치의학석사 학위논문

Effect of working time on the film thickness of dental resin cement

레진 시멘트의 혼합 후 시간에 따른
피막도 변화

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서울대학교 대학원
치의학대학원 치의학과
이 유 승

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지도 교수 김 성 훈

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치의학대학원 치의학과

이 유 승

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Yuseung Yi

Graduate School, Seoul National University

(Directed by Prof. ***Sung-Hun Kim****, D.D.S., Ph.D)*

ABSTRACT

Purpose. The objective of this study was to measure the film thicknesses of several resin cements altering the time duration between finishing of mixing and load.

Materials and Methods. Four kinds of resin cements were mixed according to the instruction manufacturers recommended and 5 kilogram loads were applied to the specimens for different lengths of time (20, 40, 60, 80, 100, 120, 140, 160 and 180 seconds. Film thicknesses were measured using a micrometer with an accuracy of 1 μm and the results were statistically analyzed by linear regression analysis and the working time recommended by manufacturers and the attained value calculated in this study were compared by Wilcoxon test ($\alpha = 0.05$).

Results. The film thicknesses of all resin cements increased as the time intervals between finishing of mixing or dispensing of cements and loading become longer and showed linear function patterns with positive correlation. There were discrepancies between the working time we calculated based on the results and the values manufacturers recommended.

Conclusion. As the time interval between the finishing of the mixing and load application be longer, the film thickness of resin cement increases. When we use resin cements in clinics, we should note that the working time, the duration from after the

mixing until the loading on the cement, at which the film thickness becomes 50 μm could be different from the value manufacturers recommend.

- **KEY WORDS:** Resin cement; Film thickness; Working time
- **STUDENT NUMBER:** 2010-22486

CONTENTS

I. INTRODUCTION

Indirect restorations with resin cement have been demonstrated to be effective with reduced micro leakage, increased retention and improved physical properties [1-5]. The success of prosthesis for a single tooth restorative treatment depends on retention and resistance of the prosthesis, the amount of tooth reduction, structural rigidity and marginal integrity, conservation of periodontal tissue and oral health management of patients [6]. Especially marginal integrity has crucial influence on successful prosthetic treatment [7]. However, the space between the prepared tooth and the prosthesis should be filled with cement or luting agent, therefore even the marginal integrity of prosthesis is perfectly accomplished, it is impossible to obtain complete marginal sealing and the more perfectly fit the inner surface of the prosthesis to the prepared abutment tooth, the lesser marginal sealing and adaptation for the reason of not escaping of the cement material from the interface [8]. Therefore, luting agents, joining indirect restorations to their preparations, are critical to their effectiveness [9,10].

For long-term clinical success rate of indirect restorations, in addition to possessing the low solubility and high ultimate strength, cement materials must also maintain a minimal film thickness over a long enough interval that restorations can be seated completely [10]. Film thickness is determined by a variety of factors including applied force during seating [11], rheology of the cement, grain size, geometry of the tooth and restoration surfaces [12] and time interval between the finishing of the mixing and the seating prostheses. The time available for flow before set may not allow all material sufficient opportunity to reach optimal minimal film thickness [10]. An increase in the film thickness of cement can cause incomplete seating of the restoration and significant exposure of luting agent to the oral environment [13].

There have been a lot of researches and studies accomplished dealing with the factors that have effects on the film thickness of resin cement and the impacts of the film thickness on both the bond strengths and stabilities on prostheses. Several established studies dealt with the film thicknesses of resin cements however, they measured the film thicknesses of the specimens at a time which ISO standard presents, if not, adopted a long time interval between the mixing and the load application [10]. That is, the duration after the mixing of cements which could affect the film thickness has not been focused. Therefore, in this study, we measure the film thicknesses of several resin cements clinically used recently according to the time duration proceeded after mixing and seating of the prostheses.

It has been suggested that the working time of a luting cement be defined as the time after the start of mixing in which a film thickness of 25 μm for water-based luting cements [14] and of 50 μm for resin-based cements which is reflected in current ISO standards [15]. According to the test described in ISO for the measurement of the film thickness of resin-based luting materials, after the mixing of cements, a 150 N load is applied for 180 seconds and then light curing is accomplished without load pressure and it is not mentioned about the time interval between the mixing and load application. Instead, 50 N load (5 kilograms) was used in this study, which based on the Zortuk *et al.* that the finger pressure applied during cementation by clinicians approximately ranged from 12 to 65 N [16], and light curing carried out simultaneously through the hole on the center of the load which make it more alike to the clinical condition.

The purpose of this study was to verify this hypothesis: film thickness of each cement increases as the time interval between the finishing of the mixing and load application be longer.

II. MATERIALS AND METHODS

The film thickness was determined using the test described in ISO for polymer-based restorative materials [15], except that the loading applied was 5 kilograms and using Mylar matrix strip for the cements not to stick to glass plates. Four adhesive luting resins were evaluated in this study (Table 1). Tests were conducted at 20℃ ± 2℃ and with a relative humidity of 50% ± 10%, and all materials were manipulated exactly as described in the manufacturer's instructions using supplied dispensers and/or auto-mixing syringe tips when applicable. When required, hand mixing was performed for the recommended time after extrusion onto a sheet of coated paper.

Ten specimens were designed for each cements. After each manufacturer's recommended mixing time, the materials were placed between two identical translucent Mylar matrix strips on the optically flat glass plate. For hand-mixed cements, the cement mix was applied to the Mylar matrix strip immediately after mixing completed, whereas the auto-mixed cements were dispensed directly onto the bottom strip. The amount of cement was not determined exactly; instead, the minimum amount was placed not to overflow over the strips. All these procedures were processed by one trained operator to avoid bias. A defined load was applied in 20, 40, 60, 80, 100, 120, 140, 160, 180 and 200 seconds after the finishing of mixing or dispensing for each cement. Covering the specimens with a flat glass plate, cylinder shaped static load of five kilograms, which had a hole in the center was applied vertically to the glass plate for a period of curing with LED through the hole for 5 seconds. All these procedure repeated three times.

Film thickness was measured using a micrometer (Absolute Digimatic, Model 293-805; Mitutoyo Corp, Kanagawa, Japan) with an accuracy of 1 μm. Three individual thickness measurements were made on each polymerized specimen followed by subtraction of thickness of Mylar strips and then averaged to represent the thickness of each specimen.

The results of this study were statistically analyzed using linear regression analysis and the working time recommended by manufacturers and the attained value calculated in this study were compared by Wilcoxon test ($\alpha = 0.05$).

Table 1. Luting cements tested in the study

Product	Manufacturer	Characteristics	Mixing method	Lot No.
Panavia F2.0	Kurary America, NY, USA	Composite resin Dual polymerized	Proprietary dispenser, Hand mixed 20 sec	A: 00543A B: 00104A
Clearfil SA luting	Kurary America, NY, USA	Self-adhesive resin Dual polymerized	Proprietary dispenser, Hand mixed 10 sec	0008BA
Zirconite	BJM Lab., Israel	Self-adhesive resin Dual polymerized	Proprietary dispenser, Automixed	400050
RelyX U200	3M ESPE, MN, USA	Self-adhesive resin Dual polymerized	Proprietary dispenser, Hand mixed 10 sec	493661

III. RESULTS

The film thicknesses of all of the cements tested are given in Fig. 1. The line of 50 μm is also represented for comparison with because the proposed ISO resin cement specification specifies 50 μm film thickness limit [15]. For all cements a minimum film thickness of 50 μm or lower was obtained, but the maximum time which sustained below 50 μm was different. The film thicknesses of all resin cements tested increased as the time intervals between finishing of mixing or dispensing of cements and loading with light curing longer and showed linear function patterns with positive correlation. Fig. 2 represents the film thicknesses of each cement in the study and Table 2 shows linear regression analysis results.

RelyX U200 shows the most linear increasing pattern with $R^2 = 0.950$ and more rapid increase than any other cement on the linear regression analysis. The moment when the thickness reached to the value of 50 μm was recorded more fast than any other cement, which 75.3 seconds calculated from the linear regression equation (Fig. 5).

Clearfil SA luting shows linear pattern but not as much as RelyX U200 ($R^2 = 0.624$). It tends to increase rapidly for initial 60 seconds and be sustained at the level and increase again after 140 seconds. The moment when the thickness reached 50 μm was 169.4 seconds based on the linear regression analysis (Fig. 3).

Zirconite increases very linearly as a function of time initially although suddenly rises after 180 seconds which makes the lowest R^2 value of 0.720. The moment when the thickness reaches 50 μm was 119.88 seconds based on the linear regression analysis (Fig. 4).

Panavia F2.0 also increases in linear pattern with $R^2 = 0.785$ and the moment when the thickness reaches 50 μm was 140.5 seconds (Fig. 2)

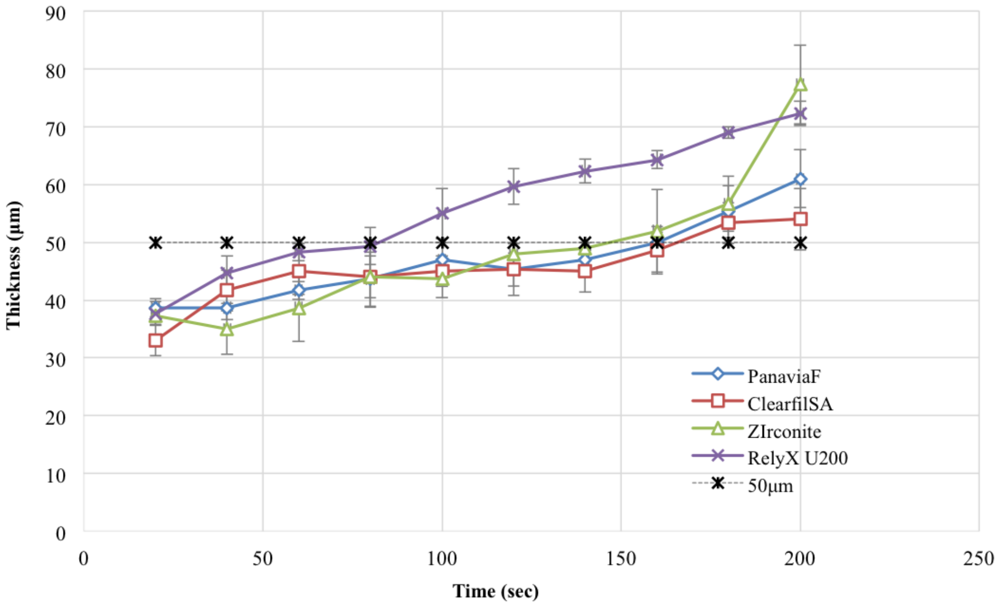

Fig. 1. The film thickness in relation to time after the mixing completed. All data represent the mean values of the results of each material.

Table 2. Linear regression analysis of Panavia F 2.0

Variables	Correlation coefficients		*B*	β
	Film thickness	Time		
Film thickness	-			
Time	.886**	-	.111**	.886
		Constant	= 34.58	
Mean	46.83	110.0		R^2 = .785
S.D.	7.35	58.43		*Adjusted* R^2 = .777
				R = .886

* *P* < .05, ** *P* < .01
B: Unstandardized regression coefficient; β: standardized regression coefficient

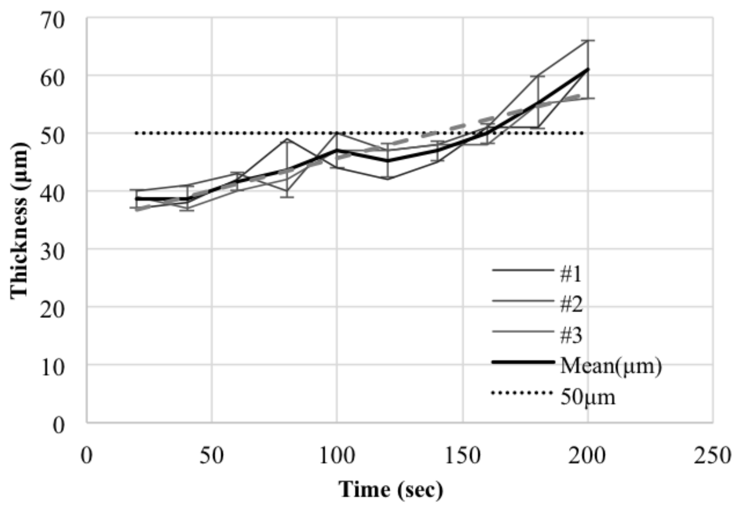

Fig. 2. The film thickness of Panavia F 2.0 in relation to time after the mixing completed. These show results of three times experiments and the mean values of those results. Square-dotted lines represent trend lines according to linear regression analysis.

Table 3. Linear regression analysis of Clearfil SA Luting

Variables	Correlation coefficients		*B*	β
	Film thickness	Time		
Film thickness	-			
Time	.790**	-	.089**	.790
		Constant =	35.76	
Mean	45.50	110.0		R^2 = .624
S.D.	6.55	58.43		*Adjusted* R^2 = .610
				R = .790

* *P* <.05, ** *P* <.01
B: Unstandardized regression coefficient; β: standardized regression coefficient

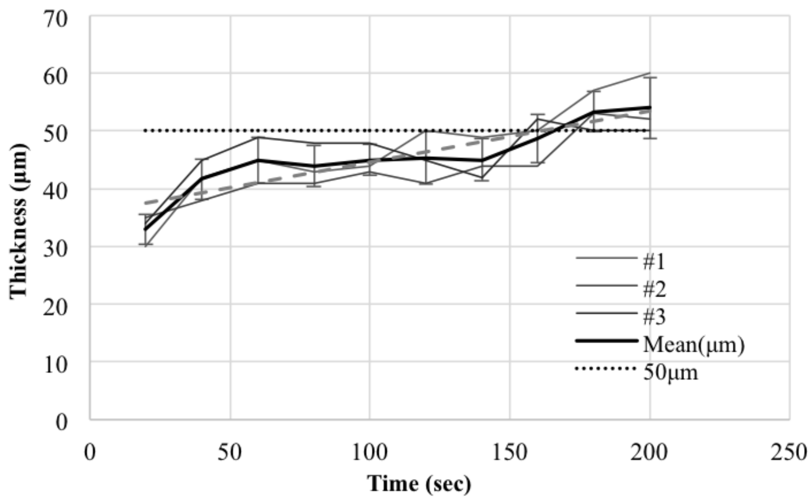

Fig. 3. The film thickness of Clearfil SA Luting in relation to time after the mixing completed. These show results of three times experiments and the mean values of those results. Square-dotted lines represent trend lines according to linear regression analysis.

Table 4. Linear regression analysis of Zirconite

Variables	Correlation coefficients		*B*	β
	Film thickness	Time		
Film thickness	-			
Time	.849**	-	.181**	.849
		Constant =	28.24	
Mean	48.17	110.0		R^2 = .720
S.D.	12.47	58.43		*Adjusted* R^2 = .710
				R = .849

* *P* <.05, ** *P* <.01
B: Unstandardized regression coefficient; β: standardized regression coefficient

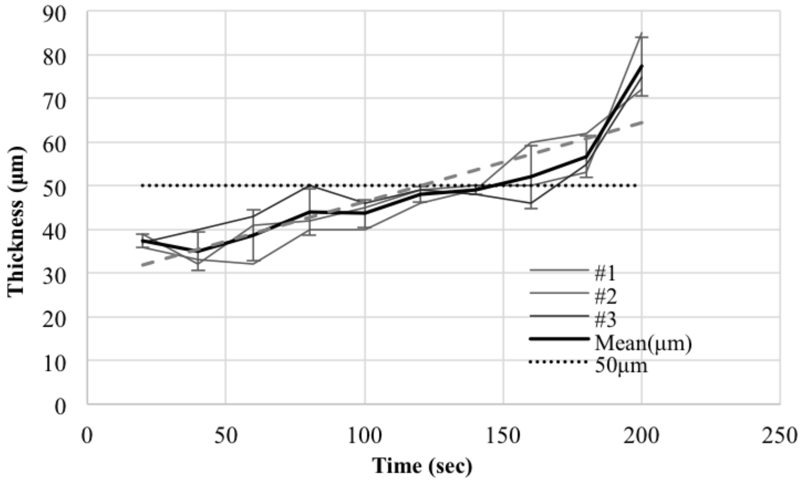

Fig. 4. The film thickness of Zirconite in relation to time after the mixing completed. These show results of three times experiments and the mean values of those results. Square-dotted lines represent trend lines according to linear regression analysis.

Table 5. Linear regression analysis of RelyX U200

Variables	Correlation coefficients		*B*	β
	Film thickness	Time		
Film thickness	-			
Time	.975**	-	.184**	.975
		Constant =	36.07	
Mean	56.27	110.0		R^2 = .950
S.D.	11.00	58.43		*Adjusted* R^2 = .948
				R = .975

* *P* <.05, ** *P* <.01
B : Unstandardized regression coefficient; β : standardized regression coefficient

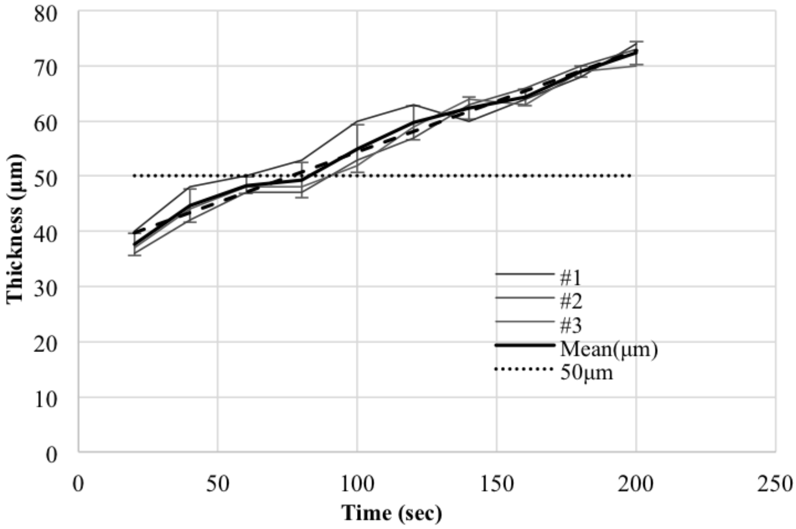

Fig. 5. The film thickness of RelyX U200 in relation to time after the mixing completed. These show results of three times experiments and the mean values of those results. Square-dotted lines represent trend lines according to linear regression analysis.

IV. DISCUSSION

As expected, film thickness of all cements in this study were influenced by time interval between the finishing of the mixing and the load application, which shows linear function patterns with positive correlation. Almost resin cements used in clinics are Type III dual-cure cements, which means they start polymerizing with start of mixing. Therefore it is obvious that the longer the time delay before load application, the thicker the resin cement which makes bonding strength and other physical properties weaker. For this reason the manufacturers recommend appropriate working time for each resin cement which reflect the ISO standard for Type I and Type III cements [15]. As mentioned above, we examined the working time when the film thicknesses get to 50 μm in the way of more similar to clinical condition. Table 6 shows the manufacturer recommended working time and the time evaluated the at which film thickness reached 50 μm in this study and statistical analysis is also represented. All the asymptotic significances are 0.109 which means there is no significant difference between the working time manufacturers' recommended and the attained value in this study.

Table 6. Working time manufacturer recommended and the time the thickness reached 50 μm

Product	Recommended working time	Attained value		Mean Differences	*P* value
			Mean		
Panavia F2.0	3 min. after mixing	149.71 121.47 150.39	140.52 (16.50)*	-39.48	.109
Clearfil SA Luting	60 sec. after mixing	182.12 133.91 192.14	169.39 (31.13)	+109.39	.109
Zirconite	1 - 2 min.	117.99 124.25 117.40	119.86 (3.80)	+29.88	.109
RelyX U200	2 min. from start of mixing	80.60 84.02 61.35	75.32 (12.22)	-34.68	.109

*Figures in parenthesis refer to standard deviation.

According to the manufacturers of each resin cements, Panavia F2.0 has the longest working time of 3 minutes after mixing and Clearfil SA Luting has the shortest working time of 60 seconds after mixing. However, recorded results in this study have not reflected the manufacturers' recommended working time represented through the ISO standard. Clearfil SA Luting and Zirconite show the attained working time in this study greater or similar to the value manufacturers recommended, though Panavia F2.0 and RelyX U200 have shorter working time attained than the recommended values. Zirconite has the recommended working of a wide range, 1-2 minutes, therefore when compare to the average, 90 seconds, the attained value in this study was 30.1 seconds greater than the manufacturers' recommended working time and it is the lowest difference among the all cements studied. Clearfil SA Luting has longer working time than any other cement used in this study and it shows the greatest difference from the manufacturers' recommendation.

Several established studies dealt with the film thicknesses of resin cements however, they measured the film thicknesses of all the specimens just once after identical time interval, if not, adopted a long time interval between the mixing and the load application. Therefore, this study represents in a regard that the film thicknesses of resin cements were measured in a more segmented time interval, 20 seconds, between after the mixing and the loading. The load applied, 5 kilograms, was more approximated to the clinical condition, rather other studies have used 150 N for loading.

In our study, we measured the changes of film thicknesses of resin cements during cementation in the clinical environment and calculated more appropriate working time than the manufacturers' specified based on the ISO standard. Though, there is a limitation that the environment is different at each clinics and it could be varied the properties of resin cements according to the clinicians treating the materials.

In summary, we have shown that the film thicknesses of resin cements increase as the time intervals between the finishing of mixing and the load application are prolonged in a linear function patterns. Also we calculated the working time based on the ISO definition that the time interval which film thickness approaches 50 μm. And as a result of the experiment carried in the more clinical condition, there were discrepancies between the values we calculated and the working times manufacturers recommended. We found that Clearfil SA luting and Zirconite have greater or similar working time that the values manufacturers specified, and Panavia F2.0 and RelyX U200 attained shorter working time. Therefore, when we use Panavia F2.0 and RelyX U200 in clinics, it is needed to treat the materials until seating the prostheses more rapidly than the working time manufacturers recommended.

V. CONCLUSION

As the time interval between the finishing of the mixing and load application be longer, the film thickness of resin cement increases. When we use resin cements in clinics, it should be noted that the working time, the duration from after the mixing until the loading on the cement, at which the film thickness becomes 50 μm could be different from the value manufacturers recommend. Therefore, it is needed to treat the materials until seating the prostheses more rapidly than the working time manufacturers recommended.

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국문초록

레진시멘트의 혼합 후 시간에 따른 피막도의 변화

서울대학교 대학원
치의학대학원 치의학과
지도교수 김 성 훈
이 유 승

연구목적: 본 연구의 목적은 임상에서 이용되는 몇 가지 레진 시멘트를 이용하여 혼합 후 부하를 가하기까지 소요되는 시간에 변화를 주어 그에 따른 경화 후의 피막도를 측정하여 혼합 후 작업시간과 레진 시멘트의 피막도의 관계를 보고자 하는 것이다. 또한 실험결과를 이용하여 적합한 작업시간을 계산하여 제조사가 제시한 작업시간과 비교해 보고자 한다.

재료 및 방법: 네 종류의 레진시멘트(Panavia F2.0, Clear SA Luting, Zirconite, RelyX U200)를 제조사의 지시대로 혼합한 후 투명한 두 장의 Matrix Mylar strip 사이에 소량의 시멘트를 넣고 다양한 시간간격(20, 40, 60, 80, 100, 120, 140, 160, 180, 200 초) 후에 5 킬로그램의 부하를 가하여 광중합을 시행하였다. 경화 이후 피막도는 1 μm 단위까지 측정 가능한 디지털 피막도 측정기를 이용하여 각 시편 당 세 번씩 측정하여 평균값을 선택하였으며, 각각의 측정결과는 선형회귀분석을 시행하였다($\alpha = 0.05$).

결 과: 본 연구에 사용된 모든 레진 시멘트에서 혼합 후 부하를 가하기까지 걸린 시간이 길어질수록 경화 후의 피막도가 증가하는 결과를 보였으며,

선형회귀분석결과 양의 상관관계를 갖는 일차함수적 패턴을 얻을 수 있었다. 혼합 후 지연된 시간에 대한 피막도의 선형 추세선에서 작업시간에 대한 ISO기준에 따라 피막도가 50 μm가 되는 지점의 시간을 계산해본 결과 각 레진 시멘트의 제조사가 제시한 작업시간과 본 연구 결과를 토대로 계산한 작업시간사이에는 차이가 존재하였다. 제조사가 제시한 작업시간과 가장 유사한 값을 보인 것은 Zirconite였으며, Clearfil SA Luting은 실험결과 더 큰 값을 나타내었고, Panavia F2.0과 RelyX U200은 더 작은 값을 보였다.

- 주요어: 레진시멘트, 피막도, 작업시간
- 학 번 : 2010-22486