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THE COLOR STABILITY OF AESTHETIC RESTORATIVE MATERIALS RESULTING FROM ACCELERATED AGING

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INTRODUCTION

In anterior tooth restoration composite, resin and porcelain are useful material. Composite resin is used for direct restoration, and prosthetic composition for indirect restoration. Commonly used all-ceramic restoration is aesthetic and less plaque accumulative. In addition, its color stability and wear resistance are high. However, stress accumulation over the time and difficulty in repair after setting in the mouth are disadvantageous. Composite resin is highly workable, can be repaired directly in the mouth. It is aesthetic, and the antagonist tooth wears out less. But its disadvantages are decreased color stability over time and a possibility of marginal leakage.

Color stability of restorative materials is quite different between direct restorative composite resin and 2nd-generation prosthetic composites. It also depends on experimental conditions such as accelerated aging and thermocycling. Composite resin showed a higher degree of discoloration than porcelain[1-3]. In an experiment on the color stability of composite resin, SYNERGY Duo produced relatively satisfactory results.[3] Compared to other dental porcelain, IPS Empress 2 is easy to observe fine bubbles in a process of polishing which is sometimes necessary before setting the restoration, and they might affect color stability.

In the measurement of color change using the CIELAB color system, if ΔE is higher than 1, the discoloration is perceptible, and it is clinically acceptable up to 3.3.[4]

We measured color change and microhardness of five aesthetic restorative materials (IPS Empress 2-glazed, IPS Empress 2-polished, Sinfony, TESCERA ATL and SYNERGY Duo) before and after accelerated aging. Surface changes of the materials were observed using a scanning electron microscope (SEM). The objective of this study was to evaluate intrinsic color stability and affecting factors on the esthetic restorative materials when subjected to accelerated aging.

EXPERIMENT MATERIALS AND METHODS

1. Experiment materials

Five materials (IPS Empress 2-glazed, IPS Empress 2-polished, Sinfony, SYNERGY Duo and TESCERA ATL) were used (Table I). All specimens were prepared as a disk of 12 mm in diameter and 2 mm in thickness, according to the manufacturer's instruction. After specimens were fixed, the surfaces were ground with the grinding wheel (diamond wheel, SD800N100R6, Woosin, Republic of Korea) of a horizontal-type surface grinding machine (Alpha Precision, Republic of Korea), and then both sides of disks were polished respectively with 6 ㎛ and then 1 ㎛ diamond abrasives (Norpon, USA). After polished, specimens of IPS Empress 2-glazed were glazed. Thirty specimens of each material were prepared for accelerated aging. For control, additional 5 specimens of each material were prepared. All the specimens were dipped in distilled water for 24 hours before the experiment.

2. Experiment methods

1) Accelerated aging

Specimens were subjected to an accelerated aging machine (Suntest CPS, Atlas electronic devices, USA).

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Using light filtering equipment of the machine, Xenon light having a wavelength longer than 310 nm was irradiated. Black standard temperature was set to be 70° C and the temperature of distilled water was 38° C. The test cycles were 40 minutes light only, 20 minutes light plus water immersion, 60 minutes light only, and 60 minutes dark plus water immersion. The test was run for a radiant energy of 397.98 KJ/mm and a total exposure time of 300 hours. Color change and microhardness of specimens were measured at intervals of 100 hours.

2) Specimen measurements

① Color

The color was measured according to the CIEL*a*b* color scale relative to D65 on a reflected spectrophotometer (CM-3500d, Minolta, Japan) with the spectral component excluded (SCE) geometry. Measurement diameter was 8 mm. Diffuse illumination/8° viewing angle was used. Each specimen was measured 3 times at different parts. And software of Spectra Magic Version 1.01 (Minolta, 1997) was used.

Changes in overall ΔE were calculated as follows;

$$\Delta E = \{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}^{1/2}$$

Color change of the specimens were measured before accelerated aging and after 100 hours, 200 hours and 300 hours of accelerated aging.

② Microhardness

Microhardness was measured using a microhardness tester (HMV-2 Series, Shimadzu Co. Japan). The measurements were performed for 5 seconds with a Vickers indenter of 2.942 N (HV0.3), and each specimen was measured twice at different parts of the surface. Microhardness change of the specimens were measured

Table I. Materials tested

Product	IPS Empress 2-glazed	IPS Empress 2-polished	Sinfony	SYNERGY Duo	TESCERA
Manufacturer	Ivoclar Vivadent AG, Liechtenstein	Ivoclar Vivadent AG, Liechtenstein	3M ESPE, Germany	Coltène/Whaledent AG, Switzerland	Bisco, USA
Batch number	G05928 F59999 G07204	G05928 F59999	181932	NC352	20009755
Photoinitiator		N/A	N/A	Camphoroquinone (1%)	Camphoroquinone Tertiary amine
Heat Initiator					TBPZ
Shade	IPS Empress 2 Incisal chromascop S1 IPS Empress 2 build up liquid Universal glaze and stain liquid	IPS Empress 2 incisal chromascop S1 IPS Empress 2 build up liquid	E2	A2/B2	Body A2
Monomer matrix			Mixture of Aliphatic monomers & Cycloaliphatic monomers	TEGDMA BisGMA BisEMA	TEGDMA BisGMA
Inorganic fillers	N/A	N/A	Strontium aluminium borosilicate glass, silanised Pyrogenic silica, silanised Special glassionomer	Barium glass, silanised Strontium glass, silanised Amorphous silica, hydrophobed	Amorphous silica Glass frit
Filler size in ㎛ (average)	N/A	N/A	0.05,0.5 - 0.7	0.04 - 2.5 (0.6)	0.05 - 17
Filler weight in %	N/A	N/A	50	74	72
Filler volume in %	N/A	N/A		59	53
Polymerization type or firing	Heat (825° C) Heat (785° C)		Light Vacuum	Light	Light (130 W) Pressure (80 PSI) Heat (132° C)
Type			Ultra-fine particle hybrid composite	Fine hybrid composite	Reinforced microfilled hybrid composite

TEGDMA: Triethylene glycol dimethacrylate
BisGMA: Bisphenol -A-diglycidylether methacrylate
TBPZ: Tertiary butyl peroxybenzoate

before accelerated aging and after 100 hours, 200 hours and 300 hours of accelerated aging.

③ SEM photography

We used the ion sputter (Polalon Division, Bio Rad. USA) 120 seconds to coat the mounted specimens in gold before they go into the SEM. The 5 kinds of specimens were photographed using a SEM at a magnification of 100, 1000 and 8000 before and after 300 hours of accelerated aging.

3. Statistical analysis

Color changes (ΔE) of products during accelerated aging were compared by Scheffe s method. Microhardness changes and microhardness percent changes of them during accelerated aging were also compared. Correlation between color changes and microhardness was analyzed (Pearson correlation coefficient) and effect of materials and times after accelerated aging on color change were evaluated with ANOVA.

EXPERIMENT RESULTS

1. Color change

During 300 hours' accelerated aging, color change was high in order of IPS Empress 2-glazed < IPS Empress 2-polished = Sinfony < TESCERA ATL < SYNERGY Duo ($P < .05$). ΔE values were 3.3 or lower in IPS Empress 2-glazed, IPS Empress 2-polished and Sinfony. TESCERA ATL and SYNERGY Duo were in the range of ΔE 3.6-6.4 and showed very obvious color changes (Table II).

2. Microhardness

During accelerated aging, TESCERA ATL showed an increase in hardness during the first 100 hours, no change during the next 100 hours, and a decrease during the last 100 hours ($P < .05$, Table III). During 300 hours' accelerated aging, the microhardness of surface increased by 3.21 - 19.64% in all kinds of composite resin specimens. The rate of increase was high in order of TESCERA ATL = IPS Empress 2-glazed = SYNERGY Duo $\leq$ Sinfony = IPS Empress 2-polished ($P < .05$, Table IV).

Significant correlation was observed between the color change and the baseline microhardness change after 200 and 300 hours of accelerated aging of Sinfony and TESCERA ATL, and between the color change and the microhardness percent change after 200 and 300 hours of accelerated aging of Sinfony and SYNERGY Duo ($P < .05$, Table V).

During accelerated aging, variables such as the kind of specimen and treatment time had a significant effect on color change, and the effect was high in order of time, products and time-products (Table VI).

3. Visual examination and SEM examination

The surface of IPS Empress 2-glazed was uneven but showed no bubble before accelerated aging, There was a large number of bubbles in IPS Empress 2-polished, and few bubbles in Sinfony. It was confirmed that SYNERGY Duo formed a few bubbles and TESCERA ATL had 1 - 2 bubbles, and gloss after polishing was similar between the two. In IPS Empress 2-glazed, IPS Empress 2-polished and Sinfony, no change in gloss after accelerated aging was observed, but in TESCERA ATL gloss was lost, and SYNERGY Duo showed the largest loss of gloss.

On SEM examination in IPS Empress 2-glazed, particles may be fallen off from other composites during accelerated aging, were caught in the recesses of surface which were made by glazing over bubbles exposed during polishing (Fig. 1). IPS Empress 2-polished did not show any surface change of surface after accelerated aging (Fig. 2). In the control specimens of Sinfony, small defects were observed on the interface between the fillers and the substrate, and no change was observed after accelerated aging (Fig. 3). The control specimens of SYNERGY Duo had a large number of defects, smaller than fillers (Fig. 4). After accelerated aging, fillers exposed coarsely over the entire surface, and around 0.5 ㎛ wide and tens of ㎛ long microcracks happened in large numbers (Fig. 5). The control specimens of TESCERA ATL had few bubbles, and had around 17 ㎛ large or smaller glass frits of various irregular forms. After accelerated aging, filler particles slightly embossed from the composite matrix, and there were many tens of ㎛ long fine cracks on the interface between the substrate and fine

Table II. Color changes (Δ E $\pm$ SD) of products during the accelerated aging process

Products	Δ E $\pm$ SD			
	0 hrs	100 hrs	200 hrs	300 hrs
IPS Emp. 2-gla.	0 ^{A,a}	1.0 ^{A,b} $\pm$ 1.0	1.26 ^{A,b} $\pm$ 1.1	1.1 ^{A,b} $\pm$ 1.2
IPS Emp. 2-pol.	0 ^{A,a}	1.6 ^{A,b} $\pm$ 2.5	2.52 ^{B,b} $\pm$ 3.0	2.5 ^{B,b} $\pm$ 2.9
Sinfony	0 ^{A,a}	0.9 ^{B,b} $\pm$ 0.3	2.05 ^{A,B,c} $\pm$ 0.7	3.3 ^{B,d} $\pm$ 0.7
SYNERGY Duo	0 ^{A,a}	4.4 ^{B,b} $\pm$ 0.6	5.70 ^{D,c} $\pm$ 0.5	6.4 ^{C,d} $\pm$ 0.9
TESCERA	0 ^{A,a}	4.1 ^{B,b} $\pm$ 0.3	4.08 ^{C,b} $\pm$ 0.5	3.6 ^{B,c} $\pm$ 0.6

For comparisons between durations of aging process (rows), means with same lower case letter are not statistically different at $P = .05$ using the Scheffé F-test.
For comparisons between products (columns), means with same capital letter are not statistically different at $P = .05$ using the Scheffé F-test.

Table III. Microhardness of products under the accelerated aging process, mean $\pm$ SD

Products	Microhardness			
	0 hrs	100 hrs	200 hrs	300 hrs
IPS Emp. 2-gla.	491.6 $\pm$ 59.8 ^{D,a}	476.5 $\pm$ 40.0 ^{C,a}	495.0 $\pm$ 23.6 ^{C,a}	605.1 $\pm$ 30. 2 ^{D,b}
IPS Emp. 2-pol.	409.5 $\pm$ 22.3 ^{C,a}	470.9 $\pm$ 40.0 ^{C,b}	492.2 $\pm$ 23.6 ^{C,b}	565.7 $\pm$ 36.9 ^{C,c}
Sinfony	31.0 $\pm$ 5.3 ^{A,a}	35.7 $\pm$ 3.6 ^{A,b}	34.9 $\pm$ 3.5 ^{A,b}	34.5 $\pm$ 3.4 ^{A,b}
SYNERGY Duo	59.8 $\pm$ 8.0 ^{B,a}	63.4 $\pm$ 6.6 ^{B,a,b}	65.0 $\pm$ 4.7 ^{B,b}	65.3 $\pm$ 4.8 ^{B,b}
TESCERA	67.7 $\pm$ 3.8 ^{B,a}	73.4 $\pm$ 3.3 ^{B,b}	75.1 $\pm$ 3.2 ^{B,b}	69.7 $\pm$ 2.7 ^{B,a}

For comparisons between products (columns), means with same capital letter are not statistically different at $P = .05$ using the Scheffe' s test.
For comparisons between durations of aging process (rows), means with same lower case letter are not statistically different at $P = .05$ using the Scheffe' s test.

Table IV. Microhardness percent changes of products under accelerated aging Process, mean

Products	Accelerated aging time		
	100 hrs	200 hrs	300 hrs
IPS Emp. 2-gla.	-1.5 ^{B,a}	2.1 ^{B,a}	24.8 ^{A,B,b}
IPS Emp. 2-pol.	15.3 ^{A,b}	20.5 ^{A,b}	38.5 ^{A,c}
Sinfony	19.6 ^{A,a}	17.0 ^{A,a}	14.9 ^{B,C,a,}
SYNERGY Duo	7.4 ^{A,B,a}	10.4 ^{A,B,a}	11.2 ^{B,C,a}
TESCERA	8.6 ^{A,B,b}	11.2 ^{A,B,b}	3.2 ^{C,a}

For comparisons between products (columns), means with same capital letter are not statistically different at $P = .05$ using the Scheffé F-test.
For comparisons between durations of aging process (rows), means with same lower case letter are not statistically different at $P = .05$ using the Scheffé F-test.

Table V. Correlation coefficient (r) of color changes and the baseline microhardness changes (microhardness percent change)

Products	Accelerated aging time		
	100 hrs	200 hrs	300 hrs
IPS Emp. 2-gla.	-0.09 (-0.11)	0.18 (-0.02)	-0.03 (-0.08)
IPS Emp. 2-pol.	-0.29 (-0.08)	-0.04 (0.08)	-0.23 (-0.09)
Sinfony	0.08 (0.10)	-0.47† (-0.47‡)	-0.18 (-0.39†)
SYNERGY Duo	0.28 (-0.08)	-0.13 (-0.37†)	-0.31 (-0.50‡)
TESCERA	-0.22 (-0.32)	0.27 (0.09)	0.42† (0.08)

† $P < .05$, ‡ $P < .01$

Table VI. ANOVA table for two way of color changes (Δ E)

Variables	Mean square	F-value	*P* - value
Aging Time	354.81	232.27	.000
Products	201.97	132.22	.000
Aging Time* Products	29.95	19.61	.000
Residual	2.40		

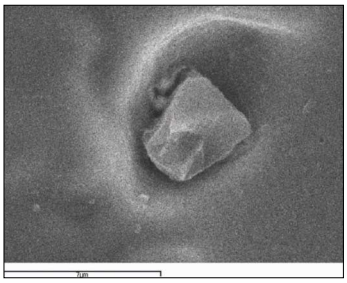

Fig. 1. IPS Empress 2-glazed. after accelerated aging (× 8000).

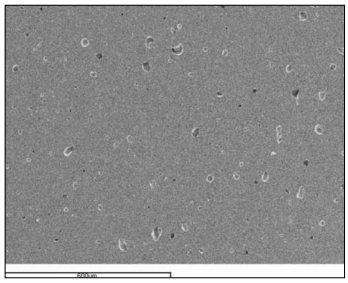

Fig. 2. IPS Empress 2-polished. after accelerated aging (× 100).

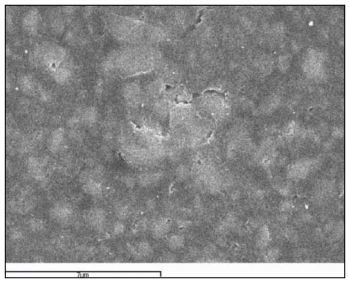

Fig. 3. Sinfony control specimen (× 8000).

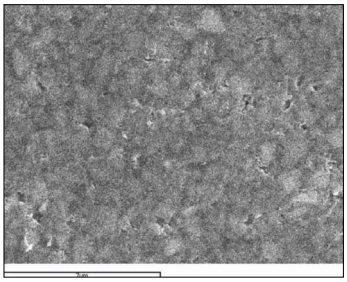

Fig. 4. SYNERGY Duo control speccimen(× 8000).

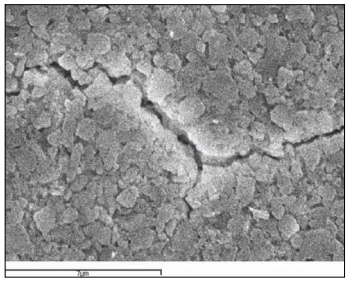

Fig. 5. SYNERGY Duo, after accelerated aging (× 8000).

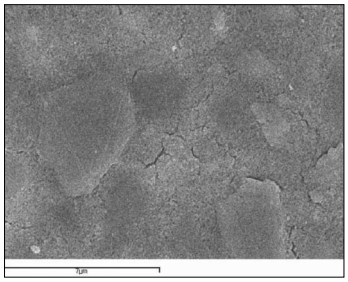

Fig. 6. TESCERA ATL, after accelerated aging (× 8000).

particles and between coarse particles and the substrate (Fig. 6).

DISCUSSION

Esquivel *et al.*[5] reported that when Procera, Duceratin and Vita VMK 68 were precipitated in distilled water, Δ E values was below 0.88. Lee *et al.*[6] reported that when IPS Empress 2 was thermocycled, Δ E values was below 2.29. Kim *et al.*[3] reported that after 400 hours' accelerated aging of Ceramco, Δ E values was 1.43 and prosthetic composites were more stable in color than composites resin. In this study, during 300 hours' accelerated aging, the maximum Δ E values were 1.26 in IPS Empress 2-glazed, and 2.52 in IPS Empress 2-polished, showing a significant difference. Lee *et al.*[6] interpreted that during thermocycling of IPS Empress 2, color changes is caused by the adsorption of stained substance from other composites to micropores formed during the firing procedure. In the SEM photograph of Empress 2-glazed in our study, there were particles caught in recesses on the surface, which are believed to have fallen off from TESCERA ATL during accelerated aging.

The discoloration of composite resin is induced by internal causes and external causes such as the absorption and adsorption of coloring agent.[7] Color change resulting from accelerated aging is related to the internal color stability. Camphoroquinone, a photopolymerization initiator in TESCERA ATL and Sinfony, is a factor degrading the stability of color.[8] Light attacks substance added as a colorant to composite resin, decomposes amine, and produces a yellowish brown oxide.[9] Dietschi *et al.*[10] reported that resistance to discoloration of composites was related to low water sorption, high filler-resin ratio, reduced particle size and hardness, and optimal filler-matrix coupling system. The degree of polymerization is different among

photopolymerized composite resin products. Reported degree of conversion of posterior composites was 36 - 74%.[11,12] It was reported that if the kinetic of dimethacrylate monomer is limited, polymerization cannot increase any more, but if temperature rises, the kinetic of dimethacrylate monomer increases and the flexibility of polymer chain in the network is enhanced, and as a result, the degree of polymerization increases by post cure.[13] Power *et al*.[14,15] reported that after accelerated aging under xenon light and 90 percent relative humidity, the matrices of resin eroded away and the filler was exposed, and as a result, the resin showed chalky surface and the roughness of the surface increased.[16] The absorption of water by composite resin is affected by the hydrophobic of the resin matrix. The effect of Oxygen in room air was responsible for the formation of a polymerization inhibited zone on the surface of the resin.[17] Bubbles contained in specimens can make oxygen inhibition zones composed of unhardened substances and absorb a large amount of water. Absorbed water causes separation between the fillers and the resin matrix or even hydrolytic degradation of the fillers.[18] SYNERGY Duo had a relatively large number of bubbles. And our results of the visual examination and SEM observation of changes caused by accelerated aging were consistent with the reports of Power *et al*. above.[14,15] Occurrence of numerous microcracks and defects on the surface of SYNERGY Duo and TESCERA ATL after accelerated aging is probably because of the erosion of the resin matrix, hydrolysis of the fillers and polymerization shrinkage related to additional polymerization involving xenon light, heat, etc. Microcracks were much wider and longer in SYNERGY Duo than in TESCERA ATL, and the surface of SYNERGY Duo had many changes including the exposure of fillers over the substrate. Kim *et al*.[11] reported that high degree of conversion can lead to clinically unfavorable condition such as polymerization contraction.

Compared to resin having Bis-GMA and TEGDMA, resin having non-aromatic monomers such as UEDMA and HEMA enhances the degree of polymerization because it maintains flexibility at the last stage of polymerization when residual double bond makes cross linkage.[19] And Stansbury[20] reported that dimethacrylate monomers with a high tendency of cyclopolymerization can be used to increase the degree of conversion without polymerization contraction. Contrary to the SYNERGY Duo and TESCERA ATL, Sinfony did not show any surface change after post cure which showed increased microhardness during accelerated aging. It is probably because there was little contraction caused by polymerization and the monomer matrix of Sinfony is composed of aliphatic monomers and cycloaliphatic monomers whereas matrix of SYNERGY Duo and TESCERA ATL was composed of Bis-GMA and TEGDMA.

As to changes resulting from accelerated aging, Powers *et al*.[15,16] reported that color change in conventional composite resin was affected more by the surface deterioration which has been shown to increase lightness and decrease chroma of restorative resins, and in fine-particle-type composite resin showing a small color change, the color change was caused by the continuous formation of colored degradation product rather than by the erosion of surface. And Douglass[1] reported that, different from that in hybrid composite resin, color change in ceramic-polymer was mostly a chromatic change with little change in value. Sinfony is composed of ultra-fine particle hybrid composites, and showed little change in the form of surface and no change in microhardness after 100 hours but continuous change in color. This suggests that the change of color was caused by the continuous formation of colored degradation product.

In previous research, color changes happened mostly within 300 hours of accelerated aging.[2,4] In accelerated aging, Ruyter *et al*.[4] reported that color changes were observed mostly during the first 300 hours, and reached the plateau after 1440 hours. Kim *et al*.[2] reported that the largest difference was observed in 100 hours, and no significant difference in 400 hours. In our study as well, color changes were largest during the first 100 hours in IPS Empress 2-glazed, IPS Empress 2-polished, SYNERGY Duo and TESCERA ATL. And it reached the plateau after 100 hours in IPS Empress 2-glazed and TESCERA ATL, and after 200 hours in IPS Empress 2-polished. In Sinfony and SYNERGY Duo, discoloration continued for the 300 hours of accelerated aging, without reaching plateau.

Wendt reported that when secondary polymerization by heat was applied after the photopolymerization of photopolymerized composite resin, it improved physical properties of wear, hardness and color stability.[21] Ferracane *et al*.[22] reported that post-cure heat treatment increases in

microhardness ranging from 7 - 47% and averaging about 25% for all composites and conditions. In research with 10 specimens of composite resin treated through accelerated aging for 122 hours and with a total of 150 KJ/m^2, percent hardness increased by 4.8 - 32.1%.[23] In our study as well, During 300 hours' accelerated aging increased the microhardness of surface by 3.21 - 19.64% in all kinds of composite resin specimens. This is considered the effect of post cure resulting from accelerated aging.

Schulze *et al*.[23] reported that there is no correlation between hardness and color change and between hardness during accelerated aging, but in our experiment, correlation was observed between the color change and the baseline microhardness change after 200 and 300 hours of accelerated aging in Sinfony and TESCERA ATL, and between the color change and the microhardness percent change after 200 and 300 hours of accelerated aging in Sinfony and SYNERGY Duo. Further study might be needed to explain the effect of microhardness on color change of these materials.

CONCLUSIONS

We applied accelerated aging to 5 kinds of aesthetic restorative materials, measured their color and microhardness and performed SEM examination. From these procedures results were obtained as follows.

1. After 300 hours' accelerated aging, IPS Empress 2-glazed, IPS Empress 2-polished and Sinfony showed no morphological changes under SEM images and their color changes were considered to be clinically acceptable.
2. After 300 hours' accelerated aging, their surface gloss were lost and surface changes including microcracks were observed in TESCERA ATL and SYNERGY Duo, and their color changes ranged between 3.58 and 6.40 ΔE units.
3. During 300 hours' accelerated aging, the microhardness of surface increased by 3.21 - 19.64% in all kinds of composites resin. There was significant correlation between microhardness changes and color changes of composites ($P < .05$).

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THE COLOR STABILITY OF AESTHETIC RESTORATIVE MATERIALS RESULTING FROM ACCELERATED AGING

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STATEMENT OF PROBLEM: The discoloration of anterior teeth restoration is one of the material problems demanding retreatment. **OBJECTIVES:** To evaluate the color stability and affecting factors on esthetic restorative materials when subjected to accelerated aging. **MATERIAL AND METHODS:** This study was conducted using porcelain disks (IPS Empress 2-glazed, IPS Empress 2-polished), direct restorative resin disks (SYNERGY Duo) and indirect restorative resin disks (Sinfony, TESCERA ATL). Accelerated aging was done by precipitating the specimens in 38°C distilled water and irradiating with xenon light, and the total irradiation was 397.98 KJ/mm. Color and microhardness change of the specimens were measured before accelerated aging and after 100 hours, 200 hours and 300 hours of accelerated aging, and Surface of the specimens were examined with SEM before and after 300 hours of accelerated aging. **RESULTS:** 1. After 300 hours' accelerated aging, a ΔE value was 3.3 or lower in IPS Empress 2-glazed, IPS Empress 2-polished and Sinfony. 2. After 300 hours' accelerated aging, gloss was lost and surface changes including microcracks were observed in TESCERA ATL and SYNERGY Duo, and color changes of them ranged between 3.58 and 6.40 ΔE units. 3. During 300 hours' accelerated aging, the microhardness of surface was increased by 3.21 - 19.64% in all kinds of composites resin. **CONCLUSION:** After 300 hours' accelerated aging, SEM images IPS Empress 2-glazed, IPS Empress 2-polished and Sinfony showed little morphological change and their color changes were considered to be clinically acceptable. And there was significant correlation between microhardness changes and color changes of composites ($P < .05$).

KEY WORDS: Color stability, Accelerated aging, SEM, Composite resin, Porcelain

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