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Accelerated Aging Effects on Color Stability of Potentially Color Adjusting Resin-based Composites

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Clinical Relevance

The overall color stability as a result of artificial aging helps determine the long-term viability of resin-based composites in clinical application scenarios. Omnichroma and Venus Pearl presented superior color stability as compared with other established resin brands.

SUMMARY

The aim of this study was to compare the effects of accelerated aging on the overall color stability of potentially color adjusting commercial resin-based composite resins. Thirty specimens (10 mm diameter and 2.5 mm thick; n=6) were fabricated using five different materials: Estelite Omega, GC Kalore, Venus Pearl, Harmonize, and Omnichroma. Color measurements were taken for each sample using a spectrophotometer before and after submitting samples through the artificial aging process (Q-sun Xenon Test Chamber, 102 min light at 63°C black panel temperature; 18 min light and water spray per ASTM G155) for a total of 300 hours (12.5 days). The total color difference (ΔE*ab) was calculated using SpectraMagic NX software and analyzed using one-way analysis of variance and Tukey test. The results for color change (ΔE*ab) were statistically significant. Omnichroma and Venus Pearl presented superior color stability and the lowest overall color change, whereas GC Kalore and Harmonize presented significant color change that would be considered clinically unacceptable (ΔE*ab > 3.3).

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INTRODUCTION

Several different esthetic resin-based composites are currently available in the market. Color matching and long-term stability are some of the greatest challenges newer formulations attempt to address as color mismatch and composite discoloration are still considered major causes for restoration replacement.[1]

Composite resins that exhibit color adjustment potential intend to simplify options for esthetic and restorative procedures. The term popularly advertised as *chameleon effect* is more appropriately described as blending effect, a perceptual phenomenon in which

colors are perceived to have a better match than if they were observed separately, acquiring a color resembling that of the adjacent tooth structure.[2] The clinical advantages of such materials include an improved esthetic appearance, simplified shade matching, a reduction on the number of shade guide tabs, and compensation for small color mismatches.[2]

Recently, a newly introduced composite (Omnichroma, Tokuyama Dental, Tokyo, Japan) claims to use "structural color" technology to esthetically match every patient shade (from A1 to D4) with a single shade. Structural color is expressed only by the physical properties of light and not by pigments or dyes. Different wavelengths of light are amplified or weakened by the structure of the material itself, expressing colors other than what the material may actually be.[3] This material has shown promising results in terms of color adjustment potential,[4] shade matching (pre- and postbleaching),[3] and clinical applications,[5] but the fundamental question regarding its long-term color stability has yet to be published.

Color stability is defined as the ability of a material to resist changes in its apparent color after being exposed to prolonged challenging conditions.[6] These factors often include ultraviolet (UV) light exposure, humidity, changes in temperature, acidity, mechanical stress, and chromogens from ingested food. Several studies have previously shown that color stability is significantly affected by the artificial aging process due to the chemical modification of various resin components and changes in the surface microstructure.[6-9]

The aim of this study was to evaluate the color stability of commercial resin-based composites with claimed color adjustment potential after accelerated aging. The null hypothesis was that accelerated aging would not result in differences in color stability between the different materials tested.

METHODS AND MATERIALS

Specimen Preparation

Five commercially available resin-based composite systems were evaluated (see Table 1). These systems were selected to represent a range of products regarded as possessing color adjustment potential (chameleon effect) properties by their respective manufacturers.

Cylindrical composite samples (10 mm diameter, 2.5 mm thick; n=6) were fabricated using a custom-made silicone matrix. All specimens were fabricated in shade A2 (with the exception of Omnichroma). The materials were adapted into the silicone matrix, and a glass plate was placed on the uncured composite and finger pressed to the thickness of the mold. The samples were light cured through the glass plate for 40 seconds using an LED curing lamp (VALO Grand, Ultradent, South Jordan, UT, USA). A calibrated radiometer (MARC Resin Calibrator, BlueLight Analytics Inc) was used to monitor the light-curing unit output, confirming a constant irradiance value between 1000 and 1100 mW/cm^2. The glass plate provided a flat and polished composite surface and prevented the creation of an oxygen inhibition layer. Excess material was removed from the samples via wet sanding with silicone carbide papers on the unpolished side to ensure uniform thicknesses (2±0.025 mm), which was confirmed using a digital thickness scale (Mitutoyo, Japan). All specimens were subsequently coded to ensure color measurements were made at the same surface for accurate comparison of the specimen's initial and final color after accelerated aging. Specimens were stored in individual containers at 37°C protected from light until being subjected to the baseline color analysis.

Color Measurements

Initial Color Measurement—

Color measurements were performed using a spectrophotometer (CM2600d, Konica Minolta, Osaka, Japan) under standardized ambient conditions according to the Commission Internationale de l'Eclariage (CIE) L*a*b* system. Measuring conditions were set as follows: CIE L*a*b* color notation system, D65 standard light source; 2° standard observer, specular component, and 100% UV included and a small area view of 3.0 mm. The specimen reading was performed over a white background.

The spectrophotometer was calibrated as per manufacturer's instructions, and the color of each specimen was measured three times and automatically averaged by the accompanying software (SpectraMagic NX, Konica Minolta). The spectrophotometer was recalibrated after color values were collected for each group. The results of color measurements were quantified in terms of three coordinate values (L*, a*, b*), as established by CIE, in which the L* axis represents the degree of lightness and ranges from 0 (black) to 100 (white); the -a* plane represents the degree of green-red color (-a = green; +a = red), and the b* plane represents the degree of blue-yellow (-b = blue; +b = yellow) color within the sample.

Accelerated Aging—

Accelerated aging was performed in a weathering chamber (Q-Sun Xenon Test Chamber, Model Xe-3; Q-Panel Lab Products). The Xenon Test Chamber reproduces the entire spectrum of solar radiation in

Table 1. *Composite resins used in the present study*

Product/ Shade	Type	Matrix	Filler type/particle size	Filler Content	Manufacturer	Lot
Omnichroma	Supra-nano filled	UDMA, TEGDMA	Spherical SiO_2-ZrO_2 Particle size of 260 nm	79 wt%/ 68 vol%	Tokuyama Dental, Shibuya, Tokyo, Japan	007E29
Estelite Omega	Supra-nano filled	Bis-GMA, TEGDMA	Spherical SiO_2-ZrO_2 Particle size of 0.2 μm	82 wt%/ 71 vol%	Tokuyama Dental, Shibuya, Tokyo, Japan	124E39
Harmonize	Nano-hybrid	Bis-GMA, Bis-EMA, TEGDMA	Spherical SiO_2-ZrO_2 Barium glass (inorganic filler) Particle size of 5-400 nm	81 wt%/ 64.5 vol%	Kerr Co., Orange, CA, USA	7111912
Venus Pearl	Nano-hybrid	TCD-DI-HEA, UDMA	Ba-Al-F-glass, SiO_2 nanofiller Particle size of 5 nm-20 μm	82 wt%/ 64 vol%	Heraeus Kulzer GmbH, Hanau, Germany	K010056
GC Kalore	Nano-hybrid	UDMA, DMA, DX-511	Fluoroaluminosilicate glass Prepolymerized filler (strontium glass and lanthanoid fluoride) silicone dioxide Particle size of 16 nm to 17 μm	82 wt%/ 69 vol%	GC International, Tokyo, Japan	1804091

Abbreviations: Bis-GMA, bisphenol A diglycidylmethacrylate; Bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; DMA, dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; TCD-DI-HEA: Bis-(acryloyloxymethyl) tricyclodecane; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

the UV, visible, and infrared, from 295 to 800 nm using three xenon arc lamps and optical filters. The irradiance of the three lamps is reflected by the mirror walls inside the equipment, which generates the same energy in the entire irradiation surface. A weathering cycle consisted of 102 minutes of UV irradiation (with an average irradiance of 0.35 W/[m^2·nm] at 340 nm) at the temperature of 38°C (equivalent to a black panel temperature of 63°C) and 50% + 10% relative humidity, followed by 18-minute light and water spray (according to ASTM G155) for a total of 300 hours (or 150 cycles).

Final Color Measurement—

The differences in the values of L* (ΔL), a* (Δa), and b*(Δb) (where ΔL* is lightness, Δa* the differences in green-red, and Δb* the differences in the blue-yellow coordinates) were determined for each specimen, comparing the baseline values with the values obtained after the accelerated aging protocol. The overall difference in color change (ΔE*ab) was calculated by SpectraMagic Software using the following formula: $\Delta E^*ab=[(\Delta L)^2+ (\Delta a)^2+ (\Delta b)^2]^{1/2}$.

Clinically applicable thresholds of color stability were considered for comparisons. ΔE*ab values from 1 to 3 were considered perceptible to the naked eye and acceptable,[10] and ΔE*ab values greater than 3.3 were considered clinically unacceptable.[11]

Statistical Analysis

The ΔL*, Δa*, Δb*, and ΔE*ab values of each resin brand were compared using a one-way analysis of

variance (ANOVA). A Tukey post hoc test was used to further compare the ΔL*, Δa*, Δb*, and ΔE*ab values between individual brands. All calculations were performed using IBM SPSSStatistics version 26.0.0.0 for Macintosh with a significance level of 0.05.

RESULTS

The means and standard deviations (SD) for the ΔL*, Δa*, Δb*, and ΔE*ab values are recorded in Table 2.

According to the one-way ANOVA, there was a statistically significant difference in the ΔL* values between groups ($F(4,25)=27.484$, $p=0.000$). A Tukey post hoc test revealed significant differences in the ΔL* values of Estelite-Harmonize ($p=0.000$), Estelite-Venus (p=0.000), Harmonize-Kalore ($p=0.000$), Harmonize-Omnichroma ($p=0.000$), Harmonize-Venus ($p=0.030$), Kalore-Venus ($p=0.000$), and Omnichroma-Venus ($p=0.012$). Omnichroma showed the lowest average amount of change in ΔL* (-0.272±0.376), while Harmonize showed the greatest amount of change (2.347±0.539).

There was a significant difference in the Δa* values between groups according to the one-way ANOVA ($F(4,25)=520.506$, $p=0.000$). A Tukey post hoc test revealed significant differences between the Δa* values of Estelite-Harmonize ($p=0.000$), Estelite-Kalore ($p=0.000$), Estelite-Omnichroma ($p=0.000$), Estelite-Venus ($p=0.000$), Harmonize-Kalore ($p=0.000$), Harmonize-Omnichroma ($p=0.000$), Harmonize-Venus ($p=0.000$), and Omnichroma-Venus ($p=0.036$). Omnichroma showed the lowest average change in Δa* (-0.032±0.136), while Harmonize showed the greatest amount of change (-3.770±0.169).

There was a significant difference in the Δb* values between groups according to the one-way ANOVA ($F(4,25)=6.073$, $p=0.001$). A Tukey post hoc test revealed significant differences in between the Δb* values of Estelite-Harmonize ($p=0.028$), Harmonize-Kalore ($p=0.004$), Kalore-Omnichroma ($p=0.026$), and Kalore-Venus ($p=0.027$). Harmonize showed the least amount of change in Δb* (-0.200±2.055), while Kalore showed the greatest change (3.372±1.886).

There was a significant difference in ΔE*ab between groups according to the one-way ANOVA ($F(4,25)=11.422$, $p=0.000$). A Tukey post hoc test revealed significant differences between Estelite-Harmonize ($p=0.038$), Harmonize-Omnichroma ($p=0.000$), Harmonize-Venus ($p=0.000$), Kalore-Omnichroma ($p=0.006$), and Kalore-Venus ($p=0.024$). Omnichroma showed the least amount of change in ΔE*ab (1.302±0.607), while Harmonize showed the greatest amount of change (4.848±0.224).

DISCUSSION

The long-term color stability of commercial resin-based composite materials used in restorative esthetic procedures is still a concern as discoloration and inappropriate color match are considered one of the major reasons to replace resin-based composite restorations.[1] This laboratory study compared the effects of accelerated aging on the color stability of five composite resin materials. The methodology used in the present study was in accordance with previous studies that used spectrophotometry and the CIE L*a*b* coordinate system, which is a recommended method for dental purposes. The CIE L*a*b* coordinate system is well suited for the determination of small color changes and has advantages such as repeatability, sensitivity, and objectivity.[12] For this study, ΔE values ranging from 1 to 3 were considered perceptible to the naked eye,[10] and ΔE values greater than 3.3 were considered clinically unacceptable.[11]

Several factors contribute to composite discoloration including intrinsic discoloration and extrinsic staining of the material. The chemical stability of the resinous matrix and the interface between the matrix and particles are some of the intrinsic factors,[13] while the absorption of staining solutions due to the patient's diet, hygiene, or smoking habits is considered an extrinsic factor.[14] Accelerated aging protocols expose

Table 2. *The mean values (±SD) of ΔL*, Δa*, Δb*, and ΔE*ab for each of the composite resin brands tested*[a]

Resin Composite Brand	ΔL* (±SD)	Δa* (±SD)	Δb* (±SD)	ΔE*ab (±SD)
Estelite Omega	-0.942 (±0.489) a	0.965 (±0.096) a	2.632 (±1.046) a,b	2.984 (±1.072) a,c
Harmonize	2.347 (±0.539) b	-3.770 (±0.169) b	-0.200 (±2.055) c	4.848 (±0.224) b
GC Kalore	-0.987 (±1.039) a	-0.207(±0.322) c	3.372 (±1.886) a	3.641 (±1.921) b,c
Omnichroma	-0.272 (±0.376) a	-0.032 (±0.136) c	0.518 (±1.353) b,c	1.302 (±0.607) a
Venus Pearl	1.112 (±0.728) c	-0.377 (±0.164) c	0.535 (±1.005) b,c	1.647 (±0.554) a

[a] *Values designated by the letters represent statistically distinct groups.*

the materials to temperature challenges, humidity, and light irradiation but do not represent the complete behavior of the materials in the oral environment as it addresses only the intrinsic discolorations factors.[15]

Although the clinical relevance of this method is unknown, and none of the artificial environments can precisely simulate the actual oral conditions,[16] several studies have evaluated the optical behavior of composites with different protocols and periods of accelerated aging as a means to predict, within a short time, the effects of long-term exposure and possible alterations of the color properties of resin-based composite materials in a clinical environment.[13,17-20]

It was previously suggested that 300 hours of accelerated aging simulates approximately 1 year of clinical service.[20-23] The conversion between hours of accelerated aging and clinical service must be carefully interpreted as an empirical comparative dataset however, as one is an intense, constant condition, while the other can be subjected to numerous variables.[24] Moreover, there is no standardization regarding the aging time necessary for promoting color alteration, and it is not clear at which moment of the aging process the discoloration would be considered clinically unacceptable.[20,25] Previous studies stated that the color change produced by accelerated aging was induced in the first 100 to 300 hours[16] and that no differences in color changes were observed with accelerated aging times ranging from 300 to 900 hours.[26]

The color stability of resin-based composite materials is determined by several factors including the degree of conversion and chemical characteristics. Higher monomer conversion indicates low amount of unreacted monomer, lower solubility, and higher color stability.[27] Unconverted double carbon bonds involve residual monomers being trapped in the composite, rendering the material more susceptible to staining,[28] while hydrophilic organic matrices (that favor water absorption) may lead to degradation of the polymeric network and subsequent release of by-products that cause discoloration (i.e., formaldehyde and methacrylic acid).[29-30] Increased water sorption results in poor color stability due to the increase in free volume of the formed polymer and greater space for the water molecules to diffuse into the polymeric network, thus contributing first to its degradation and second to discoloration.[28]

The specimens in this study were subjected to the main environmental factors involved in the hydrolysis, degradation, and further discoloration of composite restorations (UV radiation, temperature changes, and water/humidity)[31] for 300 hours. After accelerated aging, Omnichroma and Venus Pearl presented excellent color stability and the smallest changes in color (ΔE^*ab of 1.302 [±0.607] and 1.647 [±0.554], respectively), well below the ΔE^*ab = 3.3 clinically acceptable threshold.

Omnichroma consists of a mix of a uniformly sized suprananospherical filler of silicon dioxide (SiO_2) and zirconium dioxide (ZrO_2) with a particle size of 260 nm plus a round-shaped composite filler.[32] Since the color stability of resin-based composite materials is determined by not only the organic matrix and filler composition but also relatively minor pigment additions and other chemical components,[33] it is important to note that Omnichroma does not contain pigments in its formulation (according to the manufacturer), and this fact might help explain its behavior in terms of color stability.

Also, due to the structural color property that is produced by the diffraction and scattering caused by the microscopic structures of the materials, it is possible that the small color changes presented by Omnichroma might be even less perceivable clinically, as the material was able to maintain its color match after the adjacent tooth structure was bleached, demonstrating true color adjustment potential.[3] Omnichroma is also based on urethane dimethacrylate (UDMA) chemistry, a hydrophobic monomer that has the ability to increase the hydric stability of the restorative material,[34] rendering it less susceptible to degradation and further color alteration.[35,36]

Venus Pearl is composed of both UDMA and Bis-(acryloyloxymethyl) tricyclodecane (TCD-DI-HEA) monomers, with about 64% by volume of barium aluminum fluoride glass fillers having a size range of 5-20 μm. Previous studies demonstrated that the UDMA + TCD-DI-HEA combination yield significantly higher degrees of conversion and lower concentration of double bonds,[37-40] thus leading to better color stability than bisphenol A diglycidylmethacrylate (Bis-GMA) due to its lower viscosity and water sorption.[37,41-43] Very good mechanical stability was also observed after aging,[44-45] demonstrating good chemical stability, probably as a result of the big molecular size of the TCD-urethane and the absence of diluting agents.[43]

Estelite Omega occupies an intermediate position among the tested materials. No statistically significant difference between Estelite Omega, Venus Pearl, and Omnichroma was observed, despite the higher ΔE^*ab = 2.984 (±1.072). The greater color change of Estelite Omega might be explained by the presence of the more hydrophilic monomer Bis-GMA.[46,47] Additionally, no statistical difference was observed between Estelite Omega and GC Kalore.

GC Kalore and Harmonize presented significant ΔE^*ab values that were beyond the acceptability

threshold level (ΔE*ab of 3.641 [±1.921] and 4.848 [±0.224], respectively). The organic matrix of GC KALORE contains a high resin monomer content mixture of UDMA, DMA, and the DX-511 monomer. When the degree of conversion of GC Kalore was investigated, it exhibited the lowest degree of conversion values.[48] GC Kalore was also found to have an ununiform dispersion of fillers consisting of prepolymerized fillers and different size fillers that might lead to voids or nonbonding spaces at the filler/matrix interface that increase water sorption.[49,50] It can be assumed that such molecular behavior led to its poor color stability performance.

The resin matrix of Harmonize is composed of Bis-GMA, triethylene glycol dimethacrylate (TEGDMA), and bisphenol A polyethylene glycol diether dimethacrylate (Bis-EMA). TEGDMA is the most hydrophilic monomer and is included to adjust Bis-GMA's viscosity. TEGDMA and Bis-GMA are more hydrophilic than UDMA[51,52] and resulted in higher water sorption and increased solubility of the polymer than UDMA.[53,54]

TEGDMA is also present in Omnichroma (1%-5%) and Estelite Omega (5%-10%). Small differences in the percentage of TEGDMA present in their basic chemical composition could explain their different discoloration values.[35] It has been reported that an increase in the proportion of TEGDMA from 0% to 1% increased the water uptake in Bis-GMA–based composite resins from 3% to 6%.[55]

Accelerated aging resulted in a lightness (L*) shift toward darker values for all materials except for Harmonize and Venus Pearl. This effect could be related to a delayed dark curing mechanism that would further consume initiators or to residual molecules that were excited under the intense light of the accelerated aging procedure, resulting in this lightening effect.[56]

Estelite Omega was the only composite that became redder (increase in a* coordinate values), whereas all others slightly shifted toward green (decrease in a* coordinate values) with the exception of Harmonize, which presented a more significant Δa* value decrease (Δa*=-3.770 [±0.169]). Harmonize was also the only material that showed a slight shift toward blue (decrease in b* coordinate values). All other materials became yellower (increase in b* coordinate values), some of them significantly (Estelite Omega Δb*=2.632 [±1.046] and GC Kalore Δb*=3.372 [±1.886], respectively). The presence of residual camphorquinone (a yellow-color compound in which degradation results in color alteration) and residual tertiary amines (accelerators) that also cause discoloration of composite materials under the influence of light and heat, as well as unreacted C=C oxidation, explains this trend toward yellowing.[14,18,57-59]

The effects of accelerated aging as observed in the present study are related to the behavior of the tested materials under the specific aging protocol used. It is difficult to directly compare the results of this study with data from the literature, as there are limited publications available that studied the same composite systems. The results were material dependent but overall in accordance with previous studies that demonstrated that accelerated aging generally resulted in a decrease of L* and a* values and an increase of b* values.[60-63]

A limitation of this study is that other color-altering agents and conditions that also affect the long-term color stability of composite materials, such as chemicals or staining agents (food or drinks), artificial saliva, changes in pH levels, and enzymes, could have been used as part of the accelerated aging process and would have contributed for a better simulation of clinical conditions.

CONCLUSION

According to the results obtained from the experimental groups, the null hypothesis that accelerated aging would not result in color stability differences of the tested composite resins was rejected.

Within the limitations of this laboratory study, the following conclusions were drawn:

1. Accelerated aging effects on the color stability of the tested composites were material dependent.
2. Omnichroma and Venus Pearl exhibited significantly lower overall color change ($p<0.001$).
3. GC Kalore and Harmonize displayed significantly greater ($p<0.001$) overall color change that would be considered clinically unacceptable (ΔE*ab>3.3).

Conflicts of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or companythat is presented in this article.

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