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Tam Seramik Sistemlerinde Üç Farklı Yaşlandırma Yönteminin Renk Değişimi Üzerine Etkileri

Effects of Three Aging Methods on Colour Differences of All-Ceramic Materials

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Özet. *Amaç*. Çalışmanın amacı; iki farklı kalınlıkta (0.5 ve 1.0 mm) hazırlanan üç farklı tam seramik sisteminde, üç farklı yaşlandırma yönteminin renk değişimi (**Δ**E_{00}) üzerine etkilerini test etmektir.

Materyal ve metod. İki farklı kalınlıkta hazırlanan Zirkonya Katana UTML (ZK), Prettau Anterior (PA) ve IPS-e.max press (EP), in vitro yapay hızlandırılmış yaşlandırma (AAA), termal döngüyle yaşlandırma (TC) ve otoklavda yaşlandırma (AA) yöntemleriyle test edilmiştir. Renk (CIE L * a * b *) değişimi üzerindeki etkileri bir spektrofotometre kullanılarak belirlenmiş ve varyans analizi (ANOVA) ile değerlendirilmiştir.

Bulgular. Yaşlandırma protokolleri, seramik sistemlerinin **Δ**E_{00}-değerlerini farklı düzeylerde (0.64-2.69) etkilemiş ve seramik kalınlıkları istatistiksel olarak anlamlı bulunmuştur (P <0.5). Genellikle (0.5 mm EP grubu hariç), yaşlandırma yöntemleri arasında en yüksek **Δ**E_{00} değeri AAA uygulanan örneklerde, ardından TC ve AA uygulamalarında olmuştur. Ayrıca, yaşlandırma protokollerinin test edilmesinde seramik kalınlığının anlamlı olduğu bulunmuştur. Ek olarak, test edilen seramik materyallerin **Δ**E_{00} değerleri klinik olarak kabul edilen seviyenin altındadır. *Sonuçlar*. İki farklı kalınlıkta hazırlanan ve bir yıllık in vitro yaşlandırma protokolü uygulanan

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üç seramik türü, test edilen yaşlandırma yöntemlerinden farklı düzeylerde etkilenmiş ve tüm seramik örnekleri arasında önemli renk farklılıkları belirlenmiştir.

Klinik Önemi: Üç yaşlandırma protokolünün (AAA, TC ve AA) bir yıllık uygulamaları, seramik sistemlerin ΔE_{00} değerlerinde (ZK, PA ve EP) önemli farklılıklara yol açmıştır. Tüm seramik tiplerinde yaşlandırma testleri arasındaki ΔE_{00} değerleri ile tam seramik kalınlıkları arasındaki farklar anlamlıydı.

Anahtar Kelimeler: Hızlandırılmış yaşlandırma, Otoklavda yaşlandırma, Termal döngüyle yaşlandırma, Renk değişimi, Dental seramikler.

Abstract. *Objective*. The aim of the study is to test the effects of three different aging methods on colour change (ΔE_{00}) in three different all-ceramic systems prepared in two thicknesses (0.5 and 1.0 mm).

Materials and methods. Zirconia Katana UTML (ZK), Prettau Anterior (PA) and IPS-e.max press (EP) prepared in two thicknesses were tested using *in vitro* artificial accelerated aging (AAA), thermocycling (TC) and autoclave aging (AA) methods. The effects on the colour (CIE L*a*b*) differences were determined using a spectrophotometer and evaluated by analysis of variance (ANOVA).

Results. The aging protocols affected the ΔE_{00}-values of the ceramic systems at different levels (0.64–2.69), and the ceramic thicknesses were statistically significant ($P < 0.5$). Generally (excluding the 0.5-mm EP group), the highest ΔE_{00} value among the methods of aging was in the AAA applied samples, followed by those in the TC and the AA. Also, the ceramic thickness was found to be significant in testing aging protocols. In addition, the ΔE_{00}-values of the ceramic materials tested were below the clinically accepted level.

Conclusions. Three ceramic types – prepared in two different thicknesses and applied for three 1-year *in vitro* aging protocols – were affected at different levels by the aging methods tested, and significant colour differences were determined among all the ceramic samples.

Clinical Significance: One-year applications of three aging protocols (AAA, TC and AA) resulted in significant differences in the ΔE_{00}-values of ceramic systems (ZK, PA and EP). The differences between the ΔE_{00}-values among the aging tests in the all-ceramic types and the all-ceramic thicknesses were significant.

Keywords: Accelerated aging, Autoclave aging, Thermocycling, Colour stability, Dental ceramics.

1. Introduction

Biocompatible and long-lasting full ceramic restorative materials have become more popular in meeting the aesthetic expectations of physicians and patients [1, 2]. The provision of colour harmony between natural teeth and ceramic restorations and the long-term preservation of this harmony are considered important criteria for aesthetic success [3]. The use of all-ceramic restorative materials is steadily increasing due to its light reflection and light transmission properties that are similar to natural teeth. Dental ceramic materials colour stability is important for the long-term protection of the aesthetic appearance of teeth [4].

Ceramic materials with different properties and contents are used in dentistry. These materials are in contact with the mouth tissues and liquids [5] while they perform their aesthetic and functional tasks and tend to age in the oral environment. Since aging can affect the ΔE values of all-ceramic materials, it is necessary to determine the behaviour of dental materials *in vitro*.

Aging generally simulates the effects of prolonged exposure to light, temperature and moisture in an artificial aging process [6]. Different aging methods are applied in *in vitro* studies to simulate the oral environment such as autoclaving [7-9] ultraviolet aging [10, 11], thermal-cycling [12-14], and artificial accelerated aging [8, 15-18].

In dental studies, glass infiltrated ceramics, zirconia-supported alumina, lithium disilicate-reinforced glass ceramics and dense sintered zirconia-based ceramics are used [19]. A lithium disilicate-reinforced IPS-e.max press (EP) heat-pressed whole ceramic material is preferred due to its durable, bio-compatibility and aesthetic appearance. Zirconia Katana UTML (ZK) is among the computer-aided design/computer aided manufacturing (CAD/CAM) systems. It is composed of coloured zircon blocks fabricated with yttrium-stabilized tetragonal zirconia polycrystal (Y-TZP) materials and is also used due to its good aesthetic and mechanical properties [20]. The Prettau Anterior (PA) system, which is not a CAD/CAM system, is mechanically produced. The milling process is manually done by a technician [21].

CIE is an organization established to set standards on colour and appearance. In dental research, the CIE L*a*b* colour system is used to evaluate colour changes [22]. This system defines the colour with three coordinate values (L*a*b*) obtained from spectral reflectance measurements taken with a spectrophotometer. These values are a numerical description of the colour position in a three-dimensional colour space [23]. The ΔE, between the two samples is expressed; it is derived using the formula ΔE_{00} [24] and refers to the total colour change [25]. A large value of ΔE means that the colour change between two samples is excessive.

In the literature, the effects of aging methods on the various properties of dental ceramics have been tested, while the effects of aging methods on ΔE-values have not been compared. The hypothesis of this study was intended to establish that the aging tests do not affect the colour stability of different ceramic materials and that the thickness and colour changes do not affect the colour stability of the ceramic materials. For this purpose, the effects of the three different aging methods on the ΔE_{00}-values of the three different full ceramic systems were examined at two thicknesses.

2. Material and Method

In this study, the ZK, PA and EP all-ceramic systems were used, and the technical and chemical contents are given in Table 1. Each material was prepared at two thicknesses (0.5 mm and 1 mm) to form the test groups and at 13 mm in diameter to match the spectrophotometer used for colour measurements. A total of 18 groups were studied; 180 samples, 10 in each group, were tested (Figure 1). The A2 colour was selected for all the samples, which were polished by a Renfert (GMBH, Germany) polishing disc.

Table 1. *Ceramic systems used in the study*

Type	Thicknesses	Brand	Producer	Composition
Yttrium stabilized zirconia	0.5 mm 1 mm	Katana	Noritake Dental Co, Nagoya, Japan	(ZrO2+HfO2+Y2O3)> %99, Yttrium oxide (Y2O3)>4.5-≤%6, Hafnium oxide (HfO2) ≤%5.0, other oxides ≤1.0%

Yttrium stabilized zirconia	0.5 mm 1 mm	Prettau Anterior	Zirkonzahn GmbH, Bruneck, Italy	Y2O3<%12, Al2O3<%1, SiO2 max 0.02%, Fe2O3 max %0.01, Na2O max %0.04
Lithium-disilicate cam ceramic	0.5 mm 1 mm	IPS-e.max press	Ivoclar Vivadent, Schaan, Liechtenstein	SiO2 %57-80, Li2O %11-19, K2O %0-13, P2O5 %0-11, ZrO2 %0–8, ZnO %0–8 and other oxides and ceramics pigments %0-≤10

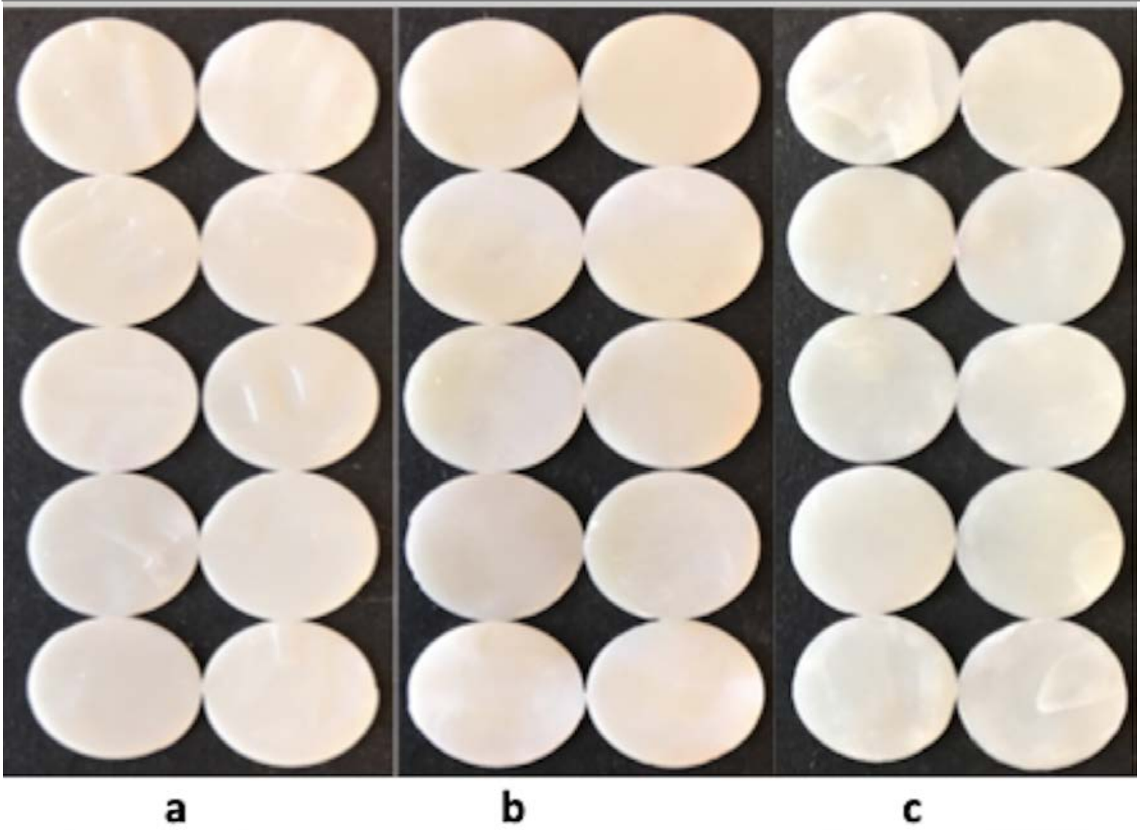

Figure 1. *ZK (a), PA (b), EP (c) all-ceramic specimens*

Preparation of specimens

Katana UTML (ZK)

In the ZK study, a Noritake Dental Supply Co. Ltd. Ultra-translucency multi-layer block product with 94.4% ZrO_2 and 5.4% Y_2O_3 content developed by a Japanese company was used. ZK is one of the CAD/CAM systems that can be produced from zirconia blocks. A CAM system can do five axis millings. ZK discs with thicknesses of 0.5 mm and 1 mm were cut, along with 2-mm and 1-mm diameter burs, on the Wieland Dental Zenotec

Select Hybrid CAD-CAM. The 1 mm cuts were done in 13 minutes and placed in an oven at 1520 °C where they were sintered for 6 hours. They were then smoothed with bur and sphere.

Prettau Anterior (PA)

In the PA study, 2 mm and 1 mm diameter zirconia polycrystal (ZP; lot: 98h14; factor: 1.2262) blocks were cut using a Rolland dwx51d CAD/CAM device to produce porcelain discs with a diameter of 13 mm and 0.5 mm and 1 mm thicknesses. After one 15-minute scraping in the Vita Zyrcomat 6000 ms oven at 1530 °C, they were sintered for 6 hours. They were then smoothed with bur and sphere. The samples were coloured with PA A2 colour solution (Prettau colour liquid, Zirkonzahn GMBH).

IPS e.max Press (EP)

In the EP study, 0.5-mm and 1-mm-thick porcelain discs (from an HT ingot) that were 13 mm in diameter were cut using a Roland dwx51d CAD/CAM device. The 1 mm disc (from a 12 x 98 mm x 12 mm Z-wax block) was machined using 2 mm and 1 mm diameter burs. Then, the discs were channelled and cuffed with the revetment. In the casting furnace, heat treatment was carried out at 850 °C for 1 hour, followed by 25 minutes at 910 °C in an Ivoclar EP 5010 furnace. After cleaning in the sand blanket, the discs were smoothed with appropriate brooms and sphered.

Aging Tests

Studies to test the physical effects of long-term clinical usage of dental materials *in vitro* for short durations and controllable conditions are commonly referred to as aging tests. In this *in vitro* study, the artificial accelerated aging (AAA), thermocycling (TC) and autoclaved aging (AA) methods were tested with 1-year aging protocols.

Artificial accelerated aging (AAA) tests

The specimens were subjected to an aging procedure consisting of exposure to ultraviolet light (UV) and water spray in the weathering machine (Atlas Xenontest Alpha high-energy weather device, Chicago USA) for 300 hours. According to the manufacturer, this period corresponds to 1 year in the mouth [26].

Thermocycling (TC) tests

The specimens were submitted to a thermocycling regimen of 10000 cycles in a 5–55 °C distilled water bath (Universal-Testing Machine MTD-500 Plus). In the TC test, it is reported that 10000 cycles are equivalent to approximately 1 year of function *in vivo* [27].

Autoclaved aging (AA) tests

The AA group was subjected to an accelerated aging test in an autoclave (Melag Vacuklav 23B +, Berlin, Germany) at 134 °C under 0.2 MPa pressure for 30 minutes, which would roughly correspond to 2 years. It was reported that a 30-minute period was sufficient to simulate 1 year of aging [28, 29].

Colour Measurements

To evaluate the colour change, CIELAB coordinates (L*, a*, b*) were recorded for each sample before and immediately after the aging procedure by means of a spectrophotometer (Lovibond RT Series Reflectance Tintometer UK). The L*a*b* values for the specimens were measured 3 times, and the mean colour coordinate values were recorded**.** The ΔE_{00} value of each specimen was calculated using the CIE Lab2000 formula $\Delta E_{00} = [(\Delta L/k_L\text{-}S_L)^2 + (\Delta C/k_C\text{-}S_C)^2 + [(\Delta H/k_H\text{-}S_H)^2 + R_T - (\Delta C/k_C\text{-}S_C) \times (\Delta H/k_H\text{-}S_H)]^{0.5}$ [24].

Statistical Analysis

The analysis of variance (ANOVA) for the repeated measurements in the comparison of ΔE_{00}-values for the different ceramic thicknesses (one-way ANOVA), materials type and the aging tests (multiway ANOVA) were performed using the IBM SPSS 20.0 package program, and a Tukey multiple comparison test was applied. Statistical significance was determined given a p-value less than 0.05.

3. Results

The highest ΔE_{00}-values in the PA and ZK systems were seen in the AAA protocol, in TC, and AA applications, respectively (Table 2, Figure 2). The highest colour changes for all samples were found in the TC group ZK 0.5 mm samples (ΔE_{00} = 2.69) and in the AAA group EP 0.5 mm samples (ΔE_{00} = 2.62), and the AA group EP 0.5 mm sample (ΔE_{00} = 2.15) value was even below the clinically acceptable level of perception of the 18 groups (ΔE < 3.5). However, the minimum colour change for all the samples was found in the AA group ZK 1 mm samples (ΔE_{00} = 0.64), the TC group PA 1 mm samples (ΔE_{00}

= 0.81), and in the AAA group PA 0.5 mm samples (ΔE_{00} = 1.20). All the 0.5-mm-thick all-ceramic samples (except the 0.5 mm PA aged AAA) showed more colour change than the 1-mm-thick ceramic samples (Table 2, Figure 2). The colour difference caused by the aging (according to the thickness of the ceramic) varied due to the ceramic types and the aging methods (Table 2, Figure 3). After the TC 0.5- and 1-mm groups, the AAA and the AA groups showed more colour changes, respectively (Figure 3). The effects of ceramic thickness on the ΔE_{00}-values were important ($P<.05$) in all three aging methods. The aging methods (AAA, TC and AA) affected the ΔE_{00}-values of ceramic systems at different levels (in the range 0.64–2.69). In other words, the aging protocol should be evaluated together with the aging protocol, ceramic type and thickness.

Table 2. *ΔE_{00}-values of aging tests in 3 all-ceramic systems*

Aging method	Ceramic types	Thickness mm	Min	Max	Standard deviation	Average
AAA	ZK	0.5	1.28	4.38	0.24	2.47
AAA	ZK	1.0	0.67	3.85	0.27	1.93
AAA	PA	0.5	0.33	2.12	0.44	1.20
AAA	PA	1.0	0.39	3.40	0.16	2.22
AAA	EP	0.5	0.65	4.38	0.33	2.62
AAA	EP	1.0	0.69	3.06	0.34	1.74
TC	ZK	0.5	0.30	5.89	1.04	2.69
TC	ZK	1.0	0.19	3.18	0.27	1.27
TC	PA	0.5	0.39	2.87	0.40	0.99
TC	PA	1.0	0.40	1.12	0.14	0.81
TC	EP	0.5	0.82	4.17	0.37	2.44
TC	EP	1.0	0.79	4.44	0.36	1.83
AA	ZK	0.5	1.10	3.35	0.20	1.54
AA	ZK	1.0	0.20	2.06	0.65	1.23
AA	PA	0.5	0.30	2.15	0.47	1.00
AA	PA	1.0	0.21	1.76	0.32	0.64
AA	EP	0.5	0.72	3.94	0.73	2.15

AA	EP	1.0	0.45	4.25	0.54	1.83

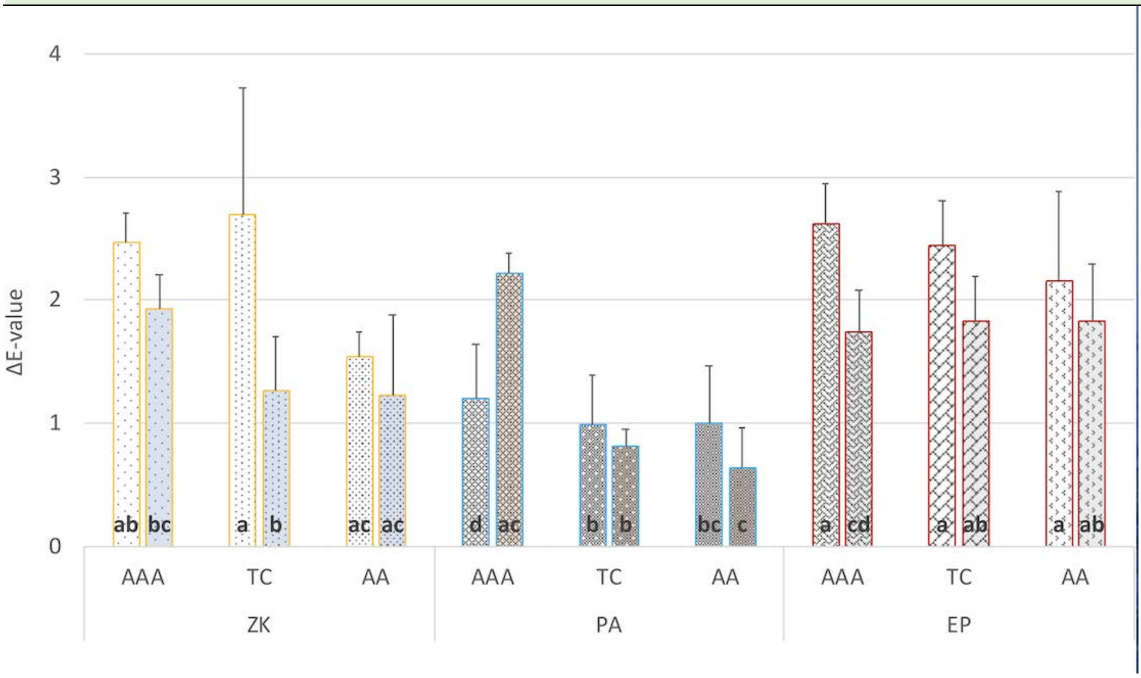

Figure 2. *The comparison of the ΔE-values of 0.5 and 1 mm thick all-ceramic types as split experiment. The ΔE-values indicated in the first bars are 0.5 mm, and the second bars are 1 mm thick all-ceramic specimens. LSD in the ZK and in the EP all-ceramic specimens was insignificant (P<.05), and in the TC and AA tests applied specimens was also insignificant (P<.05), and that was in the AAA test applied PA all-ceramic specimens was 0.95. LSD between the aging methods in the ZK and in the EP all-ceramic specimens was insignificant (P<.05), and it was 0.78 in the PA all-ceramic specimens tasted 3 aging methods.*

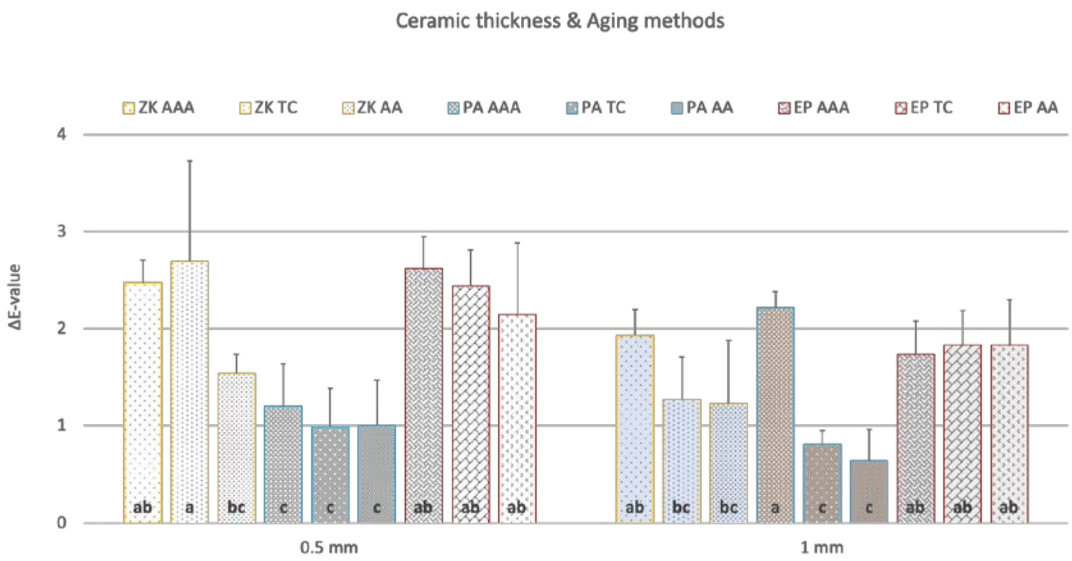

Figure 3. *The ΔE_{00}-values of 3 all-ceramic systems by aging methods, the letter indicated the multiway ANOVA of LSD (P<.05) for AAA was 0.65, for TC was 0.84, and for AA was 1.07. LSD (P<.05) between aging methods was 1.10.*

4. Discussion

The ceramics samples prepared in two different thicknesses at the beginning of the study do not show colour changes higher than ΔE = 3.5 (clinically detectable), as revealed by O'Brien [30] after three different aging protocol applications and ceramics with different thicknesses. The hypothesis that ΔE_{00}-values would be below the clinically perceptible level was accepted for the 18 groups. Previous studies have indicated that the CIE L*a*b* colour space of dental ceramics systems [31] are affected by different variables, such as the ceramic brand [32, 33], the thickness of the ceramics [33-35], the colour of the ceramics [33, 35], the particle size, the sintering temperature and duration [36] and the ZrO_2 and Y_2O_3 concentrations [37].

It was reported that the clinical change was clinically unacceptable when the colour change (ΔE) was greater than 3.5 [30]. It was also reported that the options of ceramic thickness may cause expanding of the optical properties of the full ceramics [38, 39]. In previous studies [40-42], the thickness effect was statistically significant in all the parameters analysed, and the colour differences they found in their studies were due to the differences in ceramic thickness.

It has been reported that changes in ceramic thickness can cause significant changes in the optical properties of all-ceramic materials [38, 39, 43]. It was also reported that the ΔE-value changes due to the thickness of glass-infiltrated aluminium oxide, lithium disilicate and yttrium-stabilized zirconia ceramic materials were not statistically significant among the samples in the same ceramic group, but were significantly affected among the different ceramic groups [20]. These results were similar only in the PA all-ceramic system, which was aged by the TC test, and the ΔE values of different ceramic thicknesses were found in different statistical groups in the other eight combinations.

Yttrium-stabilized zirconia is a chemically inert material with a high density. These characteristics may explain why the optical changes of the ZK and PA ceramic samples were so limited in the autoclave aging test according to the EP samples.

The colour change after aging in the autoclave depends on the pigment breakage (burning of metallic oxides) during the exposure to heat [44]. It is possible that the changes resulting from aging can only occur on the sample surface. Therefore, the effects of aging on the more homogeneous surface of zirconia are also less common. The various T-m factors affecting the phase transformation, the ZrO_2 particles [45] the size, the shape and the location, and the type and amount of stabilizing oxide [46] are important, but these factors cannot be controlled. Only technical approaches [47, 48], including surface treatments and polishing and laboratory and clinical procedures for zirconia [49-51], may be able to control it. Therefore, the different colour changes obtained in the results of different studies should be viewed with caution.

The higher colour change in all the ceramic systems with AAA may be due to UV and easier decomposition of metal oxides under UV. For EP, the maximum colour change of the samples (2.62) in the AAA application was different in terms of the chemical composition of the material (lithium disilicate glass ceramic), the lower durability of the zirconia [19], the penetration of water in the silica web [52] and, in particular, the easier degradation of the fine samples.

Due to the nature of the study, some limitations should be considered. In the *in vitro* experiment, three different all-ceramic materials were prepared in two different thicknesses and were exposed to three different aging stresses. The aging methods used in our study do not reflect the clinical conditions. It is very difficult to evaluate the colour change due to the long-term use of the ceramics that are clinically used in dentistry. In addition to future studies to address all these variables, *in vivo* studies to validate the results obtained here are required for more precise assessments.

5. Conclusion

Within the limitations of this study, it is concluded that the three aging methods significantly affected the ΔE_{00}-values of the ceramic specimens at different levels. The response of ceramic types with different levels of ΔE_{00}-values as a result of this *in vitro* aging tests showed that the colour change depends on the type of material. The fact that the standard error values in the ΔE_{00}-values obtained from the study were relatively high, and at different levels according to the ceramics types, caused the material type to be affected by the fabrication process and consequently to the aging practices. ΔE_{00}-values obtained from ceramic systems aged in three different methods by applying 1-year aging protocols are at different levels (0.64-2.69), indicating that all aging tests are applicable in *in vitro* aging studies of dental ceramics. Ceramic thicknesses were significantly differed ΔE_{00}-values in all-ceramic samples. The decrease in colour change with increasing ceramic thickness showed that the results obtained from 1 mm thick PA samples aged with AAA protocol were not always correct, and the results were dependent on the type of the all ceramic material and type of the applied aging protocol. The three tested all-ceramic systems acted independently in the different aging tests.

Disclosure Statement

The authors do not have any financial interest in the companies whose materials are included in this article.

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