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Effect of Aging on Fluorescence of Some Dental Ceramics

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Abstract

Fluorescence of natural teeth and its alterations due to environmental factors should also be taken into account in restorations. This study aims to assess fluorescence changes of three types of dental ceramics. 36 Ceramics of Feldspathic, Emax, and Enamic in 10 mm diameter and the thicknesses of 0.5 and 1mm with glaze and polish finishes were studied.

Fluorescence was measured before and after aging and were analyzed using Anova test.

The results showed that ceramics became opaque as the result of aging. This was more

noticeable for Enamic ceramic with the lightness increase of 4.10 and 4.86 for the UV

excited and non-excited measurements, respectively. Moreover, Enamic ceramic

experienced significant changes fluorescence due in its aging

(Average Fluoresence Change $_{Enamic} = -10.2$) while Emax (Average Fluoresence Change $_{Emax} = -3.6$) and

feldspathic (Average Fluoresence Change $_{Feldspathic} = -0.4$) ceramics were superior to Enamic in

this respect. In Feldspathic ceramic, due to the presence of feldspar phase, high resistance

to aging process was observed. While, severe shifts in fluorescence due to aging were

observed in Enamic ceramic. Several factors affect color and fluorescence change of

ceramics due to aging. Preparations stresses, adhesion of glazes and the formation of

micro cracks on the ceramic surfaces are among the factors can influence on this

behavior. In case on Enamic ceramic caveat should be taken as it can change its

appearance due to fluorescence hue shift probably as a result of surface scattering of UV

light. According to the findings of this study Feldspathic, Emax and Enamic are

respectively preferable in terms of fluorescence stability during aging process.

Keywords: dental ceramics, aging, fluorescence, glazing, polishing

1. Introduction

Simulation of natural tooth appearance by dental restorative materials has always been a

challenge for dental clinicians, and can only be achieved successfully by efficient clinical

and laboratory cooperation. Due to complex optical properties of tooth color,

3

determination of color parameters and achieving a color match are fundamental in esthetic dentistry. Color is an important parameter taken into account by patients when judging about the quality of a restoration. Thus, achieving excellent color match with natural adjacent teeth is a major goal for both dental clinician and patient [1-5].

Application of indirect dental restorations such as ceramic restorations has increased in the past years, which may be due to the high failure rate of direct composite restorations, their marginal discoloration, marginal fracture, and wear, as well as the excellent esthetics and biocompatibility of ceramics [6-9]. Evidence shows that dental ceramics are highly accurate materials that can mimic the appearance, color, and texture of natural teeth [10, 11].

Cosmetic dental restorations should have the optical properties of natural teeth such as their translucency, opacity, fluorescence, and opalescence, in order to be able to beautifully restore or replace the tooth structure [12, 13]. Among different optical properties required for an ideal restoration, fluorescence of restorative materials, along with the conventional parameters such as gloss, lightness, and purity can be named [11]. Aside from color match, color stability is another critical factor for primary and long-term success of dental restorations [11, 14]. In other words, color match of ceramic restorations with the adjacent natural teeth not only depends on color and translucency, but also on color change that occurs in ceramics over time as a result of aging of material [11, 14].

Natural teeth have a blue fluorescence under ultraviolet light; resultantly, the teeth appear whiter and lighter under daylight [15-17]. Fluorescence in natural teeth results in conversion of energy absorbed in lower wavelengths to light. It really is as if the teeth are

converted to a light source. When dentin is irradiated with a monochromatic light with 365 nm wavelength, fluorescence occurs with a peak at 440±10 nm [18, 19]. Fluorescence of dental materials is detected through the presence/absence of ultraviolet component of light in a spectrophotometer [6, 20, 21].

Restoration adjustments are often imperative to correct occlusal interferences, inadequate contours, and ceramic restoration margins, and improve the esthetic appearance and surface smoothness of ceramic restorations [22, 23]. Thus, the efficacy of finishing and polishing of ceramic restorations has been investigated to achieve an optimally smooth surface in glazed porcelain. The effects of different polishing techniques have been investigated, and their application as an alternative to glazing has been suggested for ceramic restorations [24-28].

Surface smoothness is an important parameter in color of restorations, because smooth surfaces reflect higher amounts of light compared with rougher surfaces [29, 30]. Some reports are available regarding different polishing techniques for ceramic restorations, emphasizing on polishing as an alternative to glazing [25, 27, 28]. Such studies compared polishing techniques with glazing of ceramic using profilometer, scanning electron microscope, and optical assessments.

Following the accelerated aging process, dental restorative materials undergo significant color change both in vitro and in the clinical setting [31, 32]. Accelerated aging refers to methods employed to simulate the clinical setting to allow assessment of color change of materials over time. In this process, dental materials are subjected to several parameters such as ultraviolet radiation, thermal alterations, constant humidity, and many other factors to simulate the intraoral conditions as much as possible [33]. Next, the color

stability of dental materials is assessed by comparing their color parameters after aging with baseline.

Small changes in thickness of transparent ceramic layers affect the final color of ceramic restorations [34, 35]. No study has addressed the effect of polishing on fluorescence of ceramics. To the best of the authors' knowledge, only one study has assessed the effect of different ceramic polishing systems on color and surface texture of Feldspathic porcelain [36], which reported that different polishing techniques yielded a smoothness comparable to that achieved after glazing on the ceramic surface. Also, the color change (ΔE) in all polishing techniques was acceptable.

The effect of accelerated aging on color change of composite resins has been previously evaluated. A study showed that accelerated aging increased chroma and opacity of composite resin [32]. Another study assessed the stability of fluorescence and translucency of direct and indirect composite restorations after aging. The results showed that the stability of optical properties of resins was variable based on their brand and color, and aging process significantly affected the fluorescence. However, the effect of this process on translucency of composite resins was not significant [37]. Another study assessed the optical properties of ceramics used in laminate veneers after aging, and showed that aging increased the masking ability of ceramics as well as their opacity, redness, and yellowness [38]. The effects of accelerated aging on color stability and surface roughness of three different dental ceramics were also evaluated in another study; the results indicated that accelerated aging had no significant effect on color stability of any of the three ceramic types; although it significantly decreased the surface roughness of nano-ceramics [39]. A previous study evaluated the change in color and fluorescence

of zirconia after aging, and found significant changes in color, lightness, chroma, and gloss after aging. However, aging had no significant effect on fluorescence [40].

To the best of the authors' knowledge, studies regarding the effect of accelerated aging on fluorescence are limited [38-40]. Thus, this study aimed to assess the effect of accelerated aging on fluorescence of ceramics. To this end the color and lightness change due to aging before and after UV excitation were studied. Moreover, the effect of aging SCR on the change of fluorescence was investigated.

2. Experimental

This in vitro, experimental study was conducted on three ceramic types of feldspathic Vita VM9 ceramic (A2; Vita Zahnfabrik, Bad Säckingen, Germany), IPS Emax (Ivoclar HT, A2; Ivoclar Vivadent, Schaan, Lichtenstein), and Vita Enamic (A2; Vita Zahnfabrik, Bad Säckingen, Germany). Table 1 presents the characteristics of ceramics.

Twelve disc-shaped specimens with 10 mm diameter were fabricated from each ceramic type with two different thicknesses of 1 mm (n=6) and 0.5 mm (n=6). Each thickness group was randomly divided into two subgroups to undergo polishing (n=3) and glazing (n=3).

Table 1. Chemical compositions and some structural data on the Feldspathic, Emax and Enamic samples.

Ceramic type	Chemical or phase Composition
Feldspathic	KALSi ₃ O ₃ 'NAlSi ₃ O ₃ 'Potassium feldspar 'Leucite '15-25% quartz '
	Metal oxides pigments
IPS Emax	SiO ₂ ,Li ₂ O,K ₂ O,P ₂ O ₅ ,ZrO ₂ ,ZnO
Enamic	SiO ₂ ,Al ₂ O ₃ ,Na ₂ O,K ₂ O,B ₂ O ₃ ,CaO,TiO ₂ ,PMMA

2.1. **Preparation of specimens**

2.1.1. Feldspathic ceramic

In this group, cylindrical silicone molds with 0.5 and 1 mm depth and 10 mm diameter were first used to standardize the size of specimens. Porcelain powder was mixed with distilled water as instructed by the manufacturer, and applied in the mold. Excess water was removed by vibration. The porcelain was condensed in the mold, and after removal from the silicone mold, it was sintered according to the manufacturer's instructions (80) mbar vacuum, starting at 450 °C and terminating at 919 °C). Ceramic temperature increased at a rate of 55 °C/minute until it reached 920 °C. Ceramic remained at 920 °C Nanu for 90 seconds.

2.1.2. IPS Emax ceramic

Wax model was first designed using computer-aided design system. It was then transferred to a milling machine, and discs were fabricated in desired dimensions. The wax patterns were sprued, flasked using 100 g flask of the device, and sintered at 700 °C for wax burnout. Ceramic ingot was then placed in the furnace and after heating at 910 °C under vacuum, the ingot was injected into the sprue by the plunger. After compression and cooling, gypsum particles were removed by 100-µm aluminum oxide particles (sandblasting) with 2.5 bar pressure. The specimens were then placed in Invex liquid followed by ultrasonic bath for 4 minutes. They were then rinsed and dried. The residual impurities were removed by using 100 µm aluminum oxide particles with 2.5 bar pressure. The sprues were cut with a diamond disc under water coolant, and the specimens were heated in a furnace (80 mbar vacuum, starting at 410 °C and terminating at 725 °C). Ceramic temperature increased at a rate of 60 °C/minute until it reached 730

°C. Ceramic remained at 730 °C for 60 seconds.

2.1.3. Enamic ceramic

Ceramic specimens with the desired dimensions were designed in a computer-aided design system. The blocks were placed in a milling machine, and the discs were fabricated with the desired dimensions. Their final thickness was controlled by a digital caliper (Mitutoyo Co., Tokyo, Japan).

One group of specimens was glazed according to the manufacturers' instructions, and the other group was polished. Polishing of IPS Emax and feldspathic ceramics was performed using Panter polishing kit (Carsten Fischer, Sirius ceramics Frankfurt, Germany). Vita Enamic ceramic specimens were polished with Vita Enamic Clinical Polishing Set (Vita Zahnfabrik, Bad Säckingen, Germany).

2.2. Aging process

To perform aging, after assessing the color measurement of the specimens, they were placed in the holder of Xenotest Alpha Chamber (Heraeus Kulzer, Hanau, Germany) which has a xenon lamp and a filter that corrected xenon light irradiation similar to daylight in visible light spectrum. The specimens were fixed with clamps under similar conditions such that the ceramic surface was completely exposed to light. The device was adjusted according to ISO 7491 for assessment of discoloration at 37 °C and 100 % humidity. The specimens were subjected to aging for 300 hours to assess their color stability.

2.3. Color measurements

The method of measurement of fluorescence of specimens has been described elsewhere [41]. Feldspathic sample, polished with the thickness of 0.5mm measured reflectance has been illustrated in Figure 1. As it could be seen the typical behavior of fluorescent samples is present in this figure. The UV part of the spectrum has been absorbed and remitted back at blue region specifically at 430 nm for the polished Feldspathic ceramic.

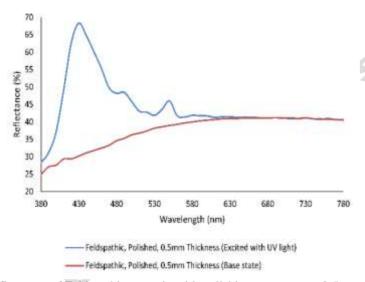


Figure 1. Reflectance of Feldspathic ceramic with polishing treatment at 0.5 mm thickness.

After aging, the color coordinates of specimens were measured again by a spectrophotometer (CS-2000; Konica Minolta, Japan). Prior to measurements, the specimens were cleaned in an ultrasonic bath for 10 minutes. Fluorescence was measured using Error! Reference source not found., which is the color difference in presence (excited) and absence of ultraviolet light (base):

$$FL = \left(\left(L_{excited}^* - L_{base}^* \right)^2 + \left(a_{excited}^* - a_{base}^* \right)^2 + \left(b_{excited}^* - b_{base}^* \right)^2 \right)^{0.5}$$
 (1)

Finally, the results were statistically analyzed and compared.

2.4. Statistical analysis

The results of fluorescence were analyzed by SPSS version 25. Data analysis was performed using three-way ANOVA. Also, LSD post-hoc and test and pairwise comparisons were applied for further studying of the significant of differences.

3. Results and Discussion

3.1. Qualitative results

The color changes of Emax, Enamic and Feldspathic samples are shown in Figure 2. In Figure 2-a, the color change of the samples are shown in a three-dimensional diagram. Moreover, in Figure 2-b, Figure 2-c and Figure 2-d, the two-dimensional diagram of the color change of the samples is shown in terms of a*b*, L*b* and L*a*. In these diagrams, each point represents the average color coordinate of three repeats. The color of the samples in Figure 1 is the actual color of the samples, which is rendered by assuming that the display color primaries are close to the sRGB color space primaries. The gray color of the samples before the excitation is due to the measurement arrangement specific to this study, and the samples have been rendered darker than their nominal color due to the lack of background and high translucency. Moreover, the color of the stimulated samples is representative of the real color of the samples after excitation, assuming the similarity of the primary colors of the display with the sRGB color space. The green arrows indicate the color coordinate shift of unexcited samples due to aging and the red arrows indicate the color shift of UV-excited samples due to aging. The black dashed lines show the color shift of the aged samples due to UV excitation and the continuous blue lines show the color shift of the unaged samples due to

UV excitation. Regarding the continuous blue lines and black dashed lines, the directions of the lines are not shown due to the crowding of the figure. However, by comparing the two ends of these lines, one can easily distinguish the direction from the color of the samples. In other words, the direction of these lines is from the gray sample to the blue ones.

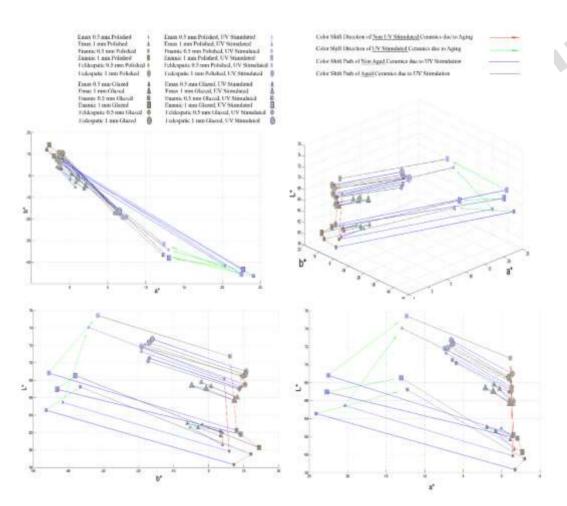


Figure 2. Illustration of color shifts due to aging of the excited and non-excited samples.

3.2. Quantitative results

3.2.1. Aging effect on changing the lightness of the samples - without UV excitation

Since aging is effective on the surface structure of ceramics and changing the surface causes more opacity, the effect of aging on ceramics has been investigated by studying

Lightness and color changes In order to investigate the change in the Lightness of the samples, a three-way ANOVA analysis was performed on the lightness due to the aging (without UV excitation). The results are shown in Table 2.

Table 2- Three-way Anova test for the effect of aging on the lightness of unexcited samples.

	Tests of Betw	een-Subject	s Effects		
Dependent Variable: dL*					
	Type III Sum of				
Source	Squares	df	Mean Square	F	Sig.
Model	446.895a	12	37.241	19.362	.000
Ceramic type	114.520	2	57.260	29.770	.000
Treatment	117.903	1	117.903	61.299	.000
Thickness	3.809	1	3.809	1.980	.172
Ceramic type * Treatment	109.355	2	54.678	28.428	.000
Ceramic type * Thickness	1.805	2	.903	.469	.631
Treatment * Thickness	.258	1	.258	.134	.717
Ceramic type * Treatment *	8.899	2	4.449	2.313	.121
Thickness					
Error	46.162	24	1.923		
Total	493.056	36			

Since the effects of ceramic type $\left(p_{\Delta L^*, \text{CeramicType}} = 0.000\right)$ and surface treatment factors $\left(p_{\Delta L^*, \text{Treatment}} = 0.000\right)$ were significant and these factors also have significant interaction with each other $\left(p_{\Delta L^*, \text{CeramicType* Treatment}} = 0.000\right)$, therefore the difference in lightness observed at different levels of these factors was investigated. Figure 3 shows the lightness difference in the aged samples (without excitation).

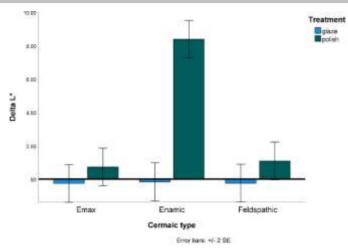


Figure 3. Lightness difference of the studied ceramics due to aging (without excitation).

3.2.2. Effect of aging on the color change of samples - without UV excitation

Although the monitoring of color coordinates L*, a* and b* is useful to justify the color changes on the surface of ceramics, from the quality of the treatment observed by the patient point of view, just the difference in color is important. To this end, the color difference of ceramics in the unexcited state due to aging was studied. The results of the three-way ANOVA on the color difference data are shown in Table 3. Moreover, the difference in colors caused by aging in different levels of the studied ceramics and finishes is shown in Figure 4.

Table 3- Three-way Anova test to investigate the effect of aging on the color difference of unexcited samples.

Tests of Between-Subjects Effects									
Dependent Variable: dE*									
	Type III Sum of								
Source	Squares	df	Mean Square	F	Sig.				
Model	646.227a	12	53.852	41.841	.000				
Ceramic type	271.621	2	135.811	105.520	.000				
Treatment	16.480	1	16.480	12.804	.002				

Thickness	.000	1	.000	.000	.988			
Ceramic type * Treatment	14.949	2	7.475	5.807	.009			
Ceramic type * Thickness	.589	2	.294	.229	.797			
Treatment * Thickness	.039	1	.039	.030	.864			
Ceramic type * Treatment *	.190	2	.095	.074	.929			
Thickness								
Error	30.889	24	1.287					
Total	677.116	36						
a. R Squared = .954 (Adjusted R Squared = .932)								

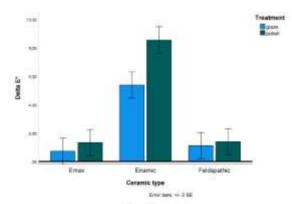


Figure 4- Colors difference of the studied ceramics due to aging (without excitation).

As it can be seen, the difference in the color difference created in $\mathrm{Emax}\left(p_{\Delta \mathrm{E}^*,\mathrm{Glazed},\mathrm{Emax}\,\mathrm{vs},\,\mathrm{Polished},\mathrm{Emax}}=0.355\right) \qquad \text{and} \qquad \mathrm{Feldspathic}$

 $\left(p_{\Delta E^*,Glazed,Feldespatic vs.\ Polished,Feldspathic} = 0.677\right)$ ceramics between the two finishes of glaze and polishing is insignificant. This is despite the fact that there is a significant difference $\left(p_{\Delta E^*,Glazed,Enamic vs.\ Polished,Enamic} = 0.000\right)$ between two different finishes in Enamic ceramic.

Also, the post hoc test showed that the color difference between Feldspathic and Emax ceramics is insignificant $\left(p_{\Delta E^*, \text{Feldspathic vs. Emax}} = 0.626\right)$, but there are significant differences between the pairs Enamic, Emax $\left(p_{\Delta E^*, \text{Enamic vs. Emax}} = 0.000\right)$ and Enamic, Feldspathic $\left(p_{\Delta E^*, \text{Enamic vs. Feldspathic}} = 0.626\right)$. The results of the investigations showed that the

thickness has no significant effect on the observed color difference $(p_{\Delta E^*, Thickness} = 0.988)$.

3.2.3. Aging effect on changing the lightness of samples - with UV excitation

As mentioned, the roughness of the surface due to aging causes more light to spread and as a result, the translucent sample (without background) becomes lighter as surface roughness increases. Therefore, in order to investigate the effect of aging on the samples, a three-way ANOVA test was performed on the results of the difference in lightness of the samples. The results of the test are shown in Table 3.

Table 4- A three-way method to investigate the effect of aging on the lightness of excited samples.

	Tests of Betw	een-Subjects	s Effects		
Dependent Variable: dL*					
	Type III Sum of				
Source	Squares	df	Mean Square	F	Sig.
Model	386.346a	12	32.196	21.636	.000
Ceramic type	197.650	2	98.825	66.411	.000
Treatment	44.489	1	44.489	29.897	.000
Thickness	5.003	1	5.003	3.362	.079
Ceramic type * Treatment	48.212	2	24.106	16.199	.000
Ceramic type * Thickness	2.973	2	1.486	.999	.383
Treatment * Thickness	1.563	1	1.563	1.050	.316
Ceramic type * Treatment *	.030	2	.015	.010	.990
Thickness					
Error	35.714	24	1.488		
Total	422.060	36			

ANOVA analysis showed that unlike Thickness $\left(p_{\Delta L^*, \text{Thickness}} = 0.626\right)$ the type of finish and the type of ceramic have significant effect $\left(p_{\Delta L^*, \text{Ceramic type}} = 0.000, p_{\Delta L^*, \text{Treatment}} = 0.000\right)$ on the lightness change. As these two aforementioned factors interact $\left(p_{\Delta L^*, \text{Ceramic type * Treatment}} = 0.626\right)$, L* difference is studied at different levels of the factors. Figure 2 shows the difference in

lightness of excited samples due to aging.

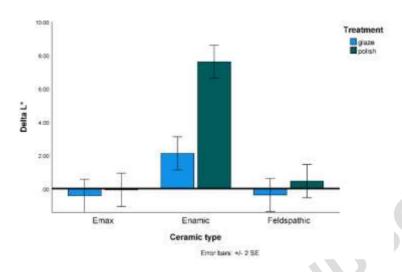


Figure 5. Lightness difference of the studied ceramics due to aging (with excitation).

Pairwise comparisons showed that the lightness change observed in glazed and polished **Emax** ceramics excited state due in the aging is to not $significant(p_{\Delta L^*,Glazed,Emax \, vs. \, Polished,Emax} = 0.619)$ and so as the same for the lightness change glazed Feldspathic between polished and ceramic in the **UV-excited** state $\left(p_{\Delta L^*, \text{Glazed}, \text{Feldespatic vs. Polished}, \text{Feldspathic}} = 0.250\right)$. On the contrary, in Enamic ceramic there is a significant difference between the lightness changes of the polished and glazed samples $\left(p_{\Delta L^*, Glazed, Enamic vs. Polished, Enamic} = 0.000\right)$.

Also, the post-hoc test with LSD method showed that there is a significant difference between Enamic and Emax ceramics $\left(p_{\Delta L^*, Enamic \, vs. \, Emax} = 0.000\right)$ and also between Enamic and Feldspathic ceramics $\left(p_{\Delta L^*, Enamic \, vs. \, Feldspathic} = 0.000\right)$ regarding the lightness change of excited ceramic due to the aging. However, comparison of the lightness change observed between Emax and Feldspathic ceramics was insignificant $\left(p_{\Delta L^*, Emax \, vs. \, Feldspathic} = 0.578\right)$.

3.2.4. Aging effect on the color change of samples - with UV excitation

Since the goal of the restoration is always the color matching of the restored tooth with the adjacent teeth in daylight conditions and this similarity should be maintained over time, the color difference of the samples due to aging was studied in the excited state with UV light. A three-way ANOVA was used to compare the factors of ceramic type, Tript thickness and glaze type. The results are shown in Table 5.

Table 5- Three-way ANOVA on color difference data due to aging under UV excitation.

	Tests of Betw	een-Subject	s Effects		
pendent Variable: dE_star					
	Type III Sum of				
ource	Squares	df	Mean Square	F	Sig.
odel	2929.530a	12	244.128	60.617	.000
eramic type	1257.085	2	628.542	156.067	.000
reatment	6.254	1	6.254	1.553	.225
nickness	.401	1	.401	.100	.755
eramic type * Treatment	18.102	2	9.051	2.247	.127
eramic type * Thickness	.941	2	.471	.117	.890
reatment * Thickness	15.384	1	15.384	3.820	.062
eramic type * Treatment *	56.655	2	28.328	7.034	.004
nickness					
ror	96.657	24	4.027		
otal	3026.188	36			
	3026.188		4.027		

As can be seen in Table 4, only the difference between the types of ceramics is significant. Also, since the interaction of the all three factors is also significant, so the investigations are carried out in different modes of combination of factors.

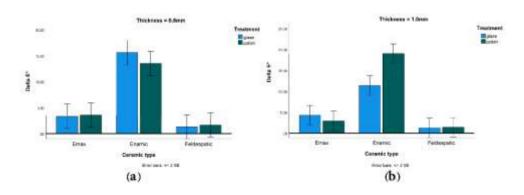


Figure 6- Color difference of the studied ceramics due to aging at different thicknesses(with excitation).

The result of the pairwise comparison of the glaze and polishing finishes for different substitutions of other factors also showed that there is a significant difference between the glaze and polishing finishes only in Enamic ceramic at a thickness of 1 mm ($p_{\Delta E^*,1.0 \text{mm},\text{Enamic},\text{Glazed vs. }1 \text{mm},\text{Enamic},\text{Folished}} = 0.000$). However, no significant difference was observed for other studied ceramics. Moreover, pairwise comparison between different finishes showed that there is a difference between thicknesses of 0.5 mm and 1.0 mm of glazed Enamic ($p_{\Delta E^*,\text{Enamic},\text{Glazed},0.5 \text{ mm vs. }\text{Enamic},\text{Glazed},1.0 \text{ mm}} = 0.017$) and thicknesses of 0.5 mm and 1.0 mm of polished Enamic ($p_{\Delta E^*,\text{Enamic},\text{Glazed},0.5 \text{ mm vs. }\text{Enamic},\text{Glazed},1.0 \text{ mm}} = 0.003$) and all other pairs were insignificant.

3.2.5. Effect of aging on changing the fluorescence of samples

One of the most important factors in dental ceramics is the preserve of the fluorescence change after aging. This is important as the degree of uniformity of the color of the restoration changes as a result and so does the color of the restoration. This causes the loss of the beauty of smile.

To investigate the fluorescence change due to aging, the color difference between the

ground and excited states is generally considered as fluorescence. Therefore, comparing the color difference of the samples due to excitation is actually an investigation of the effect of aging on fluorescence. It should be noted that despite the changes in color coordinates, which are summable, the differences in color are not like this. In other words, in order to study the fluorescence change due to the aging, the overall direction (not the arrows head direction) of aging and fluorescence changes in CIELAB color space should be the same so that these color differences can be subtracted just like scalar quantities. Since most of the samples become lighter due to aging and bluer due to fluorescence, this condition is relatively true. Figure 2 intuitively proves this claim.

Using the above description and assuming the same direction of the change in fluorescence and aging, in order to investigate the effect of aging on the fluorescence, the color difference of the samples between the UV-excited state and the base state were compared before aging and after aging. In other words, florescence at the base state were considered as standard and is subtracted from the florescence at the excited state. For this comparison, a three-way ANOVA test was performed on the color difference results. The results are shown in Table 6.

Table 6. Three-way ANOVA on fluorescence difference due to aging.

Tests of Between-Subjects Effects									
Dependent Variable: Fluorescence Difference									
	Type III Sum of								
Source	Squares	df	Mean Square	F	Sig.				
Model	1807.082a	12	150.590	23.787	.000				
Ceramic type	596.078	2	298.039	47.078	.000				
Treatment	100.656	1	100.656	15.900	.001				
Thickness	1.016	1	1.016	.160	.692				
Ceramic type * Treatment	160.675	2	80.337	12.690	.000				
Ceramic type * Thickness	5.041	2	2.520	.398	.676				
Treatment * Thickness	32.785	1	32.785	5.179	.032				

Ceramic type * Treatment *	99.023	2	49.511	7.821	.002		
Thickness							
Error	151.937	24	6.331				
Total	1959.019	36					
a. R Squared = .922 (Adjusted R Squared = .884)							

As can be seen, although the factors of ceramic type $\left(p_{\Delta E_{omin}^*, Ceramic type} = 0.000\right)$ and treatment $\left(p_{\Delta E_{nature}^*, Treatment} = 0.001\right)$ have a significant effect, as the interactions of the all three factors is $significant \left(p_{\Delta E_{aging}^*,Ceramic\ type^*Treatment^*Tickness} = 0.002 \right) \quad and \quad the \ two \ factors \ of \ Treatment \ and$ Thickness, $\left(p_{\Delta E_{aring}^*, Treatment*Tickness} = 0.032\right)$ and also interaction of Ceramic type and Treatment $\left(p_{\Delta E_{\text{attail}}^*, \text{Ceramic type*Treatment}} = 0.000\right)$ are significant, factors were studied in different factorial combination. Results are shown in Figure 7.

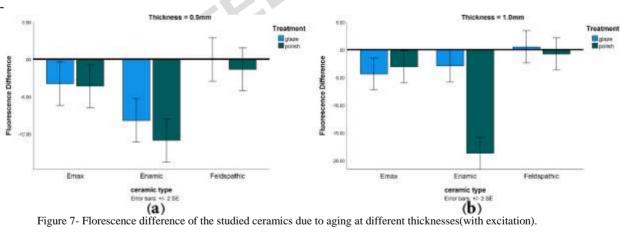


Figure 7 shows that Enamic ceramic exhibits extremely lower performance in retaining fluorescence due to aging as compared to other ceramics. Results of pairwise comparison confirms this such that except at thickness 1mm and glaze treatment the which there is insignificant difference between Enamic **Feldspathic** ceramics and

$$\left(p_{\Delta \mathrm{E}_{aing}^*, \mathrm{Glazed}, \mathrm{Imm}, \mathrm{Enamic}} = 0.109\right),$$
 and Enamic and Emax

 $\left(p_{\Delta E_{oging}^*, Glazed, 1mm, Enamic vs. Glazed, 1mm, Emax} = 0.478\right)$ ceramics all other combinatorial comparisons of

Enamic ceramic are significant. On the contrary non Enamic combinatorial comparisons

except Emax and Feldspathic at the polish treatment are insignificant

$$\left(p_{\Delta E_{aging}^*, Polished, 1mm, Feldspathic vs. Glazed, 1mm, Emax} = 0.271, p_{\Delta E_{aging}^*, Polished, 0.5mm, Feldspathic vs. Glazed, 0.5mm, Emax} = 0.284\right).$$
 More

details are listed in Table 7.

Table 7- Pairwise comparison of fluorescence changes due to aging in different levels of factors.

	Pairwise Comparisons								
Dependent Variable: diff flourscence									
							95% Confiden	ce Interval for	
				Mean			Differ	ence ^b	
Treatment	Thickness	(I) Ceramic type	(J) Ceramic type	Difference (I-J)	Std. Error	Sig.b	Lower Bound	Upper Bound	
glaze	0.5mm	Emax	Enamic	4.945*	2.054	.024	.705	9.185	
			Feldspathic	-3.245	2.054	.127	-7.485	.995	
		Enamic	Feldspathic	-8.190*	2.054	.001	-12.430	-3.950	
	1.0mm Emax		Enamic	-1.481	2.054	.478	-5.721	2.759	
			Feldspathic	-4.899*	2.054	.025	-9.139	659	
		Enamic	Feldspathic	-3.417	2.054	.109	-7.657	.823	
	0.5mm	Emax	Enamic	7.268*	2.054	.002	3.028	11.508	
polish			Feldspathic	-2.251	2.054	.284	-6.491	1.989	
		Enamic	Feldspathic	-9.518*	2.054	.000	-13.758	-5.278	
	1.0mm	Emax	Enamic	15.642*	2.054	.000	11.402	19.882	
			Feldspathic	-2.313	2.054	.271	-6.553	1.927	
		Enamic	Feldspathic	-17.955*	2.054	.000	-22.195	-13.715	

Based on estimated marginal means

4. Discussion

4.1. Qualitative results discussion

^{*.} The mean difference is significant at the 0.05 level.

b. Adjustment for multiple comparisons: Least Significant Difference (equivalent to no adjustments).

In this part, the ceramics color change observed in Figure 2 is qualitatively studied. First of all, in the studied samples, the glaze layer can create a special complication in the analysis of the obtained results. Due to its protective properties, glaze can be effective in preventing ceramic surface destruction, and on the other hand, the lack of proper adhesion between glaze and ceramic surface can cause moisture to be trapped in the interface between glaze and ceramic, and with the aid of temperature, it causes more surface destruction. On the other hand, due to contact with humidity and temperature, ceramics undergo hydric phase transformations and the formation of compounds such as AlOOH, which make the surface of the sample blue-red.

The polishing process had a greater impact on the chromatic properties compared to glazing. Maciel et. Al. [42] showed a close relationship between surface roughness and color changes. It seems that the main reason for the color difference of the polished samples is the problem of higher roughness under polishing conditions. Also, polished surfaces are more susceptible to pigmentation [43]. It seems that the maximum changes in the polished Enamic sample is due to the synergism of the remaining mechanical stresses during the machining with the polishing process. According to Figure 4 and comparing the glazed samples, the effect of machining in producing the sample has been high enough that the color difference has been maximum even in the case of the glazed Enamic sample.

In Figure 2, it can also be seen that Feldspathic and Emax ceramics have undergone a slight chroma change due to aging. This can be seen by comparing the length of the black dashed line and the continuous blue line in Figure 2-b. Meanwhile, in the case of Enamic samples, a yellow-red color change has occurred due to aging in the unexcited state

(change in the direction of the continuous blue lines and the black dashed lines for Enamic samples in Figure 2-b). In unglazed Enamic samples, due to the lack of moisture retention in the space between the glaze and the ceramic surface, the phase transformation on the ceramic surface is negligible, while in the fully glazed Enamic samples, a yellow-red tone shift has been observed, which could be related to phase transformation. Meanwhile, the polished Enamic samples have a blue tint, which is probably due to the formation of AlOOH groups on the Enamic ceramic surface.

Emax samples show better aging resistance than Enamic ceramic samples. This could be easily understood by noticing the length of the red lines in Figure 2-a. It can also be seen in Figure 2-a the least color change due to aging is related to the Feldspathic ceramic, which means that this ceramic has the greatest resistance to aging. This could be due to the phase composition of the Feldspathic sample. The presence of the feldspar phase (Table 1) encourages the liquid phase sintering, which itself leads to the formation of a dense body that is resistant to aging conditions. Feldspathic samples in two thicknesses of 0.5 and 1 mm in the unexcited state have close color coordinates, which is due to the higher opacity of these samples. In other words, when it comes to color, opacity causes insensitivity to thickness. Moreover, due to high opacity of these samples, the surface destruction has not been able to change the opacity and as a result, the lightness has left unchanged, indicating the possibility of applicability of Feldspathic ceramic in low thicknesses. It should be noted that these results are only related to color change, and other tests, including mechanical tests, should also be performed in this regard for the final evaluations.

In all three types of ceramics, it can be seen that there is a fluorescence shift as samples

age. In the case of the samples of Enamic and Emax, those exhibit extreme color change in the unexcited state, the highest amount of color change is also observed due to fluorescence (the length of the green lines in Figure 1-a). The color change in Enamic samples is more than Emax ceramic. The reason for this is that the red color change made in the Enamic samples due to aging causes the more absorption of the blue part (and probably UV parts) of the spectrum and as a result higher decrease in the fluorescence emission. The change of fluorescence due to aging becomes more important in Enamic ceramic as the fluorescence emission of Enamic samples (especially glazed ones) has undergone a hue shift due to aging (change of hue angle of fluorescence shift in Figure 1-b). This shift in the hue angle changes the color of the restored tooth and makes the work of the dentist and the laboratory more difficult.

The other reason of the change of fluorescence in Enamic ceramic during aging may be due to the formation of surface microcracks and damages during the machining process [44]. The microcracks are the location of the accumulation of hydrated ions that change the fluorescence behavior by surface chemical degradation during the aging process. In the case of Feldspathic ceramic, just like the color change in the unexcited estate due to aging, the stimulated samples show the greatest resistance to age among all of the three studied ceramics.

4.2. Quantitative results Discussion

4.2.1. Effect of aging on Lightness of the samples - without UV excitation

As can be seen in Figure 2 and 3, all the polished samples, without exception, in the non-excited state with UV, became lighter due to the aging. The reason for this is the destruction of the ceramic surface due to aging and the ability to scatter more light from

the ceramic surface. In other words, since there was no background behind the samples in the measurement of the reflection of the samples, the opacity of the samples caused the samples to become lighter. Meanwhile, in the glazed samples, due to the resistance of the glaze to degradation to aging, the surface of the sample has not changed (matted), and as a result, the glazed samples exhibit minute change in lightness (Figure 3). The increase in the lightness difference in polished state compared to glaze was insignificant for Emax $\left(p_{\Delta L^*, \text{Glazed}, \text{Emax vs. Polished}, \text{Endespathic}} = 0.23\right)$ and Feldspathic $\left(p_{\Delta L^*, \text{Glazed}, \text{Feldespathic}} = 0.11\right)$ ceramics and significant for Enamic $\left(p_{\Delta L^*, \text{Glazed}, \text{Enamic vs. Polished}, \text{Enamic}} = 0.00\right)$ ceramic. The reason for this can be seeked in the higher surface destruction of Enamic samples compared to the other two ceramics, which has caused more opacity and as a result, the sample is become lighter.

4.2.2. Effect of aging on the color change of samples - without UV excitation

A comparison of Figure 3 and 4 shows that the trend in lightness difference and color difference due to aging is almost the same for the samples (considering the fact that color difference is always positive). The only exception in this regard is glazed Enamic ceramics, in which color difference can be attributed to the phase transformation process that occurred in Enamic ceramics. In other words, although in the glazed Enamic ceramic, due to the protection of the glaze from the ceramic surface, surface corrosion did not occur, the phase transformation that occurred led to a change in chromaticity $\left(\left(\Delta a^* + \Delta b^*\right)^{0.5}\right)$ and, as a result, a high color difference.

4.2.3. Aging effect on changing the lightness of samples - with UV excitation

As seen in Figure 5, Enamic ceramic will have more fluorescence change in the excited state than the other two ceramics due to aging. Also, by comparing Figure 5 and Figure 3, it can be seen that in the glazed Enamic sample due to aging, the difference in lightness due to aging has increased as compared to the unexcited state. This observation can be justified by the fact that the glaze makes UV light to fall into the trap between the glaze and the ceramic surface and causes more excitation with successive reflections between these two surfaces. As a result of this excitation, the luminescence of the Enamic glazed sample has increased, which resulted in an increase in the lightness of the sample. The trapping of light in this interval is a function of the refractive index of the ceramic. This is despite the fact that this did not happen for the Emax and Feldspathic samples, probably due to the difference in the refractive index with the Enamic sample. This rationalization is fully aligned with the fact that the glazed and stimulated Emax and Feldspathic samples (see and compare Figure 5 with Figure 3) have the same lightness difference as their unexcited state (Figure 3) due to aging.

4.2.4. Aging effect on the color change of samples - with UV excitation

As can be seen in Figure 6, the reason for the interference of Ceramic type * Treatment * Thickness observed is the different behavior of Enamic samples in two thicknesses of 0.5 and 1 mm. In order to investigate the cause of this issue, the color coordinates of anemic samples due to aging (before and after excitation) are shown in Table 8.

Table 8- Color changes of Enamic ceramic due to aging before and after UV excitation.

	Before Excitation			After Excitation				
	ΔL^*	Δa^*	Δb^*	ΔE^*	ΔL^*	Δa^*	Δb^*	ΔE^*
Enamic,glaze,0.5mm	1.20	-1.22	4.81	5.11	2.67	-11.82	9.74	15.55
Enamic,glaze,1.0mm	-1.54	-0.62	5.24	5.50	1.56	-9.70	5.31	11.17
Enamic,polish,0.5mm	8.28	0.25	-1.39	8.40	8.60	-7.40	7.37	13.53
Enamic,polish,1.0mm	8.47	0.40	-1.88	8.69	6.60	-10.17	13.96	18.49

As shown in

Table 8, the different behavior in color difference observed in the case of Enamic ceramics is caused by the different behavior in Δb^* of the Enamic samples after excitation. Polished Enamic samples become completely opaque due to aging. This issue can be investigated by examining the ΔL^* of the samples before excitation. The opacity, which is mainly due to the scattering of the light from the surface of the samples, also causes the reflection of the UV part of the light from the surface. This prohibits penetration of UV parts of the incident lights into the deep parts of the ceramic and causes lack of excitation in these parts of the ceramics. Accordingly, Enamic ceramic loses its fluorescence power after aging. This issue can be investigated by controlling of Δb^* in

Table 8.

4.2.5. Effect of aging on changing the fluorescence of samples

As can be seen, in the thickness of 1 mm, the polished Enamic ceramic shows the highest fluorescence loss due to the aging. The reason for this can be attributed to the destruction of the surface of Enamic ceramic due to aging and the reflection of UV light before reaching the depth of the ceramic due to scattering. In other words, in polished Enamic

ceramics, due to light scattering from the surface of the ceramic, the UV light does not reach the deep parts of the ceramic. In spite of this, in 0.5 mm Enamic ceramic, this effect is less dominant due to the low thickness of the ceramic.

It can also be seen that in the polished ceramics, Emax and Feldspathic ceramics have less florescence loss than Enamic ceramics.

5. Conclusion

Three common ceramics in restorative dentistry, including Emax, Enamic and Feldspathic, in two thicknesses of 0.5 and 1 mm, were investigated in this study. The results showed that the Enamic ceramic exhibit weaker color fastness due to aging than the other two ceramics (in basic and excited states) and its ability to maintain fluorescence after aging. This is while Feldspathic ceramic showed good color stability against aging. The efficiency of Emax ceramic was between the two mentioned ceramics. It was observed that aging causes the ceramic to lighten due to surface destruction. In Enamic ceramic, a difference in the behavior of glazed and polished ceramic was observed due to aging. The blue tint in polished ceramic and the yellow-red tint in glazed ceramic respectively represent the formation of AlOOH groups and phase transformation due to penetration and moisture retention under the glaze layer. Two other ceramics showed much better stability due to the aging process.

Regarding the change of fluorescence of investigated ceramics due to aging, it was observed that Enamic ceramic exhibits a very intense color shift due to aging. This means that the restoration made with this ceramic will be prone to severe color change over time. This is while the other two investigated ceramics showed better stability against the

aging process and regardless of the mechanical parameters; they are better options than Enamic ceramic.

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