

Original research article

The impact of adding nano zirconium dioxide fillers on color change, water sorption and solubility for denture liners

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ABSTRACT

OBJECTIVE: Zirconium dioxide (ZrO₂) nanofillers were added to denture liners to improve their physical properties. The main goal of this research was to evaluate the impact of adding nano zirconium oxide (ZrO₂) fillers on color change, water sorption and solubility of silicone and acrylic denture liners.

MATERIALS AND METHOD: The surface of ZrO₂ nanoparticles was modified via treatment with the silane coupling agent (3-aminopropyl) triethoxysilane. Fourier transform infrared spectroscopy analysis was performed to characterize surfaces untreated or treated with ZrO₂. After modification, these nanoparticles were incorporated into silicone and acrylic denture liners. Three subgroups were assigned for each test method. Kruskal-Wallis and Mann-Whitney U tests were used for the statistical analysis ($p \leq 0.05$).

RESULTS: Water sorption and solubility in water values decreased with the addition of modified ZrO₂ nanofiller in both test groups (respectively, acrylic-based tissue conditioner: $p = 0.040$ and $p = 0.020$; silicone-based denture liner: $p < 0.001$ and $p = 0.017$). The acrylic-based tissue conditioner test group with 1% modified ZrO₂ nanofiller showed the greatest color change ($\Delta E = 37.94 \pm 6.62$), whereas the silicone-based denture liner group with 0.5% modified ZrO₂ nanofiller presented the lowest ($\Delta E = 5.02 \pm 2.30$).

CONCLUSION: Modified ZrO₂ nanofiller-incorporated silicone-based denture liners might show better clinical success than acrylic ones according to the evaluated parameters.

KEYWORDS: Color; denture liners; Fourier transform infrared; nanoparticles; zirconium oxide

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INTRODUCTION

Denture liners is often preferred in prosthetic dentistry to reshape tissue surfaces of dentures in contact with the oral cavity.¹ They are used to repair inflamed mucosa, distribute the functional load equally on the prosthesis, and improve the fitting and retention of the denture.² The most frequent difficulties related to the clinical application of these materials are loss of resilience, hardening, water sorption, proneness to microbial adhesion and growth, color change, and separation from the denture base resin.^{3,4}

To overcome the problems associated with soft lining, various agents or fillers such as silver nanoparticles,⁵ photocatalysts,⁶ chlorhexidine,⁷ and seed oil,⁸ have been incorporated into these materials and their properties evaluated. Despite substantial research related to denture liners, few studies have addressed the addition of ZrO₂ nanofiller into these materials.^{9,10}

ZrO₂ nanofiller has high mechanical strength, superior biocompatibility, good surface and biological properties,¹¹ making it useful in the field of dental materials.¹² Therefore, when used as a filler in prosthetic bases, it improves mechanical properties.¹³

The current research aimed to investigate color change, water absorption, and solubility of an acrylic-

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based tissue conditioner and a silicone-based soft denture liner incorporating modified ZrO₂ nanofiller. The null hypothesis was that the incorporation of modified ZrO₂ nanofillers into denture liners would have no effect on the evaluated properties of these materials.

MATERIALS AND METHOD

Preparation of ZrO₂ nanofiller using silane coupling agent

Stabilized ZrO₂ with 3 mol % yttrium (Sigma-Aldrich, St. Louis, MO, USA) has a mean nanoparticle size of ≤100 nm and a specific surface area of 10–25 m²/g. The addition of a silane coupling agent (3-aminopropyltriethoxysilane, 99%, Sigma-Aldrich, St. Louis, MO, USA) to ZrO₂ nanoparticles results in bonding between the silanol groups of the silane and the hydroxyl groups on the nanoparticle surface. This process provides adequate bonding between the soft liner and the nanofillers, as well as the homogeneous distribution of these nanofillers in denture liners. The washing process of the experiment was carried out with toluene (≥99.7%, Sigma-Aldrich) as an organic solvent. The ZrO₂ nanofiller was modified using the following technique.

Pure toluene solvent in the amount of 5 mL and ZrO₂ nanofiller in the amount of 250 mg were placed into a glass beaker and ultrasonicated for 20 minutes. The beaker was placed on a magnetic stirrer (WiseStir MSH-20A, Daihan Scientific, South Korea, stirring speed 0–1,500 rpm). Then, 13.21 µL of silane was added by using a sterile syringe under a rapid stirrer. The slurry was left for two days with the beaker covered. After that, the slurry was subjected to vacuum at 60 °C in a rotary evaporator (Büchi Rotavapor R-210, Labortechnik AG, Flawil, Switzerland) with a rotation speed of 150 rpm for 30 minutes. Finally, the silanated ZrO₂ nanofiller was dried in a vacuum oven (Binder; vacuum drying model VD 53, Tuttlingen, Germany) for 20 h at 60 °C, then stored at room temperature (21 °C) before adding denture liners.

Application of Fourier transform infrared spectroscopy method

The formation of a chemical bond between the silane coupling agent and ZrO₂ nanofiller was evaluated using Fourier transform infrared (FTIR) analysis. For the FTIR measurements, ZrO₂ nanofiller was positioned on a Perkin-Elmer FTIR spectrometer (Nicolet iS5, Thermo Scientific, Madison, WI, USA) with an attenuated total reflectance crystal setup. Forty scans were recorded at a spectral range from 4500 to 450 cm⁻¹, with a resolution of 1 cm⁻¹.

Processing of test samples

Two structurally different materials were chosen for this study: a silicone-based soft liner [Ufi Gel P (UGP), VOCO GmbH, Cuxhaven, Germany] (lot no:1610000172) and an acrylic-based tissue conditioner [Visco-gel (VG),

Table 1. Tested denture liner material groups and their coding according to the added ZrO₂ amount

Group code	Denture liners
UGP 0	autopolymerized silicone based soft denture liner (control)
UGP 1	autopolymerized silicone based soft denture liner added with 0.5wt % ZrO ₂ nanofiller
UGP 2	autopolymerized silicone based soft denture liner added with 1wt % ZrO ₂ nanofiller
VG 0	autopolymerized acrylic based tissue conditioner (control)
VG 1	autopolymerized acrylic based tissue conditioner added with 0.5wt % ZrO ₂ nanofiller
VG 2	autopolymerized acrylic based tissue conditioner with 1wt % ZrO ₂ nanofiller

Dentsply DeTrey GmbH, Konstanz, Germany] (lot no:1645226). After ZrO₂ nanofillers were modified with a silane coupling agent, they were weighed and prepared in concentrations of 0.5% or 1% wt of both tissue conditioner and soft liner (Table 1). Three subgroups were created for each denture liner. The control group contained denture liners without nanofiller (UGP 0 and VG 0), while the prepared test groups contained 0.5 (UGP 1 and VG 1) and 1 wt % modified ZrO₂ nanofillers (UGP 2 and VG 2).

Hexane solvent (Sigma-Aldrich, St. Louis, MO, USA) and modified ZrO₂ nanofiller were placed into a glass beaker. This solution was ultrasonicated for 20 minutes to separate individual nanoparticles. After the catalyst of UGP test material was added to the solution, these mixtures were ultrasonicated for a further 20 minutes. The hexane was then evaporated from the mixtures at room temperature. The catalyst and modified ZrO₂ nanofiller were mixed with the base of UGP. The obtained mixture was prepared in a weight ratio of 1:1 for Group UGP according to the manufacturer's instructions.

Similarly, ZrO₂ nanofiller was modified with the aforementioned method. The liquid of VG test material and modified ZrO₂ nanofiller were mixed with the powder of VG at a 3:2.2 powder to liquid ratio.

Test methods

Water sorption and solubility test

The experiment was conducted according to the International Organization for Standardization (ISO)¹⁴ Standard No. 10139-2. Disc-formed samples (diameter: 50 ± 1 mm; thickness: 0.5 ± 0.1 mm) were prepared in a stainless steel standard mold. Sixty samples were prepared (n=10 in each subgroup). Firstly, the samples were placed in a desiccator containing freshly prepared silica gel (Sigma-Aldrich, St. Louis, MO, USA). This desiccator was stored in a thermostated oven (24 h at 37 °C). Whole test samples were exposed to the desorption process until they reached a constant mass. The weight of each sample (M_i) was measured digitally using an analytical balance (Dikomsan, Istanbul,

Turkey). After a constant mass was determined, the samples were dipped into distilled water at $37 \pm 1^\circ\text{C}$ throughout one week. Afterward, these samples were drawn out of the water and wiped with dry tissue paper to remove remaining moisture. They were then shaken in the air for 15 seconds. Following this, the weight of each sample was measured within one minute of removal from the water (M_2). Finally, all samples were kept until a constant mass was obtained in the desiccator as described, and weighed once more (M_3). Numerical data for water sorption and solubility (Wsp and Wsl, respectively) were attained based on the ratio of relevant mass differences to volume for each sample (V) using the following equations:

$$\text{Wsp } (\mu\text{g}/\text{mm}^3) = M_2 (\mu\text{g}) - M_3 (\mu\text{g}) / V (\text{mm}^3)$$

$$\text{Wsl } (\mu\text{g}/\text{mm}^3) = M_1 (\mu\text{g}) - M_3 (\mu\text{g}) / V (\text{mm}^3)$$

Color stability test

Sixty disc-shaped samples were prepared in the size of a 10 x 2 mm using Teflon mold. After the whole test samples were kept in distilled water at about 37°C throughout one day, color stability testing was carried out. A spectrophotometer (Specord 210 Plus, Analytik Jena, Japan) was used to assess color change. Color measurement for each sample was performed by the same examiner. CIELAB color system was used in the present study.

The color differences of the test samples were assessed by obtaining coordinate values via the formulae:

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

Sample size estimation

The current research has a factorial study design. The primary outcome of the study was to determine the differences in terms of any main outcome variable (i.e., color change, water sorption and solubility) among groups. A total sample size of 48 (8 per group) was required to detect at least a 0.50 effect size (Cohen's F) according to the interaction between materials and

concentration levels with a power of 85% at the 5% significance level. We decided to enroll 10 specimens for each sub-group. Sample size estimation was performed using G*Power (Franz Faul, Universität Kiel, Kiel, Germany) version 3.1.9.1 and, based on two-way analysis of variance (ANOVA) method. The effect size of 0.50 was considered on the basis of our clinical predictions.

Statistical methods

SPSS version 17.0 software (IBM, Armonk, NY, USA) was used for detailed data analysis. The Shapiro–Wilk test was performed to check all continuous variables for normality. The homogeneity of variances was evaluated using the Levene test. Data were represented as the median and interquartile range (IQR). A comparison of each evaluated test parameter was performed with the Mann–Whitney U test. The Kruskal–Wallis test compared the differences in continuous variables between more than two groups. When the p-values were at a significant level, the Kruskal–Wallis test was followed by Conover's method due to pairwise comparisons. If the P-value was less than 0.05, it was accepted as significant. However, the Bonferroni correction was applied for controlling Type I error for all possible multiple comparison.

RESULTS

FTIR analysis

Figure 1 shows the FTIR spectra of ZrO₂, silane, and ZrO₂ nanofiller with silane coupling agents. For the modified ZrO₂ nanofiller, a broad peak at 3,341 cm⁻¹ could correspond to the OH-stretching vibration onto the ZrO₂ surface. The two strong peaks at 2,980 and 2,885 cm⁻¹ may result from the stretching vibration of C–H in silane. The peaks seen at 1,457 and 1,388 cm⁻¹ represent the C–H bending vibration. A bending vibration related to the –NH₂ group of silane can be evidenced by the intensity of peak at 1,543 cm⁻¹. The presence of the Si–O–Zr bond stretching vibration is demonstrated by the appearance of a peak at 1,073 cm⁻¹. The peaks in these wavelengths might indicate the successful silanization of the particle surface; the peaks of silane and ZrO₂ on the surface are marked in black and red stars, respectively.

Water sorption and solubility test

According to the statistical results of water sorption, no significant differences were found between the UGP 0 and VG 0 groups ($p = 0.023$). The UGP 1 and 2 test groups showed statistically significant lower water sorption values than the VG 1 and 2 groups (respectively, $p = 0.015$; $p < 0.001$). In the VG groups, the adding of modified ZrO₂ nanofiller (0.5% and 1%) into denture liners significantly decreased the mean water sorption values of the test materials ($p < 0.001$ and $p < 0.001$). The UGP 2 test group showed significantly lower water sorption than the UGP 1 and UGP 0 test groups ($p < 0.001$ and $p < 0.001$). No statistically meaningful

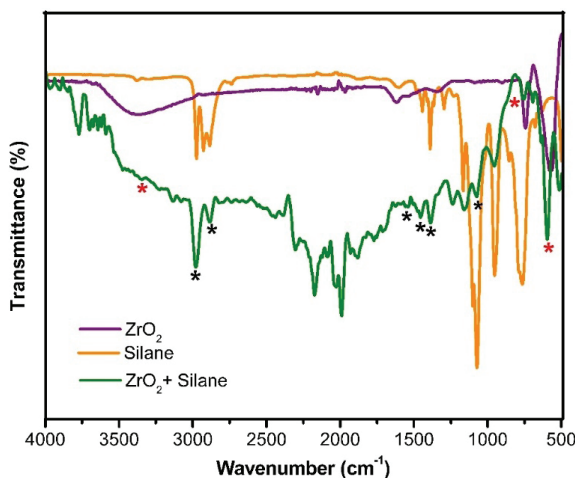


Figure 1. FTIR spectrum of ZrO₂ nanofiller and modified ZrO₂ nanofiller with silane coupling agent

difference was determined between the UGP 1 and UGP 0 test groups ($p = 0.850$). Among the samples with 1% modified ZrO_2 nanofiller, compared to VG, the UGP group showed lower statistically significant water sorption levels ($p = 0.002$) (Table 2).

Within the VG groups, a statistically significant difference was found in water solubility ($p = 0.002$). The VG 1 and VG 2 test groups had statistically lower values than the VG 0 group ($p < 0.001$ and $p = 0.007$). The VG 1 group showed the lowest water solubility values (9.88 ± 20.25). Likewise, the water solubility results for the UGP groups showed a statistically significant difference ($p = 0.017$). The water solubility values of the UGP 2 group were lower when compared with those of the UGP 1 and UGP 0 groups ($p < 0.001$; Table 3).

Color stability test

In both 0.5% and 1% modified ZrO_2 nanofiller added test samples, color change for the UGP subgroups was significantly lower than those for the VG group ($p < 0.001$). The color change for the VG 2 test group was higher than the VG 1 group ($p < 0.001$). However, in the statistical comparison of the UGP 1 and UGP 2 experimental groups, no meaningful difference was found in color change results ($p = 0.190$) (Table 4).

Table 2. Median values of water absorption for the test materials

	VG	UGP	P-value †
VG 0/ UGP 0	148.18 (69.30) ^{a,b}	-2.80 (40.71) ^b	0.023
VG 1/ UGP 1	17.45 (29.48) ^a	-5.09 (3.67) ^c	0.015
VG 2/ UGP 2	43.06 (25.78) ^b	-12.99 (3.31) ^{b,c}	<0.001
P-value ‡	0.004	<0.001	

†Comparison between VG 1,2 and UGP 1,2 groups showed the results to be statistically significant according to the Mann-Whitney U test and Bonferroni Correction ($p < 0.0167$), ‡Likewise, a comparison between VG 1,2 and UGP 1,2 groups showed the results were of statistical significance for $p < 0.025$ according to the Kruskal Wallis test and Bonferroni Correction

Table 3. Median values of water solubility for the test materials

	VG	UGP	P-value †
VG 0/ UGP 0	88.31 (41.85) ^{a,b}	-7.39 (58.65) ^b	0.063
VG 1/ UGP 1	9.88 (20.25) ^{a,c}	-11.52 (28.94) ^c	0.089
VG 2/ UGP 2	62.83 (11.75) ^{b,c}	-20.89 (7.44) ^{b,c}	0.002
P-value ‡	0.002	0.017	

Values followed by the same superscript are statistically significant [a: ($p < 0.001$), b: ($p < 0.01$), c: ($p < 0.001$)]

Table 4. Median values of colour changes for the test materials

	VG	UGP	P-value †
VG 1/ UGP 1	24.90 (7.21)	5.02 (2.30)	<0.001
VG 2/ UGP 2	37.94 (6.62)	5.81 (1.94)	<0.001
P-value ‡	<0.001	0.190	

According to Bonferroni correction, the results were accepted as statistically significant for $p < 0.025$

DISCUSSION

In the present study, modified ZrO_2 nanofiller was added into denture liners to enhance the physical features of the prosthetic liners. Surface functionalization of the ZrO_2 nanofiller with a silane agent provides better dispersion, eliminates clustering, and prevents interruption of the continuity of the polymer. The null hypothesis of our research, that the incorporation of modified ZrO_2 nanofiller into denture liners would not affect the evaluated parameters, was rejected. Water sorption and solubility values decreased with the addition of modified ZrO_2 nanofiller in both test groups. The VG 2 test groups showed the greatest color change, while UGP 1 presented the lowest.

The water sorption values of dental materials are related to their level of hydrophobicity and porous structure.^{15,16} In both groups with and without added ZrO_2 nanofiller, the UGP groups absorbed less water than the VG groups. The VG material is acrylic-based and hydrophilic, while the UGP test material is silicone-based and hydrophobic,¹⁷ so water sorption values of the UGP group can be expected to be lower. The presence of a crosslinking agent also reduced water sorption.¹⁵ The addition of modified ZrO_2 nanofillers to the test materials increased the hydrophobicity of these soft lining materials. Hayakawa *et al.*¹⁵ reported that a denture liner material containing a crosslinking agent exhibited less water sorption than a material without this agent. This finding is similar to the results of our study.

The UGP test groups showed less water sorption; therefore, less color change than the VG groups was observed. High water sorption in the VG test group may lead to the accumulation of colorants and further color change. The color results of our study are consistent with this opinion. Based on the evaluated data, the UGP test material is expected to have better durability, meaning that the clinical use for this material will be longer than VG. Negative water sorption values were obtained in all subgroups of the UGP group. If the amount of water absorbed is less than the leached-out components, negative water sorption values are obtained.¹⁸

Solubility refers to component loss during submersion. The elution of leachable ingredients occasionally irritates the tissue around dentures.^{15,16} The gradual release of plasticizers and monomer residues from the denture liner results in some clinical problems.¹⁶ The UGP 2 group showed the lowest water solubility values for all test materials. Given the results obtained from this study, the UGP 2 group can be expected to cause less irritation by the prosthesis to the tissue surface than other test materials.

The water solubility values of the UGP group were lower than those of the VG group. Silicone-based lining materials usually display lower water sorption and solubility than acrylic-based ones.¹⁹ Silicone-based lining materials contain dimethylsiloxane, which is

hydrophobic; therefore, the water sorption is low.¹⁷ In addition, due to the presence of a crosslinking agent in their composition, they do not contain plasticizer, which also reduces the water sorption and solubility quantity. Water sorption values for an ideal resilient liner should be low and they should not have soluble components.²⁰ According to the data obtained from our study, the water sorption and solubility values for all UGP subgroups met ISO standards.

Clinically acceptable water sorption and solubility thresholds of short-term tissue conditioner have not yet been established by an international standard. Due to the variety of sample sizes and the lack of a standardized protocol in the studies previously conducted, it would not be correct to compare the VG used in this study directly.

The water sorption and solubility values of the investigated samples in the VG and UGP groups were observed to decrease with the addition of ZrO₂ nanofiller. The lowest values were found in the VG 1 and UGP 2 groups. ZrO₂ nanofiller is chemically inert.²¹ When modified ZrO₂ nanofiller was added to the denture liner material, the surface energy of test materials was reduced, leading to reduced water sorption.

Kasuga *et al.*¹⁶ evaluated water sorption and solubility for two long-term experimental fluorinated resilient liner materials (containing dodecafluoroheptyl methacrylate or tridecafluorooctyl methacrylate) and an acrylic-based and a silicone-based prosthesis. In their study, the ranges of values for the water sorption and solubility for the test groups were from 3.75 µg/mm³ to 18.34 µg/mm³, and from 2.33 µg/mm³ to 6.13 µg/mm³, respectively. Our results for UGP water sorption and solubility values were lower than those reported by Kasuga *et al.*¹⁶ The reason for this difference in findings could be the polymerization method of test materials. In addition, Kasuga *et al.*¹⁶ added a fluorinated monomer to soft denture liners, whereas we added modified ZrO₂ nanofiller into denture liners. Adding different materials may have led to different results for water sorption.

An ideal prosthetic lining material should have high color stability.¹⁶ The effect of the ZrO₂ nanofiller addition on color change was significantly greater in the VG group than the UGP group. This can be related with the differences in the chemical content of these experimental groups. Acrylic denture liners exhibited lower color stability than silicone ones.²²

Maciel *et al.*²³ stated that the liquid content of VG is butyl phthalate butyl glycolate (86.9%), dibutyl phthalate (8.2%), and ethyl alcohol (solvent; 4.9%). Color stability may be related to the chemical content of the monomer.¹⁵ The UGP group contains dimethylsiloxane. The higher concentration of plasticizer and ethanol content in the VG test group and the easy release of ethanol are considered to be the reason for the greater color change than in the UGP group.

ΔE values were found to be higher than 3.7 in both test groups. The color change was clinically noticeable.

Studies on the discoloration of the denture liners are generally evaluated after immersion into a solution,^{4,5} beverage^{24,25} or artificial saliva.²² Therefore, the data found in our study cannot be directly compared with these studies.

Only a single brand of both healing and soft denture liners was tested in the present research. Therefore, it is not correct to relate the outcomes of this study directly to various prosthetic lining products. The color change, solubility, and water sorption results of the investigated liners may be different from those under clinical conditions. Because when these materials are used in the mouth, they are affected by many factors such as temperature and pH changes, masticatory force, and saliva. For these reasons, simulating clinical conditions is required to obtain more predictable results. Future *in vitro* and *in vivo* research should be performed for prosthetic liners with added modified ZrO₂ nanofillers to examine their physical properties.

CONCLUSION

To overcome the water sorption and solubility problems related to the use of denture liners, addition of modified ZrO₂ nanofiller might be a promising agent.

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Nano zirkonyum dioksit doldurucu ilavesinin protez astarlarının renk değişimi, su emilimi ve çözünürlüğüne etkisi

ÖZET

AMAÇ: Zirkonyum dioksit (ZrO₂) nanodoldurucuları, protez astarlarına fiziksel özelliklerini iyileştirmek amacıyla ilave edilmiştir. Bu çalışmanın amacı, ZrO₂ nanodoldurucu ilavesinin silikon ve akrilik protez astarlarının renk değişimi, su emilimi ve suda çözünürlüğü üzerindeki etkisini değerlendirmektir.

GEREÇ VE YÖNTEM: ZrO₂ nanopartiküllerinin yüzeyi, silan bağlama ajanı (3-aminopropil) trietoksilan ile işlemde geçirilerek modifiye edildi. Muamele edilmemiş ve ZrO₂ ile muamele edilmiş yüzeyleri karakterize etmek için Fourier dönüşümü kızılötesi spektroskopi analizi gerçekleştirildi. Modifikasyondan sonra, nanopartiküller silikon ve akrilik esaslı protez astarlarına ilave edildi. Her test yöntemi için üç alt grup oluşturuldu. İstatistiksel analizde, Kruskal-Wallis ve Mann-Whitney U testleri kullanıldı ($p \leq 0.05$).

BULGULAR: Her iki test grubunda da ZrO₂ nanodoldurucu ilave edilmesiyle su emilimi ve sudaki çözünürlük değerleri azaldı (sırasıyla akrilik esaslı doku düzenleyici: $p = 0.040$ ve $p = 0.020$; silikon esaslı protez astarı: $p < 0.001$ ve $p = 0.017$). %1 modifiye edilmiş ZrO₂ nanodoldurucu ilave edilmiş akrilik esaslı doku düzenleyici test grubu en fazla renk değişimini gösterirken ($\Delta E = 37.94 \pm 6.62$), %0.5 modifiye edilmiş ZrO₂ nanodoldurucu içeren silikon esaslı protez astarı en az renk değişimi gösterdi ($\Delta E = 5.02 \pm 2.30$).

SONUÇ: Modifiye edilmiş ZrO₂ nanodoldurucuların protez astarlarına ilavesi, bu malzemelerin fiziksel özelliklerini olumlu yönde etkilemiştir.

ANAHTAR KELİMELER: Renk; protez astarları; nanopartiküller; Fourier transform kızılötesi; zirkonyum oksit