EFFECT OF PRE-HEATING ON SOME PHYSICAL PROPERTIES OF COMPOSITE RESIN

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ABSTRACT

The purpose of this study was to evaluate the effect of pre-heating on some physical properties of composite resin.

Eighty extracted, noncarious human molars were used in the present study. Four different temperatures of composite resin were used: 4℃, 17℃, 48℃, and 56℃. The 4℃ and 17℃ values represented the refrigerator storage temperature and room temperature respectively. For 48℃ and 56℃, composite resin was heated to the temperatures. As physical properties of composite resin, shear bond strength, microhardness, and degree of conversion were measured. The data for each group were subjected to one-way ANOVAs followed by the Tukey's HSD test at 95% confidence level.

Both in enamel and dentin, among composite resin of 4℃, 17℃, 48℃, and 56℃, the pre-heated composite resin up to 56℃ revealed the highest shear bond strength, and pre-heated composite resin to the higher temperature revealed higher shear bond strength.

Microhardness value was also higher with composite resin of higher temperature.

Degree of conversion was also higher with composite resin of the higher temperature.

In this study, it seems that pre-heating composite resin up to the higher temperature may show higher shear bond strength, higher microhardness value, and higher degree of conversion. Therefore, when using composite resin in the clinic, preheating the composite resin could be recommended to have enhanced physical properties of it. (J Kor Acad Cons Dent 34(1):30-37, 2009)

Key words: Composite resin, Pre-heating, Shear bond strength, Microhardness, Degree of conversion.

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I. INTRODUCTION

The ultimate objective of composite resin restoration is an ideal restoration that is relatively easy to place, convenient to cure, long lasting, and esthetic. The parameters of effective practice have placed the additional burdens of technique and dental material awareness on the practitioner.

Polymerization of photo-activated restorative dimethacrylate-based materials leaves a significant proportion of methacrylate groups unreacted depending upon monomer and filler composition, filler particle size and type, interactions between monomers and filler initiator system, and light-curing procedure[1-4]. It exhibited incomplete degree of conversion, approximately 45% to 70%. The reason of incomplete conversion may be related to an increase in the viscosity of the rapid formed-highly cross-linked polymeric network which prevents the initiator from coming into contact with the carbon groups[5]. As the level of unreacted or residual monomer increases, the mechanical characteristics of the restoration may be decreased. Degree of conversion, independent of cure method, has a critical effect on the final mechanical

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properties and wear rate of composite[6,7]. A higher conversion ratio at a greater depth was found to increase the material modulus, resulting in less flexure and less potential for restoration fracture under loading[8].

To increase the rate of polymerization, the resulting cross-linking density, and the ultimate degree of conversion of dimethacrylate-based monomers and resins, raising the temperature after initial curing was tried at which the photopolymerization occurs[9]. Heat curing has been another method to enhance some physical properties of resin. Some advantages of heat-curing composites are already well known for a long time in the manufacture of extraorally fabricated inlays and onlays[10]. Such studies found some mechanical properties to be enhanced with elevated temperature: those were improved diametral tensile strength, increased fracture toughness and increased hardness[11-13]. The main effect of post-cure heating was to relieve stresses formed during the gel stage of polymerization[14]. Such a homogenization of stress would then account for the immediate property enhancement of post-cure heated specimens over that of the light-cured material alone.

Among the methods to increase of the degree of conversion, pre-heating composite resin may be one of the most applicable method in clinic, and it will be necessary to investigate the properties of the directly-placed composite under varying thermal conditions. Therefore, the purpose of this study was to investigate the effect of pre-heating on some physical properties of composite resin.

Ⅱ. MATERIALS AND METHODS

Tetric Ceram,TM shade A3 (Ivoclar Vivadent, Schaan, Liechtenstein, Germany) was used as a composite resin.

Four different temperatures of composite resin were used: 4℃, 17℃, 48℃, and 56℃. The 4℃ and 17℃ values represented the refrigerator storage temperature and room temperature respectively. For 48℃ and 56℃, composite resin was heated to the temperatures with Calset™ heating unit (AdDent, Inc., Danbury, CT, USA).

1. Shear bond strength test

Eighty extracted, noncarious human molars stored in isotonic saline at 4℃ were used in the study. During the last 24 hours before the experiment, teeth were kept in distilled water. The teeth were embedded in auto-polymerizing acrylic resin (Orthodontic Resin, Dentsply/DeTray, Konstanz, Germany) molds so that the prepared enamel and dentin surface were 2 mm above the acrylic resin cylinders, and were placed in tap water to reduce the temperature rise from the exothermic polymerization reaction. After the acrylic resin mold had completely polymerized, the occlusal surfaces of the teeth were ground perpendicular to the long axis of the tooth with a water-cooled, precision low-speed diamond saw (Isomet, Buhler, Lake Bluff, IL, USA) to make enamel and dentin surfaces for bonding. The surfaces of enamel and dentin were hand finished with wet #240- and #600-grit silicon carbide abrasive papers using twenty 15 cm-long strokes for 15 seconds. After ultrasonic cleaning with distilled water for 3 minutes to remove the excess debris, these surfaces were washed and dried with oil-free compressed air (Hotman, Dentro, Tokyo, Japan).

The teeth were randomly divided into 8 groups of 10 teeth (Table 1). The enamel and dentin surfaces were etched with a 37% phosphoric acid gel (ETCH-37™, Bisco, Schaumburg, IL, USA) for 30 seconds and 15 seconds respectively, and were thoroughly rinsed for 5 seconds and blot dried with cotton pellets leaving a visibly moist surfaces. Two coats of acetone-based one-bottle adhesive system (One-Step, Bisco, Schaumburg, IL, USA) were applied on teeth surfaces with a microbrush (International Corp., Durgavan, Waterford, Ireland) and light cured for 15 seconds using LED-light curing unit (Bluephase™, Ivoclar Vivadent, Schaan, Liechtenstein, Germany).

Specimens were placed in mount jigs (Ultradent Product Inc., Jordan, Utah, USA). The jig was then lowered and secured to the teeth surface. Plastic mold with an internal ring of 2.3798 mm in diameter and height of 2.0 mm was placed against the tooth surface. Composite resin was packed into the mold and light cured for 40 seconds. The samples were carefully separated from the mold by lifting the jig

while securing the sample with a rounded hand instrument to allow the bond to remain undisturbed. After twenty-four hours of water storage, the specimens in each group were tested in shear mode using a chisel-shaped rod in an Instron testing machine (Type 4411, Instron Corp., Canton, Massachusettes, USA) at a cross-head speed of 1 mm/min.

2. Vickers microhardness test

Teflon molds with a diameter of 6 mm and thickness of 2 mm were used to make composite resin specimens. Five disc-shaped specimens were made with composite resin (Tetric Ceram™, A3 shade, Ivoclar Vivadent, Schaan, Liechtenstein, Germany) for four different resin temperature groups. The molds were filled with composite resin and covered with clear Mylar strips lying on glass plate and light cured for 40 seconds using LED-light curing unit (Bluephase™, Ivoclar Vivadent, Schaan, Liechtenste in, Germany). The bottom surfaces of the specimens were ground and polished on silicon carbide abrasive papers of grit size #220, 600, 800, and 2000 sequentially. All specimens were stored in distilled water at room temperature for 24 hours before any further procedure.

For the microhardness test, a Vickers microhardness was measured using Microhardness Tester FM™ (Future-Tech Corp., Kawasaki, Japan) with a 300 g load and a 10 seconds loading time. Two microhardness measurements per specimen were obtained on the bottom surface of composite resin. Each microhardness determination consisted of two evenly-spaced indentation measurements over the polished surface of each specimen. The resultant dimensions were measured as the lengths of the diagonals of the indentation marks with the aid of an optical microscope (300 ×) and expressed as a Vickers Hardness Number (VHN).

3. Measurement of degree of conversion

The degree of conversion was determined using a FT-IR spectrometer using ten experimental resin specimens of the four different temperatures.

The FT-IR spectra were recorded with 16 scans at a resolution of 4 cm^{-1}. First, the spectrum of the unpolymerized resin was measured. After the resin specimen was irradiated for 40 seconds with a light curing unit, the spectrum of the polymerized resin was measured. According to the formula DC= [1-(cured/uncured)] × 100, the ratio between aliphatic (1640 cm^{-1}) and aromatic (1610 cm^{-1}) carbon double bonds stretching was used for calculating the degree of conversion[15].

4. Statistical analysis

The data of shear bond strength, Vickers microhardness, and degree of conversion from each group were analyzed by one-way ANOVAs and Tukey's HSD comparison test to compare them between different temperature groups (α = 0.05).

Ⅲ. RESULTS

Shear bond strength

The results of the shear bond strength tests to enamel are shown in Figure 1.

Among the temperatures of 4℃, 17℃, 48℃, and 56℃, pre-heated composite to 56℃ revealed the highest

Table 1. Summary of experimental design

Shear bond strength		Vickers microhardness	Degree of conversion	Conditioning of composite resin
Enamel group	Dentin group	Resin group		(temperature)
E1	D1	R1	R1	Refrigerator (4℃)
E2	D2	R2	R2	Room temperature (17℃)
E3	D3	R3	R3	Pre-heating to 48℃
E4	D4	R4	R4	Pre-heating to 56℃

shear bond strength ($P < .05$). The higher temperature of pre-heated composite resin yielded the higher shear bond strength to enamel. There were statistically significant differences ($P < .05$) between all groups except between groups of 4℃ and 17℃.

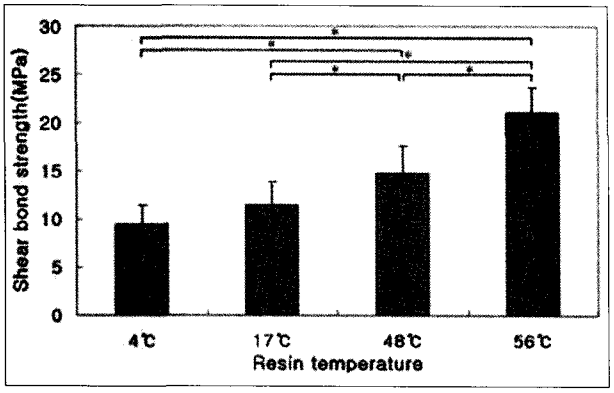

Figure 1. Shear bond strength of pre-heated composite resin to enamel. Mean ± S. D., n =10.
* significantly different at $P < .05$.

The results of the shear bond strength tests to dentin are shown in Figure 2.

Among the temperatures of 4℃, 17℃, 48℃, and 56℃, pre-heated composite to 56℃ revealed the highest shear bond strength ($P < .05$). The higher temperature of pre-heated composite resin yielded the higher shear bond strength to dentin. There were statistically significant differences ($P < .05$) except between 4℃ and 17℃ groups ($P < .05$) and between 48℃ and 56℃ groups ($P < .05$).

Microhardness

The results of the Vickers microhardness value are shown in Figure 3. The higher temperature of pre-heated composite resin yielded higher Vickers microhardness value. There were statistically significant differences between all groups except between 4℃ and 17℃ groups ($P < .05$). Among the experimental groups, pre-heated to 56℃ group yielded the highest value.

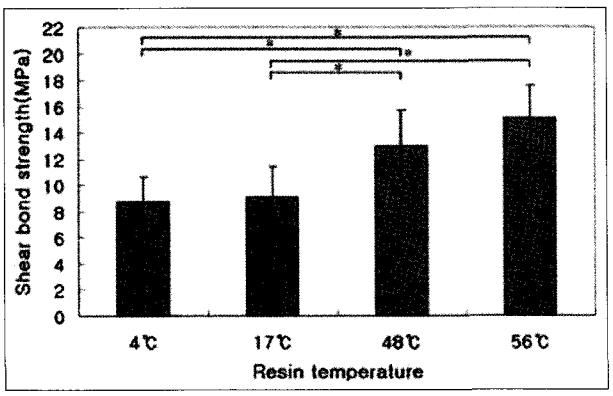

Figure 2. Shear bond strength of pre-heated composite to dentin. Mean ± S. D., n =10.
* significantly different at $P < .05$.

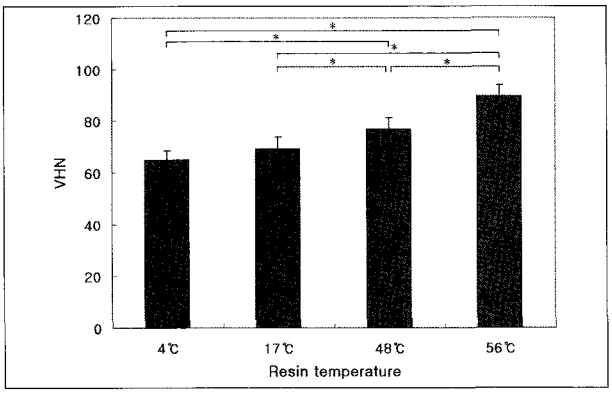

Figure 3. Vickers microhardness value. Mean ± S. D., n =10.
* significantly different at $P < .05$.

Degree of conversion

The results of degree of conversion are shown in Table 4. The higher temperature of pre-heated composite resin yielded higher degree of conversion. There were statistically significant differences between all groups except between 4℃ and 17℃ groups ($P < .05$). Among the experimental groups, pre-heated to 56℃ group yielded the highest value.

Ⅳ. DISCUSSION

This study demonstrated the influence of temperature on the shear bond strength, Vickers microhardness and degree of conversion of pre-heated composite resin. In the present study, higher temperature of

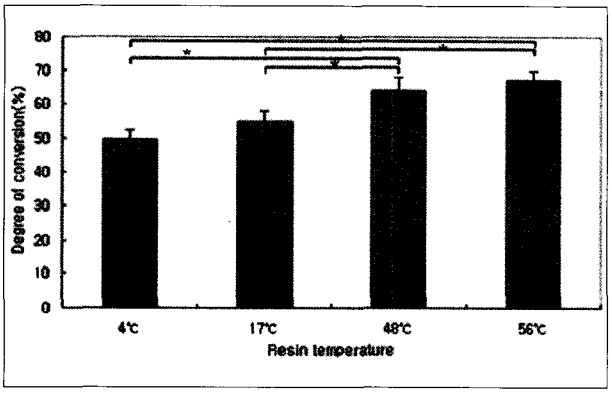

Figure 4. Degree of conversion of composite resin. Mean ± S. D., n =10.
* significantly different at $P < .05$.

pre-heated composite resin yielded higher shear bond strength, microhardness and degree of conversion. These result may be due to the fact that elevated temperature of composite increase the mobility of the radical and ensue additional polymerization[16]. Photo-activated polymerization of dimethacrylate-based materials is based on free-radical formation and leads to a strong, cross-linked network. The reaction kinetics of these multifunctional monomers used for dental restorations is a multifaceted process exhibiting complex features such as autoacceleration, autodeceleration, limited final conversion, cyclization, and radical trapping[17] This complex behavior arises from the decrease in mobility of reaction media by network formation as polymerization proceeds, leading to the onset of autoacceleration from the very beginning of polymerization[18]. This phenomenon, also called the gel effect, corresponds to a sudden increase in reaction rate, despite the monomer's being consumed. It is generally accepted that autoacceleration occurs due to changes in the termination rate constant, and a consequent increase in the concentration of free-radicals.

It was shown that increasing the temperature of the composite resin with a warmer, Calset™ composite warmer (AdDent, Inc., Danbury, CT, USA) increased the flow of composites up to 68%[19]. When a composite resin becomes more flowable, the composite may have better adaptation to the tooth structure, which may decrease microleakage[20]. Because composite resin is a viscoelastic material, it may exhibit decreased viscosity and greater flowability with an increase in temperature[19]. A previous study revealed that the film thickness of a microhybrid composite was decreased by approximately 30% when the material was heated to 54℃. However, even though the flowability of a composite resin increases with heating, the degree of flow varies among brands and composite classifications, and the flowability of pre-heated composite cannot reach to that of a flowable composite material[21].

It was reported that warming of composites to body temperature or somewhat higher level immediately before placement with heat-producing unit has been shown to improve composite properties and to reduce curing times[22,23]. Walker *et al.*[24] reported that for some composite tested, a significant increase in flexural modulus was observed when specimens were made at simulated intraoral temperature and humidity levels with respect to specimens made at ambient temperature and humidity.

In this study, preheating the composite to the higher temperature induced higher degree of conversion. It was probable that by increasing temperature, greater segmental mobility of pendent groups is possible, resulting in a higher probability of free-radical collision with unreacted methacrylate unit[25,26]. The greater number of radicals might present in the gel state, the greater is the opportunity for them to react when heated and raised to a more mobile condition[27].

If we take the amount of cross-linking into consideration, increasing the degree of conversion by increasing the temperature of the composite will be quite beneficial. Polymerization rate reaches its maximum value, and then the reaction proceeds with deceasing rate (autodeceleration), as propagation also becomes diffusion-controlled. Decreased reaction rate during autodeceleration is attributed to reduced mobility of both monomer and unreacted pendant double-bonds, and decreasing dissociation efficiency of photo-initiators in the viscous medium[28]. Upon continued reaction and cross-linking, mobility is reduced further, and the system becomes even more entangled and viscous until the reaction stops due to polymer vitrification. The onset of vitrification occurs when the increasing glass transition temperature of the reaction mixture reaches the polymerization temperature. The reaction rate will undergo a significant

decrease after vitrification, and the reaction becomes very slow as it is controlled by the diffusion of the reactive species. The diffusion-controlled effect, which produces a slow-down of the polymerization process, will also determine the final degree of conversion. Therefore, increasing the degree of conversion would be quite beneficial, since the amount of cross-linking would increase, and the amount of leachable monomer would be decreased. As the temperature is increased, the reaction rate and final conversion achieved during the resin photopolymerization increased significantly in this study. Therefore, enhancement of strength and microhardness and degree of conversion could be partially attributed an increase in monomer conversion.

Another hypothesis for superior physical properties of pre-heated composite resin in the present study is that the photoinitiator efficiency, particularly in the case of the two-component camphoroquinon/ tertiary amine initiator system, might be slightly enhanced by the reduced resin viscosity. Thus, enhanced mobility at higher temperatures of both monomer and polymer could produce a significant effect of delaying the vitrification point to higher conversion[28]. It is possible that enough camphoroquinone molecules might be converted to the excited triplet state in pre-heated composite to allow adequate propagation of the polymerization reaction for these materials.

In composite resin flowability, filler particle content, shape, and size also may influence composite resin flow[29]. In general, filler loading level, irregular surface contour, and the type of filler size distribution may impact the ability of particles to easily slide past one another[30]. Heating would not directly affect the glassy particle itself, because, within the temperature range imparted at clinically relevant temperatures, the viscosity of the filler particle remains unchanged. Coatings on the filler particle could affect the ease with which a filler particle would move in the warmed resin fluid. Particles not silanated would be more difficult to move than those that are coated, as silanization imparts better resin wetting and, thus, ease of fluid movement around the particle[31].

In the past, heavily filled materials, particularly packable materials, have had difficulty in achieving good marginal adaptation[26]. Therefore, improved flowability of pre-heated microhybrid or packable resin may result in more accurate adaptation to the marginal area or sharp line angle of cavity. As each increment of composite is placed and cured at its ideal polymerization temperature, curing at the elevated temperature might have provided improved physical and mechanical properties.

In the present study, with preheating the composite, the microhardness of the composite was related with the degree of conversion measured by the FT-IR spectra. The finding of the present study supports that the microhardness may be a good indicator of conversion of double bonds[32,33]. Asmussen *et al.*[12] also showed that, for a given resin composite, mechanical properties were a good indicator of degree of conversion.

From the results of the present study, it seems that pre-heating of composite resin up to the higher temperature may induce higher shear bond strength, higher microhardness value, and higher degree of conversion as well. Therefore, when using composite resin in the clinic, pre-heating the composite resin could be recommended to have enhanced physical properties of it. Further study is needed to understand the effect of temperature on the other properties such as volume change and internal stress release of the pre-heated composite resin.

Ⅴ. CONCLUSION

In the present study, both in enamel and dentin, among the composite resin of 4℃, 17℃, 48℃, and 56℃, the pre-heated composite resin up to 56℃ revealed the highest shear bond strength, and pre-heated composite resin to the higher temperature revealed the higher shear bond strength.

Microhardness value and the degree of conversion also were shown to be higher with composite resin of higher temperature.

Within the limitations of the present study, it seems that pre-heating of composite resin up to the higher temperature may induce higher shear bond strength, higher microhardness value, and higher degree of conversion as well. Therefore, when using composite resin in the clinic, pre-heating the compos-

ite resin could be recommended to have enhanced physical properties of it.

Further studies will be needed to assess the effect of pre-heating on other properties of composite resin.

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국문초록

중합 전 열처리가 복합레진의 일부 물성에 미치는 영향

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본 연구의 목적은 복합레진의 광중합 전 열처리가 복합레진의 일부 물성에 미치는 영향을 평가하는 것이다.

우식이 없는 여든 개의 발거된 치아를 사용하였다. 네 가지 온도의 복합레진, 즉, 냉장 보관되어 있던 4℃ 복합레진, 상온 17℃의 복합레진, Calset™를 이용하여 48℃까지 전열처리한 복합레진, 그리고 56℃까지 전열처리한 복합레진을 사용하였다. 복합레진의 물성으로서 치질과의 전단결합강도, 미세경도, 그리고 이중결합 전환율을 측정하였다.

법랑질과 상아질에서 공히 4℃, 17℃, 48℃, 그리고 56℃의 온도 중에서 56℃까지 전열처리한 복합레진이 가장 높은 전단결합강도를 보였으며, 복합레진의 온도가 높을수록 더 높은 전단결합강도를 나타내었다.

복합레진의 온도가 높을수록 더 높은 미세경도를 나타내었다.

복합레진의 온도가 높을수록 이중결합의 전환율이 더 높게 나타났다.

제한된 여건에서 행해진 본 연구의 결과를 통하여 볼 때, 냉장고 또는 실온에 보관되어 있던 복합레진을 미리 열처리하면 치질과의 전단결합강도, 복합레진의 미세경도 및 이중결합 전환율이 증가할 것으로 보인다. 따라서 임상에서 복합레진을 사용 시 복합레진의 물성을 향상시키기 위하여 레진을 미리 열처리하는 것을 추천할 수 있겠다.

주요단어 : 복합레진, 전열처리, 전단결합강도, 미세경도, 이중결합 전환율.