

EFFECT OF ARTIFICIAL AGING AND STAINING ON COLOR STABILITY AND SURFACE ROUGHNESS OF POLYETHER ETHER KETONE - AN INVITRO STUDY

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## **ABSTRACT**

**Objectives**: This study aimed to evaluate the effect of artificial aging and immersing solutions on the color stability and surface roughness of polyether ether ketone material in relation to surface polishing and glazing.

**Materials and Methods**: a total number of 80 plates were sliced out of reinforced polye ther ether ketone PEEK blanks. The samples were divided into two main groups according to polishing and glazing protocols (n=40) then each group was further divided into four subgroups according to the artificial aging and staining protocols (n=10). Artificial aging was done by aging by exposure to light for 120 min and water spraying for 18 min under artificial daylight (D65 illuminant). For staining, samples were subjected to thermos-cycling in colored solutions (coffee and cola) at different temperatures (50°C and 5°C). The average color difference ( $\Delta E$ ) was calculated, profilometer was used to measure the Surface roughness. The Kruskal-Wallis test was used to statistically analyze the data. The significance level was set at P ≤ 0.05.

**Results:** The mean values of the average color difference revealed that artificial aging and staining significantly affected PEEK color stability and surface roughness.

**Conclusions:** artificial aging and staining have a noticeable effect on the surface roughness and color stability of Polyether ether ketone material. Also, the Glazing of Polyether ether ketone has better surface roughness and color stability results.

KEYWORDS: PEEK; color stability; staining; surface roughness; artificial ageing

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# INTRODUCTION

PEEK is a polyaromatic semi-crystalline thermoplastic polymer. It's a biocompatible material with excellent chemical stability, mechanical properties, and good resistance to wear. PEEK is superior from a biomechanical perspective to other materials like glass ceramics and zirconia because it has an elastic modulus (4 GPa), comparable to human bone and dentine. The chewing forces are therefore cushioned through the material, transferring less force to the abutment teeth.<sup>(1)</sup>

PEEK was first used as dental material to manufacture implant superstructures by the company Bredent. Also, PEEK could be considered a good option for the fabrication of restorations in load-bearing areas. The maximum fracture resistance of PEEK is up to 1200 Newtons.<sup>(2)</sup>

Color stability is an important feature for restorations made from PEEK polymers. <sup>(3)</sup> Any changing in restoration color from the basic standard indicates aging or degradation of the material.<sup>(3)</sup> The color stability of PEEK significantly depends on the surface roughness and surface-free energy during surface processing. Many cleaning procedures can be used safely for PEEK polymers considering their color stability and surface roughness. <sup>(3)</sup>

Dental prostheses can become stained for a variety of reasons, including intrinsic factors such as the size of the fillers and the type of the resin matrix also the processing mode of the restoration, or extrinsic factors such as staining from food pigments like nicotine, anthocyanidins, tannins, and caffeine in drinks, mouthwash, and smoking.<sup>(4)</sup>

The mechanism behind PEEK discoloration still needs to be clarified. Surface roughness and surface treatments significantly affect color stability. Numerous research revealed a link between high surface roughness and denture resin discoloration, rationalized by the increased contact area.<sup>(5)</sup> This study aimed to examine the effects of aging

and immersing solutions on PEEK material's color stability and surface roughness in relation to surface processing (polishing or glazing).

# MATERIALS AND METHODS

## **Samples Preparation**

PEEK blanks (Amann Girrbach AG, Kobach, Austria) were sliced into 80 square-shaped samples of 1 mm thickness that were checked using a digital caliper. Polishing of all samples was done using grinding machine # 600 grit SiC paper for 20s (Ecomet 6, Buehler, Lake Bluff, IL) at 100 rpm, ten a low-speed handpiece with PoGo Discs (Dentsply Sirona, Charlotte, NC) used for 20s. Scooba Ultrasonic Cleaner ( Magpie Tech Corp, California, USA) was used to clean the samples using deionized water for 15 min. Baseline color measurements were recorded. Then samples were divided into 2 main groups (n = 40) according to the surface treatment protocol: Polishing only (P) and polishing then glazing (G). A single layer of resin glaze primer (Shofu, Kyoto, Japan) was applied to the samples and left for 60s to dry, followed by the application of two layers of glaze liquid (Shofu, Kyoto, Japan); polymerization was done for 180 s each in GC Labolight Duo Light-Curing Unit (GC, Tokyo, Japan), with a wavelength between 380-510 nm. The specimens of each group were randomly subdivided into four subgroups according to the staining and aging protocols (n = 10). The subgroup (a) specimens represent the control group, subgroup (b) represents samples soaked in distilled water and subjected to an artificial aging, subgroup (c) represents samples subjected to artificial aging and thermocycling in hot coffee, while subgroup (d) represents samples that subjected to artificial aging and thermocycling in cold cola.

## **Ageing Protocol**

In accordance with the International Organization for Standardization (ISO) 4892–2 standard the artificial aging was done to specimens of subgroups b–d. Xenon lamp weathering and light fastness test chamber (Suntest XXL+, Ametek Atlas, Mt. Prospect, IL) were used <sup>[27]</sup>. The cycle of artificial aging consisted of exposure to light for 120 minutes, followed by water spraying for 18 minutes under artificial daylight (CIE D65 illuminant). Humidity (50%) and temperature (37 °C) at constant temperature ( $\pm$ 3 °C), with a black panel temperature of 65 °C and irradiance control in the 300- to 400-nm interval of 60 W/m2. The total delivered energy was 300 kJ/m2.

#### **Staining Protocol**

Specimens of subgroup (c) were subjected to thermao-cycling in a hot coffee (50°C) and then distilled water (37°C), while subgroup (d) samples were subjected to thermao-cycling in a cold cola (5°C) and then distilled water (37°C). Each cycle lasted for 60s in the beverage, then 30 s in the distilled water, and then back to the colored beverage. To simulate 2 min per day of contact with colored beverages inside the patient's mouth,720 cycles were used. The coffee solution consisted of instant espresso coffee powder (illy, Trieste, Italy) A 5g / 120 mL of boiling water. Coca-Cola soft drink was used as a cold, carbonated beverage (Coca-Cola Company, Atlanta, GA, USA).

#### **Color Change Measurements**

The color of all the samples was measured using Vita Easyshade IV spectrophotometer (Vita Zahnfabrick, Bad Säckingen, Germany). Before each measurement, the spectrophotometer was calibrated. A white background (CIE L\*= 88.81, a\*= -4.98, b\*= 6.09) and standard illuminant D65, were selected, all measurements were done according to

the CIE L\*a\*b\* color space. the degree of lightness is referred to by the letter L\* (0-100), the color on the red/green axis is referred to by the letter a\*, and the color yellow/blue axis is referred to by the letter b\*. The measurements were taken from the middle of each sample. The color change ( $\Delta E$ ) of each sample was evaluated as follows:

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

The amount of color change was measured using the National Bureau of Standards (NBS) system. To relate the color change to a clinical standard, the  $\Delta E^*$  values were converted into NBS units where NBS =  $\Delta E^* \times 0.92^{[34-38]}$ . The NBS approach classifies changes as follows: (0.0–0.5) indicates extremely slight change,( 0.5–1.5 ) slight change, (1.5–3.0) perceptible difference; (3.0–6.0) marked change, (6.0–12.0) extremely marked change and (12.0 or more) indicates the change to another color.

## **Surface Roughness Measurements**

A contact profilometer with a 2mm stylus was used to assess the surface roughness of the specimens at baseline and after immersion. Mitutoyo 0.75mN Retractable Type SJ-210 Portable Surface Roughness Tester 178-563-11A (Mitutoyo, Kawasaki, Japan). measurements of the arithmetic average roughness (Ra) and the highest absolute vertical roughness (Rz) were made in 5 distinct directions. For each sample, the mean value of all measurements was calculated.

#### **Statistical Analysis**

The IBM (IBM Corporation, NY, USA) SPSS (Statistical Package for the Social Science; IBM Corp, Armonk, NY, USA) release 22 for Microsoft Windows was used to do all statistical calculations. The mean and standard deviation (SD) were used to describe the data statistically. The Shapiro-Wilk test was used to assess whether numerical data supported

the normality assumption. In order to compare the research groups, the Kruskal-Wallis test was used. The Wilcoxon signed-rank test was used for paired (matched) samples to compare variables within groups. P values of 0.05 or below were considered statistically significant.

#### RESULTS

For groups that Thermo-cycled in coffee and cola, NBS units calculated noticeable color changes with statistically significant differences between the different groups. The glazed samples showed better results than polished samples but there was no statistically significant difference between these two groups. (p = 0.063). Fig (1)



Fig. (1) Levels of color change for each subgroup, according to NBS.

Ra values showed a significant difference between the control, stained, and aged groups. The Ra values in coffee increased as it aged and thermo-cycled. Ra and Rz are highly positively correlated (r = 0.937). The difference in roughness between different surface processing groups (polishing and glazing groups) is significant (p = 0.02). Additionally, there is a great relation (0.978) between the color stability ( $\Delta E$ ) of the samples and the surface abrasion (Ra) regardless to surface polishing or glazing. Fig (2, 3)



Fig (2): Ra values (mean  $\pm$  SD) for all tested groups.



Fig (3): Rz values (mean  $\pm$  SD) for all tested groups.

#### DISCUSSION

The way aesthetic dental restorations behave while being used clinically is crucial. That differs according to many factors, such as the Type of the material and the technique of processing. In vitro studies always try to imitate the oral environment to get reliable results which help in producing reliable restorations. Studies used thermao-cycling tests to simulate changes in the oral cavity in order to estimate the long-term performance of tested restorative materials. <sup>(7)</sup> Thermo-cycling is a practical technique for accelerating the samples' artificial aging. This is beneficial because it can simulate the temperature in the oral cavity, which has a great effect on long-term deterioration, <sup>(8)</sup> and assess clinical performance. Both the colour stability and roughness of PEEK can be affected by artificial ageing. This effect is observed regardless of surface processing. <sup>(9)</sup> Studying the effects of artificial aging protocols on tooth-colored restorative materials has shown that color can be significantly affected. <sup>(10)</sup> Because of its low modulus of elasticity and biocompatibility, PEEK is advised for use in fixed and removable dental prostheses constructed by CAD/CAM technology. However, clinical trials are required to evaluate the long-term performance of restorations and prosthesis made of PEEK material. <sup>(11)</sup>

Coffee and Coca-Cola are common and widely used acidic colored beverages. Coffee is composed of caffeine and some acids such as tannic, citric, and chlorogenic acid. It's considered a mild acid (PH=4.8-5.1), while Coca-Cola is a highly acidic beverage (PH=2.6-2.7), it's composed of carbonated water, phosphoric acid, caramel color, and sugar. <sup>(12)</sup> Aesthetic restorative materials undergo changes in surface roughness and color due to exposure to the oral cavity.<sup>(11)</sup> The discoloration of dental restorations can have many different causes, including the type of material, surface treatment, the patient's nutritional routine, and oral hygiene. Discoloration of dental materials by strongly colored liquids like coffee and Coca-Cola can affect the aesthetic result and lead to patient dissatisfaction with dental restorations.<sup>(13)</sup>

Low-PH acidic drinks may result in increased wear and surface roughness leading to discoloration. <sup>(14)</sup> Therefore, this in vitro study aimed to determine how coffee and Coca-Cola affected PEEK's surface roughness and color stability. The current research provides a controlled and standardized process of production and eliminates other factors, which is crucial to provide results closely related to the clinical situation. <sup>(15)</sup> the results of this study agreed with a study done by Porojan L et al. (2021) <sup>(16)</sup>, which revealed that artificial aging has a great effect on the optical properties of PEEK material.

Another study done by Alsilani R. et al (2022) <sup>(17)</sup> discussed the color stability of PEEK in comparison

to Vita Enamic samples when immersed in different solutions and the results revealed that Vita Enamic samples and PEEK samples both showed color change above the clinically acceptable level.

Surface roughness and color stability are highly integrated. Rougher surfaces tend to attract more bacterial biofilms and stains <sup>(18)</sup>. Many studies have proved the effect of surface finishing and polishing on the material's color stability and surface roughness. Therefore, in this study polishing of the samples was done according to the manufacturer's instructions by the same operator for standardization.<sup>(13)</sup> Also glazing was performed according to the manufacturer's instructions <sup>(19)</sup>.

Since the color of the restoration is also influenced by surface roughness, the roughness characteristics were also taken into account in the present study. the rougher the surface, the less the reflected light reflects less light. Measurement of surface roughness is done by tracing the highest peaks and lowest valleys of the surface profile <sup>(20)</sup>. The average roughness (Ra) was calculated. Ra values below 0.2  $\mu$ m are clinically accepted <sup>(21)</sup>. The mean Ra values for all subgroups were within the clinically accepted range (below 0.15  $\mu$ m). In this study, the maximum absolute vertical roughness (Rz) was also recorded and a strong correlation was found <sup>(22)</sup>.

In accordance with the current studySahin O et al. (2016) agreed that artificial aging and thermaocycling in hot and cold colored solutions lead to an obvious increase in roughness values <sup>(21)</sup>.

In 2020, Kurahashi K et al. Suggested that the polishing and glazing protocols for PEEK achieve a clinically acceptable surface roughness.<sup>(24)</sup>

## CONCLUSIONS

Within the limits of this in vitro laboratory study, the following can be concluded:

 The glazing of PEEK has a positive effect on surface roughness, regardless of artificial aging process or staining techniques.

- Artificial aging negatively affects the surface roughness and color stability of PEEK, despite of different surface processing.
- Colored orally consumed solutions lead to obvious discolorations of PEEK material.

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