

# Comparison of the Effect of Denture Cleansers on Long-Term Water Sorption and Solubility of Polyetheretherketone with other Denture Base Materials

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## ABSTRACT

**Objective:** In this in vitro study, the effect of three denture cleansers (DCs) after immersion in a chemical solution applied to polyetheretherketone (PEEK) and other denture base materials (DBMs) on long-term water sorption and solubility was compared.

**Methods:** Disk-shaped specimens (50±1.0-mm diameter and 0.5±0.1-mm thickness) were prepared from four DBMs (n=48). All specimens were randomly subdivided into four storage media groups (n=12): Corega tablet (CT), Protefix tablet (PT), 1% sodium hypochlorite (NaOCl) solution (SH), and control (distilled water, DW). Storage media were renewed thrice a day for 120 days and simulated for 1-year use of overnight immersion. Water sorption and solubility ( $\mu\text{g}/\text{mm}^3$ ) of DBMs before and after immersion in storage media were examined and obtained data were statistically analyzed using a multivariate analysis of variance, followed by multiple comparisons by a posthoc Tukey's test ( $p < 0.05$ ).

**Results:** From statistical analysis, the effect of different DCs on the water sorption and solubility of DBMs revealed a statistical difference ( $p < 0.05$ ). The PEEK group exhibited a statistical difference in mean water sorption values among all cleanser groups ( $p < 0.05$ ). For the PEEK group, a statistical difference was observed in the DW group among SH and CT groups in terms of the mean solubility values ( $p < 0.05$ ), while a statistically significant difference was not observed in the PT group among SH and CT groups ( $p > 0.05$ ).

**Conclusion:** DCs affect PEEK and other DBMs in terms of water sorption and solubility in the long-term follow-up.

**Keywords:** Denture base material, denture cleanser, PEEK, solubility, water sorption

## 1. INTRODUCTION

Owing to the increase in the life expectancy and number of elderly individuals, implant or tooth-supported removable prostheses are used in dentistry as an alternative to fixed prosthetic restorations (1,2). There is a 13–29% incidence in partial or complete removable prostheses among adults (2). Polymers such as polymethyl methacrylate (PMMA), polyamide, and polyetheretherketone (PEEK) can be used in removable prostheses (3,4). Owing to its low density, aesthetics, cost-effectiveness, and facile manipulation, PMMA has been used in removable prostheses for a long time. However, its water sorption, solubility, impact and bending strength, residual monomer, and polymerization shrinkage still need to be improved (4). On account of these disadvantages, high elasticity polyamides have been widely used due to their high impact strength, reduction of polymerization shrinkage and associated deformation, and absence of residual monomers. However, this material

has been reported to exhibit various issues such as water sorption, surface roughness, bacterial contamination, discoloration, and difficulty in polishing (5,6).

Biocompatible metals such as cobalt-chromium or titanium are preferred framework materials in removable prostheses (1). However, even titanium, which is known to be corrosion resistant, can compromise their biocompatibility as it causes galvanic corrosion with the combination of different metals in the oral environment in the case of polymetallism (7-9). In addition to the risks of hypersensitivity and corrosion, other disadvantages of metal-framework removable prostheses include aesthetic problems with a metal appearance, adverse tissue reactions, loss of abutment teeth, and biofilm production. In addition, it is difficult and expensive to produce metal frameworks in removable prostheses by using computer-aided design/computer-aided manufacturing (CAD-CAM) systems (1,9-11).

Owing to the disadvantages of metal frameworks in removable prostheses, the development of a high-performance polymer was investigated using metal-free materials, such as PEEK, for application in removable prostheses (1,10). Compared to metal frameworks, polymer-based frameworks exhibit advantages such as better aesthetics (translucency and color), lower cost, higher elasticity, light-weight nature, lower water sorption and solubility, and ease of manufacturing and repair (1,6,9-12). Particularly, polymers exhibit significant advantages in removable prosthetic treatment due to their design control, manufacturing flexibility, and reproducible precision potential with CAD-CAM systems. In addition, mechanical and physical properties of PEEK are similar to those of bone and tooth hard tissues, thereby permitting the more stable and atraumatic production of removable prostheses. In addition, PEEK polymer is resistant to heat and can offer denture disinfection with autoclave (1,3,10). However, additional studies are needed for the application of PEEK in removable prostheses.

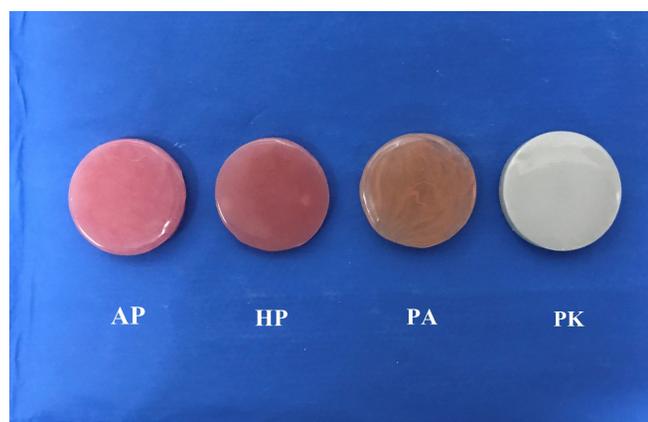
It is key to ensure prosthetic hygiene and protect the health of the oral mucosa of implant or tooth-supported removable denture users. Otherwise, oral and systemic infections may develop in these individuals (3,13). Hence, complete or partial removable dentures should be cleaned regularly and efficiently to prevent oral and systemic diseases. Denture cleaning with chemical solutions is an important factor that contributes to the oral health, prosthesis lifespan, and overall quality of patients. Chemical denture cleansers are categorized into different groups according to their chemical compounds such as alkali peroxides, acids, enzymes, and alkali hypochlorites (3,14). Ideally, chemical DCs with different contents in the market should be effective for discoloration and biofilm production without causing the physical and mechanical damage of denture base materials (DBMs) (15). In an ideal case, DBMs should contain insoluble components and exhibit low water sorption. However, these materials may be exposed to saliva, food, water, and cleansers throughout their lifetime, leading to water sorption and loss of other soluble components. In addition, these factors may affect the color stability of DBMs and may not satisfy the aesthetic expectation of patients (16).

DBMs have been evaluated in terms of color stability, surface roughness, hardness, water sorption, and solubility (3,5,12-17). However, these studies that compare the effect of DCs, PEEK, and other DBMs in terms of water sorption and solubility are not available. In only one study, Liebermann et al. (17) have evaluated the effect of different aging regimes/times of sodium chloride, artificial saliva, physiological saliva, and distilled water in the range of 1–180 days on different CAD/CAM polymers based on PEEK, hybrid and nanohybrid composite resins, and PMMA in terms of surface roughness, water sorption, solubility, Martens hardness, and indentation modulus. In this study, storage media did not exhibit any effect on the surface roughness and water sorption, and water sorption significantly increased with the storage period. In addition, PEEK exhibited the lowest water sorption and solubility values.

The aim of this in vitro study was to compare the effect of PEEK and other DBMs on water sorption and solubility after immersion three cleansers storage media. The null hypothesis was that different cleansers will not affect the water sorption and solubility of the DBMs.

## 2. METHODS

In this study, the effect of three DCs on the water sorption and solubility of PEEK (PK group), injection-molded polyamide (PA group), auto-polymerized (AP group), and heat-polymerized resin PMMA (HP group) was examined (Figure 1). Forty-eight disc-shaped specimens ( $50\pm 1.0$ -mm diameter and  $0.5\pm 0.1$ -mm thickness) were prepared from each DBM group ( $n=48$ ) according to EN ISO 20795 and randomly subdivided into four storage media groups ( $n=12$ ): Corega tablet (CT), Protefix tablet (PT), 1% sodium hypochlorite (NaOCl) solution (SH), and control (distilled water, DW). Table 1 lists the material type, composition, and manufacturer for the materials and solutions used herein.



**Figure 1.** Denture base material specimens. AP: Auto-polymerized, HP: heat-polymerized resin polymethyl methacrylate (PMMA), PA: Injection-molded polyamide, PK: Polyetheretherketone (PEEK)

In PA, AP, and HP groups, 144 disc-shaped stainless-steel mold specimens ( $50\pm 1.0$ -mm diameter and  $0.5\pm 0.1$ -mm thickness) were prepared by a CNC device (Takisawa Machine Tool Co., Okayama, Japan) for the preparation of DBM specimens. AP and HP group specimens were prepared according to the manufacturer's recommendations. Group HP specimens were polymerized under pressure in a hot water bath at  $100^{\circ}\text{C}$  for 20 min. Group PA specimens were prepared in accordance with the manufacturer's recommendations by using a micro-injection molding system at  $280^{\circ}\text{C}$  for 15 min. After polymerization, the specimens were stored in distilled water at  $37^{\circ}\text{C}$  for 24 h for the elimination of the residual monomer. The excess base resin was trimmed using a tungsten steel bur using a hand piece at a low speed.

PK specimens were designed in the stereolithography (STL) format using AutoCAD software (Autodesk, San Rafael, CA, USA). STL files were transferred to a CAD/CAM milling machine (Ceramill Motion 2, Amann Girrbach AG, Koblach,

Austria), and the specimens were milled from a PEEK dental disk (Juvora Dental Disc; Juvora, London, UK).

All specimens were polished using 600-grit, 800-grit, and 1200-grit waterproof silicon carbide paper by using a polishing device (EcoMet 30; Buehler Ltd., Lake Bluff, IL, USA). They were then polished with a high gloss agent (KMG; Candulor AG, Zurich, Switzerland). A digital caliper (IP54 Digital caliper, SHAN, Columbus, OH, USA) was utilized to ensure a uniform specimen sizes (50-mm diameter and 0.5-mm thickness). The specimens were ultrasonically cleaned for 10 min and dried with a paper towel. All specimens were thermally cycled for 5000 cycles between 5°C and 55°C with a 20-s dwell time and a 10-s transfer time from one bath to another bath (Thermocycler THE 1100; SD Mechatronik Feldkirchen-Westerham, Germany).

After thermal cycling, the same procedure was applied for two effervescent cleanser tablets (Corega and Protefix, respectively) using cleanser solutions prepared according to manufacturer's recommendations by adding one tablet to 200 mL of warm tap water. All specimens were placed in storage environments to simulate 8 h of overnight use. The solutions were renewed thrice a day, and the specimens were washed each time and placed back in the storage media. This procedure was repeated for 120 days to simulate 1 year of use. All experimental processes were performed by the same operator to maintain standardized operations.

Weight measurements were performed using a precision scale (XB 220A; Precisa, Zurich, Switzerland) with an accuracy of 0.1 mg until a constant mass value was obtained on the weight scale screen before immersion and recorded as " $m_1$ " for each specimen. After 120 days of immersion, the specimens were washed with distilled water, gently dried, and the second immersion measurements were performed, and the weight after water sorption were recorded as " $m_2$ ." The thickness and diameter of all specimens were measured with a digital caliper with an accuracy of 0.01 mm. All specimens were dried in a desiccator with silica gel at 37°C for 24 h and weighed again ( $m_3$ ).  $V$  is the volume of the specimens in  $\text{mm}^3$ . The water sorption ( $W_{sp}$ ) and solubility ( $W_{st}$ ) values obtained in  $\mu\text{g}/\text{mm}^3$  were calculated for each specimen by the following formulas:

$$\text{Water sorption: } (\Delta W_{sp}) = (m_2 - m_1) / V$$

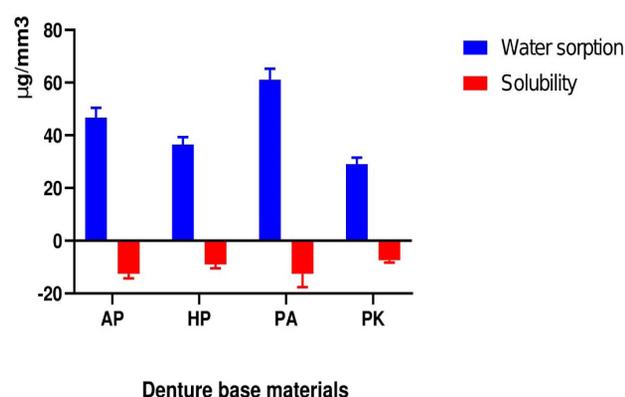
$$\text{Solubility: } (\Delta W_{st}) = (m_3 - m_1) / V$$

## 2.1. Statistical Analysis

Data were evaluated using a statistical software program (IBM SPSS Statistics, v20.0; IBM Corp). Normality analysis of the data was performed by the Kolmogorov–Smirnov distribution test. Data exhibited a normal distribution. Data for water sorption and solubility values were statistically analyzed using a multivariate analysis of variance, followed by multiple comparisons by a post-hoc Tukey's test. The statistical significance level was set at 0.05.

## 3. RESULTS

Tables 1 and 3 list the water sorption and solubility values of all DBM specimens in all cleanser baths, respectively (Figure 2). As a result of the two-way analysis of variance performed according to the obtained data, the effect of different DCs on the water sorption and solubility of the DBMs was statistically significant ( $p < 0.05$ ). For the AP group, there was a statistically significant difference in water sorption values among DW, CT, and PT groups ( $p < 0.05$ ). For the HP group, a statistically significant difference in the mean water sorption values among all cleanser groups was observed ( $p < 0.05$ ). For the PA group, a statistically significant difference in the mean water sorption values between all cleanser groups ( $p < 0.05$ ) was observed. For the PK group, a statistically significant difference in mean water sorption values between all cleanser groups ( $p < 0.05$ ) was observed.



**Figure 2.** Mean values of water sorption and solubility of the tested materials in  $\mu\text{g}/\text{mm}^3$ .

AP and PA groups showed higher mean solubility values in cleanser baths. The PK group showed higher mean solubility values than the other DBM groups ( $p < 0.05$ ).

**Table 1.** Product names, manufacturers, composition properties of test materials, and procedures used in this study.

Product	Type	Manufacturer
SR Triplex Hot	Heat-polymerized PMMA	Ivoclar Vivadent AG., Schaan, Leichenstein
SR Triplex Cold	Auto-polymerized PMMA	Ivoclar Vivadent AG., Schaan, Leichenstein
Deflex	Injection molded polyamide	Nuxen SRL, Buenos Aires, Argentina
PEEK	Unfilled PEEK CAD/CAM disc	Juvora Dental Disc; Juvora, London, UK
Corega	Potassium Monopersulfate; Sodium Bicarbonate; Sodium Lauryl Sulfoacetate; Sodium Perborate Monohydrate; Sodium Polyphosphate	Stafford-Miller Limited, Waterford, Ireland
Protefix	Sodium bicarbonate, Potassium caroate, Sodium perborate, Citric acid, Sodium laurylsulfate, Aroma	Queisser Pharma, Flensburg, Germany
1% NaOCl	Sodium hypochlorite	Aklar Kimya, Ankara, Turkey

PMMA: Polymethyl methacrylate, PEEK: Polyetheretherketone, CAD/CAM: Computer-aided design/computer-aided manufacturing, NaOCl: Sodium hypochlorite.

**Table 2.** Mean water sorption values ( $\mu\text{g}/\text{mm}^3$ ) of all specimens in different denture cleansers.

Groups	Mean $\pm$ SD			
	DW	CT	PT	SH
AP	26.08 $\pm$ 3.12	57.23 $\pm$ 4.72 <sup>A</sup>	53.03 $\pm$ 4.08 <sup>AB</sup>	26.35 $\pm$ 3.20 <sup>BC</sup>
HP	24.74 $\pm$ 3.12	42.58 $\pm$ 2.74 <sup>aA</sup>	31.70 $\pm$ 2.18 <sup>aAB</sup>	47.31 $\pm$ 3.13 <sup>aABC</sup>
PA	63.07 $\pm$ 5.19 <sup>ab</sup>	85.37 $\pm$ 3.93 <sup>abA</sup>	43.49 $\pm$ 4.51 <sup>abAB</sup>	52.62 $\pm$ 3.14 <sup>abABC</sup>
PK	27.09 $\pm$ 1.72 <sup>c</sup>	21.60 $\pm$ 2.68 <sup>abCA</sup>	36.36 $\pm$ 2.91 <sup>abCB</sup>	31.23 $\pm$ 2.64 <sup>abCABC</sup>
TOTAL	35.24 $\pm$ 16.61	51.70 $\pm$ 23.71	41.14 $\pm$ 8.82	39.38 $\pm$ 11.39

SD: Standart Deviation, AP: Auto-polymerized, HP: heat-polymerized resin polymethyl methacrylate (PMMA), PA: Injection-molded polyamide, PK: Polyetheretherketone (PEEK), CT: Corega tablet, PT: Protefix tablet, SH: Sodium hypochlorite (NaOCl) solution, and DW: Distilled water.

Within the same column or row, the same superscripted letters indicate significant differences ( $p < 0.05$ ). a:AP, b:HP, and c:PA. Statistically significant differences between denture base material specimens (within the same denture cleanser). A:DW, B:CT, and C:PT. Statistically significant differences between denture cleansers (within the same denture base material specimens).

**Table 3.** Mean solubility values ( $\mu\text{g}/\text{mm}^3$ ) of all specimens in different denture cleansers.

Groups	Mean $\pm$ SD			
	DW	CT	PT	SH
AP	9.40 $\pm$ 1.70	13.15 $\pm$ 2.04 <sup>A</sup>	16.19 $\pm$ 2.25 <sup>AB</sup>	9.50 $\pm$ 0.97 <sup>BC</sup>
HP	8.45 $\pm$ 1.61	8.13 $\pm$ 1.42 <sup>a</sup>	10.73 $\pm$ 2.38 <sup>ABa</sup>	8.64 $\pm$ 0.71 <sup>c</sup>
PA	16.59 $\pm$ 3.84 <sup>ab</sup>	10.99 $\pm$ 7.53 <sup>ab</sup>	11.64 $\pm$ 2.06 <sup>ab</sup>	10.91 $\pm$ 6.99 <sup>ab</sup>
PK	8.41 $\pm$ 1.69 <sup>c</sup>	6.47 $\pm$ 0.83 <sup>Aabc</sup>	7.72 $\pm$ 1.10 <sup>Babc</sup>	6.82 $\pm$ 0.66 <sup>Aabc</sup>

SD: Standart Deviation, AP: Auto-polymerized, HP: heat-polymerized resin polymethyl methacrylate (PMMA), PA: Injection-molded polyamide, PK: Polyetheretherketone (PEEK), CT: Corega tablet, PT: Protefix tablet, SH: Sodium hypochlorite (NaOCl) solution, and DW: Distilled water.

Within the same column or row, the same superscripted letters indicate significant differences ( $p < 0.05$ ). a:AP, b:HP, and c:PA. Statistically significant differences between denture base material specimens (within the same denture cleanser). A:DW, B:CT, and C:PT. Statistically significant differences between denture cleansers (within the same denture base material specimens).

## 4. DISCUSSION

In this study, significant differences were observed between DBMs in terms of water sorption and solubility after immersion in three cleanser solutions. The null hypothesis that DCs would have no effect on the water sorption and solubility of DBMs was rejected.

Water sorption and solubility, clinically acceptable values of DBMs are determined by international specifications. The International Organization for Standardization (ISO) specification EN ISO 20795 for DBMs proposes the calculation of water sorption and solubility in units of  $\mu\text{g}/\text{mm}^3$  according to the volume of the specimens (18). In some studies, water sorption and solubility values were evaluated using the surface area in units of  $\text{mg}/\text{cm}^2$  obtained from DBM specimens in specification no.12 in accordance with the American Dental Association (ADA) (19). In another method suggested by Kazanji and Watkinson (20), it is beneficial to determine the long-term water sorption and solubility of DBMs in percentages; however, it does not provide complete standardization. In this study, the effect of DCs on DBMs was evaluated for a long term of 120 days using EN ISO 20795 for specimen preparation.

DBMs should ideally contain insoluble components and exhibit low water sorption. However, their lifetime exposure to factors such as saliva, pH, food, water, and cleanser can lead to the loss of water sorption, plasticizers, and other soluble ingredients (16). The maximum acceptable ISO standard for DBMs is 32  $\mu\text{g}/\text{mm}^3$  for water sorption and 1.6  $\mu\text{g}/\text{mm}^3$  for solubility (18). According to the results obtained herein, water sorption and solubility values were greater than the ISO values in some groups, possibly because 1 year of use of overnight immersion coincides with a 120-day cleanser bath.

In this study, the same polishing and smoothing process was applied to surfaces of all specimens for standardization before the experiment. Bollen et al. (21) have reported that the surface roughness of PMMA is affected by polishing abrasives used during standardization. As PMMA can be easily polished, its initial roughness is less. However, polyamides exhibit a fibrous, semi-flexible structure as well as low surface hardness (22). Although the Vickers hardness number of PEEK and PMMA materials is similar, PEEK exhibits a surface topography different from that of PMMA (17). Therefore, a different surface polishing procedure may be required. In parallel, Kurahashi et al. (23) have reported that a clinically acceptable surface roughness can be achieved using a soft polishing brush and agent for greater than 3 min for polishing PEEK. Heimer et al. (24) have evaluated the effects of laboratory and chairside polishing methods on the surface roughness and surface free energy of PEEK, auto-polymerized PMMA, and a composite resin and reported that compared to laboratory methods, chairside polishing methods for PEEK render lower surface roughness values. In this study, the same polishing process was applied to all DBMs. Unfortunately, there is no completely acceptable procedure for polishing PEEK compared to other DBMs.

Durkan et al. (25) have evaluated the effect of the 20-day application of DCs on the surface roughness, hardness, and color stability of a butadiene styrene copolymer PMMA, heat-polymerized PMMA, and two polyamides (Deflex and Valplast, respectively) and reported that polyamides significantly affect surface roughness, hardness, and color stability after immersion in a cleanser bath. In addition, in this study, DC-containing sodium perborate (Corega, Protefix) increase the surface roughness of polyamides and PMMA. Aging or wear of DBMs depends on several factors such as discoloration, water sorption, solubility, surface roughness, and hardness. Accordingly, in this study, DCs are thought to cause surface roughness in the DBMs with a polished surface after 120 days of bath, thereby leading to high water sorption and solubility values.

Song et al. (26) have evaluated the physical and mechanical properties of four injection-molded DBMs (i.e., polyamide, polyester, acrylic resin, and polypropylene, respectively), and the water sorption and solubility values of these materials were in the range of 6.17–24.38  $\mu\text{g}/\text{mm}^3$  and 0.76–3.11  $\mu\text{g}/\text{mm}^3$ , respectively. The higher water sorption and solubility values in their study compared to this study can be explained by measuring  $M_2$  and  $M_3$  in the long-term. In addition, Nguyen et al. (27) have reported that polyamide does not reach saturation for 8 weeks and continues to absorb water, which is in agreement with result of present study.

Liebermann et al. (17) performed an in vitro study and reported that the solubility of PEEK in physiological saliva and distilled water is less than those of PMMA and composite resin-based materials. In our study, in parallel with the results of this study, the solubility values of the PK group in distilled water were found to be similar to the HP group. In addition, lower solubility values of PK were found in cleanser baths compared to other DBMs. In our study, higher water sorption and solubility values were observed in comparison with those obtained in this study primarily because DCs affect surfaces of PEEK and PMMA. The water sorption and solubility of dental polymers can cause molecular imbalance, which can affect their mechanical properties, dimensional stability, and biocompatibility, thereby resulting in crack formation and subsequent reduction in mechanical properties. Water sorption not only affects its physical and mechanical properties but also reduces surface hardness and elastic modulus (17,28). In this study, the water sorption and solubility values of the PEEK group can be attributed to the molecular imbalance occurring on the PEEK surface. Owing to the lack of studies on the effect of DCs on the water sorption and solubility of PEEK, comparison with other DBMs for PEEK is difficult. However, the effect of the PEEK surface topography on water sorption and solubility should be examined in future studies.

Zissis et al. (29) have evaluated the release of residual monomers by the gas-liquid chromatography of four DBMs (i.e., three heat-polymerized PMMA and one auto-polymerized PMMA) and one hard liner over 1 week, 12 months, and 38 months after curing and reported that the

release of residual monomers in heat-polymerized PMMA is less than that in auto-polymerized PMMA. In addition, in this study, a statistically significant amount of the residual monomer in auto-polymerized PMMA was reported in the first 12 months. In particular, 1 week after curing, about 2.5% of the residual monomer was observed. Several studies have reported a relationship between residual monomer and water sorption. In the case of a residual monomer, less monomer conversion occurs, possibly leading to increased water sorption and solubility (30,31). In our study, parallel to these studies, a significant difference was observed in the water sorption and solubility in the AP group compared to the HP group depending on the residual monomer amount in chemical cleanser baths.

While DC tablets are a highly recommended hygiene practice by dental health professionals, Axe et al. (32) have reported that only ~24% of removable prosthesis users utilize this approach frequently. In a systematic review, Papadiochou et al. (33) have evaluated hygiene practices in removable prostheses and reported that brushing the prosthesis is the most common cleaning method in removable prostheses and that >50% of removable prosthesis users do not remove their prosthesis at night. In this study, considering the above-mentioned oral hygiene habits, the use of DCs and simulating an 8-h overnight immersion per day is an ideal practice, but it may be partly a limitation of this study in practical terms.

One of the limitations of this study is that the specimens were produced and tested under ideal conditions that may not reflect actual clinical conditions. Other limitations of this study include the inability to completely simulate the oral environment, such as temperature, humidity, pH, bacterial acids, and denture biofilm, possibly affecting water sorption and solubility values. The possible effects of the compounds in DCs on PEEK are not completely known. In addition, with respect to the PEEK polymer, possible effects of different DCs in terms of discoloration, surface roughness, and hardness should be examined in future studies.

## 5. CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn: The water sorption and solubility of DBMs increased due to DCs with different contents during long-term follow-up of 120 days. However, future experimental and clinical studies that investigate the effect of DCs with different contents on the color stability and surface topography of PEEK are required to confirm the results of this study.

## Conflicts of interest

None declared.

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