치의학석사 학위논문

Rheological properties of dental resin cements during polymerization

경화 중 레진 시멘트의 유변학적 특성

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서울대학교 대학원

치의과학과 치과보철학 전공

이 재 림

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Rheological properties of dental resin cements during polymerization

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CONTENTS

ABSTRACT

Purpose. The purpose of this study was to observe the change of viscoelastic properties of dental resin cements during polymerization.

Materials and Methods. Six commercially available resin cement materials (Clearfil SA luting, Panavia F 2.0, Zirconite, Variolink N, RelyX Unicem clicker, RelyX U200) were investigated in this study. A dynamic oscillation-time sweep test was performed with AR1500 stress controlled rheometer at 32°C. The changes in shear storage modulus (G'), shear loss modulus (G"), loss tangent (tan δ) and displacement were measured for twenty minutes and repeated three times for each material. The data were analyzed using one-way ANOVA and Tukey's post hoc test (α=0.05).

Results. After mixing, all materials demonstrated an increase in G' with time, reaching the plateau in the end. RelyX U200 demonstrated the highest G' value, while RelyX Unicem (clicker type) and Variolink N demonstrated the lowest G' value at the end of experimental time. Tan δ was maintained at some level and reached the zero at the starting point where G' began to increase. The tan δ and displacement of the tested materials showed similar pattern in the graph within change of time.

The displacement of all 6 materials approached to zero within 6 minutes. Except Variolink N, initial setting time of the other five resin cement materials was less than 6 minutes.

Conclusion. Each dental resin cement showed differences in rheological properties and handling characteristics. Compared to other resin cements used in this study, RelyX U200 maintained plastic property for a longer period of time. When it completed the curing process, RelyX U200 had the highest stiffness. It is convenient for clinicians to cement multiple units of dental prostheses simultaneously.

Keywords: Dental resin cement, Rheological properties, Film thickness, Viscoelasticity
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I. INTRODUCTION

To make a successful restoration, complete seating of the fixed dental prostheses to abutments is as important as fabricating the accurate casting. Unless excess cement escape entirely during cementation, crown will be elevated from the correct position [1]. There were many efforts to improve the seating of crowns. The methods include increasing cement space [2], venting [3], oscillation loading [4], high seating force [5], applying vibration at the beginning of cementation [6], using pressure applicator [7], selecting and applying of appropriate dental cements. Manipulation of dental cements is concerned with types, film thickness and rheological properties such as flow, viscosity and elasticity [8]. Most of the cements flow easily after mixing. Therefore, dentists are able to completely seat the fixed dental prostheses. As the setting procedure progress, viscosity of cement materials increases which makes it easier to flow under pressure. There are time-dependent properties that affect working time and setting time [9].

The type of dental cements which were commonly used in the past was zinc phosphate cement and resin modified glass ionomer cement. But, they serve as luting agent, not chemically bonding to teeth. Therefore, retention form of prepared tooth is a critical factor to long term success [10]. Their solubility, acidity at early period is also a limitation of zinc phosphate cement [9]. Currently the use of resin cement have been increased due to their superior strength, improved retention, ability to modify color and insolubility in the oral condition [10]. Most of the resin cements have an unacceptable film thickness which is higher than ANSI/ADA specification No. 96, hence it frequently leads to incomplete seating of crown [9, 11]. There are many studies which have been reported on the rheological properties of dental cement such as viscosity and elasticity. They were time-dependent properties that affect working time and setting time [9].

However, there are few studies that dealt with rheological properties of resin cements. The purpose of this study was to observe the change of viscoelastic properties of resin cement materials during polymerization. The null hypothesis to be tested is that there is no difference in the rheological properties of dental resin cements during polymerization.

II. MATRIALS and METHODS

Six commercially available resin cement materials were investigated in this study. Their proprietary names, composition, filler contents, particle size and manufacturers are listed in Table 1. All materials were mixed in room temperature at 25^{o}C ± 1^{o}C according to the manufacturer recommendation and 60% relative humidity as the manufacturer directed. For Variolink N and Panavia F 2.0, base and catalyst ratio (1:1) was followed using 1 mL syringe.

To monitor the rheological properties in the mixed resin cement materials during polymerization, a dynamic oscillation-time sweep test was performed using a AR1500 stress controlled rheometer (TA instruments, Leatherhead, Surrey, UK). A parallel stainless steel plates with diameters of 25 mm were used and the gap between the two plates was set as 500 μm. In order to reproduce an environment of oral cavity, the temperature of Peliter plate was set at 32 ^{o}C. After loading 0.25 mL of mixed resin cement onto the lower plate, the changes in storage modulus (G'), loss modulus (G''), and loss tangent (tan δ) were recorded for twenty minutes at an oscillating frequency of 1 Hz and a maximum torque of 3000 μNm. The time from the starting of mixing to the first recording of rheological properties was less than 70 seconds. Measurements were taken every 9 seconds during 20 minutes. Above process were repeated three times for each material.

The data were analyzed using one-way ANOVA and Tukey's post hoc test ($\alpha = 0.05$). In Addition, a correlation analysis was performed to examine the relations between experimental results and setting time provided by the manufacturers.

Table 1. Dental resin cement materials included in this study.

Material	Manufacturer	Bat. No.	Composition	Filler contents	Mean particle size(μm)
RelyX U200 (Automix)	3M ESPE (MN, USA)	487972	Bi-functional (meth)acrylate	43 wt%	12.5
RelyX Unicem (Clicker type)	3M ESPE (MN, USA)	474045	Bi-functional (meth)acrylate	70 wt%	12.5
Clearfil SA Luting (Clicker type)	Kuraray (Noritake Dental Inc., Okayama, Japan)	0374AA	Paste A : Bis-GMA, TEGDMA, 10-MDP, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal sillica, di-Camphorquinone, Initiators, Benzoyl peroxide Paste B : Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatix dimethacrylate, Silanated barium glass filler, Silanated colloidal sillica, Accelerators, Pigments	44 vol% 65 wt%	2.5

Table 1 (Continued). Dental resin cement materials included in this study.

Panavia F 2.0	Kuraray (Medical Inc., Osaka, Japan)	0558AA 0104AC	A paste: 10-MDP, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, Silanated silica filler, Silanated colloidal silica, di-Camphorquinone, catalyst, Initiator B paste: Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Silanated barium glass filler, Surface treated sodium fluoride, Catalyst, Accelerators, Pigments	59 vol%	0.04 - 19
Zirconite (Automix)	BJM LAB (Israel)	4194HQB ARCZKR	Bis-GMA, UDMA Oligomer, TEGDMA, 4-META, Methacrylated phosphoric acid esters, [3-(Methacryloyloxy)propyl]trimethoxysilane, photoinitiator, Co-initiator, Benzoyl peroxide, Barium aluminoborosilicate glass, fumed silica	-	-
Variolink N	Ivoclar vivadent (Schaan, Liechtenstein)	P72407 (Base) R68646 (Catalyst)	Bis-GMA, UDMA, TEGDMA, Inorganic filler(barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, pheroid mixed oxide, Initiator, Stabilizer, Pigment	44 vol% 71 wt%	0.7 (0.04 - 3.0)

III. RESULTS

The changes of rheological parameters in the mixed resin cement materials during polymerization are shown in Fig. 1 to Fig. 4. After mixing, the G’ remains almost zero at first. However, each material exhibited an increase in G’ with time. In the end, the G’ value reached the plateau. The change of G’ value demonstrated sigmoidal curve. Each material differs significantly in the amount of required time to reach the plateau and maximum value of the shear storage modulus ($P < 0.05$) (Fig. 1). RelyX U200 demonstrated the highest G’ value while RelyX Unicem and Variolink N demonstrated the lowest G’ value. To arrange in order of G’ at the plateau, RelyX U200, Zirconite, Panavia F, Clearfil SA luting, RelyX Unicem and then Variolink N were followed.

Initial setting time is defined as amount of time until beginning of the development of elasticity, such as increase of the value of G’ or decrease of the value of tan δ. The RelyX Unicem was the first to begin setting reactions. It was followed by Clearfil SA luting, Zirconite, RelyX U200, Panavia F 2.0 and Variolink N ($P < 0.05$). However, there were no significant differences between RelyX Unicem, Clearfil SA luting and

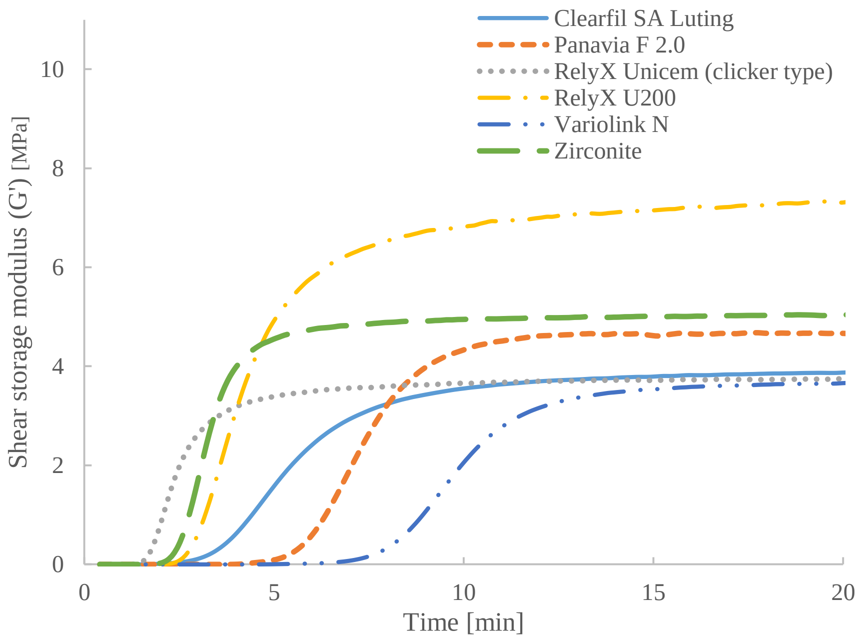

Fig. 1. The representative of shear storage modulus (G') of dental resin cements investigated. The increase of G' means the progression of the cross-linking and entanglements of polymer chains. It means that the material shows the onset of elasticity or loss of fluidity. Initial setting time is defined as amount of time until beginning of the development of elasticity, such as increase of the value of G' or decrease of the value of tan δ.

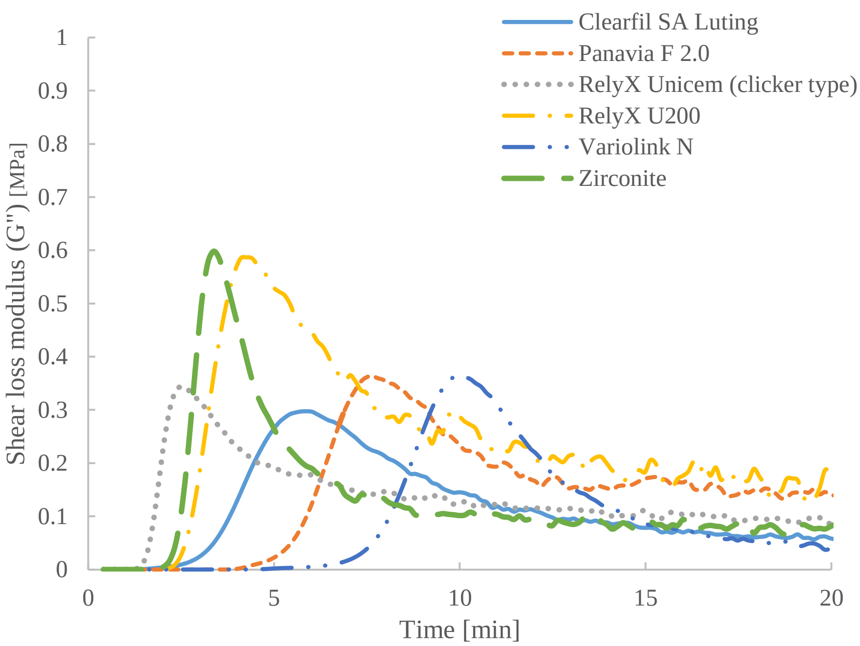

Fig. 2. The representative of shear loss modulus (G") of dental resin cements investigated.

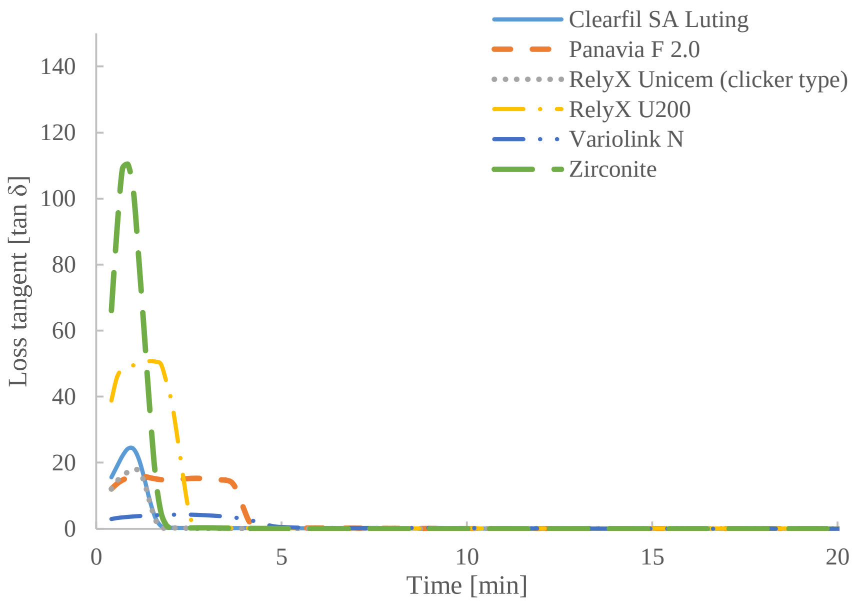

Fig. 3. The representative of loss tangent (tan δ) of dental resin cements investigated. The value of tan δ depends on the ratio of G" to G'. It represents loss of plasticity and corresponds with tendency of displacement curve.

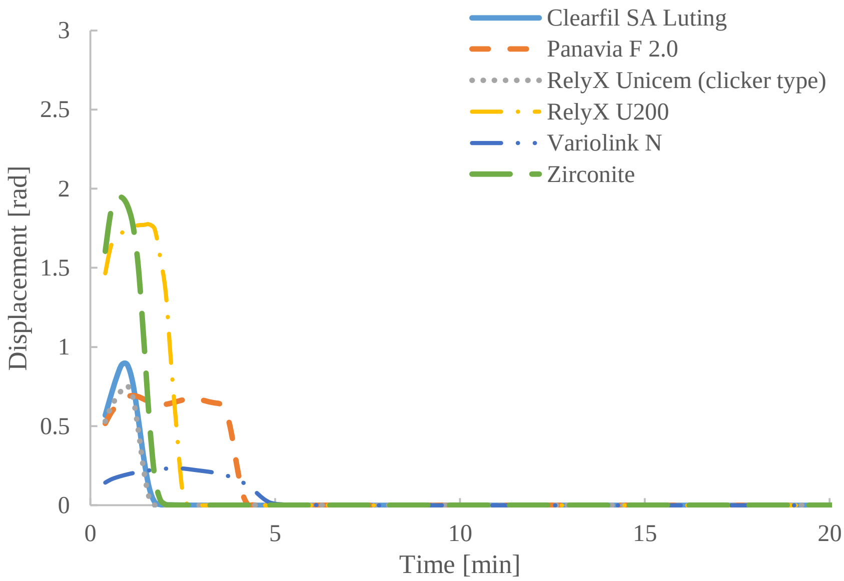

Fig. 4. The representative of displacement of dental resin cements investigated.

Zirconite. The values obtained were longer than the working time which was provided by manufacturers, but still both numbers showed high correlation (r = 0.978, $P < 0.05$).

In order to estimate the setting time, the time required to reach the plateau was needed. However, the setting reaction of the dental resin cement materials may last for hours. Therefore setting time is considered as the time required to reach 90 % of maximum shear storage modulus (Table 2). All the materials show a different setting time ($P < 0.05$). Against the manufacturer's instruction, the value obtained in this study was longer (1.17 to 2.65 times). In addition, contrast to the working time, setting time showed low correlations (r = 0.118, $P > 0.05$).

RelyX Unicem is fastest setting resin cement, while Variolink N exhibits slowest ($P < 0.05$). The G" increases until reaching a peak value, and then decreases to some level gradually (Fig. 2).

Tan δ was maintained at certain level for a certain period of time until storage modulus started to increase then it reached zero. Zirconite shows highest peak in tan δ, then rapid decrease with time. RelyX U200, Panavia F 2.0 and Variolink N maintained plateau at low value of tan δ

Table 2. The storage moduli (G') and loss tangent (tan δ) at different elapsed times, setting times for the dental resin cement materials during polymerization at 32 °C.

Material	t = 1 min		t = 5 min		t = 20 min	
	G' (Pa)	tan δ	G' (Pa)	tan δ	G' (Pa)	tan δ
RelyX U200 (Automix)	0.41	50.04	5.07E+06	0.10	7.33E+06	0.02
RelyX Unicem (clicker type)	2.81	18.46	3.41E+06	0.06	3.74E+06	0.02
Clearfil SA Luting	2.55	21.66	1.71E+06	0.16	3.88E+06	0.01
Panavia F 2.0	3.46	16.14	1.14E+05	0.24	4.65E+06	0.03
Zirconite	0.30	81.45	4.59E+06	0.05	5.04E+06	0.02
Variolink N	57.91	3.83	5.75E+03	0.46	3.67E+06	0.01

during long duration. Especially, Variolink N shows lowest value of tan δ for a longest duration.

The tan δ and the displacement of the tested materials showed similar pattern in the graph within change of time. The displacement of all six materials approached to zero within 6 minutes after start of mixing. According to Table 3, five materials except Variolink N had the setting time less than 6 minutes. After the initial setting, change in the displacement was not observed.

Table 3. The initial setting time, setting time and G' at the plateau different elapsed times, setting times for the dental resin cement materials during polymerization at 32 °C.

Material	Initial setting time (s)	Setting time at T_{90} (s)	Shear storage modulus (G') (MPa)
RelyX U200	197.50 (13.42)^{c}	538.13 (24.95)^{g}	7.25 (0.22)^{i}
RelyX Unicem	150.16 (5.04)^{d}	352.70 (22.61)^{h}	3.73 (0.11)^{l}
Clearfil SA Luting	156.73 (8.92)^{d}	613.20 (5.45)^{f}	3.84 (0.10)^{l}
Zirconite	168.22 (76.82)^{d}	356.46 (9.95)^{h}	5.02 (0.07)^{j}
Panavia F 2.0	303.37 (5.23)^{b}	635.6 (14.57)^{f}	4.66 (0.10)^{k}
Variolink N	338.80 (11.20)^{a}	801.98 (24.86)^{e}	3.62 (0.09)^{l}

· Numbers in parentheses are standard deviations.
· The same superscript letters in the same column indicate no significant differences respectively (Tuckey's post hoc test: $P > 0.05$).

IV. DISCUSSION

Film thickness, working time and setting time are important characteristics of dental cement when the clinician uses the materials during clinical process. There were many studies to monitor the development of elasticity and to determine the working time and setting time of dental cements [9, 12-17].

Batchelor and Wilson used the parallel plate plastimeter to measure the effect of humidity and temperature in zinc oxide-eugenol cements. However the parallel plate plastimeter ran short because they could not measure the rate of flow [18]. Plant *et al.* compared the various cements using the oscillating rheometer. The change of fluidity of dental cements was observed according to the type of cement, temperature and mixing ratio of powder/liquid [19]. Vermilyea *et al.* measured the viscosity of zinc phosphate and polycarboxylate cements by rotational viscosimeter during setting. Change of viscosity was monitored according to the increasing shear rate and temperature. However, rotational viscosimeter had limitation of low shear rate at the margin of crown comparing to the clinical condition during cementation. The data from this study does not matched actual clinical practice [20]. Lorton *et* *al.* developed a new rheometer that is essentially a ram and piston penetrometer. They made a change in crosshead speed and monitored a resistance of various cements at a specific crosshead speed which was determined from the preliminary studies. The shear rate dependence on the viscosity of dental cement materials was considered for a clinical evaluation of optimal utilization [15].

Vlachodimitropoulos and Wilson used displacement rheometer to monitor the development of elasticity of dental cement materials such as glass-ionomer and polycarboxylate cements. This instrument could detect and monitor the viscoelastic changes of dental cements. Therefore the working time of the cements could be inferred [13].

Osman *et al.* used a controlled stress rheometer to monitor the changes in rheological properties such as viscosity and tan δ, in various types of dental luting cements including resin cements. The objective is to compare the film thickness and rheological properties of dental luting cements. As a result, resin base luting cements performed low film thickness equivalent to the traditional zinc phosphate cement. Film thickness was dependent on a complicated effect of rheological properties and it was unable to be predicted a single major contributing factor such as viscosity or tan δ [14].

Recently, many studies have been reported for evaluating rheological properties of dental materials using a controlled stress rheometer [21, 22]. The controlled stress rheometer with an oscillatory mode has advantages against understanding rheological properties of dental elastomer during polymerization. A shear force at fixed torque value was applied between Peltier plate and deformation was recorded as setting reaction was progressed. The minute viscoelastic changes of setting elastomers were able to detect in real time and were plotted as a graph on the software. Furthermore stress controlled rheometer could provide a value of shear storage modulus, shear loss modulus and loss tangent so that the clinician could predict the information about setting time and working time. Using this instrument, we could monitor the various rheological properties in real time.

The dynamic oscillatory time sweep test is important when setting elastomer or polymer that may undergo macro- or micro-structural rearrangement with time. These arrangements directly influence rheological behavior. This test directly provides the necessary information about how a material changes with time. In this study, a dynamic oscillation time sweep test was conducted with AR 1500 stress controlled rheometer. The G’, G”, tan δ and displacement of dual cure resin cement materials were measured during polymerization.

After mixing as manufacturer directed, mixed materials were put on the lower Peltier plate which was in controlled temperature. The upper plate began to descent and applied oscillatory shear strain such as sinusoidal movement to the lower plate. If test materials were 100 % elastic, they react exactly like the strain given regardless of time. The more elastic the material is, the closer the phase angle becomes 90˚. However, dental resin cements were viscoelastic so the response of deformation was observed with change of phase angle (δ) and amplitude (σ) as setting procedures were progressed. The elastic and viscous component could be calculated separately at specific point of time.

$$\text{Total stress} = \sigma + \tau = A\,(E \sin \omega t + \eta\omega \cos \omega t)$$

$$= A\,\sqrt{E^2 + (\eta\omega)^2}(\cos\delta\ \sin\omega t + \sin\delta\ \cos\omega t)$$

$$= \|\sigma *\| \sin(\omega t + \delta)$$

$$\{\|\sigma *\| = A\sqrt{E^2 + (\eta\omega)^2}\}$$

$$\text{Elastic component (E)} = \frac{\|\sigma *\|}{A} cos\delta$$

$$\text{Viscous component } (\eta) = \frac{\|\sigma *\|}{A} sin\delta$$

We obtained value of G', G" and tan δ of the tested dental resin cements. The G' remains almost zero immediately after mixing, increases with time and finally reaches to a plateau. In curing progress of dual cure resin cement, G' remains low until crosslinks and entanglements are formed between the growing chains which allow the material to resist against the applied stress. The increase of G' with time means the progression of the cross-linking and entanglements of polymer chains [17, 23]. The completion of the polymerization is referring to the plateau of storage modulus. The definition of setting time was the point at which the increasing slope meets the plateau [22]. However, the setting reaction of dental resin cements lasted more than twenty minutes. Therefore we measured the amount of time it takes for the shear storage modulus to reach 90 % of maximum. The setting time obtained in this study was longer than that of manufacturer's instruction. Against the manufacturer's information, the value obtained in this study was longer (1.17 to 2.65 times). In addition, there was significantly no correlations between them (post hoc test; r = 0.118, P > 0.05). The dental resin cements to be tested were dual-cure cement that passes through a process of chemical cure and light cure. To evaluate the initial setting time, setting reaction by light cure was ruled out. Therefore the setting time would be abbreviated in clinics by light curing. RelyX Unicem and Zirconite set faster than other dental resin cements. On the other hand, Clearfil SA Luting and Variolink N set slowly without curing by light.

The average value of G’ of the last three minutes refers to rigidity or stiffness of resin cement materials after setting. RelyX U200 shows highest shear storage modulus and shows more rigid properties after polymerization. It is relevant to remove the excess cement after cementation.

The initial setting time is defined as the initial point at which the G’ increases. This is explained by onset of elasticity and loss of fluidity and also equal to working time. In all of the resin cement materials tested in this study, the time when G’ value began to increase was longer than the working time provided by the manufacturer. However initial working time obtained from this study demonstrated high correlation with the working time which is provided by manufacturers (r = 0.978).

Loss tangent was maintained at a certain level for a short time and then reached the zero. The point when loss tangent reached zero and initial starting point of storage modulus were similar. Zirconite shows the highest peak value and rapid decrease with time. Zirconite is closest to complete viscous body among the tested materials and rapid decrease in a loss tangent means rapid development elasticity after cementation. The slope of shear storage modulus in the course of increasing was steepest. Zirconite has low resistance during cementation that results in complete seating of crown. Once it started the polymerization, the process quit quickly. Variolink N, Panavia F 2.0 and RelyX U200 maintained plateau of tan δ for a long time. It means that these remain plastic for some time before setting, therefore they may be an advisable characteristics for clinical use. It provided longer manipulation time to a clinician before cementation to the abutments. As the development of elasticity, the fixed dental prostheses could not be on the correct position or could rebound. Therefore, the dental resin cements should be used before the development of elasticity. Judging from change of tan δ, the displacement of Peltier plate and resin cement materials occurred around 6 minutes. Therefore, the fixed partial denture being cemented should be provided adequate pressure on the prostheses in the mouth at least 6 minutes. In addition, the procedure of light curing could facilitate the curing process.

In this study, the effect of light curing was excluded because the light could not pass through stainless steel plate. Further studies are necessary to evaluate effect of light curing on rheological properties of resin cements.

V. CONCLUSION

Within the limitation of this study, all dental resin cements tested in this study showed differences in rheological properties and handling characteristics. Both obtained working time and setting time are different from the manufacturer's instruction. Comparing to other resin cements, RelyX U200 showed longer period of plastic property when it cured without light curing. Starting the curing reaction, it cured with faster rate. When it completed the curing process, RelyX U200 had the highest strength. RelyX U200, Variolink N and Panavia F 2.0 maintained plateau of loss tangent for a long time. It is convenient for clinicians to cement multiple units of dental prostheses simultaneously.

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- 국문초록 -

경화 중 레진 시멘트의 유변학적 특성

서울대학교 대학원 치의과학과 치과보철학 전공

(지도교수 이 재 봉)

이 재 림

연구목적: 본 연구의 목적은 임상에서 이용되는 몇 가지 레진 시멘트를 이용하여 혼합 후부터 경화 완료까지 시간에 따른 레진 시멘트의 점탄성 특성의 변화를 관찰하여 제조사가 지시하는 초기 경화 시간, 작업 시간, 최종 경화 시간 등과의 연관성을 살펴보고 유변학적 특성과 관련하여 사용시 주의사항에 대해 알아보고자 한다.

재료 및 방법: 6 종류의 레진시멘트 (Clearfil SA luting, Panavia F 2.0, Zirconite, Variolink N, RelyX Unicem clicker, RelyX U200) 를 제조사가 지시한 혼합비, 혼합 방법, 혼합 시간으로 혼합한 후 AR 1500 stress controlled rheometer의 하부 스테인리스 스틸 평행판 위에 0.25mL 적용 후 1Hz의 주기로 3000μN의 전단력을 가하여 dynamic oscillation time sweep test를 시행하였다. 평행판은 직경 25mm였고 두 평행판 사이의 간격은 500μm로 설정하였다. 실제 임상 상황을 재현하기 위하여

혼합은 실온(25°C)에서, 경화과정은 구강 내 온도인 32°C에서 진행하였다. 시멘트를 혼합하기 시작한 시간부터 첫 번째 데이터를 얻기까지의 시간은 70초이내로 일정하게 유지하였다. 시간에 따라 경화 반응이 진행되고 전단 저장 계수 (G'), 전단 손실 계수 (G"), 손실 탄젠트 (tan δ)와 같은 유변학적 특성을 나타내는 지표들의 변화를 20분 동안 9초 간격으로 기록하였다. 동일한 방법으로 6개의 재료마다 세 번씩 측정하였고, 각각의 측정 결과는 일원배치분석 및 Tukey's hoc test로 사후 검정을 시행하였다(α = 0.05).

결과: 본 연구에 사용된 모든 레진 시멘트에서 혼합 후 시간에 따라 G'값이 증가하여 안정상태에 도달하였다. G'값의 변화는 sigmoidal curve 형태로 나타났다. 각각의 재료는 G'이 최대값이 이르는데 걸리는 시간이 달랐다($P < 0.05$). 실험 종료 시점에서 RelyX U200은 가장 높은 G'값을 나타냈고, RelyX Unicem(clicker type)과 Variolink N이 가장 낮은 G'값을 나타냈다. 초기 경화 시간은 탄성이 발현되기 시작하기 직전까지의 시간으로 정의될 수 있는데, 이는 G' 값의 증가 또는 tan δ 값의 감소 시점으로 알 수 있다. RelyX Unicem은 가장 먼저 경화반응이 시작되었다. 초기 경화 시간과 제조사에서 지시한 작업시간 간에 높은 상관관계를 보였다($r = 0.978$, $P < 0.05$). 경화 반응이 수시간 동안 지속되기 때문에 실험 종료 시 G'값의 90%에 해당하는 값에 도달하는데 걸리는 시간을 측정하였고 이는 제조사에서 제시하는 경화시간과는 낮은 상관관계를 보였다(r =

0.118, $P > 0.05$). Tan δ는 일정 수준의 값을 유지하다가 G’이 증가하기 시작하는 시점에서 0에 도달하였다. 이는 변위량이 0에 도달하는 지점과 거의 일치하였으며, 그 시간은 6분 내외였다.

결론: 각각의 레진 시멘트는 서로 다른 유변학적 특성 및 조작성을 보였다. 본 연구에서 RelyX U200은 다른 레진 시멘트와 비교하여 가장 오랜 시간 동안 소성을 유지하고, 경화 완료 후 가장 높은 강도를 보였다. 따라서 여러 개의 보철물을 동시에 합착해야 하는 경우에 RelyX U200이 유용할 것으로 사료된다.

· 주요어 : 레진시멘트, 유변학적 특성, 피막도, 점탄성
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