

Investigation of the Effect of Preheating Before Polymerization on the Color and Gloss of Composites

Polimerizasyon Öncesi Ön Isıtmanın Kompozitlerin Renk ve Parlaklığı Üzerindeki Etkisinin İncelenmesi

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ABSTRACT

Objective: The aim of this study was to investigate the effects of preheating on the color stability and surface gloss of composite resins before polymerization.

Methods: A total of 90 samples (8 mmx 2 mm) of three composite resins with different particle sizes were prepared. Half of the samples were prepared from composite tubes at room temperature and the other half were prepared from composite tubes preheated to 60°C with the Ease-It™ heating device (n=15). After polymerization and polishing, color measurements were performed using a spectrophotometer and surface gloss was measured with a glossmeter. All samples underwent 10,000 thermal cycles, after which measurements were repeated. The Kolmogorov-Smirnov test was used to assess the normality of data distribution, and Levene's test was applied to evaluate homogeneity of variance ($P<.05$).

Results: There was no statistically significant difference in the gloss values of preheated and unheated composite resins ($P>.05$). Heat treatment did not cause a statistically significant difference between the composite types ($P>.05$), but it caused a statistically significant difference in the mean color change values ($P<.001$). The average color change values of the preheated groups were significantly lower than the unheated groups ($P<.05$).

Conclusion: Preheating composite resins prior to polymerization did not significantly affect surface gloss but significantly reduced discoloration.

Keywords: Composite resin, Heating, Polymerization, Staining, Surface gloss.

ÖZ

Amaç: Bu çalışmanın amacı, polimerizasyon öncesi ön ısıtmanın kompozit rezinlerin renk stabilitesi ve yüzey parlaklığı üzerindeki etkilerini araştırmaktır.

Yöntemler: Farklı partikül boyutlarına sahip 3 farklı kompozit rezinden toplam 90 örnek (8mmx 2 mm) hazırlandı. Örneklerin yarısı oda sıcaklığındaki kompozit tüplerden, diğer yarısı ise Ease-It™ ısıtma cihazı ile 60°C'ye ısıtılmış kompozit tüplerden hazırlandı (n:15). Polimerizasyon ve parlatma işlemlerinden sonra spektrofotometre ile renk ölçümleri ve Glossmetre ile yüzey parlaklığı ölçümleri yapılmıştır. Tüm numuneler 10.000 döngümlük bir termal döngüye tabi tutulmuş ve ölçümler tekrarlanmıştır. Sayısal verilerin gruplar arasındaki normal dağılımını değerlendirmek için Kolmogorov-Smirnov testi kullanılırken, varyansın homojenliğini değerlendirmek için Levene testi kullanılmıştır ($P<.05$).

Bulgular: Önceden ısıtılmış ve ısıtılmamış kompozit rezinlerin parlaklık değerlerinde istatistiksel olarak anlamlı bir fark yoktu ($P>.05$). Isıl işlem kompozit tipleri arasında istatistiksel olarak anlamlı bir farka neden olmazken ($P>.05$), ortalama renk değişim değerlerinde istatistiksel olarak anlamlı bir farka neden olmuştur ($P<.001$). Isıtılan grupların ortalama renk değişim değerleri ısıtılmayan gruplardan anlamlı derecede düşüktü ($P<.05$).

Sonuç: Ön ısıtma işlemi kompozit rezinlerin parlaklık değerleri üzerinde anlamlı bir fark yaratmamıştır; ancak renk değişimini önemli ölçüde azaltmıştır.

Anahtar Kelimeler: Kompozit rezin, Isıtma, Polimerizasyon, Boyanma, Yüzey parlaklığı.

INTRODUCTION

With the development of aesthetic standards in dentistry, various approaches and techniques have been developed to improve the cosmetic quality of composite resins. One of the current methods introduced during the development of these approaches and techniques is the heating of composites prior to application.

Heating composite resins before application improves the physical and mechanical properties of these materials by increasing the degree of monomer conversion and provides superior aesthetic results.¹⁻³ The viscosity and free radical mobility of the material increases with increasing composite body temperature. The polymerization reaction is completed by increasing the spread of polymer chains due to the increase in viscosity. More double bonds develop, leading to a decrease in the amount of unreacted monomer. Preheating of resin-based materials not only increases conversion, but also improves marginal adaptation and reduces polymerization shrinkage.^{1,4,5}

The gloss of a resin composite affects its aesthetic qualities, its applications in dentistry and the long-term reliability of the composite restoration. Therefore, gloss is an important factor for visual standards.⁶ As the gloss increases, the materials adapt better to the adjacent tooth and the tooth on which it is placed. Even though there is a perfect color match between the tooth and the tissue, differences on surface gloss can be detected.⁷

Surface discoloration is a significant aesthetic problem associated with composite resins. Therefore, long-term color permanence is very important for these materials. Staining; It is affected by the resin formulation, filler particle ratio, and filler particle size. Although there have been many improvements in materials to reduce surface discoloration, discoloration is still a cause of clinical failure for resin composite materials.⁸

The durability of a dental restoration using resin composites may be related to physicochemical and surface properties. Although numerous studies have examined the effect of preheating on various restorative materials, there is insufficient data on surface quality and durability.¹ This study examined how preheating affects the gloss and color stability of composite resins with various filler types and sizes (Harmonize by Kerr, G-aenial Anterior by GC, and Filtek Universal by 3M-ESPE). The first null hypothesis of this study states that there will be no significant difference in gloss between composite resins with and without preheating. The second null hypothesis states that there will be no significant difference in color changes between composite resins with and without preheating treatment.

METHODS

In this study, three different composites were used: Harmonize (nanohybrid, NH), G-aenial Anterior (micro-hybrid, MH), Filtek Universal (nanofilled, NF)). The composites used for the study are shown in Table 1. The composites were divided into two subgroups due to whether preheating was done or not.

Table 1. Composite materials used in the study and content information

Composite	Filler type	Composition	Brand (Lot number)
G-aenial Anterior	Micro-hybrid	UDMA, dimethacrylate co monomers, Prepolymerized silica, strontium and lanthanoid fluoride-containing fillers 76 wt% Fumed silica	GC Europe, Leuven, Belgium
Harmonize	Nano-hybrid composite	Silica-zirkonia filler Barium glass filler Silica and ytterbium trifluoride Inorganic fillers 75% by weight, 55% by volume Bis-EMA, Bis-GMA, TEGDMA, and/or UDMA	Kerr, Orange, USA
Filtek Universal Restorative	Nano-filled composite	20nm silica filler 4-11nm zirkonya filler Ytterbium trifluoride filler inorganic filler 76.5% by weight/ 58.4% by volume AUDMA, AFM, diurethane-DMA, and 1,12 dodecane-DMA.	3M-ESPE, St. Paul, MN, USA

Group 1 (Preheated Group): For the preparing of Group 1 samples, composite resin tubes were heated to 60°C for 15 minutes using a heating device (Ease-It™ Heating Unit, Ronvig Dental, Daugaard, Denmark) (Figure 1). The heating device alerts when the temperature of the tubes reaches 60°C. To avoid heat loss when preparing samples from the composite tubes, the tube was quickly removed from the heating device and the required composite material was placed into the Teflon mold with a metal spatula and then quickly placed back into the device. To obtain a flat and smooth surface and to remove residual material, the composite material was placed between transparent polyethylene strips on the top and bottom and sandwiched between two glass coverslips. Composite samples were polymerized for 20 s using a 1000 mW/cm² LED light device (DTE LUX E, Borkstrasse, Muenster, Germany) in standard mode, keeping the tip in contact with the glass coverslip (n = 15).

Group 2 (Unheated Group): For the preparing of Group 2 samples, the composite resin tubes were kept at room temperature (20-22 °C) until the polymerization stage. The samples were prepared by placing the composite material into teflon molds using a metal spatula. To obtain a flat and smooth surface and to remove residual material, the composite material was placed between transparent polyethylene strips on the top and bottom and sandwiched between two glass coverslips. Composite samples were polymerized for 20 s using a 1000 mW/cm² LED light device (DTE LUX E, Borkstrasse, Muenster, Germany) in standard mode, keeping the tip in contact with the glass coverslip (n=15).

All specimens were finished and polished using a two-stage spiral polishing system (ClearfilTwist Dia, Kuraray, Japan). Dark blue and light blue spiral tires, respectively, were applied to the specimen surface in circular motions at 10,000 rpm for 30 seconds under water cooling. The spirals were renewed after every five specimens. Specimens were ultrasonically cleaned (Amsco, Reliance Sonic 250, Steris Corp., Mentor, OH, USA) for 10 minutes in deionized water to get rid of polishing residue after the polishing procedure. Samples were then immersed in distilled water at 37°C in an incubator (FN 500, Nüve, Turkey) for 24 hours. Then initial color and gloss measurements were calculated.

Evaluation Methods

1. Color measurement

A color cabinet was used to maintain uniformity in color measuring. The inside of the color measurement cabinet was covered with a gray background, and measurements were conducted on a white background. The light power inside the color measurement cabinet was adjusted to 6500K (MasterTL-D 90 Graphica 18W/965, Philips, Signify, Poland) following CIE standards.

Color measurements were repeated three times from the center point of each sample using a spectrophotometer (Vita Easysshade V, VITA Zahnfabrik, Germany). Before sample measurements, the spectrophotometer was calibrated with the indicated calibration plate. The average value of the measurements taken was accepted as the final value for each sample. The CIE-L*a*b* color system, defined as a 3D measurement system, was used to evaluate the numerical data obtained: "L" stands for luminance, "a" for the red-green ratio, and "b" for the yellow-blue color ratio. The CIE 2000 formula is the most up-to-date method for the calculation of colour differences and is recommended by CIE due to its superior performance compared to CIELAB and better compatibility with visual assessments. Therefore, calculations were made using the CIE2000 formula over the CIE-Lab values.^{9,10}

2. Gloss Measurements

The surface gloss of the samples was measured using a gloss meter (Novo-Curve, Rhopoint Instrumentation, Bexhill on Sea, UK) with a 2 mm × 2 mm area and 60° geometry. Three measurements of each sample were taken at 60° angles of light incidence and reflection.¹¹ During the measurements, a black opaque plastic cup was placed over the samples to eliminate the effect of ambient light. The obtained gloss values were recorded in GU units.¹² A statistical analysis was performed on the mean values of the measurements

Following the initial color and gloss measurements, all samples were subjected to 10,000 thermal cycles (Mechatronic thermo-cycler, Seelbach, Germany) to simulate the 1-year thermal cycles occurring in the oral cavity. Samples were subjected to 10,000 thermal cycles in a thermal cycler (Thermocycler the 1100, SD Mechatronik, Germany) between 5°C-55°C with a dwell time of 30 seconds and a transition time of 10 seconds.¹³ The final color and gloss measurements of all samples were reassessed following the aging process with a thermal cycle.

Statistical Analysis

In this study, the IBM SPSS v25 program (IBM SPSS Corp., Armonk, NY, USA) was used for statistical analysis. Before looking at how important numerical data was for each group, the Kolmogorov-Smirnov test and Levene's Test for Homogeneity of Variance were used to see if the data were normally distributed. A two-way analysis of variance (ANOVA) in repeated measures examined changes in brightness and color values. Bonferroni correction was used for multiple comparisons. The results of the analysis are presented as the mean ± standard deviation.

The sample size required for the study was made for the F test by taking $\beta = 0.80$, $\alpha = 0.05$ and the effect size = 0.35 as a result of the literature research (thesis proposal, article, bibliography) with the G*Power 3.1 programme and a total of 84 samples (n = 28 in each group) should be taken. For this reason, a total of 90 samples, 30 for each group, were planned in the study. Each group was divided into two subgroups as heated and unheated (n=15).¹⁴

The assumptions of normal distribution ($P > .05$) and homogeneity of variance ($P > .05$) were met. A two-way analysis of variance (ANOVA) with repeated measures was used to look at how the gloss and color of composite resin samples changed depending on whether they were heated or not and after being aged by thermal cycling. The results were evaluated at a 95% confidence interval, with significance at the $P < .05$ level. While interpreting the results, 0.05 was taken as the significance level, and it was stated that there was a difference in the case of $P < .05$ and no significant difference in the case of $P < .05$

RESULTS

In our examination the gloss values of composite groups with and without heat treatment; there is a statistically significant difference between composite types ($P < .001$) (Table 2). However, no statistically significant difference was observed between the gloss values of composite resins with and without heat treatment (Figure 1) ($P = .344$). In addition, the interaction between the different composite types used in the study and heat treatment was statistically significant ($P < .001$). A statistically significant difference was observed in NH and NF composites ($P < .001$). The average gloss value of the heated group was lower than the unheated group in the NH composite, while in the NF composite, the gloss values of the heated group were higher than the unheated group (Figure 1, Table 2).

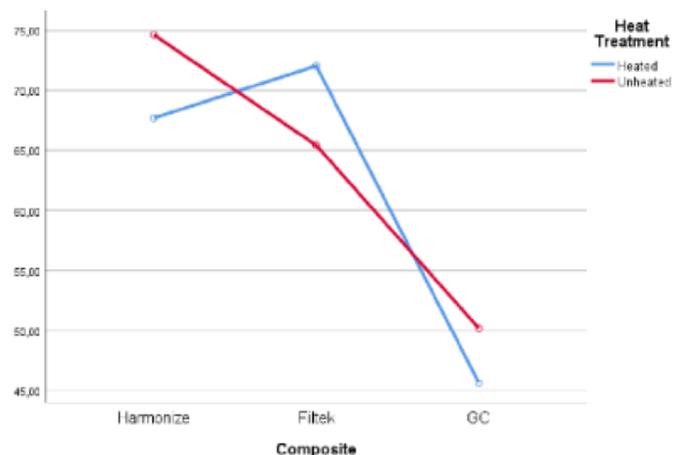


Figure 1. Gloss variation of composite types according to heat treatment or not

When we look at the effect of the aging process on gloss values, the aging process has a statistically significant effect on gloss values ($P < .001$) (Figure 2, Table 2). When we look at the interaction for 'composite' and 'aging', statistically significant differences were obtained ($P = .042$). Also, to find out why this was important, we saw that the gloss values dropped significantly after the aging process for all composite types. The NH composite group had the biggest decrease in gloss values (mean change 11.655±.) (Figure 2, Table 2).

Table 2. Mean gloss values of samples before and after thermal aging

Descriptive Statistics					
Aging	Composite	Heat Treatment	Mean	Std. Error	N
Before thermal aging	Harmonize	Heated	71,8093	13,01181	15
		Unheated	82,1727	5,81421	15
		Total	76,9910	11,21739	30
	Filtek	Heated	76,4087	9,79443	15
		Unheated	70,6247	9,86910	15
		Total	73,5167	10,09871	30
	GC	Heated	48,5143	7,16240	14
		Unheated	53,0180	6,86071	15
		Total	50,8438	7,25247	29
	Total	Heated	65,9652	15,84226	44
		Unheated	68,6051	14,27216	45
		Total	67,3000	15,04146	89
After thermal aging	Harmonize	Heated	67,6693	10,08833	15
		Unheated	60,2127	13,03018	15
		Total	63,9410	12,06142	30
	Filtek	Heated	63,5780	11,94395	15
		Unheated	67,0933	6,91847	15
		Total	65,3357	9,75565	30
	GC	Heated	67,6693	10,08833	15
		Unheated	60,2127	13,03018	15
		Total	63,9410	12,06142	30
	Total	Heated	42,7621	5,42429	14
		Unheated	47,3327	4,83606	15
		Total	45,1262	5,54589	29
Total	Heated	58,3495	14,40205	44	
	Unheated	58,2129	12,05400	45	
	Total	58,2804	13,19116	89	

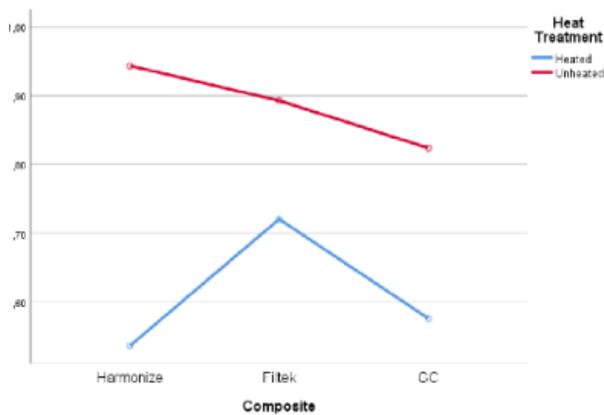


Figure 2. Gloss change in composite types after aging proces

Following analyzing the color changes, no statistically significant difference was found between composite types ($P=.47$) (Figure 3, Table 3). However, heat treatment has a statistically significant effect on the average values of color change ($P<.001$). In addition, when we look at the interaction between the different composite types used in the study and heat treatment applications, there is no statistically significant difference ($P=.388$). The color change values of the heat-treated groups were found to be statistically significantly lower than the unheated group (Figure 3, Table 3). There was a statistically significant change between the color values of the heated groups and the unheated groups ($P<.001$) (mean change 0.275) (Table 3).

Table 3. Mean values of color change

Descriptive Statistics				
Composite	Heat Treatment	Mean	Std. error	N
Harmonize	Heated	0,54	0,16	15
	Unheated	0,94	0,39	15
	Total	0,74	0,36	30
Filtek	Heated	0,72	0,21	15
	Unheated	0,89	0,40	15
	Total	0,81	0,32	30
GC	Heated	0,58	0,23	14
	Unheated	0,82	0,49	15
	Total	0,70	0,40	29
Total	Heated	0,61	0,21	44
	Unheated	0,89	0,42	45
	Total	0,75	0,36	89

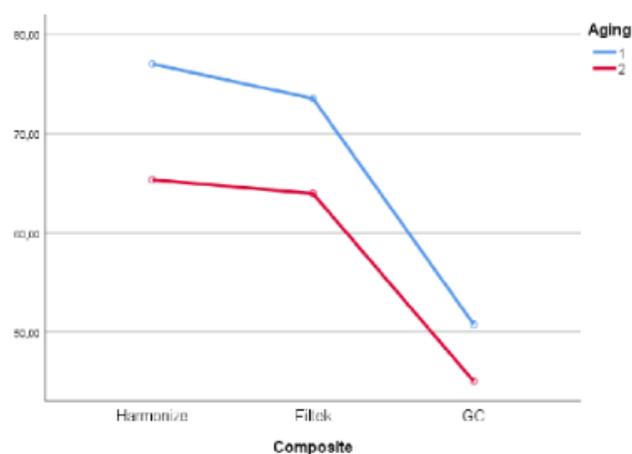


Figure 3. Mean values of color change of heated and unheated composite types

DISCUSSION

Gloss is a very important factor for an aesthetic and natural appearance on composite resin restorations.¹⁵ The greater amount of light reflected directly onto any surface, the higher gloss value recorded for that surface. A glossy surface is also an indicator of a smooth surface.^{16,17} In addition to improving the aesthetic appearance, increased surface gloss also prevents plaque retention and the formation of colored biofilms over time.¹⁸

In this study, no significant difference was found in the gloss of the composite resins as a result of the preheating process applied to the resin samples. The fact that the resin composite depends on the filler material rather than the resin matrix, which is the part of the resin composite sensitive to preheating, may explain the lack of a significant effect of preheating on gloss.¹⁹ Therefore, the first null hypothesis of the study was accepted.

Although no significant difference was found on gloss, when the data obtained were evaluated; the gloss of MH resin composite was lower than NF and NH resin composites at all time points, although not significantly. The inorganic filler size of MH resin composite is larger than NF and NH composites. Gloss is affected by several factors such as filler size distribution, filler refractive index and viscosity of resin matrix components.²⁰ When the filler size increases, light can be reflected more diffusely. Therefore, gloss values may have been recorded lower.^{6,21} Also as another result; preheating decreased the gloss of NH and MH resin composites, while it increased the gloss of NF resin composites. Composite resins with different organic matrix and particle sizes exhibit different properties.^{22,23} This is thought to be due to the smaller particle size of the NF composite. However, further research on this subject is required to reach definitive conclusions.

While gloss values can be obtained using various measurement methods, they also vary depending on experimental conditions such as the spectral distribution of light and viewing angle. Gloss measurements at 60° angle are considered more reliable clinically as they are closer to the angle at which an average person would observe any surface, and therefore in this study, surface gloss measurements were performed at 60° angle.²¹

Surface quality and gloss of resin composites are known to be related to material type, polishing procedure,²⁴ light source and aging procedure.^{25,26} The polishing system applied with a polishing kit containing diamond particles was applied as the standard protocol in this study due to their ability to produce smooth and shiny surfaces on various resin composites.^{21,27,28} A single operator polished all of the specimens, and the same light source cured them all to ensure consistency in the experimental procedure.

The thermal cycle is a current and widely used method to simulate the aging effect of daily thermal changes in the oral environment on resin materials. With this method, it is possible to understand the long-term performance of composite materials. According to studies, exposing the samples to 10,000 thermal cycles at 5-55 C° simulates 1-year thermal aging under clinical conditions. In this study, the color and gloss changes of resin samples after 1 year of thermal aging were examined, so the samples were exposed to 10,000 cycles of thermal cycling.²⁸ Studies on this subject have shown that thermal change and water absorption on resin samples over time can cause changes in the microstructure, leading to an increase in coloration and gloss loss. In other words, thermal cycling can affect the surface morphology of the composite material, causing light to scatter in different ways on the

surface.²⁸ In this study, as shown in previous studies,^{28,29} all composite samples lost gloss at the end of thermal aging. The highest gloss loss after thermal cycling was obtained in the NH composite group (mean change 11.655). Unlike other composite groups, NH composite resin contains Bis-GMA, Bis-EMA, and TEG-DMA hydrophilic monomers that increase the water absorption capacity of resin molecules. There is also no UDMA monomer, which is less hydrophilic compared to other monomers. Therefore, NH composite may have shown higher water absorption and consequently more gloss loss than other composite resins when exposed to thermal cycling.^{30,31}

The temperature of preheating range for composite resins has been accepted as 54-68°C as it does not cause pulpal irritation.¹ Therefore, in this study, preheating of composite samples was performed at 60°C. Although preheating times have been reported in different studies ranging from 40 seconds to 24 hours, it has been reported that 15 minutes is the most appropriate time in the clinical setting.¹ Therefore, in the present study, preheating was performed for 15 minutes.

In this study; significant differences were found between composite resins with and without preheating treatment in terms of color changes and the second null hypothesis of the study was rejected. However, as a striking result, it was found that the heated groups were significantly less colored than the unheated groups in all composite types. It was thought that this may be due to the improved physical and chemical properties due to the increase in the degree of monomer conversion as a result of preheating.^{2,3} The CIELAB color space is a three-dimensional color measurement system consisting of a lightness scale (L*) and a contrasting color axis for both redness-greenness (a*) and yellowness-blueness (b*).³² The differences between the set of L*a*b* values (ΔL^* , Δa^* , Δb^*) can be used to calculate the color difference (E*). Importantly, relating b findings to clinical significance depends on how the human eye is able to perceive color differences. Paravina et al.⁹ reported a color perceptibility threshold (ΔE_{00}) of 0.8 and an acceptability threshold of 1.8. Perceptibility and acceptability tolerance thresholds need to be discussed to determine which differences in color or surface gloss can significantly affect the aesthetic outcome of a dental restoration.^{9,33} Considering the results of this study, the coloration of all specimens was below the acceptable threshold. This result is thought to be due to the fact that the samples were not exposed to a coloring solution. However, another striking result was obtained when analyzed in terms of the perceptible threshold value. All of the heated resin samples were colored below the detectable threshold value (ΔE_{00} : 0.8), regardless of the composite type, while all of the unheated samples showed coloration above the detectable threshold value. This result is in agreement with previous studies reporting that the degree of polymerization increases the resistance to discoloration.³³⁻³⁵ Sousa et al.³³ put heated composites in cola and fruit juice for 7 days and found that the heated (68°C) groups lost less color. Mundim et al.,³⁶ on the other hand, didn't see a big difference in the color stability of composites heated at 60°C compared to those heated at 8 and 25°C. The results found by different studies may vary depending on the preheating method, temperature, type of solution stored, duration, or aging method.

Within the limitations of the present study, controlling the temperature is a major challenge and a major limitation of this study. Another important limitation of this study is that the effect of mechanical modification of these materials during tooth brushing was not considered. If the surface roughness of the samples had been measured, it would have allowed us to obtain more detailed information on whether the loss of gloss was due to the surface

properties of the samples or to water absorption. The conditions of this study were in vitro. The presence of other aging agents should also be evaluated to further adapt the results to clinical conditions. Despite these limitations, this study proved that preheating has positive effects on the surface coloration of selected composite materials. The effect of preheating on the gloss of the composites was also simultaneously investigated. By selecting composites with different filler types in the study, it was also possible to evaluate materials with different indications. From all these perspectives, this study is one of the rare studies examining the effects of preheating on clinical practice.

CONCLUSION

Although preheating did not have a significant effect on the gloss of the composites, it reduced surface discoloration. Therefore, the inclusion of heating in addition to the techniques applied in the fight against external discoloration of composite resin materials will provide more clinically successful results.

Ethics Committee Approval: Since this study did not involve any living subjects and was conducted entirely in vitro, ethical approval was not required.

Informed Consent: There are no participants in the study.

Peer-review: Externally peer-reviewed.

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