대한치과재료학회지 44(2) : 129-139, 2017
ISSN:2384-4434 (Print); 2384-3268 (Online)
Available online at http://www.kadm.org
https://doi.org/10.14815/kjdm.2017.44.2.129

나노복합레진과 저수축 복합레진의 복합 층으로 이룬 시편이 색과 물리적 성질에 미치는 영향

박완기[1], 최안나[1], 손성애[1], 권용훈[2], 강은숙[3], 박정길[1*]

[1] 부산대학교 치의학전문대학원 치과보존학교실
[2] 부산대학교 치의학전문대학원 치과재료학교실
[3] 인제대학교 의과대학 치과학교실

〈Abstract〉

Effect of layer combinations with nanocomposite and low-shrinkage composite resins on their color and mechanical properties

Wan-Ky Park[1], An-na Choi[1], Sung-Ae Son[1], Yong Hoon Kwon[2], Eun-Sook Kang[3], Jeong-Kil Park[1]*

Department of conservative dentistry[1], Department of Dental Material[2],
School of Dentistry, Pusan National University, Yangsan, Korea
Department of Dentistry[3], College of Medicine, Inje University, Haeundae, Busan, Korea

본 연구는 silorane을 기질의 저수축레진과 나노복합레진의 이층 (two-layer) 구조를 갖는 시료의 색과 물리적 성질을 평가하기 위함이다.

실험을 위하여 4종의 나노복합레진(Aelite LS, Grandio, Tetric EvoCeram, Filtek Z350XT)과 1종의 silorane 기질의 복합레진(P90)을 택하고 이들로 층의 두께가 각기 다른 여러 가지 조합의 이층을 만들었다. 색과 굴곡성질 평가를 위해서 나노복합레진과 silorane 레진을 0.5+1.5mm, 1+1mm, 1.5+0.5mm 두께의 조합을 만들었다. 압축성질 평가를 위하여 1.5+4.5mm, 3+3mm, 4.5+1.5mm 두께의 조합을 만들었다. 두께가 각각 2mm (굴곡성질 평가용)와 6mm (압축성질 평가용)인 단층의 시료는 대조군으로 사용하였다. 이들 시료를 이용하여 색, 투명도, 굴곡강도와 굴곡계수 및 압축강도와 압축계수를 측정하였다. 그 결과, 사용된 시료들은 모두 동일한 A3 색조이지만 L^*, a^*, b^*의 색좌표계 값들은 모두 달랐다. 층을 이룬 시료들의 L^*, a^*, b^*값은 위쪽에 위치하는 시료의 두께가 증가할수록 위쪽 레진의 L^*, a^*, b^*값에 가까웠다. 이층을 이룬 시료들의 투명도는 1.92-6.33 이었는데 각층을 이루는 두 시료의 b^*값 차이가 클수록 투명도도 증가하였다($p<0.05$). 이층을 이룬 시료들의 굴곡강도와 굴곡계수는 각각 108.8-134.0 MPa와 10.95-18.39 GPa이었고 압축강도와 압축계수는 각각 108.3-254.9 MPa와 3.13-4.11 GPa로써 단층 상태의 굴곡강도와 굴곡계수 [(123.2-161.0 MPa)와 (11.64-21.58 GPa)] 그리고 압축강도와 압축계수 [(327.4-405.4 MPa)와 (4.02-5.29 GPa)] 보다 모두 낮았다.

주제어: 색상, 적층시편, 물리적 성질, 저수축복합레진, 나노복합레진

Ⅰ. INTRODUCTION

Transformation of a paste-like composite resin into a rigid body is initiated by the activation of a photoinitiator using a blue light through a polymerization process. The free radicals formed by the activated photoinitiator and amine accelerator transform monomers into a polymer network via a chain reaction. This process involves

---

* Correspondence: 박정길 (ORCID ID: 0000-0001-6333-8138)
(50612) 경상남도 양산시 물금읍 금오로 20부산대학교 치의학전문대학원 치과보존학교실
Tel: 055-360-5221, Fax: 055-360-5214
E-mail: Jeongkil@pusan.ac.kr

Received: May. 29, 2017; Revised: Jun. 19, 2017; Accepted: Jun. 19, 2017

reduction of intermolecular distance due to a change from van der Waals to covalent bonding, and thus, macroscopic shrinkage (Ferracane, 1988).

Reduction of intermolecular distance by polymerization shrinkage causes shrinkage stress, cusp deflection, enamel cracks, postoperative sensitivity, restoration fractures, and caries recurrence (Kemp-Scholte & Davidson, 1988; Lai & Johnson, 1993; Davidson & Feilzer, 1997). To overcome these unwanted problems, many trials, such as incremental filling or the use of various curing modes, low shrinkage monomers, or the inclusion of nanofillers, have been attempted (Satsangi et al., 2004; Smith et al., 2004; Kim et al., 2007). Among the various composite resins, silorane-based composite resins have shown lower polymerization shrinkage than other dimethacrylate-based composite resins. As a mixture of siloxane and oxirane molecules, silorane-based Filtek P90 (P90, 3M ESPE, St. Paul, MN, USA) exhibits little water sorption due to the hydrophobic nature of the siloxane component and low shrinkage due to the ring-opening, flattening, and extending of oxirane molecules (Weinmann et al., 2005; Ilie & Hickel, 2006; Lien & Vandewalle, 2010). Many recent studies focused on the low shrinkage characteristic of this material and tested its performance for minimal shrinkage restoration (Lührs et al., 2011; Hamano et al., 2012; Wiegand et al., 2012).

Teeth are subjected continuously to thermal and mechanical stresses during daily function by exposure to food and beverages, and through mastication. For these reasons, restored teeth with a shrinkage gap at the resin-tooth interface can exhibit bond failure (Kemp-Scholte &, Davidson, 1988; Lai & Johnson, 1993; Davidson & Feilzer, 1997). In addition, repair or replacement of a filled material may be required due to wear and degradation (Tyas et al., 2000; Gordan et al., 2009; Moncada et al., 2009; Maneenut et al., 2011; Staxrud & Dahl, 2011). In a situation of resin repair, using the same resin product both on the upper and lower layers are important in terms of consistency. However, in many cases, using the same material used in the original restoration is not simple because the original product may be unknown or no longer be available. Furthermore, if P90 is used as an underlying material for the repair, the interface between overlying and underlying resins may be affected in terms of mechanical properties and longevity of the repaired restoration since P90 is completely different from that to dimethacrylate-based composite resins. In addition, optically the color perception of layered composite restorations is dependent on the thickness of each layer due to translucency of the materials (Friebel et al., 2012). Since composite resins available do not have the same color coordinate values even though they have the same shade denomination, the resultant color of the layered specimens may be affected by the combination of composite resins with different thickness of layers. The purpose of the present study was to evaluate the color and mechanical properties of layered specimens which are composed of P90 as an underlying material and dimethacrylate-based composite resin as an overlying material.

II. MATERIALS AND METHODS

1. Composite resins and light source

For this study, silorane-based P90 (P90) was used as an underlying material. Four nanocomposite resins [Aelite LS (AL), Grandio (GD), Tetric EvoCeram (TC), Filtek Z350XT (Z3)], all of A3 shade, were used as overlaying materials; details are shown in Table 1. A quartz-tungsten-halogen (QTH) light-curing unit (Optilux 501; Kerr, Orange, CA, USA) with a light intensity of 900 mW/cm^2 was used for light curing.

Table 1. Materials used in the study

Code	Composition	Filler type	Filler content vol%/wt%	Manufacturer
AL	Bis-EMA, TEGDMA	glass frit, amorphous silica	74/88	Bisco, Schaumburg, IL, USA
GD	Bis-GMA, TEGDMA, UDMA	Ba-Al-Borosilicate glass filler, SiO_2 nonofillers	71.4/87	VOCO, Cuxhaven, Germany
P90	Silorane	silanized quartz, yttrium fluoride	55/76	3M ESPE, St. Paul, MN, USA
TN	Bis-EMA, TEGDMA, UDMA	Barium glass, YbF_3, SiO_2	57/80.5	Ivoclar Vivadent
Z3	Bis-EMA, Bis-GMA, TEGDMA, UDMA	zirconia/silica, nanofillers	59.5/78.5	3M ESPE, St Paul, MN, USA

P90; Filtek P90; AL: Aelite LS Posterior; GD: Grandio; TN: Tetric N Ceram; Z3: Filtek Z350
Bis-EMA: ethoxylated bisphenol A glycidyl methacrylate; Bis-GMA: bisphenol A glycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate.

2. Evaluation of color

To evaluate color, disc-type specimens (diameter: 8 mm, thickness: 2 mm) were prepared by layering two composite resins with different combinations [nanocomposite resin+P90: 0+2, 0.5+1.5, 1+1, 1.5+0.5, and 2+0 mm]. To make these specimens, ring-type metal molds of different thicknesses were used (inner diameter: 8 mm, thicknesses 0.5, 1, 1.5, and 2 mm). Initially, one metal disc was filled using P90. Both top and bottom surfaces were covered using two thin glass slides to flatten surfaces and the resin was then light cured for 40 s. The cured specimen was then removed from the mold, plugged into the bottom of a 2-mm thick mold, and the upper remaining space was filled using a nanocomposite resin, which was then covered with a thin glass slide, pressed firmly to flatten the surface, and light cured for 40 s. The cured specimen was removed from the mold and stored for 24 h at 37°C in a dry, dark chamber. Colors of specimens (n=7) were measured in reflectance (%R) mode, and CIE$L^*a^*b^*$ color values were evaluated from the obtained %R values. The translucency parameter (TP) was obtained using the formula

$$TP = [(L^*_B - L^*_W)^2 + (a^*_B - a^*_W)^2 + (b^*_B - b^*_W)^2]^{1/2},$$

where subscript B refers to color coordinates obtained using a black background (L^*=2.93, a^*=0.38, and b^*=-0.34) and W refers to those obtained using a white background (L^*=93.26, a^*=-0.61, and b^*=2.09).

3. Evaluation of flexural properties

A three-point bending test was performed to determine flexural properties [flexural strength (FS) and modulus (FM)]. To produce specimens (nanocomposite resin+P90: 25×2×2 mm in the thickness combinations 0+2, 0.5+1.5, 1+1, 1.5+0.5, and 2+0 mm), stainless steel molds with different thicknesses (25×2×1 mm and 25×2×1.5 mm) were filled using P90. Both top and bottom surfaces were then flattened using two thin glass slides, and light cured for 200 s (40 s × 5) by overlapping exposed areas. The cured specimen was then removed from the mold, plugged into the bottom of 2-mm thick mold, and the top surface of the P90 was then thinly treated with bonding agent (Clearfil SE Bond, Kuraray, Tokyo, Japan), which was then light cured for 10 s. The upper remaining space was then filled using a nanocomposite resin, covered using a thin glass slide, pressed firmly, and light cured for 200 s using five overlapping exposures. Making a P90 specimen sized

25×2×0.5 mm was not easy because cured specimens were easily broken during removal from the mold. Thus, a 25×2×1.5 mm nanocomposite specimen was made first, plugged into the bottom of 2-mm thick mold, pasted with bonding agent, and then treated with P90 on the remaining upper space. After light curing, specimens were removed from molds and stored for 24 h in a 37°C dry, dark chamber. Some other specimens were immersed in 37°C distilled water for 2 weeks. After storage (n=7) or immersion (n=7), specimens were loaded into a universal test machine (Instron 3345, Grove City, PA, USA) and tested 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 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 of the linear portion of the load-displacement curve. Through the study non-layered specimen means non-layered 2-mm thick overlying nanocomposite resin or underlying P90.

4. Evaluation of compressive properties

To measure compressive properties [compressive strength (CS) and modulus (CM)], two matching stainless steel hemicylinders (inner diameter: 3 mm, outer diameter: 10 mm, height: 3, 4.5, and 6 mm)

were manufactured to form a cylinder. To produce specimens of diameter 3 mm and height 6 mm (nanocomposite resin+P90) in the combinations 0+6, 1.5+4.5, 3+3, 4.5+1.5, and 6+0 mm, the empty inner space of the hemicylinders (height: 3 and 4.5 mm) was filled using P90. Then it was exposed to light on its top and bottom surfaces for 5 s. One of the two hemicylinders was removed, and the uncovered lateral surface was exposed to light for 40 s. The remaining hemicylinder was then removed and its 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 high mold, the upper surface was thinly pasted with bonding agent, light cured for 10 s, the upper space was filled with nanocomposite resin, covered using a thin glass slide, pressed firmly to make a flat surface, and light cured for 5 s. Light curing of the nanocomposite resin was performed as described above. Making a 1.5-mm thick P90 specimen was not easy because the cured specimens were easily broken during removal from the mold, and thus, specimens were prepared by making the nanocomposite resin at 4.5 mm first, and filling with P90. After light curing, specimens were removed from the mold and stored for 24 h in a 37°C, dry, dark chamber. Other specimens were immersed in 37°C distilled water for 2 weeks. After storage (n=7) or immersion (n=7), the specimens were loaded onto a universal test machine for compression testing at a crosshead speed of 1 mm/min. CS (σc in MPa) values of specimens were obtained using the following formula

$$\sigma_c = P/A$$

where P is maximum failure load (N) and A is specimen cross-sectional area. CM (in GPa) was defined as the slope of the linear portion of the load-displacement curve. Through the study non-layered specimen means non-layered 6-mm thick nanocomposite resin or underlying P90.

Table 2. CIE$L^*a^*b^*$ and translucency parameter (TP) of layered specimens with different combinations

Layers		Thickness	Black background			White background			TP
Over	Under	(Over+Under)	L^*	a^*	b^*	L^*	a^*	b^*	
AL	P90	0 + 2	53.46 ±0.30	0.78 ±0.35	17.43 ±1.14	56.69 ±0.25	3.41 ±0.23	22.12 ±0.70	6.29± 0.55^{a}
		0.5 + 1.5	58.37 ±0.87	0.77 ±0.10	9.49 ±0.28	60.45 ±0.63	2.46 ±0.11	11.78 ±0.33	3.54± 0.37^{b}
		1 + 1	60.77 ±1.16	1.51 ±0.59	11.05 ±1.57	61.80 ±0.99	2.70 ±0.35	12.38 ±1.21	2.06± 0.51^{c}
		1.5 + 0.5	60.71 ±0.59	1.49 ±0.10	11.31 ±0.69	61.63 ±0.23	2.66 ±0.14	12.48 ±0.73	1.92± 0.20^{c}
		2 + 0	61.03 ±0.50	2.09 ±0.12	12.46 ±0.36	61.84 ±0.40	3.43 ±0.17	13.81 ±0.50	2.07± 0.21^{c}
p-value			〈0.001						
GD	P90	0 + 2	53.46 ±0.30	0.78 ±0.35	17.43 ±1.14	56.69 ±0.25	3.41 ±0.23	22.12 ±0.70	6.29± 0.55^{a}
		0.5 + 1.5	54.65 ±1.14	0.27 ±0.07	7.20 ±0.31	57.23 ±1.14	2.04 ±0.10	11.06 ±0.36	4.99± 0.45^{b}
		1 + 1	56.91 ±1.99	-0.25 ±0.22	5.89 ±0.92	59.02 ±1.42	1.52 ±0.45	9.87 ±0.33	4.84± 0.97^{b}
		1.5 + 0.5	54.60 ±0.42	-0.80 ±0.10	4.02 ±0.12	57.65 ±0.44	1.31 ±0.16	9.15 ±0.38	6.33± 0.59^{a}
		2 + 0	53.70 ±0.35	-1.27 ±0.07	4.22 ±0.16	58.30 ±0.35	1.34 ±0.07	10.62 ±0.42	8.30± 0.51^{c}
p-value			〈0.001						
TN	P90	0 + 2	53.46 ±0.30	0.78 ±0.35	17.43 ±1.14	56.69 ±0.25	3.41 ±0.23	22.12 ±0.70	6.29± 0.55^{a}
		0.5 + 1.5	54.18 ±0.53	0.18 ±0.11	10.85 ±0.38	57.50 ±0.61	2.15 ±0.24	14.17 ±0.29	5.09± 0.28^{bc}
		1 + 1	56.69 ±1.95	-0.08 ±0.13	9.73 ±0.34	59.59 ±1.86	1.54 ±0.43	12.79 ±0.47	4.54± 0.82^{b}
		1.5 + 0.5	54.55 ±0.53	-0.21 ±0.06	7.71 ±0.52	58.09 ±0.30	1.92 ±0.12	11.98 ±0.36	5.94± 0.34^{ac}
		2 + 0	54.36 ±0.29	-0.44 ±0.09	8.44 ±0.15	58.96 ±0.41	2.39 ±0.12	14.63 ±0.23	8.22± 0.32^{d}
p-value			〈0.001						
Z3	P90	0 + 2	53.46 ±0.30	0.78 ±0.35	17.43 ±1.14	56.69 ±0.25	3.41 ±0.23	22.12 ±0.70	6.29± 0.55^{a}
		0.5 + 1.5	55.24 ±0.92	0.41 ±0.04	11.62 ±0.65	57.92 ±0.70	2.13 ±0.13	14.89 ±0.74	4.58± 0.31^{b}
		1 + 1	57.28 ±0.80	0.54 ±0.11	11.72 ±0.48	59.17 ±0.54	1.98 ±0.11	14.26 ±0.52	3.48± 0.44^{c}
		1.5 + 0.5	56.44 ±0.64	0.40 ±0.15	10.94 ±0.40	59.01 ±0.40	2.21 ±0.06	14.50 ±0.33	4.75± 0.45^{b}
		2 + 0	55.11 ±0.17	-0.00 ±0.06	11.27 ±0.16	58.77 ±0.21	2.41 ±0.05	16.28 ±0.20	6.66± 0.33^{a}
p-value			〈0.001						

Table 3. Flexural properties of layered specimens with different combinations before and after immersion in distilled water for 2 weeks

Layers		Thickness	Control		Immersed	
Over	Under	(Over+Under)	Strength (FS)	Modulus (FM)	Strength (FS)	Modulus (FM)
AL	P90	0 + 2	134.6 ± 11.3^{a}	12.87 ± 0.88^{a}	121.6 ± 9.2^{a}	11.70 ± 0.80^{a}
		0.5 + 1.5	122.3 ± 12.3^{a}	16.43 ± 0.44^{bc}	113.7 ± 13.5^{a}	15.25 ± 2.88^{bc}
		1 + 1	122.7 ± 21.0^{a}	16.07 ± 0.33^{b}	110.4 ± 8.8^{a}	14.32 ± 1.34^{ab}
		1.5 + 0.5	117.9 ± 4.9^{a}	18.39 ± 1.43^{cd}	107.2 ± 10.9^{a}	17.69 ± 0.31^{c}
		2 + 0	138.2 ± 6.6^{a}	20.47 ± 1.67^{d}	123.7 ± 7.4^{a}	21.80 ± 1.65^{d}
p-value			0.223	〈0.001	0.003	〈0.001
t-test: control vs. immersed			FS: Significantly different		FM: Not significantly different	
GD	P90	0 + 2	134.6 ± 11.3^{ab}	12.87 ± 0.88^{a}	121.6 ± 9.2^{a}	11.70 ± 0.80^{a}
		0.5 + 1.5	110.9 ± 16.2^{a}	15.34 ± 1.15^{b}	109.7 ± 19.0^{a}	13.64 ± 1.04^{b}
		1 + 1	124.5 ± 20.2^{a}	15.77 ± 1.59^{b}	105.2 ± 13.2^{a}	14.31 ± 0.96^{b}
		1.5 + 0.5	134.0 ± 10.8^{a}	17.08 ± 0.96^{b}	106.6 ± 11.6^{a}	16.98 ± 0.83^{c}
		2 + 0	161.0 ± 11.7^{b}	21.58 ± 1.45^{c}	108.2 ± 9.2^{a}	18.74 ± 0.68^{d}
p-value			〈0.001	〈0.001	0.376	〈0.001
t-test: control vs. immersed			FS: Significantly different		FM: Not significantly different	
TN	P90	0 + 2	134.6 ± 11.3^{a}	12.87 ± 0.88^{ab}	121.6 ± 9.2^{a}	11.70 ± 0.80^{a}
		0.5 + 1.5	125.9 ± 18.6^{a}	13.35 ± 0.26^{a}	88.1 ± 14.1^{b}	10.96 ± 0.39^{a}
		1 + 1	124.8 ± 14.3^{a}	12.50 ± 1.95^{ab}	89.8 ± 20.4^{b}	10.99 ± 2.65^{a}
		1.5 + 0.5	108.8 ± 29.1^{a}	10.95 ± 1.7^{b}	90.7 ± 20.3^{b}	9.56 ± 2.24^{a}
		2 + 0	123.2 ± 2.8^{a}	11.64 ± 0.40^{ab}	104.8 ± 5.0^{ab}	9.13 ± 0.44^{a}
p-value			0.223	0.027	0.017	0.148
t-test: control vs. immersed			FS: Significantly different		FM: Significantly different	
Z3	P90	0 + 2	134.6 ± 11.3^{a}	12.87 ± 0.88^{a}	121.6 ± 9.2^{a}	11.70 ± 0.80^{ab}
		0.5 + 1.5	129.4 ± 8.6^{a}	13.65 ± 1.10^{a}	100.6 ± 20.2^{a}	12.07 ± 1.41^{ab}
		1 + 1	128.3 ± 19.7^{a}	13.24 ± 1.29^{a}	104.1 ± 16.5^{a}	10.19 ± 2.07^{a}
		1.5 + 0.5	133.4 ± 6.7^{a}	13.57 ± 1.02^{a}	116.5 ± 12.4^{a}	12.62 ± 0.51^{b}
		2 + 0	153.5 ± 11.9^{a}	14.74 ± 0.86^{a}	110.7 ± 8.8^{a}	12.20 ± 0.82^{ab}
p-value			0.086	0.454	0.166	0.064
t-test: control vs. immersed			FS: Significantly different		FM: Significantly different	

5. Statistical analysis

Test results were analyzed by one-way ANOVA for layering combination. A post-hoc Tukey test was followed for a multiple-comparison. The student's t-test was used to determine the significances of differences between control and immersed specimens. All tests were analyzed at $p〈0.05$.

III. RESULTS

Table 2 shows the CIE$L^*a^*b^*$ color coordinate values for two different backgrounds (black and white) and TP values of the layered specimens. Overlying non-layered specimens (nanocomposite resins) had L^*, a^* and b^* values that differed from those of underlying bulk P90, although all tested products were of the same shade. The L^* values of layered specimens were not very different from that of bulk P90 (54.18-60.77 and 53.46 for layered specimens and P90, respectively.), whereas b^* values differed markedly (4.02-11.72 and 17.43, respectively). The TP values of layered specimens ranged from 1.92 to 6.33. Of the combinations tested, AL+P90 and GD+P90 combinations showed the lowest and highest TP values, respectively.

Table 3 shows the flexural properties of layered specimens before and after immersion. FS values of non-layered nanocomposite resins ranged from 123.2 to 161.0 MPa (before immersion) and from 104.8 to 123.7 MPa after immersion for 2 weeks. On the other hand, FS values of layered specimens ranged from 108.8 to 134.0 MPa before immersion and from 88.1 to 116.5 MPa after immersion for 2 weeks. Control FS values (obtained from non-layered state) decreased approximately 1.1-32.8% after immersion depending on condition. Non-layered GD (2+0 mm) and layered TN (0.5+1.5 mm) showed the greatest FS decrease (32.8% and 30.0%) after immersion.

FM values of non-layered specimens were 11.64-21.58 GPa before immersion and 9.13-21.80 GPa after immersion for 2 weeks. Layered specimens had FM values of 10.95-18.39 GPa before immersion and 9.56-17.69 GPa after immersion for 2 weeks. After immersion, FM values decreased approximately 0.6-23.0% depending on condition. In the case of non-layered AL, FM increased by 6.5%.

The compressive properties of layered specimens before and after immersion are shown in Table 4. CS values of non-layered and layered specimens were 315.3-405.4 MPa and 108.3-254.9 MPa, respectively. After immersion, these values changed to 307.1-412.6 MPa and 132.4-272.1 MPa. In many cases, CS values increased from 0.4 to 52.7% for non-layered and layered specimens. The GD+P90 and TN+P90 combinations showed consistent CS increase as nanocomposite resin thickness increases.

CM values ranged from 3.74 to 5.29 GPa and from 3.13 to 4.11 GPa for non-layered and layered specimens, respectively, and after immersion, these values decreased by 0.8 to 11.6% depending on condition.

Ⅳ. DISCUSSION

Low shrinkage composite resins such as AL and P90 have been reported to result in substantially less polymerization shrinkage than their counterparts. When these materials are used and need to be repaired intraorally, color matching and the mechanical properties of the original and repair composite resins are important in terms of aesthetics and consistency of mechanical properties. The present study investigated how the layering combinations of different composite resins affect the color and mechanical properties of layered specimens when P90 is placed at the bottom of layered structure.

All resin products tested in the present study were of shade A3. However, since manufacturers differed, the resins had different L^*, a^*, and b^* values. Of the resins tested, AL and P90 had the highest (61.0) and lowest (53.5) L^* values, respectively, whereas P90 and GD had the highest (17.4) and lowest (4.2) b^* values, respectively. The color coordinate differences (ΔL^*, Δa^*, and Δb^*) between the tested non-layered specimens (2-mm thick) ranged 0.24-7.57, 0.78-2.05, and 4.97-13.21, respectively. In the case of layered specimens, the highest ΔL^* and Δb^* were found at AL+P90 (7.57) and GD+P90 (13.21), respectively. Since $+b^*$ represents the degree of yellowness, P90 was the least bright and most yellow resin among the tested specimens. Regarding the color coordinates of layered specimens, Z3+P90 and GD+P90 combinations showed the least (5.71-6.49) and greatest (10.23-13.41) b^* differences if compared to that of non-layered P90. These results show that the resultant color coordinates of layered specimens are influenced not by P90, but by those of the overlying

Table 4. Compressive properties of layered specimens with different combinations before and after immersion in distilled water for 2 weeks

Layers		Thickness	Control		Immersed	
Over	Under	(Over+Under)	Strength (CS)	Modulus (CM)	Strength (CS)	Modulus (CM)
AL	P90	0 + 6	315.3 ± 31.4^{a}	3.74 ± 0.31^{ab}	307.1 ± 44.7^{a}	3.68 ± 0.22^{ab}
		1.5 + 4.5	108.3 ± 14.2^{b}	3.54 ± 0.37^{a}	165.4 ± 34.0^{b}	3.44 ± 0.32^{a}
		3 + 3	209.2 ± 19.4^{c}	3.75 ± 0.32^{ab}	207.4 ± 14.0^{b}	3.67 ± 0.36^{ab}
		4.5 + 1.5	189.7 ± 24.3^{c}	4.11 ± 0.26^{b}	170.4 ± 15.7^{b}	3.99 ± 0.36^{b}
		6 + 0	327.4 ± 11.1^{a}	5.29 ± 0.13^{c}	346.1 ± 48.9^{a}	4.68 ± 0.31^{c}
p-value			〈0.001	〈0.001	〈0.001	〈0.001
t-test: control vs. immersed			CS: Not significantly different		CM: Not significantly different	
GD	P90	0 + 6	315.3 ± 31.4^{a}	3.74 ± 0.31^{a}	307.1 ± 44.7^{a}	3.68 ± 0.22^{ab}
		1.5 + 4.5	122.0 ± 13.3^{b}	3.52 ± 0.33^{a}	164.5 ± 27.3^{b}	3.46 ± 0.37^{a}
		3 + 3	203.6 ± 23.9^{c}	3.94 ± 0.12^{a}	215.9 ± 34.1^{bc}	3.80 ± 0.26^{ab}
		4.5 + 1.5	215.7 ± 22.4^{c}	4.00 ± 0.16^{a}	248.1 ± 31.4 ^{ac}	3.96 ± 0.54^{b}
		6 + 0	351.9 ± 28.0^{a}	4.96 ± 0.17^{b}	394.5 ± 41.6^{d}	4.76 ± 0.11^{c}
p-value			〈0.001	〈0.001	〈0.001	〈0.001
t-test: control vs. immersed			CS: Not significantly different		CM: Not significantly different	
TN	P90	0 + 6	315.3 ± 31.4^{a}	3.74 ± 0.31^{ab}	307.1 ± 44.7^{a}	3.68 ± 0.22^{a}
		1.5 + 4.5	126.9 ± 25.3^{b}	3.29 ± 0.24^{a}	158.0 ± 14.0^{b}	2.91 ± 0.39^{b}
		3 + 3	212.8 ± 30.0^{c}	3.41 ± 0.26^{a}	229.5 ± 24.7^{c}	3.34 ± 0.12^{ac}
		4.5 + 1.5	246.1 ± 15.2^{c}	3.58 ± 0.33^{ab}	272.1 ± 37.4^{ac}	3.31 ± 0.06^{ab}
		6 + 0	340.8 ± 29.8^{a}	4.02 ± 0.36^{b}	342.3 ± 39.2^{a}	3.63 ± 0.10^{a}
p-value			〈0.001	=0.010	〈0.001	〈0.001
t-test: control vs. immersed			CS: Not significantly different		CM: Not significantly different	
Z3	P90	0 + 6	315.3 ± 31.4^{a}	3.74 ± 0.31^{a}	307.1 ± 44.7^{a}	3.68 ± 0.22^{a}
		1.5 + 4.5	171.3 ± 21.0^{b}	3.13 ± 0.18^{b}	132.4 ± 9.6^{b}	2.78 ± 0.76^{b}
		3 + 3	236.9 ± 25.5^{c}	3.79 ± 0.13^{a}	208.7 ± 23.1^{c}	3.72 ± 0.10^{ac}
		4.5 + 1.5	254.9 ± 15.7^{c}	3.95 ± 0.46^{a}	241.4 ± 29.6^{ac}	3.92 ± 0.15^{ac}
		6 + 0	405.4 ± 33.1^{d}	4.55 ± 0.17^{c}	412.6 ± 34.2^{d}	4.43 ± 0.17^{c}
p-value			〈0.001	〈0.001	〈0.001	〈0.001
t-test: control vs. immersed			CS: Not significantly different		CM: Not significantly different	

product. So, as the overlying product thickened, the resultant color coordinates closed to those of the overlying product. Accordingly, the color of overlying product is more important than that of underlying P90 in terms of color matching with neighboring teeth. TP values of layered specimens ranged from 1.92 to 6.33. With the exception of AL+P90, layered specimens had high TP values (3.48-6.33) due to high TP values of the corresponding overlying products in their non-layered state (6.66-8.30 for Z3, TN, and GD). The highest TP value (6.33) was obtained for the GD+P90 combination, 1.5+0.5 mm, due to it's the greatest Δb^* for black and white backgrounds.

Flexural properties of specimens are related to the ability of a material to resist external stresses without fracture. The FS values of non-layered nanocomposite resins were lower

(TN: 123.2 MPa), similar (AL: 138.2 MPa), or higher (GD: 161.0 MPa and Z3: 153.5 MPa) than P90 (134.6 MPa). However, all layered specimens had a lower FS value than P90 regardless of layering combination or whether the bulk nanocomposite resin had a higher FS, although it should be added that FS differences were not significant ($p > 0.05$). In the case of FM, the values of layered specimens were greater than that of underlying P90 when the FM value of non-layered state was greater than that of underlying P90. Flexural properties were also evaluated after immersion in distilled water for 2 weeks. FS values were found to decrease significantly, but for FM, only the TN+P90 and Z3+P90 combinations showed significant decreases. FS and FM values obtained from layered specimens ranged from 88 to 116 MPa and from 11 to 18 GPa, respectively. These values seem to satisfy ISO 4049 requirement for occlusal areas (>80 MPa) and compatible with the FM of dentin (17-25 GPa), respectively (Xu et al., 1998; ISO 4049, 2000; Mahoney et al., 2000; Kinney et al., 2003).

The compressive properties are related with the material's ability to resist sustained heavy loads along the longitudinal direction during mastication (Anusavice, 2003). The CS of layered specimens was significantly lower (28-67%) than that of the corresponding overlying specimens. If the CS of the overlying products is greater than that of underlying P90, the CS of layered specimens increases as the overlying product thickens, even though the CS value (108-255 MPa) of layered specimens is much lower than that of the corresponding non-layered specimens (315-405 MPa). In the case of CM, even though the CM value of layered specimens (3.13-4.11 GPa) is lower than that of the corresponding non-layered specimens (3.74-5.29 GPa), a linear CM increase was found as thickness of the overlying layer increased if the CM of the overlying non-layered specimen is greater than that of underlying P90. In any cases, the immersion situation did not induce any significant changes of the compressive properties (CS and CM) and the evaluated CM values were much lower than that of dentin (11.0-18.5 GPa) (Craig & Peyton, 1958; Watts et al., 1987).

V. CONCLUSIONS

For various layered specimens using nanocomposite resins and P90 (low shrinkage composite resin) to form overlying and underlying structures with different thickness combinations, respectively, color, TP, flexural and compressive properties of layered specimens were affected by the overlying nanocomposite resins. However, the layered specimens had less translucent and lower mechanical properties than those of P90 regardless of the mechanical properties of overlying products.

VI. REFERENCES

Anusavice KJ (2003). Phillips' science of dental materials. 11th edition. Philadelphia: W.B. Saunders Co.; 2003.

Craig RG, Peyton FA (1958). Elastic and mechanical properties of human dentin. *J Dent Res* 37: 710-718.

Davidson CL, Feilzer AJ (1997). Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives. *J Dent* 25: 435-440.

Ferracane JL. Current trends in dental composites (1995). *Crit Rev Oral Biol Med* 6: 302-318.

Friebel M, Pernell O, Cappius HJ, Helfmann J, Meinke MC (2012). Simulation of color perception of layered dental composites using optical properties to evaluate the benefit of esthetic layer preparation technique. *Dent Mater* 28: 424-432.

Gordan VV, Garvan CW, Blaser PK, Mondragon E, Mjör IA (2009). A long-term evaluation of alternative treatments to replacement of resin-based composite restorations:

results of a seven-year study. *J Am Dent Assoc* 140: 1476-1484.

Hamano N, Chiang YC, Nyamaa I, Yamaguchi H, Ino S, Hickel R, Kunzelmann KH (2012). Repair of silorane-based dental composites: influence of surface treatments. *Dent Mater* 28: 894-902.

Ilie N, Hickel R (2006). Silorane-based Dental Composite: Behavior and Abilities. *Dent Mater* 25: 445-454.

ISO 4049 (2000): Dentistry-polymer-based filling, restorative and luting materials. International Organization for Standardization Geneva, Switzland, 2000.

Kemp-Scholte CM, Davidson CL (1988). Marginal sealing of curing contraction gaps in class V composite resin restorations. *J Dent Res* 67: 841-845.

Kim JJ, Moon HJ, Lim BS, Lee YK, Rhee SH, Yang HC (2007). The effect of nanofiller on the opacity of experimental composites. *J Biomed Mater Res B:* Appl Biomater 80: 332-338.

Kinney JH, Marshall SJ, Marshall GW (2003). The mechanical properties of human dentin: a critical review and reevaluation of the dental literature. *Crit Rev Oral Biol Med* 14: 13-29.

Lai JH, Johnson AE (1993). Measuring polymerization shrinkage of photo-activated restorative materials by a water-filled dilatometer. *Dent Mater* 9: 139-143.

Lien W, Vandewalle KS (2010). Physical properties of a new silorane-based restorative system. *Dent Mater* 26: 337-344.

Lührs AK, Görmann B, Jacker-Guhr S, Geurtsen W (2011). Repairability of dental siloranes in vitro. *Dent Mater* 27: 144-149.

Mahoney E, Holt A, Swain M, Kilpatrick N (2000). The hardness and modulus of elasticity of primary molar teeth: an ultramicro-indentation study. *J Dent* 28: 589-594.

Maneenut C, Sakoolnamarka R, Tyas MJ (2011). The repair potential of resin composite materials. *Dent Mater* 27: e20-e27.

Moncada G, Martin J, Fernandez E, Hempel MC, Mjor IA, Gordan VV (2009). Sealing, refurbishment and repair of class I and class II defective restorations: a three-year clinical trial. *J Am Dent Assoc* 140: 425-432.

Satsangi N, Rawls HR, Norling BK (2004). Synthesis of low-shrinkage polymerizable liquid-crystal monomers. *J Biomed Mater Res B Appl Biomater* 71: 153-158.

Smith RE, Pinzino CS, Chappelow CC, Holder AJ, Kostoryz EL, Guthrie JR, Miller M, Youtee DM, Eick JD (2004). Photopolymerization of an expanding monomer with an aromatic dioxirane. *J Appl Polym Sci* 92: 62-71.

Staxrud F, Dahl JE (2011). Role of bonding agents in the repair of composite resin restorations. Eur *J Oral Sci* 119: 316-322.

Tyas MJ, Anusavice KJ, Frencken JE, Mount GJ (2000). Minimal intervention dentistry - a review. *Int Dent J* 50: 1-12.

Watts DC, el Mowafy OM, Grant AA (1987). Temperature dependence of compressive properties of human dentin. *J Dent Res* 66: 29-32.

Weinmann W, Thalacker C, Guggenberger R (2005). Siloranes in dental composites. *Dent Mater* 21: 68-74.

Wiegand A, Stawarczyk B, Buchalla W, Tauböck TT, Özcan M, Attin T (2012). Repair of silorane composite--using the same substrate or a methacrylate-based composite? *Dent Mater* 28: e19-e25.

Xu HH, Smith DT, Jahanmir S, Romberg E, Kelly JR, Thompson VP, Rekow ED (1998). Indentation damage and mechanical properties of human enamel and dentin. *J Dent Res* 77: 472-480.

ABSTRACT

Effect of layer combinations with nanocomposite and low-shrinkage composite resins on their color and mechanical properties

Wan-Ky Park[1], An-na Choi[1], Sung-Ae Son[1], Yong Hoon Kwon[2], Eun-Sook Kang[3], Jeong-Kil Park[1]

Department of conservative dentistry[1], Department of Dental Material[2],
School of Dentistry, Pusan National University, Yangsan, Korea
Department of Dentistry[3], College of Medicine, Inje University, Haeundae, Busan, Korea

This study investigated the colors and mechanical properties of layered dental composites. Four nanocomposite resins (Aelite LS, Grandio, Tetric EvoCeram, Filtek Z350XT) and a silorane-based composite resin (P90) were used for overlying and underlying materials, respectively, with different thickness combinations. Colors, translucency parameter (TP), flexural and compressive properties were evaluated. All tested specimens had different color coordinates, although all were of A3 shade. Color coordinates and TP values of layered specimens better matched those of the corresponding overlying product as the thickness of the overlying product was increased. High TP values were related with high b* value differences between specimens ($p<0.05$). Both flexural strength and modulus, compressive strength and modulus of layered specimens with different thickness combinations were mostly lower than those of the corresponding overlying products, respectively, in their non-layered state.

Key Words: Color, Layered specimens, Mechanical properties, low-shrinkage composite resin, Nanocomposite resins