ATATÜRK ÜNİVERSİTESİ YAYINLARI
ATATURK UNIVERSITY PUBLICATIONS

Research Article *Araştırma Makalesi* DOI: 10.17567/currresdentsci.1617446

Influence of Preheating Procedure and Polymerization Modes on Degree of Conversion of Contemporary Resin Composites

Ön Isıtma İşlemi ve Polimerizasyon Modlarının Güncel Rezin Kompozitlerin Dönüşüm Derecesi Üzerindeki Etkisi

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ABSTRACT

Objective: To evaluate the effects of preheating procedure and polymerization modes on degree of conversion (DC) of resin composites.

Methods: Three different types of composites - a conventional (GrandioSO - [GSO]), a bulkfill (GrandioSO x-tra - [GSX]), and a thermoviscous bulkfill (VisCalor bulk - [VCB]) - were investigated. Three specimens were prepared for each combination of the variables (composite type*preheating procedure*polymerization mode). Photo-polymerization was performed using an LED operated in standard (SM), high power plus (HM), and x-tra power (XM) modes. DC was measured with Fourier Transform Infrared Spectroscopy after 5 min and 24 h post-irradiation. Furthermore, three-way analysis of variance followed by a Tukey test at a significance level of $P < .05$ were performed to compare the DC values.

Results: Considering the DC values at two post-irradiation time, preheated VCB group polymerized with HM gave the highest DC value, while GSX cured with XM, regardless of the preheating exhibited the lowest one. Preheating application caused a statistically significant increase in the DC values of the VCB*SM, VCB*HM, and GSO*HM groups at both evaluation periods, and the GSX*SM group at 24h post-irradiation. Regarding the polymerization mode, the ranking depending on the significant differences among all preheated composites tested at both time intervals was HM > SM > XM.

Conclusion: Preheating generally exhibited favorable effects on the DC of all tested composites, with the most significant impact observed in VCB. Additionally, polymerization with x-tra power mode is not recommended to achieve a sufficient DC required for clinically acceptable restorations.

Keywords: bulk fill composite; degree of conversion; FTIR; polymerization mode; preheating; thermoviscousity.

ÖZ

Amaç: Bu çalışmanın amacı ön ısıtma işlemi ve polimerizasyon modlarının rezin kompozitlerin dönüşüm dereceleri üzerindeki etkilerini değerlendirmektir.

Yöntemler: Üç farklı kompozit türü - geleneksel (GrandioSO - [GSO]), bulkfill (GrandioSO x-tra - [GSX]) ve termovisköz bulkfill (VisCalor bulk - [VCB]) - incelendi. Değişkenlerin her bir kombinasyonu (kompozit türü*ön ısıtma işlemi*polimerizasyon modu) için üç örnek hazırlandı. Foto-polimerizasyon, standart (SM), yüksek güç (HM) ve x-tra güç (XM) modlarında çalışan bir LED kullanılarak gerçekleştirildi. Dönüşüm dereceleri, polimerizasyondan 5 dakika- ve 24 saat- sonra Fourier Dönüşümlü Kızılötesi Spektroskopisi ile ölçüldü. Ayrıca, dönüşüm derecesi değerlerini karşılaştırmak için $P < .05$ anlamlılık düzeyinde üç yönlü varyans analizi ve ardından Tukey testi uygulandı.

Bulgular: Polimerizasyon sonrası iki farklı zamanda elde edilen değerler göz önüne alındığında, ön ısıtma uygulanan VCB grubunun HM ile polimerize edildiğinde en yüksek değeri verdiği, ön ısıtma işleminden bağımsız olarak XM ile polimerize edilen GSX'in ise en düşük dönüşüm değerini sergilediği bulundu. Ön ısıtma uygulaması, her iki değerlendirme döneminde de VCB*SM, VCB*HM ve GSO*HM gruplarında, ayrıca polimerizasyondan 24 saat sonra GSX*SM grubunda dönüşüm derecesi değerlerinde istatistiksel olarak anlamlı bir artışa neden oldu. Polimerizasyon modu açısından, her iki zaman diliminde test edilen tüm ön ısıtma uygulanan kompozitler arasında anlamlı farklara bağlı sıralama HM> SM> XM olarak belirlendi.

Sonuç: Ön ısıtma, test edilen tüm kompozitlerin dönüşüm dereceleri üzerinde genel olarak olumlu etkiler göstermiş olup, en belirgin etki VCB'de gözlenmiştir. Ayrıca, klinik olarak kabul edilebilir restorasyonlar için gereken yeterli dönüşüm derecesine ulaşmak amacıyla x-tra güç moduyla polimerizasyon önerilmemektedir.

Anahtar Kelimeler: bulk fill kompozit; dönüşüm derecesi; FTIR; ön ısıtma; polimerizasyon modu; termoviskozite.

Geliş Tarihi/Received 19.09.2023
Revizyon Talebi/Revision Requested 20.09.2023
Son Revizyon/Last Revision 20.10.2023
Kabul Tarihi/Accepted 31.10.2023
Yayın Tarihi/Publication Date 20.01.2025

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Cite this article: Yenidünya ÖG, Erdem A, Akgül N, Yazkan B, Özcan M. Influence of Preheating Procedure and Polymerization Modes on Degree of Conversion of Contemporary Resin Composites. *Curr Res Dent Sci* 2025;35(1): 65-70.

INTRODUCTION

Restorative dentistry has witnessed substantial innovation in composite resins, resulting from advancements in their physico-mechanical properties and manufacturing technologies in recent years. As a result of these improvements, composite resins have been acknowledged as dependable materials for direct restoration.[1] With trends aiming at expediting the restoration process, bulk fill composites that can be placed and light-cured up to 4 or 5 mm without stratification were introduced to the dental profession.[2,3] These contemporary restorative materials are characterized by marked differences from their conventional counterparts in terms of various changes in the chemistry of the monomer, modified inorganic fillers, addition of new photoinitiators, and enhancement of translucency.[4,5]

A crucial parameter for resin-based materials is their degree of conversion (DC), which represents the ratio of unreacted carbon double bonds (C=C) in a polymerized specimen relative to the uncured material.[6] DC values for resin composites, which generally range between 50% and 75%, influence the rheological and mechanical features of the polymer, thereby affecting its clinical performance and functionality.[7,8] Although the minimum DC required for clinically admissible restoration has not yet been specified, DC values below 55% are usually not recommended for occlusal restorative layers.[9] Furthermore, DC is known to be considerably impacted by variables of the curing unit such as light intensity, wavelength, light curing method, curing time, light tip size, irradiation distance, and the chemistry of resin-based restorative materials comprising of the monomer composition, filler size and amount, and type of photo-initiators.[8,10-12]

There are a variety of methods for specifying the DC of light-cured materials. Recently, Fourier Transform Infra-Red Spectroscopy (FTIR) coupled with Attenuated Total Reflectance (ATR) accessories, which is able to quantify infrared light absorbance and transmittance, has emerged as the most widely used among vibrational spectroscopic methods for DC determination.[2,13-15] The rationale behind the ATR-FTIR technique relies on the measurement of alterations in the dipole moment of the bonds in molecules that exhibit vibrational patterns before, during, and after curing.[13]

Due to the increment thickness of bulk fill composites, the requirement for increased light transmission is of paramount importance for achieving adequate DC, which eventually ensures the longevity of the restorations.[16,17] In this regard, it is worth mentioning that polymerization reaction of composite resins is induced by light-curing devices at different energy densities and exposure durations.[18] As a consequence of the latest advancements in the field of light curing units (LCUs), light-emitting diodes (LED) characterized by higher irradiance within a short time interval, which anticipates increased polymerization efficiency and diminished chairside treatment duration, are currently available on the market.[19,20] Considering this context, it ought to be emphasized that, given the importance of their role in clinical applications, dental clinicians should further question characteristics and technical details of LCUs not to compromise patient's health and longevity of restorations.[21]

Preheating application prior to light-curing ensures reduction of material viscosity, enhancement of marginal adaptation, decrease in microleakage, and increases in both radical and monomer mobility, resulting in higher DC values and thereby better physical and mechanical properties of restorative material.[8,22-24] The aforementioned phenomenon may be elaborated as follows: the increased mobility of monomers by means of elevated temperature can lead to a delay in the auto-deceleration stage of the polymerization reaction, resulting in higher monomer conversion.[25-28] This preheating technique may be conducted by inserting syringes or compules of resin-based materials into commercially existing preheating devices set at a temperature range of 39–68 °C.[25] The present study uses VisCalor bulk (VOCO GmbH, Cuxhaven, Germany)—a bulk fill composite specifically designed for preheating—which is considered a noteworthy innovation in terms of dental materials science. Although the preheating procedure has been used in the field of restorative materials for many years, the available literature pertaining to the thermoviscous bulk fill composite is insufficient due to the recent introduction of the material to the dental market, and more investigation is needed to corroborate the effects of promising preheating application.

The present study aimed to investigate the effect of different polymerization modes and preheating application on the DC of novel resin composites. The null hypotheses tested in this study were that the DC is not impacted by 1) the resin composite type, 2) the preheating procedure, and 3) the polymerization mode at two different time intervals (5 min and 24 h post-irradiation).

METHODS

This *in vitro* study investigated three types of resin composites—a conventional universal nanohybrid (GSO; GrandioSO; VOCO GmbH, Cuxhaven, Germany), a nanohybrid bulk fill (GSX; GrandioSO x-tra; VOCO GmbH, Cuxhaven, Germany), and a thermoviscous bulk fill (VCB; VisCalor bulk; VOCO GmbH, Cuxhaven, Germany) (Table 1). Drawing on the results reported by Kincses et al.[26] and using analysis of variance (ANOVA: fixed effects, special, main effects and interactions, $\alpha = 0.05$, power $[1-\beta] = 0.95$, effect size = 0.639), the total sample size required for DC analysis was established as 53 (G*Power version 3.1; Heinrich-Heine-Universität Düsseldorf). Therefore, the experiment was conducted with n = 3 for each resin composite*preheating procedure*polymerization mode subgroup. Eighteen specimens of each resin composite, constituting a total of 54 specimens, as presented in Figure 1, were randomly separated into six subgroups (n = 3) in accordance with the preheating application (p+: with preheating/p-: without preheating) and polymerization mode (SM/HM/XM) interactions.

Table 1. Specifications of tested composite resins

Material	Type	Composition	Filler content %	Lot number	Manufacturer
GrandioSO [GSO]	Universal nanohybrid composite	**Matrix:** Bis-GMA, BisEMA, TEGDMA **Filler:** Glass ceramic, silicon dioxide	89 (w/w)	2108726	VOCO, Cuxhaven, Germany
GrandioSO x-tra [GSX]	Nanohybrid bulk fill composite	**Matrix:** Bis-GMA, BisEMA, aliphatic dimethacrylate **Filler:** Inorganic filler, organically modified silica	86 (w/w)	2112568	VOCO, Cuxhaven, Germany
VisCalor bulk [VCB]	Thermoviscous nanohybrid bulk fill composite	**Matrix:** Bis-GMA, aliphatic dimethacrylate **Filler:** Inorganic filler	83 (w/w)	2111548	VOCO, Cuxhaven, Germany

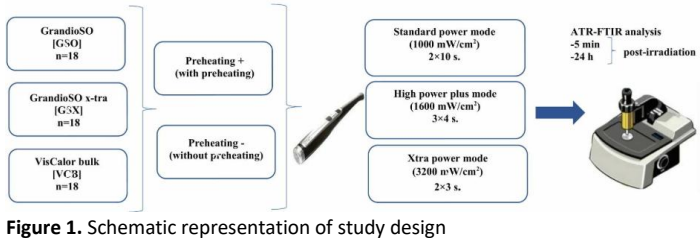

Figure 1. Schematic representation of study design

According to the manufacturer's instructions, a VisCalor Dispenser (VOCO GmbH, Cuxhaven, Germany) was used to preheat the composite compules by selecting setting 1 (for the VisCalor bulk, 30s) and setting 2 (for other studied composites, 70s) as required. In the non-preheated subgroups, the composite compules were maintained at room temperature (25 °C) with no preheating, and were included as a control group in this experiment. In each 'composite type*preheating procedure' subgroup, the uncured material was inserted into cylindrical stainless-steel molds of 8 mm × 4 mm and 8 mm × 2 mm for the bulk fills and their conventional counterparts, respectively.

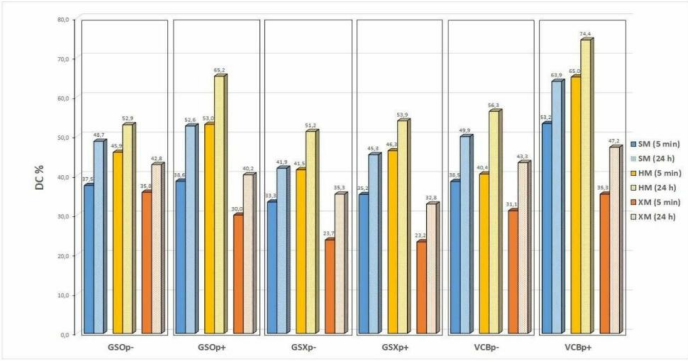

Figure 2. The mean DC of investigated materials according to the preheating procedure and polymerization mode at two-time intervals

Infrared spectra of the specimens' top surfaces were collected by employing Fourier Transformed Infrared Spectroscopy (FTIR, Nicolet iS50, Thermo Scientific, USA) equipped with a universal ATR accessory along with a diamond crystal (Thermo Fisher Scientific Co., Waltham, MA, USA) in absorbance mode. Subsequently, 32 scans at a resolution of 4 cm^{-1} within a wavelength spectrum of 4000–400 cm^{-1} were obtained. Furthermore, a background FTIR spectrum was recorded prior to each measurement. A preliminary readout of the uncured specimen was considered the unpolymerized reference. Photo-polymerization was performed using a LED unit VALO® Cordless (Ultradent Inc., South Jordan, UT, USA) operated in standard mode (1000 mW/cm^2 - [2 × 10s]), high power plus mode (1600 mW/cm^2 - [3 × 4s]), and x-tra power mode (3200 mW/cm^2 - [2 × 3s]). The specimens were stored in a lightproof oven within a desiccator with silica gel at 37°C for either 5 min or 24 h before DC measurement. Additional spectra of the cured specimens at 5 min and 24 h post-irradiation were obtained under the aforementioned conditions. Moreover, the crystal plate was cleaned cautiously with absorbent paper and ethyl alcohol between each sequence of monomer-polymer conversion.

Using a standard baseline technique,[29] the percentage of DC on the tested surfaces was calculated by determining the variation in the ratio of absorbance intensities (peak heights) of the aliphatic C=C stretching vibrations at 1636 cm^{-1} and aromatic C...C stretching vibrations at 1608 cm^{-1} using the following equation:

$$DC\ (\%) = (1 - \frac{(A_{1636} / A_{1608})after\ curing}{(A_{1636} / A_{1608})before\ curing}) x\ 100$$

Statistical Analysis

The data were analyzed with IBM SPSS version 23 (IBM Software Group, Chicago, IL, USA), while a Shapiro-Wilk test was implemented to examine the distribution normality. Furthermore, a three-way analysis of variance (ANOVA) (with resin composite type, preheating procedure, and polymerization mode as the main factors) followed by a Tukey test were used to examine the DC values among the experimental groups at two post-irradiation time. The significance level was established as $P<.05$.

RESULTS

The three-way ANOVA revealed significant effects of not only the three main factors but also the interactions of "resin composite* preheating procedure" as well as "preheating procedure* polymerization mode" at the two different time intervals, and interaction of "resin composite*polymerization mode" at 5 min post-irradiation (P <.001). Additionally, triple interaction was identified to be statistically significant at both 5 min and 24 h post-irradiation (in order of P =.001, P <.001). The results of the DC_{5min} and DC_{24h} values are presented in Table 2 and Table 3, respectively. Furthermore, Figure 2 depicts the DC values of each investigated variable in pairs, with reference to the two-time intervals. Considering the DC values at 5 min and 24 h post-irradiation separately, the highest DC value was observed in the preheated VCB group polymerized with HM, while the lowest degree of conversion was detected in the GSX cured with XM, regardless of the preheating procedure (P <.001). Preheating application caused a statistically significant increase in the DC values of the VCB*SM, VCB*HM, and GSO*HM groups at both evaluation periods, while that of the GSX*SM group exhibited statistical significance at 24 h post-irradiation (P <.001). Concerning the polymerization mode, their ranking based on the significant differences among all preheated composites tested at both time intervals was HM > SM > XM (P <.001).

Table 2. Mean degree of conversion (%) and standard deviation (mean ± SD) of each investigated variable at 5 min post-irradiation

Polymerization mode	Preheating procedure	Composite type		
		GSO	**GSX**	**VCB**
SM	**p-**	37.5 ± 0.7^{DEF}	33.3 ± 0.8^{FG}	38.5 ± 2.7^{DEF}
	p+	38.6 ± 1.2^{DEF}	35.2 ± 0.3^{EFG}	53.2 ± 4.0^{B}
HM	**p-**	45.9 ± 0.6^{C}	41.5 ± 0.7^{CD}	40.4 ± 2.6^{CDE}
	p+	53.0 ± 0.9^{B}	46.3 ± 0.3^{C}	65.0 ± 2.3^{A}
XM	**p-**	35.8 ± 1.1^{DEFG}	23.7 ± 1.1^{H}	31.1 ± 2.3^{G}
	p+	30.0 ± 0.9^{G}	23.2 ± 0.3^{H}	35.3 ± 4.5^{EFG}

Notes: A-H: There is no difference between the interactions (resin composite*preheating procedure*polymerization mode) with the same upper case letters.
Abbreviations: GSO, GrandioSO; GSX, GrandioSO x-tra; VCB, VisCalor bulk; SM, standard mode; HM, high power plus mode; XM, x-tra power mode; p-, without preheating; p+, with preheating.

Table 3. Mean degree of conversion (%) and standard deviation (mean ± SD) of each investigated variable at 24 h post-irradiation

Polymerization mode	Preheating procedure	Composite type		
		GSO	**GSX**	**VCB**
SM	**p-**	48.7±0.6^{EFG}	41.9±0.8^{IJ}	49.9±1.1^{DEF}
	p+	52.6±0.7^{CDE}	45.3±0.2^{GHI}	63.9±3.7^{B}
HM	**p-**	52.9±0.3^{CDE}	51.2±0.8^{DEF}	56.3±0.6^{C}
	p+	65.2±0.2^{B}	53.9±0.1^{CD}	74.4±3.4^{A}
XM	**p-**	42.8±1.1^{IJ}	35.3±1.2^{K}	43.3±1.2^{HIJ}
	p+	40.2±1.2^{J}	32.8±0.3^{K}	47.2±1.6^{FGH}

Notes: A-K: There is no difference between the interactions (resin composite*preheating procedure*polymerization mode) with the same upper case letters.
Abbreviations: GSO, GrandioSO; GSX, GrandioSO x-tra; VCB, VisCalor bulk; SM, standard mode; HM, high power plus mode; XM, x-tra power mode; p-, without preheating; p+, with preheating.

DISCUSSION

Effect of chemical composition on DC

Within the limits of this research, the first part of the null hypothesis was rejected since the current study confirmed that the DCs of the tested materials were impacted by the resin composite type. From a theoretical standpoint, DC (%) differences which are closely related with intricate polymerization process are expected as the monomer composition, inorganic filler characteristics vary on a large scale from material to material.[13] In a previous study, it was specified that increasing filler-matrix ratio proportionally reduces degree of conversion, because raised amounts of inorganic fillers are an impediment for polymeric chain propagation.[27] Taking into consideration of tested composites in this study, the ranking of the numerical values of DC was mostly VCB > GSO > GSX. In view of this, it is not surprising that VCB composites exhibited the highest DC values as a result of their lower filler loading (83% wt.). Notably, a discrepancy was noted between the DC values and the filler amounts of the GSO (89% wt.) and GSX (86% wt.) groups. The explanation for the higher DC values in the GSO may be attributed to its application at 2 mm increment thickness, and the presence of TEGDMA, which could have positively affected the DC in the monomer structure of the GSO.

Furthermore, the DC of composite resins is essentially impacted by the nature and quantity of each monomer in their composition.[6] Ultimately, the DC of the different monomer systems diminishes in the following order: TEGDMA > UDMA > Bis-EMA > Bis-GMA.[2] Unfortunately, the lack of data provided by the manufacturers in the package inserts and on their websites about the exact ratio of the assorted monomers, as they were proprietary, made the interpretation of the existing results an arduous task.

DC change depending on preheating

Preheating application is an innovative approach that could ameliorate the handling characteristics of dental materials, as well as their physical and mechanical properties.[30] Based on the effects of the preheating procedure on DC observed in the current study, the second null hypothesis was rejected. Our findings coincide with those of various studies[23,24,31,32] reporting that preheating causes increased molecular mobility, thereby allowing the system to attain higher level monomer conversion before vitrification. On the other hand, in several studies[15,28,33,34], it was emphasized that no significant effect was stated about the preheating procedure on monomer conversion. For instance, unlike the present study, Kincses et al.[26] specified that preheating had no beneficial impact on the DC of thermoviscous VisCalor bulk. Furthermore, a previous study that investigated the effects of pre-heating time and exposure duration on the post-irradiation properties of a thermoviscous resin composite declared that the DC did not alter with preheating application and time (no heat, T3-30 s, T3-3 min), since a reduction in the composite temperature was observed after its removal from the heating instrument.[8] This inconsistency between the findings of the current study and former investigation may have resulted from the use of different heating devices. It is important to mention that compules do not need to be removed from VisCalor Dispenser used in this study, and that's why this device can maintain increased temperature throughout the procedure as distinct from the other heating instruments.[26] In another study by Tauböck et al.[22] in which they evaluated influence of preheating on shrinkage force and monomer conversion of high-viscosity bulk fill resin composites, it was notified that preheating prior to photoactivation either maintains or increases the DC.

Influence of polymerization mode on DC

In spite of the fact that the polymerization reaction is completely chemical, dental practitioners are still regulating several momentous points of the reaction such as the curing time and radiant energy.[1] Comparisons of alternative polymerization protocols are generally made by changing the light activation time and irradiance while preserving stationary radiant exposure.[20] Considering the impact of polymerization modes in this study, especially in all the preheated groups, the high power plus mode presented a significantly higher DC than the standard mode and x-tra power modes, respectively. In line with the data obtained from the current study, the third part of the null hypothesis, which emphasizes that the DC is not affected by polymerization modes, was rejected.

In a previous study conducted by Sadeghyar et al. [3] who examined limited reciprocity in curing efficiency of bulk fill resin composites, three LCUs of rising radiant emittance capacity (1200, 2000 and 3200 mW/cm^2) were utilized. It was stated that irradiance for 10 s at 1200 mW/cm^2 displayed mostly better hardness values than by using LCUs of higher radiant emittance with reciprocally declined curing times, to sustain stationary dose of energy density. Furthermore, Ilie and Stark claimed that to preserve the mechanical features of resins in depth, the necessary energy density should be achieved at moderate irradiance coupled with enhanced exposure time.[4] A study that investigated the effect of light-curing protocols (standard and x-tra power modes) on the mechanical behavior of bulk fill resin composites reported that higher irradiance in a short period jeopardizes the mechanical properties of composite resins, leading to undesirable clinical outcomes.[5] As an explanation of the aforementioned finding, it could be pointed out that low power irradiance in a longer time duration ensures leisurely polymerization, which improves the mechanical behavior of composites, since extended chains with higher molecular weight are created in comparison with high power irradiance. The results of this investigation are consistent with those of Daugherty et al.[11] who stated that bulk fill composites polymerized with high irradiance*short/ultra-short curing time combinations may not provide a sufficient degree of polymerizetion, in turn, leading to undesirable clinical features. On the other hand, Atria et al. [35] have accentuated that different curing modes (High- 1200 mW/cm^2; Low- 650 mW/cm^2; Soft-start- [650-1200] mW/cm^2; and Turbo- 2000 mW/cm^2) with the same exposure time do not significantly impress the DC values of a composite resin. Drawing on the above observations, it is crucial to underline that dental professionals should have notice of technic elaborations and properties of LCUs by the side of the curing time and radiant irradiance suggested by the manufacturer of each dental material.[21]

For all composite resins analyzed in this study, the DC at 24 h post-irradiation was greater compared to those obtained immediately post-cure. This finding is in harmony with the studies conducted by Yang et al.[8,28] As corroborating with the literature, it was concluded that DC may not be optimized 5 min post-irradiation, and it should be anticipated for up to 24h in order to obtain eventual degree of post-polymerization conversion.[17]

A limitation of this experiment is that only one brand of composite resin was tested. Furthermore, the use of different specimens at the two time intervals (5 min and 24 h post-irradiation) may be preferred because removal of the cured specimens from the ATR crystal for re-evaluation at 24 h post-irradiation could be detrimental to the specimens, resulting in poor spectra recordings. Moreover, the outcomes of the current research should be corroborated with *in vivo* studies because they simulate intraoral environment conditions

completely and prevent erroneous prediction from the results of *in vitro* methodologies.

CONCLUSION

Within the limitations of this laboratory study, the following conclusions can be deduced:

1. VisCalor bulk indicated the highest degree of conversion in comparison with other tested composites when preheated as suggested.

2. Preheating procedure had generally favorable effects on DC of the tested composites, besides that the most significant impact was observed in VisCalor bulk group.

3. X-tra power mode, which has higher irradiance in a short time interval may not be recommended for adequate degree of conversion.

Etik Komite Onayı: Pamukkale Üniversitesi Etik Kurulu'ndan etik onay alındı (12/08/2020; E-60116787-020/47590).
Hasta Onamı: Deneysel çalışma laboratuvar çalışması olduğu için hasta onayı gerekmemektedir.
Hakem Değerlendirmesi: Dış bağımsız.
Yazar Katkıları: Fikir, O.G.Y., N.A. ve M.O.; Tasarım, O.G.Y., A.E. ve B.Y.; Denetleme, A.E., N.A. ve M.O.; Fon Sağlama, O.G.Y.; Materyaller, O.G.Y.; Veri Toplama ve/veya İşleme, O.G.Y, A.E. ve B.Y.; Analiz ve/veya Yorumlama A.E. ve N.Y.; Literatür Taraması, O.G.Y.; Makale Yazım, O.G.Y.; Eleştirel İnceleme, O.G.Y., N.A., B.Y. ve M.O..
Çıkar Çatışması: Yazarlar herhangi bir çıkar çatışmasının olmadığını beyan ederler.
Finansal Destek: Araştırma Pamukkale Üniversitesi Bilimsel Araştırma Projeleri tarafından desteklenmiştir [Proje numarası: 2020BSP017].

Ethics Committee Approval: Ethical approval was obtained from the Ethics Committee of the Pamukkale University (12/08/2020; E-60116787-020/47590).
Informed Consent: Since the experimental study is a laboratory study, patient consent is not required.
Peer-review: Externally peer-reviewed.
Author Contributions: Conception, O.G.Y., N.A., and M.O.; Design, O.G.Y., A.E., and B.Y.; Supervision, A.E., N.A., and M.O.; Funding Acquisition, O.G.Y.; Materials, O.G.Y.; Data Collection and/or Processing, O.G.Y., A.E., and B.Y.; Analysis and/or Interpretation A.E. and N.Y.; Literature Review, O.G.Y; Writing – Original Draft Preparation, O.G.Y.; Critical Review, O.G.Y., N.A., B.Y., and M.O..
Conflict of Interest: The authors declare that they have no conflict of interest.
Financial Disclosure: The research was supported by the Pamukkale University, Scientific Research Projects [Project number: 2020BSP017].

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