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Influence of thermocycling on the optical properties of laboratory resin composites and an all-ceramic material

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The purposes were to determine the difference of color and translucency of dental laboratory resin composites and an all-ceramic material, and to compare the changes in optical properties after thermocycling. Three shades for enamel appearance of two laboratory resin composites (ART; Artglass, TES; Tescera) and all-ceramic material (EMP; Empress 2) were chosen, and three disk specimens for each shade and thickness (0.8, 1.3 and 1.8 mm) were made. Color and spectral reflectance were measured before and after thermocycling over white and black backgrounds. Contrast ratio (CR) and translucency parameter (TP) were calculated. Before and after thermocycling, CR of 1.8 mm specimens did not differ between the materials. After thermocycling, CR and TP of TES were not changed significantly; however, ART and EMP showed increased CR and decreased TP in some shades ($p = 0.05$). Color change of three materials after thermocycling was not significant. CR increased in inverse proportion to TP ($r = -0.93$, $p < 0.01$). In TES, change of translucency after thermocycling was lower than those of other materials regardless of the specimen thickness. TES seemed to yield more predictable curing performance.

1. Introduction

Dental ceramic and laboratory resin composites have been used for the veneering of anterior tooth. Dental ceramic has high compressive strength and resistance to discoloration [1]. However, the abrasiveness of porcelain is destructive to opposing natural teeth, and porcelain has relatively low fracture resistance [2]. Laboratory resin composites have been presented as an alternative to overcome some of the problems inherent in dental porcelain. Although they are essentially still a composite of resin matrix with differing filler components, properties of these materials have been improved by a hybridization of composite and ceramic technologies [3]. Therefore laboratory resin composites show higher flexural strength and wear resistance than those of resin composites for direct filling [4, 5]. Recently new curing technology was adopted to reduce voids and increase strength of laboratory resin composite. Restoration is subjected to pressure and light for initial curing, and additional pressure, light and heat curing under water is followed [6]. Optical properties of this new composite may differ from those of conventional laboratory resin composites.

Normally light penetrates through the enamel into dentin before being reflected outward. This affords the lifelike esthetic vitality characteristic of natural teeth [7]. Since enamel has inherent translucency, dental restorations should reproduce the translucency of natural tooth [8, 9]. Translucency of esthetic dental materials is usually evaluated with contrast ratio (CR) and translucency parameter (TP) [1, 10].

Relatively high variability in translucency changes has been shown among the products of resin composite, with some products increasing and others decreasing in TP after immersion in water [11]. Properties of resin composites were changed after thermocycling by thermal fatigue, and the influence of thermal fatigue was different by the properties of material [12]. Color change of resin composite was perceivable after thermocycling [13]. Color stability of conventional laboratory resin composites was evaluated after accelerated aging, and the color stability was different by the material [14]. The post-curing properties of resin composites were found to be influenced by the type of laboratory curing unit [15]. Hence, large changes in optical properties that occur when laboratory resin composites are stored or processed under various environmental conditions indicate that such changes should be taken into account when these materials are used. However, there are few reports on the color and optical properties of recently introduced laboratory resin composites, which adopted a new curing technology.

The purposes of this study were to determine the difference of color and translucency of laboratory resin

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composites and an all-ceramic material of several shades and thickness, and to compare the changes in optical properties after thermocycling.

2. Materials and methods

Three shades for enamel appearance of two laboratory resin composites (ART, TES) and one incisal porcelain for all ceramic material (EMP) were studied (Table I). The shades of ART were EL, EM and ED; those of TES were Clear, Yellow and Gray; and those of EMP were S1, S2 and S3.

Three disk specimens per each shade and thickness (0.8, 1.3 and 1.8 mm) were made with Teflon molds. ART was filled into the Teflon mold. The mold was pressed between two glass plates and placed in the UniXS unit (Heraeus Kulzer, Hanau, Germany), which is the proprietary photo-curing unit for ART. Resin filled mold was exposed to curing light from the top and bottom for 90 s, respectively. In case of TES, curing procedure consisted of two steps. Proprietary curing unit (TESCERA™ ATL, Bisco, Schaumburg, IL, USA) was used with the light cup and heat cup. In the first step, using the light cup and curing unit, specimen was subjected to pressure cycle only to reduce the defects and porosity, and then light-curing procedure was applied under the same pressure to polymerize the material. In the second step, using the heat cup and curing unit, the specimen was immersed in water, and heat was transferred quickly to the material via water. In case of incisal enamel porcelain for EMP, slurry of ceramic was prepared and then condensed into the molds of 18 mm in diameter to compensate for shrinkage. After condensation, the disk was transferred to sagger tray and fired according to the manufacturer's instructions.

After curing or firing the specimens, they were ground with abrasive papers fixed to a rotating wheel. Final dimension of all specimens was 12 mm in diameter and 0.8, 1.3 or 1.8 mm in thickness. Surface was flooded with water during the polishing, and the final polishing was carried out with #2000 silicone carbide paper. Before color measurement, the specimens were stored in distilled water for 24 h at 37 °C. Color was measured three times per each specimen before and after thermocycling. Thermocycling was performed for 2000 cycles between 5 and 55 °C distilled water with the dwell time of 30 s.

Color was measured according to the CIELAB color scale relative to the standard light source D65 against a white background (CIE $L^* = 96.68$, $a^* = -0.18$, $b^* = -0.22$) and a black background (CIE $L^* = 1.15$, $a^* = -0.11$, $b^* = -0.50$) on a reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan). A drop of optical fluid (1.5 index of refraction fluid, Cargille Lab, Cedar Grove, NJ, USA) was applied to the standard background. Optical fluid was placed between the background and specimens for an optical connection [16]. The aperture diameter of the measuring port of the reflection spectrophotometer was 8 mm. Reflectivity was measured every 20 nm intervals between 400 and 700 nm. The software was Spectra-Magic version 1.01 (Minolta, 1997). Color differences (ΔE^*_{ab}) were calculated by the equation, $\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

TABLE I Materials used in this study

Code	Brand name	Shade	Batch number	Manufacturer
ART	Artglass	EL	330151	Heraeus/Kulzer, Hanau,
		EM	330152	Germany
		ED	330153	
TES	Tescera	Clear	T-130CLP	Bisco, Schaumburg, IL,
		Gray	T-130GRP	USA
		Yellow	T-130YLP	
EMP	Empress 2	S1	D65943	Ivoclar Vivadent, Schaan,
		S2	D63918	Liechtenstein
		S3	E54570	

CR is the ratio between the reflectance of a specimen when backed by a black standard and the reflectance of the same specimen when backed by a white standard having a daylight apparent reflectance of 70% (or sometimes 100%) relative to magnesium oxide [1]. TP is obtained by calculating the color difference between the specimen on a white background and the specimen on a black background: $\mathrm{TP} = [(L^*_\mathrm{B} - L^*_\mathrm{W})^2 + (a^*_\mathrm{B} - a^*_\mathrm{W})^2 + (b^*_\mathrm{B} - b^*_\mathrm{W})^2]^{1/2}$, where the subscript $_\mathrm{B}$ refers to the color parameters on a black background and the subscript $_\mathrm{W}$ refers to those on a white background [10].

Means and standard deviations of changes of CR, TP and color (ΔE^*_{ab}) were calculated. Values were analyzed by Student *t*-test, analysis of variance and Scheffe multiple range test at the significance level of 0.05. The relationship between CR or TP and material thickness was analyzed by a simple regression analysis. A multiple regression analysis was used to test the relationship between the changes of CR or TP and the changes of CIE L^*, a^* and b^* values.

3. Result

(1) Effect of shade on the CR and TP

CR values by the wavelength of ART (EM shade) and TES (Yellow shade) are in Figs. 1 and 2. CR value decreased as the wavelength increased. Changes after thermocycling were different by the material and thickness of specimen.

CR and TP values before and after thermocycling are in Tables II and III, respectively. In ART, CR values of

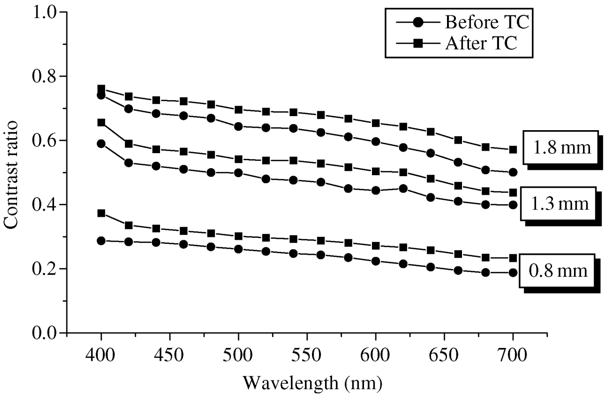

Figure 1 Contrast ratio of ART (EM).

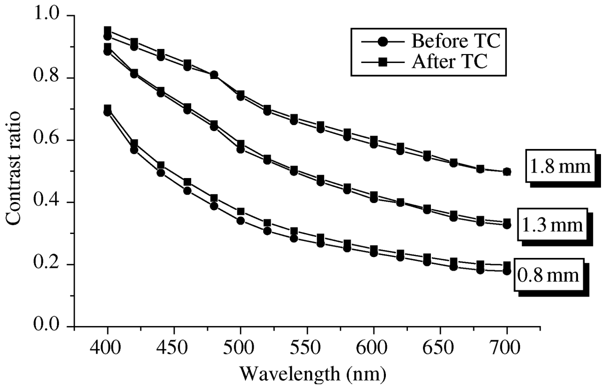

Figure 2 Contrast ratio of TES (Yellow).

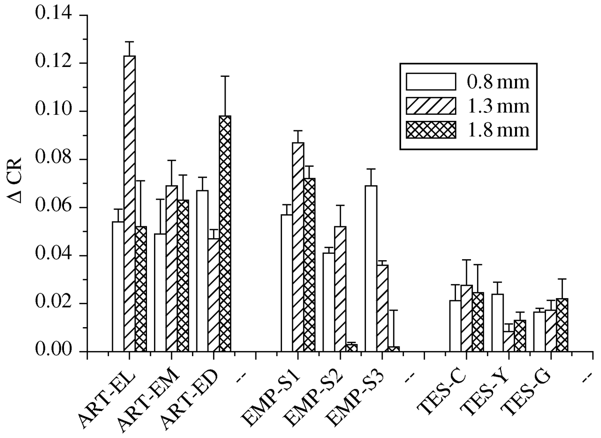

Figure 3 Changes of contrast ratio after thermocycling.

ED shade were higher than those of other shades, and TP values of ED shade were lower than those of other shade in before and after thermocycling conditions. In TES, CR values of Yellow shade were higher than those of other shades, and TP values of Yellow shade were lower than those of other shades.

(2) Effect of specimen thickness on the CR and TP

Thickness was positively correlated with CR [correlation coefficient $(r) = 0.92$–0.94 in ART, 0.93–0.96 in EMP and 0.91–0.93 in TES], and negatively correlated with TP ($r = -0.92$ to -0.94 in ART, -0.93 to -0.97 in EMP and -0.94 to -0.97 in TES). All the correlation coefficients were significant at the level of $p = 0.01$.

(3) Effect of thermocycling on the CR and TP values

Changes in CR after thermocycling (= CR value after thermocycling – CR value before thermocycling) varied by the shade and thickness, and the highest value was 0.12 in ART, 0.09 in EMP and 0.02 in TES (Fig. 3). Changes in TP after thermocycling ranged from -5.11 to -0.46 in ART, from -3.76 to -1.15 in EMP and from -1.04 to -0.22 in TES (Fig. 4). In TES, changes in CR and TP were lower than those of other two materials regardless of the specimen thickness.

Generally CR increased and TP decreased after thermocycling. After thermocycling, CR and TP values of TES showed no significant change regardless of shade and thickness ($p > 0.05$). However, in ART and EMP, CR increased and TP decreased significantly in some shades and thickness after thermocycling ($p < 0.05$).

(4) Effect of material on the CR and TP

Before thermocycling, CR was 0.19–0.33, 0.33–0.53 and 0.61–0.69 in specimens with a thickness of 0.8 mm,

TABLE II Mean (standard deviation) of contrast ratio before and after thermocycling (TC)

Code	Shade	TC	0.8 mm	1.3 mm	1.8 mm
ART	EL (1)[a]	Before	0.17 (0.03)[b]	0.39 (0.06)[b]	0.62 (0.06)
		After	0.22 (0.03)	0.51 (0.06)	0.67 (0.06)
	EM (2)	Before	0.24 (0.04)	0.46 (0.06)	0.62 (0.07)[b]
		After	0.29 (0.04)	0.53 (0.06)	0.68 (0.06)
	ED (3)	Before	0.27 (0.04)	0.53 (0.06)	0.68 (0.06)
		After	0.34 (0.10)	0.57 (0.07)	0.78 (0.07)
	DG[c]	Before	1 < 2.3	1 < 2 < 3	NS[d]
		After	1.2 < 3	1.2 < 3	1.2 < 3
EMP	S1 (1)	Before	0.19 (0.04)	0.33 (0.07)[b]	0.66 (0.06)
		After	0.25 (0.04)	0.41 (0.07)	0.73 (0.05)
	S2 (2)	Before	0.20 (0.03)	0.36 (0.06)	0.66 (0.06)
		After	0.24 (0.03)	0.41 (0.06)	0.66 (0.05)
	S3 (3)	Before	0.20 (0.03)[b]	0.40 (0.06)	0.66 (0.06)
		After	0.27 (0.04)	0.43 (0.07)	0.67 (0.05)
	DG	Before	NS	NS	NS
		After	NS	NS	NS
TES	Clear (1)	Before	0.27 (0.14)	0.41 (0.19)	0.61 (0.18)
		After	0.28 (0.14)	0.43 (0.19)	0.64 (0.19)
	Yellow (2)	Before	0.33 (0.15)	0.53 (0.18)	0.69 (0.15)
		After	0.35 (0.15)	0.54 (0.18)	0.72 (0.22)
	Gray (3)	Before	0.26 (0.14)	0.42 (0.17)	0.61 (0.20)
		After	0.28 (0.14)	0.40 (0.18)	0.63 (0.19)
	DG	Before	3.1 < 2	3.1 < 2	3.1 < 2
		After	3.1 < 2	3.1 < 2	3.1 < 2

[a] This numeric codes are used in DG.

[b] This value changed significantly after thermocycling (t-test, $p < 0.05$).

[c] DG; Different groups and " < " means significantly different group marker from Scheffe's multiple comparison test ($p < 0.05$).

[d] No significantly different groups.

TABLE III Mean (standard deviation) of translucency parameter before and after thermocycling

Code	Shade	TC	0.8 mm	1.3 mm	1.8 mm
ART	EL (1)[a]	Before	37.6 (3.7)[b]	19.4 (1.0)[b]	10.1 (4.2)
		After	32.5 (2.9)	14.9 (2.4)	8.4 (2.5)
	EM (2)	Before	30.1 (4.2)	16.6 (1.7)	9.5 (1.3)
		After	27.0 (4.3)	13.8 (1.7)	8.2 (0.8)
	ED (3)	Before	28.4 (3.9)	12.9 (1.4)	7.5 (1.2)
		After	26.0 (2.2)	12.4 (1.7)	5.9 (1.7)
	DG[c]	Before	3.2 < 1	3 < 2 < 1	NS[d]
		After	3.2 < 1	3.2 < 1	3.2 < 1
EMP	S1 (1)	Before	32.3 (3.7)	21.2 (1.7)	15.6 (2.5)
		After	30.5 (1.8)	19.2 (1.6)	9.7 (2.2)
	S2 (2)	Before	33.3 (2.8)	21.1 (1.3)	15.0 (2.8)
		After	30.8 (2.9)	19.6 (2.9)	9.1 (0.8)
	S3 (3)	Before	34.8 (1.0)[b]	20.1 (3.1)	15.9 (2.5)
		After	29.1 (4.6)	18.9 (2.9)	9.1 (1.5)
	DG	Before	NS	NS	NS
		After	NS	NS	NS
TES	Clear (1)	Before	35.6 (2.6)	25.3 (3.7)	15.4 (2.4)
		After	34.5 (3.4)	24.8 (0.9)	15.2 (1.8)
	Yellow (2)	Before	32.3 (2.8)	21.2 (0.7)	13.9 (1.5)
		After	31.6 (3.5)	20.3 (0.9)	13.0 (1.5)
	Gray (3)	Before	34.7 (4.3)	25.5 (1.5)	16.7 (2.1)
		After	34.3 (4.0)	24.5 (2.5)	15.9 (2.8)
	DG	Before	NS	2 < 1.3	2 < 1.3
		After	NS	2 < 3.1	2 < 1.3

[a] This numeric codes are used in DG.

[b] This value changed significantly after thermocycling (*t*-test, $p < 0.05$).

[c] DG; Different groups and " < " means significantly different group marker from Scheffe's multiple comparison test ($p < 0.05$).

[d] No significantly different groups.

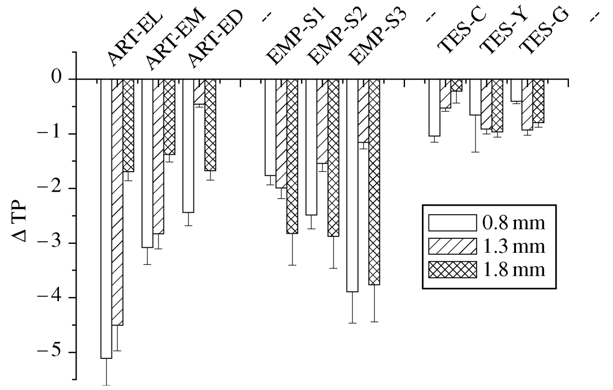

Figure 4 Changes of translucency parameter after thermocycling.

1.3 mm and 1.8 mm, respectively, and was varied by the material. There was no significant difference in CR of specimens with a thickness of 1.8 by material. After thermocycling, though there was difference by the shade, CR of EMP was generally lower than those of other materials with the thickness of 0.8 and 1.3 mm ($p < 0.05$). Before thermocycling, TP was 28.4–37.6, 12.9–25.3 and 7.5–16.7 in specimens with a thickness of 0.8 mm, 1.3 mm and 1.8 mm, respectively, and was varied by the material. Except EL shade of 0.8 mm, TP of ART was generally lower than those of other materials regardless of thermocycling and specimen thickness ($p < 0.05$).

(5) Influencing variable on translucency change

The influence of the change in CIE L^*, a^* and b^* values after thermocycling on the change of CR or TP was determined. Changes in CIE L^*, a^* and b^* values were negatively correlated with the change of CR [multiple correlation coefficient $(r) = -0.84$, $p < 0.01$] and were positively correlated with the change of TP ($r = 0.90$, $p < 0.01$). CR increases in inverse proportion to TP ($r = -0.93$, $p < 0.01$).

TABLE IV Mean (standard deviation) of color changes (ΔE^*_{ab}) after thermocycling

Code	Shade	Thickness	ΔE^*_{ab}
ART	EL	0.8 mm	2.2 (0.5)
		1.3 mm	1.5 (0.4)
		1.8 mm	2.0 (0.4)
	EM	0.8 mm	1.7 (0.7)
		1.3 mm	1.4 (0.8)
		1.8 mm	2.3 (0.8)
	ED	0.8 mm	2.4 (0.6)
		1.3 mm	1.3 (0.8)
		1.8 mm	1.2 (0.7)
EMP	S1	0.8 mm	1.5 (0.7)
		1.3 mm	1.8 (0.3)
		1.8 mm	2.1 (0.3)
	S2	0.8 mm	1.6 (0.5)
		1.3 mm	2.5 (1.0)
		1.8 mm	1.2 (0.1)
	S3	0.8 mm	2.1 (0.4)
		1.3 mm	2.3 (0.5)
		1.8 mm	1.4 (0.3)
TES	Clear	0.8 mm	1.4 (0.0)
		1.3 mm	1.5 (0.7)
		1.8 mm	1.8 (0.6)
	Yellow	0.8 mm	2.3 (0.6)
		1.3 mm	2.4 (0.2)
		1.8 mm	1.8 (0.5)
	Gray	0.8 mm	2.0 (0.1)
		1.3 mm	2.3 (0.9)
		1.8 mm	2.2 (0.8)

(6) Color change (ΔE^*_{ab}) after thermocycling

After thermocycling, ΔE^*_{ab} was 1.20–2.38 in ART, 1.20–2.29 in EMP, and 1.42–2.44 in TES (Table IV). Any trends by the material or thickness could not be drawn.

4. Discussion

CR and TP values were calculated to evaluate the translucency of laboratory resin composites. Although direct measurement of light transmittance can be used to evaluate translucency [8, 9], this method requires more complicated apparatus. CR is one of the most widely used parameter to compare relative translucency, and CR value 1 means complete opacity. CR is determined as the ratio of reflectance $(\mathrm{Y_B}/\mathrm{Y_W})$ when the specimen is placed over black and white backgrounds [1]. TP is the color difference between a uniform thickness of material over black and white backgrounds, and corresponds directly to common visual assessment of translucency [10, 11]. In the present study, TP values of ART were lower than those of other two materials in each corresponding thickness except a few cases $(p < 0.05)$. This means that ART is less translucent than other two materials.

After thermocycling, CR values of ART and EMP increased, and there was a significant increase of CR in three cases of ART and two cases of EMP. However, there was no significant increase of CR in TES. After thermocycling, TP values of all three materials decreased, and there was significant decrease of TP in two cases of ART and one case of EMP; however, there was no significant decrease in TES $(p = 0.05)$. In TES, change of translucency after thermocycling was smaller than that of other materials (Figs. 3 and 4). This implies that new curing method adopted in TES resulted in higher degree of polymerization than that with light curing only, or diffusion of water was limited in TES. In case of ART, voids might be entrapped during the curing procedure. Although the number of cycles likely to be experienced *in vivo* has not been determined, a provisional estimate of approximately 10 000 cycles per year was suggested [17]. 2000 cycles of the present study might simulate relatively short period, therefore longer effect should be further studied. The effect of thermocycling on the properties of resin composites showed discrepancy by the regimens of thermocycling and materials [18, 19]. In the present study, the effect of thermocycling was different by the material. TES showed smaller changes in translucency.

Translucency of composite materials can vary with the degree of polymerization [20]. Although ART is light-cured material, its degree of polymerization may be further increased after long-term thermocycling procedure, especially in 55 °C water. Reduction in translucency seemed to be caused by the increased polymerization of the material and corresponding change of the refractive index of the resin matrix [20]. This would have produced a greater difference in refractive index between the resin matrix and the inorganic filler. Water sorption also might lead to discoloration and change in translucency. It was reported that various composite materials have different levels of water sorption depending on the type of monomer they are made of [21]. In the present study, the changes in TP after thermocycling of ART might reflect the increased polymerization after thermocycling. However, the changes in translucency and color of two laboratory composites were comparable to those of all ceramic material. The sintered glass-ceramic of EMP veneering material features a composite microstructure consisting of glass and glass-crystal phase, and the lithium disilicate glass-ceramic demonstrated a good translucency [22]. The changes of translucency and color of EMP might reflect the fact that water was diffused into the interface of composite structure.

Optical properties of resin composites changed depending on the properties of material, manipulative variables and environment [23]. Changes of color and TP of dental resin composites that occurred after submersion in water were evaluated. As a result, color change was prominent after storage in water [24]. Color stability of conventional laboratory resin composites was evaluated after accelerated aging. After 300 h of accelerated aging, color changes ranged between 0.62 and 3.40 ΔE^*_{ab} units [14]. After UV irradiation, accelerated aging and storage in varied solutions, the color stability of laboratory resin composites was evaluated. It was concluded that it remains essential to improve the color stability of the composite resin materials used for facings [25]. In the present study, color change (ΔE^*_{ab}) after 2000 cycles of thermocycling was 1.20–2.54. Since it was reported that ΔE^*_{ab} less than 3.3 units was clinically acceptable [26], all the color changes were less than values considered clinically unacceptable. However, since the sizes of ΔE^*_{ab} values for perceptible or acceptable color-difference were varied and dependent on chromaticity [27], further improvement in color stability is needed.

Changes of CIE L^*, a^* and b^* values negatively correlated with the change of CR $(r = -0.84)$, and positively correlated with the change of TP $(r = 0.90)$. This means high color change is related to low change in CR and high change in TP. After thermocycling, CR and TP of TES showed significantly lower changes than those of other materials regardless of the specimen thickness $(p < 0.05)$. In the present study, CR and TP values were also influenced by the shade of the material. As the lightness (CIE L^* value) of shade increased, CR decreased and TP increased. There was significant difference in CR and TP among shades of ART and TES; however, there was no significant difference among shades in EMP $(p = 0.05)$.

CR increased in inverse proportion to TP, and the correlation coefficient was -0.93 $(p < 0.01)$ in the present study. Therefore, CR and TP can be used to determine the translucency of veneering materials interchangeably.

As to the translucency of human enamel, the translucency of wet enamel was higher than that of dehydrated one. Transmittance increased with increasing incident wavelength [28]. In the present study, to minimize the effect of water content on the optical properties, optical properties were measured immediately after blot drying after specimens were immersed in distilled water for 24 h. As can be seen from Figs. 2 and 3, CR value decreased as the wavelength increased. This reconfirms the result of previous report.

From this study, translucency of 1.8 mm specimens showed small difference between the materials compared to those of 0.8 mm and 1.3 mm specimens. CR increased in inverse proportion to TP ($r = -0.925$, $p < 0.01$). In TES, change of translucency after thermocycling was lower than those of other materials regardless of the specimen thickness. Color change of three materials after thermocycling was not significant.

References

1. R. G. CRAIG and J. M. POWERS, in "Restorative dental materials", 11th edn (CV Mosby, St. Louis, 2002) p. 39.
2. P. MAGNE, W. S. OH, M. R. PINTADO and R. DELONG, *J. Prosthet. Dent.* **82** (1999) 669.
3. B. TOUATI and N. AIDAN, *J. Esthet. Dent.* **9** (1997) 108.
4. R. S. FREIBERG and J. L. FERRACANE, *Am. J. Dent.* **11** (1998) 214.
5. M. KERN, J. R. STRUB and X. Y. LU, *J. Oral Rehabil.* **26** (1999) 372.
6. R. FOSTER, *Restorative Quart.* **5** (2002) 3.
7. T. M. ROBERSON, H. O. HEYMANN and E. J. SWIFT, JR in "Art and Science of Operative Dentistry", 4th edn (CV Mosby, St. Louis, 2002) p. 598.
8. S. J. PAUL, P. PLISKA, N. PIETROBON and P. SCHARER, *Int. J. Periodont. Restorative Dent.* **16** (1996) 164.
9. H. ARIKAWA, K. FUJII, T. KANIE and K. INOUE, *Dent. Mater.* **14** (1998) 405.
10. W. M. JOHNSTON, T. MA and B. H. KIENLE, *Int. J. Prosthodont.* **8** (1995) 79.
11. W. M. JOHNSTON and M. H. REISBICK, *Dent. Mater.* **13** (1997) 89.
12. A. U. YAP, X. WANG, X. WU and S. M. CHUNG, *Biomaterials* **25** (2004) 2179.
13. Y. K. LEE, B. S. LIM, C. W. KIM and J. M. POWERS, *J. Biomed. Mater. Res. Appl. Biomater.* **58** (2001) 613.
14. R. D. DOUGLAS, *J. Prosthet. Dent.* **83** (2000) 166.
15. N. TANOUE, M. ATSUMA, H. MATSUMURA and M. ATSUTA, *J. Oral Rehabil.* **30** (2003) 832.
16. R. R. SEGHI, E. R. HEWLETT and J. KIM, *J. Dent. Res.* **68** (1989) 1760.
17. M. S. GALE and B. W. DARVELL, *J. Dent.* **27** (1999) 88.
18. L. H. MAIR and R. W. VOWELS, *Dent. Mater.* **5** (1989) 247.
19. A. U. YAP, E. C. WEE and S. H. TEOH, *Oper. Dent.* **27** (2002) 25.
20. T. NAKAMURA, O. SAITO, M. MIZUNO and H. TANAKA, *Int. J. Prosthodont.* **15** (2002) 494.
21. V. M. MIETTINEN, K. NARVA and P. K. VALLITU, *Biomaterials* **20** (1999) 1187.
22. W. HOEMLAND, M. SCHWEIGER, M. FRANK and V. RHEINBERGER, *J. Biomed. Mater. Res. Appl. Biomater.* **53** (2000) 297.
23. Y. K. LEE and J. M. POWERS, *Am. J. Dent.* **14** (2001) 349.
24. W. BUCHALLA, T. ATTIN, R. D. HILGERS and E. HELLWIG, *J. Prosthet. Dent.* **87** (2002) 264.
25. T. STOBER, H. GILDE and P. LENZ, *Dent. Mater.* **17** (2001) 87.
26. I. E. RUYTER, K. NILNER and B. MOLLER, *ibid.* **3** (1987) 246.
27. R. D. DOUGLAS and J. D. BREWER, *J. Prosthet. Dent.* **79** (1998) 254.
28. R. H. BROADBELT, W. J. O'BRIEN, P. L. FAN, J. G. FRAZER-DIB and R. YU, *J. Dent. Res.* **60** (1981) 1749.

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