Original Article

HEALTH SCIENCES MEDICINE

DOI: 10.32322/jhsm.1532620

J Health Sci Med. 2024;7(5):571-580

Evaluating the clinical significance of color stability in nanohybrid composite resins: a comparative study of local and international brands

Hatice Tepe, Özge Çeliksöz, Batu Can Yaman

Department of Restorative Dentistry, Faculty of Dentistry, Eskisehir Osmangazi University, Eskişehir, Turkiye

Cite this article as: Tepe H, Çeliksöz Ö, Yaman BC. Evaluating the clinical significance of color stability in nanohybrid composite resins: a comparative study of local and international brands. *J Health Sci Med.* 2024;7(5):571-580.

Received: 13.08.2024 • **Accepted**: 18.09.2024 • **Published**: 27.09.2024

ABSTRACT

Aims: The objective of this study was to evaluate the color stability of five nanohybrid composite resins, including locally produced Turkish brands and an international brand, after exposure to staining and brushing simulations over time.

Methods: Five nanohybrid composite resins; Filtek Z550 (FLT), Dolgunn (DLG), RubyComp Nano (CMP), Nova Compo C (NVC), and Parion (PRN) were tested. Specimens underwent staining and brushing simulations designed to replicate clinical oral conditions. The staining process involved immersing the specimens in a coffee solution for 8 hours per day over a 10-day period to simulate short-term staining (t1), and for 12 days to simulate long-term staining equivalent to 1 year (t2). Brushing cycles were used to replicate the mechanical wear caused by daily oral hygiene practices. Color measurements were recorded at baseline (t0), after 10 days of staining (t1), and after 1 year of simulated staining and brushing (t2). Additional analyses were conducted using Energy-dispersive X-ray spectroscopy (EDX) and Scanning Electron Microscopy (SEM) to investigate structural and surface characteristics. Statistical analysis was performed using generalized linear models and two-way robust ANOVA to assess the significance of differences in color stability, with a significance level set at $p<0.05$.

Results: The main effect of the composite was not found to be statistically significant on the median ΔE values ($p=0.078$). The main effect of time was found to be statistically significant on the median ΔE values ($p<0.001$). Additionally, the interaction between composite and time was statistically significant on the median ΔE values ($p=0.001$). The highest median value of 3.12 was observed in the NVC composite from ΔE2, while the lowest median value of 1.285 was observed in the PRN composite from ΔE1. EDX and SEM analyses provided insights into the material compositions and their influence on color stability.

Conclusion: The findings suggest that locally produced Turkish nanohybrid composites offer competitive color stability, making them viable alternatives to international brands for aesthetic dental restorations. This study highlights the need to consider both local and international materials for their clinical performance and cost-effectiveness in restorative dentistry. Future research should explore the long-term performance and clinical implications of these materials further.

Keywords: Nanohybrid composite resin, color stability, staining simulation, brushing simulation, spectrophotometer

INTRODUCTION

Nanohybrid composite resins are among the most commonly used materials in restorative dentistry due to their combined aesthetic and mechanical advantages. These materials not only meet high aesthetic expectations, such as natural translucency and shade matching, but also provide durability and versatility in various clinical situations. Thanks to their small filler particle size and homogeneous distribution, nanohybrid composites are suitable for use in both anterior and posterior restorations, offering enhanced polishability and wear resistance.[1]

In restorative dentistry, color stability is a critical factor for the long-term success of aesthetic restorations. Changes in the color of dental materials over time can significantly impact the aesthetic appearance of restorations, leading to dissatisfaction among patients and potentially necessitating retreatment.[2] Composite resins are particularly vulnerable to staining from daily consumption of food and beverages such as coffee, tea, and wine.[3] Therefore, it is essential to evaluate the ability of these materials to maintain their color stability in clinical settings to ensure patient satisfaction and the longevity of restorations.

In this study, coffee was chosen as the staining agent due to its widespread consumption and its high staining potential. Coffee contains tannins, which are known to cause significant extrinsic staining of dental materials.[2-4] The use of a staining agent that mimics real-life dietary habits allows for a more

Corresponding Author: Hatice Tepe, htepe@ogu.edu.tr

accurate assessment of the color stability of composite resins under simulated oral conditions. Color measurements were taken at baseline (t0), after 10 days (t1), and after 1 year (t2) of simulated brushing and staining, representing immediate, short-term, and long-term color changes in the materials. This timeline was designed to capture how composite resins react to staining and brushing in both early and prolonged exposures, providing comprehensive data on their performance over time.

Brushing plays a crucial role in plaque control and maintaining oral hygiene, but it can also affect the surface properties and color stability of restorative materials.[5] Studies have shown that factors such as the abrasiveness of the toothpaste, brushing technique, and frequency can influence not only the wear of dental hard tissues but also the discoloration and surface texture of composite resins.[6,7] It is important to understand how these factors interact with restorative materials like nanohybrid composites, particularly in terms of their ability to resist color changes caused by daily oral hygiene practices.

The evaluation focuses on analyzing how color changes over time in samples subjected to staining and brushing simulations. The assessment of color changes was performed using the CIEDE2000 color difference formula, which provides a measurement more closely aligned with human visual perception of color differences.[2,8]

Energy-dispersive X-ray spectroscopy (EDX) and Scanning Electron Microscopy (SEM) analyses were conducted to examine the structural and surface characteristics of the samples. The EDX analysis determined the elemental composition of the materials, while the SEM analysis assessed surface morphology and structural integrity.[9,10] These analyses were used to understand the potential effects of the composite resins on color stability.

The composite resins used in this study, such as Dolgunn (HIMG Ceramic and Medical Composite Industry and Trade Limited Company, Turkiye) (DLG), RubyComp Nano (İnci Dental, Turkiye) (CMP), Nova Compo C (Imicryl Dental, Turkiye) (NVC), Parion (Dentac T-Resto, Turkiye) (PRN), are locally produced. The use of locally produced composite resins offers advantages such as cost-effectiveness and easy accessibility. Evaluating the competitiveness of locally produced composites against international standards contributes to the development of dental materials in Turkiye. Comparing the clinical performance of local composites with international brands can yield significant economic and clinical outcomes.

The composite resins used in this study include both locally produced Turkish materials-Dolgunn (DLG), RubyComp Nano (CMP), Nova Compo C (NVC), and Parion (PRN)- and an internationally recognized composite resin, Filtek Z550 (FLT). Evaluating the performance of locally produced composites in comparison to international brands could provide valuable insights for clinical practice, particularly regarding cost-effectiveness and accessibility in regions where international brands may not be readily available.

The primary hypothesis of this study is that significant differences in the color stability of the five nanohybrid composite resins will be observed after exposure to staining and brushing simulations over time. The secondary hypothesis posits that locally produced Turkish nanohybrid composites will exhibit comparable or superior color stability to the international composite resin FLT, given their similar formulations and intended clinical use.

This in vitro study aims to provide a comprehensive comparison of the color stability of locally produced and internationally recognized nanohybrid composite resins, evaluating their performance under simulated clinical conditions. By determining whether locally produced resins can perform as well as or better than international brands, this study seeks to support the use of cost-effective, aesthetically satisfactory alternatives in restorative dentistry.

METHODS

Study Design

Only restorative materials were used in this study. It was not tested on humans or animals and no materials derived from humans or animals were used. Therefore, ethics committee approval is not required. All procedures were carried out in accordance with the ethical rules and the principles of the Declaration of Helsinki. Five nanohybrid composite resin FLT, DLG, CMP, NVC and PRN were analyzed in this study. The category manufacturers, lots, and compositions of the composite resins are presented in Table 1. Figure 1 describes the study design, which shows the flow of the specimens through the different stages of the study. All specimens were subjected to staining and brushing simulation treatment. Color measurements were performed with spectrophotometer at baseline (t0), after 10 days brushing and staining (t1) and after 1 year brushing and staining (t2).

Specimens Size Calculation

The specimen size was calculated using G* Power statistical software. Based on the reference study values, a large effect size (f=0.80) was used. With 95% confidence (1-α) and 95% test power (1-β), the minimum sample size required for each group was 6, resulting in a total sample size of 30 for the one-way ANOVA. To account for potential specimen loss, the study was designed with 10 specimens per group.

Specimens Preparation

A total of 10 specimens (n=10) were prepared using silicon molds of 10x2 mm.[2] After the resin composite was placed in the molds with a slight overflow, a mylar strip and microscope slide were placed on the upper surfaces of the materials and polymerized for 10 s using a curing light (SmartLite Focus, Dentsply Sirona, USA). The slide was then removed, and the materials were polymerized by applying the curing light for 10 s over the mylar strip, according to the manufacturer's instructions and . The same curing light was used for all polymerization steps and the output of the light was controlled periodically using a radiometer (Woodpecker LED-F, Woodpecker Medical Instrument Co., China) to ensure an intensity of at least 1000 mW/cm^2 throughout the preparation of the specimens. Following the polymerization process,

Table 1. The category, manufacturers, lot numbers, and compositions of the composite resins

Material	Code	Material type	Composition	Filler content		Filler size	Shade	Manufacturer	Lot number
				wt%	vol%				
Filtek Z550	FLT	Nanohybrid	BIS-GMA UDMA BIS-EMA PEGDMA TEGDMA	81.8%	67.8%	0.01-3.5 µm	A2	3M ESPE, USA	N728631
Dolgunn	DLG	Nanohybrid	BIS-GMA UDMA TEGDMA SiO_2 Barium Alumino boro silicate glass powder Initiators Stabilizers	80%	61%	0.01-3 µm	A2	HIMG Ceramic and Medical Composite Industry and Trade Limited Company, Turkiye	626/0224
RubyComp Nano	CMP	Nanohybrid	BIS-GMA Methacrylate polymers (20%) Inorganic fillers (80%) Barium glass mixed oxides and copolymers, No photo-initiators and stabilizers	80%	-	0.02-0.7 µm	A2	İnci Dental, Turkiye	RCYA2275
Nova Compo C	NVC	Nanohybrid	BIS-GMA UDMA BIS-EMA PEGDMA TEGDMA Dimethacrylates (18-22% weight) ULS (Ultra Low Shrinkage) Monomer Barium glasses, Ytterbium Prepolymer	78%-83%	68%	0.4-0,7 µm	A2	Imicryl Dental, Turkiye	22M410
Parion	PRN	Nanohybrid	UDMA Bis-GMA TEGDMA Silica Quartz Pigments Initiators	77%-78%	-	0.1 µm	A2	Dentac T-Resto, Turkiye	PN220112

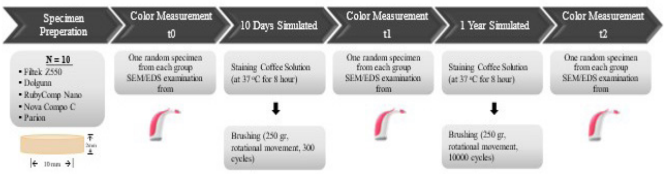


Figure 1. Flow chart of the study design

each of the specimens was polished with usin polishing disc (Optidisc, Kerr Corporation, USA) from extra-coarse to extra-fine at speed 10,000 rpm and 10 s each. A new disc was used for each specimen. The specimens were rinsed with water for 10 seconds to clean debris from the restoration surface then were kept in distilled water at 37° C in an incubator for 24 h post-polymerization.[2] All the procedures on the materials were applied by a single operator. In order to control the effect of press-on force on the polishing accuracy, the initial and final measurements of the thickness of each specimen were carried out 3 times by a single operator using an industrial type screw thread digital caliper (0.01 mm) with 0-150 mm measuring range.[11]

Staining Procedure

For the preparation of the coffee solution, 3,6 g of coffee was used per 300 ml of 100° C boiling water.[4] All solutions were allowed to reach 37° C. Eppendorf tubes were preferred to immerse the specimens individually in the study. 1.5 mm eppendorf tubes were filled with the solution and one specimen was placed inside. The tubes were kept in an oven at 37° C for 8 hour (t1-10 days) 12 days (t2-1 year) to replicate intraoral conditions. Specimens were turned over and immersed in fresh solutions every day to ensure uniform contact of the specimen with the staining solution and prevent contamination with bacteria and fungus.[2,4]

Brushing Procedure

The specimens removed from the solutions were subjected to brushing simulation with the MF-100 (Mod Dental, Esetron Smart Robotechnologies, Turkiye) brushing simulator. Toothbrush (Colgate Extra Clean 1+1, Colgate Palmolive, USA) and toothpaste (Sensodyne Çok Yönlü Koruma, Haleon, United Kingdom) with a relative dentin abrasivity (RDA) of 142 diluted 1/3 by volume were used in the brushing simulation. The toothpaste used in this study, Sensodyne (RDA 142), was selected because it represents a moderate level of abrasivity, which is common in commercially available toothpastes and reflects typical daily oral hygiene practices. The specimens were subjected to 300 (t1-10 days) and 10,000 (t2-1 year) cycles of brushing under a load of 250 g, with a circular motion with a movement diameter of 19 mm, and a movement speed of 30 mm/s, simulating 1 year of brushing. The toothbrush and paste were changed for each specimen.[12-14]

Color Assessments

The color of the specimens was assessed at three time points: t0, t1, and t2, using a digital spectrophotometer (Vita

Easyshade V, Vita Zahnfabrik, Germany). The color evaluation was based on the CIE Lab system, a three-dimensional color space where lightness (L*), red-green (a*), and yellow-blue (b*) components are represented. Specifically, L* indicates lightness on a scale from 0 (dark) to 100 (bright), a* reflects the red-green chromaticity, and b* corresponds to the yellow-blue chromaticity. For each specimen, three readings were taken from the center, and the average values of the L*, a*, and b* coordinates were recorded. Calibration of the instrument occurred between measurements. Color data were collected using an 18% grey card (L*=50, a*=0, b*=0) (JJC Photography Equipment Co. Ltd, China) as a reference.

The color differences between the measurements taken at different time points were calculated using the CIEDE2000 color difference formula.[15] ΔE was calculated by the CIEDE2000 formula using an online ΔE calculator (http://www.colormine.org/delta-e-calculator/Cie2000).

Color measurements were taken at three different time points: baseline (t0), after 10 days of brushing and staining simulation (t1), and after 1 year of brushing and staining simulation (t2). The color differences (ΔE) between these time points were calculated using the CIEDE2000 formula. ΔE1 represents the color difference between baseline (t0) and after 10 days (t1), while ΔE2 represents the color difference between baseline (t0) and after 1 year (t2). Additionally, ΔE3 represents the color difference between 10 days (t1) and 1 year (t2). These values were used to assess both short-term and long-term color stability of the composite resins.

Scanning Electron Microscope Imaging and Energy-Dispersive X-ray Examination

One specimen from each group were analyzed by SEM (Regulus 8230 FE-SEM, Hitachi High Tech Corporation, Japan) at time periods t0, t1 and t2 at ×5000 magnification, and images were recorded. Before the examination, the specimens were surface coated with 4 nm gold/palladium particles (Leica EM ACE600C, Leica Microsystems Inc., Canada) for surface conductivity. Subsequently, the representation of elemental presence in the composites was analyzed using EDX (X-Max 20, Oxford Instruments, Abingdon, UK).

Statistical Analysis

The data were analyzed using Minitab 14 and R software. Normality was assessed with the Shapiro-Wilk test. For parameters that followed a normal distribution according to composite and time, the Generalized Linear Models method was used for comparison, and multiple comparisons were performed with the Tukey test. For parameters that did not follow a normal distribution according to composite and time, the Two-Way Robust ANOVA method was used for comparison, and multiple comparisons were performed with the Bonferroni test. The results of the analyses were presented as mean ± standard deviation and median (min-max). The significance level was set at $p<0.05$.

RESULTS

The color difference measurements (ΔE) were calculated for three different intervals: ΔE1 (t0-t1), ΔE2 (t0-t2), and ΔE3 (t1-t2). ΔE1 represents the initial color changes observed after 10 days of staining and brushing simulation, while ΔE2 shows the long-term color changes after 1 year. ΔE3 reflects the additional color changes that occurred between 10 days and 1 year.

The main effect of the composite was not found to be statistically significant on the median ΔE values ($p=0.078$). The main effect of time was found to be statistically significant on the median ΔE values ($p<0.001$). Additionally, the interaction between composite and time was statistically significant on the median ΔE values ($p=0.001$). The highest median value of 3.12 was observed in the NVC composite from ΔE2, while the lowest median value of 1.285 was observed in the PRN composite from ΔE1 (Figure 2) (Table 2).

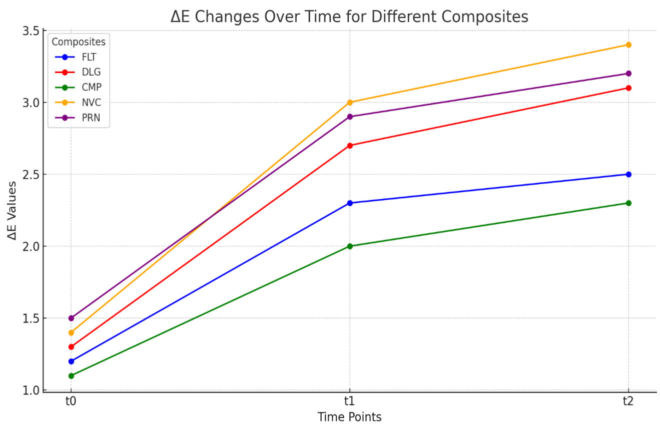


Figure 2. ΔE changes over time for different composites

During the ΔE1 time interval, the DLG group exhibited the highest color change value with a ΔE of 1.67 (1.36-2.33), while the PRN group showed the lowest value with a ΔE of 1.285 (1.05-1.79). Post-hoc analysis revealed no statistically significant differences among the composites during this interval ($p>0.05$ for all pairwise comparisons) ($p=0.078$).

Table 2. Comparison of ΔE values by composite and time

Time	Composite					Total		q	p
	FLT	DLG	CMP	NVC	PRN				
ΔE1 (t0-t1)	1.385 (0.74-1.9)^{ab}	1.67 (1.36-2.33)^{abc}	1.6 (1.18-2.49)^{ab}	1.495 (1.13-2.11)^{ab}	1.285 (1.05-1.79)^{b}	1.495 (0.74-2.49)^{a}	Composite	2.096	0.078
ΔE2 (t0-t2)	2.07 (1.64-2.46)^{acd}	2.7 (2.31-3.8)^{e}	2.385 (1.97-2.67)^{cde}	3.12 (2.38-3.59)^{de}	2.955 (2.51-3.81)^{e}	2.61 (1.64-3.81)^{b}	Time	37.334	<0.001
ΔE3 (t1-t2)	1.595 (1.04-3.24)^{abc}	1.675 (0.65-3.42)^{abc}	1.47 (0.47-2.49)^{abc}	2.04 (1.47-2.83)^{abcd}	2.2 (1.61-2.79)^{abcde}	1.785 (0.47-3.42)^{c}	Composite* time	26.676	0.001
Total	1.65 (0.74-3.24)	1.895 (0.65-3.8)	1.855 (0.47-2.67)	2.055 (1.13-3.59)	2.2 (1.05-3.81)	1.905 (0.47-3.81)			

Q: Two-way robust ANOVA; Median (min-max), $^{a-c}$: No difference between main effects with the same letter; $^{a-e}$: No difference between interactions with the same letter, FLT: Filtek Z550, In the ΔE2 time interval, the DLG group exhibited the highest color change value with a ΔE of 2.7 (2.31-3.8), whereas the FLT group had the lowest value with a ΔE of 2.07 (1.64-2.46). Statistically significant differences were observed among the groups in this time interval (p<0.001). Post-hoc analysis indicated that there was a significant difference between the DLG and FLT groups (p=0.02). Additionally, a significant difference was observed between the DLG and CMP groups (p=0.03). No significant differences were found between the other composites (p>0.05).

During the ΔE3 time interval, the PRN group showed the highest color change value with a ΔE of 2.2 (1.61-2.79), while the CMP group exhibited the lowest value with a ΔE of 1.47 (0.47-2.49). Significant differences were noted among the groups in this time interval (p=0.001). Post-hoc analysis revealed a significant difference between the PRN and CMP groups (p=0.015). A significant difference was also observed between the PRN and FLT groups (p=0.04). No significant differences were found between the other composites (p>0.05).

The main effect of the composite was found to be statistically significant on L mean value (p<0.001). The main effect of time was also significant on L value (p<0.001). Additionally, the interaction between composite and time was statistically significant (p=0.039). A decreasing trend in L values from t0 to t2 was observed for each composite (Table 3).

The main effect of the composite was found to be statistically significant on the median a value (p<0.001). The main effect of time was also found to be statistically significant on the median a value (p=0.027). Additionally, the interaction between composite and time was statistically significant on the median a value (p<0.001) (Table 4).

The main effect of the composite was found to be statistically significant on the median b values (p<0.001). The main effect of time was also found to be statistically significant on the median b values (p<0.001). Additionally, the interaction between composite and time was statistically significant on the median b values (p<0.001) (Table 5).

The SEM and EDX analyses, as presented in Figures 2-8, were performed to investigate the surface morphology and elemental composition of the restorative materials. SEM analysis, conducted at 5000x magnification, provided detailed topographical images, while EDX spectroscopy enabled the identification and quantification of elements present within the samples. Carbon and oxygen are predominant in all groups, with silicon and barium also being significant in certain groups. The presence of aluminum and additional elements like zirconium, fluorine, and sodium further differentiates the groups. These elemental compositions reflect the diverse material properties and potential applications in dental restorative materials.

In more detail, FLT group is distinguished by the presence of zirconium at 6.47%, which is not found in any other groups. The DLG group contains aluminum at 2.05%, with this element also appearing in CMP, NVC, and PRN groups, albeit in different proportions. The CMP group has aluminum at 2.72%, while the NVC group contains 2.54% aluminum, alongside unique elements such as fluorine at 2.88% and sodium at 0.38%. The PRN group does not have any unique elements compared to the other groups, but the proportions of the elements present differ. These findings highlight the distinctive elemental combinations and proportions within each group, underscoring their specific material characteristics and suggesting various potential applications in restorative dentistry.

DISCUSSION

The study's results support the primary hypothesis that significant differences in the color stability of the five nanohybrid composites were observed after staining and brushing simulations. Statistical analysis showed a significant main effect of time on median ΔE values (p<0.001) and a significant interaction between composite and time (p=0.001), indicating that color changes varied by composite type over time. At the ΔE1 and ΔE3 time points, all composites had similar color changes to FLT, but at ΔE2, FLT and CMP

Table 3. Comparison of L values by composite and time

Time	Composite						f	p
	FLT	DLG	CMP	NVC	PRN			
t0	78.14±0.78^{a}	71.23±1.18^{fgh}	77.13±1.31^{ab}	74.69±0.95^{c}	74.37±0.73^{cd}	Composite	239.52	<0.001
t1	76.27±0.51^{b}	69.69±1.24^{h}	75.63±1.35^{bc}	73.07±0.79^{de}	72.76±0.75^{ef}	Time	107.52	<0.001
t2	76.41±0.84^{b}	68.1±1.14^{i}	74.24±1.3^{cde}	71.45±0.67^{fg}	70.68±0.97^{gh}	Composite* time	2.11	0.039

f: Generalized linear models, mean ± SD, $^{a-i}$: No difference between interactions with the same letter

Table 4. Comparison of a values by composite and time

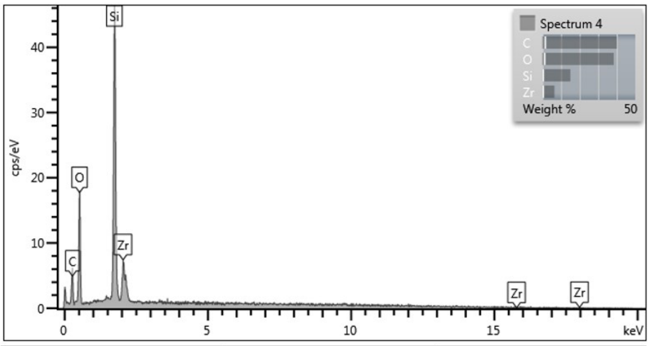
Time	Composite						q	p
	FLT	DLG	CMP	NVC	PRN			
t0	0.79 (0.6 - 0.83)^{a}	-0.42 (-1.13 - -0.17)^{de}	1.42 (1.02 - 1.93)^{g}	-2.2 (-3.1 - -1.6)^{i}	0.32 (0.1 - 0.93)^{be}	Composite	174.073	<0.001
t1	0.7 (0.65 - 0.77)^{a}	-1.27 (-1.69 - -0.73)^{df}	1.5 (1.1 - 1.83)^{gh}	-2.04 (-2.43 - -1.57)^{fi}	0.49 (0.2 - 0.9)^{bc}	Time	3.597	0.027
t2	0.5 (0.3 - 0.65)^{bc}	-1.54 (-1.77 - -1.07)^{f}	1.97 (1.37 - 2.2)^{h}	-1.23 (-2.14 - -0.93)^{df}	1.07 (0.26 - 1.5)^{acg}	Composite* time	76.177	<0.001

Q: Two-way robust ANOVA, median (min-max), $^{a-i}$: No difference between interactions with the same letter,

Table 5. Comparison of b values by composite and time

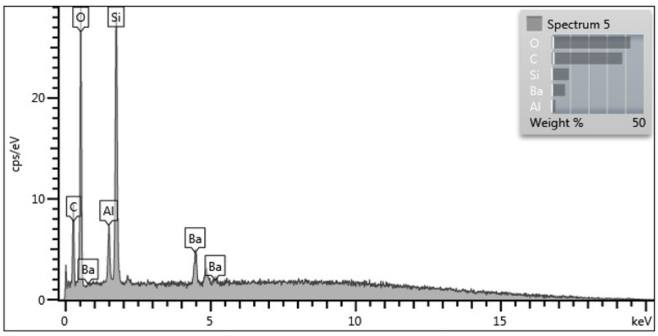
Time	Composite							
	FLT	DLG	CMP	NVC	PRN		q	p
t0	20.17 (19.9-21)[a]	11.49 (10.3-12.2)[c]	20.55 (19.03-23.2)[abef]	12.07 (9.8-3.7)[cd]	16.5 (15.57-17.4)[e]	Composite	259.765	<0.001
t1	20.4 (18.87-21.5)[a]	11.23 (10.3-11.87)[cd]	18.83 (17.4-20.87)[aef]	11.44 (9.9-12.63)[cd]	16.77 (14.43-17.27)[e]	Time	14.603	<0.001
t2	23.44 (22.13-24.57)[b]	12.43 (11.17-13.73)[cd]	20.35 (19.37-22.47)[a]	12.47 (11.32-13.73)[d]	18.6 (16.43-19.23)[f]	Composite* time	188.19	<0.001

Q: Two-way robust ANOVA, Median (min-max), [a-f]: No difference between interactions with the same letter



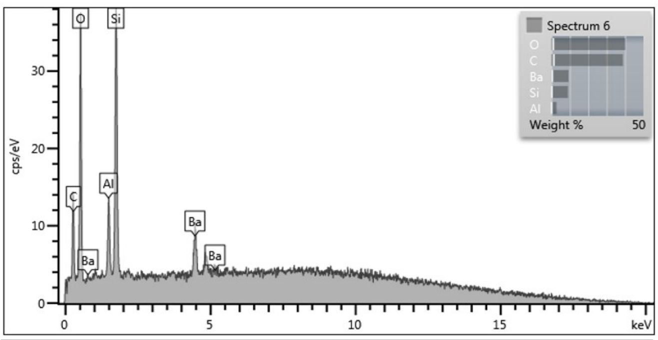
Element	Wt%	Wt% Sigma
C	40.07	1.59
O	38.45	1.19
Si	15.02	0.46
Zr	6.47	0.43
Total:	100.00	

Figure 3. Energy-dispersive X-ray spectroscopy (EDS) analysis of the FLT group



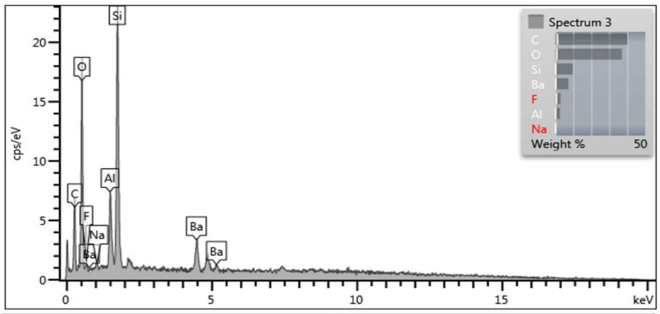
Element	Wt%	Wt% Sigma
C	38.38	1.12
O	42.85	0.91
Al	2.05	0.11
Si	9.38	0.23
Ba	7.34	0.35
Total:	100.00	

Figure 4. Energy-dispersive X-ray spectroscopy (EDS) analysis of the DLG group



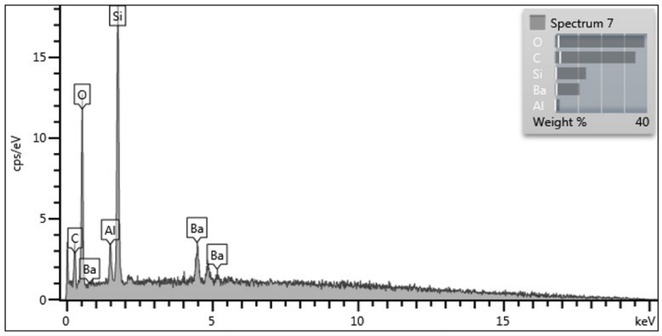
Element	Wt%	Wt% Sigma
C	38.85	1.36
O	40.19	1.07
Al	2.72	0.15
Si	8.94	0.27
Ba	9.31	0.49
Total:	100.00	

Figure 5. Energy-dispersive X-ray spectroscopy (EDS) analysis of the CMP group



Element	Wt%	Wt% Sigma
C	40.13	1.13
O	37.11	0.90
F	2.88	0.59
Na	0.38	0.12
Al	2.54	0.12
Si	9.73	0.25
Ba	7.23	0.33
Total:	100.00	

Figure 6. Energy-dispersive X-ray spectroscopy (EDS) analysis of the NVC group



Element	Wt%	Wt% Sigma
C	34.92	2.30
O	38.84	1.66
Al	2.07	0.20
Si	13.50	0.58
Ba	10.68	0.77
Total:	100.00	

Figure 7. Energy-dispersive X-ray spectroscopy (EDS) analysis of the PRN group

outperformed DLG, NVC, and PRN, partially supporting the secondary hypothesis.

The findings show that all composite resins experienced color change, but the extent varied significantly across materials over time. At the ΔE1 time point (t0-t1), initial color changes were minor and similar across all composites, indicating comparable short-term color stability. This likely reflects early surface reactions to staining agents without deep material penetration.

By the ΔE2 time point (t0-t2), more pronounced differences in color stability emerged. The FLT and CMP composites showed superior color stability compared to the DLG, NVC, and PRN composites. This suggests that the formulation and material properties of FLT and CMP provide better resistance to prolonged exposure to staining agents, likely

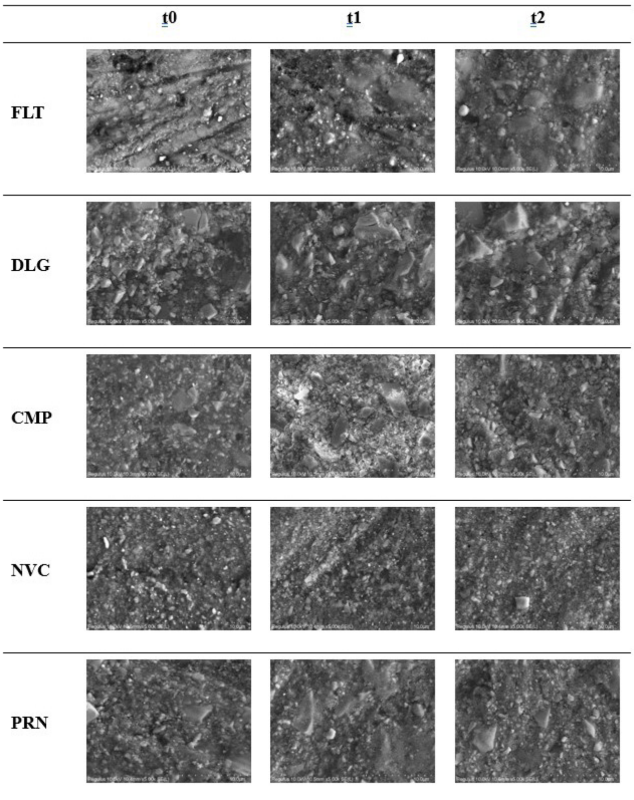


Figure 8. SEM analysis at 5000x magnification reveals the surface morphology of the groups

due to factors such as filler content, matrix composition, and surface properties that influence stain resistance and material degradation. These results align with previous research showing varying levels of color stability among different composite resins.[2,4,16]

At the ΔE3 time point (t1-t2), composites showed varying levels of color change, with PRN maintaining better color stability, likely due to its unique composition. The superior long-term performance of some composites highlights the importance of selecting materials with proven color stability for clinical applications.

Coffee was selected as the staining agent due to its common consumption and its known high potential for causing extrinsic dental stains.[2,3] This choice is relevant as it reflects the impact on the color stability of dental restorations over both short-term and long-term periods. The study's findings on how different composites responded to coffee staining provide valuable insights into their performance over short-term and long-term use, particularly for patients who frequently consume coffee and other staining beverages.

After staining with coffee, a decrease in L values was observed across all materials at all time points. This decrease indicates that the materials became darker due to the absorption of pigments. The consistent reduction in lightness across the different materials suggests that the coffee pigments effectively adhered to the surface of the restorative materials, leading to noticeable discoloration over time.[17,18] This outcome highlights the susceptibility of the materials to extrinsic staining in environments simulating real-world conditions where coffee is commonly consumed.

Overall, the significant differences observed in the color stability of the tested nanohybrid composite resins highlight the importance of considering material-specific properties when selecting dental composites for restorative procedures.[1,17] The findings support the primary hypothesis that staining and brushing simulations result in varied color stability among composite resins. The partial acceptance of the secondary hypothesis indicates that locally produced Turkish nanohybrid composites can match or exceed international standards in color stability under certain conditions.

In this study, brushing was incorporated as part of the simulation of intraoral conditions to evaluate the color stability of composite resins. Brushing is known to cause mechanical wear on tooth surfaces, which can potentially affect the color stability of composite materials by making them more susceptible to staining.[19] The toothpaste and brushing protocol used were carefully selected to simulate daily oral hygiene practices, closely reflecting real-world conditions.

Simulated brushing abrasion is widely recognized in the literature as an established in-vitro wear model that mimics clinical conditions.[14] In a study, it was noted that a typical patient performs approximately 15 brushing strokes per session, which equates to around 10,000 cycles over the course of a year with twice-daily brushing and brushing was simulated for 5,000, 10,000, and 20,000 cycles, corresponding to approximately 6, 12, and 24 months of brushing, respectively.[13] These findings highlight the importance of evaluating the resistance of composite materials to brushing when considering their long-term color stability, which is crucial for the success of aesthetic restorations in clinical practice.

Regarding the parameters for simulated toothbrushing, the present study aimed to systematically evaluate the influence of brushing time and load on the color stability of various composite materials. The 250 g load used in this study aligns with the range suggested in other research and is within the force limits commonly observed in clinical settings. Clinical studies have shown that brushing loads among individuals can vary widely, typically ranging from 140 g to 720 g, with a mean load comparable to 350 g.[20,21] The ISO technical specification for wear testing by toothbrushing recommends a force between 50 g and 250 g.[22] Previous studies have employed brushing forces of 200 g, 250 g, 350 g, and even 500 g.[21,23,24] The choice of 250 g in this study was made to balance the clinical relevance with the standardized conditions outlined in the literature.

The results indicate that locally produced Turkish nanohybrid composite resins (DLG, CMP, NVC, and PRN) exhibited varying degrees of color stability compared to the internationally recognized composite resin FLT. In the short term (ΔE1, t0-t1), all composites showed similar color changes, and the differences among them were not statistically significant. This suggests that locally produced composites can meet initial aesthetic expectations. However, in the long-term evaluation (ΔE2, t0-t2), FLT and CMP composites demonstrated superior color stability compared to DLG, NVC, and PRN composites. The superior performance of FLT and CMP is likely due to their optimized filler content and well-engineered matrix composition, highlighting the need for further development of locally produced composites to enhance their long-term performance.

Despite differences, some locally produced composites, particularly CMP, performed comparably to FLT under certain conditions. This suggests that with improved formulation and manufacturing, these composites could match international brands. Their cost-effectiveness and easy accessibility offer economic benefits and support national industry development, especially in regions with limited access to international brands.

The differences in color stability observed among the composite resins can be attributed to variations in their filler content, resin matrix composition, and overall formulation, even when filler size is similar.[25] For example, although both FLT and DLG have comparable filler sizes (0.01-3.5 μm and 0.01-3 μm, respectively), their filler content and resin matrices differ. FLT, with a higher filler volume (67.8% vol) compared to DLG (61% vol), likely exhibited superior color stability due to reduced resin matrix exposure, making it less prone to absorbing staining agents. The increased filler content helps form a smoother surface, limiting the penetration of extrinsic staining materials and improving resistance to mechanical wear.

In contrast, DLG's lower filler volume may lead to more exposure of the resin matrix, which is known to absorb water and staining agents over time, contributing to greater color changes. Additionally, the resin matrix composition of each composite plays a crucial role in color stability. FLT contains BIS-GMA and UDMA, both of which are known for their lower water sorption rates, enhancing its ability to maintain color over time. DLG, while also containing BIS-GMA and UDMA, includes alumino boro silicate glass powder, which may contribute to its differing performance in terms of color retention compared to FLT.

Similarly, CMP demonstrated competitive color stability, likely due to its well-distributed inorganic fillers and its barium glass mixed oxides, which contribute to the material's stain resistance. NVC, with its inclusion of ultra-low shrinkage (ULS) monomer and ytterbium, showed better performance in terms of long-term color stability, likely due to reduced polymerization shrinkage and its advanced filler technology.

Although there were differences among the composites, particularly with regard to their filler content and resin matrices, some locally produced composites, such as CMP, performed comparably to FLT under certain conditions. This demonstrates that with targeted improvements in formulation, locally produced composites can compete with international brands in terms of color stability. The competitive performance of these locally produced materials highlights their potential as cost-effective alternatives for aesthetic dental restorations.

In conclusion, locally produced Turkish nanohybrid composites showed comparable performance to FLT under specific conditions, highlighting their potential and the need for ongoing research to optimize their properties. Clinics can consider these composites as viable alternatives, particularly where cost and accessibility are key factors.

The structural and surface characteristics of the composite resins were examined using EDX and SEM.[9] These analyses provided crucial insights into the elemental composition and surface morphology of the materials, which influence their color stability. EDX analysis revealed the presence of various elements, including carbon, oxygen, silicon, barium, and aluminum.[9] The distribution and concentration of these elements varied among the composites. For example, the FLT contained zirconium, which was not found in the other composites. This unique elemental composition might contribute to FLT's superior color stability over time.[26]

SEM analysis revealed that FLT and CMP exhibited smoother surfaces with fewer pores, correlating with their lower ΔE values. These smoother surfaces reduce the likelihood of trapping staining agents, as well as minimizing the penetration of these agents into the material.[9] In contrast, DLG and PRN exhibited more porous and irregular surfaces, which might explain their higher ΔE values. The rougher surface and increased porosity observed in these composites likely facilitate greater staining agent retention, contributing to their more pronounced color changes over time.[4,18] The combination of EDX and SEM analyses allowed for a comprehensive understanding of the structural and surface properties of the composite resins and their impact on color stability. Composites with higher barium content, like FLT and CMP, tend to have greater radiopacity and surface integrity, which may reduce surface wear and enhance resistance to staining. In contrast, DLG and PRN contained less favorable filler compositions, which could lead to greater degradation under oral conditions, increasing their susceptibility to discoloration. These findings highlight the critical role that both surface morphology and elemental composition play in determining a composite's ability to maintain color stability. By linking the SEM and EDX findings to the ΔE values, it becomes evident that smoother surfaces, lower porosity, and favorable elemental compositions such as the presence of zirconium and higher barium content contribute to improved color stability. These structural and compositional advantages allow materials like FLT and CMP to better resist staining over time, while composites with less favorable properties show greater susceptibility to color changes.

The clinical relevance of the in vitro findings and their implications for real-world dental practice are crucial aspects of this study. While the results provide valuable insights into the color stability of different nanohybrid composite resins, it is essential to consider the need for further in vivo research.

The color change was calculated using the CIEDE2000 formula, which offers a better fit and is an update to the CIE Lab* formula, accounting for both the uniformity of the CIELAB color space and variations in lighting conditions.[2,8] The study's findings offer valuable guidance for clinical decision-making. The significant differences in color stability observed among the tested composites highlight the importance of material selection in achieving long-term aesthetic outcomes. Clinicians should consider both the initial color stability and the potential for long-term color changes when choosing composite resins for restorative procedures.

The importance of using locally produced composite resins in clinical applications is worth emphasizing. When selecting these materials for treatments, dentists should take into account factors such as initial and long-term color stability, mechanical properties, and cost-effectiveness. Locally produced composite resins offer cost-effective and readily accessible alternatives, providing significant advantages in clinical practice in Turkiye. These materials may be particularly beneficial in applications like anterior restorations, where achieving long-term aesthetic outcomes is crucial.

While there are limited studies directly evaluating locally produced Turkish composite resins, particularly regarding their surface properties and monomer structures, this study serves as a foundational reference by focusing on color stability an area where no prior research has been conducted on local composites.[10,27] By comparing these findings with international literature, we can better understand the position and potential of these local materials in the global market. Future research should continue to build on this work to further validate and optimize the performance of locally produced composites.

Clinical Significance

This study is clinically significant as it offers valuable insights for selecting composite resins in restorative dentistry. Understanding the color stability of various nanohybrid composites helps clinicians choose materials that maintain aesthetic quality over time. If locally produced Turkish composites demonstrate similar or superior color stability to international brands, they can provide cost-effective, readily available alternatives without sacrificing appearance, leading to improved patient satisfaction and potentially lower treatment costs. Additionally, the findings can guide manufacturers in enhancing composite resin formulations and performance, ultimately improving clinical outcomes and durability.

Limitations

This study has several limitations that should be considered when interpreting the results. The in vitro design does not fully replicate the complex oral environment, as factors like saliva, pH fluctuations, and enzymatic activity were not included, potentially affecting in vivo color stability. Additionally, the absence of mastication forces and enzymatic activity in the in vitro setup means that the mechanical wear and biochemical processes that occur in the oral cavity were not fully replicated. These factors can affect surface roughness and increase susceptibility to staining, potentially leading to different outcomes in vivo compared to those observed in the laboratory setting. Thermal cycling, which simulates temperature changes in the oral cavity, was also omitted, though these fluctuations can significantly impact the physical properties and color stability of dental materials. The laboratory conditions under which specimens were tested may not perfectly mirror clinical conditions, including factors like mastication wear, interaction with various substances, and patient-specific variables. The focus on specific nanohybrid composite resins limits the generalizability of the findings, as differences in formulation and filler content can lead to variability. Although a 1-year evaluation period was included, it may not fully capture the long-term color stability of the materials. Extended observation periods and more comprehensive simulations of the oral environment are recommended for future research to better reflect clinical conditions.

CONCLUSION

In conclusion, this study underscores the need for further research to fully understand the long-term performance of nanohybrid composite resins in clinical settings. By addressing the limitations of in vitro studies and conducting more extensive in vivo research, we can better inform clinical practices and improve patient outcomes. The findings from this study provide a foundation for future research and highlight the potential of locally produced composites in achieving comparable or superior performance to international brands.

ETHICAL DECLARATIONS

Ethics Committee Approval

Only restorative materials were used in this study. It was not tested on humans or animals and no materials derived from humans or animals were used. Therefore, ethics committee approval is not required.

Informed Consent

It was not tested on humans or animals and no materials derived from humans or animals were used. Therefore, informed consent is not required.

Referee Evaluation Process

Externally peer-reviewed.

Conflict of Interest Statement

The authors have no conflicts of interest to declare.

Financial Disclosure

The authors declared that this study has received no financial support.

Author Contributions

All of the authors declare that they have all participated in the design, execution, and analysis of the paper, and that they have approved the final version.

REFERENCES

1. Alzraikat H, Burrow MF, Maghaireh GA, Taha NA. Nanofilled resin composite properties and clinical performance: a review. *Oper Dent*. 2018;43(4):E173-E190.
2. Paolone G, Formiga S, De Palma F, et al. Color stability of resin-based composites: Staining procedures with liquids-a narrative review. *J Esthet Restor Dent*. 2022;34(6):865-887.
3. Ipek İ, Bilge K. The effect of different liquids on the surface roughness and color stability of single shade and nanohybrid resin composites: an AFM and SEM analysis. *Microsc Res Tech*. 2024;87(9):2063-2071.
4. Ertaş E, Güler AU, Yücel AC, Köprülü H, Güler E. Color stability of resin composites after immersion in different drinks. *Dent Mater J*. 2006;25(2):371-376.

5. Figuero E, Nóbrega DF, García-Gargallo M, Tenuta LM, Herrera D, Carvalho JC. Mechanical and chemical plaque control in the simultaneous management of gingivitis and caries: a systematic review. *J Clin Periodontol.* 2017;44 Suppl 18:S116-S134.
6. Wiegand A, Burkhard JP, Eggmann F, Attin T. Brushing force of manual and sonic toothbrushes affects dental hard tissue abrasion. *Clin Oral Investig.* 2013;17(3):815-822.
7. Kumar S, Kumar Singh S, Gupta A, Roy S, Sareen M, Khajuria S. A profilometric study to assess the role of toothbrush and toothpaste in abrasion process. *J Dent (Shiraz).* 2015;16(3 Suppl): 267-273.
8. Garg SA, Chavda SM. Color masking white fluorotic spots by resin infiltration and its quantitation by computerized photographic analysis: a 12-month follow-up study. *Oper Dent.* 2020;45(1):1-9.
9. Abd Mutalib M, Rahman M, Othman M, et al. Scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) spectroscopy. Membrane characterization. Elsevier, 2017,pp.161-179.
10. Ucuncu MK, Celiksoz O, Sen E, et al. Investigation of the degree of monomer conversion in dental composites through various methods: an in vitro study. *Appl Sci.* 2024;14(11): 4406.
11. Tepe H, Can Yaman B, Akyüz İ, Ozer F. Effect of rotation frequency of polishing discs on the surface roughness of resin composite material. *Proc Inst Mech Eng H.* 2024;238(7):803-813.
12. Heintze SD, Forjanic M, Ohmiti K, Rousson V. Surface deterioration of dental materials after simulated toothbrushing in relation to brushing time and load. *Dent Mater.* 2010;26(4):306-319.
13. Hazar A, Akgül S, Koçak S, et al. Effect of brushing time with a whitening dentifrice on surface roughness of anterior composite resins. *J Dent Indones.* 2022;29:1.
14. Monteiro B, Spohr AM. Surface roughness of composite resins after simulated toothbrushing with different dentifrices. *J Int Oral Health.* 2015;7(7):1-5.
15. Lee YK. Comparison of CIELAB DeltaE(*) and CIEDE2000 color-differences after polymerization and thermocycling of resin composites. *Dent Mater.* 2005;21(7):678-682.
16. Poggio C, Vialba L, Berardengo A, et al. Color stability of new esthetic restorative materials: a spectrophotometric analysis. *J Funct Biomater.* 2017;8(3):26.
17. Alkhadim YK, Hulbah MJ, Nassar HM. Color shift, color stability, and post-polishing surface roughness of esthetic resin composites. *Materials (Basel).* 2020;13(6):1376.
18. Leite MLAES, Silva FDSDCME, Meireles SS, Duarte RM, Andrade AKM. The effect of drinks on color stability and surface roughness of nanocomposites. *Eur J Dent.* 2014;8(3):330-336.
19. Dindar MB and Atay MT. The effect of brushing force on the surface properties and color stability of dental enamel. *NEU Dent J.* 2023;5(3):167-172.
20. Van der Weijden GA, Timmerman MF, Danser MM, Van der Velden U. Relationship between the plaque removal efficacy of a manual toothbrush and brushing force. *J Clin Periodontol.* 1998; 25(5):413-416.
21. Tanoue N, Matsumura H, Atsuta M. Wear and surface roughness of current prosthetic composites after toothbrush/dentifrice abrasion. *J Prosthet Dent.* 2000;84(1):93-97.
22. ISO TR. Dental materials guidance on testing of wear: Part 1: wear by tooth brushing. Technical Report. doi:ISOTR.2007;14569:1
23. Teixeira EC, Thompson JL, Piascik JR, Thompson JY. In vitro toothbrush-dentifrice abrasion of two restorative composites. *J Esthet Restor Dent.* 2005;17(3):172-182.
24. Cho LR, Yi YJ, Heo SJ. Effect of tooth brushing and thermal cycling on a surface change of ceromers finished with different methods. *J Oral Rehabil.* 2002;29(9):816-822.
25. Nasim I, Neelakantan P, Sujeer R, Subbarao CV. Color stability of microfilled, microhybrid and nanocomposite resins-an in vitro study. *J Dent.* 2010;38 Suppl 2:e137-e142.
26. Wang Y, Hua H, Liu H, Zhu M, Zhu XX. Surface modification of ZrO_2 nanoparticles and its effects on the properties of dental resin composites. *ACS Appl Bio Mater.* 2020;3(8):5300-5309.
27. Üçüncü MY and Üçüncü MK. Comparison of the mechanical properties of various microhybrid dental composites. *EDR.* 2023;22(2):58-64.