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EVALUATION OF COLOR CHANGE AND SURFACE ROUGHNESS OF TWO INDIRECT ESTHETIC MATERIALS SUBMITTED TO DIFFERENT IMMERSION PROTOCOL

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ABSTRACT

Aim: This study aimed to evaluate the color stability and surface roughness of hybrid ceramic and resin composite blocks materials after different immersion protocol.

Methods: Forty samples were prepared and divided into 2 groups according to the type of material. Group A hybrid ceramic (Cerasmart) and Group B composite resin blocks (Brilliant Crios). Samples of each group were subdivided into 4subgroups (n=5) according to immersion protocol. subgroup I artificial saliva, subgroup II artificial saliva and citric acid, subgroup III artificial saliva followed by citric acid and tea without sugar and subgroup IV artificial saliva followed by citric acid and tea with sugar. All subgroups were submitted to the immersion protocol for 28 days. Then samples were tested for color stability and surface roughness measurements before and after immersion by spectrophotometer and stylus profilometer respectively.

Results: For color stability and surface roughness, all subgroups for both groups showed statistically significant mean values comparing before. And after immersion the ΔE and Ra values for group B in all subgroups were significantly higher than group A. The ΔE and Ra values of subgroup III in both groups had the highest mean values.

Conclusion: Immersion protocol associated with tea without sugar showed higher stainability and affect surface roughness more than tea with sugar. Moreover, both materials exceed the clinical acceptance level (ΔE=3.7) of color change.

KEY WORDS: Hybrid ceramics, CAD/CAM composite, Color stability, Surface roughness, Tea.

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INTRODUCTION

Dental ceramics have extensively use in fixed esthetic prosthetic restoration. All ceramic materials were divided into three categories; glass ceramic, polycrystalline ceramics and hybrid ceramics. High mechanical properties, premium esthetic, color stability and excellent biocompatibility are all features of dental ceramics. However, brittleness, high hardness and ability to wear against natural teeth are some of their drawbacks. [1]

Recent advances in digital technology led to widespread use of computer aided design/ computer aided manufacturing (CAD/CAM) in the fabrication of indirect dental restorations. Hybrid ceramic materials have been introduced dentally as they have many benefits such as high flexural strength, modulus of elasticity which is comparable to dentin and the ability to improve strength after bonding. Another newly introduce indirect esthetic restorative material is CAD/CAM composite. When comparing to conventional ceramics, CAD/CAM composite is less brittle and has a lower modulus of elasticity, which is comparable to that of tooth structure. On the other hand, it has low wear resistant and questionable color stability. [1]

Regarding to complicity oral environment, color stability is a key to the long term success of dental esthetic restoration. Changes in color can be caused by exogenous and endogenous factors of material. The staining solutions found in coffee, tea and other beverages are examples of exogenous ones. The chemical composition, crystallinity and Prescence of different phases are examples of endogenous factors [2]. Moreover, color stability is directly related to the surface roughness of the material. Surface roughness is considered as cornerstone in evaluating many surface characteristics as color, hardness, wear and material degradation. Rough restoration surface can influence plaque accumulation, susceptibility to discoloration, abrasion probability of opposing teeth and also reduce degradation resistance of the material. [3] Dental restorations in the oral cavity suffer from continuously changing in stresses, fluctuation in temperature and pH. Artificial aging is used to simulate oral environmental conditions extra-orally and improve time preservation in testing the material over large period of time. Artificial aging can be done by several methods as mechanical, electromagnetic wave, thermal, thermo-mechanical, chemical aging by acidic or alkaline solutions and biological aging by different types of bacteria. [4]

Preservation of surface characteristic and color matching between fixed prosthetic restoration and natural teeth considered one of the important challenged in dentistry. For this reason, this study was conducted to compare the effect of artificial aging on color change and surface roughness of hybrid ceramic and indirect composite resin blocks.

The null hypothesis of this study was that no changes in color stability and surface roughness were observed for both material with different immersion protocols.

MATERIALS AND METHODS

The Minia University Faculty of Dentistry's ethical committee provided approval for this study under protocol number 506/2021.

The materials used in this study were hybrid ceramic blocks (Cerasmart) group A and composite resin blocks (brilliant crios) group B. A total number of 40 samples (n=20 for each group) with dimensions 14X12X1mm were sectioned using linear precision saw machine (IsoMet 4000 microsaw, Buehler, USA). For all samples, finishing and polishing were carried out according to the manufacturer's recommendations.

Each sample in both groups was measured for color stability and surface roughness before immersion protocol by spectrophotometer (Cary 5000 UV-Vis-NIR spectrophotometer from Agilent Technologies, USA) and stylus profilometer

(Portable surface roughness, Surftest SJ-21, profilometer Mitutoyo, USA) respectively.

After measurements, each group was divided into 4 subgroups (n=5) according to type of immersion protocol.

- Subgroup A I and BI (n=5 for each): artificial saliva for 24hr
- Subgroup A II and B II (n=5 for each): 2% citric acid pH=4 for 8hr, followed by artificial saliva for 16hr
- Subgroup A III and B III (n=5 for each): 2% citric acid pH=4 for 8hr, followed by artificial saliva for 13.5hr, followed by tea without sugar for 2.5hr
- Subgroup A IV and B IV (n=5 for each): 2% citric acid pH=4 for 8hr, followed by artificial saliva for 13.5hr, followed by tea with sugar for 2.5hr.

Artificial saliva was prepared from 220.5g calcium chloride powder, 124.2g sodium dihydrogen orthophosphate powder and 11.1g potassium chloride powder. A digital balance (Radwag digital balance, Radom, Poland) was used to weigh each of its parts. Then it was dissolved in 900 ml of distilled water and using a magnetic stirrer (Heidolph MR 3001, Heidolph, Germany), the speed was adjusted to 500 rpm to achieve total dissolution. Using sodium bicarbonate solution as a buffer, the pH of the mixture was adjusted to 7. The solution volume is then increased to 1000 ml using distilled water.

Citric acid was prepared from 20g citric acid powder and 5.32g sodium chloride powder. Citric acid was prepared using the same method as artificial saliva however, the pH of solution was adjusted to pH 4 using sodium bicarbonate solution as a buffer.

Tea was prepared by dipping a 2g sagged of tea into 300 ml of boiling distilled water. And tea with sugar was prepared previously mentioned and 10 g of white sugar was added with the sagged and stirred for 60s. After 1hr, the solution reached to room temperature and the sagged was removed. Then the solution was filtered through filter paper.

All immersion solutions were measured by pH meter (Adwa instruments, Hungary, Romania). All samples were stored in 20 ml of any immersion solution. All samples were washed with copious amount of distilled water for 60s and dried gently between each immersed solution. The solutions were renewed daily for all subgroups.

Color stability measurements

The measurement was done three times for each sample and the mean was recorded as sample reading by the Commission International D' Eclairage (CIE) LAB system using spectrophotometer before and after immersion. Using the following formula, the color difference (ΔE) between the two-color measurements taken before and after immersion was calculated: $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$

Surface roughness measurements

The stylus of profilometer was adjusted 90^0 to the sample surface and the traveling distance 10mm and 4mN measuring force. For each sample, this procedure was repeated three times. The average values of these measurements were considered to be the Ra record of the sample.

RESULTS

Data analysis was performed with the aid of IBM SPSS version 25 statistical package software (IBM SPSS Statistics for Windows, Version 25. Armonk, NY: IBM Corp.; Released 2012). Results were tabulated and statistically analyzed using one way ANOVA test followed by post Hoc LSD. The significance level was set at $P \leq 0.05$.

For color stability measurements (ΔE values). In group A the mean and standard deviation (SD) of

ΔE in subgroups I – IV were 1.05±0.11, 2.83±0.1, 4.46±0.14 and 4.36±0.1 respectively. The highest mean value was in subgroup III (4.46±0.14) and the lowest one was subgroup I (1.05±0.11). Subgroup I and II showed statistically significant mean values comparing to each other and subgroups III and IV.

In group B the mean ± SD of ΔE in subgroups I – IV were 1.24±0.13, 3.11±0.11, 5.69±0.17 and 5.5±0.14 respectively. The highest value was in subgroups III (5.69±0.17) and the lowest one was subgroup I (1.24±0.13). All subgroups showed statistically significant mean values comparing to each other. Regarding the comparison between group A and group B in each subgroup, all subgroups showed statistically significant mean as represented in table (1) and figure (1).

Surface roughness measurement Before immersion (BR)

In group A the mean ± SD of surface roughness BR in all subgroups were ranged between10.33±0.24 to 10.6±0.28 and all means were statistically insignificant to each other regarding each group.

In group B the mean ± SD of surface roughness BR in all were ranged between10.53±0.38 to 10.53±0.38 and all means were statistically insignificant to each other regarding each group.

Regarding the comparison between group A and group B in each subgroup, there was statistically insignificant difference in BR in all subgroups.

After immersion (AR)

In group A the mean ± SD of surface roughness after immersion in subgroups I–IV were 21.27±0.37, 22.13±0.69, 22.27±0.64 and 21.13±0.38 respectively. The highest mean value was in subgroup III (22.27±0.64) and the lowest one was subgroup IV (21.13±0.38). Subgroup IV showed statistically significant mean value to subgroups II and III and statistically insignificant mean value to subgroup I.

In group B the mean±SD of surface roughness after immersion in in subgroups I – IV were 22.93±0.55, 23.67±0.85, 23.53±0.69 and 22.13±0.65 respectively. The highest mean value was in subgroup II (23.67±0.85) and the lowest one was subgroup IV (22.13±0.65).

Regarding the comparison between group A and group B in each subgroup, all means values of group B were statistically significant higher after immersion protocols. For groups and subgroups consistently showed statistically significantly higher mean values of AR comparing to each BR as represented in table (2) and figure (2).

TABLE (1) Comparison of ΔE between different groups and subgroups showing the min, max, mean, and SD.

ΔE		Subgroup I	Subgroup II	Subgroup III	Subgroup IV	P value
		N=5	N=5	N=5	N=5	
Group A	Min– max	0.91-1.23	2.7-2.94	4.29-4.62	4.27-4.54	*<0.001**
	Mean ±SD	1.05±0.11 ^{c}	2.83±0.1 ^{b}	4.46±0.14 ^{a}	4.36±0.1 ^{a}	
Group B	Min -max	1.07-1.39	2.93-3.22	5.51-5.94	5.3-5.67	
	Mean± SD	1.24±0.13 ^{d}	3.11±0.11 ^{c}	5.69±0.17 ^{a}	5.5±0.14 ^{b}	*<0.001**
P value (A vs B)		*0.042**	*0.002**	*<0.001**	*<0.001**	

One Way ANOVA test for quantitative data between the four subgroups, followed by post Hoc LSD analysis between each subgroup.
Superscripts with different small letters refer to significant difference between each subgroup
Independent samples T test for quantitative data between the two groups
**** Significant level at P value < 0.05***

TABLE (2) Comparison of BR and AR between different groups and different subgroups showing min, max, mean, and SD.

		Subgroup I		Subgroup II		Subgroup III		Subgroup IV		P value		P value BR vs AR
		BR	AR	BR	AR	BR	AR	BR	AR	BR	AR	
Group A	Min- max	10-10.67	20.67-21.67	10-11	21.33-23	10-10.67	21.33-23	10.33-11	20.67-21.67	0.428	0.007*	<0.001*
	Mean± SD	10.33±0.24 [a]	21.27±0.37 [b]	10.47±0.38 [a]	22.13±0.69 [a]	10.33±0.2 [a]	22.27±0.64 [a]	10.6±0.28 [a]	21.13±0.38 [b]			
Group B	Min – max	10-11	22.33-23.67	10-11	22.67-24.67	10-10.67	22.67-24.33	10-11	21.33-23			
	Mean ± SD	10.53±0.38 [a]	22.93±0.55 [d]	10.53±0.38 [a]	23.67±0.85 [b,c]	10.4±0.28 [a]	23.53±0.69 [b,c]	10.53±0.38 [a]	22.13±0.65 [c,d]	0.912	0.012*	<0.001*
P value A vs B		0.347	<0.001*	0.789	0.014*	0.694	0.017*	0.760	0.018*			

One Way ANOVA test for quantitative data between the four subgroups, followed by post Hoc LSD analysis between each two groups.
Superscripts with different small letters refer to significant difference between each two subgroups.
Independent samples T test for quantitative data between the two groups
Paired Samples T test for quantitative data between each subgroup
**** Significant level at P value < 0.05***

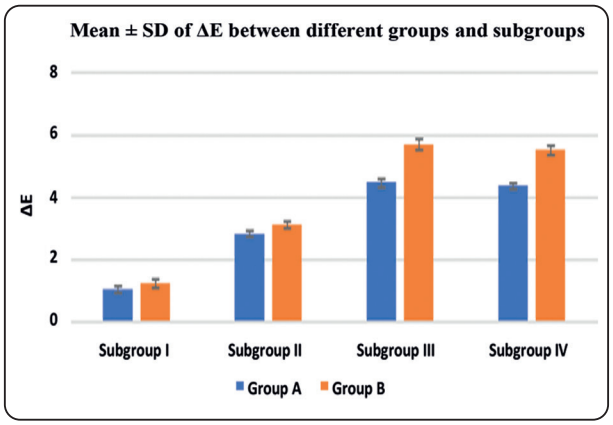

Fig. (1) Bar chart showed mean ± SD of ΔE between different groups and subgroups.

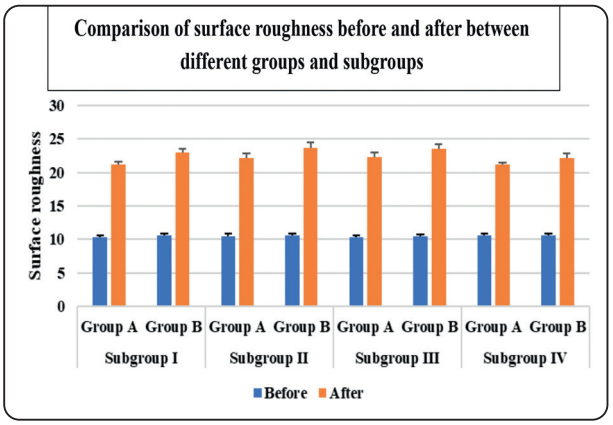

Fig. (2) Bar chart of BßR and AR between different groups and subgroups.

DISCUSSION

In order to satisfy a patient's high esthetic expectations, dental esthetics must mimic the form, color and shape of natural teeth. Therefore, one of the most significant considerations for patients and specialists is color stability. It contributes to the success or failure of esthetic restorative material, especially in the high esthetic zone. Moreover, color stability is directly related to the surface roughness of the restoration. [5,6]

One of the recent materials for esthetic restoration is hybrid ceramic, which combines the benefits of a resilient polymer with the esthetic appearance of ceramics. Furthermore, resin composite CAD/CAM block technology was developed for dental practice as an alternative to ceramics. The mechanical and optical properties of these materials are considerably better than those of conventional composite resins because they are manufactured under controlled and highly standardized conditions. The CAD/CAM resin restorations cause less wear for

opposing in addition to improve machinability and adjustment comparing to conventional ceramics [7].

Selection of sample dimension was crucial for color and surface roughness measurement. The light beam emitted from the spectrophotometer must be smaller than the surface area of the sample to avoid edge loss phenomena. [8] Furthermore, during surface roughness measurement, the stylus of the profilometer should be at least 1mm away from the sample border. [8] For those reasons, samples were prepared 12 mm in diameter. Also, machinable precision saw was used for sectioning the blocks to produce a standard section dimension for accurately testing the investigated materials. Different oral conditions and exposure times can affect color stability and surface roughness. This study was conducted to evaluate the effect of aging on the color stability and surface roughness of hybrid ceramics and reinforced composite resin block materials. Thus, all samples were stored for 28 days, which equal to 4 years, in the oral cavity regarding to **Gawrioek et al, 2012**[9] and **Erturk-Avunduk et al, 2022**[10] at different aging protocols which resemble the normal oral environment. Artificial saliva was selected as a control group and it is also a remineralizing agent. [3] Citric acid is normally found in our food as citrus foods, juices, and carbonated beverages. [11] Citric acid with pH=4 was selected in this study as a demineralizing agent [3]. The immersion protocol used in subgroup II was followed to simulate the natural process of remineralization- demineralization in the oral cavity. In this study, tea, with or without sugar addition, was selected as a staining solution because it considered one of the most popular drinks in Egypt. [12] The average consumption of tea is about 2- 3cups per day among tea drinker. [3] The actual exposure of tooth surface to tea is about 55-60s per cup. [9] Therefor the immersion protocol of subgroup III and IV in this study were done 2.5 hr per day for 28 days which is nearly equal to 4 years of tea consumption in the oral cavity. [9,10]

Rough surface may retain surface stains mechanically. Regarding to this, a smooth surface is essential for the color stability of restorations. A standard finishing and polishing procedures were done for all samples before immersion protocols. [13]

For measuring the color parameter, a desktop spectrophotometer was selected in this study. Spectrophotometer is recommended by several studies as **Mahn et al, 2021**[14], **Ersoz et al, 2021** [15]**and Paolone et al, 2023**[16] because its reproducibility, objectivity, high sensitivity and suitability for measuring small color changes comparing to other methods. Furthermore, desktop spectrophotometer ensured that the samples would not be affected by the ambient luminance of the tested region, allowing accurate measurements [15,16].

Acceptability of the clinical color change is the ability to accept or reject the color difference for the restoration. The noticeable threshold for acceptable color difference observed by a human observer is $\Delta E = 3.7$[17]. When the ΔE value is more than 3.7, the color difference is viewed clinically inappropriate. [18]

The ΔE values for group B in all subgroups were significantly higher than group A. This could be explained by the absence of Bis-GMA in composition of group A. Bis-GMA is responsible for water and stain uptake, which could affect the refractive index.[19] This result was comparable to previous studies to **Sagsoz et al, 2016** [20] and **Stamenković et al, 2021**. [21]

In addition, ΔE was exceeded 3.7 in subgroup III and IV for both groups. This could be explained by high stainability of polymers which present in both types. The result was in agreement to **Qaraghuli et al, 2022**[22], **Aydın et al, 2020**[23], **Arif et al**, **2019** [24] and disagreement with **Al Amri et al**, **2021**. [25] The disagreement might be explained by the difference among the immersion protocol time and absence of citric acid in Al Amri et al, 2021[25] study. The ΔE of subgroup III in both groups was a significantly higher than subgroup IV. This could be explained by

the stickiness effect of sugar [3] which might mask the surface roughness and reduce the staining effect of tea.[26]

Moreover, the results showed that ΔE of subgroup II in both groups was a significantly higher than subgroup I. This might be explained by presence of citric acid in subgroup II which could lead to significant changes in ΔL, Δa and Δb due to whitening effect of citric acid. [27]

The selection of a stylus profilometer to measure the surface roughness was due to the ability of the contact profilometer to detect surface roughness over large area which can give an overview to the entire surface. [28] Dental materials' surface roughness significantly impacted the buildup of plaque, staining, wear and esthetical appearance of direct and indirect restorations. Several studies revealed that the threshold of surface roughness for bacterial plaque retention was 0.2μm (200nm). [29] Furthermore, clinical research reported that a change in mean surface roughness at about 0.3μm (300nm) could be recognized by the majority of patients by the tip of the tongue. [30] In our study, all surface roughness results were below threshold of surface roughness for bacterial plaque retention.

In this study, the results showed that initial reading (BR) for all subgroups were insignificant to each other that revels excellent standardization. In addition, all readings for all subgroups after immersion (AR) were significantly higher than before immersion. In subgroup I, this could be explained by the remineralizing action of artificial saliva as it contains calcium chloride and sodium dihydrogen orthophosphate in it is composition. Both might precipitate calcium and phosphate ions on the surface of the sample [31]. This was in agreement to **Arocha et al, 2014**[32] and **Soliman et al, 2016**. [3] Although this was disagreement to **El Sokkary et al**, **2018**[33] and **waleed et al, 2021**[34] and could be explained by longer immersion time in our study.

In subgroup II, citric acid has chelating action that might be the reason for the significant increase in roughness after immersion. [35] Citric acid can dissolve silica particles and release alkaline oxides. [36] This result was in line with **AL-Thobity et al, 2022**[37] **and Munusamy et al, 2020**[38] studies. However, it is in conflict with **El Sokkary et al, 2018**[33] and **waleed et al, 2021**[34]. The conflict could be explained by variations in immersion protocol method and time.

Furthermore, in subgroup III and IV synergistic effect from the combination of citric acid and tannic acid from tea increase the surface roughness. This result was comparable to **Soliman et al, 2016**[3] and **Yousry et al**, **2023**[39] and incomparable to **Elwassefy et al, 2023**[40] due to the short immersion protocol time in their study.

Moreover, subgroup IV showed a significant decrease in surface roughness after immersion compared to subgroup II and III due to stickiness effect of sugar molecule which could block some of surface irregularities. [3] In addition, all subgroups of groupA showed significantly lower surface roughness than group B due to the difference in composition of both materials. [41]

According to the previous results and discussion of this study, the hypothesis was accepted and the null hypothesis was rejected. The result showed that after the immersion protocol, there was a significant difference in all subgroups within both groups compared to before.

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