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Evaluation of colour stability and surface roughness of three CAD/CAM materials (IPS e.max, Vita Enamic, and PEEK) after immersion in two beverage solutions: An *in vitro* study

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Abstract
Objectives: To evaluate the colour stability and surface roughness of 3 different CAD/CAM materials (IPS e.max, Vita Enamic, and PEEK) after immersion in two beverage solutions (Coffee, Coca-Cola).
Methodology: Samples from a lithium disilicate glass-ceramic (IPS e.max CAD), a hybrid ceramic (Vita Enamic), and PEEK (BioHPP) were evaluated for colour change and surface roughness under immersion in two beverage solutions (Coffee, Coca-Cola). A total of 30 samples were prepared n=10 each. Spectrophotometer analysis was used to measure colour parameters (a*, b*, c*) before and after the immersion cycles and the average colour difference (Delta E*) was calculated after immersion in different beverages. Surface roughness was measured with a non-contact profilometer. Statistical analysis was performed using the Kruskal Wallis test with the Mann Whitney test as posthoc. The significance level was set at $P \leq 0.05$.
Results: There was a significant colour change between the materials. Vita Enamic and PEEK samples showed the highest colour change value with coffee immersion (ΔE) (7.96± 3.86), (6.07± 3.12) respectively. While IPS e.max. CAD revealed the lowest colour change value (2.62 ±1.54) following coffee immersion with a statistically significant difference in between ($P < 0.011$). After immersion in different media, there was a significant difference in surface roughness between the materials ($p < 0.05$).
Conclusion: Vita Enamic and PEEK showed colour change beyond the clinically acceptable level, while IPS e.max CAD was the most stable material in colour and surface roughness.

Keywords: Colour stability, surface roughness, Vita Enamic, PEEK, beverage solutions

Introduction

The advent of Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) and the increase in patients' aesthetic demands have resulted in the creation of a variety of dental ceramic materials [1].

Colour stability is considered an important factor for the success and longevity of aesthetic restorations. In addition, the surface texture of a tooth-coloured restoration influences plaque accumulation, discolouration, and aesthetic appearance [2].

Despite the great mechanical properties of new ceramic restorations, they are still susceptible to change in colour and surface roughness, either due to external or internal factors. The oral environment is moist, warm and undergoes tremendous changes in PH due to different exposures to extrinsic factors such as caffeine-containing beverages, coloured beverages. The degree of discolouration depends mainly on the material composition, type of solution and exposure time [3].

Glass-ceramics have been widely used due to their excellent aesthetics and natural appearance. However, they have been shown to have limitations such as their low tensile strength, which leads to fracture owing to brittleness [4]. As a result, CAD/CAM hybrid ceramics have been created as an alternative to glass-ceramics [5].

Vita Enamic was introduced in 2013, it is a CAD/CAM hybrid ceramic manufactured by VITA Zahnfabrik. It is a dual network structure composed of a dominant ceramic matrix (Feldspar) infiltrated by polymer (UDMA and TEGDMA).

This material acquires the characteristics of both ceramic and composite; with the high strength and resistance to wear of ceramics and the elasticity of composite that makes the material resistant to fracture [6]. Vita Enamic is considered to have mechanical properties close to natural teeth [7].

Another recent CAD/CAM material came into the market called Polyetheretherketone (PEEK). PEEK comes from the Polyaryletherketones (PAEK) family. It is a thermoplastic semi-crystalline polycyclic aromatic polymer, which consists of two ether groups and one ketone group between aryl rings. PEEK was found to have many unique features such as biocompatibility, strength, wear resistance, and thermal and chemical stability [8]. Moreover, it has a unique colour which is greyish to brown in its pure form. Therefore, many scientists started to add fillers such as ceramic and titanium oxides to improve their colour and mechanical properties [2, 9].

BioHPP (High Polymer Performance) (Bredent GmbH Senden, Germany) is a modified PEEK form that has recently been widely used in fixed and removable dental prosthesis frameworks. Up to 30% of the material is made up of ceramic fillers ranging in size from 0.3 to 0.5 microns. BioHPP material has several advantages, including low density, biocompatibility, mechanical properties similar to enamel and dentin, and the ability to be layered with a composite material. Therefore, it is a good option for patients who suffer from bruxism or clenching because it prevents the antagonist from wearing out and the prosthesis from breaking [10].

However, there are not enough data currently in the literature comparing the effect of beverage solutions on the colour stability and surface roughness of hybrid ceramic and PEEK.

Therefore, the first null hypothesis was that there will be no difference in colour stability of the three CAD/CAM materials (IPS e.max CAD, Vita Enamic, and PEEK) after immersion in two beverage solutions (Coffee- Coca-Cola).

The second null hypothesis was that there will be no difference in Surface roughness of the three CAD/CAM materials (IPS e.max CAD, Vita Enamic, and PEEK) after immersion in two beverage solutions (Coffee- Coca-Cola).

Materials and Methods

Study Design

Three different materials, IPS e.max CAD; (Ivoclar Vivadent AG) with shade A2 LT and block size 14, hybrid ceramic (Vita Enamic, VITA Zahnfabrik) with shade 2M2-T and block size 14, PEEK (brecam BioHPP; bredent) with a white opaque shade layered with A2 composite, were used (n=10 each) to test their colour stability and surface roughness after immersion in coffee or Coca-Cola for 3 weeks. Summary of materials used and their composition is seen in Table (1).

Sample preparation and distribution

A total of thirty samples (10 X 14) mm in dimensions for IPS e.max CAD and Vita Enamic and (10 x 10) mm in dimension for PEEK in 1mm thickness were designed and constructed in a standardized manner. Samples were divided into three main groups (n=10) each, according to the type of material used. Group I: IPS e.max CAD, Group V: Vita Enamic, and Group P: PEEK. Each group was further subdivided randomly into two equal subgroups (n=5) each, according to the immersion media. C: Coffee, and L: Coca-Cola.

PEEK (breCAM.BioHPP) (Bredent GmbH &Co.KG, Senden, Germany) being supplied by the manufacturer in the form of blanks, so to make a block, autodesk Meshmixer software was used to design a 3D shape resembling a block of 16 mm length and (10 x10) in dimensions, which was then saved in STL file format and sent to the CAD/CAM 5-axis milling machine (SHERA Eco-mill 5X, Bimedis, Germany). The milling process was performed under a copious amount of water irrigation. After the completion of the milling process, the milled BioHPP block was separated from the blank with a diamond cutting instrument.

The block of each material was then sliced into square-shaped samples of 1mm thickness using the electric isoMet 4000 microsaw (Buehler, USA). Cutting was done using Buehler diamond disc (Renfert GmbH, Germany) at a speed of 2500 rpm under water coolant and a feeding rate of 13.7 mm/min. A digital caliper (6"150 mm stainless steel, micrometer gauge, tricicle, China) was used to verify the thickness after each cutting process.

Table 1: materials used in this study:

Materials (Commercial names)	Type/description	Chemical composition (In wt%)		Manufacturer
IPS e.max CAD blocks	Lithium disilicate glass-ceramic shade LT A2 size: C14	SiO_2	57.0-80.0	Ivoclar Vivadent, Schaan, Liechtenstein. Canada
		Li_2o.	11.0-19.0	
		K_2o	0.0-13.0	
		P_2O_2	0.0-11.0	
		ZrO_2	0.0-8.0	
		ZnO	0.0-8.0	
		other	0.0-10.0	
		colouring oxides	0.0-8.0	
Vita Enamic CAD/CAM blocks	Innovative hybrid ceramic shade: 2 M2-T size: EM-14	86% fine-structure feldspathic ceramic (58-63) SiO_2		VITA Zahnfabrik, Spitalgasse 3. Germany
		AL_2O_3	20-23	
		NaO_2	9-11	
		K_2O,	4-6	
		ZrO_2	0-1	
		14 polymer: (urethane dimethacrylate) UDMA, (triethylene glycol dimethacrylate) (TEGDMA)		
PEEK blanks breCAM.BIOHPP blank	High performance polymer modification of the PEEK Polymer	Partly crystalline poly ether ether ketone (PEEK) that is strengthened using 20% ceramic filler with a grain size between 0.3 to 0.5 µm		Bredent, Senden, Germany
Visio.link primer	Light cured primer for PMMA and composite which may also be used intraorally for bonding resin cement to BioHPP PEEK copings	Mixture of MMA, PETIA, dimethacrylates, photoinitiators and stabilizer used for bonding of composite to BioHPP.		

Crea.lign Opaquer	The dual opaquer ensures complete hardening at the bonding surface and provide shade reliability. Together with Visio.link primer, it forms optimum bonding.		
Crea.lign dentin	Composite resin Shade: A2, available in gel and paste form with particle size of 40 nm. Prior to polishing, the average surface roughness is approximately Ra 0.05 µm.	50% opalescent ceramic filler and high strength oligomer matrix.	
Artificial saliva	Artificial saliva	2.2 g/L gastric mucin, 0.381 g/L sodium chloride, 0.231 g/L calcium chloride, 0.738 g/L potassium phosphate, 1.114 g/L potassium chloride, 0.02 sodium azide, trace of sodium hydroxide. PH=7.0.	Biochemistry lab, faculty of medicine Cairo university, Egypt
Coffee	Nescafe Gold Instant soluble freezes dried coffee	Caffiene, theobromine, theophylline, thiamine, xanthine, tannin, tannic acid, citric acid, chlorogenic acid, spermidine, acetaldehyde, spemine, scopoletin and phenols. PH-5.8	Nestle, Switzerland
Coca-Cola	Coca-Cola beverage	Carbonated water, sugar, phosphoric acid, caramel color, natural flavours, and sodium PH=2.5	Cairo, egypt

Surface treatment of samples

The ten IPS e.max CAD samples were in the pre-crystallized blue stage form. Therefore, a crystallization cycle was done in order to attain their full strength and turn into the selected shade. They were placed in the summit press furnace (IBEX dental technology, USA) and fired on a crystallization tray (SUMMIT Press furnace; IBEX Dental Technologies, USA). For the crystallization cycle, samples were pre-dried under vacuum at 403° C for 2 minutes then the heating temperature was increased at a rate of 50° C/min until a temperature of 850° C was reached and held for 9 minutes to complete the crystallization of the IPS e.max CAD. After crystallization has been completed, the furnace was opened and the samples were left inside the furnace on the tray to cool down for 10-15 minutes.

To standardize the veneering process of PEEK (BioHPP) samples with composite resin, two samples of 1mm PEEK were used to create a mould of 2 mm depth, which was made by mixing a putty and catalyst of silicone impression material (Zetaplus, Zermakh) according to the manufacturing instructions. The impression was then left until its complete setting. After a full set, one of the PEEK samples was removed to be compensated with a uniform layer of composite (crea.lign) of 1mm in thickness (Figure 1).

In order to enhance the adhesion between veneering composite and PEEK (BioHPP), the samples were first sandblasted with 110 µm AL_2O_3 particles using oxyker duet micro-sandblaster (Fili Manfredi, Italy) at 0.25 MPa, at an angle of 45°, and from a distance of 3 cm for 5 seconds until the surface appeared matt. The samples were then conditioned with a visio.link adhesive (bredent, Germany), which was applied with a brush and the excess was removed until no shiny surface appeared, then was cured for 90 seconds by a special light polymerization device (Bre-lux Power Unit, 2, bredent, Germany) at wavelength: 370-500nm, and intensity: 220 mW/cm^2. This was followed by the application of crea.lign opaker 2, which was then cured for 180 sec. Finally, crea.lign dentine A2 composite was injected through the syringe into the mould. A circular plastic sheet was then placed over the composite to ensure a uniform, flat surface with no voids, and it was cured for 5 seconds to stabilize it. The sheet was then removed and final polymerization was done for 360 seconds with the light polymerization device, according to manufacturing instructions.

For standardization, finishing and polishing were done by the same operator, till a flat surface was achieved which was necessary for colour parameters measurement so as to allow the contact tip of the spectrophotometer to contact the surface without any angulation. IPS e.max CAD samples were polished by using a cotton puff wheel brush. The polishing manner was performed in a circular motion from the centre to the periphery of the samples in one direction. A glaze was added on one surface (clear glaze, Cerabien ZR, FC, Kuraray) and fired on a furnace tray (SUMMIT Press furnace; IBEX Dental Technologies, USA) according to manufacturing instructions. Vita Enamic samples were manually polished by Vita Enamic Polishing Set technical (VITA Zahnfabrik) according to the manufacturer instructions. It is a two-step polishing system. First, the pre-polishing pink silicon carbide discs were used at 10,000 rpm using a micromotor, followed by the high gloss polishing grey discs of the technical kit at 5000 rpm as suggested by the manufacturer. The veneering composites over the BioHPP samples were polished by visio.lign polishing Tool kit (bredent, Germany) using carbide tungsten mills, bims goat brushes, cotton puff wheel brushes and polishing paste (acrypol) to ensure a perfect lustrous finish.

Fig 1: The mould for PEEK layering with composite

Samples Immersion

The investigator under supervision assigned all steps of samples selection, randomization and preparation. Blinding was done by the technician (single-blind). Randomization was generated using Excel software with an allocation ratio of 1:1 to different groups. All the samples were immersed in 20 ml of artificial saliva for 24 hours, prior to assessment (baseline). The samples were then removed from the artificial saliva using a tweezer and blotted dry using tissue paper. The colour of each sample was assessed using a spectrophotometer (Model RM200QC, X-Rite, Neu-Isenburg, Germany), then the surface roughness was assessed using a 3D non-contact optical profilometer (Scope Capture Digital Microscope, Guangdong, China).

A 5g Nescafe Gold coffee powder (Nestle, Switzerland) was dissolved in 200 mL boiling distilled water according to the manufacturer's instructions. After 10 minutes of stirring, the solution was filtered through a filtered paper.

A plastic syringe was then used to extract 1mL of coffee to fill up fifteen glass containers, five samples from each group were immersed separately in a firmly closed glass container. Samples were kept at a constant temperature at 37°C in an incubator (CBM.Torre Picenardi (CR), Model 431/V, Italy) for 3 weeks, which was stated to be equivalent to drinking 1 cup of coffee per day for 21 months [11].

The solutions were shaken every 3 hours to provide homogeneity, Coffee solutions were changed regularly every day to avoid their fermentation and deposition. After 3 weeks, samples were then removed by a tweezer, washed under runny distilled water, and dried using tissue paper. A Coca-Cola bottle (Cairo, Egypt) of 250 mL were used in this study. A plastic syringe was used to extract 1 mL of Coca-Cola to fill up fifteen glass containers, five samples from each group, which were immersed separately in Coca-Cola in a firmly closed glass container. Samples were stored at 37° ± 1° C in an incubator for 3 weeks, which simulate the consumption of this beverage solution for 21 months [11]. Tested solutions were changed every day, then after 3 weeks, all samples were removed by a tweezer and washed under runny distilled water and dried using tissue paper.

Colour measurement

The colour of all the samples was measured using a Reflective spectrophotometer (Model RM200QC, X-Rite, Neu-Isenburg, Germany). The aperture size was set to 4 mm and the samples were positioned in the centre of the measuring port. A white background (CIE L*= 88.81, a*= -4.98, b*= 6.09) was selected and measurements were made according to the CIE L*a*b* colour space relative to the Commission Internationale de l'Eclairage (CIE) standard illuminant D65, where L* refers to the degree of lightness (0-100), a* to the colour on the red/green axis and b* to the colour yellow/blue axis. The spectrophotometer was calibrated before each measurement. Three measurements were taken for each sample from the middle in a triangle shape and the average was recorded.

The colour changes (ΔE) of the samples were evaluated using the following formula:

$\Delta E = [(L^* \text{ after staining} - L^* \text{ baseline})^2 + (a^* \text{ after staining} - a^* \text{ baseline})^2 + (b^* \text{ after staining} - b^* \text{ baseline})^2]^{1/2}$. Many articles have referenced the acceptable threshold of ΔE=3.3 which was used as a limit in this study in which 50% of the human eyes would receive it as a clinically unacceptable (Quek 2018; Sarikaya and Yerliyurt 2018)

Where: L* = lightness (0-100), a* = (change the colour of the axis red/green) and b* = (colour variation axis yellow/blue).

Surface roughness measurement

For quantitative characterization of surface Topography, a 3D non-contact optical profilometer was used (U500X, Digital Microscope, Guangdong, China). All samples were positioned perpendicularly into a digital microscope. All thirty Samples were photographed using a USB Digital microscope with a built-in camera connected with an IBM compatible personal computer using a fixed magnification of 90X. 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 the area of roughness measurement.

The cropped images were analyzed using WSxM software. Within the WSxM software, all limits, sizes, frames and measured parameters were 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. 3D images were collected for each sample, both in the central area and in the sides at areas of 10 µm × 10 µm. Each sample was imaged twice, after storage in saliva and after immersion in beverage solutions (coffee, Coca-Cola). WSxM software was used to calculate average roughness expressed in µm which could be assumed as a reliable index of surface roughness.

Statistical Analysis

Data were statistically described in terms of mean and standard deviation (± SD), or median and range when appropriate. Numerical data were tested for the normal assumption using Shapiro Wilk test. Comparison between the study groups was done using Kruskal Wallis test with Mann Whitney test as a posthoc multiple 2-group comparison after applying the Bonferroni adjustment of multiple comparisons.

Within-group comparison was done using Wilcoxon signed rank test for paired (matched) samples. Correlation between various variables was done using Spearman rank correlation equation for non-normal variables/non-linear monotonic relation.

Two-sided p values less than 0.05 was considered statistically significant. All statistical calculations were done using the computer program IBM (IBM Corporation, NY, USA) SPSS (Statistical Package for the Social Science; IBM Corp, Armonk, NY, USA) release 22 for Microsoft Windows.

Results

For samples immersed in Coffee, the highest colour change values were seen in Vita Enamic with ΔE (7.96± 3.86), followed by PEEK with ΔE (6.07±3.12), while IPS e.max CAD showed the lowest colour change values ΔE (2.08±0.83) with a statistically significant difference in between them ($P \leq 0.05$) (Table 2, Figure 2).

While for samples immersed in Coca-Cola, higher colour change values were seen in Vita Enamic and PEEK (4.30±1.18), (5.28±2.55) respectively, than those observed in IPS e.max CAD (3.15±1.98) with no statistically significant difference in between them (Table 2, Figure 2).

Vita Enamic and PEEK showed lower colour change values with Coca-Cola than with Coffee, while IPS e.max CAD revealed higher colour change values with Coca-Cola than with Coffee with no statistically significant difference between them (Table 2, Figure 2).

For surface roughness, before the immersion in beverage solutions, there were no statistically significant differences between the surface roughness of different ceramic types. After immersion in beverage solutions, there was a statistically significant difference in surface roughness between the materials (p≤ 0.05). IPS e.max CAD showed the lowest (Ra) values (0.256±0.002), Whereas Vita Enamic showed the highest (Ra) values (0.259±0.003) followed by PEEK (Ra) value was (0.275±0.002) (Table 3, Figure 3)

There was a statistically significant direct (positive) correlation between colour changes (ΔE) and surface roughness (Ra) regardless of the material and beverage solution type. Where the increase in colour change was associated with an increase in surface roughness and vice versa (Table 4).

The roughness of the three tested samples surfaces was imaged and then interpreted as peaks and valleys using a USB Digital microscope, where the increased values indicated higher roughness (Figure 4, 5, 6, 7, 8, 9, 10, 11, 12).

Table 2: The mean, standard deviation (±SD) values and results for Comparison of (ΔE) of different interactions.

Beverage Solution	IPSe. max CAD		Vita Enamic		PEEK		p value
	Mean	±SD	Mean	±SD	Mean	±SD	
ΔE of coffee	2.08	0.83	7.96	3.86	6.07	3.12	0.011*
ΔE of Coca Cola	3.15	1.98	4.30	1.18	5.28	2.55	0.101
p value (between beverage solutions)	0.465		0.117		0.754		

Table 3: The mean, standard deviation (±SD) values and results of Comparison of surface roughness (Ra, µm) between the three different materials regardless of the beverage solution type:

	IPS e.max CAD		Vita Enamic		PEEK		p value
	Mean	±SD	Mean	±SD	Mean	±SD	
Surface roughness before Immersion in two beverage solutions	0.254	0.003	0.256	0.002	0.257	0.002	0.107
Surface roughness after immersion in two beverage solutions	0.256	0.002	0.259	0.003	0.257	0.002	0.026*

Table 4: Results of Pearson's correlation coefficient for the correlation between colour changes and surface roughness

Correlation coefficient	P-value
0.366	0.047*

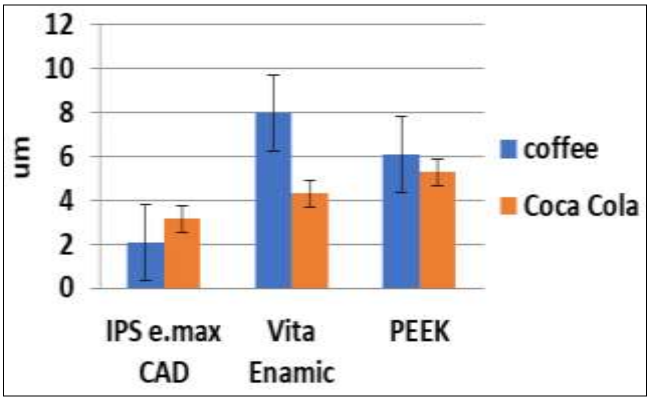

Fig 2: Bar chart showing the mean ΔE of different interactions

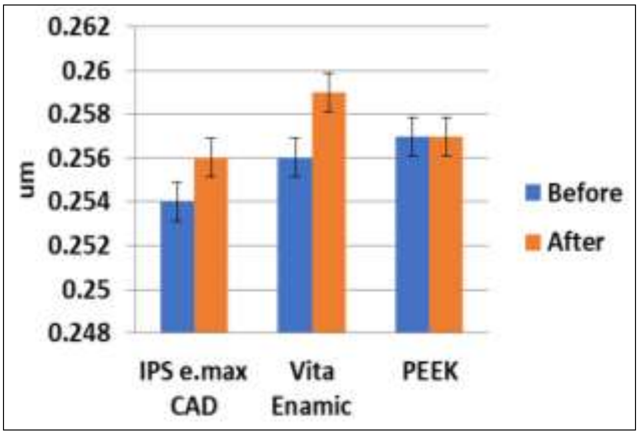

Fig 3: Bar chart showing the mean of surface roughness (Ra, µm) between the three different materials regardless of the beverage solution type

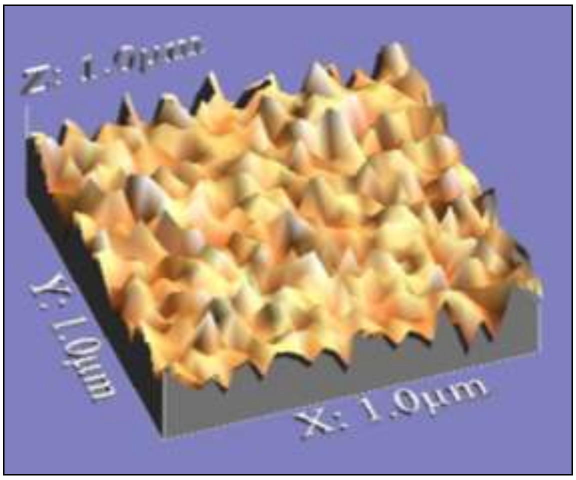

Fig 4: 3D non-contact optical profilometer interference microscope showing topographic micrograph of IPS e.max CAD sample before immersion in coffee and Coca-Cola.

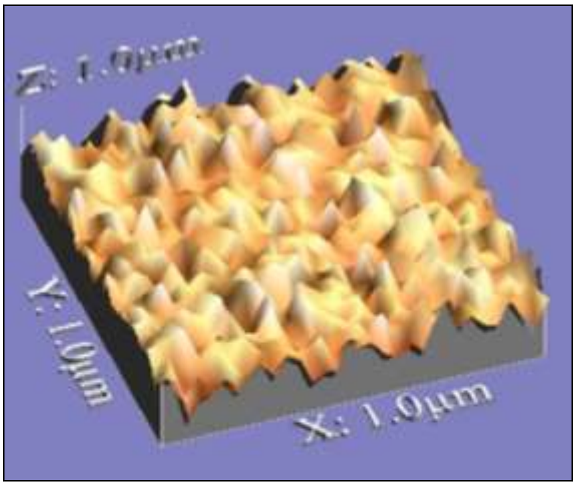

Fig 5: 3D non-contact optical profilometer interference microscope showing topographic micrograph of IPS e.max CAD sample following Coffee immersion.

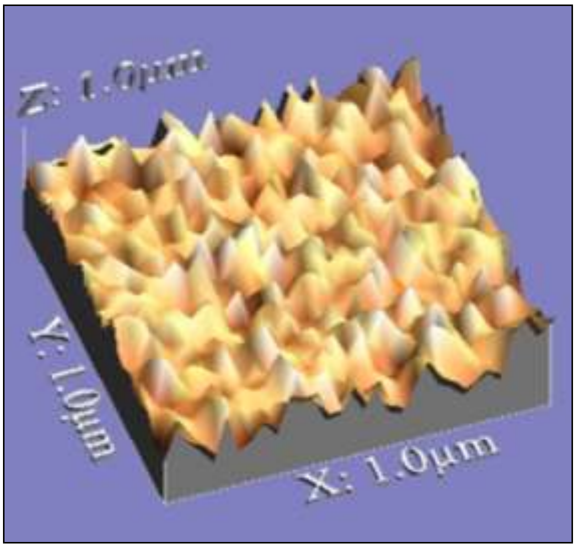

Fig 6: 3D non-contact optical profilometer interference microscope showing topographic micrograph of IPS e.max CAD sample following Coca-Cola immersion.

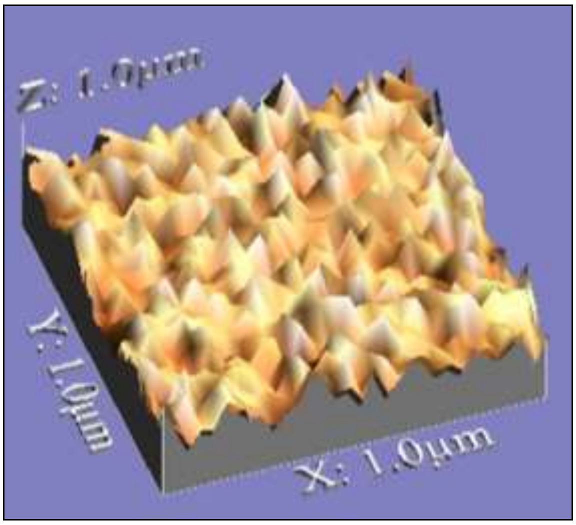

Fig 9: 3D non-contact optical profilometer interference microscope showing topographic micrograph of Vita Enamic sample following Coca-Cola immersion

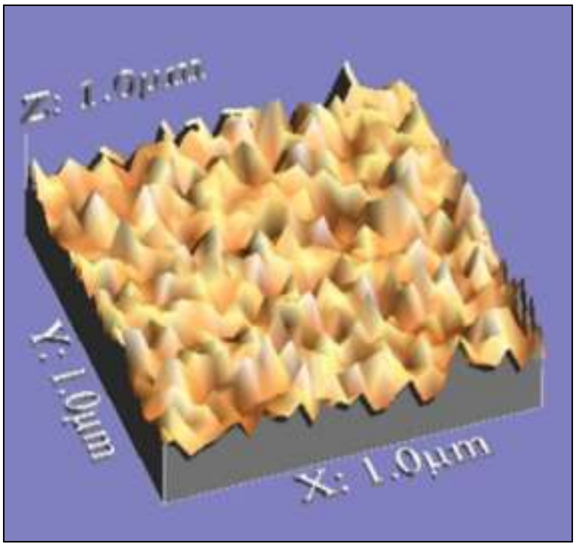

Fig 7: 3D non-contact optical profilometer interference microscope showing topographic micrograph of Vita Enamic sample before immersion in coffee and Coca-Cola solutions

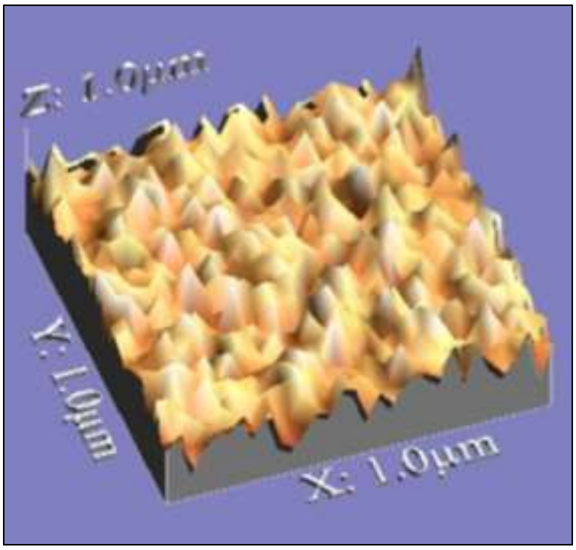

Fig 10: 3D non-contact optical profilometer interference microscope showing topographic micrograph of PEEK sample before immersion in coffee and Coca-Cola

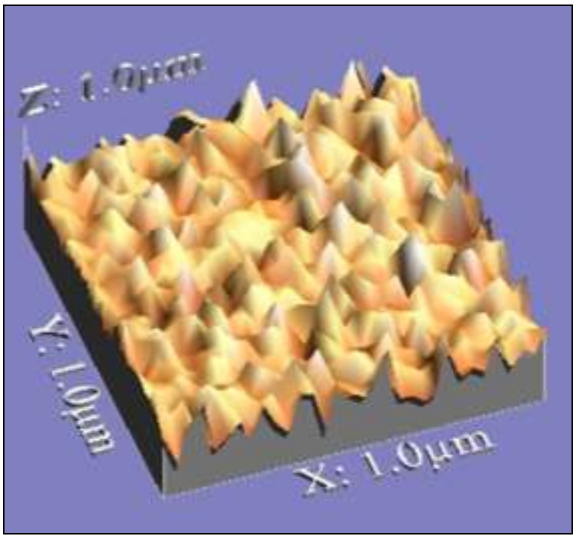

Fig 8: 3D non-contact optical profilometer interference microscope showing topographic micrograph of Vita Enamic sample following Coffee immersion

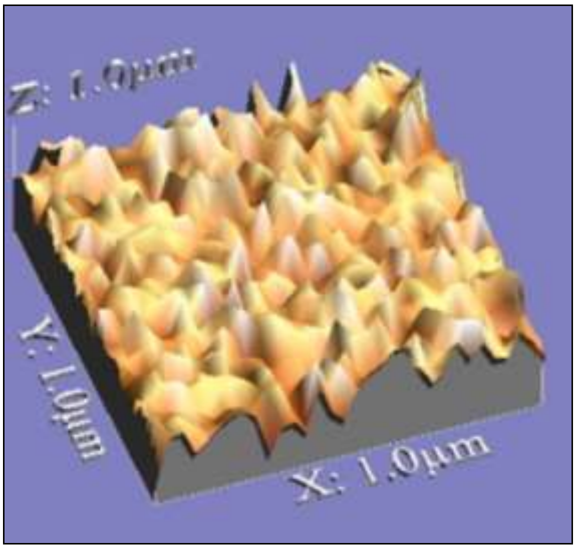

Fig 11: 3D non-contact optical profilometer interference microscope showing topographic micrograph of PEEK sample following Coffee immersion

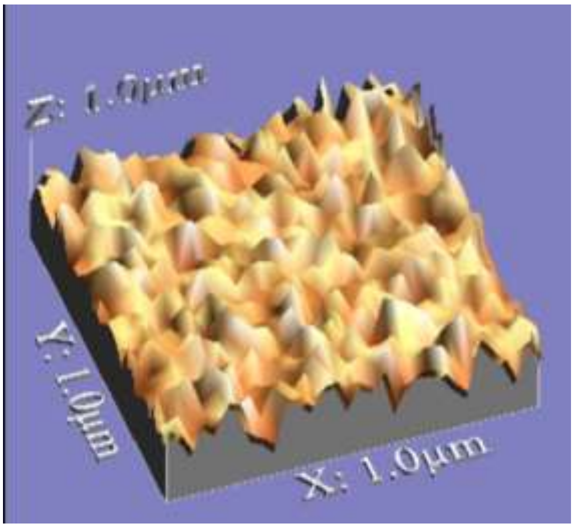

Fig 12: 3D non-contact optical profilometer interference microscope showing topographic micrograph of PEEK sample following Coca-Cola immersion

Discussion

Colour stability is considered to be an important aspect in the success or failure of any restoration. With today's advanced technology, the need for long-lasting aesthetic restoration has grown and is becoming a demand among patients [13].

Over the last decade, there has been a vast development of new CAD/CAM materials that have proven their efficacy. Therefore, their optical behaviour must be well investigated in order to meet patients' high aesthetic demands [14].

IPS e.max CAD, Vita Enamic, and PEEK materials were tested in this study for their unique structure and reported advantages.

IPS e.max CAD shade LT A2 size C 14 and Vita Enamic shade A2 T size 14 were selected as these shades are the most commonly used in crowns fabrication. In addition, BioHPP, which is white and opaque, had to be layered with a composite. Shade A2 was selected to match the other materials used.

Coffee and Coca-Cola were selected in this study being the most commonly consumed acidic beverages by individuals. Coffee is a mild acid with PH=5.8, composed of caffeine, tannic acid, citric acid, chlorogenic acid, while Coca-Cola is a strong acid with PH=2.5 composed of phosphoric acid, carbonated water, sugar, and caramel colour [15, 16]. Dental restorative materials when exposed to the oral cavity may develop a change in colour and surface roughness, which is caused by a variety of reasons, including the type of the material, surface finishing, patient's dietary habits and oral hygiene. Discolouration of dental materials due to the effects of strong coloured beverages such as coffee and Coca-Cola may affect the aesthetic outcome and result in patient dissatisfaction with the restorations [3, 17, 18]. Low PH acidic beverages might also deteriorate the material surface integrity leading to increased surface roughness, wear, and subsequent discolouration [19].

Therefore, this study aimed to evaluate the effect of two beverage solutions (coffee, and Coca-Cola) on the colour stability and surface roughness of three aesthetic materials, IPS e.max CAD, Vita Enamic, and PEEK.

The present analysis is an *in vitro* study, which offered a controlled and standardized method of fabrication and prevented different natural tooth variables, which is essential to provide information closer to that of the clinical situation [20].

The IPS e.max CAD, Vita Enamic, and the constructed PEEK blocks were cut by isoMet system which is a low-speed sectioning machine that cuts in precision different types of materials with minimal deformation and waste [1].

For this study, the thickness of IPS e.max CAD, Vita Enamic, and PEEK was determined to be 1mm, which was the minimum recommended thickness for these materials to be used in fixed restorations. While the dimensions were (10 x14) and (10 x10) mm, which allowed the testing machine to record the readings easily [15].

A digital calliper was used to verify the thickness of each sample in order to eliminate any variables that could affect the final results of colour change and surface roughness [17].

Surface roughness and colour stability are highly integrated. Rougher surfaces tend to attract more bacterial biofilms and stains [7]. Many studies have emphasized the effect of the polishing materials and surface finishing procedures on the colour change and surface roughness of the material. Therefore, in this study polishing of the samples were done according to manufacturer instructions of each company to attain a smooth surface, and was performed by the same technician for standardization purposes to achieve flat surfaces [17]. For IPS e.max CAD samples, glazing was performed according to the manufacturing instructions. The samples were fired in a furnace using a firing tray that stored heat and provided tension-free cooling of the glass-ceramic. It has been shown that glazing generated smoother surfaces than polishing with glass-ceramics [21].

For Vita Enamic samples, polishing set technical kit was used, which was developed by (VITA Zahnfabrik) specifically for polishing Vita Enamic. It has been investigated and proved to attain a smoother material surface instead of glazing [22].

BioHPP (PEEK samples) were sandblasted with AL_2O_3 particles to increase the surface area for bonding, and MMA-based adhesive (visio.link) was applied, which has been shown to provide the strongest chemical bond with PEEK. Since PEEK is not an aesthetic material, it had to be layered with nano-composite material (crea.lign), which enhanced the aesthetic properties and stain resistance. Polishing of composite was done with a Visio.lign tool kit polishing kit and acrypol polishing paste to ensure a perfect finish and a surface with a high plaque resistance [23, 24].

This study measured colour difference and surface roughness after 3 weeks of immersion in coffee and Coca-Cola. It has been documented in the literature that one week of immersion in a laboratory is equivalent to seven months of drinking one cup of coffee and Coca-Cola a day. In the present study, immersion for 3 weeks, which was equivalent to 21 months of beverage consumption in a clinical situation [11, 25].

Prior to immersion in beverage solutions**, S**amples were immersed for 24 hours in artificial saliva to clean the samples and simulate the neutralizing effect of saliva [26]. The coffee solution was prepared and stirred for 10 min afterwards to ensure a homogenous solution, and then filtered through a filter paper to remove any residues [27]. Coffee solutions were shaken every 3 hours to avoid precipitation and renewed every 2 days to avoid yeast fermentation of coffee [15].

Coca-Cola solution was also changed regularly every two days to provide a freshly mixed solution. All samples were kept in an incubator at 37º C to simulate and maintain the optimal temperature of the oral cavity [26].

In the present study, a spectrophotometer was used for the measurement of colour difference as it has been shown to be more accurate than other shade-taking methods because it is reproducible and eliminates subjective errors. A Spectrophotometer can detect a colour difference of 1 ΔE, while visual methods detect a colour difference of 3.3 ΔE [28].

There is a consensus among the studies that the clinical acceptability threshold of ΔE ranges from 2 to 4. Many articles have referenced the acceptable threshold of ΔE=3.3, which was used as a limit in this study where 50% of human eyes would receive it as clinically unacceptable [7, 12].

We used the classical CIELab formula ΔE_{ab} in obtaining the colour difference as it is the oldest and most commonly used method in studies [29].

Surface roughness was measured using a 3D non-contact optical profilometer interference microscope, which has been shown to be preferable and more reliable in quantitative surface topographic analysis. It is also connected to a camera that captures a 3D surface texture image of the whole sample, resulting in a precise qualitative representation of the samples [30, 31].

Results showed that IPS e.max CAD, Vita Enamic, PEEK, and beverage solutions had a significant effect on colour change values.

After immersion in coffee solution, results revealed a statistically significant difference between the three materials in colour change ($P \leq 0.05$). IPS e.max CAD was the most colour stable material (ΔE= 2.08), which was below the clinical acceptance level (ΔE=3.3). On the contrary, Vita Enamic and PEEK samples revealed high colour change values when immersed in Coffee. The mean ΔE values of Vita Enamic was = 7.96 and for PEEK was = 6.07. This was considered to be above the clinically acceptable limit.

Therefore, the first null hypothesis that there will be no difference in colour change of the three CAD/CAM materials (IPS e.max, Vita Enamic, and PEEK) after immersion in Coffee was rejected.

Coffee showed a high stain impact on resin containing materials (Vita Enamic and PEEK in this study), which could be attributed to the presence of yellow pigments with low polarity facilitating its penetration into the resin matrix [15]. These findings were in agreement with Eldwakhly *et al.* and Stamenkovic *et al.* [14, 18] who proved that the greatest colour change of hybrid ceramics and nanocomposites were seen with coffee beverages, and the least colour changes were seen with lithium disilicate materials and they related their findings to the microstructure of the material.

In the present study, the high discolouration of Vita Enamic with coffee could be attributed to its composition, being a porous ceramic structure infiltrated by a polymer [17]. The polymer tends to absorb water and pigments from the highly discoloured chromogenic food. Studies found the TEGDEMA component of the polymer in Vita Enamic is the one responsible for colour absorption due to its hydrophilic nature. Moreover, another explanation for the high stainability of Vita Enamic might be related to the effect of acids in wearing down the polymer content and leaving a rough surface of ceramic, which increased its staining liability [12, 18].

The high Color change of PEEK (BioHPP) might also be due to the water absorption nature of the veneering composite material, as it is composed of BISGMA and TEGDMA, which shows high hydrophilicity [32]. This was in agreement with Hussein *et al.* [33] and Badran *et al.* [34] who proved the stainability of PEEK (BioHPP) layered with the crea.lign composite and they related their findings to the composite hydrophilicity. However, PEEK exhibited lower colour difference values than Vita Enamic in this study. This might be attributed to the composition of the layering composite used (crea.lign) (Bredent, Germany) as it contained 50% nano-ceramic fillers with a 40 nm filler size that enhanced its aesthetic properties and stain resistance [32]. This was in agreement with Aydin *et al.* [35] and Ziada and Beleidi [32] who revealed that the colour stability of composite is affected by the filler type and resin matrix

The results of this study were in disagreement with Acar *et al.* [37] who compared the effect of coffee staining on IPS e.max CAD, Vita Enamic, Lava Ultimate, and nanocomposites at different thicknesses (0.5 mm, 1 mm, and 1.2 mm). They found that Vita Enamic revealed a clinically perceivable but acceptable colour change following IPS e.max CAD, while nanocomposite showed an unacceptable colour change when stained with coffee. This could be due to the short time period of testing the materials and the different thicknesses they used. Staining decreased with polymers when the thickness increased to 1.2 mm.

After immersion in Coca-Cola solution, results revealed no statistically significant difference between the three materials ($p \geq 0.05$). IPS e.max CAD showed the lowest colour change (ΔE=3.15), which remained below the clinically acceptable level. While Vita Enamic and PEEK revealed a higher colour change. The mean ΔE values of Vita Enamic was = 4.30 and for PEEK was =5.28, which exceeded the clinically acceptable level and represented an unacceptable colour change.

Therefore, the first null hypothesis that there will be no difference in colour change of the three CAD/CAM materials (IPS e.max, Vita Enamic, and PEEK) after immersion in (Coca-Cola) was accepted.

Coca-Cola is a highly acidic beverage but showed lower colour difference values in the present study with resin-containing materials (Vita Enamic and PEEK) than with coffee. This might be attributed to the high polarity of Coca-Cola beverages, which reduced their absorption effect and limited their adherence to the surface and likely being easily removed when washed [36]. In addition, the presence of phosphate ions in Coca-Cola seemed to have similar effect on teeth surfaces [14]. These findings were consistent with many studies Quek *et al.*; Sarikaya and Yerliyurt; Eldwakhly *et al.*; Seyidaliyeva *et al.*; Younes *et al.* [7, 12, 14, 25 ,36] who found that Vita Enamic color change was higher with coffee than in Coca-Cola.

Furthermore, Coca-Cola caused more colour changes in IPS e.max CAD than coffee. This could be related to the composition of IPS e.max CAD, which contained phosphorus pentoxide (P_2O_2) that might have a higher affinity for the phosphoric ions in Coca-Cola [14]. These findings were in agreement with Eldwakhly *et al.* [14] and Abdalkadeer *et al.* [37] who found that Coca-Cola beverages produced more colour changes in porcelain materials than coffee beverages.

The findings of this study were also in accordance with Shetty *et al.* [38] who evaluated the colour stability of crowns obtained by layering composite over zirconia and Polyetheretherketone (PEEK) Copings after thermocycling and immersion in hot and cold beverages for 24 hours. The results showed PEEK to have a high colour stainability, which was attributed to the natural greyish background of the used PEEK and the layering composite's liability to absorb pigments.

The results of surface roughness in the present study demonstrated a statistically significant difference between the three materials after immersion, regardless of the beverage solution type (Table 3, Figure 3). IPS e.max CAD exhibited the lowest surface roughness (0.256μm), while Vita Enamic

and Veneered PEEK revealed higher changes in surface roughness (0.259µm), (0.257µm) respectively.
Therefore, the second null hypothesis that there will be no difference in surface roughness of the three CAD/CAM materials after immersion in two beverage solutions (Coffee-Coca-Cola) was rejected.
Despite statistical significance, the difference was clinically trivial as the values were near the threshold (0.2µm), which was considered to be a clinically acceptable limit for surface roughness. Meaning that if the value has exceeded this threshold it will be prone to plaque accumulation and stain formation [30, 39, 40].
The reason for the low changes in surface roughness could be related to the effect of surface finishing where glazing IPS e.max CAD showed the lowest change in surface roughness, this was in agreement with many studies (Maciel *et al.*; Abu-obaid *et al.*; Aldosari *et al.*) [13, 21, 29] who reported that glazed IPS emax CAD produced smoother surfaces than polished ones.
Polishing Vita Enamic revealed a slight increase in surface roughness, this could be due to the use of Vita Enamic Polishing kit technical recommended by the manufacturer. The results of the present study were in accordance with Ozarslan *et al.* [22] who showed that Vita Enamic Polishing kit technical revealed a lower surface roughness and smoother material surface than the glazed ones.
Contrary to the results of the present study, (Tekçe *et al.*; Abu-obaid *et al.*; Aldosari *et al.*) [13, 30, 29] found that the polishing of Vita Enamic resulted in a higher colour change and surface roughness than glazing. This could be due to different polishing materials and techniques used.
The slight increase in surface roughness of IPS e.max CAD and Vita Enamic after immersion in Coca-Cola and coffee could be also attributed to the effect of acid in degrading the ceramic matrix and exposing rough surfaces [41]. This was in agreement with (Branco *et al.*; Hussein *et al.*) [42, 33] who stated that the increase in surface roughness of ceramics was due to the dissolution of the ceramic material by the effect of acids.
The results of surface roughness were supported by the 3D digital topographical micrographs of the non-contact profilometer. IPS e.max CAD showed a slight increase in the number of peaks and valleys with blunt shaped peaks and shallow valleys with coffee more than in Coca-Cola (Figure 4, 5, 6). Together with Vita Enamic samples, which showed an increase in the number of peaks and valleys after immersion in coffee, with sharp, pointed peaks and deeper valleys, whereas they were more blunt and shallower after immersion in Coca-Cola (Figure 7, 8, 9).
Polishing PEEK with visio.lign toolkit and acrypol polishing paste has caused no change in surface roughness. This is because it ensures a perfect finish with high plaque resistance, identical to ceramics as claimed by the manufacturer [23].
The topographical 3D digital micrographs of PEEK samples showed a slight increase in the number and length of peaks with few pointed edges together with a slight increase in the depth of valleys after coffee and Coca-Cola immersion (Figure 10, 11, 12).
The results of Pearson correlation revealed a statistically significant direct (positive) correlation between colour change (ΔE) and surface roughness (Ra) ($p< 0.05$), where the value of the Correlation coefficient was $r = 0.366$ meaning that there is a moderate relation, and this could be explained by the fact that the surface roughness is an important causative factor for the colour change, but not the only factor [21].
One of the limitations of this study, being an *in vitro* study as it didn't reflect the real conditions in the oral cavity and the other affecting factors such as saliva, water, PH level, temperature changes, and occlusion. The flat samples also didn't simulate the 3D shape of the tooth structure with its contours and convexities. Therefore, further *in-vivo* studies for better correlation with clinical settings should be carried out. PEEK digital layering techniques should be also more investigated in terms of colour stability. Further studies are needed to assess the effect of glazing and polishing on the colour stability and surface roughness of ceramic materials.

Conclusion

Within the limitations of this study the following conclusion and clinical recommendation could be drawn:

1. All the tested restorative materials (IPS e.max CAD, Vita Enamic, and PEEK) were susceptible to colour changes after being subjected to coffee and Coca-Cola, and that was related to their microstructure.
2. IPS e.max CAD material exhibited the least change in colour and surface roughness.
3. Vita Enamic and layered PEEK (BioHPP) revealed an unacceptable colour change with coffee and Coca-Cola. Therefore, are not recommended to be used in an aesthetic zone due to their high stain susceptibility, and their use should be limited to posterior teeth, with frequent polishing and finishing to attain a smooth surface.
4. There is a positive correlation between colour changes and surface roughness for the tested restorative materials.
5. Clinicians should consider the dietary habits and oral hygiene of individuals when selecting aesthetic materials
6. Dentists should warn patients about the effects of highly discoloured beverages on hybrid ceramics and composite restorations.

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Conflict of Interest

The authors have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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