Microhardness and microleakage of composite resin according to the change of curing light intensity

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국무초록

다양한 중합광도에 따른 복합레진의 미세 경도와 변연 누출도

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심미성 수복재 중합시 사용되는 광원은 다양한 요인들에 의해 선택되어지고 있지만 이러한 요인들은 앞으로도 논란의 여지를 많이 남겨두고 있다. 또한 현재 사용되고 있는 중합법들이 제각기 독특한 장점들을 갖고 있기 때문에 최적의 중합법 결정이 필요한 시점이다.

이에 본 연구에서는 중합광의 강도(50, 100, 200, 300, 400, 600mW/cm²)와 중합시간(10, 20, 40초)을 다양하게 변화시켰을 때 복합레진의 미세경도와 미세누출도가 어떠한 양태를 보이는지를 관찰하고자 하였다. 본 실험에서는 A3 색상의 혼합형 복합레진인 국산 DenFil과 미세입자형 복합레진인 Esthet X를 사용하였다.

중합 1일 후 복합레진의 Vickers 경도는 다이아몬드형 압흔 길이를 측정하여 평가하였으며 미세누출도는 1% methylene blue 용액을 이용한 방법과 주사전자 현미경을 이용한 수복물과 치질간의 최대 이개도 평가법의 두 가지 방법으로 측정하였다.

본 연구의 결과는 다음과 같다;

- 1. 미세경도는 시편 상면이 하면에 비해 단단한 양태를 보였으며 상, 하면에 관계없이 혼합형 복합레진인 국산 DenFil이 미세입자형 복합레진인 Esthet X에 비해 높은 경도치를 보였다.
- 2. 모든 미세 경도 실험에서 너무 낮은 광도(50mW/cm², 100mW/cm²)로 중합한 군에서는 대조군에 비해 유의하게 낮은 미세 경도를 보였다(p(0.05).
- 3. 상면의 경우, DenFil은 중합시간과 관계없이 200mW/cm^2 이상의 광도에서 대조군(600mW/cm^2 광도로 40 초간 중합)과 유의차 없는 미세경도치를 보였으나, Esthet X는 200mW/cm^2 이상의 광도로 40 초 동안 중합한 군에서 만 대조군과 유의차 없는 미세경도치를 보였다.
- 4. 하면의 경우, DenFil은 300mW/cm² 이상의 광도로, Esthet X는 200mW/cm² 이상의 광도로 각기 40초 동안 중합한 군에서만 대조군과 유의차 없는 미세경도치를 보였다.
- 5. 법랑질 변연부에서는 색소 침투가 전혀 없었으나 상아질 변연부에서는 정도의 차이는 있었지만 모든 시편에서 색소 침투를 보였다.
- 6. 통계적 유의차는 없었지만 저광도에서 우수한 미세누출도를 보였으며 색소 침투법과 최대 이개도 평가법간에는 상 관성이 매우 낮았다 (p=0.801).

주요어: 광도, 미세경도, 변연누출도

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I. Introduction

Use of composite resin has been increased steadily for the purpose of restoring lost tooth strength and esthetics. Visible light-cured materials have come to dominate the market because of the convenience by their command set nature. Many of the advantages of these composites are dependent on the adequate polymerization.

The polymerization shrinkage of a composite resin, however, occurs when the monomer of the composite polymerizes. This shrinkage is still a major problem of producing marginal gaps around composite restorations and may also contribute to postoperative pain, tooth fracture, and secondary caries.

An optimal degree of conversion and minimal polymerization shrinkage are generally antagonistic, as increased monomer conversion invariably leads to elevated polymerization shrinkage values. However, they are indispensable for an optimal resin composite restoration. A number of approaches^{1,2)} have been used to reduce the stress on the restoration-cavity wall interface, such as dentin bonding agents, stressabsorbing liners, three-sited light-curing technique, incremental filling technique, low-intensity curing lights, and so on.

The clinical performance of light curing composite resin is largely influenced by the quality of the curing unit used and the source of the visible light, especially the wavelength and the intensity of the light source. Curing lights have been developed with varying intensities and curing cycles to speed up curing and reduce marginal gaps in composite restorations³⁾.

Polymerization with high-intensity light has been related to increased depth of cure and improved mechanical properties. However, this type of curing has also been associated with greater polymerization shrinkage⁴⁾. Literature^{5,6)} has shown that slowing down the curing rate of light-initiated resin composites by lowering the light intensity of the light source improved the integrity of the adhesive composite-cavity interface as a result of a more gradual contraction stress development.

Actually, the selection of a curing light is a multifactorial decision that will undoubtedly provide thought-provoking debate in the near future. While each method of polymerization presents unique clinical benefits, the optimal light-curing technique remains to be determined⁷¹.

The purpose of this study was to verify whether there might be any differences of microhardness and microleakage when the curing condition was varied with the light intensity of 50, 100, 200, 300, 400, 600mW/cm² and curing time for 10, 20, 40 seconds. A group cured for 40 seconds with the light intensity of 600mW/cm² was considered as a control group.

I Material & Methods

1. Specimen Preparation

1) Microhardness

Submicron-microfill type composite resin of Esthet X (Dentsply, Germany) and hybrid type of DenFil (VERICOM, Korea) were used, same color of A3 was used.

Composite resin was filled in a mold with cylindrical cavity (diameter; 8mm, thickness; 2mm) which was placed on the glass slide, after that, the upper surface was pressed with a mylar strip and the glass slide. Upper surface was irradiated with intensities of 50, 100, 200, 300, 400, 600mW/cm² each using an experimental light curing unit. Curing times were 10, 20, 40 seconds with each light intensity. Total 360 resin specimens were made.

After curing, every specimen was stored in a saline contained light-proof container for 24 hours. After then, diamond-shaped indentations (Schimadzu microhardness tester type-M (Schimadzu, Japan)) were made with a load of 25 gf for 15 seconds at randomly selected 3 sites each in the upper and lower surfaces (Fig. 1). Vickers hardness was calculated from this length of indentation using the following equation.

 $Hv = 1854.4 P/d^2$

Hv: Vickers hardness P: Testing load (gf)

d: Length of indent (μm)

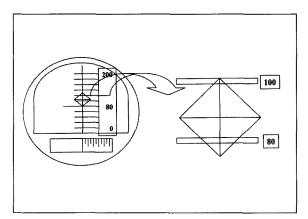


Fig. 1. Schematic drawing of checking dimension of indent

2) Microleakage

Box-shaped class 5 cavity (mesiodistal width: 3 mm, occlusogingival width: 2mm, depth: 1.5mm) was prepared on the buccal and lingual surfaces of molar teeth. The center of cavity was located on the cementoenamel junction.

After treatment of cavity walls according to the manufacturer's instructions, Esthet X was bulk filled and cured under the following conditions: light intensity of 200, 300, 400, 600mW/cm² for 40 seconds and 600mW/cm² for 20 seconds. These conditions were chosen from the result of microhardness test because of no statistical difference in microhardness values of lower surfaces between groups. After storage in a saline contained light-proof container for 1 day, finishing and polishing of the restoration was made with a Sof-Lex disk (3M, U.S.A.). Ten restorations were made in each group. Teeth were thermocycled from 5°C to 55°C for 1000 cycles with a dwell time of 30 seconds at each temperature.

For obtaining an undamaged image, resin replica was made with a polyurethane die material (MOD-RALIT®-3K, DREVE-DENTAMID-GMBH, Germany) from a vinyl polysiloxane impression material (Aquasil™, Dentsply, Germany). Each die was trimmed until a little piece of specimen around gingival margin was remained. Specimen was coated in a vacuum evaporator with a thin film of gold. Observation was done under the scanning electron microscope (JSM-5200, JEOL Ltd, Tokyo, Japan) at an operating magnification of x1000. The maximum

gap width between restoration and cavity wall was measured.

For evaluation of degree of dye penetration, surrounding tooth surface was covered with a nail varnish 2 times except areas 1 mm far from the margin. Afterwards, tooth was immersed in 1% methylene blue solution for 24 hours. After washing and drying, the superficial layer of dye was removed. Tooth was then sectioned from the buccal or lingual surface in a vertical plane parallel to the mesiodistal axis of the tooth. Degree of dye penetration was scored under a binocular microscope (SZ40, Olympus, Japan) and was rated according to the following scores: 0 = no dye penetration: 1 = dye penetration to 1/2 of the occlusal or gingival wall: 2 = dye penetration to the full length of the occlusal or gingival wall: 3 = dye penetration including axial wall.

3) Statistical analysis

Data of microhardness were analyzed using one-way ANOVA, and multiple comparisons were done with a Scheffe test in 95% significance level. Statistical difference of dye penetration between groups was verified using Kruskal-Wallis Spearman's rho test was used to correlate dye penetration test and maximum gap width test.

II. Results

1) Microhardness

Table 1 shows microhardness values of all groups. Generally, hybrid-type composite resin, DenFil showed harder surface than that of submicron microfill-type composite resin, Esthet X irrespective of the location of the surface. Upper surface was generally harder than lower surface.

Curing time and light intensity were positively correlated with the increase of microhardness value, that is, the longer curing time and the stronger light intensity were, the harder surface could be obtained.

In the upper surface of DenFil, we could get 9 subsets of hardness values (Table 2). There was no difference if cured with an intensity of over 200 mW/cm² irrespective of the length of curing time. But in a case of Esthet X, this could be found in a condition of curing for 40 seconds only (Table 3).

Table 1. Microhardness values of test groups (Mean (S.D.)) (kg/mm²)

Intensity	Curing time	Uppe	r surface	Lower s	urface
(mW/cm ²)	(seconds)	DenFil	Esthet X	DenFil	Esthet X
	10	53.60 (13.62)	24.49 (6.41)	5.13 (2.81)	1.97 (0.61)
50	20	66.89 (10.29)	36.18 (7.02)	19.74 (4.81)	5.65 (3.33)
	40	82.61 (6.17)	45.30 (4.78)	36.41 (5.42)	13.71 (3.61)
	10	73.55 (8.58)	41.46 (4.40)	24.13 (6.52)	9.92 (5.45)
100	20	76.53 (11.80)	52.31 (5.65)	29.75 (7.19)	22.61 (4.50)
	40	83.28 (5.08)	56.88 (3.92)	61.54 (4.87)	36.01 (4.15)
	10	90.08 (3.97)	57.60 (5.60)	42.00 (3.99)	35.90 (2.35)
200	20	100.13 (4.78)	66.08 (5.64)	66.47 (3.53)	44.60 (3.17)
	40	99.39 (5.68)	73.95 (2.03)	79.28 (4.87)	61.17 (2.70)
	10	84.00 (5.02)	63.27 (2.97)	68.86 (4.34)	45.81 (3.13)
300	20	93.80 (4.88)	69.77 (5.08)	83.54 (4.38)	50.52 (2.11)
	40	99.22 (3.37)	76.37 (5.05)	91.16 (6.08)	66.89 (4.07)
	10	93.99 (6.64)	70.13 (3.05)	68.71 (8.56)	50.16 (3.56)
400	20	93.27 (4.06)	67.39 (4.38)	83.16 (11.81)	51.44 (7.61)
	40	106.66 (3.58)	77.63 (6.06)	97.31 (4.25)	64.86 (6.73)
	10	99.90 (6.82)	67.33 (8.89)	82.91 (3.21)	53.68 (8.67)
600	20	95.81 (7.18)	78.28 (3.82)	86.49 (7.14)	64.79 (5.76)
	40	99.06 (6.74)	83.19 (9.71)	94.60 (4.72)	65.82 (9.48)

Table 2. Homogeneous subsets of the upper surface of DenFil

Group				Subset	for alpha =	0.05		
(Intensity-Curing time)	N							
(mW/cm ² -seconds)		1	2	3	4	5	6	7
50~10	10	53.60						
50~20	10	66.89	66.89					
100~10	10		73.55	73.55				
100~20	10		76.53	76.53	76.53			
50~40	10		82.61	82.61	82.61	82.61		
100~40	10		83.28	83.28	83.28	83.28	83.28	
300~10	10			84.00	84.00	84.00	84.00	
200~10	10			90.08	90.08	90.08	90.08	90.08
400~20	10				93.27	93.27	93.27	93.27
300~20	10					93.80	93.80	93.80
400~10	10					93.99	93.99	93.99
600~20	10					95.81	95.81	95.81
600~10	10					99.06	99.06	99.06
300~40	10					99.22	99.22	99.22
200~40	10					99.39	99.39	99.39
600~40	10						99.90	99.90
200~20	10						100.13	100.13
400~40	10							106.66
Significance		0.440	0.083	0.075	0.065	0.063	0.060	0.07

 $\textbf{Table 3.} \ \ \text{Homogeneous subsets of the upper surface of Esthet } X$

Group					Subset	for alpha	= 0.05			
(Intensity-Curing time)	N						,			
(mW/cm ² -seconds)		1	2	3	4	5	6	7	8	9
50~10	10	24.49								
50~20	10	36.18	36.18							
100~10	10		41.46	41.46						
50~40	10		45.30	45.30	45.30					
100~20	10			52.31	52.31	52.31				
100~40	10				56.88	56.88	56.88			
200~10	10				57.60	57.60	57.60			
300~10	10					63.27	63.27	63.27		
200~20	10						66.08	66.08	66.08	
600~10	10						67.33	67.33	67.33	
400~20	10						67.39	67.39	67.39	
300~20	10						69.77	69.77	69.77	
400~10	10						70.13	70.13	70.13	70.13
200~40	10							73.95	73.95	73.95
300~40	10							76.37	76.37	76.37
400~40	10								77.63	77.63
600~20	10								78.28	78.28
600~40	10									83.19
Significance		0.205	0.708	0.347	0.129	0.326	0.056	0.064	0.140	0.066

Table 4. Homogeneous subsets of the lower surface of DenFil

Group				-	Subset	for alpha	= 0.05			
(Intensity-Curing time) (mW/cm²-seconds)	N	1	2	3	4	5	6	7	8	9
50~10	10	5.13								
50~20	10		19.74				•			
100~10	10		24.13	24.13						
100~20	10		29.75	29.75	29.75					
50~40	10			36.41	36.41					
200~10	10				42.00					
100~40	10					61.54				
200~20	10					66.47	66.47			
400~10	10					68.71	68.71			
300~10	10					68.86	68.86			
200~40	10						79.28	79.28		
600~10	10							82.91	82.91	
400~20	10							83.16	83.16	
300~20	10							83.54	83.54	83.54
600~20	10							86.49	86.49	86.49
300~40	10							91.16	91.16	91.16
600~40	10								94.60	94.60
400~40	10									97.31
Significance		1.000	0.627	0.206	0.209	0.968	0.141	0.265	0.296	0.064

In the lower surface, DenFil should be polymerized for 40 seconds with an intensity of over 300mW/cm² or for 20 seconds with an intensity of 300mW/cm² and 600mW/cm² to get the same microhardness value as control group cured from 600mW/cm² for 40

seconds (Table 4). Esthet X also needs a polymerization time for 40 seconds when using the intensity of over 200mW/cm² or a condition of an intensity of 600 mW/cm² for 20 seconds should be kept (Table 5).

Groups cured with very low light inten sities of 50

Table 5. Homogeneous subsets of the lower surface of Esthet X

Group				Subset	t for alpha =	0.05		
(Intensity-Curing time)	N					per		
(mW/cm ² -seconds)		1	2	3	4	5	6	7
50~10	10	1.97						
50~20	10	5.65						
100~10	10	9.92						
50~40	10	13.71	13.71					
100~20	10		22.61					
200~10	10			35.90				
100~40	10			36.01				
200~20	10			44.60	44.60			
300~10	10			45.81	45.81			
400~10	10				50.16	50.16		
300~20	10				50.52	50.52		
400~20	10				51.44	51.44		
600~10	10				53.68	53.68	53.68	
200~40	10					61.17	61.17	61.17
600~20	10						64.79	64.79
400~40	10						64.86	64.86
600~40	10							65.82
300~40	10							66.89
Significance		0.072	0.559	0.329	0.518	0.144	0.123	0.98

Table 6. Degree of dye penetration in all groups

Intensity (time)	Margin	N	Mean (S.D.)
200 (40)	Occlusal	10	0.00 (0.00)
200 (40)	Gingival	10	2.70 (0.48)
200 (40)	Occlusal	10	0.00 (0.00)
300 (40)	Gingival	10	2.50 (0.53)
400 (40)	Occlusal	10	0.00 (0.00)
400 (40)	Gingival	10	2.90 (0.32)
(00 (00)	Occlusal	10	0.00 (0.00)
600 (20)	Gingival	10	2.90 (0.32)
(00 (40)	Occlusal	10	0.00 (0.00)
600 (40)	Gingival	10	2.70 (0.67)



Fig. 2. Methylene blue dye penetration through dentinal margin

Table 7. Ranks of dve penetration

Intensity (time)	Margin	N	Mean Rank
200 (40)	Gingival	10	24.15
300 (40)	Gingival	10	19.25
400 (40)	Gingival	10	29.05
600 (20)	Gingival	10	29.05
600 (40)	Gingival	10	26.00

(Kruskal-Wallis test)

Table 9. Maximum gap of all groups (μm)

Intensity (time)	Margin	N	Mean (S.D.)
		- 14	
200 (40)	Gingival	10	16.88 (7.80)
300 (40)	Gingival	10	22.49 (11.83)
400 (40)	Gingival	10	23.79 (7.72)
600 (20)	Gingival	10	26.42 (13.66)
600 (40)	Gingival	10	16.90 (8.72)

Table 10. Homogeneous subset

T-+:	N	Subset for alpha $= .08$		
Intensity (time)	1/1	1		
200 (40)	10	16.88		
600 (40)	10	16.90		
300 (40)	10	22.49		
400 (40)	10	23.79		
600 (20)	10	26.42		
Sig.		.374		

and 100mW/cm^2 showed significantly lower microhardness values(p(0.05) irrespective of the location of the surface and the material.

2) Microleakage

There was no dye penetration in enamel margin, but all dentin margins were dye penetrated. We could see an obvious problem of microleakage when the cavity, which has enamel and dentin margins simultaneously, was bulk filled (Table 6, Fig. 2).

Even if there was no significant difference between

Table 8. Statistics of K-W test

	dye penetration
Chi-Square	5.670
df	4
Asymp. sig.	.225

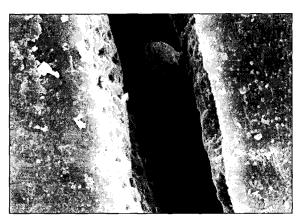


Fig. 3. SEM image revealing maximum gap: Traces of tubule orifices could be seen

Table 11. Correlation between SEM and dye penetration

		Dye	SEM
Spearman's rho Dye	Correlation	1.000	036
	Coefficient		.801
	Sig. (2-tailed)	50	50
	N		

groups, polymerization with a lower light intensity might result in a less microleakage than control group (Table 7, 8).

Table 9 shows the amount of maximum gap around gingival margin from SEM examination (Fig. 3). ANOVA test revealed that there was no difference between groups, but the smallest gap was found in a group polymerized with an intensity of 200mW/cm² for 40 seconds (Table 10).

Spearman's rho test showed that the correlation between the results of dye penetration and SEM examination was very low (Table 11).

IV. Discussion

The use of light curing units has increased tremendously over the past few years with the introduction of photoactivated composite restorative resins. The advantages of light-cured composites are well documented especially the ease of placement of restorations as a result of extended working time and control of setting. Many of the advantages of these composites are dependent on the adequate polymerization and therefore the source of the visible light, especially the wavelength and the intensity of the light source.

With the introduction of photosensitive (light-cured) restorative materials in dentistry, various methods were suggested to enhance the polymerization of these materials including layering⁸⁾, use of light transmitting wedge²⁾, and the use of more powerful light-curing devices⁹⁾.

Actually, the selection of a curing light is a multi-factorial decision that will undoubtedly provide thought-provoking debate in the near future. While each method of polymerization presents unique clinical benefits, the optimal light-curing technique remains to be determined.

The rate of polymerization of a resin-based composite known to be accompanied by high contraction stress was examined by its surface hardness⁵⁾. It was reported that the degree of polymerization of esthetic restorative materials depends on the light intensity and the curing time. The value of microhardness was increased with the increase of curing time, but there was no difference if cured with an intensity of over 200~300mW/cm² according to restorative materials in this study. This was similar to the result of Rueggeberg¹⁰⁾ and Bayne¹¹⁾. They reported respectively at least 280 and 300mW/cm² was needed to correctly polymerize 2mm thickness of universal color of composite resin. Furthermore, lower light intensities of 200~300mW/cm² could get similar microhardness with control group cured from 600mW/cm² for 40 seconds in this study. This result was very unique and needed further study. In a study of Pires et al¹²⁾, it was reported that the microhardness of the lower surface decreased significantly if the thickness was increased. We could see the same result in this

study.

It is often assessed by penetration methods using radioisotopes, dyes or silver nitrate as the diffusing species. Penetration tests are very useful in characterizing the extent and location of microleakage around a restortation but suffer from the disadvantage that specimens must be sectioned to evaluate leakage.

Lutz et al¹³⁾ recommended the use of light reflecting wedge in class I restoration to get the advantage of reducing marginal gap by reflecting curing light. However, Losche²⁾ revealed this effect was not from light-reflecting, but from the reduced light intensity. Another study⁵⁾ showed curing with a lower intensity could get better microleakage than conventional method. Conflict result was made by Friedle et al14. They reported starting curing with a lower intensity did not give any effect on marginal adaptation. In this study, no dye penetration was done on the enamel margin, but penetration was made on the all the specimens of dentinal margin. We could see an obvious problem of microleakage when the cavity, which has enamel and dentin margins simultaneously, was bulk filled from this result. We have to use incremental technique to prevent this phenomenon. Even if there was no significant difference between groups, polymerization with a lower light intensity might result in a less microleakage than control group. Results from SEM examination was similar.

It is a little strange there was no correlation between the degree of dye penetration and the maximum gap. However, it could be understandable if we think there may be cluster areas or foci of adhesion that hold the restorative material in place, yet allow microleakage around these areas¹⁵⁾.

With the spread of light polymerization in dentistry for curing light-activated filling and luting materials, the visible-light curing unit has become a standard device in the dental office. It was reported the light intensity is the main factor for the degree of polymerization. There should be more studies about curing light intensity in order to further develop lots of material properties and marginal adaptation.

V. Conclusion

Within the limit of this study, it could be concluded curing technique using a lower light intensity, such as, $200\sim300\text{mW/cm}^2$ might give similar or better result in microhardness and microleakage than currently used maximum intensity technique did.

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