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치의과학박사학위논문

**Effect of Vibration on Adaptation of
Dental Composites
in Simulated Tooth Cavities**

진동이 모형치아와동에 대한 복합레진의
적합도에 미치는 효과

2019 년 2 월

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한 선 희

Effect of Vibration on Adaptation of Dental Composites in Simulated Tooth Cavities

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Abstract

Effect of Vibration on Adaptation of Dental Composites in Simulated Tooth Cavities

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Objectives. This research investigated the effects of vibration on the rheological properties of dental composites and the adaptation of tooth cavity-composite interfaces.

Methods. A portable vibratory packing device and two composites, Filtek Z250 (Z250) and Filtek Bulk Fill Posterior (BFP), were evaluated. The frequency and amplitude of the vibratory packing device were measured. Dynamic oscillatory shear tests were conducted with varying frequency to

examine the rheological properties of the composites. Twenty identical composite teeth with a Class I cavity were prepared and filled with one of the two composites. The composite was placed into a cavity using the vibratory packing device operating in either ON or OFF mode. After light-curing of the composite, the gap between the tooth and the composite was evaluated using micro-computed tomography. Two-way ANOVA was used to evaluate the effects of vibration and composite type on tooth-composite adaptation.

Results. The frequency of the vibratory packing device was 66.8 Hz. The complex viscosity, η^* , of BFP was higher than that of Z250, and η^* of both composites significantly decreased with increasing oscillation frequency. The application of vibration did not decrease gap formation in cavity-composite interface.

Conclusion. The application of vibration (66.8 Hz) decreased the viscosity of composites, but did not enhance adaptation at the tooth-composite interface.

Keywords: Dental composite, Rheology, Vibration, Micro-CT, Gap

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1. Introduction

Unlike amalgam and gold restorations, dental composites can be directly adhered to teeth, enabling minimally invasive treatment. As material properties such as polymerization shrinkage and wear resistance have been improved and patient demand for esthetic restoration has increased, the frequency and range of composite use have also increased.¹⁻³

The handling characteristics of dental composites are very important for easy placement of filling material into a cavity and for good bonding⁴ and greatly influence the practitioner's choice of the composite. When the

composite is applied to a cavity, it tends to stick to the application instrument as well as to the tooth surface, making it difficult to manipulate and resulting in voids in the composite or decreased cavity wall adaptation.⁵ In addition, an incremental filling technique is required for good cavity wall adaptation, sufficient light-cure, and reduced polymerization shrinkage stress and leads to longer chair time.⁶

A flowable composite that can be injected into a cavity using a syringe-type device is relatively convenient. However, its reduced filler content to obtain flowability leads to an increase in the amount of polymerization shrinkage and does not have an adequate hardness as a final restoration for posterior teeth.

To date, high viscosity bulk fill composites and flowable bulk fill composites have been released in the market.^{7,8} Manufacturers claimed that those materials can be filled as a single layer by increasing the depth of cure and decreasing the amount of polymerization shrinkage. However, these new types of composites have not resolved the handling problem of conventional composites or de-bonding due to the stress caused by polymerization shrinkage.⁹

The pre-warmed method¹⁰ and sonic (or ultrasonic) vibration^{11,12} have been introduced to improve the convenience of manipulation and to increase the adaptability of dental composites to a cavity without changing the composite formulation. The heating of composites decreases their viscosity

by increasing the molecular motion of the monomer.¹⁰ A composite heated to 60 °C has increased fluidity without changes in other properties. However, during the cooling process, the viscosity of the heat-treated composite increases rapidly by 66-450%.¹³ In addition, there is the possibility of thermal damage to the dental pulp by the pre-heated hot composite and questions regarding control of the thermal volume change and cooling time during the filling process.

Another method to increase the adaptation of a composite is a handpiece-type loading device using vibration. It uses a high-viscosity composite with high filler content and reduces the viscosity of the composite with vibration to enable single-layer bulk filling.⁷ However, the device requires a specially designed, expensive handpiece, coupler, and a special composite containing a vibration modifier.

Most of current dental composites are viscoelastic materials composed of Bis-GMA (bisphenol A glycidyl methacrylate) and TEGDMA (triethylene glycol dimethacrylate) monomers and inorganic fillers of a few nm to 1 µm size. The absolute values and ratio of elasticity and viscosity of the composite prior to cure are greatly related to the handling characteristics and flowability of the composite. The type and proportion of monomers, amount and size of inorganic filler, surface morphology, temperature, vibration frequency, and shear stress directly influence the viscoelasticity of dental composites.¹⁴⁻¹⁹ In previous studies, the viscoelastic properties of dental

composites were measured with increasing oscillation frequency up to 100 rad/s (≈ 16 Hz). It was reported that the dental composite showed 'pseudoplasticity' or a 'shear-thinning' property in that the viscosity decreased with increasing oscillatory shear rate.¹⁴⁻¹⁶ However, no studies have been published to date on the viscoelasticity change of dental composites when subjected to vibrations higher than 100 rad/s (≈ 16 Hz).

Recently, a portable vibratory packing device has been introduced with the intent of increasing the adaptation of the tooth-composite interface by applying a vibration of 60 Hz or more. However, to date, no studies have assessed the utility of the device. The purpose of this study was to observe the viscoelastic change of dental composites at the frequency of the vibratory packing device and to verify the effectiveness of the device by evaluating the adaptation of the tooth-composite interface after adapting the device for composite filling.

2. Materials and Methods

2.1. Materials

A micro-hybrid composite, Filtek Z250 Universal Restorative (Z250, 3M ESPE, St. Paul, MN, USA), and a nano-hybrid composite, Filtek Bulk Fill Posterior Restorative (BFP, 3M ESPE), were used. The composition of each composite is shown in Table 1.

2.2. Vibratory packing device

A vibratory packing device (COMO, B&L Biotech, Ansan, Korea) was used for packing the composite into the tooth cavity. A rounded-end tip (diameter: 2 mm) was used among various changeable tips. After the tip of the vibratory packing device was placed on a displacement sensor (LVDT, AX-1, Solartron Metrology, West Sussex, UK), the output voltage of the sensor was measured using an oscilloscope (TDS220, Tektronix, Inc., Wilsonville, OR, USA). The frequency and amplitude of the device were recorded.

2.3. Rheological measurement

In order to measure the dynamic viscoelastic change of the composites under oscillation, a dynamic oscillatory shear test was conducted using an

ARES (Advanced Rheometric Expansion System, TA Instruments, New Castle, DE, USA) in strain-controlled mode. A parallel plate geometry (diameter: 8 mm) was selected for paste-type composites, and the temperature was set to 30 °C.

A strain sweep test was performed before the frequency sweep test. The composites were loaded on the parallel plate, the chamber was closed to block ambient light, and the gap between the plates was decreased to 1.65 mm. The shear storage modulus G' and the shear loss modulus G'' were measured for strains of 0.01-10% at a frequency of 10 Hz.

A frequency sweep test was performed to measure the change in viscoelasticity of composites with varying frequency. Based on the change of G' and G'' obtained in the strain sweep test, a sinusoidal strain of 2%, which is close to that of the linear viscoelastic region and similar to the vibration amplitude of the packing device (0.034 mm)/specimen thickness (1.65 mm), was applied. The complex viscosity η^* was measured for frequencies of 0.01-70 Hz ($n = 3$).

2.4. Gap measurement of tooth cavity-composite interface

In order to investigate whether the vibratory packing device influences the adaptability of the composite into a cavity, micro-computed tomography

(micro-CT) was used to measure the gap at the tooth-composite interface.

2.4.1. Simulated tooth cavity preparation

A Class I cavity (4 mm in mesio-distal width, 2.5 mm in bucco-lingual width, 2 mm in depth from the central pit) was prepared on the occlusal surface of an upper premolar model tooth (#15, A5AN-500, Nissin Dental Products, Inc., Kyoto, Japan) using a taper and flat end diamond bur (TF-31, Mani, Tochigi, Japan) under a water spray. After an impression of the prepared tooth was made with a polyvinylsiloxane impression material (Honigum Light, DMG, Hamburg, Germany), a flowable composite (DenFil Flow, shade A2, Vericom, Anyang, Korea) was incrementally filled and photopolymerized to obtain 20 identical simulated composite teeth. Glycerin was applied on the surface of the specimens, and they were light-cured to minimize the formation of air-inhibited, unreacted resin layers.

2.4.2. Restorative procedure

Equal amounts of composites were prepared in advance to fill the cavity. The weight difference of the tooth specimen before and after composite filling was set as the amount of composite. After the composite tooth specimen was fixed in a metal vise, the cavity was sandblasted with 50 μm

aluminum oxide (Al_2O_3) particles (BasicMobil, Renfert, Hilzingenm Germany), water cleaned, and air dried. A silane coupling agent (Porcelain Primer, Bisco Inc., Schaumburg, IL, USA) was applied and dried. A bonding agent (Scotchbond Multi-Purpose, 3M ESPE) was uniformly applied to the inner surface of the cavity and light-cured for 20 s with an LED light-curing unit (B&Lite, B&L Biotech). The vibratory packing device was equipped with a 2 mm diameter rounded tip. The filling of the composite was divided into two groups as follows.

Group 1: Either Z250 or BFP composite was placed into the cavity and packed using the packing device without vibration (OFF) (Fig. 1a).

Group 2: After placing Z250 or BFP composite into the cavity, it was packed using the packing device with vibration (ON) (for each material, n = 5).

Packing was done for 40 s with 2 stroke/s at 30 °C and light-cured for 20 s (radiant emittance = 700 mW/cm^2).²⁰

2.4.3. Evaluation of internal adaptation using micro-CT

The restored cavities were analyzed with a high-resolution micro-CT system (Model 1172, SkyScan, Aartselaar, Belgium) operating with a 100 kV

accelerating voltage, a 100 μ A beam current, a 0.5 mm Al filter, 4.84 μ m resolution, and 180° rotation with a 0.4° step. The sliced images were reconstructed into 3-D images (image pixel: 4.84 μ m in X-Y axis, 10 μ m slice thickness in Z axis) using image reconstruction software (NRecon, Ver. 1.7.0.4, SkyScan). The gap volume was measured using an analysis program (CT Analysis, ver. 1.16.4.1, Skyscan) for upper, middle, and lower layers at 0.66 mm intervals. The measured gap volume in the CT images was used to evaluate the degree of internal adaptation of composites in the cavity.

2.5. Statistical Analysis

Two-way ANOVA was applied to compare the gap volume at the tooth-composite interface according to the types of composite and vibration. Statistical analysis was performed using SPSS software (Version 23.0, SPSS Inc., Chicago, IL, USA), and the significance level for the test was set at 5%.

3. Results

The frequency and amplitude of the vibratory packing device (COMO) were 66.8 Hz and 0.034 mm, respectively (Fig. 1b). The changes of G' and G'' according to the strain are shown in Fig. 2. The G' and G'' of BFP were higher than those of Z250 and decreased when the strain increased to more than 1% in both composites.

As the frequency increased, the complex viscosity η^* decreased significantly in both Z250 and BFP (Table 2, Fig. 3). The η^* of Z250 decreased by 81.6%, from 731.58 Pa·s at a frequency of 2 Hz to 134.63 Pa·s at 70 Hz. In BFP, the η^* decreased by 89.4%, from 11,112.12 Pa·s at 2 Hz to 1174.73 Pa·s at 70 Hz. The η^* of BFP was 8.73 times higher than that of Z250 at 70 Hz, close to the vibration frequency of COMO.

The total cavity volume was 21.5 mm³. More than 80% of the total gap volume was observed in the lower third of the layers for all conditions (Table 3, Fig. 4, 5). For both Group 1 (vibration = OFF) and Group 2 (vibration = ON), there was no significant difference in gap between the two composites in the same group ($p > 0.05$). For the same composite, both Z250 and BFP showed no statistically significant difference between ON

and OFF vibration modes for each composite ($p > 0.05$). No interaction was observed between composite type and vibration mode ($p = 0.0505$).

4. Discussion

The vibratory packing device (COMO) had a DC motor and an eccentric weight to generate circular vibration. Thus, at the end of the 2 mm diameter tip of the packing device, both vertical and shear vibration were applied to the composite. In this study, the dynamic oscillatory shear test was carried out because it was reported that the viscosity change under vertical vibration had a strong correlation with that of the dynamic oscillatory shear test.¹⁷

It is possible to use various geometries such as parallel plates, cone and plate, and concentric cylinders to measure the viscoelasticity of materials.²¹ For high-viscosity dental composites, it is difficult to use the cone and plate geometry due to the limitation of sample loading and gap control.¹⁶ For this reason, the parallel plate geometry was used in this study. It was reported that the SAOS (small amplitude oscillatory shear) test did not reveal significant differences between cone and plate and parallel plates in multiphase systems such as dental composites.²¹

The G' and G'' of Z250 and BFP decreased with increasing strain amplitude greater than 1% (Fig. 2). The shear stress of the composites under shear deformation was mainly caused by the frictional force of the filler in resin matrix.¹⁸ Therefore, the decrease in G' and G'' with increasing strain

may be due to the reduction of frictional force by separation between resin monomers and filler particles or between filler particles. Z250 and BFP are different in filler content and size, especially BFP, which contains 4-11 nm zirconia/silica nanoparticles, leading to different changes of G' and G'' in shear deformation. The G' of BFP decreased steeply at over 1% strain.

The gap volume at the tooth-composite interface was largest in the lower third for both composites (Table 3), which is similar to the gap produced by the polymerization shrinkage of composite during curing.²² It was reported that the polymerization shrinkage vectors of dental composites during light-curing depend on time and the location and depth in the cavity, and that they are directed to the lower quadrant of the cavity.²³ Composite on the axial wall is able to partially dissipate the shrinkage stress through cuspal deflection along the vector from the axial wall to the inside. For the non-bonded upper part of the composite, there is room for the downward flow to relieve stress. On the other hand, there is a high possibility for de-bonding to occur by the upward stress vector at the bottom surface.

The volume percent of the gap ranged 0.95% to 2.59%, which was similar to the voids observed with a SonicFill delivery system analyzed with micro-CT.²⁴ However, the volume of the gap should be compared for materials processed under the same set of conditions, including

experimental design, CT image processing method, and threshold values on the histogram. In addition, gap formation is related to the handling characteristics of composites such as its adaptability (wettability), stickiness, and slumping resistance, and because of this, the rheological properties of composites should be analyzed together.^{25,26}

Theoretically, sonic vibration is expected to increase the flowability of the composite to enhance its adaptability.²⁴ However, the vibration did not affect the gap formation of either of the composites in this study ($p > 0.05$). In spite of the lack of a statistically significant difference, BFP tended to have less gap formation with the vibration mode ON. These results could be explained by the differences in the viscoelasticity of the composites with vibration affecting gap formation. BFP had an η^* that was 35.8 times larger than that of Z250 at a stationary state ($f = 0.01$ Hz), and the decreased η^* of BFP at 70 Hz was similar to that of Z250 at 1 Hz (Table 2). This would cause the result in the highly viscous BFP composite. In the Z250, the η^* at 70 Hz was similar to the level of a flowable composite.¹⁶ It should be noted that, as η^* decreased, not only the flowability and adaptability of composite increased, but also the stickiness of the composite to an instrument increased²⁷, which can have an adverse effect on the composite adaptability to the cavity wall.⁵

As mentioned previously, polymerization shrinkage and stress can affect the gap formation by de-bonding. There is little difference in polymerization shrinkage and shrinkage stress between BFP and Z250²⁸, and therefore its effect can be excluded.

Unlike previous studies undertaken with different cavities in extracted teeth that have different anatomical and histochemical features, in this study replicated composite teeth with the same tooth cavity were used. Therefore, the shape and size of the cavities were identical, a fixed amount of composite was used to minimize specimen errors.

Reflection optical microscopy²⁹, confocal microscopy³⁰, and scanning electron microscopy³¹ can be used to evaluate the adaptation of the composite with a sectioned specimen. With SEM, it is possible to observe both the gap at the μm level and the interfaces between tooth, adhesive, and composite.³² However, there is a possibility of contamination or damage of specimens during the sectioning process, which can lead to different results according to the cutting location. Moreover, there are limitations in evaluating the three-dimensional gap volume.

Dye penetration observes the degree of penetration to determine the adaptation of composite inside a cavity.³³ However, it has limitations in that

the results may show differences depending on external conditions such as the type and chemical properties of the tracer, the pH of the solution, and the chemical affinity of the specimen-tracer.²² Thus, the micro-CT analysis has the merit that is possible to reduce errors because there is no need to cut the specimen or to reproduce the experiment, and it can measure the micro-gap in all aspects in three dimensions.^{22,24}

To summarize, the vibratory packing device, COMO, could not enhance the adaptability of dental composites into a tooth cavity. It was reported that both conventional and bulk-fill composites showed less cuspal deflection with incremental layering techniques.¹⁹ In particular, composite core restoration after endodontic treatments or narrow-deep cavity filling like a proximal box of Class II cavity require the incremental filling technique, resulting in tight adaptation into the bottom of cavity, a reduction in the number of voids, and the attainment of the maximum degree of conversion.^{6,33} Thus, the conventional incremental layering and packing method is needed to overcome technique sensitivities and to increase the adaptability of dental composites.

5. Conclusion

The complex viscosity, η^* , of BFP was higher than that of Z250, and η^* of both composites decreased with increasing vibration frequency. The vibratory packing device oscillated at a frequency of 66.8 Hz and did not enhance the cavity adaptation of composites.

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Tables and Figures

Table 1. The resin composites used in this study

Composite (Code, Shade, Lot No.)	Composition
Filtek™ Z250	Bis-GMA, UDMA, TEGDMA, Bis-EMA
Universal Restorative (Z250, A2, N783064)	Filler: 0.01 – 3.5 µm Zirconia / silica particles (82 wt% / 60 vol%)
Filtek™ Bulk Fill Posterior Restorative (BFP, A2, N710161)	AUDMA, AFM, DDDMA, UDMA Filler: 4 - 11 nm Zirconia / silica, Ytterbium trifluoride (76.5 wt% / 58.4 vol%)

Abbreviations: Bis-GMA, bisphenol A glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; AUDMA, aromatic urethane dimethacrylate; AFM, addition-fragmentation monomer; DDDMA, 1, 1 triethylene glycol dimethacrylate 2-dodecanediol dimethacrylate

Table 2. Change in complex viscosity (η^*) of composites as a function of frequency

Frequency (Hz)	Complex viscosity (Pa·s)	
	Z250	BFP
0.01	35046.6	1254933.6
0.1	5946.3	131520.6
1.1	1145.7	19118.3
11.1	326.0	3657.8
70.0	134.6	1174.7

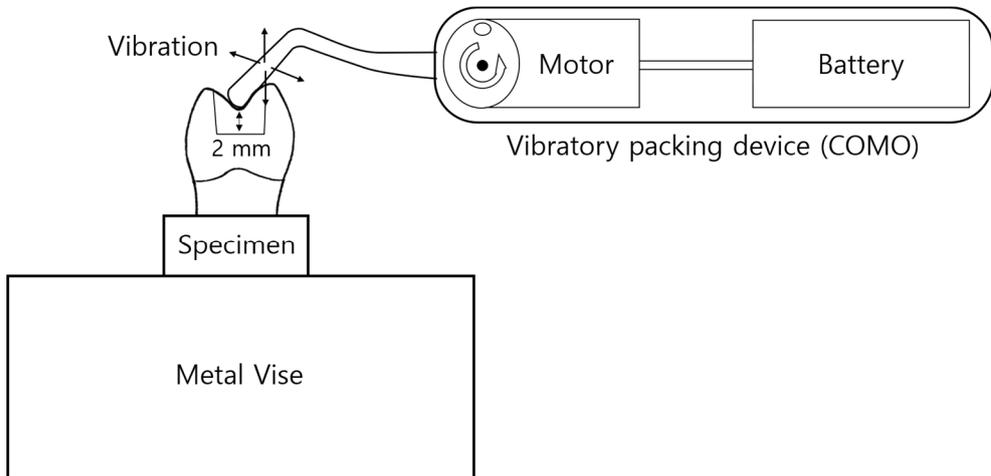
Table 3. Volume of the gap (mm³ and %) at different locations of the tooth-composite interface with or without vibration (ON or OFF) when filled with Z250 or BFP

Location	Z250		BFP	
	ON	OFF	ON	OFF
Upper 1/3	0.014 (0.014)	0.009 (0.007)	0.009 (0.004)	0.006 (0.003)
Middle 1/3	0.031 (0.030)	0.027 (0.027)	0.022 (0.023)	0.080 (0.076)
Lower 1/3	0.367 (0.107)	0.288 (0.212)	0.172 (0.182)	0.471 (0.280)
Total gap volume (mm ³)	0.412 (0.123) ^{†*}	0.323 (0.233) ^{††*}	0.203 (0.203) ^{††*}	0.557 (0.329) ^{†††*}
(Vol. %)	1.92	1.51	0.95	2.59

The same superscript ‘†’ or ‘††’ means that there is no significant difference between ON and OFF mode for each composite, respectively ($p > 0.05$).

The same superscript ‘*’ or ‘**’ means that there is no significant difference between Z250 and BFP for each packing mode, respectively ($p > 0.05$).

(a)



(b)

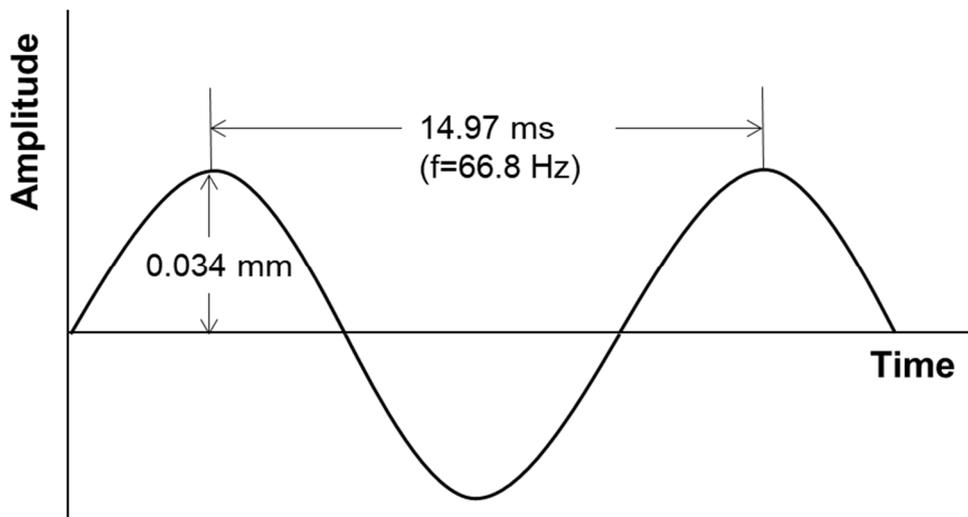
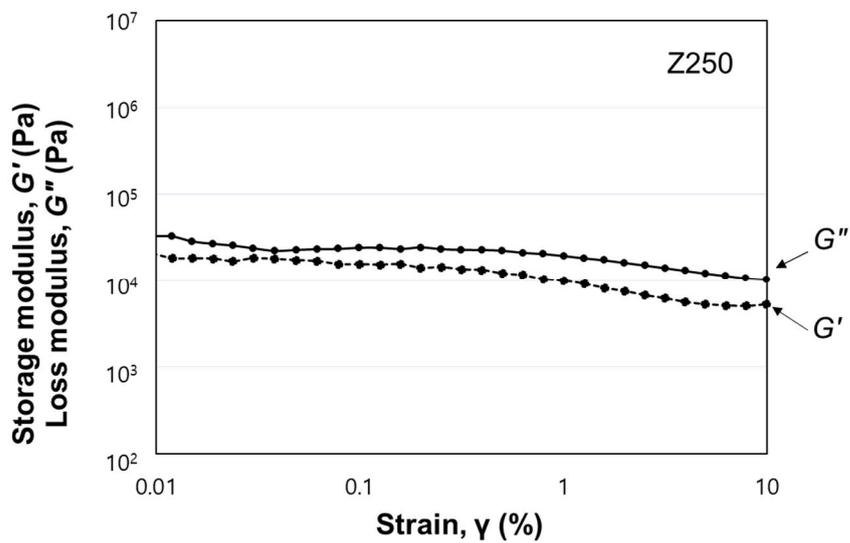


Figure 1. (a) Schematic diagram of a tooth specimen and a vibratory packing device (COMO). (b) The frequency and amplitude of vibration mode of the COMO were measured using an oscilloscope.

(a)



(b)

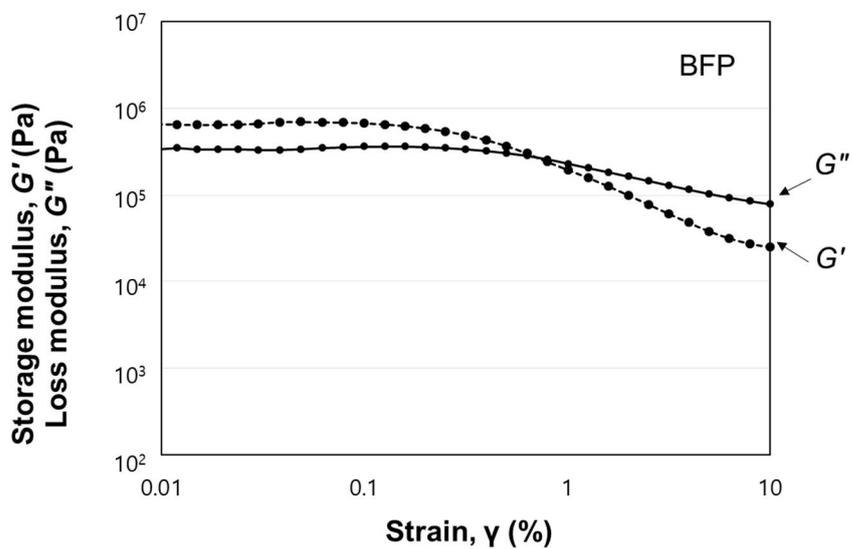


Figure 2. Strain sweep test. Shear storage modulus (G') and shear loss modulus (G'') as a function of strain in (a) Z250 and (b) BFP at $f = 10$ Hz.

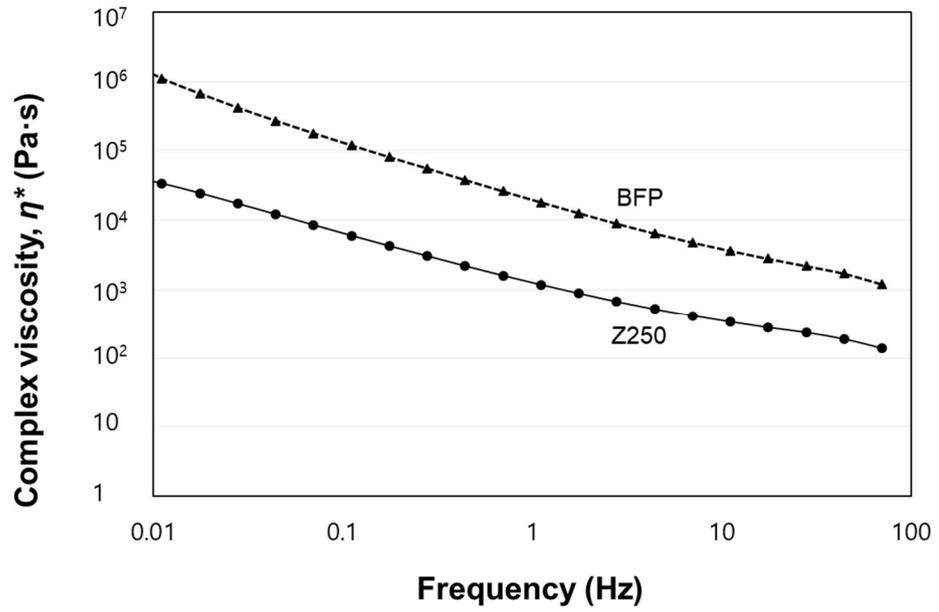
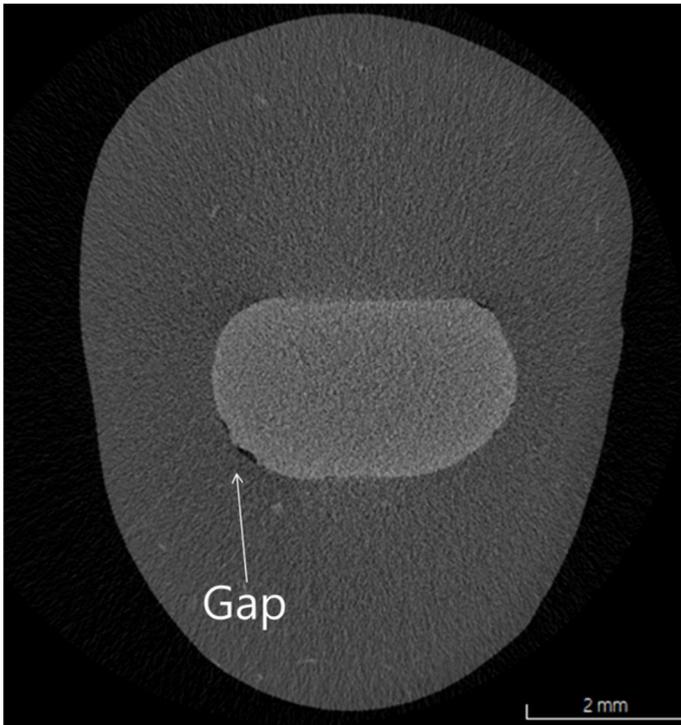
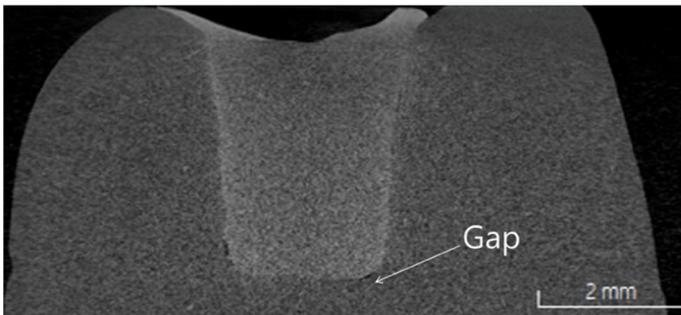


Figure 3. Frequency sweep test. The complex viscosity (η^*) of composites as a function of frequency at log scale.

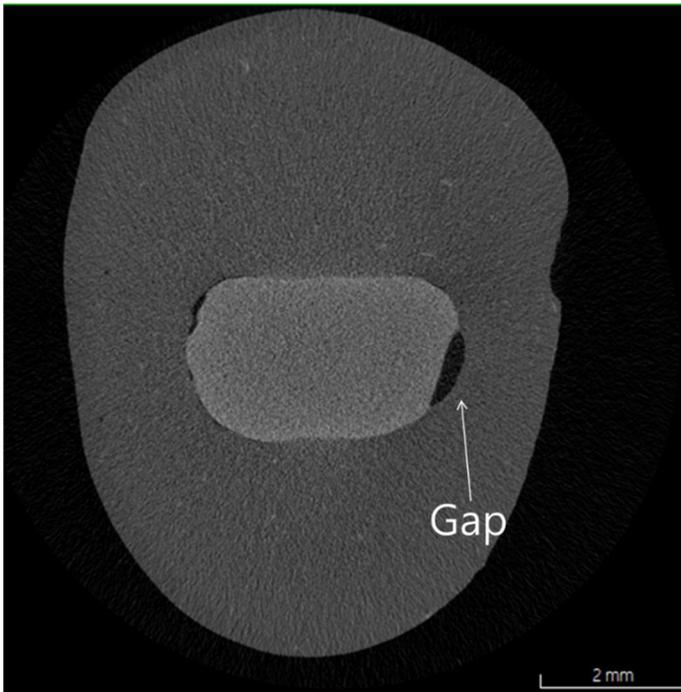
(a)



(b)



(c)



(d)

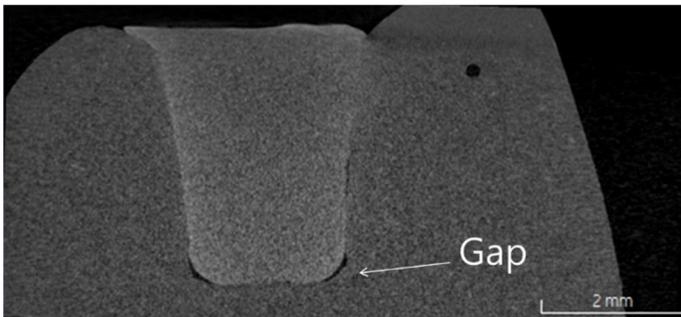


Figure 4. Micro-CT images of the gap in (a) BFP sample with vibration in a horizontal sectional view and (b) in a sagittal sectional view, (c) BFP sample without vibration in a horizontal sectional view and (d) in a sagittal sectional view.

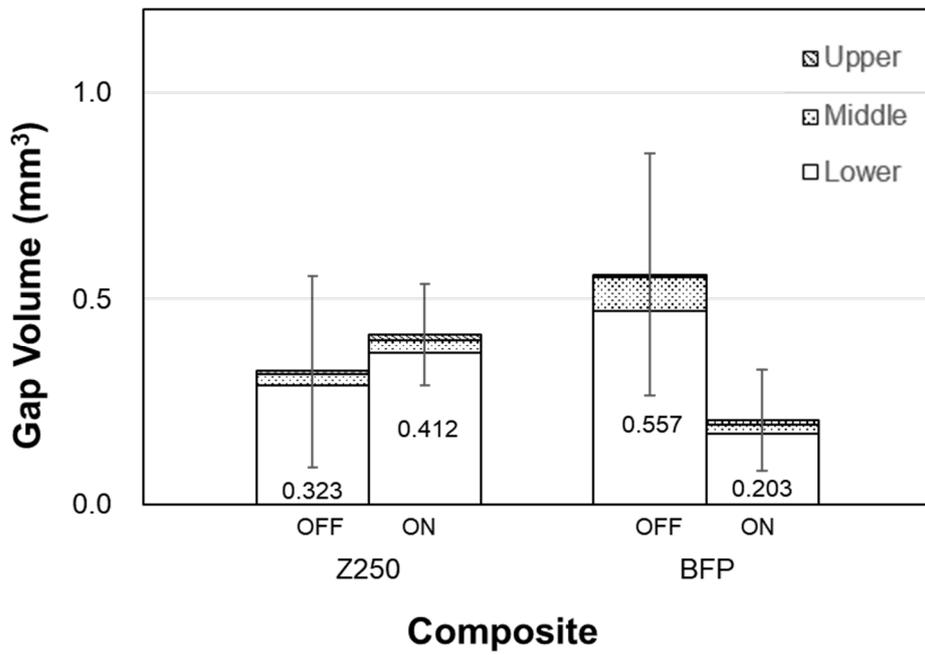


Figure 5. Volume of gap (mm^3) at the tooth-composite interface when vibration is ON or OFF.

요약(국문초록)

진동이 모형치아와동에 대한 복합레진의 적합도에 미치는 효과

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1. 연구목적

본 연구의 목적은 복합레진 충전 시 진동형 응축기구가 복합레진의 유변학적 성질과 와동-복합레진 계면의 적합도에 미치는 영향을 알아보고자 하였다.

2. 재료 및 방법

휴대용 진동형 충전기구와 Filtek Z250(Z250) Filtek Bulk Fill Posterior(BFP)을 사용하였다. 진동형 충전기구의 진동 주파수와 진폭을 측정하였다. 동적회전전단 실험으로 진동 주파수 변화에 따른 복합레진의 유변학적 성질을 측정하였다. 1 급 와동이 형성된 동일한 크기와 모양의 복합레진 모형 치아 20 개에 진동형 응축 기구의 전원을 켜고 상태와 끈 상태로 복합레진을 충전한 후, Micro-Computed tomography 를 이용하여 와동-복합레진 계면의 적합 되지 않은 부위의 3 차원적 부피를 계산하였다. Two-way ANOVA 로 진동 여부와 복합레진의 종류가 적합도에 미치는 영향을 평가하였다.

3. 결과

진동형 충전기구의 주파수는 66.8 Hz 였다. BFP 의 복소점도는 Z250 에 비해 높았고 주파수의 증가에 따라 두 복합레진의 점도가 크게 감소하였다. 충전 시 진동을 적용하는 것은 와동-복합레진 계면의 미적합부위를 감소시키지 않았다.

4. 결론

진동을 이용한 복합레진 충전법 (66.8 Hz)이 복합레진의 점도를 감소시키지만, 복합레진-와동 적합성을 증진시키지 않았다.

주요어: 치과용 복합레진, 유변학, 진동, micro-CT, 적합도

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