



EFFECT OF VARIOUS PH MEDIA ON SOME OPTICAL PROPERTIES AND SURFACE ROUGHNESS OF BILAYERED REINFORCED POLYMER AND MONOLITHIC HYBRID CERAMIC (AN IN-VITRO STUDY)

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ABSTRACT

Objective: The objective of this study was to evaluate the effect of different pH media on color stability, translucency and surface roughness on Hybrid ceramic material (Vita Enamic) and bilayered reinforced polymer (PEEK). **Materials and methods:** A total of 60 discs, 30 in each group that were designed with milled with CAD/CAM, each group was further subdivided into 3 subgroups (10 samples in each group) according to the immersing solution.(neutral, weak acid and strong acid), all samples were aged in the immersion solutions for 24 days in an incubator at 37° which simulate 2 years of clinical service. Optical profilometer was used to assess surface roughness, a spectrophotometer was used to assess color change and translucency was calculated with the tranclucency parameter equation. **Results:** The results showed that no statistical significant difference in surface roughness, color stability and translucency with Vita Enamic in different solutions, and statistical significant difference with PEEK group in different solutions. **Conclusion:** Vita Enamic has higher color stability and translucency than PEEK and can maintain smoother surface with different pH media.

KEYWORDS: ph Media, Optical Properties, Surface Roughness, PEEK, Vita Enamic

INTRODUCTION

With the increased demand in esthetics, patients and dentists are seeking perfection in restoring natural teeth. Ceramics have shown the most accurate reproduction of the appearance, color, and texture of natural teeth⁽¹⁾. Several ceramic-based materials have recently been introduced for CAD–CAM dentistry including lithium disilicate glass ceramic, yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP), monolithic zirconia, and zirconia-containing lithium silicate ceramics nano-resin ceramic, hybrid ceramic, interpenetrating phase ceramic. These materials help in the technological revolution that offers clinicians appropriate ceramics with the ease of restoration fabrication through CAD–CAM technology⁽²⁾. Vita Enamic is a member of hybrid ceramics group by VITA;

The ceramic part consists of an aluminum oxide-enriched, fine-structure feldspar matrix (86 wt.%) infused by a polymer material consisting of (14 wt%) urethane dimethacrylate and triethylene glycol dimethacrylate. Its flexural strength is 151 MPa.⁽³⁾ Both advantages of ceramic and resin materials are combined in ceramic/polymer materials such as less brittleness, excellent machinability and edge stability⁽³⁾.

New materials based on polyether ether ketone (PEEK) were introduced to the market as a potential alternative material for surgical procedures such as inter-body fusion cages or dental implants with a similar stress distribution as titanium implants⁽⁴⁾. PEEK is an organic thermoplastic material with excellent mechanical and chemical resistance properties. Another variant of PEEK is BioHPP

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developed by Bredent GmbH. BioHPP stands for Bio High Performance Polymer and has been developed especially for dental applications. The material has been proven to be very popular amongst dental technicians ⁽⁵⁾.

One of the problems that face hybrid ceramics is long term color stability, discoloration may be endogenous or exogenous. Endogenous discoloration results from chemical instability of the material, exogenous staining results from adsorption of stains from oral cavity and is affected by other conditions as surface roughness.⁽⁶⁾ With the increasing demand on aesthetic restorations, it is necessary to evaluate the color stability of these hybrid materials when it is subjected to different pH media. Commonly consumed beverages can range from neutral solutions, weak acids and strong acids⁽⁷⁾.

The aim of this study was to evaluate the effect of different pH media on color stability, translucency and surface roughness on Hybrid ceramic material (Vita Enamic) and bilayered reinforced polymer (PEEK).

MATERIALS AND METHODS

Samples grouping:

A total of 60 disc shaped samples with dimensions (10mm diameter x 1.5 mm thickness) were fabricated. They were divided into two equal groups of 30 samples each according to the type of material used: (Group I PEEK and Group II Vita Enamic). PEEK samples were composed of two layers; (substrate layer Bio HPP and the veneering layer HIPC).

Samples fabrication

One blank of each Bio.HPP and Bio.HIPC were used to obtain cylinders with diameter of (10mm), CAD/CAM was used to design and mill cylinder shapes, these cylinders were cut into 30 discs of Bio. HPP and 30 discs of HIPC with desired dimensions (10mm diameter x 0.7 mm thickness) and 30 discs

of Bio.HIPC (10mm diameter x 0.8 mm thickness) using Isomet cutting machine (Isomet 4000 linear precision saw, Lake Bluff, USA) (2500rpm with coolant), digital caliper (Pro'skit Mechanical digital caliper) was used to insure thickness and diameter of each sample. Cementation of HIPC discs on Bio. Hpp discs was done according to manufacturer instructions by the following steps.

First the veneering surface of Bio.Hpp and the luting surface of HIPC discs were abraded with airborne-particles (110 μ m Aluminum oxide) (Al₂O₃) powder (basic Quattro IS; Renfert, Hilzingen, Germany) with pressure (2 MPa), at an angle of 45° from a distance of 10 mm using customized sand-blasting machine, and subsequently put in an ultrasonic bath (L&R Transistor Ultrasonic T14, L&R, Kearny, NY, USA) filled with distilled water for 5 min, Afterwards, then handled with plastic tweezers and left to dry.

Bio.Hpp and HIPC discs were conditioned using visio.link for 1 minute and polymerized for 90 seconds at 220 mW/cm² 90 s by polymerizing light cure machine (Bre.Lux power unit 2- bredent GmbH &Co. KG).⁽⁸⁾ A thin layer of combo.lign luting cement was applied to bonding surfaces of HIPC before pressing them on the Bio.Hpp discs, then excess cement was removed by brush and then samples were polymerized by light cure machine for 180 s at 220 mW/cm² ⁽⁸⁾.

The Vita Enamic block were designed and milled by the same technique used with PEEK to obtain cylinder blocks with round diameter then these cylinders were cut by micro saw to obtain 30 round shaped samples with surface area of 10mm diameter and 1.5mm thickness.

An experienced technician carried out the finishing and polishing sequences in accordance with the manufacturer's instructions for rpm. The same straight hand piece and electric motor was used to control the rpm setting for each step.

Aging:

Base line readings (surface roughness, color and translucency) were measured for each sample before aging process, then each sample was stored in a separate container, These containers were filled with different pH solutions according to each group.

The total storage time was 24 days in an incubator at 37°C which simulate 2 years of clinical service⁽⁹⁾. The solutions were changed every 24 hours and fresh solution was prepared each time.

Surface roughness measurements:

The optical methods tend to fulfill the need for quantitative characterization of surface topography without contact⁽¹⁰⁾. Samples were photographed using optical profilometer, USB Digital microscope with a built-in camera (U500X Capture Digital Microscope, Guangdong, China) connected with an IBM compatible personal computer using a fixed magnification of 120X. The images were recorded with a resolution of 1280 × 1024 pixels per image. Digital microscope images were cropped to 350 x 400 pixels using Microsoft office picture manager to specify/standardize area of roughness measurement. The cropped images were analyzed using WSxM software (Ver 5 develop 4.1, Nanotec, Electronica, SL)⁽¹¹⁾. Within the WSxM software, all limits, sizes, frames and measured parameters are expressed in pixels. Therefore, system calibration was done to convert the pixels into absolute real world units. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software. Subsequently, a 3D image of the surface profile of the samples was created. Five 3D images were collected for each sample, in the central area and in the sides at area of 10 μm × 10 μm⁽¹²⁾.

WSxM software was used to calculate average of heights (Ra) expressed in μm, which can be assumed as a reliable indices of surface roughness⁽¹³⁾.

Color change (ΔE) measurements:

Color stability was assessed using spectrophotometer each sample was assessed before and after aging, The color was measured using a reflective spectrophotometer (X-Rite, model RM200QC, Neu-Isenburg, Germany). The aperture size was set to 4 mm and the specimens were exactly aligned with the device. A white background was selected and measurements were made according to the CIE L*a*b* color space relative to the CIE standard illuminant D65. The color changes (ΔE) of the specimens were evaluated using the following formula:

$$\Delta E_{\text{CIELAB}} = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$$

Translucency parameters (TP)

The translucency parameters (TP) values were obtained by calculating the color difference of the samples over black and white backgrounds by using the following equation: $TP = [(L_b^* - L_w^*)^2 + (a_b^* - a_w^*)^2 + (b_b^* - b_w^*)^2]^{1/2}$, where letters “b” and “w” refer to color coordinates over the black and white backgrounds, respectively.

RESULTS AND STATISTICAL TESTS

The results were analyzed using Graph Pad In-stat (Graph Pad, Inc.). A value of P < 0.05 was considered statistically significant, one-way ANOVA was done for compared time followed by Tukey’s pair-wise if showed significant. Two-way analysis of variance was performed. Student t-test was done between groups at different pH media.

Roughness

For *Gr_PEEK*: it was found that a statistically significant in roughness mean value between subgroups. For *Gr_Vita Enamic*: it was found that no statistical significant difference in roughness mean value between subgroups. All values are represented in table 1.

TABLE (1) Descriptive statistics of roughness results (Mean values± SDs) in μm for both groups at different pH media:

Variables	Baseline	pH_5.5	pH_2.5	pH_7	P value
Gr_PEEK	0.2241 ^{bB} ±0.014	0.2439 ^a ^B ±0.013	0.2189 ^{bB} ±0.008	0.2384 ^a ^B ±0.019	0.0005*
Gr_Vita Enamic	0.2453 ±0.009 ^A	0.2541 ±0.006 ^A	0.2492 ±0.013 ^A	0.2510 ±0.006 ^A	0.2259
P value	0.0013*	0.0306*	<0.0001*	0.0194*	

* Indicates the mean difference is statistically significant at the 0.05 level.

Different small letters indicate statistically significant difference in the same row. ($p\text{-value} \leq 0.05$).

Different capital letters indicate statistically significant difference in the same column. ($p\text{-value} \leq 0.05$).

Color change (ΔE) measurements:

For **Gr_PEEK**: it was found that a statistically significant in ΔE mean value between subgroups. For **Gr_Vita enamic**: it was found that no statistical significant difference in ΔE mean value between subgroups. All values are represented in table 2.

TABLE (2): Descriptive statistics of color change results (Mean values± SDs) for both groups at different pH media

Variables	pH_5.5	pH_2.5	pH_7	P value
Gr_PEEK	8.07±3.9 ^a	4.29±1.27 ^b	5.89±1.51 ^{ab}	0.0097*
Gr_Vita Enamic	7.95±2.44	5.46±1.34	7.02±4.33	0.1862ns
P value	0.9439 ns	0.059 ns	0.4473 ns	

Translucency parameters (TP):

For**Gr_PEEK**:itwasfoundthatnostatisticalsignificantdifference inTPmean valuebetween subgroups. For **Gr_Vita Enamic**: it was found that no statistical significant difference in TP mean value between subgroups.

TABLE (3): Descriptive statistics of translucency parameter results (Mean values± SDs) in TP for both groups at different pH media

Variables	Baseline	pH_5.5	pH_2.5	pH_7	P value
Gr_PEEK	7.61 ±2.32 ^B	7.76 ±2.69 ^B	6.99 ±2.49 ^B	6.97 ±3.34 ^B	0.8245 ns
Gr_Vita Enamic	16.33 ±3.21 ^A	16.72 ±4.56 ^A	14.25 ±3.07 ^A	12.82 ±3.8 ^A	0.0580 ns
P value	<0.0001*	<0.0001*	<0.0001*	0.0018*	

DISCUSSION

Color of dental restorative material is one of the optical properties, and it is the visual perception of an object, or substance regarding the light reflected, or transmitted through⁽¹⁴⁾. Tooth-colored fillings and fixed dental prostheses are important expectations to achieve patients and dentists satisfaction.

Color stability can be affected by many factors such as: incomplete polymerization, chemical reactivity, water sorption, and surface roughness of material.^(15, 16) Polishing ability and material structure are not the only parameters providing color stability for a longer period of time but also patient’s dietary habits and cleaning measures are key factors^(17, 18). With aging process in clinical use, ion leaching out occurs leads to surface roughness of the material and subsequent color change⁽¹⁹⁾.

In this study these two different materials were selected because both of them have polymer phases. PEEK (Bio.Hpp) and veneering composite (Brecam.HIPC) were used not the granules type because it is a CAD/CAM material same as Vita Enamic.

Polyetheretherketone (PEEK) is a semi-crystalline thermoplastic polymer and is considered as a promising alternative of fixed and removable Prosthetic dentistry with adequate mechanical properties to fulfill the basic requirements in the restorative field as showed by recent studies⁽²⁰⁾.

Vita Enamic is one of the polymer-infiltrated feldspathic ceramic materials, consists of 86% ceramic (by weight)⁽²¹⁾. Besides having the properties of both ceramic and composite materials, polymer-infiltrated ceramic network (PICN) materials are considered to have mechanical and aesthetic properties similar to natural teeth⁽²²⁾. Highly aesthetic milling block that provides an alternative to ceramic blocks for computer-aided design/computer-aided manufacturing (CAD/CAM) indirect restorations⁽²³⁾.

This present study was performed in vitro to evaluate the effect of different pH media on surface roughness and color stability of different hybrid materials; In-vitro testing was used because it overcomes the limitations associated with the clinical testing such as individual variation by creating a controlled environment. These tests provide a guideline about the color stability of the two materials and act as a baseline for the clinical studies⁽²⁴⁾.

The supporting structure or esthetic restorative material is a primary source of the resulting color. This color is influenced by the thickness and translucency of the final restoration, as evident by the amount of reflection and scattering of light⁽²⁵⁾.

Samples were cut into discs with the diameter 10mm and 1.5 mm thickness, these sample configuration was selected because it fits experimental system, also it could be considered adequate thickness recommended by the manufacturer for prosthetic appliance. Peek samples were veneered to simulate clinical conditions, there are different systems and techniques for the peek and veneering fabrication.

Another criterion of aesthetics is the surface roughness. Rough surfaces are more prone to coloration because of its elevated stain retention capacity⁽²⁶⁻²⁸⁾. The evidence proved that stain resistance and color stability is improved with properly polished surfaces⁽²⁹⁾. It was also reported that materials with different microstructures require different polishing techniques⁽³⁰⁾, so that polishing was per-

formed by the same operator for both groups but with different polishing kits. Peek samples were polished with Bredent polishing kit, and Vita Enamic samples were polished using the Vita Enamic technical polishing set following the manufacturer's instructions.

In the oral cavity restorative materials are continuously exposed to different pH media according to dietary habits and oral conditions. The total immersion time was 24 days is equivalent to 2 years of consumption of the beverages which might be enough time to evaluate clinical success⁽⁹⁾.

Surface roughness

Samples were photographed using USB Digital microscope because of easier access, affordability and reduced time.

It was found that in baseline readings Vita Enamic has higher statistical significant surface roughness than PEEK group, but within the subgroups there was significant change with different pH media on PEEK and no significant change on Vita Enamic group.

Between all pH media, Pepsi subgroup showed lowest statistically significant decrease in surface roughness ($0.2189 \pm 0.008 \mu\text{m}$), the low pH of Pepsi (2.5) may have affected the surface integrity of PEEK veneering resulting in smoothing of the surface⁽³¹⁾.

Cruz et al.⁽³²⁾ stated that the increase in surface roughness of Vita Enamic with exposure to strong acids was possibly due to the dissolution of the ceramic portion that constitutes most of this material. The boundaries between the ceramic and polymer portions became more evident with the dissolution of the feldspathic matrix by the acid. Furthermore, microcracks were observed on the surface, revealing that this material seems to have been the most affected by the acid.

On the other hand coffee subgroup with pH (5.5) recorded highest statistical roughness mean value with PEEK (0.2439 ± 0.013), The increase in surface

roughness with coffee subgroup may be due to acidity in coffee which alters surface topography by creating micro- and nano-scale porosities of varying depth and with water might be due to water sorption of the material⁽³³⁾.

Color

It was found that there was no statistical significant difference between both groups in color change but within the subgroups there was significant change with different pH media on PEEK and no significant change on Vita Enamic group.

Coffee subgroup recorded statistically significant highest color mean value in both groups, it may be due to smaller molecular size of coffee coupled with water absorption characteristic of the tested materials, creating a stronger staining effect^(34,35).

PEEK recorded statistically higher color mean value, this discoloration usually occurs as a result of water sorption by the resin component of the material. The type of resin matrix plays a vital role in the color sustainability of the material.

According to the manufacturer, polymer part in Vita Enamic consists hydrophobic urethane dimethacrylate (UDMA) and hydrophilic triethylene glycol dimethacrylate (TEGDMA). Accordingly, the TEGDMA in Vita Enamic blocks may have resulted in greater water sorption which may have permitted the penetration of any hydrophilic colorant into the resin matrix.

Since UDMA is more hydrophobic than TEGDMA, it is therefore more color stable. Nevertheless, it has been reported that dimethacrylates form cross-linked networks with entrapped unreacted monomers that serve as plasticizers. Such plasticization forms a more open structure, which may facilitate additional water sorption. This may explain how the resin matrix could have contributed to the higher discoloration values obtained by Vita Enamic⁽³⁶⁻⁴⁰⁾.

Previous studies observed that discoloration occurs with Vita Enamic in coffee solution which could be attributed to compatibility of the yellow colorant with the resin matrix. The degree of dye penetration into the resin matrix depends on the dye polarity. Since coffee is a solution of low polarity, its yellow colorant may have facilitated deeper colorant penetration into the resin matrix^(31,38,40,41).

Translucency

Group PEEK showed lower statistical significant value than Vita Enamic in translucency parameter which may be due to the effect of opaque base layer while Vita Enamic is a monolithic material with no base material.

Different solutions showed no significant effect on translucency in both groups, some previous studies confirmed this findings as Eldwakhly et al⁽⁴²⁾ who evaluated color stability and translucency of different restorative CAD/CAM materials before and after being subjected to different staining solutions (cola, coffee and ginger solutions). Glass ceramic (E-max), high-translucency zirconia (lavaTM Plus), resin nanoceramic (Lava Ultimate), and hybrid ceramic (VITA ENAMIC) CAD/CAM blocks, it was found that staining significantly affected the color ΔE of all specimens. No significant differences in ΔTP were found between different staining solutions.

Kurt et al⁽⁴³⁾ evaluated the effects of accelerated artificial aging on the translucency and color stability of monolithic ceramics with different surface treatments. ΔE values of the groups exceeded the clinically acceptable level and TP values were not affected by the surface treatment in either material and decreased after aging.

According to the previous results the hypothesis of this study was partially accepted as there was significant change in surface roughness with pH media on PEEK group and non significant change with Vita Enamic group. Significant change in color stability with PEEK and non significant with Vita Enamic. Different pH media did not affect the Translucency in both groups

CONCLUSION

Within the limitation of this study, it concluded that:

1. Vita Enamic can maintain smoother surface than PEEK with different pH media.
2. Vita Enamic can maintain color stability more than PEEK with different pH media.
3. Vita Enamic has higher translucency than PEEK.
4. Prolonged exposure of polymer materials to weak acids can cause increase in surface roughness, while prolonged exposure to strong acids can decrease surface roughness of the material.

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