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**Influence of inhomogeneity
of light curing unit on microhardness
of resin cements under CAD/CAM blocks**

Yu-Ra Go

The Graduate School

Yonsei University

Department of Dental science

**Influence of inhomogeneity
of light curing unit on microhardness
of resin cements under CAD/CAM blocks**

A Masters Thesis

Submitted to the Department of Dentistry
and the Graduate School of Yonsei University

in partial fulfillment of the
requirements for the degree of
Master of Dental science

Yu-Ra Go

June 2021

This certifies that the Masters thesis of
Yu-Ra Go is approved.

Sung-Ho Park

Jeong-Won Park

Kwang-Mahn Kim

The Graduate School

Yonsei University

June 2021

감사의 글

학위 논문의 마무리 하면서 감사의 글을 어떻게 쓸까 고민을 하다 보니, 짧고도 긴 수련 생활을 추억해 볼 수 있었습니다. 인턴으로 서울에 올라왔을 때부터 시작해서 보존과에 의국원으로 공부하고 진료할 수 있었던 모든 순간순간이 소중한게 느껴지고, 지금 이 순간도 매우 감사함을 느낍니다.

먼저 논문의 작성을 지도해주신 박성호 교수님께 감사의 말씀을 올립니다. 교수님께서 차근차근히 도와주시고 배려해주신 덕분에 무사히 마무리할 수 있었고, 돈으로도 살 수 없는 지식을 배웠습니다. 그리고 따뜻한 조언과 격려를 통해 논문의 완성에도 도움을 주신 박정원 교수님과 김광만 교수님께도 감사의 말씀 올립니다.

많은 가르침을 주시고 부족한 저를 이끌어주신 김정희 부장님, 김선호 과장님, 김미연 과장님, 김난아 과장님 덕분에 행복한 수련 생활을 보낼 수 있었습니다. 그리고 즐거운 추억을 만들어준 사이 좋은 보존과 의국원들에게 감사의 말씀 드리고 싶습니다. 특히 이제 가족같이 느껴지는 우리 동기 진영언니, 없었으면 어떻게 수련생활 했을까 싶을 정도로 많이 감사하고 고맙습니다. 무뚝뚝하지만 웃을 때 예쁜 수연 언니를 비롯해 재운 오빠, 철호, 중훈이도 같이 인턴으로 입사해서 4년 동안 많은 추억 만들어줘서 감사합니다.

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2021년 6월 고유라 올림

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Abstract

Influence of inhomogeneity of light curing unit on microhardness of resin cements under CAD/CAM blocks

Yu-Ra Go

Department of Dental Science, Graduate School, Yonsei University

(Directed by Professor Sung-Ho Park, D.D.S., M.S., Ph.D.)

1. Objectives

The light from light curing units passing through the restoration shows an inhomogeneous distribution. The objective of this study was to determine effects of light inhomogeneity on the polymerization of resin cement under CAD/CAM blocks of different types and thicknesses.

2. Materials and Methods

IPS e.max CAD (A3 LT, A3 HT), Celtra Duo (A3 LT), LAVA Ultimate (A3 LT) and Vita Enamic (A3 T) were used. Each block's thickness was 1.0, 1.5, 2.0 and 4.0 mm. A total of 100 specimens were produced. Variolink N (light-cured) and RelyX U200 (dual-cured) were used as resin cements. Light transmission and beam profile of each block were measured. For microhardness measurement, five points (-4 mm, -2 mm, 0 mm, +2 mm, +4 mm) that coincided with the distance from the center to the periphery of the tip were marked on the specimen's surface. At each point, microhardness was measured immediately, 24 hours and a week after polymerization.

3. Results

The resin cement's microhardness decreased as the thickness of the CAD/CAM block's thickness increased. The e.max HT groups showed the highest microhardness and the Vita Enamic groups showed the lowest microhardness ($p < 0.05$). Resin cements with thickness of 1 mm showed relatively uniform microhardness, whereas those with thickness of 2 mm or more showed inhomogeneous microhardness ($p < 0.05$).

4. Conclusion

Translucency and light transmission differed, showing an inhomogeneous distribution depending on the CAD/CAM block's thickness and types. And light inhomogeneity affected resin cement's microhardness.

Key words : CAD/CAM blocks; resin cements; inhomogeneous; microhardness

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I. Introduction

With the development and universalization of CAD/CAM technology, CAD/CAM restorations are gaining popularity in aesthetic dentistry. Accordingly, several studies

have been conducted on the durability and adhesion of CAD/CAM restorations. The clinical success of restorations with CAD/CAM blocks depends on the adhesion between restoration and teeth [1]. In general, CAM/CAM restorations are bonded with resin cement with high bonding strength, aesthetics, and low solubility [2].

The resin cement used for bonding of the restoration must not only receive sufficient light energy, but also receive energy in the wavelength range of 400-500 nm to generate an adequate number of free radicals for proper polymerization. However, light energy attenuation by restoration can cause incomplete polymerization of the resin cement, resulting in deterioration of physical properties and discoloration [3]. This deterioration of resin cement's durability can lead to permanent failure of CAD/CAM restorations.

Since the distribution of irradiance and spectral emission of many dental light beams is different, the light energy passing through the restoration shows an inhomogeneous distribution [4-6]. Differences in irradiance and light wavelengths in different areas within the same restoration can lead to differences in the degree of polymerization and reaction rate of resin cement [4]. This light distribution can adversely affect resin-based restorative materials. Sunnegårdh-Grönberg et al. [6] have measured beam profiles of four light-curing units (LCUs) and claimed that both local irradiance and spectral emission of LCUs are inhomogeneous. In this regard, the authors have stated that the inhomogeneous light output of LCUs could affect resin-based restoration's microhardness, which could lead to a deteriorated physical properties of the restoration [5].

The degree of light attenuation by the CAD/CAM block is also affected by the block's type and thickness [7, 8]. If CAD/CAM block component's types and ratios are different, there might be various differences in light transmission or light distribution. Emami et al. [7] have found that depending on the polymer content, even glass-ceramic of the same shade can show different translucency and fluorescence. In addition, the thicker the restoration, the less the number of photons passing through the restoration. This can be explained by the scattering and absorption of light by the Beer-Lambert law.

In other words, the thicker the restoration, the greater the radiation absorption, implying that light concentration and path length are different [8]. Similarly, Watts et al. [9] have analyzed light transmission of ceramic materials at a wavelength of 400-500 nm, claiming that if the surface reflection value is corrected, light transmission could be accurately expressed according to the Law of Beer-Lambert. In addition, Ilie et al. [10] have quantified the amount of light transmitted in zirconia with thicknesses of 0.5, 1, 1.5, 2, 2.5, and 3 mm. They reported that the value of transmitted irradiance decreased as the thickness of the restoration increased.

Light attenuation by the CAD/CAM block can lead to a significant reduction in resin cement's microhardness, thus negatively affecting the success rate of the restoration [11]. The resin cement polymerization rate can be explained by the total energy theory that depends on the radiant exposure determined by the interaction between the exposure duration and the radiant emittance [12, 13]. According to a previous study [14, 15], the radiant exposure required to polymerize a 2 mm thick composite resin was considered to be 16 J/cm². According to a study by Beolchi et al. [16], 12 J/cm² is required to polymerize a 2 mm of Z100 composite resin properly.

RelyX U200 is self-adhesive resin cement that does not require a separate bonding process as it contains methacrylated phosphoric ester as a functional monomer. Use of self-adhesive resin cements has become increasingly common. Their clinical success is based on reduced technical and operator sensitivity [17]. Several studies investigated shear and bond strength of RelyX Unicem to different types of ceramic. The obtained results stated that RelyX Unicem achieves bond strength that is either higher or comparable to other resin cements [18]. On the other hand, Variolink N is conventional resin cement that does not contain a functional monomer. Bis-GMA is its main component.

As described above, many studies have analyzed the effect of light energy attenuation of LCUs and microhardness of resin cement according to the type and

thickness of CAD/CAM blocks. However, studies on how the inhomogeneity of light emitted from LCUs affects the polymerization of resin cement under CAD/CAM restoration is lacking. When the type and thickness of CAD/CAM blocks are different, it would be a meaningful work to find out how inhomogeneity of light passing through them changes and how these changes affect polymerization of resin cement. Therefore, the objectives of this study were to 1) investigate effects of CAD/CAM block's type and thickness on translucency, light transmission, and microhardness of resin cement; 2) investigate effects of inhomogeneous distribution of irradiance passing through the CAD/CAM block on resin cement's microhardness; 3) to propose appropriate energy to polymerize resin cement based on from results. The null hypotheses of this study are as follows: (1) Regardless of the CAD/CAM block's type or thickness, the translucency and light transmission of the CAD/CAM block are constant; (2) Beam profile passing through the CAD/CAM block has a uniform profile regardless of block's type or thickness; (3) Regardless of the type or thickness of the CAD/CAM block, there is no difference in the resin cement's microhardness; (4) Regardless of the distance from the center of the irradiated area on the resin cement, the resin cement has a constant microhardness for all parts.

II. Materials & Methods

2.1 Preparation of CAD/CAM block specimen

Five types of CAD/CAM block were prepared (Table 1). Using a 3-axis cutting machine, specimens having a width of 10 mm x 10 mm and a thickness of 1.0 mm, 1.5 mm, 2.0 mm, and 4.0 mm were prepared. One side of each specimen was polished to mimic the surface of the polished ceramic restoration. IPS e.max CAD was subjected to sintering according to the manufacturer's instructions after preparing the specimen. Celtra Duo was performed without glazing treatment. Five specimens were fabricated in each types and thickness.

2.2 Translucency of CAD/CAM block

The specimen's translucency was measured using a VITA Easy Shade (VITA Zahnfabrik, Bad Säckingen, Germany). The sample was evaluated for translucency parameter (TP) under two backgrounds: White ($L^* = 97.0$, $a^* = 0.6$, $b^* = 3.1$) and Black ($L^* = 17.6$, $a^* = -4.4$, $b^* = 4.7$). The putty index was prepared so that each CIE $L^*a^*b^*$ value could be measured at the center of the specimen. TP was obtained using the following formula:

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

where subscript B was a value measured on a black background and subscript W was a value measured on a white background. The larger the TP value, the more transparent the specimen.

2.3 Measurement of irradiance and beam profile after passing the CAD/CAM block

Radiant power (mW) of the light source passing through the specimen was measured using a Fiber-optic Spectrometer (USB 2000; Ocean Optics, Dunedin, FL, USA). To measure LCU's radiant at the same position, the LCU was fixed to the clamp holder. The irradiance of light passing through each CAD/CAM specimen was measured with an optical fiber (diameter of 3.175 mm) attached to a USB 2000. The irradiance (mW/cm^2) was calculated by dividing the diameter of the optical fiber. The LCU used in the experiment was an LED curing unit (EliparTM DeepCure-L, 3M ESPE, St. Paul, USA) with a diameter of 9 mm. The irradiance of the LCU was measured with a Fiber-optic Spectrometer (USB 4000; Ocean Optics, Dunedin, FL, USA) and an Integration sphere. The value corrected by the diameter of the curing tip was $1,015 \text{ mW}/\text{cm}^2$.

Irradiance distribution was measured using a Laser Beam Profiler (Ophir Spiricon, Logan, UT, USA) with lens having a focal length of 25 mm. Each CAD/CAM specimen was fixed 3 mm away from the light tip of the LCU. The light was attenuated using 3.0 OD filter Reflective Neutral Density Filters (Optical density, Edmund optics, Barrington, NJ, USA). According to the manufacturer, only 0.1 % of the light passes through the 3.0 OD filter.

The beam profile passing through the CAD/CAM specimen was recorded and photographed using a BeamGauge v.6.6 software (Ophir Spiricon, Logan, UT, USA).

2.4 Measurement of microhardness of resin cements

RelyX U200 (3M/ESPE, St. Paul, MN, USA) and base pastes of Variolink N (Ivoclar-Vivadent, Liechtenstein) were used in this study. According to the CAD/CAM block type, five specimens were made. A total of 200 resin cement specimens were produced. The resin cement can have a constant thickness using a stainless steel mold

with a thickness of 200 μm (10mm x 10mm). The resin cement was mixed using a plastic spatula on a mixing pad according to the manufacturer's instructions and then placed to the mold cavity. The CAD/CAM Block specimen and mylar strip were placed on it. Resin cement was pressed with a glass plate under constant pressure to make an even and smooth surface. After fixing the light tip of LCU on a vertical line with resin cement under the CAD/CAM block, light polymerization was performed for 20 seconds (Fig. 1). The control group was polymerized without placing the CAD/CAM block.

To measure the inhomogeneous microhardness of each resin cement, microhardness was measured at five points (-4 mm, -2 mm, 0 mm, +2 mm, +4 mm; X-4, X-2, X0, X+2, X+4) according to the distance from the center of the irradiated area on the resin cement to the peripheral part (Fig. 2). Microhardness was measured at each point immediately after polymerization (T0), 24 hours after polymerization (T1), and a week after polymerization (T2). All resin cement specimens were stored in an opaque box wrapped with aluminum foil to prevent polymerization by external light. Microhardness measurement was performed on the top of each resin cement specimen using a Vickers hardness tester (MVH, Shimadzu, Japan) with a load of 0.98 N for 10 seconds.

2.5 Statistical analysis

According to the type and thickness of CAD/CAM block, translucency and light transmission were analyzed by one-way analysis of variance (ANOVA) with Post-hoc Tukey test. The microhardness of each specimen was analyzed by repeated measures ANOVA for each resin cement specimen. Bonferroni test was used as a post-hoc analysis (variables: thickness of CAD/CAM block, type of CAD/CAM block, distances from the center of the light tip to the periphery). Pearson correlation was performed to determine correlations between optical properties of CAD/CAM block and microhardness. In all cases, $P < 0.05$ was considered statistically significant.

Table 1. CAD/CAM blocks and resin cements used in this study.

Materials	Manufacturers	Primary components	Lot. number	Shade
e.max CAD LT	Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate Glass-ceramic	605330	A3 LT
e.max CAD HT	Ivoclar Vivadent, Schaan, Liechtenstein	Lithium disilicate Glass-ceramic	626409	A3 HT
Celtra Duo	Dentsply DeTrey, Konstanz, Germany	Zirconia-reinforced Glass ceramic	5365411025	A3 LT
Lava Ultimate	3M ESPE, St. Paul, MN, USA	Nano-ceramic particle Reinforced Composite	V-T- 3M2EM14	A3 LT
Vita Enamic	VITA Zahnfabrik Bad Säckingen, Germany	Polymer infiltrated ceramic	Lava-03	A3 T
RelyX U200	3M ESPE, St. Paul, MN, USA	Base : phosphoric acid, methacrylate, TEGDMA, esters Catalyst : fiberglass, substitute dimethacrylate	6751704	A2
Variolink N Base	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, urethane dimethacrylate, barium glass	YZ1288	A3

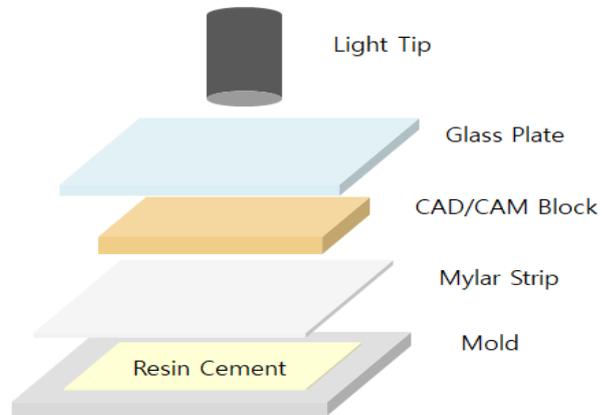


Figure 1. Diagram showing the polymerization of resin cements.

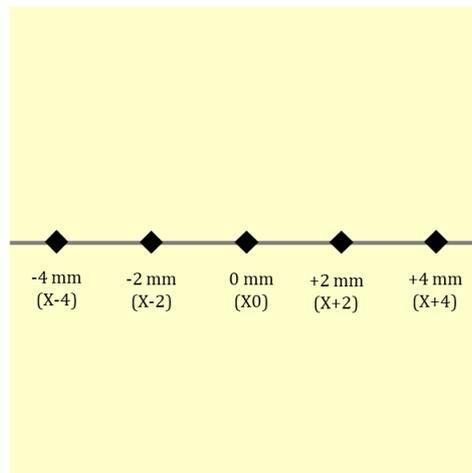


Figure 2. Diagram of a resin cement specimen with Vickers Hardness measurements made in 2 mm increments of the irradiated area.

III. Results

3.1 Translucency of CAD/CAM block

Translucency of the CAD/CAM block is shown in Table 2. The translucency of CAD/CAM block was significantly different according to the type and thickness of the block ($p < 0.05$). As the thickness of the CAD/CAM block increased, the translucency of the block decreased ($p < 0.05$). TP values decreased in the order of e.max HT \geq Lava Ultimate \geq Celtra Duo \geq e.max LT \geq Vita Enamic ($p < 0.05$).

3.2 Irradiance and beam profile after passing CAD/CAM block

Light transmission of the LCU through the CAD/CAM block is shown in Table 3. There was a significant difference in measured irradiance according to the type and thickness of the CAD/CAM block ($p < 0.05$). The LCU used in the study showed a spectral distribution with a peak at about 450 nm. As the thickness increased, the peak value decreased and the irradiance passing through the CAD/CAM block decreased ($p < 0.05$). e.max HT showed the highest irradiance, followed by Lava Ultimate, Celtra Duo, e.max LT, and Vita Enamic.

The beam profile of the light passing through the CAD/CAM block and OD filter is presented in Figure 3. Central saturation was observed for all specimens with thickness of 1, 1.5 mm and 2 mm. The beam profile for the CAD/CAM block with 4 mm thickness showed a non-uniform light distribution. A high luminance region was observed in the central region of all specimens. The further away from the central region, the lower luminance region appears. There were also differences according to the type of block. Unlike other blocks, for the 4 mm thick Vita Enamic, the hot area did not appear even in the central area. Only a low illumination area was shown.

3.3 Microhardness of resin cements

Mean values and standard deviations of the microhardness of all specimens are shown in Figures 4-9. As the thickness of the CAD/CAM block increased, the microhardness value of resin cements significantly decreased for both RelyX U200 and Variolink N in the order of 1 mm \geq 1.5 mm > 2 mm > 4 mm ($p < 0.05$). Regarding the type of the CAD/CAM block, significant differences in microhardness were found in the order of e.max HT \geq Lava Ultimate \geq Celtra Duo \geq e.max LT \geq Vita Enamic for both RelyX U200 and Variolink N ($p < 0.05$).

There were no differences in microhardness among X-4, X-2, X0, X+2, and X+4 in the resin cement specimens under 1 mm thick CAD/CAM blocks. In contrast, those under the 2 mm and 4 mm blocks showed differences among the measurement points for all blocks in both RelyX U200 and Variolink N ($p < 0.05$). For the RelyX U200 specimens under the 1.5 mm blocks, the microhardness was different among the measurement points for e.max LT, and Enamic, whereas there were no differences for e. max HT, Celtra Duo, and Lava Ultimate. In the Variolink N, they were different for all blocks. The highest microhardness was shown at point X0. The microhardness decreased as the distance from X0 increased ($X0 \geq X-2, X+2 \geq X-4, X+4$).

In the case of RelyX U200, there were significant differences between T0 and T1, and T0 and T2, but there was no significant difference between T1 and T2 ($p < 0.05$). In Variolink N, there was no significant difference between T0, T1, and T2 ($p > 0.05$).

3.4 Radiant exposure for homogenous polymerization

At thickness of 1 mm, the microhardness of the resin cement was uniform regardless of the location at a light irradiation time of 20 seconds. However, at thickness of 1.5 mm, some blocks showed non-uniform microhardness values. A relatively inhomogeneous microhardness appeared at thickness of 2 mm or 4 mm. For homogenous microhardness,

the energy value derived from the block of 1 mm thickness can be referred. The transmitted irradiance at a block thickness of 1 mm was about 300 mW/cm^2 and the energy value after light irradiation for 20 seconds was performed about 6 J/cm^2 , which can be viewed as the reference value minimum total energy.

3.5 Correlation between optical properties of CAD/CAM blocks and microhardness

According to the Pearson correlation coefficient, there was a significant correlations between the translucency of the CAD/CAM block and the microhardness in both RelyX U200 (0.727) and Variolink N (0.783) ($p < 0.01$). In addition, there was a significant correlations between the light transmission and the microhardness in both RelyX U200 (0.735) and Variolink N (0.8) ($p < 0.01$).

Table 2. Comparison of translucency parameter (TP) of CAD/CAM blocks.

Thickness (mm)	e.max LT	e.max HT	Celtra Duo	Lava Ultimate	Vita Enamic
1	17.98 ± 0.54 ^{Ac}	20.68 ± 0.38 ^{Aa}	17.76 ± 0.22 ^{Ac}	19.49 ± 0.67 ^{Ab}	14.98 ± 0.27 ^{Ad}
1.5	13.54 ± 0.35 ^{Bb}	14.88 ± 0.46 ^{Ba}	13.96 ± 0.28 ^{Bb}	14.75 ± 0.50 ^{Ba}	9.93 ± 0.61 ^{Bc}
2	6.14 ± 0.28 ^{Cc}	9.28 ± 0.56 ^{Ca}	6.36 ± 0.59 ^{Cc}	7.75 ± 0.26 ^{Cb}	5.53 ± 0.38 ^{Cd}
4	1.56 ± 0.05 ^{Dc}	3.46 ± 0.12 ^{Da}	1.98 ± 0.49 ^{Dbc}	2.87 ± 0.19 ^{Db}	0.77 ± 0.35 ^{Dd}

Different uppercase superscript letters represent statistical differences within columns (thickness) and different lowercase superscript letters represent statistical differences within rows (types) ($p < 0.05$).

Table 3. Comparison of light transmission (mW/cm^2) of CAD/CAM blocks. Light transmission of the LCU was $1,015 \text{ mW}/\text{cm}^2$.

Thickness (mm)	e.max LT	e.max HT	Celtra Duo	Lava Ultimate	Vita Enamic
1	327.6 ± 15.48 ^{Aa}	350.1 ± 16.38 ^{Aa}	330.69 ± 8.93 ^{Aa}	348.27 ± 15.3 ^{Aa}	295.90 ± 8.74 ^{Ab}
1.5	227.88 ± 8.46 ^{Bb}	258.85 ± 8.46 ^{Ba}	225.23 ± 8.31 ^{Bb}	250.31 ± 2.95 ^{Ba}	197.52 ± 5.37 ^{Bc}
2	138.55 ± 1.28 ^{Cc}	175.92 ± 0.78 ^{Ca}	145.83 ± 2.91 ^{Cb}	171.76 ± 1.21 ^{Ca}	124.02 ± 2.61 ^{Cd}
4	55.89 ± 0.52 ^{Dc}	68.22 ± 3.45 ^{Da}	60.78 ± 1.48 ^{Db}	63.71 ± 2.29 ^{Dab}	52.71 ± 0.11 ^{Dd}

Different uppercase superscript letters represent statistical differences within columns (thickness) and different lowercase superscript letters represent statistical differences within rows (types) ($p < 0.05$).

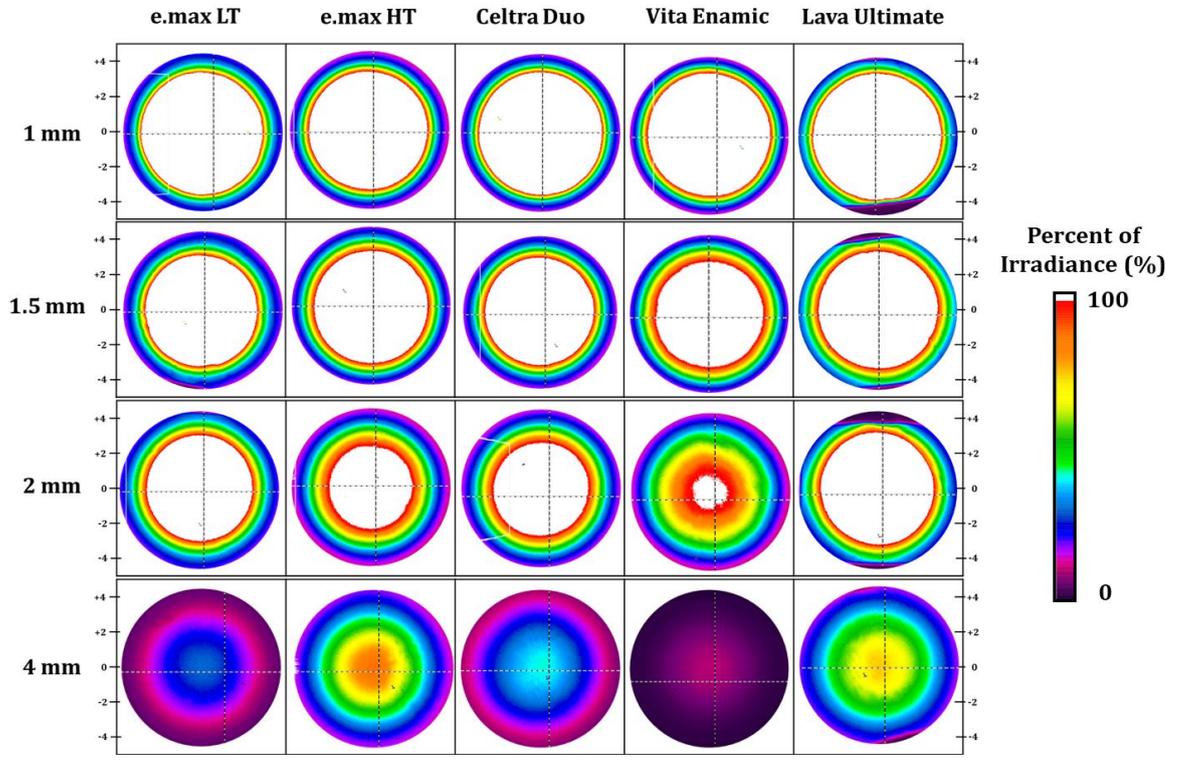


Figure 3. Beam profile through different types and thickness of CAD/CAM blocks with 3.0 OD filter. Irradiance of all blocks was not uniform. It showed dense irradiance at the center and low irradiance at the outer cold area. Central saturation was observed for all specimens with thickness of 1, 1.5 mm and 2 mm.

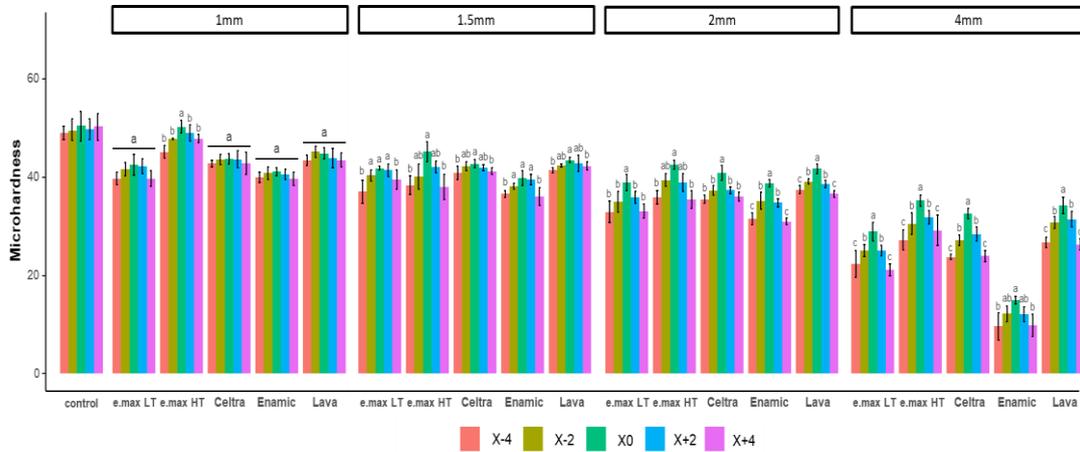


Figure 4. Immediately (T_0) after polymerization, the microhardness (HV) of RelyX U200 plotted using the mean and standard deviation. Different lowercase letters represent statistical differences between measurement points (X-4, X-2, X0, X+2, X+4).

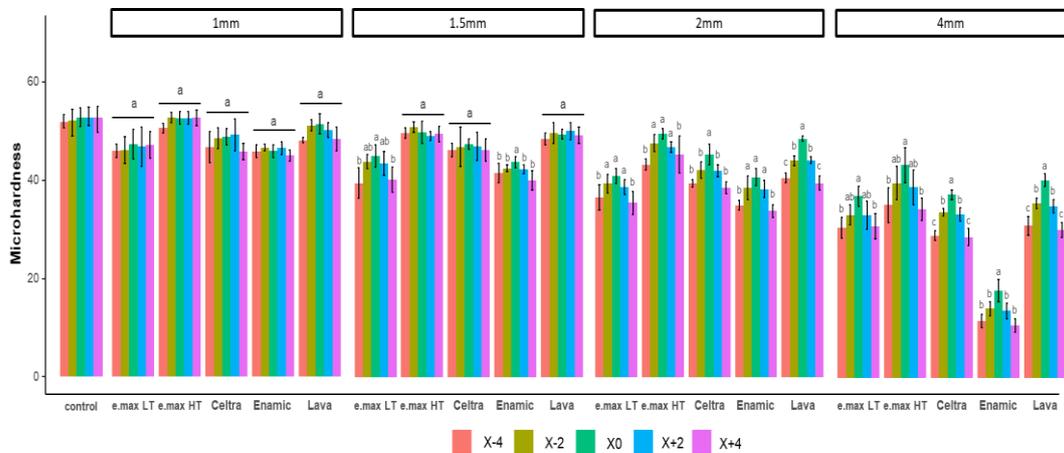


Figure 5. 24 hours (T_1) after polymerization, the microhardness (HV) of RelyX U200 plotted using the mean and standard deviation. Different lowercase letters represent statistical differences between measurement points (X-4, X-2, X0, X+2, X+4).

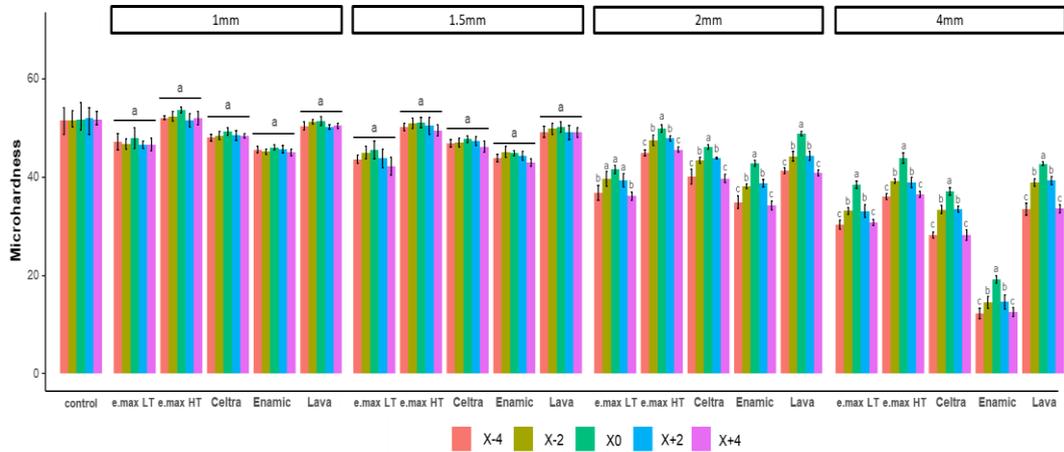


Figure 6. A week (T2) after polymerization, the microhardness (HV) of RelyX U200 plotted using the mean and standard deviation. Different lowercase letters represent statistical differences between measurement points (X-4, X-2, X0, X+2, X+4).

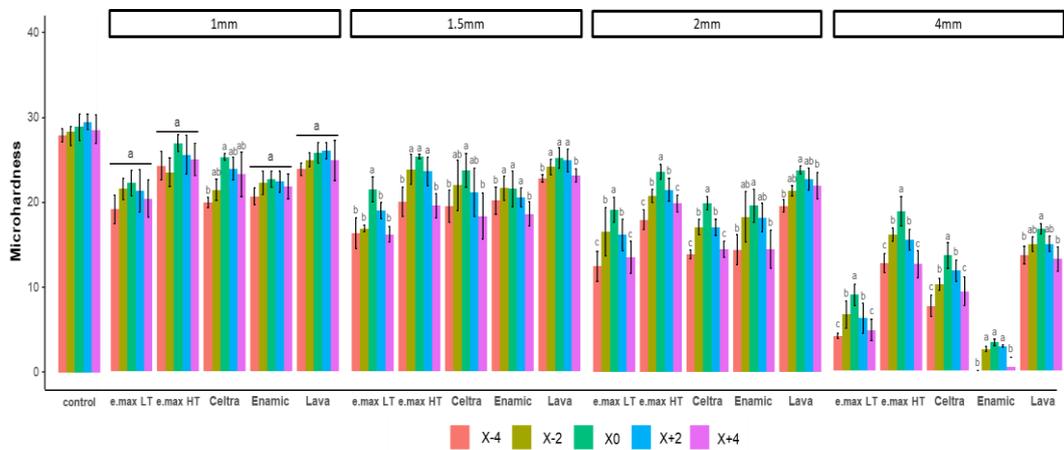


Figure 7. Immediately (T0) after polymerization, the microhardness (HV) of Variolink N plotted using the mean and standard deviation. Different lowercase letters represent statistical differences between measurement points (X-4, X-2, X0, X+2, X+4).

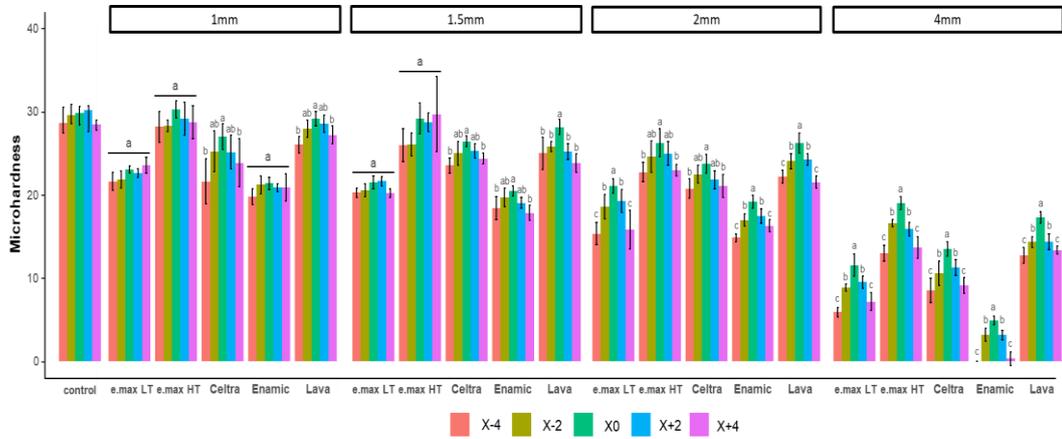


Figure 8. 24 hours (T1) after polymerization, the microhardness (HV) of Variolink N plotted using the mean and standard deviation. Different lowercase letters represent statistical differences between measurement points (X-4, X-2, X0, X+2, X+4).

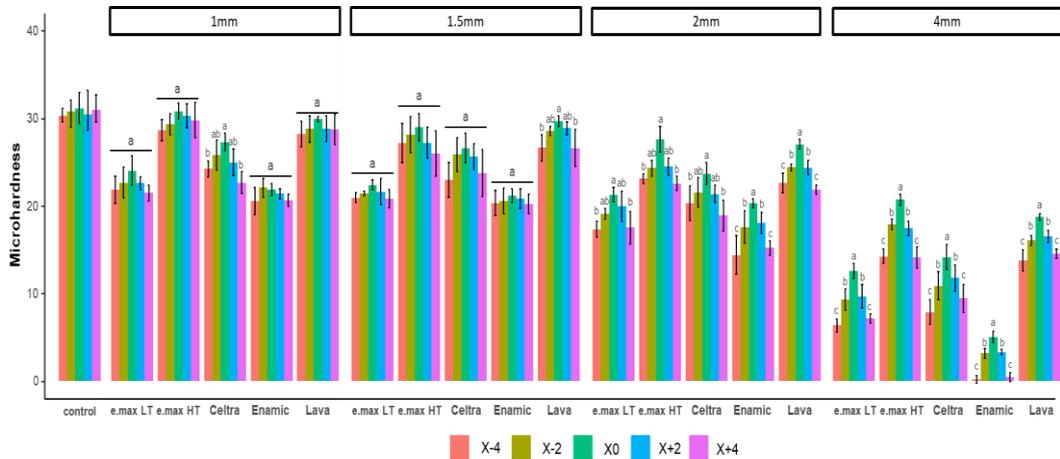


Figure 9. A week (T2) after polymerization, the microhardness (HV) of Variolink N plotted using the mean and standard deviation. Different lowercase letters represent statistical differences between measurement points (X-4, X-2, X0, X+2, X+4).

IV. Discussion

This study investigated effects of the type and thickness of CAD/CAM blocks on resin cement's microhardness. This study was meaningful in that we explored the inhomogeneity that appeared when the light energy from the LCU passed through CAD/CAM blocks and determine effect of inhomogeneity on the polymerization of resin cements.

The minimum width of margin in CAD/CAM crown should be 1.0 mm and the minimum wall thickness should be 1.5 mm. In the case of posterior inlay or onlay, the occlusal surface is required to be 2.0 mm. It is necessary to have a maximum thickness of 4.0 mm in the proximal box. Accordingly, CAD/CAM blocks with thickness of 1.0 mm, 1.5 mm, 2.0 mm, and 4.0 mm were used in this study. To consistently reproduce the thickness of resin cement, a stainless steel mold with a thickness of 200 μm was used. Besides, in this study, the microhardness of the surface was used as an index to evaluate the degree of polymerization of resin cement. Ozturk et al. [19] have performed a study on the polymerization rate and surface hardness value of resin cement under ceramic restoration and found that there is a positive correlation between the resin cement's polymerization degree and surface hardness.

Results of the present study revealed that as the thickness of the CAD/CAM block increased, the translucency and light transmission decreased showing a difference between types of CAD/CAM block ($p < 0.05$). Therefore, the first null hypothesis that translucency and light transmission were constant regardless of the CAD/CAM block's type and thickness was rejected. TP values are known to be determined by chemical structure, crystal thickness, porosity, volume, and crystalline form [20]. In particular, TP values are strongly influenced by the thickness of the restoration. Similar to results of previous studies [11, 20, 21], as the thickness increased, TP values decreased in this study ($p < 0.05$). The translucency of the CAD/CAM block can affect the attenuation of light

passing through the restoration. The higher the translucency, the deeper the light can penetrate into the resin cement specimen.

Compared with the LCU's own radiance emittance (1,015 mW/cm²), the irradiance decreased by 71.5 - 65.6% when passing through a 1.0 mm thickness block. When the block thickness increased from 1.0 mm to 1.5 mm and 2.0 mm, the irradiance decreased to 73.9 - 66.7% and 50.2 - 42.2%, respectively. In addition, a low irradiance of 52.7 - 68.2 mW/cm² was observed in all 4 mm thick specimens (Table 3). Similarly, Meng et al. [22] have reported that the light's transmittance is decreased to 100 mW/cm² in ceramics with a thickness of 3.0 mm or more. In another study [23], when three CAD/CAM materials was measured, light's transmission was significantly decreased in the order of 1.0 mm > 1.5 mm > 2.0 mm. The author stated that it was because scattering occurred inside the material.

According to the thickness of the CAD/CAM block, the decrease in transparency and irradiance had a significant correlation with the microhardness of resin cement ($p < 0.01$). The microhardness of resin cements that control group was similar to that of the passed through 1.0 mm thickness of e.max HT. As the thickness of the CAD/CAM block increased, the resin cement's microhardness tended to decrease. However, there was no significant difference in microhardness between 1.0 mm and 1.5 mm ($p > 0.05$) in most cases. This result was consistent with Volkan's experimental results showing that there was no difference in microhardness of resin cement underneath the CAD/CAM block with a thickness of 1.0 mm or 1.5 mm [24]. Also, Arrais et al. [25] have reported that there is a significant difference in microhardness between 0.7 mm and 1.4 mm thickness when the microhardness of dual-cured resin cements are measured under ceramic blocks. Regarding this, Halverson et al. have reported that 90% polymerization of resin-based composite can be achieved when an energy of 2000 mWsec/cm² is given by photopolymerization at 200 mW/cm² for 10 seconds [26]. In this study, except for Enamic, thickness of 1.0 mm or 1.5 mm had an irradiance of 200 mW/cm² or more. The energy suggested by Souza et al. [27] was satisfied by performing light irradiation for 20 seconds

(Table 3). Based on this, it could be assumed that there was no significant difference in microhardness between specimens with thickness of 1.0 mm and 1.5 mm. On the other hand, there was a significant difference in microhardness for specimens with thickness of 2 mm and 4 mm. Therefore, the third null hypothesis that resin cement's microhardness would be constant regardless of CAD/CAM type and thickness was partially rejected.

In this experiment, neutral density filters were used for beam profile measurement. According to the manufacturer, the OD filter has a transmittance according to the $T = 10^{-OD} \times 100 = \text{Percent Transmission}$ equation. Therefore a 3.0 OD filter used transmits only 0.1% of the light from the LCU. The beam profile showed that the light energy was highest in the center. It decreased toward the outside (Fig. 3). Therefore, the second null hypothesis that the beam profile passing through the CAD/CAM block would have a uniform profile regardless of the block's type or thickness was rejected. The beam profile result showed that the irradiance passing through the CAD/CAM block had a specific distribution. This particular irradiance distribution affected the activation of free radicals, which in turn affected the conversion rate and microhardness [28]. Therefore, depending on the LCU's direction for the tooth, this inhomogeneous irradiance could lead to resin cement's inhomogeneous polymerization and premature failure of the restoration [29].

In the present study, the inhomogeneous light energy that passed through CAD/CAM blocks also affected the resin cement's microhardness under thicker blocks. In both RelyX U200 and Variolink N cements, the resin cement under the CAD/CAM block had the highest at X0. It also showed an inhomogeneous microhardness that decreased toward the outer ($p < 0.05$). Therefore, the final null hypothesis that all parts would have a constant microhardness regardless of the distance to the center was rejected. All 1 mm blocks and most of 1.5 mm blocks showed relatively homogeneous microhardness. Blocks with thickness of 2.0 mm or 4.0 mm showed a non-homogeneous microhardness distribution ($p < 0.05$).

Considering the result of this study on the light beam's inhomogeneity, when a LCU is

positioned in the center of Class II indirect restoration, lower light energy would be delivered to the resin cement in the proximal box area because it would be the outer side of LCU. Moreover the thickness of indirect restoration is usually thicker than outer areas. However ISO 4049 [30] depth of cure test of luting materials assumes that the LCU's light output is uniformly distributed and that the specimen is subjected to the same irradiance over the entire surface. These shortcomings can lead to incorrect treatment in the clinic.

As a result of the present study, when using LCU with irradiance of $1,015 \text{ mW/cm}^2$, the value of 6 J/cm^2 or more described above can be viewed as the minimum total energy for obtaining a homogenous initial polymerization of resin cement with a $200 \mu\text{m}$ thickness. Similar to this study, Yap et al. [31] have reported that most resin composites with a depth of 1.5 mm are polymerized at $6\text{-}12 \text{ J/cm}^2$ and that appropriate surface hardness could be obtained by irradiation for 20 seconds at a low intensity of 200 or 300 mW/cm^2 . For uniform microhardness of resin cement, the total radiant exposure must be increased by sufficiently increasing the exposure duration. In our study, when light irradiation was performed with 2.0 mm thickness block, irradiance of about 150 mW/cm^2 was obtained (Table 3). Thus, it can be assumed that light irradiation for at least 50 seconds would be required for homogenous polymerization of resin cement. Likewise, at least 120 seconds would be required in 4.0 mm thick block. Similar to these exposure duration, an integrated review of resin cement stated the optimal conversion degree of resin cement can be achieved with LCU of 1400 mW/cm^2 and exposure time of 20 to 120 seconds [32].

The microhardness of resin cement can vary depending on the type of photo-initiators, monomer's composition, the ratio of the diluent, the content of the filler, and the polymerization method. Ruyter and Svendsen [33] have reported that the polymerization rate is decreased as the amount of aromatic monomers such as Bis-GMA is increased, while polymerization rate is increased as the amount of monofunctional and diluent monomers is increased. In addition, when radiopaque components such as Al, Ba, B, YbF₃, and Zr are included, microhardness may be changed [28]. Therefore, Bis-GMA

and Barium glass contained in Variolink N might have influenced the microhardness. Hofmann et al. [34] have measured the microhardness in three modes (dual-cured, light-cured, and chemical-cured) of resin cements, including Variolink 2. The dual-cured mode showed significantly higher values than light-cured and chemical-cured modes. The authors stated that physical properties were reduced when only the base paste was used. Similar results have been reported by Blackman and Warren [35, 36]. In our study, polymerization was carried out using only the base paste of Variolink N, which obtained relatively lower microhardness.

In Variolink N, there was no significant difference in microhardness with increasing time (T0, T1, T2) ($p > 0.05$). In RelyX U200, the microhardness value increased significantly after 24 hours (T1) and one week (T2) compared to immediately after polymerization (T0), but there was no significant difference between 24 hours (T1) and 1 week (T2) ($T0 < T1 \leq T2$) ($p < 0.05$). In response, Meng et al. [22] explained that early vitrification due to insufficient light activation can interfere with the subsequent self-polymerization, thereby reducing the dual-cured resin cement's overall polymerization. In addition, Jang et al. [37] reported on the effect of light exposure in self-adhesive dual-cured resin cement, which reduced the conversion rate with less light exposure. Therefore, the decrease in light transmittance by the CAD/CAM block would affect the initial polymerization of the dual-cured resin cement.

According to results of this experiment, since the type and thickness of the CAD/CAM block influenced the resin cement's microhardness, clinicians should be careful when using a thick or opaque CAD/CAM block. In such a case, increasing the polymerization time [26] or the temperature of the polymerization material (37 degrees or less) [38] can improve the degree of conversion of a resin cement. In addition, the type of resin cement can affect the clinical process. In experiments with light-cured resin cement and dual-cured resin cement, Koah et al. [39] have shown that sufficient polymerization could be obtained with only light irradiation up to 2.0 mm thickness.

Even with the same shade, the degree of light transmission and light reflection of the CAD/CAM block can be different depending on the composition and ratio [40]. The microhardness was decreased in the order of e.max HT \geq Lava Ultimate \geq Celtra Duo \geq e.max LT \geq Vita Enamic. In particular, in the case of 4.0 mm thick Enamic during the experiment, it was difficult to measure the microhardness for some cases because polymerization did not occur. Caprak et al. [41] measured the microhardness of dual-cured resin cement made of several types of Monolithic CAD/CAM materials. Similar to the result of the present study, Vita Enamic showed the lowest microhardness values for both dual-cured and light-cured modes [41].

LAVA Ultimate is a resin nano-ceramic CAD/CAM block containing nano-ceramic particles in a highly polymerized resin matrix. Nanofiller can improve the translucency because particles with a diameter smaller than the wavelength of visible light can generate less light scattering and absorbance [42, 43]. In contrast, Vita Enamic has a low translucency due to the relatively large amount of Al₂O₃. Therefore, Vita Enamic is used for inlays, onlays, and veneers. However, it is not recommended for full crowns due to debonding issues [44, 45].

Celtra Duo is a zirconia-reinforced lithium silicate that can be selectively heat treated. Since 10% zirconia is dissolved in the lithium silicate glass matrix, the silicate crystal is four times smaller. Thus, the glass content is high and the translucency is high. However, in Awad's study [44], heat-treated Celtra Duo shows higher translucency values than IPS e.max CAD LT. The authors explained that heat-treated Celtra Duo can increase physical properties and translucency because surface defects and porosity are reduced [44]. On the contrary, one study has shown that the translucency of silicate glass ceramic is decreased after multiple firing cycles. Previous studies have also reported less color change and translucency change in glazing-treated Celtra Duo [46, 47]. In the present study, e.max LT and Celtra Duo without heat treatment showed similar translucency values. According to an additional study following this study, translucency of Celtra Duo is decreased after glazing (Unpublished data).

This study revealed that the light inhomogeneity of the LCU might cause inhomogeneity in the polymerization of the resin cement under restoration. It was meaningful that appropriate energy for homogeneous polymerization of the resin cement was proposed. However, various studies are needed to prevent light inhomogeneity from causing problems clinically.

V. Conclusion

In the case of 2 mm and 4 mm thickness, both RelyX U200 and Variolink N showed an inhomogeneous distribution of the microhardness that decreased from the center of the light source (X0) to the periphery ($p < 0.05$). On the other hand, at thickness of 1 mm, microhardness showed relatively uniform distribution.

To obtain uniform polymerization of the resin cement under the CAD/CAM block, light energy at more than 6 J/cm^2 must be supplied to the resin cement.

The degree of polymerization of the resin cement in the center and the periphery of the irradiated region are different. Thus, it is necessary to make efforts to achieve homogeneous polymerization while moving the LCU in various positions and to increase the irradiation time for proper light energy.

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Abstract (In Korean)

광조사기 빛의 비균질성이 CAD/CAM block 하부 레진 시멘트의 미세경도에 미치는 영향

고유라

연세대학교 대학원

치의학과

(지도교수 박성호)

I. 목적

수복물을 통과하는 광중합기의 빛은 비균질한 분포를 보여준다. 본 연구의 목적은 빛의 비균질성이 각기 다른 종류와 두께의 CAD/CAM block 하부의 레진 시멘트 중합에 어떠한 영향을 미치는지 알아보기 위함이다.

II. 방법 및 재료

IPS e.max CAD, Celtra Duo, Lava Ultimate 및 Vita Enamic이 사용되었다. 각 블록의 두께는 1.0, 1.5, 2.0, 4.0 mm로 총 100개의 시편이 제작되었다.

레진 시멘트로는 RelyX U200이 이원중합형으로, Variolink N이 광중합형으로 사용되었다. 각 block의 광투과도와 beam profile이 측정되었고, 미세경도 측정을 위해 레진 시멘트 시편의 중심으로부터 주변까지 일정한 간격으로 레진 시멘트 위에 5개 지점 (-4 mm, -2 mm, 0 mm, +2 mm, +4 mm) 이 표시되었다. 각 지점에서 중합 후 즉시, 하루 뒤, 일주일 뒤에 미세경도를 측정하였다.

III. 결과

레진 시멘트의 미세경도는 CAD/CAM block의 두께가 증가함에 따라 감소하였다. e.max HT의 미세경도가 가장 높은 값을 보였고, Vita Enamic이 가장 낮은 값을 보였다 ($p < 0.05$). 1 mm 두께에서 레진 시멘트는 비교적 균일한 미세경도를 보였으나, 2 mm 이상의 두께에서는 비균질한 미세경도를 나타냈다 ($p < 0.05$).

IV. 결론

CAD/CAM block을 통과하는 beam profile은 두께와 유형에 따라 달라지며, 비균질하게 나타났다. 이는 레진 시멘트의 미세경도에 영향을 준다. 레진 시멘트의 균질한 중합을 얻기 위해서는 6 J/cm^2 이상의 광 에너지가 필요하며, 이를 위해서는 광중합 조사시간을 늘릴 필요가 있다. 또한 임상적으로는 광조사된 중심부와 주변부의 레진 시멘트의 중합 정도가 달라지므로, 광중합기를 여러 위치에서 움직이면서 균질한 중합이 이뤄지도록 노력해야 한다.

핵심되는 단어 : CAD/CAM blocks; resin cements; inhomogeneous; microhardness