Investigation of Color and Microhardness Changes in Direct, Indirect, and 3D-Printed Composites

Direkt, İndirekt ve Üç Boyutlu Baskı Kompozitlerin Renk ve Mikrosertlik Değişimlerinin İncelenmesi

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ABSTRACT

INTRODUCTION: The aim of this study was to determine the surface hardness and color changes of resin composites produced for direct, indirect, and 3D printing applications.

MATERIAL and METHODS: Forty-five samples with a diameter of 9 mm and a thickness of 3 mm were prepared. After polymerization, the samples underwent ultrasonic cleaning, polishing, and coloring procedures. Microhardness measurements were taken using a Vickers microhardness device, and color change parameters were determined using a spectrophotometer. The data were statistically evaluated using ANOVA for repeated measurements.

RESULTS: Significant differences were found in microhardness measurements among the three different composite resin groups after initial, ultrasonic cleaning, polishing, and coloring procedures ($p < 0.05$). However, there was no significant difference in microhardness measurements between the initial and ultrasonic cleaning stages ($p > 0.05$). Microhardness values differed significantly after polishing and coloring ($p < 0.05$). Orange juice did not cause a significant color change (ΔE) in any of the restorative materials ($p > 0.05$), while significant color change (ΔE) was found in samples exposed to coffee after orange juice ($p < 0.05$).

CONCLUSION: The color and microhardness of all used composites were affected by polishing and coloring procedures, with varying effects observed for each material.

Keywords: *Microhardness, Color change, Nano-hybrid composite, Universal resin composite, 3D printing*

ÖZ

GİRİŞ: Direkt, indirekt ve 3 boyutlu baskı için üretilmiş likit rezin kompozitlerin yüzey sertliği ve renk değişiminin tespiti amaçlandı.

YÖNTEM ve GEREÇLER: 9 mm çap ve 3 mm kalınlıkta hazırlanan 45 örneğin polimerizasyonunu takiben örneklerin ultrasonik yıkama, polisaj ve renklendirme prosedürü sonrası mikrosertlik ölçümleri alındı. Mikrosertlik Vickers mikrosertlik cihazıyla renk değişimi parametresi ise spektrofotometre kullanılarak tespit edildi. Elde edilen veriler istatistiksel olarak tekrarlayan ölçümler için ANOVA, Pearson korelasyon analizi ile değerlendirildi.

BULGULAR: Üç farklı kompozit rezin grubunun başlangıç, ultrasonik yıkama, polisaj ve renklendirme prosedürü sonrası alınan mikrosertlik ölçümleri arasında anlamlı fark bulundu ($p <0,05$). Bu üç restorasyon materyalinin başlangıç ve ultrasonik yıkama sonrası mikrosertlik ölçümleri arasında anlamlı fark bulunmadı ($p >0,05$). Ancak polisaj ve renklendirme sonrası mikrosertlik değerleri birbirlerinden farklıydı ($p <0,05$). Restorasyon materyallerinde ortaya çıkan renk değişim miktarları ve mikrosertlik ölçümleri arasında korelasyon bulunmadı. Portakal suyunun tüm restorasyon materyallerinde anlamlı bir renk değişimine (ΔE) neden olmadığı ($p >0,05$), kahve ve portakal suyu sonrasında kahve uygulanan örneklerde anlamlı renk değişimi (ΔE) bulundu ($p <0,05$).

SONUÇ: Kullanılan tüm kompozitlerin renginin ve mikrosertliğinin polisaj ve renklendirme prosedüründen etkilendiği ve bu etkinin her materyalde farklı olduğu izlendi.

Anahtar Kelimeler: *Mikrosertlik, renk değişimi, nano hibrit kompozit, üniversal rezin kompozit, üç boyutlu baskı*

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INTRODUCTION

Dental resin composites, first developed by Rafael Bowen by combining dimethacrylates (epoxy resin and methacrylic acid) with a certain proportion of silanized quartz, are the most widely used aesthetic restorative materials.[1] The first produced and chemically polymerized resin composites were recommended for use in class III, IV, V cavities. A significant development for dental composites occurred in the 1970s with the development of light-polymerized resins, and many initial disadvantages were overcome with continuous research and developing technology.[2] Thus, resin composites, whose indications were initially limited to small and medium-sized cavities, can now be used in large and deep cavities of the posterior region thanks to the use of polymerization systems that enable the use of different techniques during the construction phase, structural and chemical changes in resin and inorganic particles.

In the past, the biggest obstacle to the use of resin composites in deep and large cavities was the shrinkage that occurs during their polymerization and the need to improve the mechanical properties of the material.[3] For this reason, the first generation of indirect resin composites was introduced by Touati and Mormann in the 1980s for use in posterior inlay and onlay restorations.[4] In this way, it has been possible to obtain long-lasting, durable, and aesthetic restorations with reduced polymerization shrinkage, which preserve the remaining tooth structure in teeth with high material loss. Despite their advantages, the use of these materials requires additional procedures and time, as well as an extra financial burden on the patient. Consequently, advancements in technology aim to streamline the application process for indirect inlay and onlay procedures, thereby minimizing both time and financial burdens on patients. For this purpose, traditional impression and production methods, which are the application procedures for indirect resin composites, have been replaced by computer-based "Computer Aided Design and Manufacturing" (CAD/CAM) design and production methods with the introduction of various products based on new technologies. Desktop intraoral scanners are needed for CAD/CAM, which means computer-aided design and computer-aided manufacturing at the patients' chairside. This facilitates the acquisition of more precise data and the design of virtual restorations using CAD software, which is then transferred to a manufacturing process.[5] Computer-aided manufacturing can be performed by subtractive methods using a milling unit, as well as by additive method, that is, three-dimensional printing, which has recently been introduced to the market. The first three-dimensional printers that produced with three-dimensional printing technology were able to produce using "Selective Laser Sintering" (SLS) and "Fused Deposition Modelling" (FDM) methods.[6] Over time, three-dimensional printers have evolved to enable the production of materials that are more resistant to the oral environment, long-lasting and aesthetic at the same time.[6] The first three-dimensional printers used plastic or metal as raw materials. Advancements in three-dimensional printing technology have led to the creation of printers capable of integrating epoxy resin-based composite-ceramic hybrid materials, which are polymerized using heat and light. Examples of systems that use a liquid photopolymer "hybrid resin" that can be polymerized with ultraviolet (UV) light include Digital Light Projection [Digital Light Processing (DLP)] and Stereolithography (SLA) printers. With the high accuracy and resolution of these systems, biocompatible restorations can be produced in a short time and in a way that is attractive to both patients and clinicians.[7]

A good and reliable aesthetic restorative material, whether produced by the direct or indirect method, should be able to mimic the appearance of natural teeth in addition to its strong structural properties. This is related to the color matching and color stability of the material. An esthetic restorative material with advanced chemical and mechanical properties should also be resistant to erosive conditions in the mouth. Dental erosion is one of the most common pathologies in today's lifestyle where acidic foods and beverages are frequently consumed.[8] There are many studies in the literature that reveal the link between dental erosion and acidic foods and beverages. In addition to erosive foods and beverages, coffee is one of the beverages that almost everyone consumes on a daily basis in today's lifestyle. As a coloring agent, coffee consumption is second after water consumption, with an annual consumption of more than 500 billion cups by people all over the world.[9,10] Not only the chewing forces in the mouth, but also erosive and coloring agents from food and beverages are a threat to a dental restorative material. Therefore, this study aimed to see how the surface microhardness and color stability of dental resin composites produced by different techniques are affected under erosive and staining threats. For this purpose, the null hypothesis of the study is that the surface microhardness and color stability of aesthetic dental resin composites produced by different techniques are not affected by orange juice and coffee consumption.

MATERIALS and METHODS

1. Preparation of Samples

In this study, 3 different types of resin composite materials were used (Table 1). Group A was a direct universal resin composite, while group B was an indirect nano-hybrid resin composite. Group C was a liquid resin composite (3D composite) produced for 3D printing. Resin composites in all groups were used in shade A2. The flowchart of the study is shown in Figure 1. A total of 45 samples were prepared, 15 in each group.

Table 1. Materials, properties, ingredients and manufacturing companies.

	Material Type	Material Form	Brand Name	Lot Number, Shade	Ingredients	Manufacturer
Group A	Direct Resin Composite	Universal Composite	3M Filtek Supreme Ultra Universal Restorative	9406537, A2	BIS-GMA, BIS-EMA6, UDMA, PEGDMA, Triethylene Glycol Dimethacrylate, Phenyl bis (2,4,6-trimethylbenzoyl), Phosphine Oxide, Silanized Ceramic, Zirconia Silanized Silica, Filler Volume: %60-80 +1-5	3M Canada Company
Group B	Indirect Resin Composite	Nano Hybride Composite	Gradia Plus Heavy Body Dentin	2302101,H B-DA2	%1–5: BIS-GMA, %5–10: TEGDMA % 1-5 UDMA:, Ceramic-filled inorganic fillers (%71 by weight), Prepolymerized fillers (%6 by weight), Filler volume %71+%6	GC Europe, Leuven, Belgium
Group C	Liquid Resin, (for 3D Printing)	Liquid Resin	Varseo Smile Crown Plus	A2 Dentin	4′-Isopropylidenediphenol, Ethoxylated and 2-Methylprop-2-enoic Acid, Silanized Dental Glass, Methyl Benzoylformate, Diphenyl (2,4,6-trimethylbenzoyl) Phosphine Oxide, Inorganic Fillers: 30-50% by weight (particle size 0,7 µm)	BEGO, Bremen Germany

1.1 Preparation of Resin Composite Samples in Groups A and B

The resin composite samples were placed in silicone molds with an inner diameter of 9 mm and a height of 3 mm with a 2 mm layering technique. For this purpose, a setup including a transparent celluloid strip and a 1 mm thick slide was used under and over the silicone mold. The distance between the samples and the light source was standardized by using a glass slide (1 mm thickness). The placed resin composite layers were polymerized over the slide with an LED light device Blue Phase (Ivoclar/Vivadent, Liechtenstein) with a light intensity of 1200 mW/cm^2 for 20 seconds in soft start program mode according to the manufacturer's recommendation. Unlike group A, resin composite samples in group B were cured in Autoflash (Bego, Germany) for 1500 pulses x 2 times following polymerization with an LED light unit. Then, the samples in both A and B groups were stored in distilled water at 37°C for 7 days to ensure post polymerization.

1.2 Preparation of Liquid Resin Composite Samples Used with Three Dimensional Printing Method

Group C samples were designed in a CAD (Computer Aided Design) program called Autodesk Fusion 360 (Autodesk Inc., USA) with an inner diameter of 9 mm and a height of 3 mm. The samples designed in this program were made ready for printing using Bego Cam Creator (Bego, Germany), a slicing software. The samples were then produced on a compact DLP (Digital Light Processing) 3D printer Varseo xs (Bego, Germany) using Varseo Crown Plus Dentin, a permanent liquid resin. Then, they were cured in autoflash (Bego, Germany) at 1500 pulses x 2 times. Subsequently, the samples were ultrasonically irrigated.

1.3 Ultrasonic Irrigation of Samples

Since the 3D resin composite produced should be subjected to ultrasonic irrigation as a production step, all samples used in the study were irrigated with isopropyl alcohol in ProWash S (Sprintray, USA) for 15 minutes to create standardization. All samples were then kept in an incubator at 37 °C for 7 days, moistened with distilled water.

1.4 Polishing of Samples

All groups were polished using the Shofu Super-Snap Rainbow Technique polishing kit (Shofu, Japan) without water cooling at 15,000 rpm for 30 seconds.

2. Microhardness measurement

In this research, all samples were kept at 37 °C for 7 days and the microhardness measurements were carried out at pre-polishing, post-ultrasonic irrigation, post-polishing, pre-staining and post-staining stages for samples in groups A and B, and at post-ultrasonic irrigation, post-polishing, pre-staining and post-staining stages for those in group C (Figure 1). Measurements were performed 3 times at 2 mm intervals along the diameter of the sample. A Shimadzu MCT-510 (Shimadzu, Japan) microhardness tester was used for the microhardness measurements. For the measurement, a Vickers diamond tip was applied to the material surfaces for 15 seconds with 100 g load. The "trace" obtained was then imaged with the help of a digital camera M 1192 (Carl Zeiss Jena, Germany) and transferred to the computer connected to the device. These images were measured and recorded as Vickers Hardness Values (VHV) using the "Kameram" program.

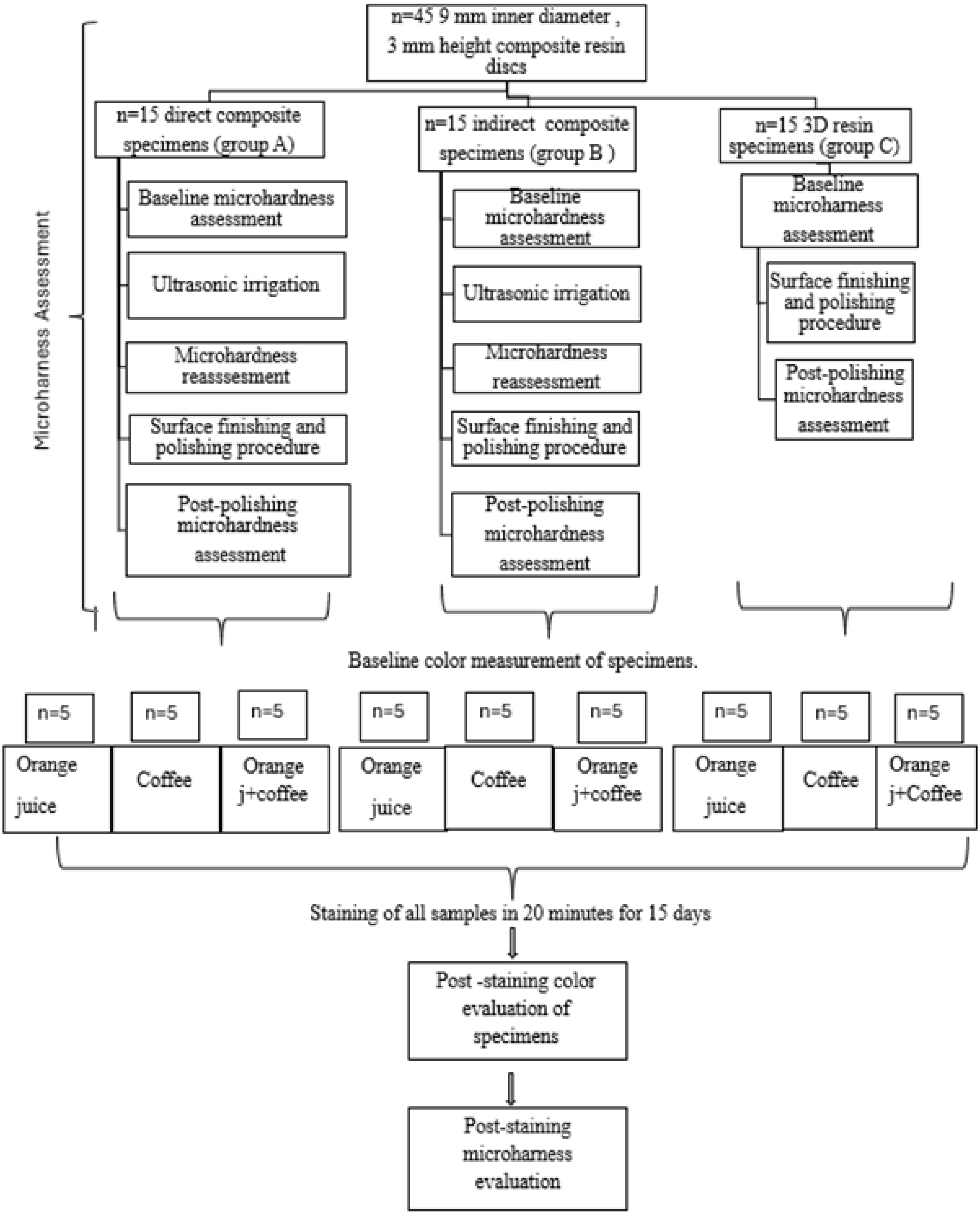

Figure 1. Flow Chart

3. Staining of Samples

A total of 45 samples were divided into groups of 5 and exposed to coffee and/or orange juice for 20 min per day for 15 days. The samples in group A1 were exposed only to orange juice (Cappy Coca-Cola Company, Istanbul, Turkey, pH 3.75), those in group A2 were exposed only to coffee, and those in group A3 were first exposed to orange juice for 20 min, then to coffee for 20 min after irrigating with distilled water. Similar solutions of 200 ml were prepared for groups B and C. The coffee solution was prepared by mixing 2 g of soluble coffee (Nescafe Classic, Nestle, Bursa, Turkey, pH 4.73) in 200 ml of hot water. A new solution was used every day. Distilled water was then used as a storage medium.

4. Color measurement

Initial color measurements of all samples were conducted after polishing, while final measurements were taken subsequent to staining. The color measurements of the samples were performed under D65 standard lighting conditions corresponding to daylight and the device was calibrated before each measurement. Measurements were performed on a standard white background (L=91.2, c=1.5, h=113.2) using VITA Easyshade® V (Vita Zahnfabrik, USA). The device was calibrated before each measurement and the average CIE L* c* h* value obtained from 3 measurements taken from 3 different points of the disc-shaped material was calculated using the CIEDE 2000 (Picture 1). ΔE values were calculated by using the formula. ΔL', ΔC' and ΔH' in the formula define the differences in lightness, chroma and hue between 2 different measurements. lightness (SL), color (SC), and tone (SH) weighting functions for color intensity and hue. RT is the turnover function describing the amount of interaction between color intensity and hue differences in the blue area in the CIE L*a*b color system. KL, KC and KH are parametric factors evaluated for brightness, color intensity and hue. In this study, KL, KC and KH were considered as "1".

5. Statistical analyses

Statistical analyses were performed using the SPSS program (version 27, IBM, Armonk, USA). The Kolmogorov-Smirnov test was used for normal distribution and Levene's test was used for homogeneity of variances. Comparison of the surface microhardness of different composites was tested by one way ANOVA on the mean data. In addition, the mean microhardness values obtained after 4 different steps (repeated measurements) were evaluated with two way ANOVA and post-hoc Tukey test by creating a generalized linear model. The correlation between microhardness data and Delta E data obtained as a result of staining was examined by Pearson correlation analysis. The statistical significance level was taken as "p<0.05". Power analysis G-power (version 3.0.1, University of Düsseldorf, Germany) was performed to calculate the sample size in the study with repeated measurements, and it was calculated that the study could be performed with a total of 27 samples with a minimum sample size of 3 for each group, an effect value (f) of 1.000173, a critical F value of 2.510157 and a power value of 98.537%. Based on these data, the study was completed with a total of 45 samples with a minimum number of 5 samples in each group with the same effect value and a critical F value of 2.2085181 and a power of 99.999%.

RESULTS

I. Microhardness

According to ANOVA for Repeated Measures, the microhardness measurements of the 3 different resin composite groups taken at baseline, after ultrasonic irrigation, after polishing and after the staining procedure indicated significant differences between the indirect, direct and 3D resin composite groups (p <0.05). However, there was no significant difference between the initial and post-ultrasonic irrigation microhardness measurements of the three different restoration materials used (p >0.05). However, the initial measurements and the microhardness values after polishing and staining were also different from each other (p <0.05). According to pairwise comparisons, these differences were observed for the direct resin composite group between coffee and orange juice in the staining procedure, which showed that orange juice reduced the microhardness more than coffee (p <0.05). Exposure to orange juice followed by coffee did not cause any additional change in microhardness (p >0.05). The microhardness changes of all three restoration materials compared to baseline were different for the three different treatments included in the staining procedure (orange juice, coffee, orange juice + coffee), (p <0.05). The most affected restoration material was the indirect resin composite for the orange juice and coffee groups and the 3D resin composite for the orange juice + coffee group (Figure 2). The percentage change of microhardness values caused by the staining procedure in the restoration materials is presented in (Table 3).

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)\right]^{1/2}$$

Picture 1. CIEDE 2000 (DE00) formula

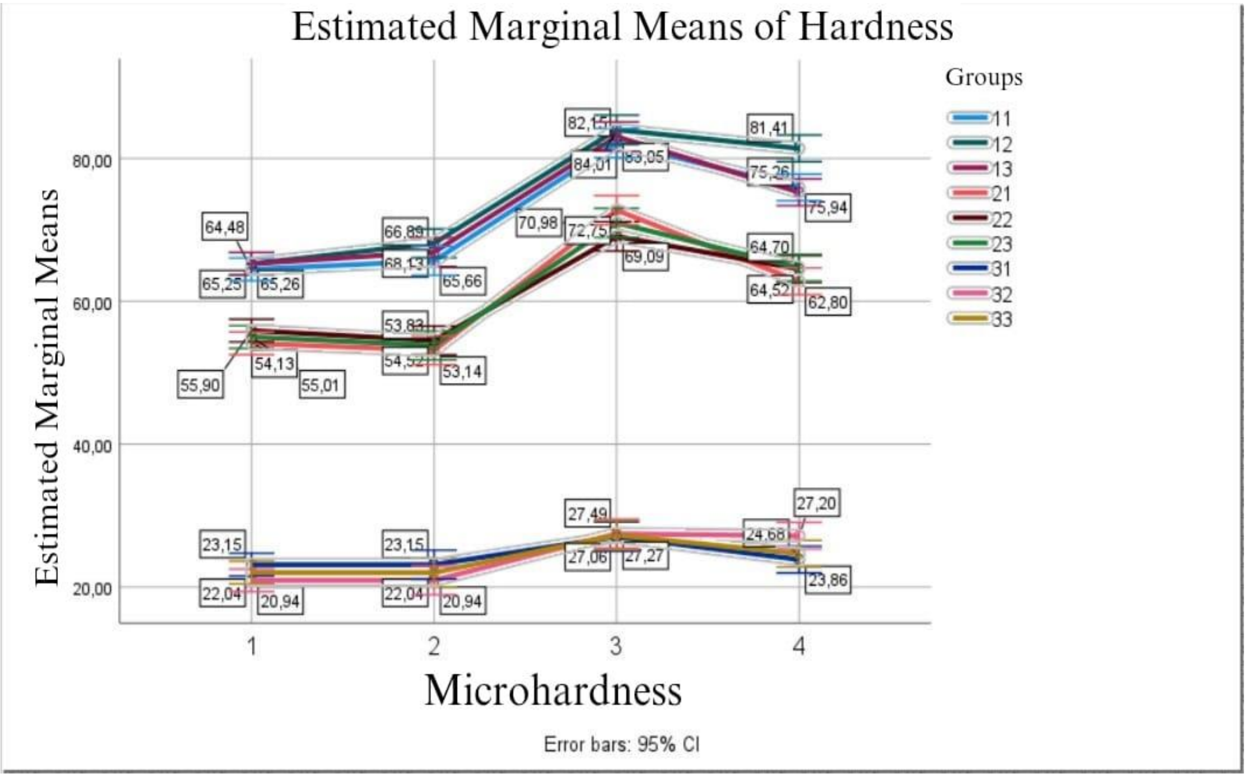

Figure 2. Microhardness values represent; Number 1 before the procedure, number 2 after ultrasonic irrigation, number 3 after polishing, number 4 after process. Groups: Group number 11 direct composite with orange juice, group number 12 direct composite with coffee, group number 13 direct composite with orange juice + coffee, group number 21 indirect composite with orange juice, group number 22 indirect composite with coffee, group number 23 indirect composite with orange juice + coffee, group number 31 3D resin with orange juice, group number 32 3D resin with coffee, group number 33 3D resin with orange juice + coffee

Table 2. Initial L-C-H Values (L mean, C mean, H mean), values after the staining procedure (L mean 2,C mean 2, H mean 2), values of shade change (ΔE).

	L (mean)	C (mean)	H (mean)	L (mean 2)	C (mean 2)	H (mean 2)	ΔE
Universal Composite							
Orange Juice	78,44	24,72	92,76	78,9	24,34	92,98	1,204
Coffee	77,56	23,5	93,53	73,74	27,33	90,54	6,21
Orange Juice and Coffee	78,22	24,42	93,06	73,88	26,90	89,68	6,19
Indirect Composite							
Orange Juice	80,14	41,68	87,34	80,6	42,3	87,74	0,924
Coffee	79,98	42,36	87,32	74,15	45,36	84,82	7,05
Orange Juice and Coffee	79,9	41,76	87,42	76,14	43,48	85,74	4,604
3D Liquid Resin							
Orange Juice	78,38	25,6	89,06	78	26,28	89,78	1.188
Coffee	78,66	25,44	89,1	74,12	27,88	88,52	5,278
Orange Juice and Coffee	78,48	25,98	89,1	73,7	28,02	87,88	5,518

II. Color change ΔE values

When the amount of color change in the restoration materials after the staining procedure was compared, it was observed that orange juice did not cause a significant color change (ΔE) in all restoration materials ($p > 0.05$), but there was a significant color change (ΔE) in all coffee-treated samples and orange juice+coffee samples ($p < 0.05$). Accordingly, the highest color change after coffee application was observed in indirect resin composite, followed by direct resin composite and 3D resin composite, respectively. In coffee application after orange juice, the highest color change was observed in direct resin composite, followed by 3D resin and indirect resin composite, respectively (Table 2).

Table 3. Microhardness changes before and after staining procedure.

	INITIAL	ULTRASONIC CLEANING	POLISHING	STAINING	CHANGE (%)
Universal Composite					
Orange Juice	64,47 ±3,12	65,66 ±4,10	82,15 ±1,16	75,94 ±1,91	7,55%
Coffee	65,25 ± 2,63	68,12 ±2,90	84,00 ± 3,35	81,40 ± 1,81	3,09%
Orange Juice and Coffee	65,26 ± 0,56	66,89 ±0,01	83,05 ±0,01	75,25 ±0,94	9,38%
Indirect Composite					
Orange Juice	54,12 ± 1,14	53,14 ±2,61	72,75 ±1,63	62,80 ±2,0	13,67%
Coffee	55,90 ± 0,47	54,52 ±1,76	69,08 ±4,95	64,51 ±3,69	6,61%
Orange Juice and Coffee	55,01 ±0,01	53,83 ±0,01	70,98 ±0,01	64,70 ±3,36	8,84%
3D Liquid Resin					
Orange Juice	23,15 ± 2,58	23,15 ± 2,58	27,06 ±1,71	23,85 ±0,84	11,85%
Coffee	20,94 ± 1,54	20,94 ±1,54	27,48 ±1,75	27,20 ±0,73	1,04%
Orange Juice and Coffee	22,04 ± 0,01	22,04 ±0,01	27,27 ±0,01	24,67 ±1,55	9,51%

DISCUSSION

This study investigated the surface hardness and color change parameters of direct, indirect, and 3D resin composites. The null hypothesis was rejected, as the polishing and staining procedures significantly influenced the color and microhardness of all composites, with variations observed among the materials. The surface hardness of a restorative material is an important factor related to the wear of the material, affecting its clinical success and providing resistance to plaque formation.[11] The Vickers microhardness apparatus was employed for microhardness assessment in this study owing to its benefits, including high precision, resistance to deformation of the diamond tip when utilized appropriately, prolonged usability, a single tip sufficing for all materials, complete adaptability to both soft and hard surfaces, and accurate measurement.[12] Visual assessment of the color stability of dental resin composite is often misleading because it is known that the human eye is closely related to the light source, gingival color, distance of the colored object from the eye, environmental factors, and the experience of the clinician trying to determine the color.[13] For precise, reliable and reproducible results, the use of a spectrophotometer was preferred in this study. The surface microhardness of the three different groups of resin composites used in this study was initially different from each other. This difference is thought to be due to the difference in the chemical properties of the materials and the type, size and proportion of inorganic filler. Indeed, Szalewski et al. concluded that the changes in the materials were related to the filler ratio and size in their study in which they examined the flexural strength and surface microhardness of these materials following staining and erosive procedures applied to different groups of nanohybrid and microhybrid resin composites.[14] The microhardness values of all resin composites examined in this study exhibited alterations from the initial values, irrespective of the staining procedure employed. Barve et al. also used staining and erosive beverages on two different types of nanofill and micro-hybrid composites noting a variation in microhardness across all experimental groups.[15] When the results of the aforementioned study and the results of this study are evaluated together, it is possible to say that the surface hardness of all resin composites subjected to staining and/or erosive procedures changes regardless of the composite type. The change in the microhardness of the material after the staining procedure is explained by the degradation of the silica matrix after the use of colorant and/or erosive beverage and the subsequent leakage of the colorant content between the organic matrix and the filler phase.[15] Yanıkoğlu et al. [16] found a significant difference between the surface hardness of the samples kept in a humid environment and the samples kept dry and found a decrease in the surface microhardness values of the samples kept in a humid environment as a result of water absorption and matrix degradation. In summary, the surface microhardness values of resin composites are related to the water absorption of the material and this change is reported to occur within the first 7 days.[17] The indirect resin composite exhibited the most significant alteration in microhardness among the material groups, attributed to its formation of prepolymerized inorganic filler and the hydrophilic TEGDMA (Triethylene glycol dimethacrylate) monomer within the organic matrix, making the material susceptible to degradation from erosive beverages. The polymerization rate of liquid resins produced for 3D printing is lower than that of conventional dental resin composites and there are more residual monomers on the surface of these composites after curing, which cause deterioration in the surface integrity of the material and softening on the surface.[18,19] As a matter of fact, in this study, the microhardness values of the 3D resin composite were found to be low compared to other composites. In the literature, phosphine oxides, which are frequently used as photoinitiators, are blamed for the low curing depth of liquid resin composites produced for 3D printing and the resulting excessive amount of residual monomer.[18] Ultrasonic irrigation is a necessity in the production stage of three-dimensional liquid resin composites. For this reason, the other composites used in this study were subjected to ultrasonic irrigation just like the 3D composites and their microhardness was measured. It was determined that polishing was the process that caused the microhardness change in all composite types. Similarly, there are results in the literature that the polishing process for resin composites increases the microhardness of the materials.[20,21] However, the material with the least microhardness difference caused by polishing is the 3D resin composite, which can be associated with the low inorganic content of the material. The increase in microhardness of the material post-polishing results from the elimination of the oxygen inhibition layer, which adversely affects the material's mechanical properties due to high resin content. [20] In the study, the microhardness values of all resin composite groups treated with orange juice showed a greater decrease compared to the groups treated with coffee. This difference may not only be related to the pH of orange juice, but also to the amount of titratable acid and chelation quality. The chelating agents in orange juice may be responsible for this change by interacting with the inorganic particles in the resin composite.[22,23] Another objective of this in vitro study was to determine the color stability of the materials following various staining procedures. When the amount of color change was compared, no statistically significant ΔE change was detected in all groups treated with orange juice, but the ΔE change was significant in all groups treated with coffee after orange juice+coffee. There was no correlation between the microhardness change caused by orange juice and color change. Likewise, studies in literature indicate no correlation between color change and variations in microhardness.[16.22] In this study, all samples underwent a daily 20-minute staining procedure for a duration of 15 days. Coffee producers report that consumers drink 2-3 cups of coffee daily, with each cup taking approximately 15 minutes to consume.[25] An in vitro application of 24 hours corresponds to an average of 1 month in the mouth. Within the limitations of this study, 300 minutes of in vitro coffee administration corresponds to an average of 6 days in the mouth. In order to ensure that the short-term application of staining and/or erosive agents was close to a procedure that could stimulate the oral environment, the samples were kept in a humidified environment with distilled water for 15 days, except for the 20-minute staining procedure. Abouelmagd et al [23], and Barve et al [15] also preferred distilled water as a storage medium in similar studies. In future studies, it would be appropriate to increase the duration of colorant and erosive beverage application. In the color measurements performed at the end of the staining procedure, it was found that 3D resin composites were less stained compared to direct and indirect composites. The assertion that the water absorption of 3D composites is elevated, leading to susceptibility to hydrolysis and structural degradation, as reported by Kim et al. in their study on the color stability of 3D resin composites, could be associated with the finding that the liquid absorption of these composites is both higher and more rapid.[19]

Due to this intense absorption, the liquid saturation of 3D composites increased and the diffusion of the colorant solution was limited, resulting in the material being less affected by the staining procedure. In this in vitro study, more color change was detected in coffee-treated samples compared to the other two application procedures. Roeder et al [24] reported that polar yellow pigments were the source of discoloration in coffee-treated samples in their study investigating the effect of various polishing systems on dental resin composites. It is known that there is a large amount of residual monomer on the surfaces of 3D resin composites.[18,19] As a result of the study, it was determined that the 3D resin composite material was more affected by coffee application after orange juice, unlike the indirect and direct composite groups. This effect is thought to be due to the lower resistance of the residual monomers on the surface of the 3D resin composite to the erosive effect. The color change after staining may be due to the silica-containing matrix and silane phase in addition to the phosphine oxide content in the universal direct resin composite group. Since the silane phase in the content allows liquid absorption, this type of composite may be more stained than 3D liquid resins. The reason why the indirect resin composite showed more discoloration after coffee application compared to the other groups may be due to the presence of camphoroquinone and TEGDMA (Triethylene glycol dimethacrylate), a hydrophilic monomer. On the other hand, the less coloration of the direct resin composite may be due to the predominance of hydrophobic monomers (BIS-EMA, zirconia, ceramic) in its structure.

Within the limits of the in vitro study;

- The microhardness values of all direct, indirect and 3D resin composites with different initial values were affected by the polishing process,
- The microhardness values of the resin composites were affected by the staining procedure and the maximum change in microhardness values was observed with the inclusion of orange juice in the procedure,
- In all resin composite groups, coffee caused the most color change and the material most affected by coffee was indirect resin composite,
- With the exception of the 3D resin composite, the coffee-on-orange juice procedure caused less discoloration than the coffee-only procedure,
- No correlation exists between the variation in microhardness and the color values of the materials.

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