COLOR STABILITY OF CURRENT PROSTHETIC COMPOSITES UNDER ACCELERATED AGING AND IMMERSION IN A COFFEE SOLUTION

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The color stability of current prosthetic composites is unknown, even though the quality of composite materials has been improving. This study examined the intrinsic color stability of various current prosthetic resins (ceramic-polymers) after an accelerated aging process and the extrinsic color stability after immersion in a coffee solution. By comparing the amount of discoloration after aging with that without aging, the effect of the accelerated aging process on external discoloration could be evaluated.

Three current prosthetic composites (Artglass, Targis, Sculpture), one light polymerized direct composite (Z100) and one dental porcelain control (Ceramco) were assessed. The color changes (ΔE) of all the specimens were determined using the CIE L*a*b* color order system with a reflected spectrophotometer.

The results were as follows:

1. The prosthetic composite materials subjected to the accelerated aging test showed no significant difference in color changes ($p > .05$).
2. In the coffee solution immersion test after the aging process, the color changes of the Targis and Artglass groups were not different from that of the Z100 group, which showed the highest color change.
3. In the immersion only test, a significantly high color change was observed in the sculpture glazing group.
4. The aging process influenced on the color changes more in the Targis, Artglass and Z100 groups than in the Sculpture and Ceramco groups.

Key Words
Prosthetic composite, Intrinsic color stability, Extrinsic color stability, Accelerated aging

Recently introduced prosthetic composite systems have been used in an attempt to resolve some of the problems inherent in dental ceramics. Porcelain has color-rendering properties and optical properties that simulate natural teeth.[1] However, weak flexural strength and fracture resistance, destructive abrasiveness when used on the opposing natural tooth, a time-consuming and technically sensitive fabrication procedure and opaque nature in metal ceramic restorations are many of its numerous undesirable characteristics.[2]

The prosthetic resins currently used were introduced in the mid 1990's and are called second generation indirect composites or ceramic-polymers (ceromer). The ceromer material contains silanized, microhybrid inorganic fillers embedded in a light curing organic matrix. Due to the higher density of inorganic filler than traditional direct and indirect composites, their physical and mechanical properties are superior.[3,4] Ceromer materials can bond to dentin and enamel to excellent retention. Their unique curing methods produce the potential for optimal curing.[3,4] Easy repair is another advantage. The thermal expansion coefficient and the elastic modulus of the ceromer are quite similar to dentin. The material exhibits a deformation capacity similar to that of a natural tooth, which reduces the fracture inducing stress between the restorations and the tooth structures. Ceromers have become popular alongside FRC (Fiber Reinforced Composite) systems for use in aesthetic prosthetic restorations such as inlays, onlays, veneering, metal-free anterior or posterior fixed prosthesis. Artglass (Heraeus Kulzer, germany), BelleGlass HP (Kerr / Syborn, USA), Herculite (Kerr, USA), Sculpture (Jentric, USA), Targis (Ivoclar, Liechenstein) are available second generation indirect composites.[4-7] Although ceramic polymers have many advantages, there are few reports on the color stability of these materials.

Discoloration is a major cause for the replacement of the resin.[10] Discoloration of the resin materials is caused by intrinsic and extrinsic factors. The intrinsic factors involve an internal reaction in the resin material, such as the alteration of the resin matrix and the matrix and filler interface. The color change may also be caused by changes in temperature, immersion in water and exposure to ultraviolet light.[11,12,19-21]

Extrinsic factors for resin discoloration include stains by colorants from exogenous sources like coffee, tea, nicotine, and other beverages. The extent of discoloration in the oral cavity relates to dental hygiene and dietary habits. The colorants accumulate on the surface and penetrate to the sub-surface by superficial degradation and a reaction of the staining agents within the superficial layer.[13,14]

Several studies on the color changes of resin composites have been reported.[11-20] However, little is known regarding the internal color stability of ceramic polymers after accelerated aging and its relationship with external discoloration.

The purpose of the this study was to evaluate the intrinsic color stability of several current prosthetic composites after 100, 200, 300, 400 hours of accelerated aging and the extrinsic color stability after immersion in a coffee solution for 12, 24, 48, 72 hours and 1 week. The effect of accelerated aging on external discoloration was also evaluated by comparing the amount of external discoloration in the immersion test after with and without aging.

MATERIALS AND METHODS

Three dentin prosthetic composites, one light polymerized direct composite (Z100, 3M, USA) and one dental porcelain (Ceramco, Ceramco, USA) were selected for this study (Table Ⅰ). The sculpture group was divided into two subgroups by the finishing method, polishing and glazing. Six disks, 2mm in thickness and 10mm in diameter in shade B2 (Vita Lumin Shade guide) were fabricated for each material. Resin specimens were fabricated using a split acrylic mold between two glass slides over a Mylar strip. These specimens were polymerized according to manufacturer's instructions. Porcelain specimens were fabricated using a larger mold (thickness 2.5mm and diameter 13mm) without a Mylar strip. The porcelain was fired once and glazed according to the manufacturer's recommendations. The specimens were ground with 600, 800 grit silicon carbide (SiC) paper and polished (Table

Table Ⅰ**.** Materials tested

Product (Manufacturer)	Type	Filler	Inorganic filler (% Wt)	Filler size
Ceramco (Ceramco, USA)	Porcelain	N / A	N / A	N / A
Z100(3M,USA)	Microhybrid	Zirconia Silica	66	0.01～3.5μm
Artglass (Kulzer, Germany)	Ceromer	Silicone dioxide Barium alumina silica glass	70	0.7μm
Targis (Ivoclar, Richitenschetein)	Ceromer	Barium alumina silica glass Mixed oxide Silicon dioxide	75～85	1mm 200nm 40nm
Sculpture (Gentric pentron, USA)	Ceromer	Barium borosilicate glass Hydrophobic amorphous silica	79	0.6μm

Table Ⅱ**.** Finishing instruments for polishing

Materials	Instruments		Manufaturer
Z100	Shofu Supersnap polishing set		Shofu, Düsseldorf, Germany
	Medium grit	5000 r.p.m. / 20s	
	Fine grit	5000 r.p.m. / 20s	
	Super fine grit	5000 r.p.m. / 20s	
Artglass	Art-Glass tool kit		Kulzer, Wehrheim, Germany
	Polisher (Prepol)	5000 r.p.m. / 120s	
	Polisher (Mepol)	5000 r.p.m. / 60s	
	Polisher (Hipol)	5000 r.p.m. / 120s	
Targis	Big silicone point (BL-R3)	5000 r.p.m. / 120s	Shofu, Düsseldorf, Germany
	Robinson brush (brown)	5000 r.p.m. / 120s	Renfert, Germany
	Silicon wheels, white		Ash / Densply, York, USA
	Linen-brush and polish paste green		Ivoclar, Schaan, Leichtenstien
	Linen-brush and polish paste blue		Ivoclar, Schaan, Leichtenstien
Sculpture	Big silicone point (BL-R3)	5000 r.p.m. / 120s	Shofu, Düsseldorf, Germany
	Robinson brush (brown)	5000 r.p.m. / 120s	Renfert, Germany
	Polishing buffers		

Fig. 1. Suntest CPS

Ⅱ). All specimens were polished on one side and randomly coded on the unpolished surface. The specimens were stored for a minimum 3 days in air at room temperature.

1. Accelerated aging

The specimens were subjected to an artificial aging process in a weathering machine (Suntest CPS, Atlas electronic devices, Chicago, IL) with exposure to a controlled-irradiance xenon arc filtered through borate borosilicate glass at 0.55 $W/m^2/nm$ at 340nm (Fig. 1). The other test conditions included: a black standard temperature of 70℃ (light) at the specimen level and immersion in 38℃ distilled water.

The test cycle was 40 minutes irradiation only, 20 minutes irradiation with water immersion, 60 minutes irradiation only and 60 minutes dark with water immersion. The total elapsed exposure was 792 kJ/m^2 for 400 hours. The accelerated aging conditions used in this study were modeled after conditions used in previous studies.[16-21]

2. Preparation of a staining solution, methods of staining and cleaning[13]

To prepare the coffee solution, 60g of instant coffee (Taster's choice, Nestle, Vevey, Switzerland) was placed on filter paper and 1L of boiling distilled water passed through the filter. The test specimens were stored in the coffee solution at 50° for 12, 24, 48, 72 hours and 1 week. Following removal from the staining solution, the specimens were dipped in a soap solution, consisting of 10 ml soap and 700 ml distilled water and sonicated for 5 min.

3. Specimen evaluation

The color was measured according to CIE L*a*b* color scale[24] relative to D65 on a reflected spectrophotometer (CM-3500d, Minolta, Japan) with the spectral component included (SCI) geometry. The aperture diameter of the spectrophotometer measuring port was 3mm. The illumination and viewing conditions of this instrument used a CIE diffuse/8° geometry. A CIE 1964 supplementary standard colorimetric observer was selected. The SpectraMagic version 1.01 (Minolta, 1997) software was used. The CIE L*a*b* is an approximately uniform color space with coordinates for lightness, i.e. white-black, L*; redness-greenness, a*; and yellowness-blueness, b*. The color changes (ΔE^*) were calculated using the equation, $\Delta E^* = [(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$.

The color measurements were made at the baseline, after 100, 200, 300, 400 hours of the accelerated aging process and after 12, 24, 48, 72 hours, and 1 week of immersion in the coffee solution. The mean CIE L*a*b* values were determined from 3 separate measurements, repositioning the specimen after each measurement.

4. Statistical analysis

The mean ΔE and standard deviation values were compared using a one-way ANOVA and the Scheffe's multiple range tests (α<0.05) were used to determine the effects of i) accelerated aging, ii) immersion in the staining solution and iii) combination of the previous two conditions on the color of the specimen groups. A comparison of the mean ΔE values of the immersion test after aging with those

Table Ⅲ. Color changes by 400 hours of accelerated aging

Subject	N	ΔE100*	ΔE200*	ΔE300*	ΔE400
Ceramco	6	0.48(0.28)[a]	0.72(0.59)[a]	0.82(0.54)[a]	1.43(1.00)
Artglass	6	1.75(0.35)[b]	2.16(0.78)[a,b]	2.02(0.85)[a,b]	1.62(0.79)
Targis	6	1.85(0.35)[b]	2.21(0.38)[a,b]	1.97(1.10)[a,b]	2.33(0.81)
S -glaze	6	1.64(0.82)[a,b]	2.39(0.70)[b]	1.67(0.69)[a,b]	2.40(0.87)
S- pol*	6	1.38(1.01)[a,b,A]	2.12(1.32)[a,b,A]	2.39(1.18)[b,A]	2.45(1.33)[A]
Z100	6	2.34(0.23)[b]	2.47(0.46)[b]	2.58(0.51)[a,b]	2.61(0.90)

- Mean values of 6 specimens with the standard deviation in parentheses.
- * Denotes statistically different ($P < 0.05$). If the difference is statistically insignificant, the result of the Scheffe test is not written.
- For comparisons between the products, the mean with the same lower case letter are not statistically different at $p = 0.05$ using the Scheffe test.
- For comparisons between the aging time, the mean with the same capital letter are not statistically different at $p = 0.05$ using the Scheffe test.
- S-glaze denotes sculpture-glaze and S-pol denotes sculpture-polishing

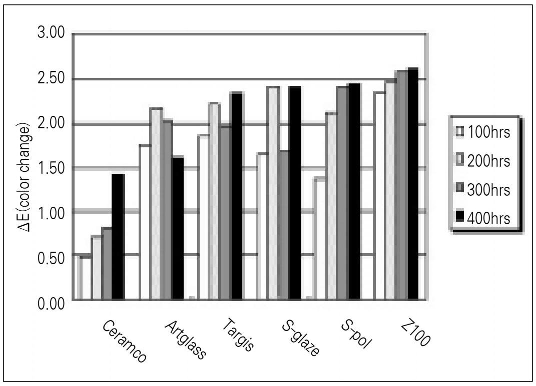

Fig. 2. Color changes of 6 materials for 400 hours of accelerated aging.

without aging was done using a student's t-test .

RESULTS

1. Intrinsic color stability after the accelerated aging process

Mean and standard deviation of the ΔE values for each specimen group after 100 hours of the accelerated aging process are listed in Table Ⅲ and Fig. 2. After 100 hours of accelerated aging, the sculpture glazing and polishing groups demonstrated less color changes than the other resin groups, which were similar to those of porcelain. However, after 200 hours, the color changes were not significantly different among the resin groups. After 400 hours, color changes were not significantly different among all the groups ($p>0.05$). As a function of aging time, only the sculpture polishing group demonstrated significant color changes. However, a statistical difference was not detected by the Scheffe test. The total color changes ranged from a magnitude of 0.48-2.61.

2. The extrinsic color stability in the staining solution after the accelerated aging process

The mean and standard deviation of the ΔE values for each specimen group immersed in the coffee solution for 1 week after 400 hours of aging are listed in Table Ⅳ and Fig. 3. During the test, the color changes were lowest in the porcelain group and highest in the Z100 group ($p<0.05$). Among the

Table Ⅳ. Color changes by immersion in the coffee solution after 400 hours of aging process

Subject	N	ΔE(12 hrs)*	ΔE(24 hrs)*	ΔE(48 hrs)*	ΔE(72 hrs)*	ΔE (1week)*
Ceramco	6	0.73(0.60)[a]	0.83(0.47)[a]	0.75(0.53)[a]	0.91(0.51)[a]	0.72(0.49)[a]
Artglass*	6	6.34(1.77)[a,b,A]	6.83(1.94)[b,c,A,B]	9.65(2.50)[b,c,A,B]	10.53(2.62)[b,c,B]	10.28(2.40)[b,c,A,B]
Targis*	6	5.98(1.65)[a,b,A]	6.71(2.09)[b,c,A]	9.53(2.45)[b,c,A]	10.09(2.53)[b,c,A]	10.67(2.39)[b,c,A]
S-glaze*	6	4.63(1.81)[a,b,A]	5.06(2.05)[a,b,A]	6.81(2.48)[a,b,A]	7.63(2.80)[a,b,A]	8.29(2.07)[b,A]
S-pol	6	4.73(2.59)[a,b]	4.67(2.05)[a,b]	5.93(2.71)[a,c]	5.93(2.71)[a,b]	7.20(3.01)[b]
Z100*	6	9.78(5.01)[b,A]	11.86(3.34)[c,A]	15.03(4.27)[c,A]	16.10(4.32)[c,A]	15.98(3.67)[c,A]

- Mean values of 6 specimens with the standard deviation in parentheses.
- * Denotes statistically different ($P < 0.05$). If the difference is statistically insignificant, the result of the Scheffe test is not written.
- For comparisons between the products, the mean with the same lower case letter are not statistically different at $p = 0.05$ using the Scheffe test.
- For comparisons between the aging time, the mean with the same capital letter are not statistically different at $p = 0.05$ using the Scheffe test.
- S-glaze denotes sculpture-glaze and S-pol denotes sculpture polishing

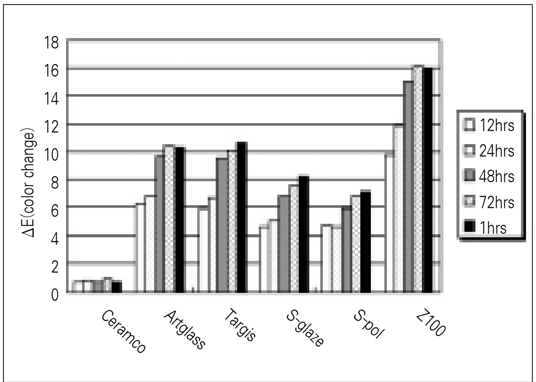

Fig. 3. Color changes of groups caused by immersion without aging

prosthetic composites, the sculpture groups especially the sculpture polishing group, showed the lowest amount of color change. After 24 hours of immersion, the color changes of the sculpture groups were significantly lower than those of the Z100 group. However, the differences were not significant for the sculpture, Targis and Artglass groups from 12 hours to 1 week of immersion. In terms of the immersion time, all the resin groups except the sculpture polishing group demonstrated significant color changes. In the Artglass group, color changes between 12 hours and 72 hours were significantly different. However, a statistically significant difference in the other resin groups was not detected by the Scheffe test. The total color changes ranged from a magnitude of 0.73-16.10.

3. External discoloration in the staining solution without the accelerated aging process

The mean and standard deviation of the ΔE values for each specimen group immersed in the coffee solution without aging for 1 week are shown in Table Ⅴ and Fig. 4. During the test, the sculpture glazing group showed the most color changes and both the Targis and Artglass groups exhibited the least color change in the prosthetic composite resin groups, which was also similar to the Ceramco group. The color changes in the sculpture polishing group were somewhere between those of the sculpture glazing group and the Targis and Artglass groups, which was similar to those of the Z100 group.

Table Ⅴ. Color changes of groups caused by immersion without aging

Subject	N	ΔE(12 hrs)*	ΔE(24 hrs)*	ΔE(48 hrs)*	ΔE(72 hrs)*	ΔE(1week)*
Ceramco	6	0.63(0.26)^{a}	0.58(0.52)^{a}	0.62(0.43)^{a}	0.52(0.51)^{a}	0.60(0.43)^{a}
Artglass*	6	0.77(0.37)^{a,b,A}	1.23(0.52)^{a,b,A}	1.57(0.78)^{a,b,A}	1.76(0.67)^{a,c,A,B}	2.99(1.17)^{a,b,B}
Targis	6	8.25(1.82)^{a}	1.02(0.80)^{a}	1.23(0.97)^{a}	1.67(0.85)^{a,c}	2.50(1.67)^{a,b}
S-glaze	6	3.28(1.54)^{c}	9.46(1.95)^{c}	9.62(1.84)^{d}	9.91(1.52)^{c}	10.95(2.11)^{d}
S-pol*	6	4.73(2.59)^{b,A}	3.50(1.72)^{b,A}	4.58(1.36)^{c,A}	5.73(1.62)^{b,A}	5.95(1.99)^{b,c,A}
Z100*	6	2.48(0.80)^{a,b,A}	3.50(0.85)^{b,A}	3.69(0.99)^{b,c,A}	4.60(1.26)^{b,c,A}	7.22(2.46)^{c,B}

■ Mean values of 6 specimens with the standard deviation in parentheses.

■ * Denotes statistically different (P <0.05). If the difference is statistically insignificant, the result of the Scheffe test is not written.

■ For comparisons between the products, the mean with the same lower case letter are not statistically different at p =0.05 using the Scheffe test.

■ For comparisons between the aging time, the mean with the same capital letter are not statistically different at p =0.05 using the Scheffe test.

■ S-glaze denotes sculpture-glaze and S-pol denotes sculpture-polishing

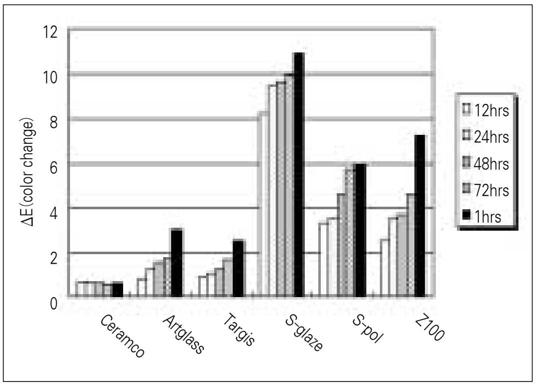

Fig. 4. Color changes of groups caused by immersion without aging.

Regarding the immersion time, the Z100, Artglass and sculpture polishing groups exhibited significant color changes. The Z100 group showed significant color changes after 1 week and the Artglass group exhibited color changes after 72 hours. A significant difference in the sculpture polishing group was not detected by the Scheffe test. The total amount of color change ranged from a magnitude of 0.62-10.95.

4. Color change difference between groups which were immersed after aging and without aging process.

The analysis by the student's t-test revealed that in the Ceramco and sculpture polishing groups, the aging process had no significant effect on the external color changes. In the Z100, Artglass and Targis groups, the changes were higher in the groups immersed in the coffee after aging than those immersed without aging. In the sculpture glazing group, the unaged specimens showed the largest color changes but after 48 hours, the differences were not statistically significant.

DISCUSSION

Prosthetic composite resin materials without metal substructures offer the optical advantage of more natural light dynamics and increased biologic compatibility. Clinical observations over a 4-year period have shown that in terms of wear, ceromer restorations had an equal longevity to ceramic restorations.[25] In clinical conditions, the character of color stability in prosthetic composites is also crucial.

Spectrophotometry is the standard method for measuring color. It is used in population samples, porcelain manufacture and the development of shade guides. A spectrophotometer with an integrating sphere can operate two different measuring geometries, the specular component included (SCI) and excluded (SCE). With the SCI geometry, the color changes, the surface roughness and geometrical shadowing function can be measured.[26]

The accelerated aging process simulates the effects of long-term exposure to environmental conditions that involves ultraviolet light exposure, temperature and humidity changes. The process was originally designed to determine how commercial products, such as automobile paints and textiles, would respond to outdoor conditions. This aging method has been used to test the intrinsic color stability of dental resins since 1978. The international organization for standardization (ISO) determines the color stability of dental polymeric materials through an aging process with radiation and circulating water[27]. Although the manufacturer of the weathering device estimates that 300 hours of weathering is equivalent to 1 year of service, a direct relation between the weathering time and the period of clinical use has not been reported.

For 400 hours of the accelerated aging process, the ΔE values of the prosthetic composite groups increased except those of the Artglass group. According to Power et al.[17-20], the color changes under the accelerated aging process were attributed to the continuing formation of colored degradation products and surface roughening caused by the wear of chemical degradation. The level of increase was highest at the first 100 hours. Öysaed and Ruyter reported that most of water absorption takes place during the first week.[28] In this study, the test was begun using dried samples. Thus, besides irradiation and temperature, the initial amount of water absorption appears to contribute to the highest color change ranging from 0.48-2.34 at 100 hours. The size of the filler particles in the composites also influenced the color stability. Smaller filler particles scatter light more effectively than larger particles and any slight discoloration may be masked.[16] This can be one of the reasons why the ΔE value was lower in the sculpture group than the Targis group after 100 hours of aging.

For 400 hours, the ΔE values increased generally but after 300 hours, a decreasing tendency was observed in the Artglass, Targis and the sculpture glazing groups. This was in contrast to Ruyter's report.[22] Most of the color change was observed to occur in the first 300 hours and reached a plateau after 1440 hours of aging. In this study, despite the decreasing tendency, the color change difference in the three groups according to aging time was insignificant. The maximum difference between 200 hours and 300 hours was 0.72 in the sculpture glazing group, which was similar to the mean standard deviation.

After 400 hours of aging, no statistically significant difference was observed among the color changes of all groups. Among the prosthetic composite groups, no difference was observed for the whole accelerated aging test. The largest ΔE value was 2.61 in the Z100 group, which is lower than the 3.3 considered to be the upper limit of acceptability in subjective visual evaluations.[16,22] By measuring the internal discoloration, discriminating the materials was difficult.

In the immersion test after aging, the sculpture group was less susceptible to the staining solution than both the Artglass and Targis groups. However, in the test without aging, the sculpture glazing group showed the highest discoloration. The color changes were not significantly different among prosthetic composites in the immersion test after aging but the difference was significant in the immersion test without aging.

Mair reported that subsurface staining was absent in composites and was an artifact observed by transmission microscopy.[29] This discolored composite layer or absorbed stains could be theoretically removed by proper polishing.[14] According to those studies, discoloration by a staining solution appeared to be affected by surface changes. Tanoue et al[30] reported that the sculpture group showed the smoothest surface even after tooth brushing in prosthetic composites, which contained microsized fillers. Cho and Yi[61] reported that polished Targis showed highest amount of change in luster due to its large filler particles. This explained the least color change of the sculpture and the resin group in the immersion test with aging.

In the immersion test without aging, the sculpture group was sensitive to water absorption. However, the specimens that were immersed after aging had been saturated with water before being soaked in the staining solution, and those that were immersed without aging absorbed the staining agent directly. This is associated with the characteristics of the composites that allow water to penetrate the matrix or filler-matrix interface.[17,18,32] Glazing liquid was also attributed to the color change. The glazing liquid used in the sculpture group was too sticky to be spread as a thin coating. This layer was sensitive to the staining agent, which contrasted to Garman's report showing that this resin coating maintained the color match of the restorations in a one year clinical study.[34] The Targis and Artglass groups showed lower color changes than the Z100 group, which were not significantly different from that of the Ceramco group.

Generally, the amount of discoloration was higher in the immersion test after aging than in the test without aging. However, in the Ceramco and sculpture polishing groups, the differences were insignificant. After 48hours, the sculpture glazing group showed no significant color difference between the group immersed after aging and that without aging. The differences were also insignificant in the Artglass, Targis and Z100 groups. The effect of the accelerated aging process was not obvious in the last three groups.

The color stability of current prosthetic composites was affected mostly by external colorants. The accelerated aging process caused few color changes itself but had influence on the external discoloration.

The resin matrix of current prosthetic composites has improved through the use of multifunctional methacrylate monomers and bifunctional monomers, and there are differences in the molecular structure and composition in the composite matrix.[9] The curing method also varies according to the material. Prosthetic composites showed improved color stability against the aging process based on these features. However, prosthetic composites have many inherent weak points from the resin material used. In this study, the factors associated with discoloration were not investigated. Further studies to assess the mechanism of prosthetic composites discoloration are recommended.

CONCLUSION

This study evaluated the intrinsic color stability of several current prosthetic composites after 100, 200, 300, 400 hours of accelerated aging and the extrinsic color stability after immersion in a coffee solution for 12, 24, 48, 72 hours and 1 week. The effect of accelerated aging on external discoloration was also evaluated by comparing the amount of external discoloration in the immersion test after aging with that without aging. Within the limitations of this study, the following conclusions were drawn:

1. In the accelerated aging test, the prosthetic composites demonstrated the most insignificant different color changes among the groups. After 400 hours of aging, the color changes of all the groups tested were no difference and were below a quantitative level that would be acceptable.
2. In the immersion test after aging, the color changes in the sculpture group showed lower values than those of both the Artglass and Targis groups. However, the differences were not significant among the prosthetic composites.

3. In the immersion test without aging, the sculpture glazing group showed the most color changes. Both the Artglass and Targis groups showed the least change next to the Ceramco group.
4. In a comparison of the color changes in the immersion test after aging with that without aging, the differences were found to be insignificant in the Ceramco and sculpture polishing groups. The Targis, Artglass and Z100 groups demonstrated more color changes in the immersion test after aging. The sculpture glazing group showed higher discoloration in the immersion test without aging until 24 hours. However, from 48 hours, the differences were insignificant.

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