# ORIGINAL ARTICLE / ARAȘTIRMA MAKALESİ

# The Effect of Different Polymerization Procedures on Color Stability of Indirect Resin Restorations

Farklı Polimerizasyon Prosedürlerinin İndirekt Rezin Restorasyonların Renk Kararlılığına Etkisi

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## ABSTRACT

**Objectives:** The aim of this study was to evaluate the effect of different polymerization procedures on color stability of indirect resin restorations.

Materials and Methods: 288 disk-shaped specimens of resin composites (Filtek Z250, Gradia Plus, Ceramage, GrandioSO) were prepared with laboratory and chairside curing device (I.Labolight Duo/II. Elipar S10). Then, baseline color measurements were performed. Specimens of each composite were randomly divided into three subgroups according to staining solution (coffee, black tea and distilled water as control group) (n=12). Color measurements were performed and  $\Delta E$  values were calculated. The data were evaluated by using ANOVA for repeated measures and multiple comparisons were determined by Bonferroni test (p<0.05).

**Results:** Staining increased over time for all groups and coffee showed the highest staining potential (p<0.05). Polymerization procedures also affected color stability (p<0.05).

**Conclusion:** It was concluded that different polymerization procedures and staining solutions might affect color stability of resin composites tested.

**Keywords:** Indirect Resin Composites, Polymerization Procedures, Color Stability, Staining Solutions, CIE Lab system.

#### ÖΖ

Amaç: Bu çalışmanın amacı, indirekt rezin restorasyonların renk stabilitesi üzerinde farklı polimerizasyon prosedürlerinin etkisini değerlendirmektir.

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Gereç ve Yöntem: Kompozit rezinlerden (Filtek Z250, Gradia Plus, Ceramage, GrandioSO) 288 adet disk şeklinde numune laboratuvar ve hasta başı polimerizasyon cihazı (I.Labolight Duo/II. Elipar S10) kullanılarak hazırlandı ve başlangıç renk ölçümleri yapıldı. Kompozit numuneleri, boyama solüsyonuna göre (kahve, siyah çay ve kontrol grubu olarak distile su) rastgele üç alt gruba ayrıldı (n=12). Renk ölçümleri yapıldı ve  $\Delta E$  değerleri hesaplandı. Veriler, tekrarlanan ölçümleri için ANOVA kullanılarak değerlendirildi ve çoklu karşılaştırmalar Bonferroni testi ile belirlendi (p<0.05).

**Bulgular:** Boyama tüm gruplarda zamanla arttı ve kahve en yüksek boyama potansiyelini gösterdi (p<0.05). Polimerizasyon prosedürleri ayrıca renk stabilitesini de etkiledi (p<0.05).

**Sonuç:** Farklı polimerizasyon prosedürlerinin ve boyama solüsyonlarının test edilen kompozit rezinlerin renk stabilitesini etkileyebileceği sonucuna varıldı.

Anahtar Kelimeler: İndirekt Kompozit Rezin, Polimerizasyon Prosedürleri, Renk Kararlılığı, Boyama Solüsyonları, CIE Lab sistemi.

## INTRODUCTION

Performing a perfect restoration with an acceptable survival rate and an esthetic appareance has become the primary goal of today's dental proffesionals. Resin composites are the most commonly used restorative materials in both anterior and posterior region for many reasons. Due to developments in physicomechanical and esthetic properties, their application fields have become widespread from direct to indirect restorations. Indirect techniques are preferred for dental restorations considering some disadvantages of direct techniques, such as difficulties of achievement of an optimal contact point, occlusal anatomy, color stability, complexity of marginal sealing and risk of moisture contamination in long chair time periods (Demarco, et al. 2012; Dourado Loguercio, et al. 2004). Another drawback of resin composites is polymerization shrinkage that generates a stress at the interface between the resin and cavity wall, leading to marginal gap formation

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and postoperative hypersensitivity(Hickel &Manhart 2001). Besides, degree of conversion (DC) has to be taken to the consideration and this could affect physical and biological properties of resin composites in terms of water sorption, hydrophilicity, color stability, wear resistance and surface microhardness (De Munck, et al. 2005). Indirect resin composite restorations are polymerized outside of the mouth in laboratory conditions to achieve optimal monomer conversion.

Direct and indirect resin composites have basically similar formulations and some of them could be used either direct or indirect techniques according to their user manuals. However, polymerization procedures and devices could be effective in their clinical behaviours. Some of them also accommodate a combination of light, heat, vacuum pressure with various polymerization modes. Although, the indirect resin composites could be polymerized with conventional polymerization units, Light Emitting Diode (LED) laboratory polymerization devices were also developed. Manufacturers equipped these devices with polywave (blue/violet) LED light sources and speculated that this technology ensures optimal hardening of resin materials with high power outlet and different curing modes.

In oral environment, color stability of resin composite restorations is affected by intrinsic factors related to resin matrix type, filler size distrubution and percentage and extrinsic factors, such as incomplete polymerization, surface roughness, water sorption, oral hygiene and food colorants (Dietschi, et al. 1994; Patel, et al. 2004). As well as, indirect resin composites gained popularity, researchers focused on effects of conventional and laboratory polymerization devices on color stability, hardness, flexural strength of these restorative materials (Grazioli, et al. 2019; Imai, et al. 2019; Nakazawa 2009). While, laboratory polymerization devices' requires additional cost and they may not always be found in dental offices, conventional devices were the most common used equipments for restorative procedures and it might be advantageous to use one device for two purposes. However, the effect of different post-polymerization techniques on physicomechanical properties of indirect resin restorations is still a controversial issue.

The aim of the present study was to evaluate the color stability of resin composites polymerized with a chair-side and a laboratory LED curing devices.

Null hypothesis tested were:

- Polymerization procedure did not affect the color stability of indirect resin restorations.
- Different immersion solutions did not affect the color stability.

RESIN COMPOSITES	MANUFACTURER	MATRIX COMPOSITION (Filler %by Weight)	LOT NUMBER			
Filtek Z250	3M ESPE, St.Paul, IL, USA	BIS-GMA, UDMA and BIS-EMA resins, Zirconium Silicate, Inorganic filler (76 wt%)	NA14156			
Gradia Plus	GC, Tokyo, JAPAN	UDMA, Dimethacrylate, Inorganic fillers (71 wt%), Prepolymerized fillers (6 wt%), Photoinitiators, Stabilisers, Pigments	1901151			
Ceramage	Shofu, Kyoto, JAPAN	UDMA, UDA, Zirconium Silicate, Pigments and Inorganic filler (73 wt%), Photoinitiators, Stabilisers	121828			
GrandioSO	Voco, Cuxhaven, GERMANY	BIS-GMA, BIS-EMA, TEGDMA, Glass ceramic and Silicone dioxide - nanoparticles, Inorganic fillers (89 wt%)	1921607			

Table 1: The resin composites, manufacturers, material composition and lot numbers used in the study.

Table 2: Polymerization units, manufacturers and details of polymerization procedures.

POLYMERIZATION	MANUFACTURER	LIGHT SOURCE, WAVELENGTH AND EXPOSURE	SERIAL NUMBER
DEVICE		DURATION	
		1200mW/cm2,	
Elipar S10	3M ESPE,	430-480nm,	939.123.015874
	St. Paul, MN, USA	5, 10, 15, 20 seconds, continuous mode (120 sec) and tack-cure	
		mode.	
		12 Blue LED's: 465 – 485 nm,	
Labolight Duo	GC	3 Violet LED's:390-400 nm	00922
	Tokyo, JAPAN	Step mode 10 sec	
		Full mode 1, 1.5, 3, 5 minute.	

# **MATERIALS AND METHODS**

The resin composites, manufacturers, material composition and lot numbers were shown in Table 1. Curing units, manufacturers and details of polymerization procedures were also presented in Table 2.

#### **Specimen Preparation**

A total of 288 disk shaped specimens from four resin composites [(Filtek Z250, NA14156, 3M ESPE, St.Paul, IL, USA), (Gradia Plus, 1901151, GC Tokyo, Japan), (Ceramage, Shofu, 121828, Kyoto, Japan), (GrandioSO, Voco, 1921607, Cuxhaven, Germany)] of A2 shade using a teflon ring mold (2 mm-thickness, 10 mm-diameter) were prepared for color stability evaluation of this study (n=12).

In each resin composite group, half of the specimens was polymerized by a chair-side cordless LED light curing unit (Elipar S10, 939.123.015874, 3M ESPE, USA). The resin composite material was placed in a plastic mold (2 mm-thickness, 10 mm-diameter) and covered with a Mylar strip and a glass slide with 1 mm thickness. The specimens were gently finger pressed to remove excess material and polymerization was performed for 20 s with Elipar S10. Then, Mylar strip and glass slide were removed and specimens were subjected to curing for 3 minutes with the same device to perform complete polymerization of resin composite. The other half was polymerized with a laboratory curing unit (Labolight Duo, 00922, GC, Tokyo, Japan). The resin composite material was placed in same manner and polymerization was performed for 10 s with initial mode and 3 minutes with full mode of Labolight Duo.

After polymerization, specimens were removed from the mold and stored in distilled water at 370 C for 24 h. Then, finishing and polishing procedures were done with aluminium oxide disc system (Supersnap, Shofu, Kyoto, Japan). Specimen preparation was performed by one operator of the research team.

### **Color Measurements**

Before the baseline color measurement, the specimens were rinsed for 10s with distilled water and dried with absorbent paper. The measurements were performed using a spectrophotometer (Vita Easy Shade Compact, VITA Zahnfabrik, Bad Säckingen, Germany) using the CIE L,a,b system. Mean color measurements were performed using a white background (Ardu, et al. 2010; Arocha, et al. 2013). The specimens of each resin composite group were divided into 3 subgroups (n=12) according to staining solutions (coffe, tea and distilled water as control). The tea solution(Yellow Label, Lipton, Rize Turkey) was prepared by immersing two prefabricated tea bags (2x2g) into 250 ml of boiling water for 1-2 minutes. The coffe solution was prepaired with 5g of instant coffee (Nescafe Classic, Nestle), poured into 250 ml boiling water. Staining solutions were kept at 370C and freshly prepared everyday to avoid any contamination (Ardu, et al. 2010; Arocha, et al. 2014; Rahim, et al. 2012).

Color measurements were then made according to same procedure mentioned above after 1 day, 7, 14, 28 days immersion periods.

Color difference  $(\Delta E^*_{ab})$  of the specimens was calculated according to following formula:

 $\Delta E^{*}_{ab} = [(\Delta L^{*})^{2} + (\Delta a^{*})^{2} + (\Delta b^{*})^{2}]^{\frac{1}{2}}$ 

(L\* Lightness, a\* red-green, b\* yellow-blue axes of Comission Internacionale de l'Eclairaga L\*a\*b\* system)

 $\Delta E$  values  $\geq$ 3.3. were considered clinically unacceptable (Malmstrom, et al. 2002).

## **Statistical Analysis**

The data were analyzed with Statistical Package for Social Sciences 18.0 (SPSS, Chicago, IL, USA) using ANOVA for repeated measures. Multiple comparisons were determined by Bonferroni test for color stability at a significance level of 0.05. The minimum number of samples required for the study was calculated using G\*Power v.3.1 software (Heinrich Heine, University of Dusseldorf, Düsseldorf, Germany). 0.05 alpha (type I error), effect size 0.25 and beta power 0.85" (1 – type II error) were determined and the minimum estimated number of samples for each group was found to be 33.

## RESULTS

The mean and standard deviations of  $\Delta E$  values were shown in Table 3.

Statistically significant differences were found among the groups tested according to polymerization procedures, except for distilled water groups (p<0.05). Filtek Z250 groups polymerized with Labolight Duo showed clinically unacceptable color changes and demonstrated statistically significant differences compared to specimens polymerized

	LABOLIGHT DUO						ELIPAR S10						
RESIN	STAINING SOLUTIONS						STAINING SOLUTIONS						
COMPOSITES	Coffee		BlackTea		Distilled Water		Coffee		BlackTea		Distilled Water		
	Mean		SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Z250	$\Delta E1$	2,35	1,54	1,02	0,85	1,37	1,99	1,55	1,14	0,58	0,60	0,80	1,03
	$\Delta E_7$	5,78	2,39	1,96	2,46	0,03	1,27	3,33	1,76	0,93	0,79	1,05	0,81
EK	$\Delta E_{14}$	7,83	3,08	1,96	1,57	0,24	1,82	4,18	1,59	1,11	0,70	0,70	1,05
FILT	$\Delta E_21$	8,43	1,35	4,34	3,05	0,31	1,42	4,41	2,08	1,75	0,71	0,51	0,86
	ΔE _28	9,68	2,18	4,69	1,95	0,81	1,92	5,00	2,28	1,59	0,81	1,30	1,20
GRADIA PLUS	$\Delta E_1$	1,47	0,62	0,77	0,88	0,39	2,03	1,31	0,81	0,22	0,93	0,30	0,69
	$\Delta E_7$	2,98	1,57	2,06	0,99	0,48	2,76	2,19	1,89	1,20	0,66	0,51	0,88
	$\Delta E_{14}$	5,68	1,75	1,85	1,49	0,75	1,93	2,45	1,44	0,73	0,70	0,37	2,30
	ΔE _21	6,61	2,87	2,57	1,19	0,24	2,87	2,30	1,31	1,75	0,70	0,88	1,51
	ΔE_28	8,27	3,16	3,92	2,24	0,71	2,59	4,02	4,81	2,83	2,21	0,91	2,38
CERAMAGE	$\Delta E_1$	1,27	2,15	0,16	1,19	0,58	0,72	0,13	1,37	0,06	0,62	0,50	0,72
	$\Delta E_7$	5,43	0,86	1,15	1,05	0,11	1,01	4,11	4,91	0,70	0,91	0,13	0,94
	$\Delta E_{14}$	7,92	1,83	1,06	1,13	0,24	1,10	4,84	5,13	1,14	2,72	0,20	0,65
	ΔE _21	9,54	1,76	1,75	1,31	0,07	0,49	6,24	10,29	1,42	1,38	0,01	0,73
	ΔE _28	10,05	2,48	3,65	1,17	0,01	0,90	7,07	7,32	3,50	3,20	0,62	0,63
GRANDIOSO	$\Delta E_1$	1,65	1,51	2,36	1,23	0,45	2,45	1,21	1,10	1,44	1,00	1,28	1,60
	$\Delta E_7$	3,90	1,70	0,81	1,44	0,27	1,28	2,14	1,63	1,83	1,35	1,12	1,18
	$\Delta E_{14}$	5,41	2,58	1,22	1,03	0,60	1,93	2,97	2,33	1,56	1,28	1,62	1,78
	ΔE _21	7,27	5,16	2,44	1,19	0,38	1,76	3,10	2,11	2,24	1,32	0,97	2,00
	ΔE _28	7,46	2,28	2,90	2,93	0,93	1,67	3,77	2,29	4,30	2,69	1,30	1,84

**Table 4:** Pairwise comparison of  $\Delta E$  values according to polymerization methods.

RESIN COMPOSITE	STAINING SOLUTIONS	POLYMERIZATION PROCEDURESLABOLIGHT DUO VS ELIPAR S10 P values						
		ΔE_1	ΔΕ_7	ΔE_14	ΔE _21	ΔE _28		
FILTEK Z250	Coffee	-	-	0.024	0.000	0.001		
	Tea	-	0.011	-	0.000	0.000		
	Distilled water	-	-	-	-	-		
GRADIA PLUS	Coffee	-	-	0.021	0.000	0.000		
	Теа	-	-	0.019	-	-		
	Distilled water	-	-	-	-	-		
CERAMAGE	Coffee	0.009	-	0.013	0.000	0.004		
	Теа	0.030	-	-	-	-		
	Distilled water	-	-	-	-	-		
GRANDIO SO	Coffee	0.013	0.029	0.009	0.000	0.003		
	Tea	-	-	-	-	-		
	Distilled water	-	-	-	-	-		

\* (-) = no statistically significant difference[p>0.05]

with Elipar S10 from 21 days immersion periods in tea solution (p<0.05). There were statistically significant differences in all groups immersed in coffee solutions due to polymerization procedures (p<0.05). It was also demonstrated that Gradia Plus and Grandio SO groups polymerized with Elipar S10

showed lower and acceptable DE values until the 28 day immersion period in coffee (Table 4).

The effects of staining progressively increased over time for all of the composites and polymerization procedures tested except for distilled water (p<0.05). Coffee solution showed the highest staining potential at any immersion period, followed by tea and distilled water and there were statistically significant differences between the subgroups (p<0.05). Multiple comparison between the resin composite groups also demonstrated statistically significant differences for both Labolight Duo and Elipar S10 groups related to the immersion solutions (p<0.05). Clinically unacceptable color differences were observed in all restorative material groups from 7 day immersion in coffee and 21 day immersion in tea ( $\Delta E \ge 3.3$ ).

# DISCUSSION

The present study evaluated the color stability of four different indirect resin composites polymerized with a chairside and a laboratory curing devices. Our results showed that color stability of resin restorative materials was affected by polymerization procedure and first hyptohesis was rejected. It was also revealed an undeniable effect of immersion solution and time on color stability of resin composites and second hypothesis was rejected.

Color change of dental restorations over time is a situation that can not be avoided and has a failure risk of an esthetic restoration. It can occur related to various factors, such as dental plaque accumulation, consumption of staining beverages, imcomplete polymerization of resin composites, water sorbtion, surface roughness, etc. (Ardu, et al. 2010; Lee, et al. 2011; Nakazawa 2009; Nasim, et al. 2010). Coffee and tea are frequently consumed beverages in worldwide and held responsible for discoloration of natural tooth and restoration surfaces. In this study, color stability was evaluated in coffee and tea, known to have potential to stain restorative materials and has already been used in a large number of studies (Domingos, et al. 2011; Falkensammer, et al. 2013; Nasim, et al. 2010; Tuncer, et al. 2013). Distilled water was used as a control solution. In previous studies, coffee found to be have much more staining potential compared to tea and our results supported these findings (Ardu, et al. 2010; Arocha, et al. 2014; Ertas, et al. 2006; Kentrou, et al. 2014). In the present study, there were significant differences in coffee groups of all resin materials, regardless of polymerization procedure and color change values were clinically unacceptable from even 7 days immersion time. Coffee contains yellow stain molecules with low polarity due to their affinity to the resin polymer networkexplained staining mechanism in two ways; absorbtion of coffee colorants and ionic interaction with amine groups in resin materials (Arocha, et al. 2014; Kissa 1995). Black tea has also a staining potential and it

might be attributed to the presence of tannins that enhances the adherence of color pigments (Quek, et al. 2018). Previous studies evaluated the color stability of indirect resin composites, demonstrated increased  $\Delta E^*_{ab}$  values in coffee more than tea solution and attributed their findings to amount and strength of color molecules in staining solution (Ardu, et al. 2010; Kentrou, et al. 2014)

Polymerization is an important phenomenon to achieve optimal mechanical and optical properties in resin composites (Yazici, et al. 2007). In the present study, polymerization procedures affected color stability in varing levels for all material groups immersed in tea and coffee. However, in distilled water groups, minimal color change occurred compared to baseline and there was no significant difference between chair-side and laboratory polymerization devices. In previous studies, discoloration was associated to polymerization factors, including photoinitiators, curing units and light application time (Asmussen 1983; Correr, et al. 2005; Kakaboura, et al. 2003). Elipar S10 has high-power LED technology to provide 1200mW/cm2 intensity between 430 and 480 nm utilizable wave range and matches the absorbtion spectrum of the most commonly used initiator, camphorquinone. Labolight Duo contains 380-510 nm blue and violet LEDs with a wide spectrum and manufacturer claims that this technology ensures optimal hardening of all light cured dental materials. Blue light activates photoinitiators (mainly CQ) with maximum absorbance of light close to 468 nm (Cramer, et al. 2011; Jandt & Mills 2013). This wide range could negatively affect photoinitiator activation due to different positioning of light emitters along the same light curing unit tip causing inhomogeneous light output (Price, et al. 2010; Price, et al. 2014). However, laboratory light curing units have to polymerize various dental materials not only resin composites and polywave LED units might be preferred to activate more photoinitiators (Magalhaes Filho, et al. 2016). Karaarslan et al. evaluated that effect of alternative polymerization techniques on color change of a resin composite and found no significant difference among the devices in terms of  $\Delta E$  values (Karaarslan, et al. 2013). They also indicated high Db values resulted in less yellow color in specimens polymerized with inlay oven, combination of light and heat under water.

Some studies indicated that the filler particle distrubution should be uniform in resin matrix and probability of the formation of filler rich or depleted areas may increase the water sorption of resin materials (Skrtic, et al. 2004). Resin matrix composition has also an important effect on water sorption and it was reported that UDMA resin monomer demonstrated lower water sorption than other dimethacrylates and resulted in a better color stability (Khokhar, et al. 1991; Ruyter, et al. 1987). In the present study, GC Gradia Plus and Ceramage were Bis-GMA free materials and contained UDMA and our results did not support the idea of UDMA was responsible for staining as a previous one (Kentrou, et al. 2014).

There were some limitations with the present study. First, only two polymerization devices were evaluated and other techniques, such as heat and vacuum pressure systems have to be studied to decide which post-polymerization technique may improve color stability of indirect restorations. Second, resin composites for indirect restorations were manufactured with a variety of enamel and body shades and due to absence of body shades A2 shade was selected for Filtek Z250 and GrandioSO. It could affect the color stability parameters and in further studies, enamel/body shades of indirect resin composites would be investigated.

## CONCLUSION

Within the limitations of this study, it can be concluded that;

- Chairside LED curing device showed good performance as laboratory LED curing device for post-polymerization of indirect restorations in equaul exposure time in terms of color stability.
- Type of staining solution had a significant effect on color stability of indirect resin composites tested and coffee was found to be the most effective solution.

## **Declaration of Interest Statement**

The authors would like to thank GC Company and Shofu Dental for providing the materials for this research. The authors do not have any financial interest in the companies whose materials are included in this article.

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