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THE COLOR CHANGE OF INDIRECT ZIRCONIA CONTAINING RESTORATIVE MATERIALS – A COMPARATIVE IN VITRO STUDY

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ABSTRACT

Aim: to evaluate the influence of immersion time in coffee staining solution on the color coordinates of two Zirconia-containing indirect aesthetic materials.

Material and methods: 20 Discs were used in the current study; samples have been divided into two groups according to the material used (monolithic Zirconia restoration group (katana) and indirect Zirconia silicate filled composite restoration group [Ceramage]. The samples were prepared and then immersed in coffee [staining solution]. Spectrophotometric analysis was made immediately after manufacturing of discs at (T0), at 7 days (T1), 14 days (T2) and at 21 days.

Results: At T0-T1, T1-T2, T0-T2 and T0-T3, the mean ΔE of Zirconia showed statistically significantly lower value than Ceramage. At T2-T3; no statistically significant difference between mean ΔE of the two ceramic types was found (*P*-value = 0.155, Effect size = 0.235). Regardless of time; Zirconia showed a statistically significant lower mean ΔE than Ceramage after staining protocol.

Conclusion: The ΔEs of tested CAD/CAM Zirconia ceramics is considered color stable in comparison to zirconia filled composite (Ceramage), and better resistant to external coffee staining. Color difference (ΔE) between different test specimens at different immersion or contact/time in coffee drinks showed clinically un acceptable range above 3.3 units). Thus, Zirconia filled lab composite is not aesthetically stable in color. Further studies are required to assess the color stability of different zirconia containing indirect restorative materials upon exposure to different oral conditions with aging inside the oral cavity.

KEYWORDS: Ceramage, Zirconia, Color stability

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INTRODUCTION

Tooth-colored restorative materials are exposed to multiple oral cavity insults, such as thermal fluctuations, mechanical stresses, staining beverages and acidic insults. These insults could affect the color stability of restorations in oral service due to repeated exposure over time. The overall success rate of these different restorations is measured in terms of mechanical and aesthetic stability over time[1,2].

Throughout the past times, the usage of indirect zirconia restorations became more popular in the restorative dental field because of its remarkable biocompatibility with oral tissues, and excellent mechanical properties. This was also accompanied with inferior esthetic properties, compared to other glass ceramics. Ceramic restoration better color stability compared to composite resin, meanwhile, they also show inferior mechanical properties due to their inherent brittleness. On the other hand, composite resin shows inferior color stability and enhanced modulus of elasticity approaching that of the tooth structure[3].

The inherent weakness points of all the above-mentioned materials led to the launching of new materials containing both elements, these materials showed satisfying aesthetic as well as mechanical properties due to improved modulus of elasticity. Categories of these restorative materials include polymer infiltrated ceramic network (PCIN), organically modified ceramic (ormocer), and ceramic optimized polymers (ceromers)[2].

The aesthetic success of the restoration can be described in two main terms, initial color matching and stability with time. Variations in the chemical formula of different restorative materials has great effect on their color stability, which includes ceramic/resin content in addition to resin type. Color stability and uptake of stains from cola, food and beverages are related to numerous factors that have been discussed in many reviews. Various in vitro procedures were put forward to produce the simulation of testing color stability of restorations intraorally, by immersing in staining factors, such as exogenous staining solutions for different durations, simulating the frequency of exposure to such solutions in the oral cavity. The discoloration occurring from coffee drinks is considered one of effective & strong extrinsic staining effects [4,5] .

Multiple methods were proposed to evaluate the esthetic long-term success of any restoration depending on measuring color differences. Such methods included conventional and digital shade recording methods. Digital methods showed accuracy of results and analysis of shade parameters [5,6]. The differences in color are described in terms of (ΔE), where (ΔE) values below the level of 3.3 is not considered to be clinically perceived, as they indicated no obvious color change, whereas higher levels indicated deterioration of color stability.

The objective of this study was to assess the effect of immersion time in coffee staining solution on the color coordinates of two Zirconia containing indirect aesthetic materials.

Null Hypothesis of this study claims that, there is no clinically perceivable color difference between zirconia and other indirect restorative material containing resinous matrix in addition to high content of Zirconia ceramic filler, when subjected to coffee staining solution, simulating the intraoral conditions at different time intervals.

MATERIALS AND METHODS

Grouping

Discs of two indirect zirconia containing restorative materials have been used within the current study (Table 1). Samples have been divided into two groups (n=10) according to the material used.

Group I (control group): 10 discs of monolithic Zirconia restoration (Katana, Kuraray Noritake Dental, Tokyo, Japan).

Group II: 10 discs of indirect Zirconia silicate filled composite restoration (Ceramage, Shofu, Kyoto, Japan).

Sample preparation

Discs of both restorative materials of shade A2 were used in this study. Samples were prepared in the form of discs with a diameter of 10 mm and a thickness of 2 mm. For the fabrication of monolithic Zirconia samples, a Teflon mold of 10 mm internal diameter and 2 mm thickness was scanned with an extraoral scanner (D/R2000, 3 shape, Copenhagen, Denmark).

The produced Standard Tessellation Language (STL) file, was exported to a milling machine (K5+, VHF, Ammerbuch, Germany). The produced Zirconia discs were glazed and sintered in the sintering furnace according to the manufacturer instructions[7].

Ceramage discs were fabricated by packing the composite material in a Teflon mold, and then two microscope glass slides with smooth polished surfaces were used to pack it. Discs were initially cured with light curing device (dentmax, LED, Korea), complete curing of the material was done in special curing unit (Solidilite V Light-Curing Unit, Shofu, Kyoto, Japan)[8].

Bonding procedures

To simulate the clinical situation, the discs of both groups were bonded to tooth structure and then exposed to the staining solution and light on one side only. Freshly extracted teeth were collected from the teeth bank, Faculty of Dentistry, Al Azhar University. Enamel was ground by a diamond disc and running water to expose the dentine. Fabricated discs were bonded to dentin by self-adhesive resin cement (Relyx adhesive, Dentsply). All discs were cured for 40 seconds (curing time), ready for staining procedures[9].

The reason for our research study, where, dentin surface of freshly extracted sterile teeth was exposed, after grinding enamel by diamond disc under running water. (Teeth bank, Al Azhar dental university) All the fabricated discs were bonded to exposed dentin by self-adhesive resin cement (Relyx adhesive, Densply). All discs were cured for 40 seconds (curing time), ready for staining procedures[9].

Staining process

The staining solution used was coffee (Nescafe Classic, Nestle, Egypt). Samples were submerged within the staining solution at ambient temperature for a duration of 21 days. Solutions were interchanged every day and were placed in sealed containers in order to avoid the evaporation of the staining solutions[,10]. Spectrophotometric examination took place immediately after manufacturing of discs at (T0), at 7 days (T1), 14 days (T2) and at 21days (T3)[10].

TABLE (1) Materials used within this study.

Material	Filler by weight	Composition	Type
Katana TM Zirconia ML (Kyoto, Japan)	> 99%	$ZrO_2 + HfO_2 + Y_2O_3$	Monolithic ytria stabilized Zirconia
	< 1 %	Other oxides	
Shofu Ceramage CE (Shofu, Kyoto, Japan)	73%	Zirconium Silicate	Micro fine ceramic polymer system with progressive fine structured fillers
	5 – 15 %	Urethane Dimethacrylate (UDMA)	

Color measurement and color change evaluation

In all groups, colorimetric assessment took place via a blind skilled operator at four investigational stages of immersion in staining solution: At baseline (T0), and after 7, 14 and 21 days (T1, T2 and T3 respectively). It was done in accordance with the CIE L*a*b* system. Prior to every assessment, specimens have been slightly washed using distilled water and air-dried [11,12].

Samples were chromatically measured four times and calculation of the mean values was performed. Usage of the CIE L* a* b* color system took place to determine the color differences. The L* value indicated "lightness"; the higher the L value, the higher is the lightness (a value of 100 matches flawless white and that of zero matches black). CIE L* a* b* values are named the "chromaticity coordinates"; "a*" displays red color on positive values and green color on negative values; "b*" displays yellow color on positive values and blue color on negative values[13].

The overall color differences (ΔE ab*) has been measured using the following equation:

$$\Delta E\ Lab^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]\ x1/2$$

Color measurements (CIE L*a*b* and **VITA-PAN** Classical shades) were attained prior to and following the contact with numerous solutions, by the means of a spectrophotometer and shade taking device (Vita Easyshade, Vita Zahnfabrik, Germany). Calibration of both instruments took place at orderly timings according to the manufacturers' instructions, and a mean sample reading was calculated from three incessant measurements[14]. Color was measured beforehand and following the exposure to several solutions, then L*, a*, b* formula was used for the determination of color change (DE) and calculating the difference; values were attained with white (DE white) and black (DE black) backgrounds. The L* value displays lightness, whilst a* represents the cyan-magenta spectrum and b* the blue-yellow parameter.

During clinical situations, Ruyter and co-workers presented that the human eye has the ability to sense Delta E of 3.3 or higher. Thus, Delta E of 3.3 was set as the boundary for detectable color change. All specimens were chromatically measured three times (one week, two weeks & three weeks) and the mean values were calculated; then the average of each color parameter for specimens of similar shades was calculated. Therefore, all the calculated values for color change, at different time intervals were statistically analyzed & tabulated for results interpretation.

RESULTS

Statistical Analysis

Numerical data has been explored for normality by checking the distribution of data and the usage of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data displayed parametric distribution. Data has been presented as mean and standard deviation (SD) values. Repeated measures Analysis of Variance (ANOVA) has been used to study the effect of ceramic type, time and their interaction on mean ΔE. Bonferroni's post-hoc test was done for pair-wise comparisons when ANOVA test showed significance. The significance level was set at $P \leq 0.05$. Statistical analysis took place by the means of IBM SPSS Statistics for Windows, Version 23.0. Armonk, NY: IBM Corp.

Repeated measures ANOVA results

The results presented that material's type (regardless of time) had a statistically significant effect on mean ΔE (*P*-value <0.001, Effect size = 0.932). Time (regardless of material type) also had a statistically significant effect on mean ΔE (*P*-value <0.001, Effect size = 0.723). The interaction between the two variables had a statistically significant effect on mean ΔE (*P*-value = 0.039, Effect size = 0.264). Since the interaction between the variables is statistically significant, so the variables are dependent upon each other. (Table 2)

Color change (ΔE)

Comparison between two groups regardless of time intervals:

Regardless of time; Zirconia (group1) showed statistically significantly lower mean ΔE than Ceramage (group 2), after staining protocol, where the (*P*-value <0.001) (Table 3) (Figure 1).

Effect of different interactions on ΔE

Comparison between ceramic types:

At T0-T1, T1-T2, T0-T2 and T0-T3; the mean ΔE of Zirconia showed statistically significantly lower value than Ceramage (*P*-value = 0.021, Effect size = 0.505), (*P*-value <0.001, Effect size = 0.964), (*P*-value = 0.013, Effect size = 0.559) and (*P*-value = 0.006, Effect size = 0.628), respectively.

At T2-T3; there was no statistically significant difference between mean ΔE of the two ceramic types (*P*-value = 0.155, Effect size = 0.235).

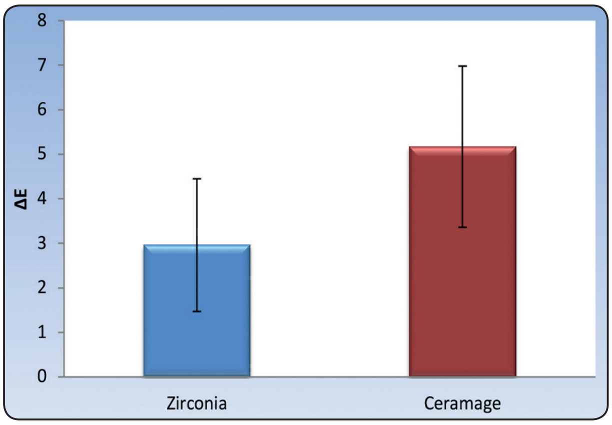

Fig. (1) Bar chart showing mean and standard deviation values for ΔE of the two materials, regardless of time

Comparison between times:

With Zirconia; there was a statistically significant difference between mean ΔE at different times (*P*-value = 0.001, Effect size = 0.959). Pair-wise comparisons between times revealed that there was no statistically significant difference between ΔE values from T0-T1, T0-T2 and T0-T3; all showed the statistically significantly highest mean

TABLE (2): Repeated measures ANOVA results for the effect of different variables on mean ΔE

Source of variation	Type III Sum of Squares	Df	Mean Square	F-value	P-value	Effect size (Partial eta squared)
Material type	60.839	1	60.839	110.428	<0.001*	0.932
Time interval	83.756	4	20.939	20.919	<0.001*	0.723
Material type x Time interaction	11.460	4	2.865	2.862	0.039*	0.264

*df: degrees of freedom = (n-1), *: Significant at P ≤ 0.05*

TABLE (3) The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between ΔE of the two ceramic types regardless of time

Zirconia (group 1)		Ceramage (group 2)		P-value	Effect size (Partial eta squared)
Mean	SD	Mean	SD		
2.96	1.49	5.17	1.81	<0.001*	0.932

**: Significant at P ≤ 0.05*

ΔE values. There was no statistically significant difference between mean ΔE from T1-T2 and T0-T3; both showed the statistically significantly lowest mean ΔE values.

With Ceramage; there was a statistically significant difference between mean ΔE at different times (*P*-value = 0.001, Effect size = 0.968). Pair-wise comparisons between times revealed that there was no statistically significant difference between ΔE values from T0-T1, T0-T2 and T0-T3; all showed the statistically significantly highest mean ΔE values. The mean ΔE from T1-T2 showed lower mean value with non-statistically significant difference from T0-T1 but a statistically significantly lower mean value compared with T0-T2 and T0-T3. The statistically significantly lowest mean ΔE was found from T2-T3. (Table 4) (Figure 2)

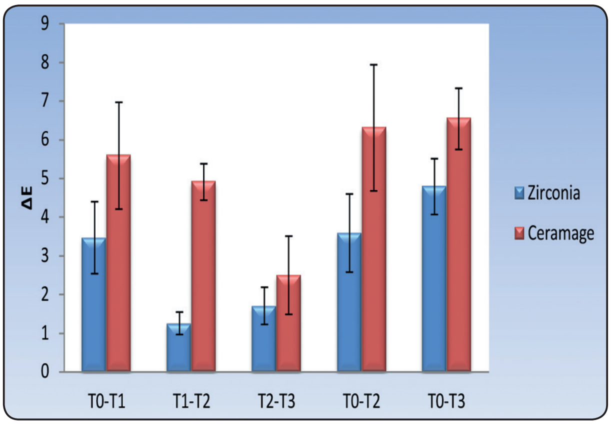

Fig. (2). Bar chart representing mean and standard deviation values for ΔE with different interactions of variables

TABLE (4) The mean, standard deviation (SD) values and results of repeated measures ANOVA test for comparison between ΔE values with different interactions

Time	Zirconia (g:1)		Ceramage (g:2)		*P*-value (Between ceramic types)	Effect size *(Partial eta squared)*
	Mean	SD	Mean	SD		
T0-T1	3.47 ^{A}	0.93	5.59 ^{AB}	1.38	0.021*	0.505
T1-T2	1.26 ^{B}	0.29	4.91 ^{B}	0.47	<0.001*	0.964
T2-T3	1.71 ^{B}	0.48	2.5 ^{C}	1.01	0.155	0.235
T0-T2	3.59 ^{A}	1.01	6.31 ^{A}	1.63	0.013*	0.559
T0-T3	4.79 ^{A}	0.72	6.54 ^{A}	0.79	0.006*	0.628
P-value (Between times)	0.001*		0.001*			
Effect size *(Partial eta squared)*	0.959		0.968			

: Significant at P ≤ 0.05, Different superscripts in the same column are statistically significantly different.

DISCUSSION

The success of the esthetic restoration is dependent on main factors as initial color match and long-term color stability[15]. Matching restorations to natural teeth depends on its own color and translucency, however, color shifts as the material service in the mouth over time[16]. Therefore, color stability rises as the main factor to the longstanding esthetic success of dental restorations [17].

Recent materials were introduced to the market with a mixture of ceramics and resin to gain the advantages of both materials. Materials used in this study were monolithic zirconia restorations (katana) and zirconia filled composites (Ceramage) with 73 % zirconium silicate filler in UDMA resin matrix. In our study, we tested the effect of such a material modification on the color stability after exposure to staining solution (coffee), through different intervals of time. Coffee (Nescafe Classic, Nestle Suisse SA, Vevey, Switzerland) has been used in this study as one of the most used solutions that can cause staining for teeth and intraoral restorations[18,19]. The staining solution was freshly prepared and changed every day to ensure maximum staining effect[20,21,22].

Material discs were fabricated according to the manufacturer instructions for each product, Ceramage composite has been initially cured, then was further subjected to an additional curing cycle to ensure total resin polymerization. The chemical durability of ceramics is primarily fine, yet, a lot of factors could have an influence over it, such as the composition and microstructure of the ceramic material, the chemical characteristics of the corrosive surrounding, and the exposure period. Unpolymerized resin was mentioned in some studies as a cause of discoloration. Monolithic Zirconia discs were fabricated using CAD/CAM system, sintered and glazed to ensure highly glazed surface resistant to staining [(13,23,24)].

Discs of both materials were bonded to freshly cut dentin using dual cured resin cement. This step was performed to simulate the oral conditions, making the studied materials exposed to staining solution from one side only, as agreed in other studies. [(3,6,16,28)]

Base color has been recorded with (Easyshade V, Vita Zahnfabril, Germany), using CIE LAB system. Shade recording was done at different intervals of time: baseline, one, two and three weeks respectively (T0, T1, T2 and T3) simulating the exposure of restorations to oral conditions, where one-week is equivalent to seven months, as mentioned in other researches. [(20, 28,30)]

Previous studies suggested that, immersion in cola and coffee, is considered a good staining tendency test and an estimate for the materials' tendency to discolor [25,26]. Many researchers approved this methodology for staining, like Chan et al., who has found that coffee caused more discoloration than tea and cola beverages[27,28]. Thus, 28 days immersion of samples within coffee for assessment of the consequential staining effect is considered an exaggerated duration to reality. For this reason, only three successive time intervals were chosen as T0, T1, T2, T3 (immediate, one week, two weeks and three weeks) respectively.

Assessment of color and shade differences were calculated as (delta E), Calculated using the equation $\Delta E = \sqrt[2]{\Delta L + \Delta a + \Delta b}$

Color difference was considered clinically perceivable when delta E exceeded 3.3 value. Another study mentioned that ΔE values greater than 3.7 units were considered as unacceptable color difference [13,29,30].

Results of this study revealed superior color stability (2.96 $\pm$ 1.49) of monolithic Zirconia over time, compared to composite filled with high percentage of zirconium silicate filler (73%) (5.17 $\pm$ 1.81). This finding was supported by many studies that documented that ceramics showed superior color stability over time upon exposure to oral conditions or staining solutions[1,13].

Studying the effect of exposure time to staining solution on the color stability of monolithic Zirconia has shown that maximum color changes occurred within the first 7 days of exposure to coffee (T0-T1 mean of 3.47$\pm$0.93), whereas significant lower mean color differences at T1-T2 (1.26 $\pm$0.29) & T2-T3 (1.71$\pm$0.48), indicating color stability upon long time exposure to coffee.

On the contrary, analysis of more significant color changes were observed in case of ceramage at the T0-T1 (5.59 $\pm$1.3) and T1-T2 (4.91$\pm$0.47), these results were confirmed by other studies [27,30,31]. Progressive significant color change with time can be attributed to the finding which states that material discoloration increases due to adsorption and surface absorption of colorants, and this outcome of fine colorant particles remain to block pitted free of colorants zones. Another explanation is that water molecules might spread in the material carrying chromogenic molecules. Further processes can similarly explain the progressive color change of ceramage, these processes include dissolution hydrolysis, expansion plasticization, microcrack formation, and fatigue that was mostly caused by water sorption [30].

Both studied materials have shown color difference below the clinically unacceptable level at the third week of exposure (T2-T3), supporting the finding which stated that most of the color changes would occur during the first two weeks of exposure to staining solution.

The null-hypothesis of the current investigation was rejected. In our study, significant differences in color stability were reported among the two tested materials and throughout disparate coffee staining time intervals. This came in accordance with previous reports that tested other materials[32,33].

It was concluded that the ΔEs of tested CAD/CAM Zirconia ceramics is considered color stable in comparison to zirconia filled composite ceramage, and better resistant to external coffee staining. Color difference (ΔE) between different test solutions at different immersion or contact/time in coffee drinks showed clinically un acceptable range above 3.3 units. Thus, Zirconia filled lab composite is not esthetically stable in color. Moreover, the 73% high filler content of Zirconium silicate inside ceramage didn't improve its resistance to staining solutions. Further studies are required to assess the color stability of different zirconia containing indirect restorative materials upon exposure to different conditions inside the oral cavity.

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