Original Article

In Vitro Evaluation of Color and Surface Roughness Changes of Polyetheretherketone, Monolithic Zirconia, and Resin Nanoceramics Exposed to Staining Liquids

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Received: 05-Apr-2024; **Revision:** 02-May-2024; **Accepted:** 10-May-2024; **Published:** 27-Jul-2024

Abstract

Background: This study aims to investigate color stability and surface roughness of polyetheretherketone (PEEK), zirconia, and hybrid ceramics while stored in different liquids. **Methods:** A total of 240 specimens were prepared from monolithic zirconia, PEEK, and hybrid ceramics. All specimens were polished using rubber sets with different grain sizes. Color parameters (L*, a*, b*) were measured three times using a dental spectrophotometer in standard D65 lightning. Each group was divided into eight different groups to be kept in eight different solutions as distilled water, cola, red wine, tea, coffee, heptane, citric acid, and 50% ethanol. Specimens were held in solutions at 37°C for 12 days. Color measurements were repeated, and color change (ΔE) was calculated using the CIE Lab formula. **Results:** The color difference of PEEK specimens was found above the clinically acceptable limit; however, color differences for monolithic zirconia produced by coffee were found within the clinically acceptable limits. ZR and HC specimens' color change values were found between threshold values ($1<\Delta E<3.3$). The differences observed in surface roughness levels amongst the ZR specimens could be caused by the polishing instrument and procedure. **Conclusion:** The color change of the materials was within acceptable limits, whereas the surface roughness increased more than 0.2 µm. Especially cola, heptane, and red wine significantly increased the mean surface roughness.

Keywords: *Color, hybrid ceramics, liquid, PEEK, zirconia*

Introduction

The advancement of digital technologies has led to the development of dental ceramics and an increase in demand for full ceramic and esthetic restorations that are both durable and have a natural appearance.[1] A variety of ceramic and polymer-based materials are now available to dentists owing to computer-aided design computer-aided manufacturing (CAD-CAM) technology.[1] The use of monolithic restorative materials eliminates chipping in layered restorations because of adhesion problems between the veneer and the core layers.[1,2] Feldspathic, lithium disilicate, zirconia-reinforced lithium disilicate, resin-matrix, zirconia ceramics, and PEEK are monolithic materials with different contents and microstructures that can be used with CAD-CAM systems.[2-4] Compared to conventional techniques, monolithic materials are used for producing more durable restorations.[3,5] Monolithic zirconia restorations have quickly gained popularity due to their high mechanical strength even at low material thickness, acceptable esthetic outcomes, short production times compared to veneered restorations, and lower costs.[5] Resin-based CAD-CAM materials can be used as alternative treatment options to ceramics in single-tooth restorations.[6] These materials have also been popular because they are easily processed in

Access this article online

Quick Response Code:

Website: www.njcponline.com

DOI: 10.4103/njcp.njcp_259_24

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How to cite this article: Demir Sevinç EH, Inal CB, Aydin C. *In vitro* evaluation of color and surface roughness changes of polyetheretherketone, monolithic zirconia, and resin nanoceramics exposed to staining liquids. Niger J Clin Pract 2024;27:918-24.

CAD-CAM systems, have elastic modulus that is close to dentin, do not cause opposing tooth wear, eliminate additional laboratory procedures, are easy to polish, and absorb functional stresses.[1] Also, current studies have focused on a new class of biomaterials such as BioHPP, which are glass-fiber or ceramics reinforced and belong to resin-based materials and polymers.[7] Peek reinforced with glass fiber or ceramics (BioHPP) improves the mechanical and biological qualities as well as its esthetic appearance.[7]

Polyetheretherketone (PEEK) is a linear polycyclic aromatic polymer and has a semi-crystalline structure. PEEK was first utilized in industry, and by the late 1990s, its stability, biocompatibility, radiolucency, and mechanical properties made it a suitable biomaterial for orthopedic and spine implants.[8,9] Today, PEEK is an alternative material in many dental applications. Because of its characteristics such as bone-like elastic modulus and low bacterial accumulation as well as its lightweight and durable structure, it is utilized as an implant material, prosthetic substructures, and as a temporary and permanent abutment material for implants.[10,11] PEEK is a biocompatible material and resistant to higher temperatures and chemicals.[6,12,13] PEEK, which has a natural color and is more biocompatible than metals, generally white or gray, and is quite opaque in terms of its optical characteristics. It needs to be veneered because it cannot be utilized in esthetic applications as monolithic restorations.[14] However, due to low surface energy and resistance to surface modification by different chemical agents, bonding between PEEK and veneer material is a clinical problem. For this reason, PEEK is usually preferred as a monolithic crown and bridge material in the posterior region.[15]

Primary factors determining the degree of discoloration are the type of solution, exposure duration, and substance of the material.[16] Food-simulating liquids are used to evaluate the chemical degradation of restoratives. The liquids approved by the Food and Drug Administration (FDA) are heptane, which imitates fatty meat and meat products; vegetal fatty foods; ethanol, which imitates alcoholic beverages; and citric acid, which imitates foods such as fruits and vegetables. These fluids affect the mechanical properties of restorative materials.[17] Even though current monolithic restorations have excellent mechanical qualities, they can still change color and surface roughness.[16] The pH of the oral environment is constantly changing because of chemical substances found in food and drink, as well as saliva in the mouth. This results in structural deformities, surface erosion, an increase in surface roughness, and color changes.[18]

The purpose of this study is to evaluate the color stability and surface roughness of PEEK, zirconia, and hybrid ceramics when stored in different liquids. The null hypothesis of this study was that the color and surface roughness of PEEK, zirconia, and hybrid ceramics would not be affected after being stored in different liquids.

Materials and Methods

Monolithic zirconia, PEEK, and hybrid ceramics were selected for the study [Table 1]. Semi-sintered monolithic zirconia blocks were cut 15 × 12 mm in diameter and 2.2 mm in thickness using a low-speed precision cutting device (Esetron Mod Dental; Ankara, Türkiye) by considering approximately 20% sinterization shrinkage. The specimens were dried in the porcelain furnace (Programat P300; Ivoclar Vivadent, Schaan, Liechtenstein), which was set to 80°C for 30 min before sintering, then sintered in a sinterization furnace (Infire HTC; Dentsply Sirona System, Bensheim, Germany) according to the manufacturer instructions. After sinterization, the final dimensions of the specimens were 12 × 10 mm in diameter and 1.5 mm in thickness. Hybrid resin nano ceramic blocks (Cerasmart; GC Company; Tokyo, Japan) and PEEK discs (Copra PEEK, Whitepeaks, Germany) were cut 14 × 12 mm in diameter and 1.5 mm in thickness. The thickness of all specimens was measured by a digital caliper (Alpha Tools, Mannheim, Germany) from three different regions and adjusted to 1.5 mm (±0.2). One surface of each specimen was smoothed with a series of 800, 1000, and 1200-grit abrasive silicon carbide papers. A total of 240 specimens were prepared, 80 for each material.

Zirconia specimens were polished using a zirconia polishing set (Amber; Whitepeaks, Germany) with varying grain sizes (coarse cyan, medium red, and fine cream). HC specimens were polished using a polishing rubber (Diatemp HP; Hybrid Resin Nano Ceramics, Eve, Germany). The PEEK specimens were polished using gray (thin grain size) and white (extra-thin grain size) colored polishing rubbers (Emerald Peek; Whitepeaks, Germany). For each experimental group, a polishing set was renewed in every 10 specimens. After completing the surface treatments, specimens were cleaned in an ultrasonic cleaner (Euronda, Eurosonic Micro; Vicenza, Italy) with distilled water for 10 min.

The color parameters were measured three times on the polished surfaces of the specimens. Measurements were performed using a dental spectrophotometer (VITA Easyshade Advance; VITA Zahnfabrik, Bad Säckingen, Germany) in D65 light conditions on neutral gray background. The spectrophotometer was calibrated after all three measurements.

The L*, a*, b* parameters of the CIE Lab color system were measured by a spectrophotometer and recorded. The following equation was used to calculate the difference between two colors in the CIE Lab system.[19]

$\Delta E^* = [(L_1^* - L_0)^2 + (a_1^* - a_0^*)^2 + (b_1^* - b_0^*)^2]^{1/2}$

In the CIE Lab color system, the L* value (0 means black and 100 means white) represents the brightness of the material, the a* value is the color density from red to green, the b* value is the color density from blue to yellow.[19] Before the testing process, the averages of L*, a*, b* color values obtained from each specimen were indicated as L_0, a_0, b_0 and recorded.

After the color measurements, the surface roughness values (Ra) were measured using a profilometer (Marsurf M300 C; Mahr GmbH, Germany) from three different regions of the polished surfaces.

Each group of materials was randomly divided into eight groups ($n = 10$) to be held in eight different solutions. The experimental groups are listed below:

1. Group: Distilled water
2. Group: Cola (Coca Cola Drink Inc., Ümraniye, İstanbul, Türkiye)
3. Group: Red wine (Kayıbağ, Denizli, Türkiye)
4. Group: Tea (Lipton; Rize, Türkiye); according to manufacturer instructions, one teapot tea was brewed in 200 mL of hot water and left to cool to room temperature
5. Group: Coffee (Nescafe, Blend 37, İstanbul, Türkiye); according to manufacturer instructions, 2 g coffee was mixed with 200 mL of hot water and chilled to room temperature
6. Group: Heptane (Tekkim Chemical Industry, Bursa, Türkiye)
7. Group: Citric acid (Aklar Chemistry; Ankara, Türkiye)
8. Group: 50% Ethanol (Aklar Chemistry; Ankara, Türkiye)

Each specimen group ($n = 10$) was placed in 50 mL falcon tubes and 20 mL of solution was added to the tubes. Specimens were held in solutions for 12 days to mimic the 12-month use of daily beverage consumption which lasts 3.2 cups each for 15 min.[20]

To imitate the intraoral environment, the temperature was held at 37°C in a stove (Edwards; Crawley, England) for 12 days. During this waiting period, all solutions were renewed every 2 days to prevent the formation of sediment caused by solutions.

The specimens were removed from the tubes on the 6^{th} and 12^{th} days and cleaned for 10 min in the ultrasonic cleaner. The measurements of color parameters and surface roughness values of the polished surfaces were repeated.

Statistical analyses

The data obtained in this study were analyzed using SPSS 22 (IBM, New York, USA) software. The normality of data was evaluated using the Shapiro–Wilk test. Because the data are not suitable for normal distribution, the Kruskal–Wallis H test was used in comparisons between three or more groups, and the Wilcoxon signed-rank test was used in the group comparisons. The results were significant for $P < 0.05$.

Results were evaluated according to the ΔE value. The clinical acceptability and perceptibility threshold values are accepted as $1 < \Delta E < 3.3$.[21]

Results

Color change results of experimental groups

Descriptive and comparative statistics of the color change values of the experimental groups are shown in Table 2. There was a significant difference between the material types in terms of color difference ($P < 0.05$).

When the color difference of the materials on the 6^{th} and 12^{th} days is examined, according to the Wilcoxon signed-rank test results ($P < 0.05$). There was a significant difference between the PEEK and ZR groups, whereas there was no significant difference between the HC groups ($P > 0.05$).

The ΔE values of the HC specimens were mostly clinically acceptable and were significantly lower than ZR and PEEK specimens on the 6^{th} and 12^{th} days ($P < 0.05$). The 12th-day ΔE values of PEEK specimens were higher than the acceptable threshold

Table 1: Brand names, manufacturers, compositions, and shades of the monolithic crown materials used in the study

Monolithic crown materials	Brand name	Manufacturer	Composition	Block shade
Zirconia	Incoris TZI (ZR)	Dentsply Sirona, Germany	$ZrO_2 + HfO_2$ + Y2O3, 99% by weight	A1
Hybrid resin nanoceramic	Cerasmart GC (HC)	GC Dental Product Corp. Japan	71% nanoparticle filling includes silica and barium glass	A1 LT 14
PEEK	Copra PEEK (PEEK)	Whitepeaks, Germany	100% PEEK	Light

Table 2: The mean color change (ΔE) values measured on the 6th day and the comparison of the specimens'ΔE values with the Kruskal–Wallis *H* test

ΔE	ZR				HC				PEEK			
	6th day		12th day		6th day		12th day		6th day		12th day	
	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med
Distilled water	1.6±0.7	1.8 A	2.3±0.6	2.4 B	0.9±0.3	0.87 C	0.81±0.47	0.60 C	1.74±0.86	1.27 B	3.06±2.26	3.57 A
Cola	1.2±0.7	1 B	2.7±1	3 B	1.07±0.98	0.86 B	1.11±0.8	0.87 C	2.62±1.66	2.69 A	3.96±0.93	4.26 A
Red wine	2.32±0.37	2.20B	2.08±0.54	2.17 B	1.07±0.64	0.97 C	1.44±0.51	1.42 C	3.78±1.9	3.96 A	3.29±1.71	3.20 A
Tea	2.67±0.50	2.73A	2.58±0.79	2.63 B	1.15±0.53	1.01 B	1.38±0.39	1.29 C	2.76±1.38	2.93 A	3.24±1.43	2.95 A
Coffee	3.14±0.82	3.19A	1.74±0.41	1.70 B	0.99±0.64	0.93 B	1.35±0.55	1.23 C	3.44±1.52	3.43 A	3.67±1.23	3.56 A
Heptane	4.2±0.43	4.23A	2.30±0.66	2.20 B	0.71±0.35	0.66 C	1.12±0.55	1.09 C	3.34±1.4	3.48 B	3.36±1.22	3.04 A
Citric acid	3.54±1.22	3.93A	2.19±0.92	2.26 A	1.45±0.32	1.35 B	1.35±0.49	1.39 B	1.88±1.36	1.56 B	1.67±0.98	1.38 B
Ethanol %50	3.08±0.91	3.19A	1.94±0.93	2.24 A	1.00±0.29	0.99 C	1.00±0.29	0.99 B	2.00±1.39	1.44 B	2.38±1.84	2.15 A

*ΔE: Color change SD: Standard deviation, ZR; zirconia, HC; hybrid ceramic, PEEK: polyetheretherketone, There is no statistically significant difference between the median values of groups with common capital letters in the ZR, HC, and PEEK groups held in the same solution for 6 and 12 days (P>0.05)

Table 3: The surface roughness (Ra) values measured on the 6th and 12th day and the comparison of the specimens' Ra values with the Kruskal–Wallis *H* test

Ra	ZR				HC				PEEK			
	6th day		12th day		6th day		12th day		6th day		12th day	
	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med	Mean (± SD)	Med
Distilled water	0.37 (± 0.07)	0.37 A	0.32 (± 0.08)	0.32 A	0.11 (± 0.04)	0.09 B	0.12 (± 0.04)	0.11 B	0.33 (± 0.12)	0.32 A	0.34 (± 0.11)	0.33 A
Cola	0.34 (± 0.08)	0.35A	0.36 (± 0.09)	0.39 A	0.11 (± 0.04)	0.11B	0.12 (± 0.04)	0.11 B	0.27 (± 0.07)	0.27A	0.30 (± 0.06)	0.29 A
Red wine	0.37 (± 0.06)	0.38 A	0.39 (± 0.07)	0.42 A	0.11 (± 0.03)	0.12 B	0.13 (± 0.03)	0.11 B	0.37 (± 0.07)	0.37 A	0.38 (± 0.08)	0.39 A
Tea	0.31 (± 0.09)	0.31 B	0.33 (± 0.09)	0.32 B	0.13 (± 0.02)	0.13 C	0.15 (± 0.03)	0.15 C	0.35 (± 0.08)	0.34 A	0.34 (± 0.04)	0.34 A
Coffee	0.41 (± 0.09)	0.45 A	0.38 (± 0.09)	0.38 A	0.12 (± 0.04)	0.11 C	0.13 (± 0.05)	0.12 C	0.25 (± 0.03)	0.25 B	0.25 (± 0.05)	0.25 B
Heptane	0.32 (± 0.09)	0.33 A	0.36 (± 0.06)	0.37 A	0.12 (± 0.03)	0.11 B	0.14 (± 0.05)	0.12 C	0.27 (± 0.05)	0.28 C	0.31 (± 0.05)	0.30 B
Citric acid	0.30 (± 0.06)	0.29A	0.29 (± 0.08)	0.31 A	0.12 (± 0.05)	0.13B	0.12 (± 0.04)	0.12 B	0.32 (± 0.05)	0.32C	0.32 (± 0.05)	0.32 C
Ethanol %50	0.32 (± 0.08)	0.32 A	0.29 (± 0.12)	0.27 C	0.12 (± 0.03)	0.11 B	0.12 (± 0.02)	0.13 B	0.35 (± 0.08)	0.31 A	0.34 (± 0.08)	0.32 A

*Ra: Surface roughness SD: Standard deviation, ZR; zirconia, HC; hybrid ceramic, PEEK: polyetheretherketone. Between ZR, HC and PEEK groups that are held in the same solution for the same period, there is no statistically significant difference between the mean Ra values with the same uppercase letters (P>0.05) in nearly all solutions, whereas the ZR specimens' ΔE values were very close to the clinically acceptable threshold.

The 12th-day ΔE values of the ZR and HC group were found between the acceptability limits (1< ΔE < 3.3), except for the distilled water group for HC specimens, which were below the perceptibility threshold (0.81 ± 0.47).

Although there was no statistically significant difference between ZR and PEEK samples ΔE values that were held in tea and coffee solutions on the 6th day, there was a significant difference between these two groups on the 12th day.

Comparison of mean surface roughness values (Ra) of monolithic zirconia, hybrid ceramic, and PEEK specimens

The results of the Kruskal-Wallis h-test. are shown in Table 3. There was a significant difference between different materials in terms of mean Ra values (P < 0.05).

Significant differences were observed on the 6th and 12th days' mean Ra values compared to all material groups held in heptane, citric acid, and coffee solutions (P < 0.05). The Ra values on the 6th and 12th days were significantly lower in the HC group than in the ZR and PEEK groups.

The PEEK and ZR groups' surface roughness was above the clinically accepted value (Ra = 0.2 µm), but the HC group's surface roughness was below this threshold. The surface roughness of ZR, HC, and PEEK is often increased by distilled water, red wine, and heptane. Tea does not affect ZR and PEEK; however, it increases the surface roughness of HC. Holding in cola, in contrast, increases the surface roughness of ZR and PEEK but not HC.

Discussion

This study evaluates the surface roughness and color changes of ZR, HC, and PEEK polymers, which are subjected to different liquids. The liquids caused changes in color and the surface roughness of monolithic specimens. Therefore, the null hypothesis was rejected.

CAD-CAM system allows the use of different metal-free monolithic restorative materials. These have become more popular because of their biological and esthetic advantages. One of the most frequently used materials is monolithic zirconia, because of its excellent mechanical properties, it becomes an ideal restorative material.[22] Monolithic ceramics, ceramic-like materials, and polymers are applied safely in posterior restorations due to their natural tooth-like appearance, reinforced composition, and superior mechanical features.[23,24] Therefore, hybrid ceramics and PEEK materials that are alternative to zirconia for monolithic restorations are used in this study. Hybrid ceramics are restorative materials that combine the benefits of ceramics and composite resins. It is utilized as a chair-side restorative material thanks to features such as easy polishing, speed milling, and the absence of additional firing.[3] PEEK, another monolithic material alternative, has already found an application in prosthetic dentistry as an infrastructure material, and it is being considered as an alternative to zirconia crowns in the posterior region.[25]

The material choice of restoration is significantly influenced by surface roughness, mechanical durability, and coloring by aging.[18] These three monolithic materials were influenced differently by various liquids with different characteristics due to their dissimilar surface properties. Restorations are exposed to a variety of substances in the oral environment. Long-term interaction with these fluids can lead to alterations in the structure of restorations, including surface roughness and color. These criteria should also be considered when choosing the restorative material.[26]

Monolithic restorations frequently exposed colorants from foods and drinks in an intraoral environment. The solutions were selected from a variety of sources, including colored beverages that are commonly consumed, acidic beverages that can cause chemical degradation, and solutions that are similar to foods and alcohol.[17,27-29] The waiting period of the specimens in these solutions was calculated according to 1 year in the clinic, 15 min per day for 12 days.[20,27,30]

According to a study, PEEK is a semi-crystalline polymer, and its degree of crystallinity may be one of the primary variables influencing its ability to absorb dyes or water, which could lead to a decrease in color stability.[31] PEEK specimens' color difference was found above the clinically acceptable limit, which was especially similar to studies that looked at how coffee affects the color of PEEK.[16,32] This is explained by coffee causing coloration in the material because of both absorption and adsorption.[33] However, coloring with coffee caused color differences within the clinically acceptable limits for monolithic zirconia used in this study.

Acceptability and perceptibility threshold values for esthetical properties of posterior restorations may be tolerated at higher levels. The CIE Lab color difference formula provides an advantage in terms of the comparability of the study results because it is extensively used in the literature and is simple to use.[32] Perceptibility and acceptability threshold values are typically regarded to be 1< ΔE <3.3 in studies that evaluate color changes in materials held in solutions.[30,34] In this study, these threshold values were accepted and those with a ΔE value above 3.3 are not clinically acceptable; those between 1 and 3.3 threshold values are stated to be clinically perceptible.

In this study, the color difference values from three various monolithic materials generally were found below the acceptable threshold (ΔE <3.3). However, the color change values for PEEK specimens in red wine, coffee, and heptane on the 6th day and cola, coffee, and heptane on the 12th day were above the acceptable threshold value. Color change values of ZR and HC specimens were between threshold values (1< ΔE <3.3). There was a significant difference between the monolithic materials used in this study. According to the results, HC specimens showed the lowest color change in all solutions, whereas PEEK and ZR generally did not show any significant difference. The biggest color change was observed in coffee, red wine, cola, and heptane in general. Although there was no significant difference between the 6th and 12th days for PEEK specimens, the color change observed on the 6th day in ZR was greater. The biggest color change occurred in the PEEK group for this study. It had been thought that this was due to a lack of ceramic fillers in the PEEK material used in the present study and that the other monolithic materials had more resistant structures to discoloration.[16] Excessive coloration in ZR and PEEK held in heptane solution could be caused by heptane influencing the surface hardness of the material.[17] According to the study, re-polishing PEEK restorations after 1 year is recommended due to an increase in surface roughness.

Both free surface energy and surface roughness have a major impact on color stability. Dental materials with smooth surfaces and low surface energy exhibit less color change.[35,36] The necessity and importance of chairside polishing are supported in *in vitro* studies.[37-39] In this study, it is believed that the polishing applied to the specimens prevents the fluids from adhering to the surface, avoiding the coloring of the material. Although the color change values of HC specimens reached the highest value with red wine on the 12th day as a result of reducing surface roughness by polishing, it is below the acceptable threshold. The cumulative effect of red wine's alcohol and pH has been predicted to cause an increase in surface roughness, which is why it affects all the materials. The highest surface roughness values were generally recorded in ZR material, PEEK, and HC materials. Although the difference between PEEK and ZR Ra values was not statistically significant, a statistically significant difference was found with HC. The polishing equipment and process are considered to have led to the difference in surface roughness levels between the ZR specimens in this study. Glazing may be recommended for zirconia restoration to avoid unexpected results of polishing.

One of the limitations of this study is the wear during the chewing function and how it will affect the color and surface roughness of the materials held in liquids. Furthermore, procedures to reach standard thickness and size may cause the inhomogeneous color of specimens. High solubility is caused by a variety of proteins found in natural saliva.[40] Using distilled water as a control group instead of saliva does not fully reflect the negative effects of saliva on dental materials. Furthermore, the study did not investigate how artificial aging affects the surface characteristics and color changes of dental materials. The impact of liquids on carbon or glass-reinforced PEEK materials' surface roughness and color change values might be evaluated in future studies.

Conclusions

Within the limitations of this study, the following conclusions were drawn:

1. PEEK and monolithic zirconia materials resulted in higher color changes and mean surface roughness values than in hybrid resin ceramics for all solutions. Although color change values were within the acceptable threshold values, mean surface roughness values were above 0.2 μm.
2. The surface roughness of all three monolithic materials showed an increase in the cola, red wine, and heptane solutions.

Acknowledgments

This work has been supported by the Gazi University Scientific Research Projects Coordination Unit under grant number 03/2020-25.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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