



유동성레진과 수복용레진으로 형성된 레진층의 굴곡 및 압축성질

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Flexural and compressive properties of layered specimens formed with flowable and composite resins

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깊은 와동을 수복할 때 베이스 재료로 유동성레진이 사용된다. 이런 경우 유동성 레진은 상부에 위치하는 다른 복합레진과 층을 이루게 되는데 본 연구에서는 그렇게 이루어진 레진층의 기계적 성질을 평가한다. 이를 위하여 5종의 유동성레진을 택하여 베이스 재료로 사용하고 2종의 복합레진을 택하여 베이스 상부에 위치하도록 하였다. 이후에 이들 각각과 10가지 조합으로 된 층 시료의 굴곡 및 압축성질을 평가하였다. 그 결과 유동성레진 자체가 높은 굴곡 강도와 modulus값을 가진 경우 복합레진과 층을 이루어도 높은 굴곡 강도와 modulus를 보였다. 그러나 압축강도의 경우는 그렇지 않아서 압축강도가 크던 작던(259.8~439.8 MPa)간에 층에 이룬 시료의 압축강도는 높지 않았고(251.4~295.3 MPa), 굴곡 modulus는 압축 modulus와 선형적으로 높은 상관관계를 보였다. 결국 복합레진과 유동성레진으로 층을 이룬 시료의 기계적 성질은 상부에 위치하는 복합레진에 상관없이 베이스의 유동성레진에 의존하였지만 그 성질은 일관성을 보이지 않았다.

색인단어 : 레진수리, 복합레진, 굴곡성질, 압축성질

INTRODUCTION

Dimethacrylate-based composite resins are popular for dental restoration due to agreeable mechanical properties, excellent aesthetics, easy handling, and quick polymerization [김사학 등, 2017; 임범순 등, 2016; 배지명 & 오승환, 2015; Ferracane, 2011; Lee et al., 2010; Wiegand et al., 2007].

As to composite resins, polymerization is a process that converts monomers to polymer network with hardening of the texture and shrinking of the volume through a shortening of the length which occurs during and after light curing. Since polymerization process involves the change of governing bond among the constituting molecules as a form of polymerization shrinkage, it produces many unwanted clinical problems, such as postoperative sensitivity, marginal leakage, recurrence of caries, or bond failure [Watts, 2005; Kemp-Scholte & Davidson; 1988; Davidson & Feilzer, 1997]. The filled composite resin within the tooth cavity inevitably

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confronts such problems due to shrinkage, repeated thermal stress by daily diets, and mechanical stress through the mastication. Failure (detachment) of the restored material can happen in unexpected situations and discoloration, wear, and degradation of the upper part of the restoration also will happen as a time-dependent process, so all these require repair or replacement depending on situation [Tyas et al., 2000; Moncada et al., 2009]. Repair of composite resins is less invasive process than replacement of the whole structure because repair involves partial modification of the exposed, defected, or aged structure, so the base material can be remained intact state if it was not damaged or detached.

The remarkable advantage of flowable resins over most composite resins is easy handling feature with excellent marginal adaptation [김영진 & 권태엽, 2016; Yazici et al., 2008; Abedian & Millstein, 2006]. Due to excellent flowable nature with low modulus, flowable resins are frequently chosen as a lining or base material for tooth cavity restoration [Ivanovas et al., 2011; Beun et al., 2012]. However, since flowable resins have low microhardness due to low filler content compared to many composite resins, generally, flowable resins are used with composite resins by forming a layer. Many recently available flowable resins have different filler content and viscosity, so they may have different mechanical properties, result in diverse mechanical properties if flowable resins are coupled with composite resins of different mechanical properties. The purpose of the present study was to evaluate the mechanical properties (flexural and compressive properties) of the layered specimens with composite resin for an overlying material and flowable resin as a base material.

MATERIALS AND METHODS

1. Specimens

For the study, two composite resins [Tetric N Ceram

(TC) and Filtek Z350XT (ZX)] and five flowable resins [Estelite Flow Quick (EQ), Premise Flow (PF), Palfique Estelite LV (PL), Revolution Formula 2 (R2), and Filtek Z350 Flow (ZF)] of all A3 shade were used for overlying and base material, respectively. The details are shown in Table 1. For the light curing of specimens, a light-emitting diode (LED) light-curing unit (LCU) (L.E.Demetron, Kerr, Orange, CA, USA) with light intensity of 900 mW/cm² was used.

2. Three-point bending test

A three-point bending test was performed to determine the flexural properties [flexural strength (FS) and modulus (FM)]. To produce specimens (25×2×2 mm with combination of 1+1 mm), at first, a stainless steel mold of 25×2×1 mm was filled using TC (ZX). Both top and bottom surfaces were covered using two thin glass slides to make each surface flat, light cured for 200 s (40 s × 5) through five exposure by overlapping exposed areas. The cured specimen was removed from the mold, plugged into the bottom of 25×2×2 mm mold. The upper empty space was filled using one of five flowable resins, covered using a thin glass slide, pressed firmly to make a flat surface, light cured for 200 s through five overlapping exposures. After light curing, the specimen (n=14) was removed from the mold and aged for 24 h in at 37°C dry, dark chamber. After aging, randomly chosen seven specimens were loaded into a universal test machine (Instron 3345, Grove City, PA, USA) at a crosshead speed of 1 mm/min. FS (σ_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

Table 1. Materials tested in the present study

	Composition	Filler content vol%/wt%	Manufacturer
TC	Bis-EMA, TEGDMA, UDMA, Barium glass, YbF ₃ , SiO ₂	57/80,5	Ivoclar Vivadent Schaan, Liechtenstein
ZX	Bis-EMA, Bis-GMA, TEGDMA, UDMA, Non-aggregated silica, zirconia/silica, nanoclusters	63,3/78,5	3M ESPE St. Paul, MN, USA
EQ	Bis-EMA, TEGDMA, UDMA, Silica-zirconia, silica-titania filler	53/71	Tokuyama Tokyo, Japan
PF	TEGDMA, Barium glass, silica filler	54,6/72,5	Kerr Orange, CA, USA
PL	Bis-EMA, Bis-GMA, TEGDMA, Silica-zirconia, silica-titania filler	50/65	Tokuyama Tokyo, Japan
R2	TEGDMA, Barium glass	41/60	Kerr Orange, CA, USA
ZF	Bis-GMA, TEGDMA, Zirconia/silica cluster fillers	55/65	3M ESPE St. Paul, MN, USA

TC: Tetric N Ceram; ZX: Filtek Z350XT; EQ: Estelite Flow Quick; PF: Premise Flow; PL: Palfique Estelite LV; R2: Revolution Formula 2; ZF: Filtek Z350 Flow

Bis-EMA: ethoxylated bisphenol A glycidyl methacrylate; Bis-GMA: bisphenol A glycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate

wt%: nominal weight (according to the information supplied by the manufacturers)

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

where P/D is the slope of the linear portion of the load-displacement curve. The remaining seven specimens were immersed in the 37°C distilled water for 2 weeks and then the same measurements were performed under the same conditions as stated above.

3. Compression test

To produce specimens (diameter: 3 mm, height: 6 mm with combination of 3+3 mm) for the measurement of compressive properties [compressive strength (CS) and modulus (CM)], two identical hemicylinders (diameter: 3 mm, height: 3 mm) were combined together to form a cylinder and the inner empty space was filled with TC (ZX). After filling the mold, both top and bottom surfaces were covered

with glass slides to produce a flat surface. Light was irradiated from the top to bottom surface for 5 s. One of the two hemicylinders was removed by sliding it, and the uncovered lateral surface was exposed to light for 40 s. The remaining hemicylinder was then removed and the uncovered lateral surface was exposed to light in the same manner. The cured specimen was removed from the mold, plugged into the bottom of the 6-mm thick mold. The empty upper space was filled using one of five flowable resins, covered using a thin glass slide, pressed firmly to make a flat surface, light cured for 5 s. The rest processes for light curing of the filled resin were the same as described above. The specimens (n=14) so produced were removed and aged for 24 h in a 37°C dry and dark chamber. After 24 h, compression tests were performed (n=7) on a universal test machine at a crosshead speed of 1 mm/min. CS (σ_c in MPa) of the

specimens was obtained using the following formula

$$\sigma_c = P/A$$

where P is maximum failure load (N) and A is the cross-sectional area of the specimen. CM (in GPa) is defined as the slope of the linear portion of the load-displacement curve. The remaining seven specimens were immersed in the 37°C distilled water for 2 weeks and then the same measurements were performed under the same conditions as stated above.

4. Statistical analysis

The obtained results were analyzed using one-way ANOVA. A post-hoc Tukey test was followed for a multiple-comparison, t-test was performed to find the statistical difference between the control and immersed specimens. All tests were analyzed at $p < 0.05$.

RESULTS

Table 2 shows the flexural properties (strength (FS) and modulus (FM)) of the bulk and layered specimens. In the

Table 2. Flexural properties of the bulk and layered specimens

		Before immersion		After immersion		
		FS (MPa) ¹	FM (GPa) ¹	FS ²	FM ²	
Bulk	TC	117.7±9.2 ^{ab}	12.24±0.44 ^a	107.6±10.1 ^a	8.93±0.85 ^a	
	ZX	164.0±8.8 ^c	15.25±0.21 ^b	93.6±7.8 ^a	11.02±0.56 ^b	
	EQ	167.9±12.4 ^c	10.58±0.38 ^c	167.3±15.0 ^b	9.20±0.28 ^a	
	PF	131.5±9.9 ^b	9.09±0.24 ^d	107.0±6.7 ^a	7.25±0.39 ^c	
	PL	108.0±4.6 ^a	4.70±0.2 ^e	96.9±2.8 ^a	4.48±0.21 ^d	
	R2	122.9±13.0 ^{ab}	6.42±0.42 ^f	113.4±9.4 ^a	5.79±0.56 ^e	
	ZF	167.9±6.1 ^c	8.61±0.28 ^d	114.5±11.9 ^a	5.89±0.11 ^e	
<i>p</i> -value		< 0,001	< 0,001	< 0,001	< 0,001	
		FS ¹	FM ¹	FS ²	FM ²	
Layered	EQ	171.2±10.4 ^{ab}	9.24±0.41 ^{abc}	145.7±21.7 ^{ab}	7.27±0.61 ^{ab}	
	PF	150.5±13.4 ^{bcd}	8.57±0.71 ^{ab}	117.4±8.7 ^{cd}	7.31±0.49 ^{ab}	
	PL	114.9±8.9 ^e	4.81±0.51 ^d	96.4±7.7 ^d	4.44±0.66 ^c	
	R2	136.3±9.93 ^{def}	7.61±0.7 ^{ae}	118.4±16.0 ^{bcd}	6.12±0.38 ^{abd}	
	ZF	163.1±15.8 ^{bcd}	8.76±0.66 ^{ab}	133.4±15.3 ^{ace}	7.28±0.42 ^a	
	EQ	170.1±16.8 ^{ab}	9.97±1.26 ^{bf}	145.3±17.7 ^a	8.92±0.99 ^a	
	PF	149.8±11.4 ^{bcd}	10.14±1.15 ^{bc}	142.1±6.1 ^{ac}	8.91±0.30 ^{bf}	
	ZX	PL	134.1±19.4 ^{ce}	5.76±0.5 ^{de}	112.0±7.6 ^{de}	5.39±0.50 ^{cd}
	R2	146.2±7.9 ^{bcd}	8.02±0.89 ^a	126.7±9.7 ^{ace}	6.23±0.57 ^{ad}	
ZF	196.5±8.7 ^a	10.85±1.00 ^{cf}	131.7±3.1 ^{ace}	8.62±0.59 ^{bf}		
<i>p</i> -value		< 0,001	< 0,001	< 0,001	< 0,001	

* Statistically significant difference for resin product is shown by superscript letters^{a,b,...}. Same letters are not significantly different ($p > 0.05$).

* On t-test, same letter is not significantly different.

bulk state, FS of two overlying composite resins was 117,7 and 164,0 MPa and the other four base flowable resins ranged 108,0-167,9 MPa. FM was 12,24 and 15,25 GPa and ranged 4,70-10,58 GPa, respectively. For the specimens layered with TC and ZX, before immersion, FS ranged 114,9-171,2 MPa and 134,1-196,5 MPa, respectively. Also, FM ranged 4,81-9,24 GPa and 5,76-10,85 GPa, respectively. Among the cases, ZX+ZF showed the highest flexural properties (FS and FM). FS and FM values of the layered specimens have similar trends of the base flowable resins (in the bulk state) regardless of the overlying composite resins. In all cases, decrease of FS and FM after immersion were significant ($p < 0,05$).

Table 3 shows the compressive properties (CS and CM) of the bulk and layered specimens. CS of two overlying

composite resins was 324,8 and 338,4 MPa and the others ranged 259,8-439,8 MPa. CM was 3,62 and 4,54 GPa and ranged 2,67-4,11 GPa, respectively. Specimens layered with TC and ZX showed CS range 251,4-282,4 MPa and 259,7-295,3 MPa, respectively. CM also ranged 2,86-3,98 GPa and 2,59-4,18 GPa, respectively. In most cases, CS values of the layered specimens were much lower than those of the base flowable resins (in the bulk state) regardless of the overlying composite resins. However, CM values of the layered specimens have similar trend to those of the base flowable resins. After immersion, bulk specimens showed insignificant CS and CM change; however, layered specimens showed significant change in CM ($p < 0,05$).

Table 3. Compressive properties of the bulk and layered specimens

		Before immersion		After immersion		
		CS (MPa) ¹	CM (GPa) ¹	CS ¹	CM ¹	
Bulk	TC	324,8±22,3 ^a	3,62±0,39 ^a	279,1±26,7 ^a	3,36±0,11 ^a	
	ZX	338,4±19,4 ^a	4,54±0,05 ^b	347,0±26,4 ^b	3,86±0,15 ^b	
	EQ	439,8±35,2 ^{bc}	4,11±0,14 ^b	456,3±31,0 ^c	3,81±0,14 ^b	
	PF	351,8±25,8 ^{ad}	3,48±0,31 ^{ad}	357,3±22,4 ^b	3,28±0,13 ^a	
	PL	381,0±32,2 ^{ab}	2,67±0,10 ^c	427,3±15,6 ^c	2,86±0,05 ^c	
	R2	259,8±38,9 ^e	3,13±0,15 ^d	287,5±35,8 ^a	2,96±0,21 ^c	
	ZF	361,5±16,9 ^a	3,34±0,23 ^{ad}	332,6±18,9 ^{ab}	3,15±0,16 ^{ac}	
<i>p</i> -value		< 0,001	< 0,001	< 0,001	< 0,001	
		CS ¹	CM ¹	CS ¹	CM ²	
Layered	TC	EQ	259,2±34,0 ^a	3,98±0,10 ^{ab}	277,7±29,0 ^a	3,56±0,25 ^{ab}
		PF	251,4±30,0 ^a	3,34±0,19 ^{cd}	255,6±35,6 ^a	3,31±0,32 ^{bc}
		PL	282,4±34,7 ^a	2,86±0,43 ^{de}	287,1±31,4 ^a	3,10±0,25 ^{bd}
		R2	263,4±26,1 ^a	3,11±0,15 ^{df}	251,1±33,5 ^a	2,76±0,36 ^{de}
		ZF	272,5±23,3 ^a	3,66±0,11 ^{ac}	279,4±31,2 ^a	3,45±0,14 ^b
	ZX	EQ	295,3±40,6 ^a	4,18±0,11 ^a	291,1±39,0 ^a	4,00±0,17 ^a
		PF	259,7±34,9 ^a	3,52±0,09 ^{bcd}	262,9±26,4 ^a	3,32±0,22 ^{bf}
		PL	276,1±24,4 ^a	2,59±0,34 ^e	256,3±19,9 ^a	2,85±0,35 ^{cdf}
		R2	262,3±18,5 ^a	3,66±0,13 ^{ac}	247,3±36,3 ^a	2,97±0,47 ^{cdf}
		ZF	262,8±3,06 ^a	4,01±0,47 ^{ab}	283,6±36,7 ^a	3,56±0,42 ^{ab}
<i>p</i> -value		= 0,50	< 0,001	= 0,05	< 0,001	

DISCUSSION

Sometimes the restored tooth with composite resins requires repair or replacement due to fracture, failure, or repeated wear of the filled composite resins. Regarding cost and treatment strategies, repair can be advantageous than replacement of whole filling. In a deep cavity, for the restoration, flowable resins are frequently used as a base material. Over this base flowable resin, composite resin of better mechanical properties can be placed to warrant high durability against high external stresses.

The flexural properties (FS and FM) are the measure of material's resist against external stress without fracture. Among the specimens, ZX, EQ, and ZF have high FS (164,0-167,9 MPa); TC and the others have low FS (108,0-131,5 MPa) value. High FS did not warrant the same high FM. ZX has both high FS and FM, whereas, EQ and ZF do not have high FM despite their high FS. To make layered specimens, two composite resins and five flowable resins were chosen for the overlying and base materials, respectively. Specimens layered with TC and flowable resins have 114,9-171,2 MPa (for FS) and 4,81-9,24 GPa (for FM). If ZX layered with flowable resins, they have 134,1-196,5 MPa FS and 5,76-10,85 GPa FM. High FS from EQ and ZF and low FS from PL were observed regardless of FS of overlying TC (117,7 MPa) and ZX (164,0 MPa). The same high FM from EQ and ZF and low FM from PL were also observed regardless of overlying product. Apparently low FS and FM from PL are probably due to insufficient (low) polymerization. Flowable resins have high content of TEGDMA to allow flowability. Since TEGDMA has low molecular weight, it allows low viscosity (high flowability), but it also has high degree of conversion and cross-link density compared to other common monomers and low Young's modulus [Sideridou et al., 2003; Rüttermann et al., 2010]. As to the tested combinations, high FS and FM can be obtained regardless of overlying composite resin if

FS and FM of the base flowable resin are high. The obtained FS values from two different layering combinations ranged 114,9-196,5 MPa depending on combination and are greater than 80 MPa, which is minimum requirement for occlusal area by ISO 4049 [ISO 4049, 2000]. The FM values, ranged 4,81-10,85 GPa, are lower than that of dentin, 17-25 GPa. After immersion, ZX+ZF combination showed the highest FS (33,0%) and FM (20,6%) decrease among the combinations. It is probably due to the highest decrease of ZX and ZF (for FS: 31,8%, FM: 31,6%) among the tested specimens in their bulk state. Except ZX+ZF case, the other combinations showed no specific and consistent changes after immersion in conjunction with the change of bulk state.

The compressive test is a valuable way to measure the material's ability to resist sustained heavy loads during mastication [Anusavice, 2003]. The tested overlying composite resins have a similar CS value. However, two base flowable resins (EQ and Z2) have much different CS values (439,8 and 259,8 MPa, respectively). After immersion, the CS of the bulk specimen had differently changed. The CS of TC and ZF had decreased, whereas the others had increased. Increase of CS after immersion would be due to additional polymerization for two weeks. Two different light-curing steps for specimen preparation in our case (two 5 s light curings on the top and bottom surfaces; two 40 s light curings of lateral sides) may leave insufficiently polymerized regions due to insufficient light curing time both on the top and bottom surfaces. So, the initiated polymerization process terminates with much incompletely polymerized region within the bulk structure. Much lower CS values (251,4-295,3 MPa) of the layered specimens compared to CS of each bulk specimen may be attributable to this. The lowest CM from PL base regardless of overlying composite resins may indicate that if CM of the base material is low, CM of the layered specimen will low as well. The obtained CM values of the layered specimens (2,59-4,18 GPa) are much lower than that of dentin (11,0-18,5 GPa), which is acceptable

for the buffering of heavy external loads against underlying dentin [Craig & Peyton, 1958; Watts et al., 1988]. FS and CS had negligibly low correlation between them, whereas FM was high linearly correlated with CM values, so it is partially practical to estimate flexural properties with pre-known compressive properties, or vice versa.

CONCLUSION

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

1. The flexural properties (FS and FM) of the layered specimens depended on the flexural properties of the base flowable resin regardless of the overlying composite resins.
2. CM of the layered specimens showed similar CM trend of the base flowable resins, whereas CS had no such trend between them.

The obtained flexural and compressive properties of the layered system satisfy the ISO requirement and compatible with the underlying dentin for buffering the external heavy loads.

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Flexural and compressive properties of layered specimens formed with flowable and composite resins

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Flowable resins can be used as a base material during the restoration of deep tooth cavity. The purpose of the present study was to evaluate the mechanical properties of the layered specimens which have flowable resin as a base material. For the study, two composite resins and five flowable resins were chosen for the overlying and base materials, respectively. Flexural and compressive properties of each bulk and ten layered specimens were measured. Layered specimens showed high flexural strength (FS) and flexural modulus (FM) if bulk state FS and FM of the base flowable resin are high. However, compressive strength (CS) was not that case. CS of the layered specimen was not high (251.4~295.3 MPa) whether CS of the bulk state is high or not (259.8~439.8 MPa). FM showed high linear correlation with CM. After all mechanical properties of the layered specimens were not consistently influenced by the mechanical properties of the base flowable resins regardless of the overlying composite resins.

Key Words : Resin repair; Composite resin; Flexural property; Compressive property
