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# **IN VITRO EVALUATION OF THE COLOR STABILITY OF TWO DIFFERENT LAYERING CERAMICS AFTER THERMOCYCLING IN COFFEE**

Mark Daou\* | Pascale Habre-Hallage\*\* | Elie Zebouni\*\*\*

## **Abstract**

Statement of the problem: Emphasis on the aesthetic result will always remain a key priority, especially in the anterior areas. Thus, the color stability of a restoration partially defines the success of this restoration over time. The most commonly used layering material is porcelain, which is in intimate contact with food and beverages in the oral cavity.

Purpose: The purpose of this in-vitro study is to assess the color stability of two different layering porcelains after thermocycling in coffee, and then compare the color stability of these ceramics with each other.

Materials and methods: Two blocks of E.max CAD shade A2 were cut in blue phase to have 40 identical 1 mm thick blocks (N = 40). The E.max blocks were then crystallized and a stainless steel device was manufactured, in which each block was inserted, and the layering ceramic was added to flare the outer edge of the device (2 mm of feldspathic porcelain in height). 20 blocks then received the type of ceramic E.max Ceram shade A1 (n = 20), and the other 20 the ceramic GC LiSi shade A1 (n = 20), then were placed in the furnace for firing and glazing according to the manufacturers' recommendations. The blocks were numbered at the upper right corner with a red ring diamond bur of diameter 016 under water irrigation. Then, a 3D model was fabricated, in which each block was inserted in a cassette and reproducible points were recorded for each measurement. Using a spectrophotometer (Vita Easyshade Advance), 3 measurements L\* a\* b\* of the color were taken for each block at time 0 (M1). All samples were then subjected to thermocycling (coffee at 55°C / distilled water at 5°C) for 1095 cycles simulating 1 year of clinical exposure to coffee. A second measurement (M2) is carried out, then the thermocycling continued until reaching 2190 cycles, at the end of which the third measurement (M3) was performed, and all the measurements transferred to Microsoft Excel software (2013). The L\*, a\* and b\* data were then collected and the color change  $\Delta E$  was calculated for each type of ceramic using the statistical software IBM SPSS Statistics (version 25.0).

Results: A statistically significant difference in color was observed within the two groups, as well as between the types of ceramics. After 2190 cycles, 65% of E.max Ceram specimens were above the acceptable threshold while 90% of GC LiSi specimens were above the same threshold.

Conclusion: The layering ceramics E.max Ceram and GC LiSi are both affected by coffee over time, but E.max Ceram seems much more stable than GC LiSi over 2 years of clinical simulation.

**Keywords:** Color stability - spectrophotometer - layering porcelain - lithium disilicate - thermocycling - staining – coffee – laboratory research.

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## **EVALUATION IN VITRO DE LA STABILITÉ DE COULEUR DE DEUX DIFFÉRENTES CÉRAMIQUES DE STRATIFICATION APRÈS THERMOCYCLAGE DANS LE CAFÉ**

### **Résumé**

Exposé du problème : L'accent mis sur le résultat esthétique restera toujours une priorité essentielle, notamment dans les zones antérieures. Ainsi, la stabilité de la couleur d'une restauration définit en partie le succès de cette restauration dans le temps. Le matériau de stratification le plus couramment utilisé est la porcelaine, qui est en contact intime avec les aliments et les boissons dans la cavité buccale.

Objectif : Le but de cette étude in vitro est d'évaluer la stabilité de la couleur de deux céramiques de stratification différentes après thermocyclage dans le café, puis de comparer la stabilité de la couleur de ces deux céramiques entre elles.

Matériels et méthodes : Deux blocs de E.max CAD de teinte A2 ont été découpés en phase bleue pour donner 40 blocs identiques de 1 mm d'épaisseur (N = 40). Les blocs E.max ont ensuite été cristallisés et un dispositif en acier inoxydable a été fabriqué, dans lequel chaque bloc a été inséré, et la céramique de stratification ajoutée pour arriver au bord extérieur du dispositif (2 mm de porcelaine de hauteur). 20 blocs ont ensuite reçu le type de céramique E.max Ceram teinte A1 (n = 20), et les 20 autres la céramique GC LiSi teinte A1 (n = 20), puis ont été placés dans le four pour cuisson et glaçage selon les recommandations du fabricant. Les blocs ont été numérotés au niveau du coin supérieur droit avec une fraise diamantée bague rouge de diamètre 016 sous irrigation d'eau. Ensuite, un modèle 3D a été fabriqué, dans lequel chaque bloc a été inséré en cassette et des points reproductibles ont été enregistrés pour chaque mesure. À l'aide d'un spectrophotomètre (Vita Easyshade Advance), 3 mesures L\* a\* b\* de la couleur ont été effectuées pour chaque bloc au temps 0 (M1). Tous les échantillons ont ensuite été soumis à un thermocyclage (café à 55°C / eau distillée à 5°C) pendant 1095 cycles simulant 1 an d'exposition clinique au café. Une deuxième mesure (M2) est effectuée, puis le thermocyclage s'est poursuivi jusqu'à atteindre 2190 cycles, au terme desquels la troisième mesure (M3) a été effectuée, et toutes les mesures transférées vers le logiciel Microsoft Excel (2013). Les données L\*, a\* et b\* ont ensuite été collectées et le changement de couleur  $\Delta E$  a été calculé pour chaque type de céramique à l'aide du logiciel statistique IBM SPSS Statistics (version 25.0).

Résultats : Une différence de couleur statistiquement significative a été observée au sein des deux groupes, ainsi qu'entre les deux types de céramiques. Après 2190 cycles, 65% des échantillons E.max Ceram étaient au-dessus du seuil acceptable tandis que 90% des échantillons GC LiSi franchissaient même seuil.

Conclusion : Les céramiques de stratification E.max Ceram et GC LiSi sont toutes deux affectées par le café au cours du temps, mais E.max Ceram semble beaucoup plus stable que GC LiSi sur 2 ans de simulation clinique.

**Mots clefs:** stabilité de couleur - spectrophotomètre - céramique de stratification - disilicate de lithium - thermocyclage - coloration – café – recherche au laboratoire

## Clinical Implications

Porcelain is the only used layering material covering indirect restorations, and therefore the most affected by extrinsic factors in the oral cavity. No study has ever reviewed the E.max Ceram and GC LiSi, (two pioneers in layering porcelain) color stability over time, when exposed to coffee. This study will help understand the behavior of those two dental materials over a 2-year clinical simulation, and bring an answer to the discoloration problem clinicians face on a daily basis.

## Introduction

Over the past three decades, drastic changes have taken place in the field of dental restorations, accompanied by rapid changes in the materials and techniques used, which have increased the demand for cosmetic dentistry. Indeed, the latter has become a very important element in contemporary society. Patients are now asking for much more targeted and precise results.

Being the most popular material in dentistry, dental ceramics, mainly composed of feldspar, quartz and kaolin [1], is best known for its aesthetic appearance, its biocompatibility and its smooth surface [2–6]. Finesse, and Omega 900 feldspathic porcelain materials were fabricated and fired according to manufacturer recommendations. Porcelain blocks were polished through 0.25  $\mu\text{m}$  diamond polishing paste. Thirty-six enamel specimens were obtained and milled to a 2 mm (+0.5 mm; the translucency, the color gradient, the surface texture and the mechanical properties place this material as a reference material in dentistry, and therefore allow the creation of ceramic crowns that mimic the shape and color of natural teeth [3–5,7]. There is a lack of information in the few studies reporting on the stainability of dental porcelain materials. However no studies have been found that investigated effects of polishing methods and staining agents

on the color stability of dental porcelains. The purpose of this study was to evaluate the effects of different polishing techniques on the color stability of various dental porcelains. From its manufacture to its placement in the mouth, this material is exposed to several factors which can however affect its optical and mechanical properties. We can divide them into intrinsic factors specific to the making of ceramics, and other extrinsic factors linked to the eating habits of individuals [5,8–11]. Cervical adaptation, occlusal adjustment and final finishing/polishing are procedures to be performed at the dental office after adhesive cementation. Final adjustments may result in loss of ceramic glaze, which requires new polishing of the ceramic surface, with special attention for selection of adequate materials and instruments. The purpose of this study was to evaluate the efficiency of different vehicles associated with diamond pastes indicated for dental ceramic polishing. Two polishing pastes (Crystar Paste and Diamond Excell). Several *in vitro* studies have demonstrated the effect of coloring drinks on the color of dental ceramics [8,9,12,13] microhardness, and color stainability of a vitreous CAD-CAM lithium disilicate ceramic with a glazed or mechanically polished surface. Material and methods. Specimens (N=160). But even if the crown's shape is kept perfectly identical to a reference tooth over time, a simple perceptible change in its color is considered a failure of the crown [9,14].

Nowadays, dental ceramics are mostly vitrocereamics; they are very aesthetic and also have high mechanical properties [15]. Lithium disilicate is the most widespread among them, in its different forms: machined, pressed and layered [16]. The latter thus constitutes the main material of layering porcelain, which gives the crown a highly aesthetic appearance. It is also the outer layer of the crown, which is in continuous intimate contact with everyday's foods and beverages.

These solutions affect natural teeth as well as the restorations present in the mouth by creating color deposits on the surfaces. Among these solutions, we can cite cocoa mixed with saliva, wine and tea, but also coffee, a drink widely used all over the world.

Several studies have studied the effect of coloring solutions on natural teeth and on direct and indirect restorations; some of them compare the impact of different solutions between each other on these restorations [8–10,13,17,18] microhardness, and color stainability of a vitreous CAD-CAM lithium disilicate ceramic with a glazed or mechanically polished surface. Material and methods. Specimens (N=160, while others compare the same category of solution and their consequences [19]. We can then note that coffee is the most used solution in all studies regardless of the protocol followed.

The published studies using coffee as the main immersion solution vary according to each protocol, but with one thing in common: the continuous immersion of specimens in the tank. One of these studies evaluates the effect of several types of coffee on composite restorations over 30 days, another over 54 hours [13] IPS e-max CERAM ([CER] n=72, others for a period of 4 weeks [9,10] or even 24 hours only [17]... according to Palla et al [13] IPS e-max CERAM ([CER] n=72, a continuous immersion of 54 hours clinically simulates the consumption of coloring drinks for 3 years. On the other hand, according to Guler et al [17], 24 hours of coffee immersion roughly simulates regular coffee consumption for 30 days. Furthermore, in Al-Samadani's study [19], an incubation bath at 37°C is used to simulate the temperature of the oral cavity, while Gürdal et al [20] adopts thermocycling in two baths, the first of 55°C and the second of 5°C.

The purpose of this *in vitro* study is precisely to assess the color stability of two different layering ceramics, after thermocycling in coffee. The null hypothesis would be that no

statistically significant difference in color will be found between the baseline measurement and the 2 year clinical simulation measurement.

## Materials And Methods

Two E-max CAD blocks of shade A2 HT (B40L, Ivoclar Vivadent AG) were selected (Figure 1). They were cut millimeter by millimeter using a microtome (Exakt) and under continuous irrigation of water to give rectangular specimens (Figure 2). Identical blocks, 1 mm thick each, 16 mm wide and 18 mm long were obtained and 40 blocks (N = 40) chosen arbitrarily.

A stainless steel device was then manufactured according to the dimensions of the blocks, in order to be able to position the block and layer the ceramic in a reproducible manner (Figure 3). Then, the blocks, still in their blue phase, were crystallized in a ceramic furnace (Programat P300, Ivoclar Vivadent AG) at 850°C for 20 minutes [8], and divided into 2 groups of 20 blocks each. In the manufactured device, each block was positioned on the internal table at 2 mm from the external edge, covered by the layering porcelain according to the manufacturer's instructions, until flaring the external edge of the device (Figure 4).

20 blocks of E.max were then layered with layering ceramic E.max Ceram (Ivoclar Vivadent) dentin of shade A1 (n = 20), and the 20 others of layering ceramic initial LiSi (GC America) dentin of shade A1 also (n = 20). The blocks of the same group were put in the furnace for firing, then glazed and tested in the device to make sure of their height. Within each group, the 20 blocks were numbered from 1 to 20 at the upper right corner using a diamond ball red ring bur on turbine (diameter 016) and under water irrigation (Figure 5). An additional point was also engraved on the upper left corner of the 20 blocks which have received the LiSi ceramic, using the same technique already mentioned. Furthermore, a virtual 3D model was made using Autocad software, from



Figure 1: E.max CAD blocks

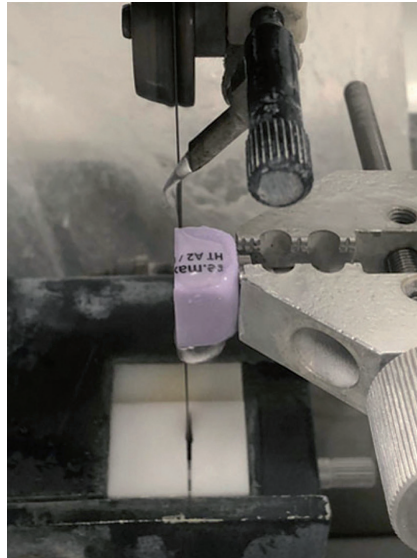


Figure 2: Exact microtome cutting E.max blocks

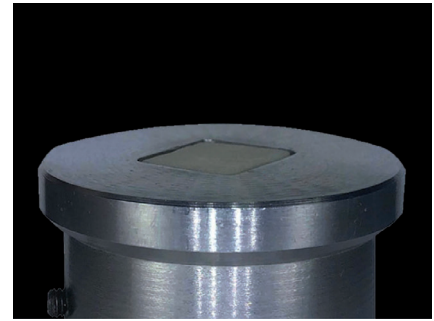


Figure 3: Manufactured stainless steel device for the reproducible ceramic layering

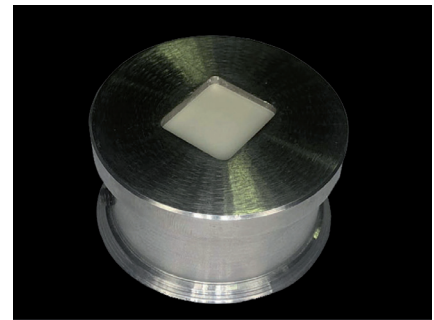


Figure 4: Ceramic build-up flaring the external edge of the device

the dimensions of the blocks, then printed in 3D with plexiglass; the three holes allow the spectrophotometer to be positioned each time in a reproducible manner so that the shade of the same fixed point can be recorded (Figure 6). The base of this model is in opaque white plexiglass and the other sides totally transparent for simple and precise access [21]; each block can therefore be inserted into a cassette in this device for reproducible recordings of its color (Figure 7). Using a spectrophotometer (Vita EasyShade Advance), 3 measurements according to the nomenclature  $L^*a^*b^*$  [22] were taken for each block at the level of 3 precise points (named 1, 2 and 3), where the spectrophotometer head was placed perpendicular in contact with the white base block [10] (Figure 8).

The baseline measurement was noted M1. The spectrophotometer was calibrated before each measurement

according to the manufacturer's recommendations, and all this was carried out by the same experienced operator.  $L^*$  indicates the brightness, and its value varies between 0 (black) and 100 (white). The values  $a^*$  and  $b^*$  indicate the color in the red-green and yellow-blue axes respectively. Positive  $a^*$  values show a shift towards red, negative ones towards green. Similarly, the positive  $b^*$  values underline a yellow spectrum, and the negative ones the blue spectrum [10,23–25]. The values thus provided were transferred to an Excel file. The coffee solution was then prepared at the rate of 300 ml of water for each 3.6 g of Nescafé (Nescafé Red Mug, Nestlé Brasil, Araras / SP, Brasil) [4,17,26], and all the specimens were inserted in an incubator containing hot coffee (at 55°C) and distilled water (at 5°C), and this for 2 minutes each (Figure 9). Fresh coffee was added every 8 hours in the coffee bath maintained

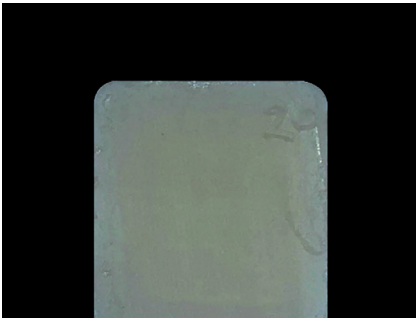


Figure 5: E.max blocks numbered on the upper right corner

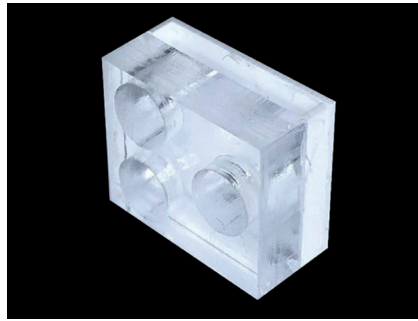


Figure 6: The manufactured plexiglass 3D model for reproducible recordings

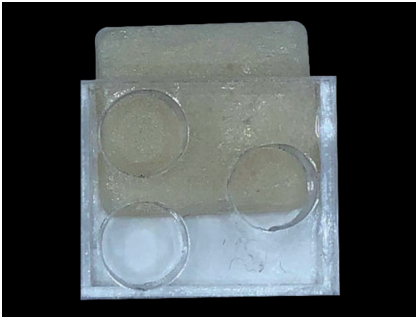


Figure 7: Insertion of the blocks in the 3D model

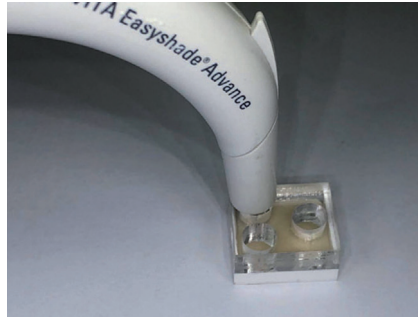


Figure 8: The spectrophotometer positioning through the access holes

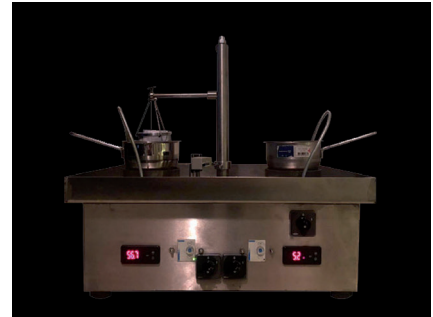


Figure 9: The thermocycling machine in hot coffee and cold water

at 55°C, and ice cubes in the distilled water bath maintained at 5°C.

After 1095 cycles, the samples were taken out and a first measurement of the same points was carried out for each block, then transferred to Excel; this measurement was noted M2. 2190 cycles were performed in total. Each cycle was then the sum of 2 minutes of immersion in coffee, 20 seconds of journey between coffee and distilled water, 2 minutes of immersion in distilled water and 20 seconds of journey back to coffee. Once the cycles were finished, a new recording was made for the 40 samples, using the device at the same 3 points, and transferred to Excel software. The color difference  $\Delta E$  was then calculated from the mathematical formula  $\Delta E = [\Delta L2 + \Delta a2 + \Delta b2]^{1/2}$ .

The Kolmogorov-Smirnov tests were conducted to assess the normal distribution of quantitative variables at the level of each group. Parametric

tests were applied since the variables followed the normal distribution. One-Sample t tests were conducted to compare the average values of  $\Delta E$  with the theoretical perceptibility threshold "2.6" and acceptability threshold "3.7" values.

## Results

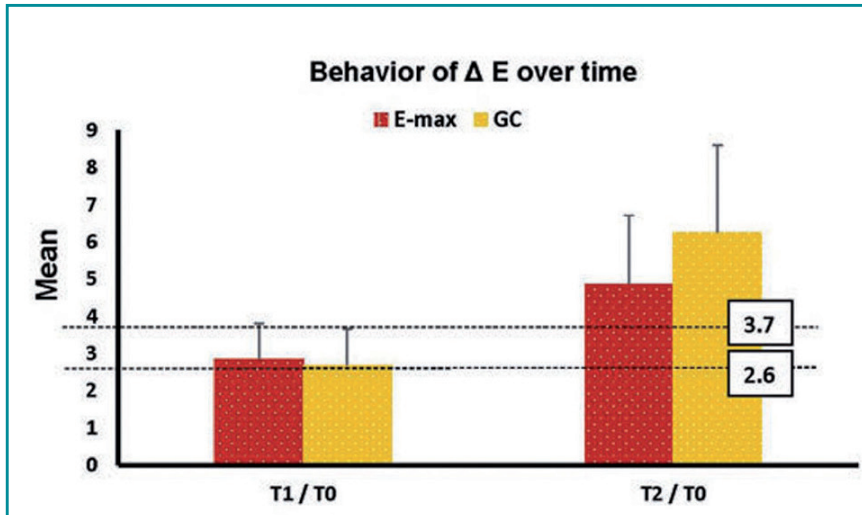
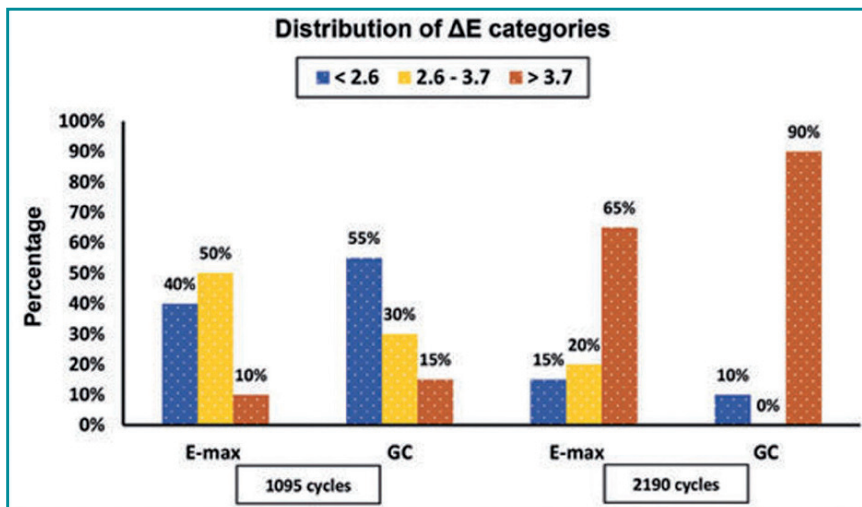
The mean and standard deviation of the  $\Delta E$  value for the E.max Ceram and GC LiSi specimens after 1095 cycles (T1) and 2190 cycles (T2) are presented in table 1. For the E.max group, the average  $\Delta E$  which was  $2.839 \pm 0.973$  after 1095 cycles increased significantly to  $4.856 \pm 1.855$  after 2190 cycles (-p-value <0.001). For the GC group, the average  $\Delta E$  which was  $2.692 \pm 0.961$  after 1095 cycles increased significantly to  $6.261 \pm 2.341$  after 2190 cycles (-p-value <0.001) (Figure 10).

The mean  $\Delta E$  was not significantly different between E.max and GC after 1095 cycles (-p-value = 0.633). However, it was significantly greater in GC specimens compared to E.max specimens after 2190 cycles (-p-value = 0.042).

This study showed that in T1 (after 1095 cycles), the mean value of  $\Delta E$  was not significantly different from the perceptible threshold "2.6", and this for both the E.max group (-p-value = 0.285) and the GC group (-p-value = 0.672). After 2190 cycles, the mean value of  $\Delta E$  was significantly above the acceptable threshold "3.7" for the E.max group (-p-value = 0.012) and the GC group (-p-value <0.001).

After 1095 cycles, the  $\Delta E$  was not different between E.max and GC. Also,  $\Delta E$  was not significantly different from the perceptible threshold "2.6" for the two types of ceramics. After 2190 cycles, the  $\Delta E$  was greater with the GC

	$\Delta E$ at T1 /T0	$\Delta E$ at T2/T0	-p-value
E.max Ceram	$2.839 \pm 0.973$	$4.856 \pm 1.855$	<0.001
GC LiSi	$2.692 \pm 0.961$	$6.261 \pm 2.341$	<0.001
-p-value	0.633	0.042	

Table 1: Variation of  $\Delta E$  over time in ceramicsFigure 10: Behavior of  $\Delta E$  over time in ceramics according to the perceptible and acceptable thresholds.Figure 11: The  $\Delta E$  categories according to the thresholds for ceramics.

compared to the E.max. However, the  $\Delta E$  was greater than the acceptable threshold “3.7” for E-max and the GC.

The specimens’ percentages, according to the thresholds’ values at T1 and T2, are shown in the figure 11. The distribution of the  $\Delta E$  categories was significantly different between the two ceramics (-p-value = 0.038).

## Discussion

In the present study, a statistically significant difference is detected between the different groups, hence the null hypothesis is rejected. This indicates that the color of the specimens of the two groups (E.max and GC) significantly increased after 2190 cycles of thermocycling in coffee simulating 2 years of consumption of this drink. All the more, the statistical results underline that the  $\Delta E$  relative to the E.max group after 1095 cycles simulating 1 year, is more or less identical to that of the GC group at the same time, indicating values below the perceptibility threshold “2.6” for 40% of the E.max samples and 55% of the GC samples [27], and above the acceptability threshold “3.7” for 10% of the E.max and 15% of the GC. On the other hand, after all the cycles completed, the  $\Delta E$  -GC- seems to be more affected by the coloring solution than  $\Delta E$  -E.max-, thus crossing the acceptability threshold for 90% of the specimens.

In this study, we decided to use CAD/CAM frameworks. These, presented in blocks in the blue phase, were easy to mill during this stage, since they have not yet acquired their final mechanical properties. It is only after the final crystallization that these properties are optimal. This is why we opted for the cutting of E.max CAD pre-crystallized blocks in blue phase, of uniform thickness of 1 mm, thus

simulating the thickness of the infra-structure of a conventional crown [13,28,29] IPS e-max CERAM [CER] n=72. The crystallization completed, these blocks were ready to receive the layering porcelain. But to be able to start this procedure in a reproducible way for each block, the creation of a stainless steel device seemed to us as the only possible solution, in which the blocks placed one by one would allow the layering of identical ceramic thickness of 2 mm [30]. At this stage, and before starting the experiment, a shade measurement of each block was imperative; we then decided to manufacture a plexiglass device perforated in 3 access points, to, on one hand, record a reproducible measurement of the exact same points, and on the other hand, increase the precision of our study.

Among the closest studies to ours, evaluating the ceramics' color stability, is that of Palla et al [13] IPS e-max CERAM [CER] n=72. The latter sought to assess the color difference of several types of E.max (CAD, Press and Press without glazing) following a thermocycling and immersion in different coloring solutions, including coffee. According to Palla et al, immersion in coloring drinks was done for 54 hours, thus simulating 3 years of clinical simulation. However, a coffee drinker drinks an average of 3 cups of coffee a day for 5 minutes each. If it is assumed that the contact between the coffee solution and the teeth is only for 2 minutes each time, the duration of continuous daily exposure of this solution to the dental surfaces would only be for 6 minutes per day. This is why in our study we considered that 1 year of exposure to coffee is equivalent to 2190 cycles.

In this same study by Palla et al, the results seem to agree with those collected in our study. Significant variation in the color of lithium disilicate ceramic occurs for samples soaked in coffee.

The closest to our study, measuring the color stability of feldspathic porcelain, is undergone by Jain et al. [30]

in 2013. It assesses the color stability of 3 different feldspathic ceramics (Vita, Ceramco-3 and Duceram Kiss), all mounted on a rigid metal base similar to ours, and soaked in coloring solutions, including coffee, for 90 days. An evaluation of the color of all specimens was recorded at 0, 45 and 90 days using a spectrophotometer. Likewise, the results of Jain et al agree perfectly with ours, demonstrating a significant change in the color of the 3 types of feldspathic ceramic (especially Ceramco-3) after 90 days in coffee.

In the literature, a large number of studies have sought to emphasize the effect of coffee consumption on dental ceramics. The most used variables in a large majority of these studies are the surface texture rather than the type of ceramic, and this in order to be able to establish a relationship between texture and color. However, the results seem close for all these studies. Furthermore, the advantage of our study is the fact of carrying out a thermocycling in coffee under conditions similar to those of the oral cavity; thermocycling, in an incubation bath of hot coffee on one hand and fresh distilled water on the other hand, has never been tested on layering ceramic. Furthermore, a comparison of two types of ceramic with each other under these same conditions has never made a subject of research.

## Conclusion

No study assessing the color stability of two brands of layering ceramic has been done in the literature. This is strange since this porcelain, which is the most used type of porcelain, is the most affected by consumed colorants in the oral cavity, as it is in intimate contact with common food and beverages. The advantage of this study is the fact that it seeks to provide an answer to a question with a direct clinical aim, and to draw a conclusion allowing the clinician to ask the laboratory technician to use this or that ceramic.

According to the results of our study, the two brands of layering ceramic

E.max Ceram and GC LiSi were affected by thermocycling in coffee, to different degrees. After 1 year, this effect is more or less similar on these ceramics. This variation in color is all the more accentuated for a period simulating 2 years of clinical situation, where the color change of the GC ceramic far exceeds that of the E.max on one hand, and the acceptable threshold for the success of restoration on the other hand. Hence the E.max Ceram layering ceramic has shown better color stability over time.



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