordingly, the user needs more attention to the detailed flexural properties of each product than nominal classification (category) of the resin products for the reliable and durable restoration.

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