

Research Article

THE EFFECT OF DIFFERENT STAINING SOLUTIONS ON THE COLOR STABILITY OF PERMANENT INDIRECT COMPOSITE RESINS PRODUCED BY ADDITIVE AND SUBTRACTIVE TECHNIQUES

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ABSTRACT

Objective: To investigate the effects of various beverages on the color stability of permanent composite resins produced by additive (AM) or subtractive manufacturing (SM) techniques comparatively.

Materials and Methods: Six composite resin materials produced by SM (Vita Enamic-VE, Cerasmart-CE, Lava Ultimate-LU) and AM (VarseoSmile Crown plus-VSC, Saremco print CROWNTECH-SPC, Formlabs 3B Permanent Crown-FPC) techniques were selected and soaked in different solutions (artificial saliva, black tea, coffee) for different times (1 and 7 days). L*, a*, b* values of the samples were recorded using a spectrophotometer. The color changes of the samples were determined using the CIELAB formula. In determining the color differences between the test materials, ANOVA was used for parametric data and Kruskal-Wallis analysis for non-parametric data.

Results: Group VE was the least stained group on the 1st and 7th day of artificial saliva solution and the 7th day of coffee solution, while Group CE was the least stained group on the 1st day of coffee solution. In the tea solution, on the 1st and 7th days, there wasn't difference in the materials' color change ($p>0.05$). Tea and coffee solutions caused statistically significantly more color change in all test materials than artificial saliva (except Group CE on the 7th day, Group VSC and FPC on the 1st day) ($p<0.05$).

Conclusion: 3D permanent composite resins generally showed more staining than CAD/CAM milled composite resins. Tea and coffee staining solutions changed the color of the materials compared to artificial saliva. As the storage time increased, more color changes were observed.

Keywords: Composite Resins, 3D printing, CAD/CAM milling, Color stability

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INTRODUCTION

Recent advancements in the digital workflow have significantly impacted research fields in dentistry today (1). Particularly, with the advancement of computer-aided design and computer-aided manufacturing (CAD/CAM), dental treatments can be performed more effectively, easily and quickly (2). Focusing on a digital workflow, CAD/CAM systems have significantly surpassed traditional prosthesis manufacturing procedures (1). Following data collection, the digital workflow uses CAD software to process the data before digitally designing the restorations. During the fabrication process, additive manufacturing (AM) or subtractive manufacturing (SM) techniques may be preferred (3).

The SM technique, causes more material to be consumed than the material to be obtained because it performs production by milling from a block of material (1,3). As such, the cost is relatively high. The AM method, which is based on layering layers on top of each other, was developed following the need to reduce costs (4). The term AM is the process of creating a three-dimensional (3D) object designed in a virtual environment with 3D printers that perform the printing process (5). It is predicted that 3D printers used in AM technology will become the main method for digital production in dentistry in the future by finding wide usage areas in many fields, especially in prosthodontics, due to their advantages such as fast and detailed production, freedom and ease of design, material and workforce saving (1, 3, 4). Stereolithography (SLA) and digital light processing (DLP) are the two AM methods most popularly utilized in resin-based dental restorations. The foundation of both technologies is the polymerization of liquid resin with a UV and/or laser source (3).

Dental material development related to AM technology has significantly increased in recent years (6, 7). A variety of printable resins have entered the market recently and are being used for producing both permanent and temporary fixed prosthetic restorations. The use of composite resin materials for permanent restorations, particularly single crowns, inlays, onlays, and veneers, has emerged as a suitable solution for restorative and prosthetic rehabilitation (8, 9). However, their use is still limited by their questionable mechanical and physical properties (10, 11), biocompatibility (12, 13), antimicrobial properties (14), and optical properties due to the presence of residual monomers formed during post-processing (15-17).

Color harmony and color stability are essential in composite materials because they are used as materials that mimic the appearance of natural teeth. One of the most important indications for the replacement of composite restorations after intraoral use is color change (18). Discoloration of restorative materials is an undesirable effect of aesthetic dental resin composites, although inevitable in the oral environment (19).

It is known that the various stain types to which the material is subjected, the surface roughness and composition, exposure frequency, and duration all have an impact on the color stability of restorative

materials (17). The color stability of conventional resin restorative materials has been shown to be considerably impacted by frequent exposure to coloring beverages like coffee and tea (19). In addition to material composition, finishing and polishing techniques can also have an impact on the quality of the composite surface and may therefore be associated with early discoloration (21). The manufacturing method used can also have an impact on the color stability of the restorative material (17). In particular, the "stair-step phenomenon" commonly observed in 3D printing technology may be important when considering the aesthetic properties of composite resin restorations and hence color stability (17, 22). Additionally, the AM technique's post-polymerization step influences the material's ultimate structure based on the quantity of residual monomer, which could directly affect the printed material's color stability (23).

Thresholds for visual color differences can be applied as a quality control measure and as a reference for choosing aesthetic materials (24). Visual assessment and digital color measurement devices are used in the evaluation of color change. In visual assessment, many factors such as light source, gingival color, distance of the colored object to the eye, environmental factors, and experience affect the color assessment of dentists (24). Therefore, digital colorimeters such as colorimeters and spectrophotometers, which can make reproducible measurements with sensitive, quantitative techniques, are preferred to obtain more precise and reliable results in the evaluation of color change (24, 25).

For measuring color difference values, the Commission Internationale de l'Eclairage (CIE) L* a* b* color system is utilized (26, 27). L* a* b* values are referred to as "chromaticity coordinates" in this 3D color system: The L* a* b* values in this 3D color system are referred to as "chromaticity coordinates":(L*) stands for "lightness"; the higher the L value, the greater the lightness. (a*) shows positive values as red and negative values as green (-a*=green; +a*=red); (b*) shows positive values as yellow and negative values as blue (b*=blue; +b*=yellow). Mean color difference values (ΔE) is a numerical value indicating the amount of perceived color difference between two objects (26, 28).

In the researches, color changes and surface roughness of resin based composite blocks produced with CAD/CAM milling systems have been investigated (26, 29). However, a scoping analysis conducted recently (30) showed that the properties of 3D printing polymers have not yet been sufficiently assessed and characterized. This knowledge is important to successfully manage these materials and the satisfaction of patients' increasing aesthetic preferences. Although no comprehensive studies are comparing permanent polymeric-based composite resins produced by both subtractive and additive digital methods, the studies that have been conducted generally include 3D composite resins used for temporary restorations (1, 31-35).

Therefore, the purpose of this study was to compare the effects of coloring-potent beverages like coffee and tea on the staining and color stability of permanent composite resins produced by the AM and SM techniques. The study's null hypothesis declares that there won't be any variations in staining properties

based on the type of restoration material used, the kind of colorant solutions employed, or how long the ingredients in the colorants are stored.

MATERIALS AND METHODS

This study comparatively investigated the staining sensitivity and color stability of permanent composite resins produced by SM (Vita Enamic-VE, Cerasmart-CE, Lava Ultimate-LU) and AM (VarseoSmile Crown plus-VSC, Saremco print CROWNTECH-SPC, Formlabs 3B Permanent Crown-FPC) method in three different solutions (artificial saliva, black tea, coffee) at different times (0, 1 and 7 days).

Table 1. Content details of composite resins in test groups

Production method	Trade name	Abbr	Material type	Matrix Type	Filler type and content	Manufacturer
SM	Vita Enamic	VE	Polymer infiltrated ceramic network	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide (86%) Silica (20nm), zirconia (4–11 nm)	Vita Zahnfabrik, Bad Sackingen, Germany
	Cerasmart	CE	Resin nano ceramic	BisMEPP, UDMA, DMA	Silica (20 nm) ve Barium glass (300 nm) (71%)	GC Corp., Tokyo, Japan
	Lava Ultimate	LU		BisGMA, UDMA, BisEMA, TEGDMA	Silica (20 nm), zirconia (4–11 nm) (80%)	3M ESPE Dental Products, St. Paul, MN, USA
AM	Saremco print CROWNTEC	SPC	3D printing composite	BisEMA	Silanized dental glass (0.7µm) (30-50%)	Saremco Dental AG; Rebstein, Switzerland
	VarseoSmile Crown plus	VSC		2-methylprop-2-enoic acid phosphine oxide	Silanized dental glass (0.7µm) (30-50%)	Bego, Bremen, Germany
	Formlabs 3D Permanent crown	FPC		50-75% esterification products of 2-methylprop-2-enoic acid (methacrylate)	Silanized dental glass (0.7µm) (30-50%)	Formlabs, Somerville, MA, US

(SM: Subtractive manufacturing, AM:, Additive manufacturing, 3D: three-dimensional , Bis-GMA: bisphenol A diglycidylether methacrylate, Bis-MEPP: 2,2-Bis (4-methacryloxypropoxyphenyl) propane, UDMA: urethane dimethacrylate, TEGDMA: triethylene glycol dimethacrylate, Bis-EMA: ethoxyl ated bisphenol-A dimethacrylate, DMA: dimethacrylate)

The minimum sample size for this study was calculated as $n = 12$ ($N = 216$) per group with an effect size of 0.50, 90% power, and an error level of $\alpha = 0.05$. Table 1 presents an overview of the properties and compositions of all the materials employed in the research.

Table 2. Production and polishing procedures of test groups

Group code	Production methods	Post-processing procedure*	Polishing procedure*
VE	CAD/CAM blocks were wet sliced with a diamond saw using a precision cutting machine (Microcut 201, Metkon, Turkey)	-	<ul style="list-style-type: none"> - The top surfaces of all samples were polished with Sof-Lex Diamond Polishing System (3M ESPE St Paul, MN, USA). - 4 different grades of abrasive aluminum oxide coated discs were applied in decreasing order of grits. - Coarse (100 μm), Medium (29 μm), Fine (14 μm), and Super Fine (8 μm) discs were used, respectively. - A single operator used a low speed hand equipment running at 15,000 rpm to apply a dry. - A new disc was used to polish each specimen. - Each disc was applied in the same direction for 15-20 seconds for standardization.
CE			
LU			
SPC	AM technique: DLP-based Printer: Asiga Max UV (Asiga, Anaheim Hills, CA, USA) Printing layer thickness: 50 μm Printing orientation: 0°	<ul style="list-style-type: none"> - Washing was performed for 2-3 minutes in 98% concentrated isopropanol using ultrasonic technology. - The Otoflash G171 device (NK Optik, Baierbrunn, Germany) was used to cure the light with 4000 lighting exposures while being in a nitrogen oxide gas atmosphere. 	
VSC	AM technique: DLP-based Printer: Varseo XS (Bego, Bremen, Germany) Printing layer thickness: 50 μm Printing orientation: 0°	<ul style="list-style-type: none"> -It was washed in a 96% concentrated ultrasonic ethanol bath for 3 minutes and then for 2 minutes. -It was pressurized with nitrogen gas (1.0–1.2 bar) in Otoflash (Bego, Bremen, Germany), which produced 1500 flashes while performing at a frequency of 10 light per second. 	
FPC	AM technique: SLA-based Printer: Formlabs (Somerville, Massachusetts, USA) Printing layer thickness: 50 μm Printing orientation: 0°	<ul style="list-style-type: none"> - Submerged in Form Wash (Formlabs, Somerville, MA, USA) for 3 minutes in 99% isopropyl alcohol. -Post-curing was performed twice in Form Cure (Formlabs, Somerville, MA, USA) at 390–405 nm, 60°C for 20 minutes. 	

* Performed in accordance with the manufacturer's instructions.(CAD/CAM: Computer-aided design and computer-aided manufacturing, AM: Additive manufacturing, DLP: Digital light processing, SLA: Stereolithography, VE: Vita Enamic, CE: Cerasmart, LU: Lava Ultimate, SPC: Saremco print CROWNTEC, VSC: VarseoSmile Crown plus, FPC: Formlabs 3D Permanent crown)

CAD/CAM blocks were wet-sliced by a diamond saw (Micracut 201, Metkon, Bursa, Turkey) and 108 samples were obtained with a rectangular shape (14 mm x 14 mm x 1.0mm). 3D printing materials (VSC, SPC, FPC) were designed in the same dimensions using a CAD software program (SolidWorks Corp. Concord, MA, USA) and transported to a 3D printer after being converted to Standard Transform Language (STL) format. Group VSC, SPC, and FPC were produced with Asiga Max UV (Asiga, Anaheim Hills, CA, USA), Varseo XS (Bego, Bremen, Germany), Formlabs (Somerville, Massachusetts, USA) 3D printer with a

print layer thickness of 50 μm and a print orientation of 0° , respectively. Post-processing of the 3D test samples was performed by each manufacturer's instructions. Table 2 contains a detailed explanation of these procedures.

For surface standardization, the samples were ground wet with 600, 800, and 1000 grit silicon carbide abrasive paper. All of the sample's dimensions were measured using a digital caliper (Mitutoyo Corp., Tokyo, Japan). Then the top surfaces of all samples were polished by a trained inspector (N.E.O) with Sof-Lex (3M ESPE Dental Products, St. Paul, Minnesota) with low-speed hand equipment. Abrasive aluminum oxide-coated discs of 4 different grades, which are Coarse (100 μm), Medium (29 μm), Fine (14 μm), and Super Fine (8 μm), were applied in descending grit order (Table 2). The produced samples were then submerged in distilled water at 37°C for 24 hours after being washed for 5 minutes in an ultrasonic cleaner.

Following a random division into subgroups ($n = 12$), a total of 36 samples (A2 shade) from each type of material were submerged in two distinct staining solutions (tea, coffee) and artificial saliva to analyze the time-dependent color difference of the samples obtained. The properties and preparation procedures of the staining solutions used are presented in Table 3.

Table 3. The properties and preparation procedures of the staining solutions

Immersion solution	Manufacturer	Chemical composition	Preparation procedure
Artificial saliva	-	KCl (0.4 g L ⁻¹) NaCl (0.4g L ⁻¹) CaCl ₂ (H ₂ O) (0.795 g L ⁻¹) NaH ₂ PO ₄ (H ₂ O) (0.69 g L ⁻¹) Na ₂ S (0.005 g L ⁻¹) Urea (1 g L ⁻¹)	To create 1 L of artificial saliva, all of the ingredients were mixed. The pH of the saliva was then determined, and 15 ml of 0.1 M NaOH was added to the mixture to obtain a pH of 6.5.
Black Tea	Yellow Label Tea; Lipton, Rize, Turkey	Tea Leaves	1 tea bag was placed in 200 mL of boiled distilled water for 10 min.
Coffee (without sugar)	Nescafe Classic; Nestle, Switzerland	Coffee powder	2 g coffee granules were placed in 200 ml boiled distilled water for 7 min and allowed to cool at room temperature.

(NaCl: Sodium chloride, KCl: Potassium chloride, CaCl₂(H₂O): Calcium chloride hydrate, NaH₂PO₄(H₂O): Sodium dihydrogen phosphate monohydrate, Na₂S: Sodium sulfide, NaOH: Sodium hydroxide)

A spectrophotometer (VITA Easyshade V; Vita Zahnfabrik, Germany) was used to record the samples' quantitative basic color parameters (L^* , a^* , and b^*) in accordance with the CIE system. Measurements were performed in standard D65 illumination (36) in a light-controlled box (31) against a neutral grey background (37). To minimize the possibility of external light reflection on the sample side and to ensure that the light

angle was maintained throughout the test procedure, a custom-made silicone mold was used to hold the sample during color measurement (33). The instrument was calibrated before each measurement, and each sample was measured three times. The values of L^* , a^* , and b^* were obtained by averaging these measurements.

Using the previous reference for artificial saliva (38), coffee and tea were prepared in accordance with the manufacturer's instructions. All samples were then stored in coloring solutions for 24 hours at 37°C in an incubator. All staining solutions were refreshed after each 24 h immersion (39). After the coloring period, each sample was cleaned under distilled water to remove any deposits of coloring solutions and dried with tissue paper before re-measurements (40). The coloring cycle was performed for 7 days and color measurements of all samples were repeated on days 1 and 7. Assuming that 1 cup of coffee is drunk in 15 minutes, it was determined that the exposure time to the coloring beverage was 48 minutes per day and 24 hours per month with an average consumption of 3.2 cups of coffee per day (41). For this reason, the first measurement period selected in this study was determined as 1 day (24 hours), which corresponds to 1 month of use in the mouth, and the other measurement period was increased to 1 week to see the degree of exposure. The color changes of the samples were obtained by calculating the ΔE value according to the CIE L^*a^*b system. In the formula, L_1^* , a_1^* , and b_1^* are the first measurement values and L_2^* , a_2^* , and b_2^* are the second measurement values.

$$\Delta E^* = [(L_1^* - L_2^*)^2 + (a_1^* - a_2^*)^2 + (b_1^* - b_2^*)^2]^{1/2}$$

Statistical analyses of surface roughness and ΔE values were calculated using the SPSS (Version 22 IBM) package program. The Shapiro-Wilk and Kolmogorov-Smirnov tests were performed to determine whether the data was distributed normally. In determining the color differences between the test materials, the Kruskal-Wallis analysis was used when a one-way analysis of variance was not available. The time-dependent color change within each material group was evaluated by using the Wilcoxon sign test when the dependent sample t-test was normal distribution ($\alpha=0.05$).

RESULTS

The ΔE values obtained from the test groups are shown in Table 4. When material groups are examined on a solution basis; in the artificial saliva solution; Group VE was stained the least on the 1st day and there is a significant difference with all other groups except Group LU and Group VSC. On the 7th day, Group CE was stained the most and had a significant difference with Group VE and Group FPC ($p<0.05$). In the tea solution, between the materials, there was no difference in color change on the 1st and 7th day

($p > 0.05$). In the coffee solution, Group CE stained the least on the 1st day and had a significant difference with Group LU and Group FPC ($p < 0.05$). On the 7th day, Group VE stained the least and there is a significant difference with Group LU which stained the most.

Table 4. ΔE values of the test groups in soaked solutions on the 1st and 7th day

Time	Materials	Staining solution			<i>p</i>
		Artificial saliva	Black tea	Coffee	
$\Delta E 0-1$	VE	0.67 ± 0.37 ^{abcAB} 0.57 (0.50)	3.07 ± 1.40 ^{aA} 2.45 (2.32)	2.37 ± 1.15 ^{aB} 2.06 (1.40)	0.000*
	CE	1.56 ± 0.77 ^{aA} 1.54 (1.00)	2.37 ± 0.89 ^{bA} 2.32 (1.16)	1.67 ± 0.61 ^{bcB} 1.78 (0.97)	0.030*
	LU	1.35 ± 0.63 ^{dA} 1.09 (1.22)	2.09 ± 0.70 ^{cB} 2.16 (1.25)	3.14 ± 1.32 ^{bAB} 2.90 (1.52)	0.000*
	SPC	1.71 ± 0.74 ^{bA} 1.57 (1.11)	2.76 ± 1.12 ^{dA} 2.86 (2.84)	2.75 ± 1.39 ^{dB} 2.93 (2.20)	0.010*
	VSC	1.85 ± 1.66 ^{eA} 1.18 (1.66)	2.06 ± 0.73 ^{eB} 1.95 (1.30)	2.69 ± 1.35 ^{eC} 2.65 (2.12)	0.278*
	FPC	2.34 ± 1.54 ^{eA} 1.90 (1.54)	3.33 ± 1.52 ^{fB} 3.07 (0.99)	3.23 ± 1.13 ^{cC} 3.38 (1.48)	0.181*
<i>p</i>		0.001**	0.027**	0.025*	
$\Delta E 0-7$	VE	1.72 ± 0.95 ^{aAB} 1.44 (1.47)	5.25 ± 1.44 ^{aA} 5.19 (2.33)	3.80 ± 1.94 ^{aB} 2.96 (3.21)	0.000*
	CE	4.66 ± 2.42 ^{abA} 4.56 (2.88)	5.31 ± 2.27 ^{bB} 5.50 (3.27)	4.87 ± 2.03 ^{bc} 4.34 (2.54)	0.776*
	LU	2.44 ± 0.88 ^{cA} 2.30 (0.87)	3.99 ± 1.59 ^{cB} 3.62 (2.31)	6.05 ± 1.74 ^{aAB} 5.96 (2.80)	0.000*
	SPC	3.31 ± 1.41 ^{dAB} 2.60 (1.29)	5.05 ± 1.87 ^{dA} 4.47 (2.27)	4.70 ± 1.85 ^{cB} 4.06 (3.50)	0.004*
	VSC	2.29 ± 1.00 ^{eAB} 2.13 (1.13)	5.13 ± 1.80 ^{eA} 4.81 (2.31)	6.00 ± 2.01 ^{dB} 5.91 (2.90)	0.000*
	FPC	2.41 ± 0.89 ^{bAB} 1.49 (0.85)	5.07 ± 1.11 ^{fA} 4.83 (2.27)	5.63 ± 1.35 ^{eB} 5.16 (2.51)	0.000*
<i>p</i>		0.000**	0.444*	0.021*	

*One-way ANOVA, ** Kruskal-Wallis H test. (VE: Vita Enamic, CE: Cerasmart, LU: Lava Ultimate, SPC: Saremco print CROWNTEC, VSC: VarseoSmile Crown plus, FPC: Formlabs 3D Permanent crown). The data are presented as mean ± standard deviation (Mean ± SD) and median and interquartile range Median(IQR). The p value is statistically significant (in bold). Mean difference significant at $p < 0.05$; means with same letters statistically different. Lowercase letters for columns, uppercase letters for rows.

When examined according to the solutions immersed within the group on a material basis; in Group VE; tea and coffee solutions caused significant color change compared to artificial saliva on the 1st and 7th day ($p < 0.05$). In Group CE; tea solution caused significant color change compared to artificial saliva on the

1st day ($p < 0.05$), while the staining effect of the solutions was not significant on the 7th day ($p > 0.05$). In Group LU, coffee showed significantly higher staining than the other solutions on the 1st and 7th day ($p < 0.05$). In group SPC, tea solution caused significant color change compared to artificial saliva on the 1st day, while coffee, as well as tea, caused significant color change compared to artificial saliva on the 7th day ($p < 0.05$). In Group VSC and Group FPC, while the staining effect of the solutions was not significant on the 1st day ($p > 0.05$), tea and coffee solutions caused significantly higher discoloration compared to artificial saliva on the 7th day ($p < 0.05$). When all test groups were analysed, 3D permanent composite resins generally showed more staining than CAD/CAM milled composite resins.

The comparison of the ΔE values of the composite samples on the 1st and 7th day within the groups are given in Figure 1. The ΔE values of all samples on the 7th day were higher than the 1st day ΔE values. Group VE, Group CE, and Group LU in black tea, Group SPC and Group VSC in artificial saliva solution, and Group VE and Group SPC in coffee showed statistical differences ($p < 0.05$).

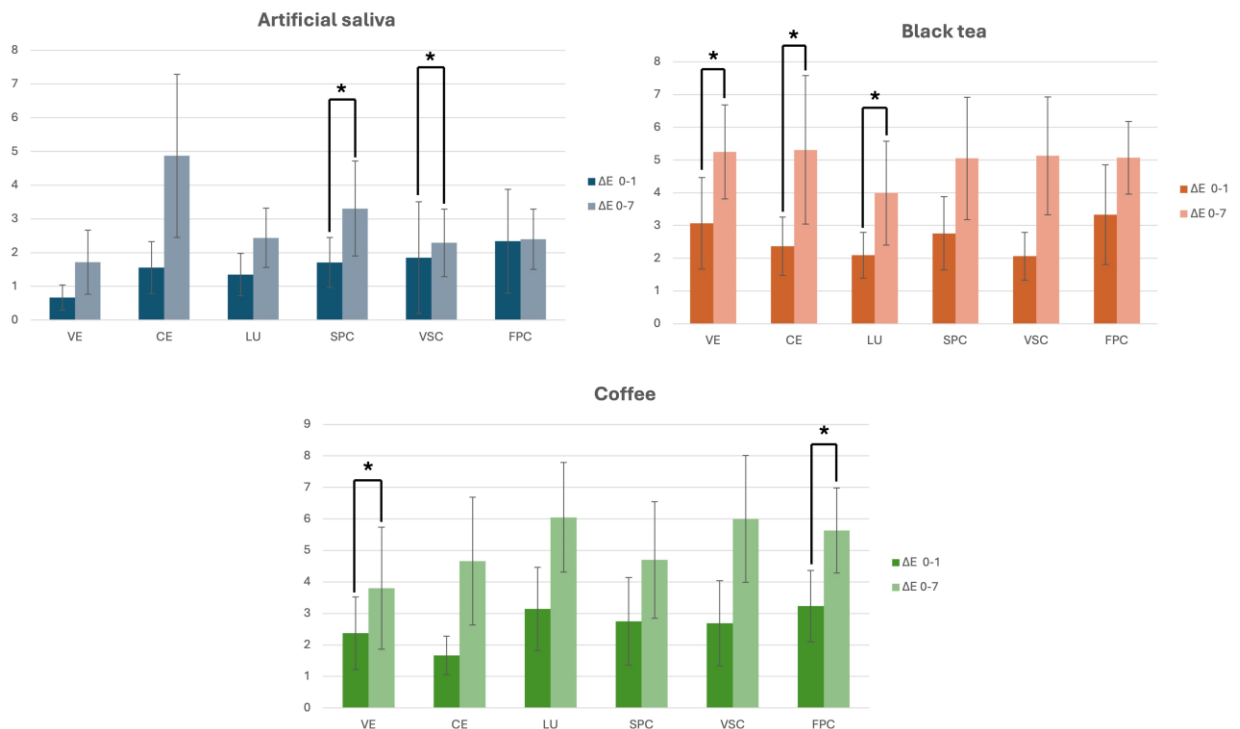


Figure 1. Comparison of ΔE values of material groups according to immersion time periods in the solutions. (VE: Vita Enamic, CE: Cerasmart, LU: Lava Ultimate, SPC: Saremco Print Crowntec, VSC: VarseoSmile Crown plus, FPC: Formlabs 3D Permanent crown) Mean \pm standard deviation (Mean \pm SD) of ΔE values. (* $p < 0.05$ indicate statistical differences between groups.)

DISCUSSION

This research compared the color stability of resins produced from permanent composites using digital techniques in different dye solutions in two different periods. The null hypothesis has been rejected in

accordance with this research's results. Significant distinctions have been found in terms of material and solution as well as storage duration.

Changes occur in the physical and chemical properties of resin composites and indirectly in their aesthetic properties due to exposure to food and beverages with coloring properties that are constantly consumed in the oral environment (42). One of the most important indications for the replacement of composite restorations after intraoral use is color change (19).

In dental material examinations, color evaluation by spectrophotometer is the most preferred and reliable method because it can obtain objective, fast and repeatable numerical data (43, 44). As a result, the spectrophotometer color-measuring device was used in this investigation to measure color.

For the numerical expression of color change, the Commission Internationale de l'Eclairage - CIE L* a* b* color system is used (18, 19). The L* a* b* values in this three-dimensional color system are known as "chromaticity coordinates" (26). ΔE is a numerical value that indicates the amount of color difference perceived between two objects and is calculated with the CIELAB formula (28). The data obtained from the study's measurements were recorded as CIE L*a*b* values and calculated using the formula as ΔE values.

O'Brien et al. evaluated the material as stable in terms of color difference if $\Delta E=0$, clinically imperceptible if $0.5 \leq \Delta E \leq 1$, clinically perceptible if $1 \leq \Delta E \leq 2$ and clinically unacceptable if $\Delta E > 3.7$ (46). In dentistry, $\Delta E \geq 3.7$ is often determined as the clinically perceivable threshold value for color difference (45, 46). In this study, the material groups immersed in all solutions used on the 1st day were colored within the clinically acceptable threshold value. Despite the composition of each material, it was found that when the storage period was prolonged, all color changes by the end of the 7th day exceeded the clinical limit in all solutions exception for artificial saliva. In addition, the color change values of all 3D printing groups except Group FPC in artificial saliva, all CAD/CAM groups (Group VE, Group CE and Group LU) in tea, and Group VE and Group FPC materials in coffee on day 7th were statistically significantly higher than those at day 1st.

Depending on the material, colorant type, and timing of storage, the degree of discoloration could differ. The color stability of nanofilled and micro-hybrid composite resins was evaluated by Erdemir et al. (46) and Al-Dharrab et al. (47) following immersion in various a beverage. They found that the material's color stability was influenced by the type of colorant used, how long it was stored, and the chemical structure of the composite resin (47, 48). These results are in line with the study's findings, which showed that colorants and storage duration significantly affected the shades of color.

When the studies evaluating the color stability of composite resins according to the beverage consumption habits of individuals in society are examined, it is seen that many different beverages are used as coloring solutions (30). In this study, coffee and tea solutions were used as coloring solutions due to their popularity and artificial saliva was used as a control. As a result, depending on the type of colorant, the

degree of staining was found to be higher in tea and coffee solutions than in artificial saliva in each material group. On a similar note, Almejrad et al (33) and Ardu et al (37) stated that artificial saliva was the less coloring agent in their studies in which resin-based dental materials were soaked in different solutions including tea, coffee and artificial saliva.

Group VE (CAD/CAM milling) was the least stained group on the 1st and 7th day of artificial saliva solution and the 7th day of coffee solution, while Group CE (CAD/CAM milling) was the least stained group on the 1st day of coffee solution. In general, the discoloration was greater in 3D printing materials (Group SPC, Group VSC and Group FPC) and their color stability wasn't as good as the materials used in CAD/CAM milling (Group VE, Group CE, and Group LU). Similar to our study, Shin et al. (35) investigated the degree of discoloration based on material type, colorant kinds (grape juice, coffee, curry), and colorant storage period for CAD/CAM blocks and 3D printing resins. It was noted that color change increased in all groups with increasing storage times. However, since color stability was found to be less in 3D materials compared to other groups, they stated that color change is a feature that should be taken into consideration when 3D printing resins are used (35). Similarly, Alharbi et al (17) evaluated the color differences of labial veneers produced using milling and 3D printing techniques in different immersion media (coffee, tea and artificial saliva) at 12 and 24 days. Because 3D-printed restorations showed are more susceptible to stains than milled restorations, it is advised that these materials should be used as temporary, short- or long-term restorations rather than permanent restorations (17).

The literature reports a variety of causes for the color instability of 3D printed resin. The surface microstructure of 3D printing has layers because it is based on the AM process (3,24,62). This situation, known as the "stair-step phenomenon", may be important when surface roughness values are considered for color stability (17, 22). The presence of micropores, residual monomers, and potentially incomplete polymerization at the layer interface could all increase the coloration potential of printed materials (17). Another reason may be related to water absorption capacity and solubility. A higher degree of water absorption leads to the absorption of coloring agents, resulting in discoloration of the material (33). Low color stability may also be caused by the 3D-printed composite resin materials' (Group SPC, Group VSC, and Group FPC) lower polymerization rates when compared to other materials (35). In the SM method; Group VE, Group CE and Group LU materials are obtained by milling from industrially produced blocks by polymerizing at high temperature and pressure. As a result, these materials have more compact structures and high polymerization rates (49). In contrast, even though 3D printing resins are applied post-curing to ensure polymerisation and reduce the amount of residual monomer, Shin et al. reported that the polymerisation rate was relatively low in their study (35). In addition to having an impact on biological processes and mechanical strength, a low polymerization rate can raise the risk of discoloration from

decreased surface integrity (23). Also, the presence of residual monomers causes deterioration of surface integrity due to surface softening as a result of hydrolysis (45).

The color and translucency stability of 3D materials for temporary crown and bridge restorations were investigated by Kim et al. (45) at various times following the procedure, including one hour, one day, one week, one month, and six months. They discovered that while the translucency of the 3D printable dental materials varied relatively little, the color stability of the materials altered considerably. After six months of water storage, they discovered that the materials generally got more darker, yellower, and opaque (45). In our study, we measured the samples only on the 1st and 7th day. One of the limitations of our investigation is that the coloration resulting from post-processing procedures could not be observed for a longer period.

The type, size, and quantity of inorganic particles incorporated into the structure of resin composites can influence how susceptible they are to coloration (16, 34). Inorganic particles on the surface may move away from the organic resin matrix during the clinical life of the material and cause the formation of a cavity in that area. As the number of these voids increases, surface irregularity increases (15).

Although the filler content of all 3D printing materials (SPC, VSC, FPC: 30-50%) is less than CAD/CAM milling materials (VE: 86%, CE: 71%, LU: 80%), the high size (SPC, VSC, FPC: 0.7 μ m) [VE: Silica (20nm), zirconia (4-11nm), CE: Silica (20 nm) and barium glass (300 nm), LU: Silica (20 nm) and zirconia (4-11 nm)] and irregular distribution of fillers can explain the reason for the high color variation of the 3D printed materials in this study. Since the filler particle sizes of CAD/CAM milling materials are smaller, they may have a lower degree of surface discoloration than other materials when separated from the surface. In parallel with the findings of our study, Vichi et al. (15) reported that composites with large inorganic particle sizes had more discoloration compared to composites containing smaller particles. We think that the color change obtained from 3D printing materials is due to the inorganic particle sizes and ratios of the composite.

In addition, it has also been reported that composites with smaller amounts of inorganic particles show increased water absorption and more discoloration due to the higher volume of the organic resin matrix (50). It has an association with the early development of 3D printed material technology, which is still limited in terms of presenting more monomer types and filler loads. The variation in monomer composition in the chemical structure can also be as influential as the organic matrix ratio. The structure of 3D printing materials includes methacrylate (Group VSC and Group FPC) and BisEMA (Group SPC). Because the Bis-EMA monomer doesn't have hydroxyl groups in contrast to the BisGMA monomer, it is less viscous and has a high fluidity, which makes it suitable for 3D printing. Prospective advancements in the types (such as UDMA or TEGDMA) and amounts of classical monomers employed in CAD/CAM composite resins signify a crucial area of research and development for 3D-printed dental materials (15).

When the material groups were analyzed based on solutions; in the artificial saliva solution, there was a difference between Group VE - Group CE, Group VE - Group SPC and Group VE - Group FPC on the 1st

day and between Group VE - Group CE and Group CE - Group FPC on the 7th day. In the tea solution, there was no difference in terms of color change between the materials on the 1st and 7th day. In the coffee solution, there was a difference between Group CE - Group LU and Group CE - Group FPC on day 1 and between Group VE - Group LU on day 7. In paired comparisons, there was nothing noticeable between the groups that were 3D printed. The reason for this may be that the chemical contents are close to each other based on the sharing of the companies. Furthermore, the resins used in this study's 3D printing were produced with specific 3D printers that were appropriate to the resins that the companies suggested.

Print orientation and thickness have an impact on the optical characteristics of dental restorative resins that are 3D printed (51). Espinar et al (16) evaluated the effect of build orientation (0°, 45°, and 90°) on different parameters, including color difference, of 3D-printed provisional resins. As a result of the study, they determined that the effect of changes in build orientation on the topography of 3D printed restorations was significant, but build orientations did not affect any of the variables examined (16). In this study, the parameters for 3D printing were set constant for all samples (Printing layer thickness: 50 µm, Printing orientation: 0°). The study of Espinar et al. which reported that printing direction did not make a difference in terms of color change, supports the use of only 0 degree printing angle in this study.

Limitations of this study include the fact that thermocycling, ultraviolet exposure, smoking and brushing regimes were not applied, which are among the factors that can affect color staining and surface properties. In addition, surface preparation methods, which are necessary to ensure long-lasting aesthetic results, have not been investigated.

Currently, there's not enough data to make a firm judgment about how well 3D-printed permanent restorative materials perform clinically (8). In-vitro studies of much longer duration than this study are needed to improve material development in restorative and prosthetic dentistry and to better understand the optical properties of 3D printed materials. It will be necessary to conduct a follow-up study to determine how various printing parameters and variables, such as light source or temperature variations, affect the color stability of 3D-printed resins during post-curing processes. By using 3D-printed crown and bridge materials, a thorough study in these areas will increase the predictability and dependability of the dental treatment procedure.

CONCLUSION

Within the limitations of this study; when compared to artificial saliva, the materials were considerably discolored by different colorants. It was observed that the color change was more as the storage time increased. The material groups immersed in all solutions used on the 1st day were colored within the clinically acceptable threshold value ($\Delta E \leq 3.7$). At the end of the 7th day, all color changes in all solutions,

except for the artificial saliva, were observed to go beyond the clinical limit. 3D permanent composite resins generally showed more staining than CAD/CAM milling composite resins.

Clinical impact

To maximize the clinical effectiveness of 3D-printed restorative dental resins, it is essential to understand their optical behavior. When 3D-printed resin restorations are utilized in clinics for aesthetic reasons, particularly in the anterior region, low color stability may make patients feel dissatisfied in terms of appearance. Therefore, while prescribing 3D-printed resins to patients, clinicians need to be concerned due to their limited color stability.

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Authorship contributions

Concept: NEB, ZS, CYC Design: NEB, ZS, CYC Data collection or processing: NEB, ZS, CYC, OA Analysis and interpretation: NEB, ZS, CYC, OA, MAK, Literature search: NEB, ZS, CYC, Writing: NEB, ZS, CYC.

Data availability statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Declaration of competing interest

There is no conflict of interest in this study.

Ethics

Since resources obtained from humans or animals were not used in this study, ethics committee approval was not obtained.

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