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Opalescence and fluorescence properties of indirect and direct resin materials

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Abstract

Objective. To measure the opalescence and fluorescence properties of indirect and direct resin materials before and after polymerization, and to determine the influence of the material and shade group combination on these properties. ***Material and methods.*** BelleGlass NG (BG, indirect resin) and Estelite Sigma (ES, direct resin), each composed in 3 shade groups (EN, OD and TL for BG; BS, AS and OP for ES) out of a total of 16 shades were investigated. Resin material was packed into a mold (the BEC condition) and polymerized with a light-polymerization unit (CWL). Secondary polymerization (CIC) was performed for BG. Color was measured in the BEC, CWL, and CIC conditions, and the opalescence parameter (*OP*) and fluorescence parameter (*FL*) were calculated. ***Results.*** For the *OP*, the mean for BG material was 24.3 before polymerization, which changed to 19.9 after polymerization (CIC). In the case of ES, the mean *OP* before polymerization was 25.6, which changed to 12.4 after polymerization (CWL). For the *FL*, the mean *FL* for BG was 2.5 before polymerization, which changed to 0.7 after polymerization. In the case of ES, the mean *FL* before polymerization was 1.2, which did not change after polymerization. Material and shade group combination influenced the *OP* and *FL* values ($p < 0.05$). ***Conclusions.*** The opalescence and fluorescence properties of resin materials varied depending on the material, shade group, and polymerization. Clinically, these properties should be considered when neighboring teeth are restored with different types of material.

Key Words: *Opalescence, fluorescence, indirect resin material, direct resin material*

Introduction

One important goal of esthetic dentistry is to restore the color and appearance of natural dentition. Esthetic considerations in restorative and prosthetic dentistry have received greater emphasis over the past several decades. Among the optical properties required for ideal restorations of natural teeth, the importance of the opalescence and fluorescence properties of restorative materials has been emphasized recently, besides traditional color parameters such as value, hue, and chroma [1,2].

The enamel of natural teeth is opalescent, where there is light scattering of the shorter wavelengths of the visible spectrum, giving a tooth a bluish appearance in reflected color and an orange/brown appearance in transmitted color [3]. This light scattering is caused by particles, smaller than the wavelength of visible light, that are dispersed throughout a translucent matrix of a lower refractive index, creating the reflected light blue-gray hues that become clearly visible at the incisal halo [4,5]. Opalescence of dental materials was determined with the opalescence parameter (*OP*), which is the difference in yellow-blue color coordinate (CIE Δb^*) and red-green color coordinate (CIE Δa^*) between the reflected and transmitted colors [6–8].

Natural teeth emit a blue fluorescence under the action of ultraviolet (UV)-light, which makes teeth whiter and brighter in daylight [9,10]. Fluorescence of natural teeth is the phenomenon that the energy absorbed is converted into light with longer wavelengths, in which case a tooth actually becomes a light source. When human dentin is irradiated by 365 nm monochromatic light, fluorescence is revealed with a peak at 440 ± 10 nm [11]. Fluorescence of dental materials was determined as the color difference caused by the inclusion or exclusion

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of the UV component of light with a reflection spectrophotometer [12–14].

The opalescence of resin materials was achieved by the different refractive indices of the resin matrix and the dispersed fillers [6]. Key factors for maximizing the opalescence in resin materials are reported to be: 1) the presence of one or more internal phases of 380–500 nm, 2) a large difference in the refractive indices between matrix and internal phase, and 3) a high dispersion in one or all of the internal phases [2]. On the other hand, the fluorescence of resin materials was achieved by special Flu-pigments rather than by the resin matrix or the fillers. During polymerization, the refractive index of the resin matrix increases while the refractive index of the fillers remains unchanged; therefore, the difference in the refractive indices between the resin matrix and the inorganic fillers, as well as the *OP* and *FL* values of the resin materials, could be changed after polymerization.

The shade groups vary within the brands of indirect and direct resin materials, and the resin matrix and the dispersed fillers are different by the materials. Depending on the material and shade group, the opalescence and fluorescence properties of resin materials may change differently after polymerization, and the values may vary by these groups. The objectives of this study were to measure the changes in opalescence and fluorescence properties of indirect and direct resin materials after polymerization and to determine the influence of material and shade group combination on these properties. We hypothesized that: (1) the amount of change in the opalescence and fluorescence properties of indirect and direct resin materials after polymerization would be the same and (2) the properties of both resin materials measured after polymerization would be the same regardless of the material and shade group.

Material and methods

Preparation of specimen

BelleGlass NG (BG, indirect resin; Kerr, Orange, Calif., USA) and Estelite Sigma (ES, direct resin; Tokuyama, Tokyo, Japan), each composed of 16 shades, were investigated (Tables I and II). Shades of BG were divided into three groups, i.e. enamel (BG-EN), opacious dentin (BG-OD), and translucent (BG-TL). Those of ES were also divided into three shade groups, i.e. basic (ES-BS), additional (ES-AS), and opaque (ES-OP).

Resin material was packed into a white polytetrafluoroethylene mold (12 mm in diameter and 1 mm in thickness) on a polyethylene terephthalate strip. After packing the material, another strip was laid on top of the specimen and pressed with a 49 N load for 3 min to produce a uniform thickness. Both of the

Table I. Shades of BelleGlass NG indirect resin material investigated.

Group	Group code	Shade	Shade code	Batch no.	Composition
Enamel	BG-EN	Cuspal	CP	445488	70 vol.% (77 wt.%) of borosilicate glass (0.4 μm) and silica nanoparticles (50 nm). Aliphatic resin and UDMA
		Gray	GR	440126	
		Light	LI	441289	
		Neutral	NT	445601	
Opacious dentin	BG-OD	A2	A2	443989	72 vol.% (87 wt.%) of barium aluminosilicate glass filler (10 μm, 0.4 μm, and 50 nm). Bis-GMA
		A3	A3	445091	
		A3.5	A3.5	446244	
		B0	B0	417283	
		C2	C2	451119	
		D2	D2	447153	
Translucent	BG-TL	A2	A2	452970	63 vol.% (78 wt.%) of barium aluminosilicate glass filler (10 μm, 0.4 μm and 50 nm). Bis-GMA
		A3	A3	445602	
		A3.5	A3.5	445722	
		B0	B0	416806	
		C2	C2	441024	
		D2	D2	443661	

Kerr, Orange, Calif., USA.
UDMA: Urethane dimethacrylate; Bis-GMA: Bisphenol A glycidyldimethacrylate.

BG and ES specimens were light cured for 40 s in three overlapping areas with a light-polymerization unit (Spectrum 800, Dentsply/Caulk, Milford, Del., USA) at an intensity setting of 400 mW/cm^2. The output of the polymerization light was checked with a radiometer (SDS/Kerr, Orange, Calif., USA). After light polymerization, specimens were released from the mold and both strips were removed. Secondary polymerization of the BG material was performed in the proprietary polymerization chamber (BelleGlass HP Polymerization Unit, Kerr) at 135°C with a pressure of 4.1×10^5 Pa in a nitrogen atmosphere for 20 min. Five specimens were produced for each shade.

Measurement of color

Color was measured according to the CIELAB color scale relative to the standard illuminant D65 on a spectrophotometer (Color-Eye 7000A, GretagMacbeth, New Windsor, N.Y., USA) for calculation of the opalescence (*OP*) and fluorescence parameters (*FL*). For calculation of the *OP*, color of a specimen was measured in the reflectance mode over a zero calibrating cylinder and in the transmittance mode under 100% UV included condition. For calculation of the *FL*, color of a specimen was measured over a white background (reference white standard, instrument serial no. 71060147K, GretagMacbeth; CIE

Table II. Shades of Estelite Sigma resin materials investigated.

Group	Group code	Shade	Shade code	Batch no.	Composition
Basic shade	ES-BS	A1	A1	AE51527S	71 vol.% (82 wt.%) of silica-zirconia filler and composite filler (0.1 to 0.3 μm). Bis-GMA and TEGDMA
		A2	A2	AE43327S	
		A3	A3	AE639Y6S	
		A3.5	A3.5	AE81266S	
Additional shade	ES-AS	Incisal	Inc	AW46047	
		B1	B1	AW65657	
		B2	B2	AW61147S	
		B3	B3	EW703B45S	
		B4	B4	AW502225S	
		C1	C1	AE051316S	
		C2	C2	AW804166S	
		Cervical	Cerv	AE00335S	
Opaque shade	ES-OS	BW	BW	AW55906S	
		OA1	OA1	EW75345S	
		OA2	OA2	AW86457	
		OA3	OA3	AW96257	

Tokuyama, Tokyo, Japan.
Bis-GMA: Bisphenol A glycidyldimethacrylate; TEGDMA: triethylene glycol dimethacrylate.

$L^\star = 94.28$, $a^\star = -0.40$ and $b^\star = 1.34$) in the reflectance mode under 100% UV-included and UV-excluded conditions. Measurements were repeated three times for each specimen.

Calculation of opalescence and fluorescence

The *OP* and *FL* values were calculated before (BEC) and after polymerization with the polymerization light (CWL) for both materials, and after polymerization in the polymerization chamber (CIC) only for BG material. For BG material, the CIC condition was regarded as the polymerized condition, while for ES material the CWL condition was regarded as the polymerized condition. The *OP* value was calculated as: $OP = [(\text{CIE } a_T^\star - \text{CIE } a_R^\star)^2 + (\text{CIE } b_T^\star - \text{CIE } b_R^\star)^2]^{1/2}$, in which subscripts $_T$ and $_R$ indicate transmittance and reflectance mode, respectively [6–8].

The *FL* value was calculated as the difference in color (ΔE^*_{ab}) depending on the inclusion or exclusion of the UV component of illuminant as $FL = [(\text{CIE } L_{100}^\star - \text{CIE } L_0^\star)^2 + (\text{CIE } a_{100}^\star - \text{CIE } a_0^\star)^2 + (\text{CIE } b_{100}^\star - \text{CIE } b_0^\star)^2]^{1/2}$, in which subscripts *100* and *0* indicate the UV 100% included and the UV-excluded condition of the CIE standard illuminant D65, respectively [15].

Statistical analysis

Changes in the *OP* and *FL* values after polymerization (BEC vs CIC for BG: BEC vs CWL for ES) were compared with a paired *t*-test at the significance level of 0.05 (SPSS 12.0; SPSS, Chicago, Ill., USA). One-way analysis of variance (ANOVA) at the significance level of 0.05 was performed for both the *OP* and *FL* values after polymerization (CIC in BG and CWL in ES) by the material and shade group combination (six groups). Means were compared using the Tukey multiple comparison test. Because there was a significant difference in optical properties by the shade group within each material, material and shade group combination, rather than type of resin material such as BG or ES, was used as an independent variable. The six groups compared were BG-EN, BG-OD, and BG-TL in BG material and ES-BS, ES-AS, and ES-OP in ES material.

Results

The *OP* values of BG and ES materials before and after polymerization are presented in Figures 1 and 2. For BG material, the range of the *OP* values was 3.7 to 36.6 before polymerization and 15.0 to 30.3 after polymerization. For ES material, the range of the *OP* values was 23.2 to 28.8 before polymerization and 10.9 to 17.3 after polymerization. Based on a paired *t*-test, the *OP* values of both resin materials changed significantly after polymerization ($p < 0.05$). After polymerization, the mean *OP* value of BG material was greater than that of ES material (19.9 vs 12.4).

The changes in the *OP* values after polymerization of the six groups are presented in Figure 3. After polymerization, there was a significantly greater decrease in the *OP* value of ES material (from 25.6 to 12.4), although BG material showed a small change after polymerization (from 24.3 to 19.9). The mean *OP* value of all the shades of BG material after polymerization (19.9) was greater than that of ES (12.4), and the standard deviation of ES material was relatively smaller than that of BG (3.7 vs 1.7).

The *FL* values of BG and ES materials before and after polymerization are presented in Figures 4 and 5. For BG material, the range of the *FL* values was

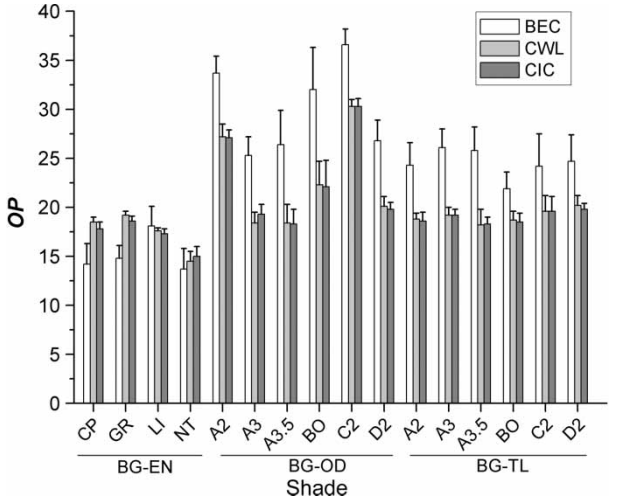

Figure 1. *OP* values before polymerization (BEC), after polymerization with a polymerization light (CWL), and after polymerization in the polymerization chamber (CIC) of the BG material. The vertical bars denote standard deviation.

0.7 to 4.0 before polymerization and 0.5 to 1.8 after polymerization. For ES material, the range of the *FL* values was 0.5 to 4.2 before polymerization and 1.1 to 1.3 after polymerization. Based on a paired *t*-test, the *FL* values of both resin materials changed significantly after polymerization ($p < 0.05$), when the mean *FL* value of BG was smaller than that of ES (0.7 vs 1.2).

The changes in the *FL* values after polymerization of the six groups are presented in Figure 6. After polymerization, there was a significantly greater decrease of the mean *FL* value in BG material (from 2.5 to 0.7), although ES material showed no change after polymerization. The mean *FL* value of all the shades of BG after polymerization (0.7) was smaller than that of ES (1.2), and standard deviation of ES material was relatively smaller than that of BG (0.4 vs 0.1).

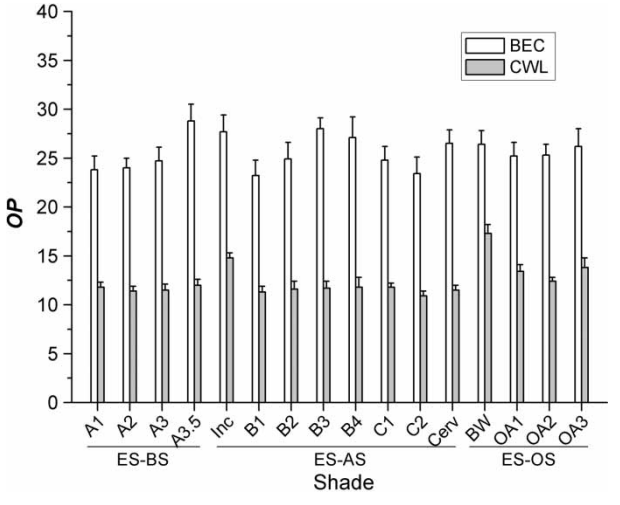

Figure 2. *OP* values before polymerization (BEC) and after polymerization (CWL) of the ES material. The vertical bars denote standard deviation.

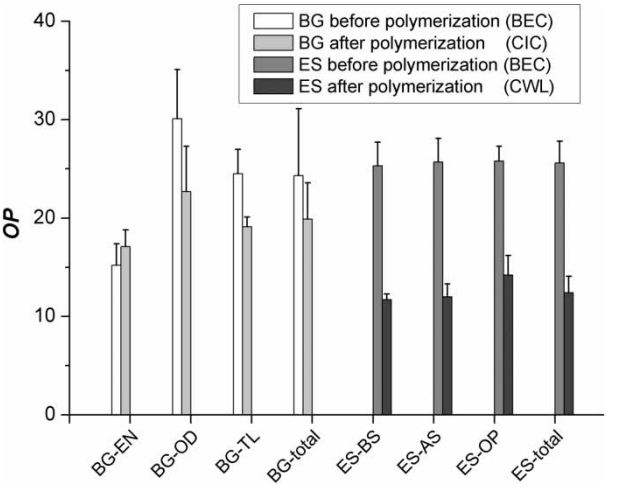

Figure 3. Mean (SD) *OP* values of each shade group and each material.

The *OP* and *FL* values after polymerization of six groups were influenced by the material and shade group combination based on one-way ANOVA ($p < 0.05$). Based on the Tukey multiple comparison test, the following homogenous subsets were observed for the *OP* value: ES–BS (mean: 11.7) = ES–AS (mean: 11.9)/ES–OP (mean: 14.2)/BG–EN (mean: 17.1)/BG–TL (mean: 19.1)/BG–OD (mean: 22.7) ($p < 0.05$). Those for the *FL* value were: BG–TL (mean: 0.60) = BG–EN (mean: 0.75) < BG–EN (mean: 0.75) = BG–OD (mean: 0.84) < ES-OP (mean: 1.14) = ES–AS (mean: 1.19) < ES–BS (mean: 1.21) ($p < 0.05$).

All shades of BG material showed fluorescence peak before polymerization, but none showed fluorescence peak after polymerization. As an example, fluorescence emission curves [ΔSpectral reflectance (UV 100 – UV 0); arbitrary unit] for the BG-TL group materials before polymerization are presented

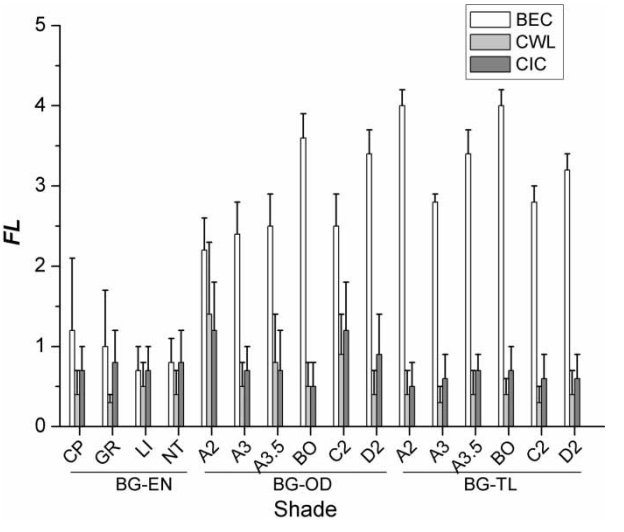

Figure 4. *FL* values before polymerization (BEC), after polymerization with a polymerization light (CWL), and after polymerization in the polymerization chamber (CIC) of the BG material. The vertical bars denote standard deviation.

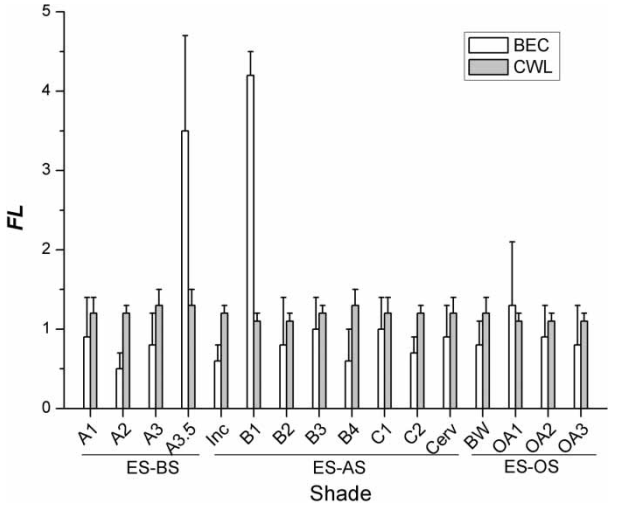

Figure 5. *FL* values before polymerization (BEC) and after polymerization (CWL) of ES material. The vertical bars denote standard deviation.

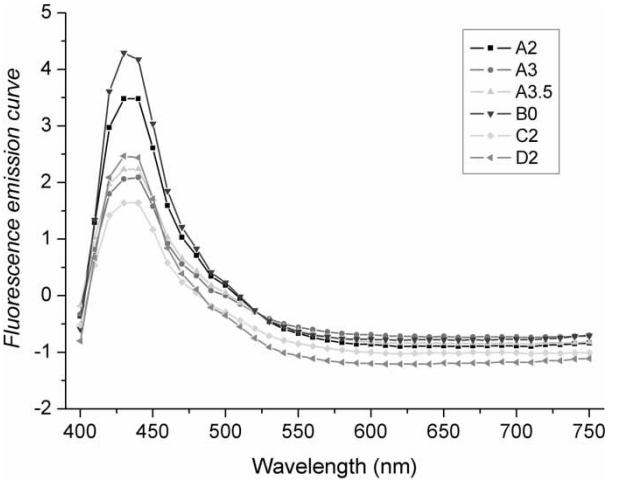

Figure 7. Fluorescent emission curve for the BG-TL shade group materials before polymerization.

in Figure 7. These curves indicate the differences in the reflectance by the inclusion or exclusion of the UV component of the standard illuminant D65 as a function of wavelength. All shades in the BG-TL group showed peaks around 450 nm in the curves. As an example of changes in the fluorescent emission curve of BG material, the fluorescence emission curves for the A2 shade are presented in Figure 8 indicating that there was a fluorescence peak around 450 nm before polymerization which disappeared after polymerization.

Discussion

Indirect resin systems were developed to overcome the negative attributes of their porcelain counterparts and to simplify fabrication, insertion, and post-delivery adjustments [16]. BelleGlass NG, as a widely used indirect resin, was reported to exhibit higher values for both hardness and degree of polymerization than packable resin materials [17]. Just like BelleGlass NG, the direct resin Estelite Sigma is also composed of three shade groups by the application area. Therefore, BelleGlass NG and Estelite Sigma were investigated in the present study as indirect and direct resin materials to compare their opalescence and fluorescence properties.

Opalescence and fluorescence properties of dental materials could be determined quantitatively with the reflection spectrophotometer [6–8,12–14]. The *OP* values of human and bovine enamel were reported in a previous study [8]; however, there were no identified *FL* values of nature tooth based on the protocol of the present study. Compared with the *OP* of human enamel (mean *OP* value $=22.9\pm 1.9$) [8], only the BG–OD group materials showed a competitive *OP* value ($=22.7$) to that of the human enamel. Other shade groups of BG material and all shades of ES material showed lower *OP* values than tooth enamel. However, it has to be considered that

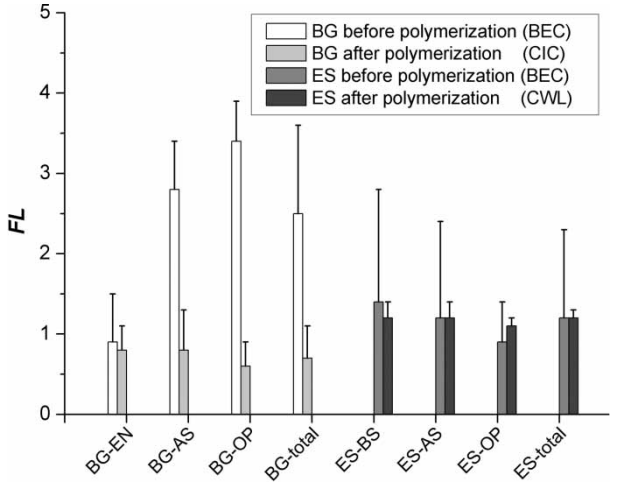

Figure 6. Mean (SD) *FL* values of each shade group and each material.

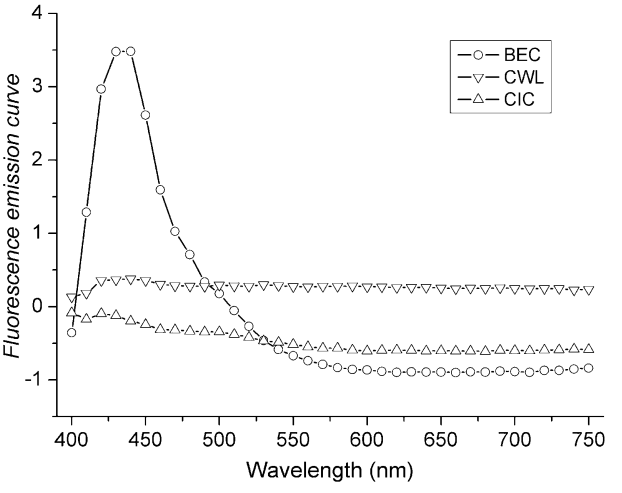

Figure 8. Fluorescent emission curve for the BG-TL A2 shade material before polymerization (BEC), after polymerization with light (CWL), and after second polymerization in the polymerization chamber (CIC).

the thickness of specimens varied by the teeth (0.9–1.3 mm) in the study on the *OP* values of human enamel, which might have influenced the *OP* values of tooth enamel and also might have contributed the differences in the *OP* values compared with the values of the present study.

Since the resin matrix and the dispersed fillers are different in the two resin materials (Tables I and II), the extent of changes in opalescence was different in the two resin materials. A decrease in the *OP* values was observed in both the resin materials investigated, and a smaller amount of decrease was observed in BG material. On the other hand, the distributions of filler size were 2500 nm, 400 nm, and 50 nm in BG material and 100–300 nm in ES material. Therefore, BG material fits previously cited opalescent conditions more precisely than ES material [2]. This may partly explain the higher opalescence in BG material than in ES material. In this regard, the addition of proper sized and well-dispersed fillers could increase the opalescence properties of this material.

The fluorescence phenomenon makes a definite contribution to the brightness and vital appearance of a human tooth [1]. In the present study, the *FL* values before and after polymerization in BG and ES materials were different ($p < 0.05$), and the ES material was more fluorescent than the BG material after polymerization. The reason could be the different composition of the two resin materials. Since the refractive index of the resin matrix changes during the polymerization process [18,19], fluorescence will also change differently because the uncured and cured resin matrices could influence the Flu-pigments differently.

Although the *FL* values in the present study were lower than the generally considered clinically perceptible limit of 3.7 [20], the characteristics of fluorescent emission under visible light condition may be different from those under another light. However, it is generally regarded that the fluorescence of a tooth contributes importantly to its appearance, even though it is not as apparent in daylight [21]. Moreover, the condition of the present study was normal daylight (D65), not intense UV-illumination, so this difference in color can be emphasized under intense black (UV) light. However, the influence of the kind of UV component, such as daylight or black light, on the fluorescent emission has not been studied so far. Therefore, perceptibility of fluorescent emission under varied illumination conditions should be studied further [13].

The polymerization process of BG material combines two different curing systems. The material is initially cured by a conventional curing light, and then in a proprietary oven at a temperature of 135°C and pressure of 4.1×10^5 Pa in a nitrogen atmosphere for 20 min. The elevated temperature and nitrogen gas increase the polymer conversion, and the pressure allows the oxygen to be purged out, which results in a higher conversion rate of the material. As for the investigated BG material in the present study, both of the changes in *OP* and *FL* values during the primary polymerization cycle were generally higher than those during the secondary polymerization cycle, and the fluorescence peaks disappeared in all the shades after primary polymerization, which might have been due to the different objectives of the two polymerization systems. Primary polymerization leads to the dominant polymer conversion and corresponding large changes in *OP* and *FL* values, while the main purpose of the secondary polymerization was to make the conversion rate even higher, thus to increase the mechanical properties of the material. The reason for disappearance of the fluorescence peaks after polymerization is unclear; however, it could have been due to the different influences of the uncured and the cured resin matrices on the Flu-pigments.

Considering these results, the first hypothesis, i.e. that the amount of changes in the opalescence and fluorescence properties of indirect and direct resin materials after polymerization would be the same, was rejected because BG showed smaller changes in *OP* but bigger changes in *FL* after polymerization than ES ($p < 0.05$). The second hypothesis, i.e. that these properties of both resin materials measured after polymerization would be the same regardless of the material and shade group, was also rejected because both of the *OP* and *FL* values were significantly influenced by the combination of material and shade group of the materials.

There are several limitations in the present study. First, an ANOVA may not have been the ideal statistical method for this study because of the identified differences in standard deviations. Both of the *OP* and *FL* values of ES material showed relatively smaller standard deviations than those of BG; however, an ANOVA assumes that the different groups have the same standard deviations. This fact should be considered when interpreting the results in the present study. Second, the influence of water sorption and/or aging on the *OP* and *FL* values was not evaluated in the present study, although it is reported that the opalescence and fluorescence properties of resin materials were influenced by these treatments [22]. Furthermore, the *OP* and *FL* values might be influenced by the thickness of the specimens, and the thickness of restored resin materials varied by patient and tooth, which should be further studied.

Within the limitations of the present study, the opalescence and fluorescence properties changed significantly during the polymerization procedure in both of the indirect and direct resin materials ($p < 0.05$), and these properties of the materials vary by the brand. Therefore, these properties should be

considered in clinic when neighboring teeth are treated with different types of resin material.

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