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In Vitro Effect of Denture Cleansers on the Color Stability of Polyetheretherketone and Other Denture Base Polymers

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Efecto *in vitro* de los limpiadores de prótesis en la estabilidad del color de la polieteretercetona y otros polímeros base de prótesis

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ABSTRACT: The aim of this *in vitro* study was to compare the long-term effect of overnight use of denture cleansers with different chemical compositions on the color stability of denture base polymers (DBPs). The four DBPs evaluated were PEEK (PK group), thermoinjection-molded polyamide (PA group), auto-polymerized polymethylmethacrylate (PMMA) (AP group), and heat-polymerized resin PMMA (HP group). The cleaning agents used were Corega tablet (CT), Protefix tablet (PT), and 0.5% sodium hypochlorite (NaOCl) solution (SH). Distilled water (DW) served as a control. Forty-eight disc-shaped specimens (10mm × 2mm) were prepared from each DBP and randomly immersed in the different storage media (n=12 in each group). Color measurements of the specimens before and after immersion in the denture cleansers were made using a spectrophotometer (VITA Easyshade V). The color changes were measured and recorded in L* a* b*. After 120 d, the CIEDE2000 formula was used to calculate color changes (ΔE_{00}). For analysis of the data, a multivariate analysis of variance was used ($p < 0.05$). The results of the statistical analysis revealed significant color change differences in the DBPs immersed in the various denture cleansers ($p < 0.05$). Groups PA and PK showed the highest values for all solutions. AP-SH showed the highest ΔE_{00} value in group AP, no significant difference was found between other solutions. In group HP, there was no statistically significant difference between the average ΔE_{00} values of all solutions. Long-term daily use of the denture cleansers affected the color stability of all the DBPs, although the level of color change was acceptable. Laboratory and clinical studies on the color stability of PEEK are needed to confirm the results of this study.

KEYWORDS: Color stability; Denture base material; PEEK; PMMA; Polymer.

RESUMEN: El objetivo de este estudio *in vitro* fue comparar el efecto a largo plazo del uso nocturno de limpiadores de prótesis con diferentes composiciones químicas sobre la estabilidad del color de los polímeros base de prótesis (DBP). Los cuatro DBP evaluados fueron PEEK (grupo PK), poliamida moldeada por termoinyección (grupo PA), polimetilmetacrilato (PMMA) autopolimerizado (grupo AP) y resina PMMA termopolimerizada (grupo HP). Los agentes de limpieza utilizados fueron Corega (CT), Protefix (PT) y la solución de hipoclorito de sodio (NaOCl) al 0,5% (SH). El agua destilada (DW) sirvió de control. Se prepararon 48 especímenes en forma de disco (10mm × 2mm) de cada DBP y se sumergieron al azar en los diferentes medios de almacenamiento (n=12 en cada grupo). Las mediciones del color de las muestras antes y después de la inmersión en los limpiadores de dentaduras se realizaron con un espectrofotómetro (VITA Easyshade V). Los cambios de color se midieron y registraron en $L^* a^* b^*$. Después de 120 d, se utilizó la fórmula CIEDE2000 para calcular los cambios de color (ΔE_{00}). Para el análisis de los datos, se utilizó un análisis multivariante de la varianza ($p < 0,05$). Los resultados del análisis estadístico revelaron diferencias significativas en el cambio de color de los DBP sumergidos en los distintos limpiadores de dentaduras ($p < 0,05$). Los grupos PA y PK mostraron los valores más altos para todas las soluciones. AP-SH mostró el valor ΔE_{00} más alto en el grupo AP, no se encontraron diferencias significativas entre las demás soluciones. En el grupo HP, no hubo diferencias estadísticamente significativas entre los valores medios de ΔE_{00} de todas las soluciones. El uso diario a largo plazo de los limpiadores de dentaduras afectó a la estabilidad del color de todos los DBP, aunque el nivel de cambio de color fue aceptable. Se necesitan estudios de laboratorio y clínicos sobre la estabilidad del color del PEEK para confirmar los resultados de este estudio.

PALABRAS CLAVE: Estabilidad de color; Material de base para prótesis; PEEK; PMMA; Polímero.

INTRODUCTION

Polymethylmethacrylate (PMMA) and polyamides are frequently used in partial or complete removable dentures (1,2). Of the two, owing to its low density, esthetics, low cost, and ease of manipulation, PMMA has long been the preferred material in removable prostheses. However, PMMA also has disadvantages, such as water sorption, solubility, insufficient impact and bending strengths, residual monomer release, and polymerization shrinkage. Various efforts have been made

to overcome the disadvantages of PMMA-based composites, including the addition of epoxy resins, polyamide, or butadiene styrene (2).

The elasticity of polyamides is higher than that of heat-polymerized PMMA. High elasticity is required to ensure resistance to impacts and breakage. As compared to heat-polymerized PMMA, polyamides have various advantages, including reduced polymerization shrinkage and deformation. In terms of their toxicological safety, they can be used in patients with allergies to residual

monomers (3). However, polyamide materials also suffer from problems, such as water sorption, surface roughness, bacterial contamination, and discoloration. Furthermore, they are difficult to polish (4).

Due to the disadvantages associated with the use of metal frameworks in partial or complete removable prostheses, metal-free materials, including high-performance polymers, such as arylketone polymers, have been studied (5,6). A number of studies confirmed that polymer-based frameworks exhibited better esthetics (translucency and color), lower cost, higher elasticity, improved lightness than metal frameworks. Other reported advantages of polymer-based frameworks included ease of production and repair (4,5-8). Polymers were reported to be particularly advantageous in removable prostheses owing to issues relating to design control and repeatable and precise production potential using computer-assisted design (CAD)/computer assisted manufacturing (CAM) systems.

PEEK, a recently developed polymer belonging to the polyaryletherketone family, has mechanical and physical properties similar to those of bone and tooth hard tissues (6). This polymer has properties such as elimination of allergic reactions and metallic taste, polishability, low plaque affinity and good wear resistance (9). More research on the use of polymers in partial or complete removable dentures is needed.

Dental prosthesis hygiene and oral mucosal health are extremely important in implant-supported and tooth-supported removable prostheses to combat oral and systemic infections, especially in geriatric and immuno-compromised patients (1,10). Thus, dental prostheses must be cleaned properly on a regular basis. Cleaning with chemical solutions can contribute to oral health, prosthetic denture life, and overall quality of life. Denture cleaning agents are divided into different

groups according to their chemical compositions (i.e., enzymes, acids, hypochlorites, and peroxides) (1,11). Denture cleaning agents with different chemical compositions are widely used in the market to prevent microorganism colonization and plaque formation, especially *Candida* species, without changing the physical and mechanical properties of the prosthetic material (12). Ideally, denture base polymers (DBPs) should contain insoluble components and exhibit low water sorption, as these materials are exposed to saliva, food, water, and cleansers, resulting in water sorption and loss of soluble components. Over time, exposure to these factors results in surface erosion of the prosthetic material, with resulting effects on the color stability and esthetic appearance of the prosthesis (13).

Previous studies have evaluated the short-term use of high concentrations of sodium hypochlorite (NaOCl) as a denture cleaning agent (14,15). For denture cleansers used daily and long term, agents with low concentrations of NaOCl are needed (16,17). A number of studies have investigated the effect of cleansers with different NaOCl contents on DBPs in terms of their color stability, water sorption, hardness, roughness, and solubility (1,3,10-20). Most of these color stability studies used the CIELab formula (ΔE_{ab}) to determine color differences (1,13,20). However, a new formula, CIEDE2000 (ΔE_{00}), has been proposed to replace the ΔE_{ab} formula for calculating color differences from L^* a^* b^* values. According to a number of studies, ΔE_{00} values are superior to ΔE_{ab} values in terms of detecting small color differences, especially visual differences in high chroma colors (21-24). For this reason, we used the CIEDE2000 formula for calculating color changes in our study.

There are insufficient studies on the long-term effects of cleansers on the color stability and optical properties of PEEK polymers are insufficient. In a previous study, color stability in different colorant media was evaluated on PEEK, PMMA and

composite resin for a short time. Subsequently, the color change of the cleaning methods was evaluated (20). Another study evaluated the long-term effect of staining and cleaning solutions on color stability in polyoxymethylene and PMMA (25). The aim of this *in vitro* study was to compare the effects of long-term use of effervescent tablets, and 0.5% NaOCl solution on the color stability of PEEK, thermoinjection-molded polyamide, auto-polymerized, and heat-polymerized PMMA polymers. The null hypothesis was that the different cleansers would not affect the color stability of the DBPs.

MATERIALS AND METHODS

GROUP ALLOCATION

Forty-eight disc-shaped specimens (10mm×2mm) were prepared from each DBP (n=48) and randomly divided into four different storage media subgroups (n=12): Corega tablet [(CT), (Stafford-Miller Limited, Waterford, Ireland)], Protefix tablet [(PT), (Queisser Pharma, Flensburg, Germany)], 0.5% NaOCl solution [(SH), (Aklar Kimya, Ankara, Turkey)], and distilled water (DW) as a control. Table 1 provides information on the compositions of the materials and the manufacturers of the prosthetic materials and denture cleaning solutions used in the study.

In each of the AP, HP, and PA groups, 144 disc-shaped wax samples (Cavex Set Up Regular; Cavex, Haarlem, Netherlands) were created in

metal molds (thickness: 2mm; diameter: 10mm). In the AP group, the specimens were prepared in the powder liquid ratio recommended by the manufacturer. In the HP group, the specimens were polymerized under pressure in a hot water bath at 100°C for 45min. In the PA group, the specimens were melted at temperatures of between 270 and 288°C for 11min according to the manufacturer's recommendations and then injected into a muffle for 3min at 6 bar pressure for 30sec. After polymerization, all the specimens were kept in DW for 24h at 37°C for residual monomer elimination. Excess resin was trimmed using a hand piece with a tungsten steel bur at low speed. In the PK group, the specimens were designed using stereolithography (STL) and AutoCAD (Autodesk, San Rafael, CA, USA). The STL files were transferred to a CAD/CAM milling machine (Ceramill Motion 2; Amann Girrnbach AG, Koblach, Austria), and the specimens were milled from a PEEK dental disk (Juvora Dental Disc; Juvora, London, U.K.).

The surfaces of all the specimens were polished with 600-grit, 800-grit, and 1200-grit waterproof silicon carbide paper (Struers, Ballerup, Denmark) using a polishing device (Phoenix Beta; Buehler, IL, U.S.A.) under running water. They were then polished with a high-gloss agent (KMG; Candulor AG, Zurich, Switzerland). A digital caliper (Digimatic Caliper; Mitutoyo Corporation, Kanagawa, Japan) was used to ensure a uniform specimen thickness of 2mm. Subsequently, the specimens were cleaned ultrasonically for 10min and dried with a paper towel.

Table 1. Commercial names, compositions, and manufacturers of the materials used in the study.

Product	Code	Type and Components	Manufacturer
SR Triplex Hot	HP	Heat-polymerized PMMA	Ivoclar Vivadent AG., Schaan, Leichens- tein
SR Triplex Cold	AP	Auto-polymerized PMMA	Ivoclar Vivadent AG., Schaan, Leichens- tein
Deflex	PA	Injection molded polyamide	Nuxen SRL, Buenos Aires, Argentina
PEEK	PK	Unfilled PEEK CAD/CAM disc	Juvora Dental Disc; Juvora, London, UK
Corega Tablet	CT	Potassium Monopersulfate; Sodium Bicarbonate; Sodium Lauryl Sulfoacetate; Sodium Perborate Monohydrate; Sodium Polyphosphate	Stafford-Miller Limited, Waterford, Ireland
Protefix Tablet	PT	Sodium bicarbonate, Potassium caroate, Sodium perborate, Citric acid, Sodiumlau- rylsulfate, Aroma	Queisser Pharma, Flensburg, Germany
0.5% NaOCl Solution	SH	Sodium hypochlorite	Aklar Kimya, Ankara, Turkey

IMMERSION PROTOCOL

The specimens were immersed in four cleaning solutions: CT, PT, SH, and DW. The same procedure was applied for two effervescent cleanser tablets (Corega and Protefix) prepared according to the manufacturer's recommendations by adding one tablet to 200ml of warm tap water. All the specimens in each of the four groups (CT, PT, SH, and DW) group were stored in the respective solution for 8h to simulate overnight use. The solutions were renewed three times a day. At this point, the specimens were removed, washed in water, and returned to the storage media. This procedure was repeated for 120d to simulate 1 y of use. The same operator performed these steps at room temperature (i.e., between 23 and 24°C) to maintain standardization and avoid temperature variations, respectively.

COLOR MEASUREMENTS

Color measurements of the specimens before and after immersion in the various cleaning agents were obtained using a spectrophotometer (VITA Easyshade V; VitaZahnfabrik, Bad Säckin-

gen, Germany). The color measurements of each sample were made with the sample placed on the same white background. To obtain the average color value of a sample, three measurements were made at three different places in the middle area of each specimen at a right angle to the surface of the specimen. The average values were then recorded. The measurements were performed under D65 standard illumination conditions, calibrating each measurement according to the manufacturer's instructions. Color changes were measured and recorded in $L^* a^* b^*$. The color change (ΔE_{00}) was calculated using the CIEDE2000 formula.

To calculate the color change (ΔE_{00}) values, we used previously reported 50% perceptibility (1.72) and acceptability (4.08) threshold values for denture acrylic-based resins (22).

STATISTICAL ANALYSIS

The normality analysis of the data was performed using the Kolmogorov-Smirnov distribution test. The data were found to have a normal distribution. The data for ΔE_{00} values were statistically analyzed using a multivariate analysis of

variance, followed by multiple comparisons by a post hoc Tukey's test. The level of statistical significance was set at 0.05.

RESULTS

The statistical analysis revealed significant differences between the average $\Delta E00$ values. The mean $\Delta E00$ values of all the denture base specimens are presented in Table 2 ($p < 0.05$).

In group PA, CT ($1,81 \pm 0,28$) showed the lowest statistically $\Delta E00$ value among all cleansers. Group PA-SH ($3,28 \pm 0,64$) and group PA-DW ($2,71 \pm 0,79$) showed the highest statistically $\Delta E00$ value among SH and DW groups. PA-CT ($1,81 \pm 0,28$) showed the statistically lowest $\Delta E00$ value for the PA group. $\Delta E00$ values of all solutions in group PA were statistically higher than group HP.

In addition, all cleansers in group PA were statistically higher than group AP, except for PA-CT.

In group PK, DW (1.76 ± 0.51), which was the control group, showed the lowest $\Delta E00$ value statistically compared to denture cleansers. Group PK-CT ($2,70 \pm 0,50$) was the statistically highest $\Delta E00$ value among CT solutions. $\Delta E00$ values of all solutions in group PK were statistically higher than group HP like group PA.

In group AP, SH (2.47 ± 0.54) statistically the highest $\Delta E00$ values were found compared to other cleansers. In group HP, there was no statistically significant difference between the average $\Delta E00$ values of all solutions. In addition, $\Delta E00$ values of all solutions in group AP were higher than group HP, but there was only a statistically significant difference between the CT and SH groups.

Table 2. The $\Delta E00$ values of the DBPs after immersion in various denture cleansers ($p < 0.05$).

Groups	Mean \pm SD			
	DW	CT	PT	SH
AP	$1,59 \pm 0,43^{aA}$	$1,67 \pm 0,31^{aCB}$	$1,47 \pm 0,33^{aC}$	$2,47 \pm 0,54^{ab,ABC}$
HP	$1,15 \pm 0,20^{bc}$	$1,14 \pm 0,23^{abd}$	$1,23 \pm 0,30^{bd}$	$1,29 \pm 0,12^{bcd}$
PA	$2,71 \pm 0,79^{ab dA}$	$1,81 \pm 0,28^{bcABC}$	$2,64 \pm 0,85^{abB}$	$3,28 \pm 0,64^{bc cC}$
PK	$1,76 \pm 0,51^{cd ABC}$	$2,70 \pm 0,50^{cd cA}$	$2,53 \pm 0,72^{cd B}$	$2,61 \pm 0,54^{dc C}$

The same superscript lowercase letters indicate statistical differences within lines, and the same superscript uppercase letters indicate statistical differences within columns. SD: standard deviation.

DISCUSSION

In this study, significant differences were detected in the color stability of PK, PA, AP, and HP samples immersed in the different denture cleaning solutions. Consequently, the null hypothesis that the denture cleansers would have no effect on the color stability of the different DBPs was rejected.

Based on a detailed literature review, only a few studies appear to have focused on color changes in PEEK material when used as a denture

base. According to the results of the present study, comparison of the $\Delta E00$ values in the various denture cleanser groups revealed the lowest mean $\Delta E00$ value in the PK-DW group. Heimer *et al.* (20) examined the effect of different cleaning methods on the color stability of PK material under different environmental conditions over a 7-d period and reported that the color stability of PK was better than that of AP and composite resin. Unlike their study, we evaluated the impact of long-term immersion in denture cleansers on color stability. As shown by the statistical analysis, the highest

ΔE_{00} values were found in the PK group compared to the HP group for all cleansers ($p < 0.05$). Similarly, the ΔE_{00} values of all the cleansers were lower for the other PMMA resin (AP) compared to the PK group. However, only the CT and PT groups were statistically different when compared to AP. It is possible that the different translucencies and optical properties of the DBPs tested in the present study affected the color measurements.

Similar to PEEK, polyetherketoneketone (PEKK), a high-temperature polymer, is a member of the polyaryletherketone family (1). Ozyilmaz *et al.* (1) evaluated the effect of denture cleansers on long-term color stability and surface topography of PA, HP, and PEKK DBPs and reported changes in these parameters in all the DBPs. It was reported that alkali peroxide-based effervescent cleansers (CT, PT) affected the color stability of PA more than PEKK and HP (1). Durkan *et al.* (19) evaluated the effect of denture cleansers applied for 20 d on the surface roughness, hardness, and color stability of two polyamides (Valpast and Deflex), a butadiene styrene copolymer PMMA (Rodex), and a PMMA polymer (Paladent). They reported that immersion in the denture cleansers significantly affected the surface roughness and color stability of the polyamides (19). Although not evaluated in our study, it is known that polyamides are more susceptible to change in water absorption and surface roughness than PMMA (26). These disadvantages of polyamides are consistent with the fact that more color changes were seen in our study. In the present study, the higher ΔE_{00} values were found in the PA group compared to the PMMA groups (AP and HP).

AP acrylic resins have a lower degree of cross-linking than HP (27). AP acrylic resins show higher water absorption and solubility than HP resins (28). More residual monomer is observed in AP resins than HP resins (29). In addition, since AP resins contain amine accelerators, they exhibit higher solubility due to their oxidation (30). In our study, the fact that the AP group showed more

color change in all cleansers compared to HP is consistent with this information.

Earlier studies reported discoloration, loss of soluble components, and water sorption in acrylic resin materials immersed in denture cleansers (13,17-19). The length of time immersed in the denture cleansers significantly affected the color stability of the acrylic resin materials (13). Based on the ΔE_{00} values, we found statistically significant color changes in all the DBPs immersed in the denture cleaning agents for 1 y of simulated use. These color changes may be the result of leaking components of the coloring material. The opacity of acrylic resin after prolonged immersion may be caused by leakage or sorption of monomers. Alkaline peroxides, available in effervescent tablets or powder form, dissolve in water and react to form a hydrogen peroxide solution, resulting in the release of oxygen. Via mechanical action, the released oxygen removes deposits on the prosthesis (18). However, it is a known fact that cleaning tablets containing sodium perborate and sodium bicarbonate increase the surface roughness of polymer-based materials (31). The cleaning tablets are colored because the released oxygen causes surface roughness (10). Davi *et al.* (16) reported that cleaning with alkaline chemical solutions stimulated the release of plasticizers and changed the surface properties of polymer-based denture base resins. Peracini *et al.* (17) reported that denture cleansers should be used carefully due to their negative effects, such as discoloration, on the physical appearance of DBPs. Additionally, it has been shown that sodium hypochlorite has a bleaching effect depending on the usage time and concentration (16). The color stability of the DBPs immersed in various denture cleaning agents in the present study differed, with values below the 50% acceptability threshold (4.08) found for all the materials. According to the results of our study, which simulated 1 y of clinical use, the denture cleansers used caused clinically acceptable discoloration of the DBPs. Daily use of denture

cleansers can affect both the physical and mechanical properties of DBPs. Color stability is an indicator of aging or damage and affects the longevity of dental prostheses (13). Subsequent studies on PEEK need to be supported by investigations of the effects of other parameters, such as the surface topography, water sorption, and solubility.

The color stability of the restorations is highly proportional to their roughness (32). In addition, microbial adhesion and products in the oral environment can change the color stability (33). Ideal restorative materials should have low liquid absorption, good polishability and color stability as well as strong mechanical strength. In particular, its polishability increases color stability and extends the life of the restoration (20). It is difficult for dental technicians and dentists to determine an ideal polishing method due to the lack of precise guidelines for PEEK material. Data are missing for the most appropriate PEEK polishing method and its effect on surface roughness and surface free energy (34). Unfilled PEEK samples were used in this study. Modification of PEEK materials with carbon and glass fibers can reduce wear with chain structures and increase physical strength (35). The addition of ceramic fillers to the polymers results in a high degree of polishability, thereby reducing plaque adhesion and providing long-term color stability (36). Polishability and color stability enable the production of partial or complete removable prostheses that are more stable and satisfactory result (1).

The present study had a number of limitations. The samples were tested under conditions that may not reflect actual clinical conditions. The tests also did not fully simulate parameters in the oral environment, such as temperature, moisture, and denture biofilm, which may have implications for the color change measurements. Another limitation of this study was that the lack of translucency of PEEK material may also have influenced the color stability measurements.

CONCLUSION

Within the limitations of this *in vitro* study, we conclude that overnight use of denture cleaning solutions with different chemical compositions caused color changes in various types of DBPs, including PEEK, but these changes were within acceptable limits. To validate the results of this study, as well as laboratory studies, clinical studies are needed to investigate the effect of different denture cleansers on the color stability of PEEK.

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CONFLICTS OF INTEREST STATEMENT

No conflict of interest

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