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Color stability of stained and glazed monolithic zirconia following thermocycling

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Thesis submitted to the faculty of the
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DISCLAIMER

ABSTRACT

Color stability of stained and glazed monolithic zirconia following thermocycling

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U.S. Army Advanced Education Program in Prosthodontics Graduate Program, 2023

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CLINICAL RELEVANCE STATEMENT

This study provides the information on color stability of extrinsic staining and glazing on monolithic zirconia restorations during artificial aging.

ABSTRACT

INTRODUCTION: Achieving satisfactory esthetics and transparency are a key requirement for zirconia restorations to be comparable to other ceramics. The extrinsic staining and glazing can fulfill the requirement but there is currently lack of information on their longevity.

OBJECTIVES: This study investigates the surface color changes (ΔE) of stained and glazed monolithic zirconia restorations during artificial aging. Three extrinsic staining and glazing systems were applied to groups of monolithic zirconia discs and the color stability was measured using a spectrophotometer.

METHODS: Ninety monolithic zirconia discs (IPS e. max ZirCAD LT A1) were prepared into three groups of extrinsic staining and glazing: Miyo, ICE Zirkon Stains Prettau, and IPS Ivocolor. The baseline was measured using the Vita EasyShade Advance 4.0 spectrophotometer. A thermocycling machine was used to simulate the oral environment and ΔE was measured after 5,000, 10,000, 20,000, 30,000, 40,000 and 50,000 thermocycles. The change in ΔE was calculated from the baseline.

RESULTS: The average change in ΔE after 50,000 thermocycles was 0.688 for Miyo, 1.267 for IPS Ivocolor and 0.726 for ICE Zirkon Stains Prettau. Each sample was scored according to perceptibility ($\Delta E = 0.8$) and acceptability ($\Delta E = 1.8$) thresholds, designated as pass or fail. Seventy-three percent of Ivocolor Zirkon Stains Prettau samples failed on perceptibility threshold ($p<0.0023$) and thirty percent failed the acceptability threshold ($p<0.0001$). The proportions for Miyo and ICE Zirkon Stains were consistently the same. Thirty-seven percent failed on perceptibility threshold, but all samples passed the acceptability threshold.

CONCLUSIONS: There was a significant difference between the systems in the proportion of samples meeting or exceeding one or more thresholds for perceptible change and acceptable change after 50,000 cycles. Miyo and ICE Zirkon Stains Prettau were not statistically significantly different and showed all acceptable samples at 50,000 cycles. In contrast, IPS Ivocolor showed a substantial proportion of unacceptable color changes.

TABLE OF CONTENTS

CHAPTER 1: INTRODUCTION

Zirconia has been the versatile material and with the help of modern technology, it became a material of choice in dentistry. Zirconia has gained popularity due to biocompatibility, high fracture toughness and radiopacity.[1] Also resembling similar optical characteristics of natural dentition, zirconia has been accepted as an anterior indirect restoration. [2,3] However, zirconia has shown low esthetic performance compared to other ceramics and still lacking on satisfactory transparency. [4,5] This becomes a disadvantage to highly esthetic demanding cases and other type of ceramics had fulfilled the role instead.

Because zirconia lacks transparency without custom characterization, manufacturers have developed numerous staining and glazing systems to overcome this esthetic deficiency. With proper custom characterization, zirconia can satisfy the high esthetic demands of modern dentistry and patients can benefit from a versatile material. Color changes of the characterization due to intraoral function over time may limit the longevity and quality of restorations.[6] It is important to know how long the characterization will remain unchanged in the oral environment.

One of the most challenging procedures in esthetically demanding dentistry is to match a restoration that resembles natural dentition. Color is defined as a phenomenon of light or visual perception that enables one to differentiate otherwise identical objects.[7] It is an important factor in dentistry to assess and correctly match a patient’s tooth shade to a restoration. Rods and cones in our retina help us with this task by correctly translating wavelengths that are not absorbed by an object and then reflected.[8] Color is described based on the Munsell color space in terms of value, chroma and hue, all of which are based off wavelengths in the visible light spectrum.[9] The difference in color can be measured objectively by calculating the ΔE and it represents the distance between two colors in three dimensions.[10]

The Commission International de l'Eclairage (CIE) developed the CIELab* system to standardize the ability to measure and calculate color differences. The CIELab* system made it possible to calculate the differences between two colors in a way that corresponded to visual perception.[11,12] Most spectrophotometers use the ΔE determined from the CIELab* value to quantify the color difference of a tooth and shade to be matched. ΔE_{ab} is calculated as follows:

$$\Delta E^*_{ab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2}$$

The L* represents the lightness and it ranges from 0 to 100 with 0 being a black object and 100 being a white object. The a* represents red or green and it ranges from -90 to 70. Positive a* represents greenness and negative a* represents redness. The b* represents yellow or blue and it ranges from -80 to 100. Positive b* represents yellowness and negative b* represents blueness.[10] In 2001, the CIE Published the CIEDE 2000 color difference formula to incorporate other variables and parameters. The formula incorporates hue and compensates for neutral colors, lightness and chroma differences. The CIEDE 2000 is calculated as follows:

$$\Delta E^*_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \frac{\Delta C'}{k_C S_C}\frac{\Delta H'}{k_H S_H}}$$

In the formula, ΔL' represents lightness, ΔC' represents chroma and ΔH' represents hue. R_T is a function that accounts for the differences between chroma and hue in the blue region.[10] The S_L, S_C, and S_H are the weighting functions for the lightness, chroma, and hue components, respectively. The values calculated for these functions vary according to the position of the sample being considered in CIELAB color space. The k_L, k_C, and k_H values are the parametric factors to be adjusted according to different viewing parameters such as textures, backgrounds, and separations for the lightness, chroma and hue components, respectively.[13]

Comparing the CIEDE2000 and CIELAB color difference formulas show that the CIEDE2000 formula provides a better fit in the evaluation of color difference threshold of dental ceramics.[14] Many studies suggest this formula because it provides better indicators of human perception and acceptance of color differences in tooth shades.[14,15,16] Based on these recommendations, the CIEDE2000 formula will be utilized in this study.

In dentistry, visual judgement is the most frequently used method of evaluating tooth shades. There are two major thresholds for assessing color differences: perceptibility threshold and acceptability threshold.[17] The smallest color difference that can be detected by 50% of observers corresponds to the 50:50% perceptibility threshold.[18] The difference in color that is acceptable for 50% of observers corresponds to the 50:50% acceptability threshold.[19] However, different studies suggest different thresholds due to the subjective nature of the process.[20] Paravina found CIEDE 2000 (ΔE_{00}) to be 0.8 with 50:50% perceptibility and 1.8 with 50:50% acceptability and CIELAB (ΔE_{ab}) to be 1.2 with 50:50% perceptibility and 2.7 with

50:50% acceptability.[21] Based on the lowest thresholds provided by Paravina, ΔE_{00} of 0.8 with 50:50% perceptibility and 1.8 with 50:50% acceptability will be utilized in this study.

In dental offices, shade matching is conducted using commercially available shade guides that rely on visual perception by the dentist, assistant, and patient. Unfortunately, this method of shade matching is unreliable and inconsistent. Research by Okubo found that both pre- and post-doctoral dentists correctly match shades only about 50% of the time.[22] A spectrophotometer can help to overcome this issue. Many studies suggest that using a spectrophotometer is a more dependable and consistent method of shade selection due to its ability to quantify color.[15,20,23] Different spectrophotometers have different reliability and accuracy. Reliability measures the consistency of a spectrophotometer to make repeated measurements of the same shade, while accuracy refers to the ability of a spectrophotometer to provide a correct shade match.[24] A study by Kim-Pusateri et al. evaluated four spectrophotometric devices. They found similar reliability of over 96% with differences ranging from 0.5% to 9% in the four devices, but variability in accuracy. The highest accuracy was found with Vita EasyShade of 92.6% and reliability of 96.4%.[24]

Thermocycling is intended to simulate the thermal stress to which restorative materials and teeth are exposed by consuming drinks and food. The process leads to aging of the specimens in a short period of time.[25] Different periods of thermocycling are among the methods used to simulate the aging process of dental materials in in-vitro studies. These methods attempt to simulate the hydrolytic degradation that occurs in the oral cavity.[26] A temperature between 5°C and 55°C with 30 seconds of dwell time and 10 seconds of transfer time was proposed in ISO 11405 recommendations.[27] Gale and Darvell postulated that every 10,000 thermal cycles approximately correspond to one year of clinical function with 20 to 50 cycles considered equivalent to a single day.[28]

The aim of this study was to investigate the surface color changes (ΔE_{00}) of stained and glazed monolithic zirconia restorations during artificial aging. Three extrinsic staining and glazing systems were applied to groups of monolithic zirconia discs and the color stability was measured using a spectrophotometer. The following two null hypotheses were made: 1) There will be no significant difference in color changes after thermocycling for 50,000 cycles within each group; 2) There will be no significant difference in color stability among three extrinsic staining and glazing systems.

CHAPTER 2: MATERIALS AND METHODS

The sample was designed using the Tinkercad Web CAD Software (Tinkercad.com, San Francisco, CA) into 11.5 mm x 11.5 mm x 2 mm disc. Ninety monolithic zirconia discs were milled from e. max ZirCAD LT A1 (Ivoclar Vivadent, Schann, Liechtenstein), using PrograMill PM7 (Ivoclar Vivadent, Schaan, Liechtenstein) and sintered using 3M ESPE Lava Therm (3M Center, St. Paul, MN) at 1500°C. Ninety samples were divided into three groups: MiYo (Jensen Dental, North Haven, CT), ICE Zirkon Stains Prettau (Zirkonzahn GmbH, Bruneck, Italy), and IPS Ivocolor (Ivoclar Vivadent, Schaan, Lichtenstein).

MiYo stain and glaze were applied to the thirty discs according to manufacturer’s instructions. First, the discs were steam cleaned and air dried. MiYo

Trans Shade A was measured using Ohaus Scout SPX2201 (Parsippany, NJ) into 0.2g and sixty drops of InSync Glaze Liquid (Jensen Dental, North Haven, CT) were added. The stain was mixed homogeneously, and Gilson Pipetman pipette (Gilson Incorporated, Middleton, WI) was used to measure 20 μL onto each sample disc. Dekema Austromat 624i porcelain furnace (Dekema Dental-Keramikofen GmbH, Freilassing, Germany) was used at 745°C to fire sample discs. Once air cooled, 0.2g of InSync Glaze Paste was mixed with sixty drops of InSync Glaze liquid. 10 μL of mixed glazed solution was applied top of the stained sample discs and fired at 745 °C with the porcelain furnace.

Zirkon stain and glaze were applied to the thirty discs according to manufacturer's instructions. First, the discs were steam cleaned and air dried. A1 Prettau Zirkonzahn was measured into 0.2g and sixty drops of Zirkonzahn ICE Stain Liquid were added. The stain was mixed homogeneously and then 20 μL was applied onto each sample disc. The porcelain furnace was used at 800°C to fire sample discs and then air cooled. 0.2g of Glaze Plus (Zirkonzahn GmbH, Bruneck, Italy) was mixed with sixty drops of Zirkonzahn ICE Stain Liquid. 10 μL of mixed glazed solution was applied top of the stained sample discs and fired at 800 °C with the porcelain furnace.

Ivocolor stain and glaze were applied to the thirty discs accordingly to manufacturer's instructions. First, the discs were steam cleaned and air dried. Ivocolor Shade Dentin SD1 was measured into 0.2g and sixty drops of Ivocolor Mixing Liquid were added. The stain was mixed homogeneously and then 20 μL was applied onto each sample disc. The porcelain furnace was used at 710°C to fire sample discs and then air cooled. 0.2g of Ivocolor Glaze Paste was mixed with sixty drops of Ivocolor Mixing Liquid. 10 μL of mixed glazed solution was applied top of the stained sample discs and fired at 710 °C with the porcelain furnace.

All discs were engraved with first letter of group name and number on the side of the disc. The baseline of L*, a* and b* were measured three times using a 3D-printed sample holder with a spectrophotometer (EasyShade 4.0, Vita Zahnfabrik, Bad Sackingen, Germany) (Figure 1). Once baseline was measured, the discs were stored in 3D-printed trays (Figure 2). A thermocycling machine (Thermocycling Test Apparatus, Sabri Dental Enterprises, Downers Grove, IL) was set at 5 °C and 55 °C water baths, with a dwell time of 30 seconds and 10 seconds of transfer time. All samples were thermocycled for 5,000, 10,000, 20,000, 30,000, 40,000 and 50,000 cycles (Figure 3). Respective L*, a* and b* data were measured on each designated number of cycles and ΔE_{00} was calculated by comparing to the baseline.

CHAPTER 3: RESULTS

The mean and standard deviation of the change in ΔE_{00} from baseline to designated cycles were plotted by group (Graph 1). All three groups showed a near linear rise in ΔE_{00} up to 20,000 cycles. Zirkon showed no further rise, MiYo declined slightly, while Ivocolor continued to rise.

1) Descriptive statistics

a) Initial samples

Descriptive statistics for the sets of 30 samples prepared with the three groups are shown in Table 1. Values for L*, a* and b* were compared for the three groups, consistent with staining for the same A3 color. All 9 sets of data showed p>0.05 for both tests for normality, and modest skewness and kurtosis, with no prominent outliers. Therefore, the values were consistent with random variance around a mean. For the larger means of L* and b*, the coefficient of variance (CV) was modest, similarly consistent with preparation variance.

MiYo showed the lowest variance for all three CIELab* color parameters, and consistent with this and the lack of outliers, it also showed the narrowest range of values. The range for L* in MiYo was nearly half that of the other two groups.

b) 50,000 cycle samples

Descriptive statistics for the sets of 30 samples prepared with the three groups are shown in Table 2. Values for L*, a* and b* were comparable for the three systems, consistent with staining for the same A3 color. All 9 sets of data showed p>0.05 for both tests for normality, and modest skewness and kurtosis, with no prominent outliers. Therefore, the values were consistent with random variance around a mean. The CV for all three parameters was modest, similarly consistent with preparation variance.

As for the initial samples, MiYo showed the lowest variance for all three CIELab* color parameters, and consistent with this and the lack of outliers, it also showed the narrowest range of values.

2) Analytic statistics

a) Inter-sample variance of L*, a* and b* color parameters of initial samples

To facilitate evaluation of the initial and 50,000 cycle color parameters, 3-D scatterplots of the L*, a* and b* values for the samples of three groups were plotted

(Graph 2). Broadly, for the initial samples, a* and b* showed a narrow distribution around a diagonal L* axis for both the initial and final cycles, with MiYo showing a narrower distribution along the L* diagonal and Zirkon showing tight clustering along the a* and b* axes. This pattern suggested a linear and near linear relationship between the parameter values for the initial samples within each group. Partial correlation analysis confirmed a strong correlation between pairs of parameters for each group (Table 3).

To investigate the relationship further, L* was plotted against a* and b* and b* against a*, for the initial samples, and the data fitted to a line model using non-linear regression. As shown in Graph 3, there is an apparent linear relationship between pairs of parameters. In all three groups, increasing values of L* were associated with a decrease in value for a* and b*, and therefore a* and b* increased together.

A line model was fitted to each set of values. Most plots showed a normal distribution for the residuals, and as shown in Table 4, most plots showed an R^2 value near 1.000, indicating a good fit to the linear model. Across the 30 samples in the initial datasets, for all three groups, L* showed a negative correlation to a* and b*, while b* showed a positive correlation to a*.

b) Inter-sample variance of L*, a* and b* color parameters of 50,000 cycles samples

A similarly high partial correlation between pairs of parameters for each system was found for the 50,000 cycle samples. The relationship between parameters was further investigated by linear regression (Table 5).

The color variance within the sets of initial and 50,000 cycles samples, likely due to variance in preparation (with other smaller error components such as random measurement error) showed a strong correlation between the L*, a* and b* color space parameters. This suggested that the variance both before and after thermocycling was due to a consistent pattern of variance along a color vector line. This interpretation was consistent with the apparent shift in samples due to thermocycling seen in the 3D scatterplots.

c) Cumulative changes in in-color parameters with thermocycling

(i) Comparison of rate of L*, a* and b* parameter change against correlated parameter (regression slope)

For L*, a* and b*, two-way ANOVAs were used to compare the regression slope values (mean, SE, n=30) at initial and 50,000 cycles for the three groups. For L* vs a*, the initial and final slopes for Ivocolor and Zirkon did not show a significant difference (Sidak's multiple comparisons test; $p \geq 0.99$), while MiYo showed a significant increase (less negative; $p = 0.026$). For L* vs. b*, and a* vs. b*, no group showed a significant change due to thermocycling ($p \geq 0.84$). Thus, with the exception of MiYo L* vs. a*, the relationships between pairs of parameters were unchanged after thermocycling. And the variance between samples in color in a group after 50,000 cycles followed the same pattern as the initial sample variance. This was again consistent with a potential shift along a color vector line due to thermocycling.

(ii) Changes in L*, a* and b*

Two-way repeated measures ANOVA was used to evaluate mean changes in the three-color parameters for the three systems. For the ANOVA assumptions, sphericity was not a concern as there were only two levels for the repeat measure (0 and 50,000 cycles). The samples passed Levene's test for homogeneity of variance ($p \geq 0.18$), and Q-Q plots showed most values lying on a diagonal line. The groups were of the same size ($n = 30$). As shown in Graph 3, there were differences between the group and in the patterns of change with thermocycling.

For L*, there was significant between subjects (group) effect ($p < 0.001$), and significant within-subjects effects for cycles ($p < 0.001$) and the interaction of cycles group ($p < 0.001$). Post hoc comparisons (Tukey's HSD correction) for group showed no significant difference between Miyo and Zirkon ($p = 0.98$), but Ivocolor was significantly different from MiYo and Zirkon ($p < 0.001$). Examination of the simple effects within group system showed several significant differences (Table 6). All three groups showed a significant decrease in L* between the initial and 50,000 cycle values. Ivocolor L* was significantly different from MiYo, with lower values, at both the initial and 50,000 cycles. In comparison to Zirkon, Ivocolor was not significantly different initially, but was significantly lower than Zirkon for comparison of 50,000 cycles samples.

For a*, there was a significant between subjects (group) effect ($p < 0.001$), and a non-significant within-subject effect for cycles ($p = 0.095$) but a significant effect for the interaction of cycles group ($p = 0.004$). Post hoc comparisons (Tukey's HSD correction) for group showed no significant difference between Ivocolor and Zirkon ($p = 0.14$), but MiYo was significantly different from Ivocolor and Zirkon ($p < 0.001$).

Examination of the simple effects within cycles system showed several significant differences (Table 7). MiYo and Zirkon did not show a significant change in a between the initial and 50,000 cycles samples. However, Ivocolor showed a modest, but significant increase. In comparison to Ivocolor and Zirkon, MiYo showed a consistently lower value of a* at both initial and 50,000 cycles.

For b*, there was a significant between subjects (group) effect ($p < 0.001$), a significant within subjects' effects for cycles ($p = 0.031$) and the interaction of cycles group ($p = 0.041$). Post hoc comparisons (Tukey's HSD correction) for group showed no significant differences between Ivocolor and Zirkon ($p = 0.68$), but MiYo was significantly different from Ivocolor and Zirkon ($p < 0.001$). Examination of the simple effects within cycles group showed several significant differences (Table 8). All three groups failed to show a significant change in b* between the initial and 50,000 cycle values. MiYo b was significantly different from the other two groups, with lower values, at both the initial and 50,000 cycles. Ivocolor and Zirkon did not show any significant differences.

d) Development of perceptible and tolerable color change with thermocycling

(i) Cumulative changes at 50,000 cycles

A perceptible change in color has ΔE_{00} of ≥ 0.8, and an acceptable color change has a limit of 1.8. These values, and an intermediate value of 1.3 were used to score the ΔE_{00} values of the group samples at 50,000 cycles to determine cumulative changes. The proportion of Ivocolor samples exceeding all three thresholds was significantly greater than the proportion of MiYo and Zirkon samples which were equal for all three thresholds. The ratio of Ivocolor fails to the other two systems was 1.97, 3.62 at the 0.8 and 1.3 thresholds respectively. MiYo and Zirkon both showed no fails at the acceptable threshold, whereas about one third of the Ivocolor samples were no longer acceptable (Table 9).

CHAPTER 4: DISCUSSION

Through spectrophotometric analysis, this study evaluated the surface color changes (ΔE_{00}) of stained and glazed monolithic zirconia discs during artificial aging through thermocycling. The collected data met the assumptions for relevant parametric tests (normal distribution and homogeneity of variance). Repeated measured tests were used to compare samples tested at cycle intervals. The initial L*, a* and b* sample values for each system showed a modest CV, consistent with variance due to sample manufacture.

Each group showed a strong linear correlation between pairs of color system parameters. This was broadly characterized by an inverse correlation between L*, a* and b* (with a* and b* positively correlated). The relationships between parameters appeared to not show any significant change after 50,00 cycles. Samples that showed a shift in color did so by translocation along the same parameter regression lines as for sample variance. That is, color change due to thermocycling appeared to be due to the same mechanism as sample color variance during sample production.

Repeated measure two-way ANOVA comparing initial, and 50,000 cycles samples showed that thermocycling induced changes in one or more color parameters. The group differed in their patterns of change, and Zirkon showed the least change in parameters overall. Therefore, there was strong statistical support for a difference in color arising as a result of thermocycling, and a difference in group response to thermocycling, with Zirkon shown the least changes.

After 50,000 cycles there was a significant difference between the group in the proportion of samples meeting or exceeding one or more thresholds for perceptible change and acceptable change. MiYo and Zirkon were not significantly different and showed no unacceptable samples at 50,000 cycles. In contrast, Ivocolor showed a substantial proportion of unacceptable color changes.

The results of this study support the rejection of the null hypotheses that thermocycling cause color changes after 50,000 cycles. The other hypothesis, that different extrinsic staining and glazing system reacted differently causing two out of three systems were acceptable after thermocycling.

CHAPTER 5: CONCLUSION

MiYo and Zirkon demonstrated excellent color stability after enhanced by artificial aging through thermocycling. All extrinsic staining and glazing system were perceptible after 5 years of simulated oral environment but color change on MiYo and Zirkon were still acceptable. Ivocolor was perceptible and unacceptable after 5 years of simulated oral environment.

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FIGURES

Figure 1: 3D-printed sample holder with disc inside. Tip of spectrophotometer inserted with lid in place for consistent orientation onto center of the disc during measurements.

Figure 2: 3D-printed trays with discs inside. A lid was placed over the tray and water draining holes were incorporated with the design.

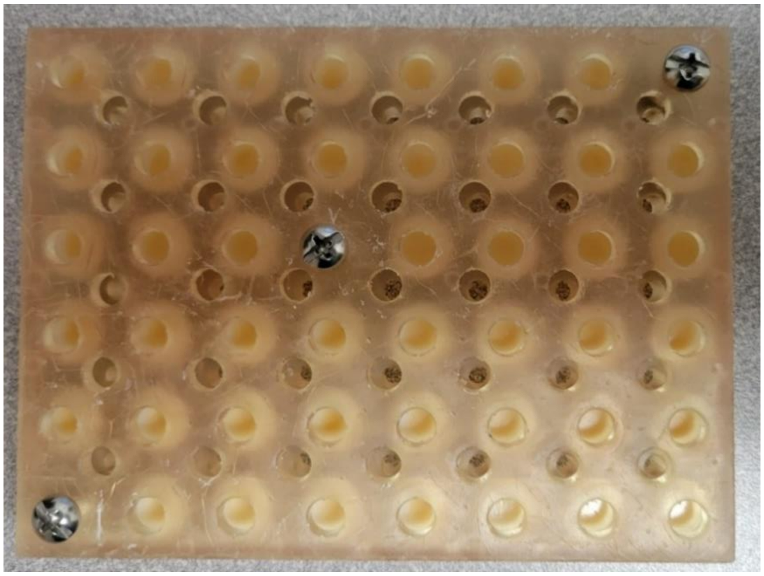
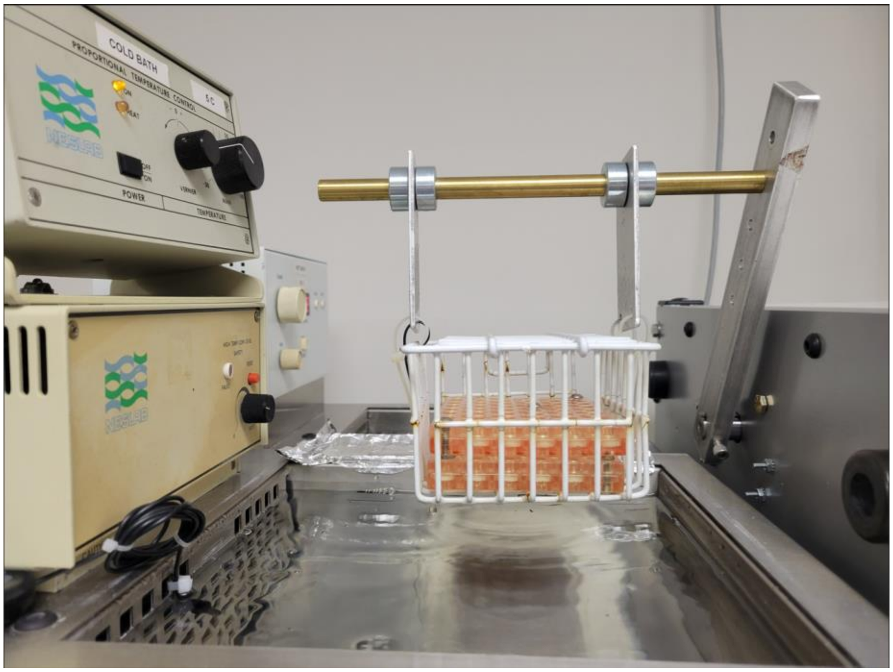
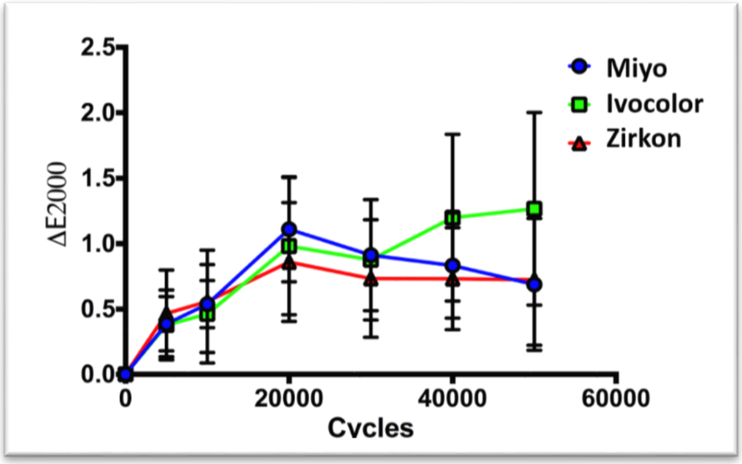


Figure 3: 3-D printed trays with samples were secured within a basket. Thermocycling machine rotates the arm between 5°C and 55°C water baths and enhanced artificial aging on the samples.

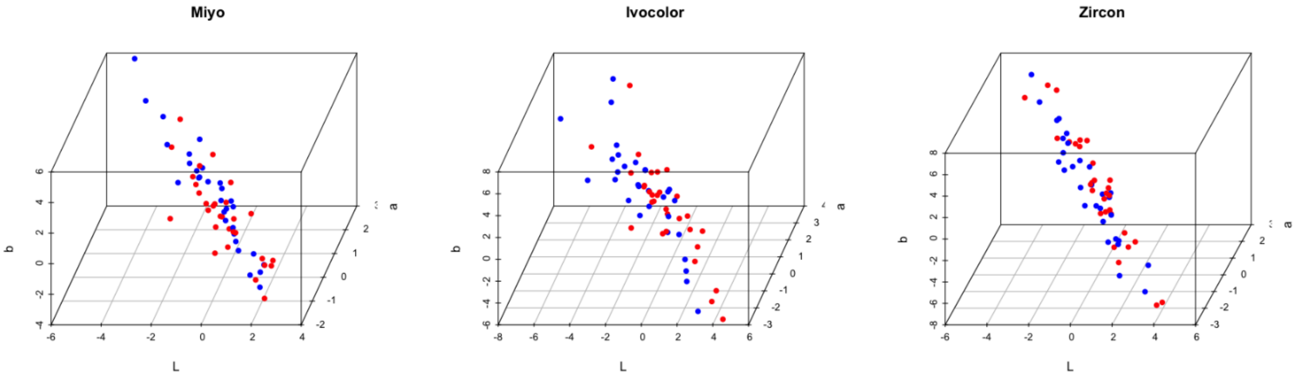


GRAPHS

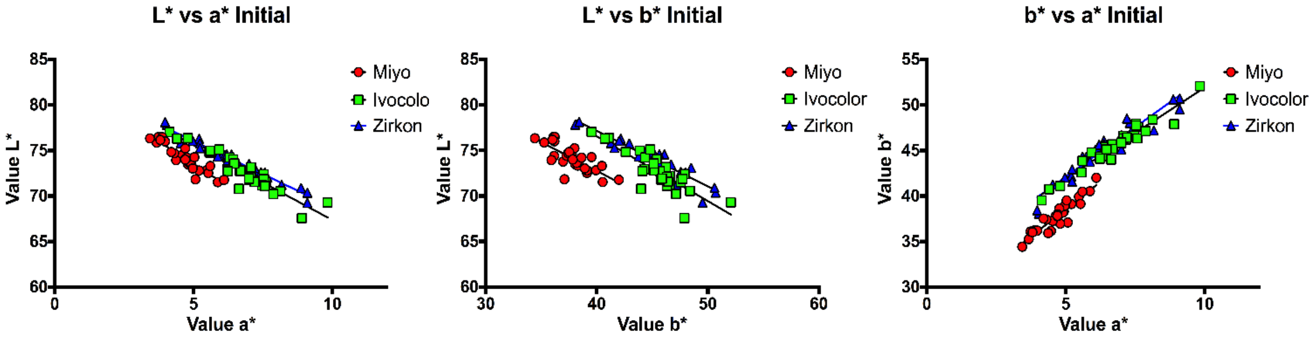
Graph 1: Mean and standard deviation of the change in ΔE_{00} from baseline to designated cycles were plotted by group.



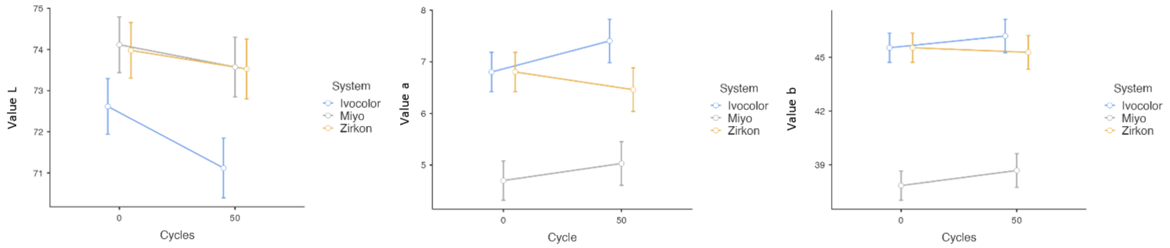
Graph 2: 3-D scatterplots of L*, a* and b* for Miyo, Ivocolor and Zirkon. Values for each of the 30 samples are plotted as residuals from the initial parameter mean. Red symbols are initial values, blue are values after 50000 cycles. Note the scales are different.



Graph 3: Plots of Initial sample L against a and b, and b against a, for Miyo, Ivocolor and Zirkon**.** Regression lines for each system are shown.



Graph 4: Plots of marginal means and standard errors for two-way repeated measures ANOVA of L*, a*, and b* for the three Systems at Initial (0 Cycles) and 50,000 Cycles.



TABLES

Table 1: Descriptive statistics of Initial Samples. Values for the groups of 30 samples are shown. [1]: Standard deviation. [2]: Coefficient of variance. [3]: The lowest p value for the D'Agostino and Pearson and Shapiro-Wilk normality tests are shown. Values>0.05 are consistent with a normal distribution. [4]: Outliers were determined using the ROUT algorithm, Q=1%.

		Mean	SD[1]	CV (%)[2]	Minimum	Maximum	Range	Normality[3]	Skew-	Kurtosis	Outlier[4]
L*	Miyo	74.12	1.391	1.88	71.53	76.47	4.94	0.36	0.090	-0.571	0
	Ivocolor	72.62	2.112	2.91	67.57	77.03	9.46	0.72	0.120	0.316	0
	Zirkon	73.98	2.012	2.72	69.27	78.13	8.86	0.69	-0.230	0.357	0
a*	Miyo	4.699	0.654	13.91	3.43	6.10	2.67	0.80	0.053	-0.215	0
	Ivocolor	6.803	1.198	17.61	4.13	9.83	5.70	0.40	-0.070	1.086	0
	Zirkon	6.416	1.358	21.17	3.97	9.10	5.13	0.54	0.301	-0.243	0
b*	Miyo	37.84	1.688	4.46	34.43	42.00	7.57	0.69	0.307	0.153	0
	Ivocolor	45.53	2.474	5.43	39.53	52.07	12.54	0.16	-0.231	1.552	0
	Zirkon	45.20	3.100	6.86	38.07	50.70	12.63	0.53	-0.410	0.225	0

Table 2: Descriptive statistics of 50000 Cycle Samples. Values for the groups of 30 samples are shown. [1]: Standard deviation. [2]: Coefficient of variance. [3]: The lowest p value for the D'Agostino and Pearson and Shapiro-Wilk normality tests are shown. Values>0.05 are consistent with a normal distribution. [4]: Outliers were determined using the ROUT algorithm, Q=1%.

		Mean	SD[1]	CV (%)[2]	Minimum	Maximum	Range	Normality[3]	Skew-	Kurtosis	Outlier[4]
L*	Miyo	73.58	1.712	2.33	69.33	76.10	6.77	0.29	-0.601	0.112	0
	Ivocolor	71.12	2.315	3.26	65.43	75.47	10.04	0.86	-0.220	-0.107	0
	Zirkon	73.53	1.923	2.62	69.47	77.23	7.76	0.84	-0.061	-0.484	0
a*	Miyo	5.030	0.895	17.79	3.60	7.47	3.87	0.27	0.564	0.585	0
	Ivocolor	7.404	1.287	17.38	4.53	10.20	5.67	0.51	0.012	0.472	0
	Zirkon	6.461	1.261	19.51	4.13	9.37	5.24	0.79	0.253	-0.347	0
b*	Miyo	38.69	2.119	5.48	34.9	43.83	8.93	0.74	0.296	0.031	0
	Ivocolor	46.19	2.604	5.64	39.63	52.10	12.47	0.28	-0.379	1.062	0
	Zirkon	45.27	2.954	6.52	39.17	51.27	12.10	0.93	-0.119	-0.324	0

Table 3: Partial correlation analysis of Initial sample parameters L, a, b. The top number is the Pearson's r, the bottom is the p value.

	L*			a*			b*		
	Miyo	Ivocolor	Zirkon	Miyo	Ivocolor	Zirkon	Miyo	Ivocolor	Zirkon
L*	*	*	*	*	*	*	*	*	*
a*	-0.922 <0.001	-0.931 <0.001	-0.980 <0.001	*	*	*	*	*	*
b*	-0.770 <0.001	-0.834 <0.001	-0.939 <0.001	0.930 <0.001	0.968 <0.001	0.957 <0.001	*	*	*

Table 4: Summary of results for non-linear regression fit to a linear model for Initial sample parameters L*, a*, b*. Values for the groups of 30 samples are shown. The null hypothesis that one curve fitted all three system data sets for a plot was tested.

Plot	Parameters	R^2	Slope	SE slope	Intercept	SE Intercept	One curve to fit all	One intercept F(2,84),	One slope F(2,84),
L* vs a*	Miyo	0.849	-1.961	0.156	83.33	0.741	p<0.0001[1]	p=0.81	0.023
	Ivocolor	0.867	-1.641	0.122	83.78	0.839			
	Zirkon	0.960	-1.451	0.056	83.29	0.365			
L* vs b*	Miyo	0.592	-0.634	0.099	98.11	3.765	p<0.0001[2]		
	Ivocolor	0.696	-0.712	0.089	105.0	4.055			
	Zirkon	0.882	-0.609	0.042	101.5	1.912			
b* vs a*	Miyo	0.865	2.403	0.179	26.55	0.851	p<0.0001[3]	0.0001	0.20
	Ivocolor	0.937	1.999	0.098	31.93	0.675			
	Zirkon	0.916	2.184	0.125	31.19	0.819			

Table 5: Summary of results for non-linear regression fit to a linear model for 50000 Cycle sample L*, a*, b* parameters. Values for the groups of 30 samples are shown. The null hypothesis that one curve fitted all three system data sets for a plot was tested.

Plot	Parameters	R^2	Slope	SE slope	Intercept	SE Intercept	One curve to fit all	One intercept F(2,84),	One slope F(2,84),
L* vs a*	Miyo	0.849	-1.512	0.146	80.99	0.75	p<0.0001[1]	p=0.055	0.45
	Ivocolor	0.867	-1.661	0.131	83.42	0.98			
	Zirkon	0.960	-1.479	0.071	83.06	0.47			
L* vs b*	Miyo	0.760	-0.625	0.066	97.58	2.57	p<0.0001[2]	p=0.26	p=0.45
	Ivocolor	0.677	-0.732	0.096	104.9	4.42			
	Zirkon	0.932	-0.628	0.032	102.0	1.46			
b* vs a*	Miyo	0.961	2.321	0.089	27.01	0.45	p<0.0001[3]	p<0.0001	p=0.004/ 0.053
	Ivocolor	0.934	1.956	0.098	31.70	0.74			
	Zirkon	0.969	2.306	0.079	30.37	0.52			

Table 6: Significant differences in L* across System and Cycle group comparisons. Simple effects were evaluated using Tukey's HSD multiple comparisons correction. The first number is the p value, the second the difference in L* between 0 and 50000 cycles. (i.e., L*0-L*50). NS: not significant (p>0.05).

L*		**Miyo vs.**		**Ivocolor vs.**		**Zirkon vs.**	
	Cycles	**0**	**50**	**0**	**50**	**0**	**50**
Miyo	**0**	x	x	x	x	x	x
	50	0.009 0.54	x	x	x	x	x
Ivocolor	**0**	0.03 1.50	ns	x	x	x	x
	50	<0.001 3.00	<0.001 2.45	<0.001 -1.50	x	x	x
Zirkon	**0**	ns	ns	ns	<0.001 -2.86	x	x
	50	ns	ns	ns	<0.001 -2.41	0.049 0.45	x

Table 7: Significant differences in a* across System and Cycle group comparisons. Simple effects were evaluated using Tukey's HSD multiple comparisons correction. The first number is the p value, the second the difference in a* between 0 and 50000 cycles. (i.e., a*0-a*50). NS: not significant (p>0.05).

a*		**Miyo vs.**		**Ivocolor vs.**		**Zirkon vs.**	
	Cycles	**0**	**50**	**0**	**50**	**0**	**50**
Miyo	**0**	x	x	x	x	x	x
	50	ns	x	x	x	x	x
Ivocolor	**0**	<0.001 -2.10	ns	x	x	x	x
	50	<0.001 -2.71	<0.001 -2.38	0.042 0.60	x	x	x
Zirkon	**0**	<0.001 -2.10	<0.001 -1.77	ns	ns	x	x
	50	<0.001 -1.76	<0.001 -1.43	ns	0.027 0.94	ns	x

Table 8: Significant differences in b* across System and Cycle group comparisons**.** Simple effects were evaluated using Tukey's HSD multiple comparisons correction. The first number is the p value, the second the difference in b* between 0 and 50000 cycles. (i.e., b*0-b*50). NS: not significant (p>0.05).

b*		**Miyo vs.**		**Ivocolor vs.**		**Zirkon vs.**	
	Cycles	**0**	**50**	**0**	**50**	**0**	**50**
Miyo	**0**	x	x	x	x	x	x
	50	ns	x	x	x	x	x
Ivocolor	**0**	<0.001 -7.69	<0.001 -6.85	x	x	x	x
	50	<0.001 -8.34	<0.001 -7.50	ns	x	x	x
Zirkon	**0**	<0.001 -7.69	<0.001 -6.85	ns	ns	x	x
	50	<0.001 -7.43	<0.001 -6.59	ns	ns	ns	x

Table 9: Number of samples exceeding thresholds after 50000 Cycles: Samples were scored according to thresholds of 0.8, 1.3 and 1.8, and designated as Pass (did not exceed threshold) or failed (exceeded threshold). The statistical significance of the proportions in each System at each threshold were tested by Fisher's exact test. As the proportions for Miyo and Zirkon were consistently the same, post hoc pairwise comparisons were not needed, and Ivocolor was the significantly different system.

Threshold	**0.8**			**1.3**			**1.8**		
System	**Pass/Fail**	**Proportion fail**	**p**	**Pass/Fail**	**Proportion fail**	**p**	**Pass/Fail**	**Proportion fail**	**p**
Miyo	19/11	0.37		26/4	0.13		30/0	0.0	
Ivocolor	8/22	0.73	0.0023	16/14	0.47	0.0049	21/9	0.3	0.0001
Zirkon	19/11	0.37		26/4	0.13		30/0	0.0	