Comparative Evaluation of Surface Hardness and Color Stability of Dental Composites with Different Photoinitiators

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Abstract

Aim This study aims to comparatively investigate the color stability and microhardness values of dental composites with different photoinitiators.

Material and method In the study, Ivoclar Tetric N-Ceram, Dentac Parion, and FGM Llis composites were used. A total of 30 composite discs were evenly divided for surface microhardness and ΔE measurements. Each subgroup contained 5 composite discs (Diameter: 5 mm, Thickness: 2 mm). Before the experimental procedures, all composite discs were aged in a coffee solution for one week. Surface microhardness was measured using the Vickers Hardness Test device, while color stability was assessed with a spectrophotometer. One-way ANOVA and Two-Way Repeated Measures ANOVA with Bonferroni-adjusted pairwise comparisons were applied.

Results No statistically significant difference was observed among the three dental composite groups in terms of color stability (p > 0.05). However, their color stability performance ranked from highest to lowest as follows: Llis (3.13 ± 0.42) > Parion (2.76 ± 0.57) > Tetric N-Ceram (2.70 ± 0.76). In contrast, a statistically significant difference was detected among the groups regarding surface microhardness (p < 0.05). Llis exhibited significantly higher values compared to Tetric N-Ceram and Parion (p < 0.05).

Conclusion The compositional structure of the composite material and the photoinitiator mechanism used are directly related to the formation of its mechanical properties.

Keywords Camphorquinone, Color stability, Dental composite, Microhardness, Photoinitiator

Introduction

Advancements in dental composites have reached an unprecedented level, with modifications in both the inorganic and organic components yielding more aesthetic and functional restorative materials (1).

Technological innovations have facilitated the transition from chemically polymerized composites to light-cured materials, significantly enhancing color stability and other mechanical properties of composite restorations (2). This pivotal shift in composite polymerization has been largely driven by the incorporation of photoinitiators such as camphorquinone, which play a fundamental role in initiating the polymerization cascade through the generation of free radicals upon light activation (3,4). Among these, camphorquinone remains the most widely employed photoinitiator (5).

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This study was granted funding under the TÜBİTAK–2209-A Research Projects Support Program for University Students as part of the 2023 second-term application cycle. Camphorquinone exhibits an absorption spectrum within the blue region of visible light, with a wavelength range spanning 360–510 nm (4,5). However, its most significant drawback lies in its intrinsic yellow coloration. The increased weight percentage of camphorquinone within the composite matrix results in a pronounced yellowish hue and reduced luminosity, which can, in turn, influence color stability, mechanical performance, and the degree of conversion (6). Resin-based composites may also incorporate various alternative photoinitiators with distinct chemical structures and formulations, such as benzophenone (7), 1-phenyl-1,2-propanedione (PPD) (8), dibenzoyl germanium (Ivocerin) (9), diphenyl(2,4,6-trimethylbenzoyl)-phosphine oxide (TPO) (10), and Lucirin TPO (11) each offering potential advantages in mitigating the limitations associated with camphorquinone.

In this context, our study aims to comparatively evaluate the microhardness values and color stability of three distinct dental composite materials, each containing a different photoinitiator, following a one-week aging process in a coffee solution. The null hypothesis of this study is stated as follows: There is no statistically significant difference among the three composite materials in terms of color stability and microhardness values.

Material and Methods

Design of study

This study was conducted at the Hard Tissue Laboratory of the Altınbaş University Faculty of Dentistry Dental Hospital.

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Furthermore, ethical approval is not required for this study, as no live animal or human-derived products were used in its execution. It incorporated composite resins with distinct photoinitiator systems, specifically Tetric N-Ceram (Ivoclar; Schaan, Liechtenstein), FGM Llis (FGM Group; Brazil), and Parion (Dentac; Turkey) (Table 1). Prior to initiating the study, a power analysis was performed using the G*Power statistical software package (G*Power Ver. 3.0.10, Germany). Based on the parameters established by Yildirim Ucuncu et al. (12), the power analysis was conducted with a Type I error probability of 0.05 (95% confidence interval), a test power of 0.8 (Type II error = 0.20), and an effect size of 1.73. Under these conditions, the required sample size was determined to be five specimens per subgroup (n = 5). The study was conducted in two separate phases.

Measurement of color stability

Baseline and post-staining color measurements were conducted under standardized conditions, at the same time of day, using a spectrophotometer (Vita Easy Shade Compact, VITA Zahnfabrik, Bad Säckingen, Germany). Prior to each measurement, the device was calibrated according to the manufacturer's instructions using the built-in ceramic reference block. Specimen preparation was carried out using Teflon molds, each with a diameter of 5 mm and a thickness of 2 mm, to ensure uniformity. Five composite resin discs were fabricated for each subgroup (n=5; per group), and this phase of the study was conducted with a total of 15 composite discs. A transparent celluloid strip was first placed on a microscope slide, followed by stabilization of the Teflon mold. Composite resin was then carefully introduced into the mold using a plastic spatula, ensuring the absence of air bubbles. Subsequently, another transparent celluloid strip and a microscope slide were placed atop the mold. Gentle finger pressure was applied to eliminate excess material and ensure a smooth, uniform surface. Following this, only the microscope slide was removed, and polymerization was carried out directly through the transparent celluloid strip, adhering to the designated polymerization protocols (ValoTM Cordless, Ultradent, Cologne, Germany). This approach effectively prevented the formation of an oxygen inhibition layer, ensuring optimal polymerization quality. The light-exposed upper surfaces of the specimens were carefully marked and removed from the molds without deformation. Each specimen was then placed in a tube containing distilled water and stored in a dark incubator at 37°C for 24 hours to ensure proper hydration and stabilization. For baseline color measurements, the specimens were removed from the distilled water, gently dried, and analyzed using the CIELAB color system. Measurements were performed three times for each specimen, precisely at the center of the resin surface. The arithmetic mean of these three measurements was calculated to obtain the average L, a, and b values. To simulate staining and aging, a coffee solution was prepared by dissolving 2 g of coffee in 200 mL of boiling water, followed by filtration through a filter paper to remove residues. The specimens were then immersed in 5 mL of the prepared coffee solution within separate tubes and stored in a 37°C incubator for 24 hours (13). At the end of the immersion period, the specimens were removed from the coffee solution, gently dried, and subjected to post-staining color measurements, following the same protocol as the baseline assessments.

Table 1: The compositional information and lot numbers of the dental composites

Brand of dental com- posites	Origin	Contain	Photoiniti- ator	Recom- mend Polym- erization Technique	Lot Number
Tetric N-Ce- ram	Ivoclar Group, Schaan, Liechten- stein	UDMA, Bis-EMA, Bis-GMA, copoly- mer, Si-Zr mixed oxide, ytterbium trifluoride, inor- ganic fillers: 54-56 vol% particle size: 0.11 μm – 15,5 μm	Dibenzoil germanium	500-900 mW/cm2 : 20 s 1000-1300 mW/cm2 : 10 s 1800-2200 mW/cm2 : 5 s	Z04ZB2
Parion	Dentac, T-Resto, Türkiye	Bis-GMA, Bis- EMA, UDMA, TEGDMA inor- ganic filler, silica, quartz Inorganic fillers: 77-78 w% & 66 v%	Camphorqui- none	≥800 mW/ cm2 20 s	PN220122
Llis	FGM Den- tal Group, Brasil	Bis-GMA, TEGD- MA, Bis-EMA, UDMA camphorquinone, co-initiators, Silane, Barium- Aluminum Silicate Glass, Silicon Dioxide Inorganic fillers: 56-59 v% & 77.5 -78.5 w% Particle size: 40 nm -0.7 μm (average size: 0.7 μm)	Cam- phorquinone + co-initiator (tertiary amine)	1200 mW/ cm2: 20 s (trans- lucent & enamel) – 40 s (dentin & body) (2.7 - 3.0 mm) 700 mW/ cm2 : 20 s (trans- lucent & enamel) – 40 s (dentin & body) (2.2 - 2.9 mm) 500 mW/ cm2: 20 s (trans- lucent & enamel) – 40 s (dentin & body) (1.9 - 2.5 mm)	310823

The changes in color coordinates were determined using the following equations:

 $\Delta L^* = L2 - L1, \Delta a^* = a2 - a1, \Delta b^* = b2 - b1$

The total color change $(\Delta E)^*$ was calculated using the formula:

$$\Delta E_{ab}^* = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$$

Measurement of microhardness

The Vickers hardness test was employed to assess the surface hardness of both the upper and lower surfaces of the specimens and to determine their hardness ratio. Specimen preparation followed the same protocol described in Section 2.2. Measurement of Color Stability, utilizing Teflon molds with a diameter of 5 mm and a thickness of 2 mm to ensure uniformity. Five composite resin discs were fabricated for each subgroup (n=5; per group), and this phase of the study was conducted with a total of 15 composite discs. The light-exposed upper surfaces of the specimens were carefully marked and removed from the molds without deformation. Subsequently, the specimens were stored in tubes containing distilled water and incubated in a dark environment at 37°C for 24 hours. Following the incubation period, hardness measurements were performed using a Vickers hardness testing device (Shimadzu HMV-2, Japan). This device features a touchscreen panel, allowing precise adjustment of test load, test duration, and other parameters. The test load is adjustable between 98.07 mN and 19.914 N, while the test duration ranges from 5 to 999 seconds. Additionally, the device is equipped with two ocular lenses providing 10x and 40x magnification. A 50 g load was applied for 30 seconds to the upper surfaces of the specimens, creating a diamond-shaped indentation. The projection corners of this indentation were marked under 40x magnification for precise measurement. Three hardness measurements were obtained from distinct locations on the upper surface of each specimen, ensuring a minimum spacing of 100 µm from the specimen's edges and between measurement points. The arithmetic mean of these three measurements was recorded as the Vickers Hardness Number (VHN) for the upper surface.

Statistical analysis

All statistical analyses were conducted using GraphPad Prism software (GraphPad Software, Inc., California, USA). Descriptive statistical methods, including mean, standard deviation, and median, were employed to summarize the study data. The normality of quantitative variables was assessed using the Shapiro-Wilk test alongside graphical inspections. For comparisons involving three or more normally distributed groups, a One-Way ANOVA was performed. In cases where measurements were taken at different time points on the same material with two independent variables, a Two-Way Repeated Measures ANOVA was utilized. To control for multiple comparisons, Bonferroni-adjusted pairwise comparisons were applied, ensuring the identification of statistically significant differences between groups. A significance threshold of p < 0.05 was considered indicative of statistical relevance.

Results

The mean microhardness values are presented in Table 2. Microhardness test results were analyzed using a Two-Way Repeated Measures ANOVA, revealing a statistically significant difference among composites aged in the coffee solution (p < 0.05). To determine the source of this variation, a Bonferroni-adjusted multiple comparison test was conducted. No statistically significant difference was observed between the microhardness values of N-Ceram and Parion dental composites (p > 0.05). However, Llis dental composite exhibited significantly higher microhardness values of

ues than both counterparts (p < 0.05) (Table 3). Post-immersion color stability, assessed through ΔE values, was analyzed via One-Way ANOVA. No statistically significant difference was detected among the dental composites in terms of color stability (F = 0.7476; p > 0.05) (Table 4). Notably, ΔE values for all three composites remained below the clinically perceptible threshold of 3.3, ranking from worst to best as Llis > Parion > Tetric N-Ceram.

Table 2: The microhardness values of the dental composites

	Microhardness (VHN)		
	Before aging	After aging	
Tetric N-Ceram	35.18 ± 3.59	35.82 ± 3.95	
Dentac Parion	36.84 ± 5.07	34.26 ± 4.26	
Llis	45.01 ± 11.21	41.86 ± 10.57	

Table 3: Post-hoc analysis multiple comparisons

Microhardness	Mean Differences	95,00% CI of dif- ferences	Adjusted P Value
N- Ceram vs. Par- ion	1.450	-0.3570 to 3.257	>0.9999
N- Ceram vs. Llis	-6.457	-11.97 to -0.9395	0.0016*
Parion vs. Llis	-7.907	-14.06 to -1.755	<0.0001*

Two-Way Repeated Measures ANOVA -Pairwise comparisons with Bonferroni correction $*p{<}0.05$

Table 4: The comparison of ΔE values

	ΔΕ	р	
Tetric N-Ceram	2.70 ± 0.76		
Dentac Parion	2.76 ± 0.57	>0.05	
Llis	3.13 ± 0.42		

One-Way ANOVA

Discussion

Based on the findings, the null hypothesis of this study was partially rejected. While no statistically significant difference was observed among the composites in terms of color stability, a significant difference was detected in microhardness values. It is well established that various systems, such as CIELAB, CIE-DE2000, and CMC, are utilized in devices designed to quantify color data and calculate ΔE values (14,15). Among these, CIELAB is recommended for dental applications, as it characterizes color based on human perception and provides a suitable framework for detecting subtle color differences (16). Spectrophotometers, developed to facilitate rapid color analysis while ensuring adequate color information, direct light onto the sample surface and provide readings in CIE L* a* b* units. In this study, the VITA Easy Shade spectrophotometer-a widely employed device in the literature—was used for this purpose (13,17,18). Extensive research has been conducted to determine the acceptability and perceptibility thresholds of different color systems (14,19). Paravina et al. (19) defined the 50:50% perceptibility threshold for CIELAB as 1.2 and the acceptability threshold as 2.7, while Ruyter et al. identified the threshold for an unacceptable ΔE value at approximately 3.3 (20). Additionally, studies have proposed higher thresholds, such as 4.2 (21) and 5.5 (22). In light of this information, composite materials with ΔE values below 3.3 were considered clinically acceptable in this study, and no statistically significant differences were found among the tested composites.

Hardness testing methods, including Brinell, Knoop, and Vickers, employ distinct techniques and application protocols (23). Among these, the Vickers test is widely used in dentistry due to its applicability across a broad range of materials. As a non-destructive method, it determines microhardness by automatically measuring the diagonal length of the quadrilateral indentation created by a diamond indenter (12,23,24). The literature indicates that various loading weights have been used in conjunction with different holding times (12,24-26), and an ISO standard has been established for this purpose (27). Based on the microhardness test results, the highest values-both before and after a one-week aging period in a coffee solution-were observed in the Llis group. Statistically significant differences were found between Llis and both Tetric N-Ceram and Parion. However, no significant difference was detected between Tetric N-Ceram and Parion, with their values exhibiting close similarity. The Llis composite, when immersed for varying durations in acidic solutions such as ferrous sulfate (pH: 4.5) and paracetamol (pH: 3.6), exhibited lower microhardness values in ferrous sulfate compared to paracetamol (26). Acidic solutions may lead to surface degradation by inducing matrix breakdown and dissolution within the composite structure, thereby compromising key properties such as surface roughness and microhardness (26,28). According to the literature, the pH of coffee is reported to range between 4.85 and 5.13. However, the absence of direct pH measurement using an indicator in our study represents a limitation. Consequently, restorative materials subjected to prolonged exposure in coffee solutions may be expected to exhibit reduced microhardness values and diminished color stability over time (29).

The inorganic filler composition of Llis comprises smaller particles compared to Tetric N-Ceram and Parion, while its volumetric filler content is notably higher. The type and content of inorganic fillers incorporated into the chemical composition of resin-matrix composites exert a direct influence on their polymerization mechanism (30). Increasing the filler content has been shown to enhance VHN (12,31), whereas the degree of conversion may remain unaffected, potentially leading to reduced polymerization shrinkage (32). This fundamental principle accounts for the superior hardness values observed in the Llis composite compared to the others. To address the inherent limitations of camphorquinone-based or camphorquinone-amine-based dental composites-such as their yellowish hue, which compromises esthetics, and their rapid photopolymerization under ambient light-alternative photoinitiators, including phenylbis(2,4,6-trimethylbenzoyl)phosphine oxide (BAPO), dibenzoyl germanium (Ivocerin), and diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide (TPO), have been introduced and integrated into composite formulations (3,9,33). Notably, TPO-containing composites have been demonstrated to offer superior color stability compared to conventional camphorquinone-based resin composites (11,33). Ivocerin, a germanium-based photoinitiator with broad-spectrum short-wavelength absorption, has been developed to enhance polymerization efficiency. Materials incorporating this photoinitiator have been reported to achieve significantly higher reactivity and polymerization efficiency than those formulated with camphorquinone or TPO (3,9). Consequently, composites containing such advanced photoinitiators are expected to exhibit superior color stability, a finding that aligns well with our study's results and existing literature.

The limitations of this study include the absence of an investigation into the mechanical properties—particularly the degree of conversion—of the composites in aqueous environments other than coffee, such as artificial saliva or effervescent tablets. Future studies may consider these factors and employ extended aging protocols. Additionally, the precise quantity of photoinitiators within the composite formulations is not explicitly disclosed on product labels. To address this limitation, high-sensitivity chromatographic or spectroscopic analyses could be conducted to accurately determine photoinitiator concentrations.

Conclusion

The compositional structure of resin-based composites, particularly the quantity and size of inorganic filler particles, directly influences their mechanical properties, such as microhardness. A dental composite incorporating an Ivocerin-based photoinitiator may yield superior outcomes in terms of color stability compared to other formulations.

Declarations

Ethics Committee Approval: Since this study did not involve the use of any live animals or human-derived materials, ethical approval was not required.

Informed Consent: Since no human-derived materials were used in this study, informed consent was not required.

Peer Review: Externally peer-reviewed.

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Conflict of Interest: Authors declared no conflict of interest.

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