Polymerization of dual cured composites by different thickness

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ABSTRACT

The purpose of this study was to evaluate the effect of thickness, filling methods and curing methods on the polymerization of dual cured core materials by means of microhardness test.

Two dual cured core materials, MultiCore Flow (Ivoclar Vivadent AG, Schaan, Liechtenstein) and Bis-Core (Bisco Inc., Schaumburg, IL, USA) were used in this study. 2 ㎜ (bulky filled), 4 ㎜ (bulky filled), 6 ㎜ (bulky and incrementally filled) and 8 ㎜ (bulky and incrementally filled)-thickness specimens were prepared with light cure or self cure mode. After storage at 37℃ for 24 hours, the Knoop hardness values (KHN) of top and bottom surfaces were measured and the microhardness ratio of top and bottom surfaces was calculated. The data were analyzed using one-way ANOVA and Scheffe multiple comparison test, with $\alpha = 0.05$.

The effect of thickness on the polymerization of dual cured composites showed material specific results. In 2, 4 and 6 ㎜ groups, the KHN of two materials were not affected by thickness. However, in 8 ㎜ group of MultiCore Flow, the KHN of the bottom surface was lower than those of other groups ($p < 0.05$). The effect of filling methods on the polymerization of dual cured composites was different by their thickness or materials. In 6 ㎜ thickness, there was no significant difference between bulk and incremental filling groups. In 8 ㎜ thickness, Bis-Core showed no significant difference between groups. However, in MultiCore Flow, the microhardness ratio of bulk filling group was lower than that of incremental filling group ($p < 0.05$). The effect of curing methods on the polymerization of dual cured composites showed material specific results. In Bis-Core, the KHN of dual cured group were higher than those of self cured group at both surfaces ($p < 0.05$). However, in MultiCore Flow, the results were not similar at both surfaces. At the top surface, dual cured group showed higher KHN than that of self cured group ($p < 0.05$). However, in the bottom surface, dual cured group showed lower value than that of self cured group ($p < 0.05$). 〔J Kor Acad Cons Dent 33(3):169-176, 2008〕

Key words : Dual cured core materials, Thickness, Filling methods, Curing methods, Polymerization, Microhardness test

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Ⅰ. INTRODUCTION

The recent development of direct core materials enables dental clinicians to restore non-vital teeth by replacing the tooth structure that was lost due to endodontic treatment[1)]. Core materials can be provided as self cured, light cured or dual cured system. Self cured composites can build up the lost tooth structure at one time, and have better marginal adaptation and present less damage to the integrity of the restored tooth[2)]. However, they have limited working time and long setting time. On the contrary, light cured composites offer a longer working time than self cured ones, but there is a possibility of incomplete polymerization especially in a deep cavity due to the limited depth of light transmission[3)]. For those reasons, dual cured systems, that combined favorable properties of both self cured and light cured systems, have been widely used as core build-up resin materials[2-4)].

Adequate polymerization of a resin composite is a critical factor to obtain adequate physical and biological properties[1,3-7)]. The effectiveness of polymerization depends on not only the chemistry of the material and concentration of the initiator, but also the filler particle type, size, and loading[1,5,8-10)]. In a light cured or dual cured version, it is also affected by the curing light irradiance, exposure time and light transmission[9)].

Dual cured composites by different manufacturers have different handling characteristics, compositions, mixing types and properties from each other. The dual cured composite at the top surface is mainly polymerized through photo-initiated chemical reactions, while at the bottom surface it is done via chemically initiated polymerization. However, the deeper region of dual cured systems may not polymerize fully, when chemical polymerization is not sufficient[4)].

There are several methods to evaluate degree of polymerization of resin materials[1,5,6,8)]. Although direct method, such as Fourier-transformed infrared (FTIR) spectroscopy or Raman spectroscopy, has been widely used and most accurate method, it is complex, expensive and time-consuming[1,5,6)]. Therefore, microhardness test is considered as a simple and, at the same time, effective method to evaluate the degree of conversion[1,5,8)]. Moreover, a positive correlation has been reported between the results of hardness value and FTIR spectroscopy or Raman spectroscopy[5,8-11)].

The purpose of this study was to examine the effect of the thickness, filling methods and curing methods on the polymerization of dual cured composites by means of microhardness test.

Ⅱ. MATERIALS AND METHODS

Two dual cured core materials, MultiCore Flow (Ivoclar Vivadent AG, Schaan, Liechtenstein) and Bis-Core (Bisco Inc., Schaumburg, IL, USA) were used in this study. MultiCore Flow is auto-mixed type and Bis-Core is hand-mixed type of two pastes. Their components and concentrations are presented in Table 1.

Specimen Preparation

Each composite was packed into 2 ㎜ (bulky filled), 4 ㎜ (bulky filled), 6 ㎜ (bulky and incrementally filled) and 8 ㎜ (bulky and incrementally filled)-thickness Teflon mold, respectively. The mold cavity was confined between opposing 0.05 ㎜ transparent polyester films (Hawe Striproll, KerrHawe SA, Bioggio, Switzerland). A glass slide was covered on top of the resin composite and pressed, permitting the excess material to extrude from the mold. The material was irradiated for 10 sec per 1 ㎜ using a light curing unit (Optilux 501, Kerr, Danbury, USA), providing a light intensity of 500 ㎽/㎠ as evaluated by a hand-held radiometer, or self cure mode (waiting for 30 min in dark at room temperature). And then the samples were removed from the mold and the upper surfaces (closer to the light source) were marked with a pen. Seven samples were assigned to each group. Samples were stored in

Table 1. Components of materials used in this study

Material	Component	Concentration (%)*	
MultiCore Flow *(batch #: H19579)*		*Base*	*Catalyst*
Monomer matrix	Bis-GMA	28.1	28.4
	Triethylene glycol dimethacrylate		
	Urethane dimethacrylate		
	Benzoyl peroxide		
Inorganic filler	Barium glass filler	33.0	32.6
	Ba-Al-fluorosilicate glass	11.8	11.7
	Ytterbium trifluoride	10.1	10.1
	Highly dispersed silicon dioxide	16.4	16.0
Etc.	Catalysts and stabilizers	0.6	1.0
	Pigments	〈 0.1	
Bis-Core *(batch #: 0600005486, 0600005599)*		*Base*	*Catalyst*
	Bisphenol A diglycidyl methacrylate	5 – 15	15 – 30
	Glass filler	50 – 80	50 – 80
	Urethane	2 – 10	
	Triethylene glycol dimethacrylate		5 – 15
	Fused silica	2 – 10	2 – 10

* Specifications are in wt %. All values were presented by the manufacturers.

Table 2. Classification of groups in this study

Group code	Thickness (㎜)	Filling method	Curing method	Curing time (sec)
M2 B2	2	Bulk	Light	20
M4 B4	4	Bulk	Light	40
M6 B6	6	Bulk	Light	60
M6I B6I	6 (4 + 2)	Incremental	Light	40 + 20
M8 B8	8	Bulk	Light	80
M8I B8I	8 (4 + 4)	Incremental	Light	40 + 40
M8S B8S	8	Bulk	Self	_

M and B mean MultiCore Flow and Bis-Core, respectively.

the distilled water at 37℃ for 24 hours. The top and bottom surfaces of samples were polished with a #2000 abrasive paper and PoGo system (Dentsply, Konstanz, Germany) to remove the oxygen inhibited layer.

Microhardness Measurement

The Knoop hardness values (KHN) of the top and bottom surfaces were measured at 50 gf load and a dwell time of 10 seconds with a digital microhardness tester (FM-7, Future-Tech Corp., Tokyo, Japan). Indentations were made at five points on each surface. The microhardness ratio of two surfaces (hardness ratio) was defined as KHN of the bottom surface/KHN of the top surface.

Statistical Analysis

Statistical evaluation of the data was performed by one-way analysis of variance (ANOVA). Following the ANOVA, Scheffe multiple comparison test (α = 0.05) was used to identify pairwise differences. All statistical analysis was performed using SPSS 12.0 for Windows (SPSS Inc., Chicago, IL, USA).

Ⅲ. RESULTS

The mean KHN and the hardness ratio of MultiCore Flow are shown in Table 3 and Figure 1. At the top surface, M8S group showed significantly lower hardness value than those of the other groups ($p < 0.05$). At the bottom surface, M8 group showed the lowest hardness value, 31.8 ± 3.3. For the hardness ratio, M8 group showed the lowest value. However, the hardness ratios of all the other groups were over 0.8.

The mean KHN and the hardness ratio of Bis-Core are shown in Table 4 and Figure 2. At the top and bottom surfaces, B8S group showed sig-

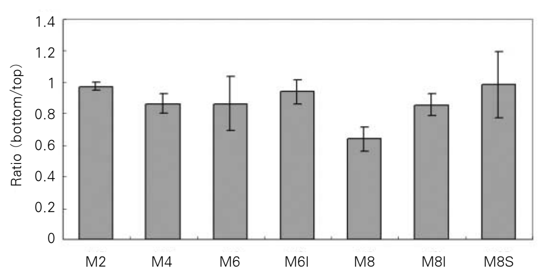

Figure 1. The hardness ratio between the top and bottom surface of each experimental group in MultiCore Flow.

Table 3. The mean KHN and the hardness ratio of MultiCore Flow (mean ± SD)

Groups	Number	Top	Bottom	Hardness Ratio
M2	7	52.6 ± 2.3^{A}	51.2 ± 2.3^{A}	0.97 ± 0.03^{A}
M4	7	53.4 ± 1.5^{A}	46.0 ± 3.1^{AB}	0.86 ± 0.06^{A}
M6	7	52.0 ± 1.8^{A}	44.8 ± 7.9^{AB}	0.86 ± 0.17^{A}
M6I	7	51.2 ± 2.5^{A}	47.9 ± 2.2^{AB}	0.94 ± 0.08^{A}
M8	7	50.0 ± 1.6^{A}	31.8 ± 3.3^{C}	0.64 ± 0.08^{B}
M8I	7	53.6 ± 2.7^{A}	45.6 ± 2.3^{AB}	0.85 ± 0.07^{A}
M8S	7	42.3 ± 8.0^{B}	40.4 ± 4.6^{B}	0.99 ± 0.21^{A}

SD means standard deviation.
The same superscript in each column is not significantly different by Scheffe multiple comparison test at α = 0.05.

Table 4. The mean KHN and the hardness ratio of Bis-Core (mean ± SD)

Groups	Number	Top	Bottom	Hardness Ratio
B2	7	68.3 ± 2.9^{A}	61.2 ± 4.1^{AB}	0.90 ± 0.09^{AB}
B4	7	64.7 ± 2.9^{A}	55.9 ± 4.4^{AB}	0.87 ± 0.08^{B}
B6	7	61.5 ± 4.9^{A}	55.0 ± 5.4^{AB}	0.90 ± 0.11^{AB}
B6I	7	62.7 ± 5.5^{A}	58.5 ± 5.0^{AB}	0.93 ± 0.06^{AB}
B8	7	65.5 ± 3.3^{A}	62.5 ± 4.2^{A}	0.96 ± 0.06^{AB}
B8I	7	67.5 ± 5.2^{A}	60.7 ± 4.2^{AB}	0.90 ± 0.09^{AB}
B8S	7	53.0 ± 3.6^{B}	54.1 ± 2.8^{B}	1.02 ± 0.06^{A}

SD means standard deviation.
The same superscript in each column is not significantly different by Scheffe multiple comparison test at $\alpha = 0.05$.

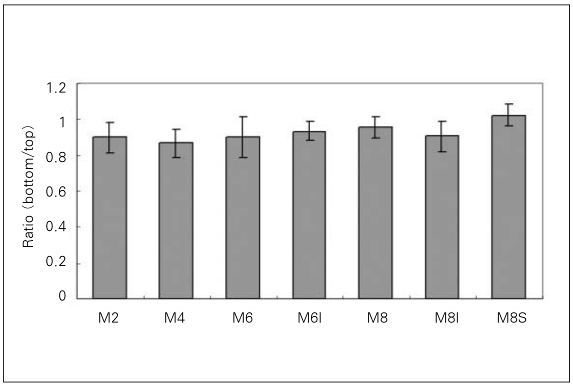

Figure 2. The hardness ratio between the top and bottom surface of each experimental group in Bis-Core.

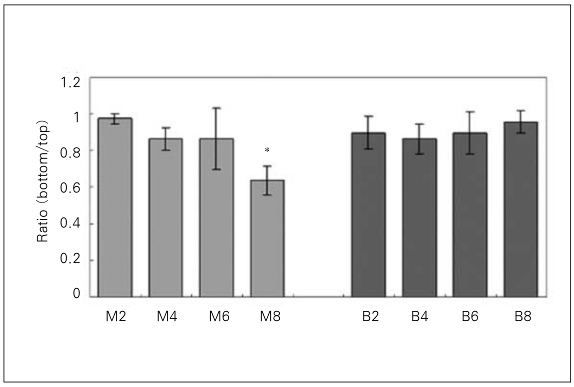

Figure 3. The hardness ratios of MultiCore Flow and Bis-Core by different thickness when the bulk technique was used. *Statistically significant in Scheffe multiple comparison test ($p < 0.05$).

nificantly lower hardness values than those of the other groups ($p < 0.05$). The hardness ratios in all groups were higher than 0.8.

Figure 3 shows the hardness ratios of MultiCore Flow and Bis-Core by different thickness when the bulk technique was used. The hardness ratio of M8 group was significantly lower than those of another three groups ($p < 0.05$). In Bis-Core, there was no significant difference between groups.

Ⅳ. DISCUSSION

Dual cured version of resin composite was introduced to combine favorable properties of both self cured and light cured systems[3,4]. However, it is still unclear whether polymerization of dual cured composites is consistent or not throughout the depth of a cavity, because of the complicated polymerization reaction and various formulation of the materials. Therefore, the aim of this study was to examine the effect of thickness, filling methods, curing methods on the polymerization of two dual cured core products by using a micro-hardness test.

In all groups except M8, the microhardness value of the cured surface was not affected by the thickness. The KHN of the bottom surface of M8 group showed lower value than those of the other groups. This implies that the polymerization of the material was not enough in the deep portion probably due to the insufficient chemical polymerization reaction. Therefore, although dual cured version has the incorporation of chemical and light curing modes in the same material, the two types of polymerization may not complement each of the other. Another explanation may be possible. Initial low intensity light curing accelerated change of the dual cure composite matrix from the gel to post-gel phase, thus the free movement of the radical might be inhibited[2,13]. On the other hands, B8 group showed no significant difference with another groups in KHN. This result indicates that the polymerization of dual cured composites especially in a deep cavity seems to be material dependent.

There have been many of studies addressing the effect of curing mode on a variety of properties of dual cured luting composites[2,13]. Some researchers proposed that the dual cured composites had inferior mechanical and physical properties when the material was only chemically cured[2,13,14]. In the present study, the microhardness value of M8S group was lower than that of light cured group. This suggests that light curing is needed to obtain good mechanical properties in the curing of dual cured materials.

The ideal hardness ratio of resin composites would be 1.0[1]. That is, the hardness of the bottom surface should be similar to that of the top surface[1,5]. However, it is not always possible to obtain such a value practically. In clinical conditions, the hardness ratio ranging from 0.80 ~ 0.90 has been employed as criteria for adequate conversion at a specific sample thickness[1,5,7,15]. In MultiCore Flow, the hardness ratio of M8 group was lower than 0.8, which means that polymerization at the bottom surface was not sufficient to provide optimal mechanical properties. On the other hand, the hardness ratios in all groups of Bis-Core showed higher than 0.8, which means that the polymerization of Bis-Core was not affected by thickness. When the bulk technique is used in a deep cavity up to 8 ㎜ depth, the material should be carefully chosen because the polymerization of dual cure version is material specific.

However, other factors, such as filler load, filler type, filler size, or resin matrix types, shall be taken into consideration when dual cured version is used[4]. Therefore, it is difficult to compare the degree of conversion between the different brands of composites only using microhardness test. Light transmission can also affect the microhardness. If light transmission of Bis-Core to the bottom surface is better than that of MultiCore Flow, the polymerization of the bottom surface of Bis-Core in a deep cavity can be better and enough to provide optimal mechanical properties. Different mixing methods were employed for two dual cured composites. MultiCore flow is auto-mixed type and Bis-Core is hand-mixed type. Bis-Core might contain more voids (porosity) than the MultiCore Flow, as the result of incorporating air while mixing the two pastes. Because the presence of oxygen in the voids inhibits polymerization, the degree of conversion can not be enough[2]. Nevertheless, in the present study, the microhardness value of Bis-Core was higher than that of MultiCore Flow. This result suggests that characteristics of material may affect more than mixing type on the microhardness although the microhardness value can be affected by their mixing types.

Within the limitations of the present study, the degree of polymerization of dual cured composite evaluated by means of microhardness test was not consistent throughout all the depth of a cavity. The incremental filling method and sufficient light curing to the materials may be recommended especially in a deep cavity to obtain adequate polymerization of a dual cured composite. However, the degree of polymerization of dual cured composites also seems to be material specific. Further researches are needed to elucidate polymerization reaction of dual cured composites.

Ⅴ. CONCLUSION

This study evaluated the effect of thickness, filling methods and curing methods on the polymerization of two dual cured core materials, MultiCore Flow and Bis-core by means of microhardness test.

The effect of thickness and curing methods on the polymerization of dual cured composites showed material specific results. In 2, 4 and 6 ㎜ groups, the KHN of two materials were not affected by thickness. However, in 8 ㎜ group of MultiCore Flow, the KHN of bottom surface was lower than those of the other groups. In Bis-Core, the KHN of dual cured group were higher than those of self cured group at both surfaces. However, in MultiCore Flow, dual cured group showed higher KHN than that of self cured group at top surface, while the opposite at bottom surface. The effect of filling methods on the polymerization of dual cured composites was different by their thickness or materials. In 6 ㎜ thickness, there was no significant difference between bulk and incremental filling groups. In 8 ㎜ thickness, Bis-Core showed no significant difference between groups. However, in MultiCore Flow, the microhardness ratio of bulk filling group was lower than that of incremental filling group.

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국문초록

두께에 따른 이중 중합형 복합레진의 중합

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본 연구는 이중 중합형 복합레진에서 재료의 두께, 충전방법 및 중합방법에 따른 중합도를 미세경도 시험을 이용하여 측정하고자 하였다. 이중 중합형 복합레진으로는 MultiCore Flow (Ivovlar Vivadent AG, Schaan, Liechtenstein)와 Bis-Core (Bisco Inc., Schaumburg IL, USA)를 사용하였다. 시편의 제작은 각각 두께가 2 (단일충전), 4 (단일충전), 6 (단일충전과 적층충전), 8 (단일충전과 적층충전) ㎜의 Teflon mold에 재료를 주입한 다음 할로겐 광중합기 (Optilux 501, Kerr, Danbury, USA)를 사용하여 광중합하거나 암실에서 30분 동안 기다린 후(자가 중합) Teflon mold에서 제거하였다. 제거한 시편은 37℃ 증류수에 24시간 동안 보관한 후 각 시편의 윗면과 아랫면을 2000번 연마제와 PoGo system (Dentsply, Konstanz, Germany)을 이용하여 마무리하였다. Digital microhardness tester (FM-7, Future-Tech Corp., Tokyo, Japan)를 이용하여 경도값(Knoop hardness number)을 측정하였으며 윗면의 경도값/아랫면의 경도값을 이용하여 경도비를 계산하였다. 계측치는 one-way ANOVA로 통계 분석 후 사후검정은 Scheffe 다중비교법을 이용하였다.

이중 중합형 복합레진의 중합도에 대한 두께의 영향을 보면 재료에 따라 다른 결과를 보였다. 2, 4, 6 ㎜ 군에서는 MulriCore Flow와 Bis-Core 모두 두께에 의한 영향을 받지 않았지만 8 ㎜ 군에서는 MultiCore Flow의 아랫면에서 다른 두께의 군보다 낮은 경도값을 보였다.

충전방법에 따른 중합도의 차이를 보면, 재료의 두께나 재료에 따라 다른 결과를 보였다. 6 ㎜ 군에서는 단일충전군과 적층충전군 사이에 차이를 보이지 않았으나, 8 ㎜ 군에서는 Bis-Core에서는 차이가 없는 반면 MultiCore Flow에서는 단일충전한 군이 적층중전한 군보다 낮은 경도비를 보였다.

중합방법에 따른 중합도의 차이를 보면, 재료에 따라 다른 결과를 보였다. Bis-Core의 경우에는 윗면과 아랫면 모두에서 이중 중합 시킨 군이 자가 중합 시킨 군보다 높은 경도값을 보였다. 그러나 MultiCore Flow의 경우, 윗면에서는 이중중합 시킨 군이 더 높은 경도값을 보였지만 아랫면에서는 더 낮은 값을 보였다.

따라서 본 연구의 결과에 따르면 코어용 이중 중합형 복합레진을 깊은 와동에 충전할 경우 적층충전이 추천되며, 또한 광중합을 해 줌으로써 더 좋은 물리적 성질을 기대할 수 있을 것으로 사료된다.

주요어: 이중 중합형 복합레진, 두께, 충전방법, 중합방법, 중합도, 미세경도 시험