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Uv–vis spectrophotometric direct transmittance analysis of composite resins

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ABSTRACT

Objectives. The objective of this experiment was to evaluate the direct transmittance of the micro-hybrid composite resins Charisma F, Solitaire II, Intens and Tetric Ceram, and the nanofilled composite resins Exthet-X and Filtek Supreme in the dentin and translucent shades.

Methods. Three samples of each composite resin and shade were obtained with dimensions of 12 mm × 12 mm and 1 mm thick. After light activation, three measurements for each sample were done with an UV–vis spectrophotometer. The spectra was analyzed by mean values for the percentage of direct transmittance at intervals of 20 nm in the range between 400 and 700 nm (%T) and the percentage of direct transmittance in the wavelengths of 400, 560 and 700 nm (T_d).

Results. The Student's *t*-test showed that dentin shade of Charisma F had significant higher %*T* than the translucent shade. For Solitaire II, Intens, Tetric Ceram and Esthet-X the translucent shade had higher %*T* than the dentin shade. For Filtek Supreme there was no statistical difference between the dentin and translucent shades. According to analysis of variance (ANOVA) and Tukey multiple-range test Charisma F had the highest %*T* and T_d at dentin and translucent shades, except for translucent shade at 700 nm, in which Esthet-X had higher T_d.

Significance. This study indicates that the percentage of direct transmittance is not directly related with the composite resin shade.

1. Introduction

Among the various restorative materials, composite resins have been widely used and have evolved a great deal, both with regard to composition and processing technology. Proof of this is the appearance of various composite resins for the direct as well as the indirect technique.

To obtain an esthetic restoration, it is important for the composite resin to be supplied with different matrixes, shades and values in order to associate different colors and thus faithfully reproduce the esthetic characteristics of the tooth.

Watts and Cash [1] demonstrated that the composition of the material is a factor determining its optical properties, so that the reflection index differs among composite

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Table 1 – Information about the research materials

Material	Shade	Classification[a]	Batch number	Manufacturer
Charisma F	A3	Micro-hybrid	023	Heraeus Kulzer, Hanau, Germany
Charisma F	Translucent	Micro-hybrid	030	
Solitaire II	A3	Micro-hybrid condensable	050499	
Solitaire II	Translucent	Micro-hybrid condensable	010224	
Esthet-X	A3	Nanofiller	01111000983	Dentsply De Trey GmbH, Germany
Esthet-X	YE	Nanofiller	0111000078	
Inten-S	A3	Micro-hybrid	D5865	Ivoclar/Vivadent, Liechtenstein
Inten-S	Incisal	Micro-hybrid	E53352	
Tetric Ceram	A3	Micro-hybrid	900513	
Tetric Ceram	Translucent	Micro-hybrid	901232	
Filtek Supreme	A3B	Nanofiller	030998	3M ESPE, St. Paul, MN, USA
Filtek Supreme	YT	Nanofiller	199602	

[a] Manufacture's information.

resins, ceramics and glass ionomer cements. For the composite resins, filler composition and filler content, as well as the shape and size of the filler are some of the factors responsible for the optical dispersion of the material [2].

In view of the importance of esthetics, various studies have assessed the optical properties of restorative materials, either using direct and total transmittance methodologies [3,4], spectrophotometry by reflectance [5], as well as the contrast index on black and white background [6,7], for the purpose of assessing material translucency and color.

Translucency is one of the various factors that determine the optical characteristics of the material and refers to the partial passage of light through a certain structure. This is an important feature of restorative materials, since the tooth allows the partial passage of light through its tissues, and may also present with different degrees of translucency, depending on the anatomic region. Therefore, the presence of different degrees of translucency in composite resins is a determining factor in the quality of esthetic reproduction of lost portions of teeth.

The objective of this study was to evaluate the direct transmittance percentage of micro-hybrid and nanofiller composite resins in the A3 and translucent shades, or similar. This research was conducted under two hypotheses: translucent composite resins have higher direct transmittance percentage than ones similar to them in the A3 shade; composite resins with different compositions have different direct transmittance percentages.

2. Materials and methods

The materials used in this study are listed in Table 1, together with shade, classification according the filler, batch no. and manufacturer. The composite resins in the A3 shade of the Vita scale, or similar, were considered as the dentin shade. Whereas the composite resins of the incisal, enamel or translucent type were considered as being of the translucent shade. The information about the filler and organic matrix (Tables 2 and 3) were obtained from the manufacturers or, when not available, from the literature [8,9].

Table 2 – Composition of the inorganic phase of the composite resins

Material	Shade	Percentage of filler (weight)	Size	Composition
Charisma F	Dentin Translucent	75[a]	0.02–2 μm (0.7)[b]	AlF, Ba, SiO_2[b]
Esthet-X	Dentin Translucent	77[a]	10 nm–0.8 μm[b]	SiO_2, Ba–Al–F–Si[b]
Intens	Dentin Translucent	82.2[a]	0.24–1.0 μm[a,b]	Ba, YbF_3[b]
Solitaire II	Dentin Translucent	75[a]	0.7–25 μm[b]	SiO_2, Ba, Al, B–Si[b]
Filtek Supreme	Dentin	78.5[a]	5–20 nm[a]	SiO_2, Zr–SiO_2[a]
Filtek Supreme	Translucent	72.5[a]	75 nm[a]	SiO_2[a]
Tetric Ceram	Dentin Translucent	78.6[a]	0.04–3 μm (0.7)[b]	SiO_2, Ba–Al–F, YbF_3[b]

[a] Manufacture's information.
[b] Sabbagh et al. [8].

Table 3 – Composition of the organic phase of the composite resins

Material	Shade	Composition
Charisma F	Dentin Translucent	Bis-GMA, TEGDMA[a]
Esthet-X	Dentin Translucent	NA
Intens	Dentin Translucent	Bis-GMA, UDMA, TEGDMA[b]
Solitaire II	Dentin Translucent	Polimatrix[b]
Filtek Supreme	Dentin Translucent	Bis-GMA, Bis-EMA, UDMA, TEGDMA[b]
Tetric Ceram	Dentin Translucent	Bis-GMA, UDMA, TEGDMA[b]

NA: not available.
[a] Zantner et al. [9].
[b] Manufacturer's information.

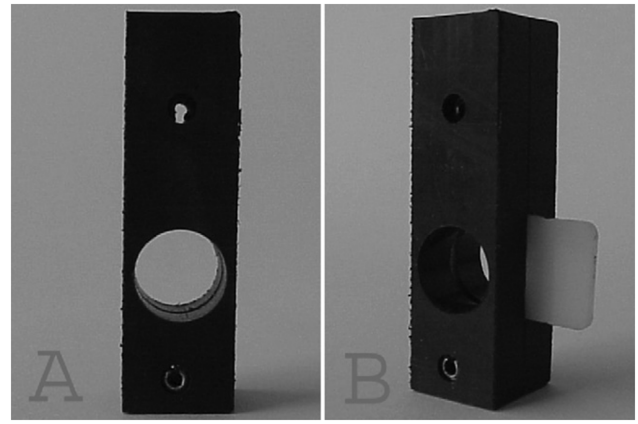

Fig. 1 – Teflon device: (A) anterior view and (B) lateral view. There is a specimen being positioned into the groove on the lateral face.

The specimens in composite resin were obtained in a metal matrix with internal dimensions of 12 mm × 12 mm and height of 1.0 mm. The matrix was placed on a glass plate and a first increment of composite resin approximately 0.5 mm thick was put inside it. The four angles and the center of this increment were light-cured for 10 s each using the XL 1500 (3M Co., St. Paul, MN, USA) visible-light-curing unit, totaling 50 s. The light intensity was controlled by the radiometer Demetron 100 (Demetron/Kerr, Danbury, CT, USA), at a minimum range of 400 mW/cm^2. An additional layer of composite resin was placed over the first layer and a second glass plate was placed over the matrix, followed by compression to run off the excess composite resin into the escape areas. After light-curing for 50 s at the same points, the top glass plate was removed and the test specimen removed from the matrix. From this procedure, the test specimens were manipulated with the use of rubber gloves, with the purpose of preventing them from being contaminated.

The specimen thickness was gauged with a digital caliper with a precision of 0.01 mm (Mitutoyo Sul Americana Ltd., Suzano, SP, Brazil) on four central axes. The maximum difference accepted was standardized at ±0.05 mm, and the specimens were stored dry at a light-proof receptacle. Three specimens were obtained for each corresponding shade of each composite resin [5,10,11,12].

Each specimen was placed into a black teflon device 42 mm high, 11.4 mm deep, 12 mm wide, with an orifice of 10 mm diameter located at 9.3 mm from the base (Fig. 1). On the lateral face of the device there was a 1.15 mm thick groove into which the test specimen was fitted. The Teflon device with the specimen was positioned in the UV–vis spectrophotometer HP 8453 (Hewlett-Packard, Paloalto, CA, USA) with diode arrangement for the direct transmittance test at a wavelength between 400 and 700 nm. Three consecutive readings were taken for each specimen [10,13,14], for a time not exceeding 5 s, with the angle of incidence and reading at 0°/0° [15] and interval of 1 nm. The equipment conditions of use include a filament heating time of not less than 5 min, temperature of 22 ± 2 °C and relative humidity in the range of 50 ± 5%.

The numerical values (percentages) of direct transmittance of the material in the range of 400–700 nm were initially stored in the form of a file in the equipment software – HP ChemStation/HP89552 K – and were afterwards transfered to the program Microsoft Excel at intervals of 20 nm for quantitative analysis of the resulting spectra [14,16–18]. The quantitative analysis of the results corresponded to the mean of the percentage values with intervals of 20 nm in the range between 400 and 700 nm. The percentage obtained was considered to be the direct transmittance for the entire spectrum studied (%T), for each specimen, in each reading. In addition, the direct transmittance (T_d) of three arbitrary points was determined in the spectra obtained, at the wavelengths of 400, 560 and 700 nm. These were chosen because they represented the extreme and intermediate points for quantitative analysis of the variations found [13,15].

Student's t-test was applied for comparison of %T between the dentin and translucent shades of each composite resin. Analysis of variance (ANOVA) combined with a Tukey multiple-range test were performed to compare %T and T_d at the wavelengths of 400, 560 and 700 nm of all composite resins at dentin and translucent shades. The statistical software SPSS version 10.0 was used to process and analyze these data.

3. Results

The Student's t-test revealed that the translucent shade had statistically higher %T than the dentin shade for Esthe-X, Intens, Solitaire II and Tetric Ceram. For the Filtek Supreme there was no statistical difference between the dentin and translucent shades, and for Charisma F the dentin shade had statistically higher %T than translucent shade (Table 4).

By the ANOVA, significant differences were found among the composite resins for %T and T_d in the three wavelengths when compared the composite resins in the dentin or translu-

Table 4 – Direct transmittance for the entire spectrum (%T) between the shades for each composite resin

Material and shade	n	Mean (S.D.)	P-value
Charisma F			
Dentin	9	3.706 (0.366)	0.01[a]
Translucent	9	1.972 (0.110)	
Esthe-X			
Dentin	9	0.449 (0.045)	0.01[a]
Translucent	9	1.556 (0.177)	
Intens			
Dentin	9	0.085 (0.017)	0.01[a]
Translucent	9	0.276 (0.022)	
Solitaire II			
Dentin	9	0.021 (0.003)	0.01[a]
Translucent	9	0.035 (0.013)	
Filtek Supreme			
Dentin	9	0.335 (0.038)	0.63
Translucent	9	0.324 (0.050)	
Tetric Ceram			
Dentin	9	0.071 (0.001)	0.01[a]
Translucent	9	0.079 (0.006)	

[a] $P \le 0.01$.

Table 5 – Direct transmittance for the entire spectrum (%T) and the direct transmittance at the wavelengths of 400, 560 and 700 nm for dentin shade (T_d)

Resina	n	Mean (S.D.)	P-value
%T			
Charisma F	9	3.706^{A} (0.366)	0.01[a]
Esthet-X	9	0.449^{B} (0.045)	
Intens	9	0.085^{C} (0.017)	
Solitaire II	9	0.021^{C} (0.003)	
Filtek Supreme	9	0.335^{B} (0.038)	
Tetric Ceram	9	0.071^{C} (0.001)	
T_d (400 nm)			
Charisma F	9	0.075^{A} (0.017)	0.01[a]
Esthet-X	9	$0.014^{B,C}$ (0.006)	
Intens	9	0.012^{C} (0.005)	
Solitaire II	9	0.011^{C} (0.001)	
Filtek Supreme	9	0.024^{B} (0.011)	
Tetric Ceram	9	0.010^{C} (0.000)	
T_d (560 nm)			
Charisma F	9	3.592^{A} (0.361)	0.01[a]
Esthet-X	9	0.169^{B} (0.024)	
Intens	9	0.042^{B} (0.010)	
Solitaire II	9	0.022^{B} (0.006)	
Filtek Supreme	9	0.213^{B} (0.022)	
Tetric Ceram	9	0.052^{B} (0.004)	
T_d (700 nm)			
Charisma F	9	8.709^{A} (0.782)	0.01[a]
Esthet-X	9	1.892^{B} (0.180)	
Intens	9	0.272^{D} (0.019)	
Solitaire II	9	0.022^{D} (0.013)	
Filtek Supreme	9	1.052^{C} (0.130)	
Tetric Ceram	9	0.178^{D} (0.001)	

No significant differences were observed between the composite resins if the value of direct transmittance is quoted with the same character.

[a] $P \le 0.01$.

cent shades. According to Tukey's multiple-range test the highest %T and T_d occurred for Charisma F, being statistically higher than the others at both shades (Tables 5 and 6). The exception was the wavelength of 700 nm in the translucent shade, in which the composite resin Esthet-X had the highest T_d. For the dentin shade, only at the wavelength of 560 nm there was no statistical difference in the T_d among the composite resins Esthet-X, Intens, Solitaire II, Filtek Supreme and Tetric Ceram.

4. Discussion

Watts and Cash [1] observed that many of the esthetic dental biomaterials may be approximately modeled by a composite structure of discrete spherical particles embedded in a matrix. For Rayleigh, scattering of light, the reduction of light intensity within the medium is given by Eq. (1), in which d is the optical path length (thickness), V_p the volume fraction of particles, r the particle radius, n_p the refractive indices of the particles, n_m the refractive indices of the matrix and λ is the wavelength of light.

$$T = \exp -2303d\left[\frac{3V_p r^3 (n_p/n_m - 1)}{4\lambda^4}\right] \quad (1)$$

It is important to point out that in this equation, the values are exponential and by the negative logarithm, thus one has one behavior when the refraction index of the matrix is greater than that of the inorganic load, and the converse if this situation is inverted. It may therefore be deduced that: (a) if the refraction index of the matrix is smaller than that of the particle, a reduction of transmittance will occur with the increase of r and V_p, and an increase in transmittance will occur with the increase of λ; (b) if the refraction index of the particle is smaller than that of the matrix, an increase in transmittance will occur with the increase of r and V_p, and an reduction of transmittance will occur with the increase of λ. In addition to this, the effect of each of the factors is differentiated, since r is raised to the third power, λ to the fourth power and V has a multiplier factor of 3. However, the application of any theoretical model follows a series of laws that are generalizations, which allow the formulation of an equation such as the one presented.

Composite resins are traditionally formulated with components, such as silica (refractive index of 1.463); Bis-GMA (refractive index of 1.551); TEGDMA (refractive index of 1.460) and UDMA (refractive index of 1.484) [19] in various combinations with other monomers, and it is difficult to quantify each separate factor exactly. Furthermore, a turbid medium does not follow any formula or equation in a straight line [16]. The difference between the refractive indices of the organic matrix and the filler influence the properties of composite resins, since the adjustment of the monomer or the filler refraction indices resulted in better properties of transmittance and polymerization depth [19,20]. This may be one of the reasons that lead to the Charisma F obtaining higher percentage of direct transmittance, irrespective of the shade, at

Table 6 – Direct transmittance for the entire spectrum (%T) and the direct transmittance at the wavelengths of 400, 560 and 700 nm for Translucent shade (T_d)

Material	n	Mean	P-value
%T			
Charisma F	9	1.972[A] (0.110)	0.01[a]
Esthet-X	9	1.556[B] (0.177)	
Intens	9	0.276[C] (0.022)	
Solitaire II	9	0.035[D] (0.013)	
Filtek Supreme	9	0.324[C] (0.050)	
Tetric Ceram	9	0.079[D] (0.006)	
T_d (400 nm)			
Charisma F	9	0.021[A] (0.005)	0.01[a]
Esthet-X	9	0.010[C] (0.001)	
Intens	9	0.014[B] (0.004)	
Solitaire II	9	0.010[C] (0.000)	
Filtek Supreme	9	0.010[C] (0.001)	
Tetric Ceram	9	0.011[C] (0.001)	
T_d (560 nm)			
Charisma F	9	1.747[A] (0.110)	0.01[a]
Esthet-X	9	0.721[B] (0.096)	
Intens	9	0.159[C] (0.012)	
Solitaire II	9	0.030[D] (0.011)	
Filtek Supreme	9	0.137[C] (0.022)	
Tetric Ceram	9	0.062[D] (0.004)	
T_d (700 nm)			
Charisma F	9	4.982[B] (0.228)	0.01[a]
Esthet-X	9	5.969[A] (0.614)	
Intens	9	0.868[D] (0.039)	
Solitaire II	9	0.031[E] (0.009)	
Filtek Supreme	9	1.341[C] (0.203)	
Tetric Ceram	9	0.178[E] (0.023)	

No significant differences were observed between the composite resins if the value of direct transmittance is quoted with the same characters.

[a] $P \leq 0.01$.

practically all of the wavelengths. The exception occurred only for the wavelength of 700 nm, in which the direct transmittance of the translucent shade was lower than that of Esthet-X, but higher than the others (Table 6; Fig. 3). As the composition of the filler is similar for all the studied materials (Ba, SiO_2), this hypothesis is based on the fact that the Charisma F has the monomers Bis-GMA and TEGDMA in its organic matrix (refractive indices of 1.463 and 1.460, respectively), while the other materials additionally have the monomer UDMA, with a refractive index of 1.484 [19]. In a composite resin with a load of Barium and Silica (Ba and SiO_2), combined with a matrix of Bis-GMA/TEGDMA or UDMA/TEGDMA, the great difference in polymerization depth (50% greater) found for the Bis-GMA/TEGDMA system was associated with the lower dispersion of light as a result of the refractive indices being closer in the Bis-GMA/TEGDMA system [21]. Therefore, the importance of the refractive indices with regard to the direct transmittance percentages found, must not be underestimated.

Another important factor is the particle size. In accordance with the Rayleigh equation, the particle size must have a great influence, as it was indicated that it would cause a decrease in transmittance with the increase of the radius (dimensions). Powers et al. [22] found less dispersion and opacity for a micro-filled composite resin in comparison with another that had macrofilled. Yeh et al. [23] also found that the optical thickness (which refers to opacity) and the scattering coefficient of a macrofilled composite resin was greater than that of a micro-filled one, but this effect was dependent on the wavelength in question. At shorter wavelengths, the micro-filled composite resin caused greater scattering of the light, while the macrofilled composite resin caused this effect at longer wavelengths. Thus, it is noted that the scattering capacity is dependant on the particle size and on the wavelength. This effect was demonstrated by Yearn [24], whose study indicated that the particles disperse more intensely as the dimensions get closer to the half of the wavelength. This effect may be observed in the Filtek Supreme. At the wavelengths of 400 and 560 nm, the dentin shade (Table 5) demonstrated higher direct transmittance percentages in comparison with the translucent shade (Table 6), and this effect may be credited to the smaller particle size of the dentin shade (5–20 nm) when compared with the translucent shade (75 nm) (Table 2). Whereas at 700 nm, the direct transmittance percentage was greater for the translucent shade, and could once again suggest that this increase in the direct transmittance percentage was caused by the phenomenon described above. However, one must be cautious about generalizing the importance of this factor. If one takes into account the results found in the comparisons of direct transmittance percentages for the composite resins of the dentin shade (Table 5), one finds that the materials with a distribution of particles of very differentiated sizes have the same behavior at the wavelengths of 400 and 560 nm (Fig. 2). This finding was also present at the wavelength of 400 nm for the composite resins of the translucent shade (Table 6; Fig. 3), but in a less pronounced way.

When analyzing only the direct transmittance percentage for the entire spectrum (%T), the particle size enables the materials to be grouped into certain categories for the composite resins of the dentin shade. Those of the nanofilled (Esthet-X and Filtek Supreme) and micro-hybrid (Tetric Ceram, Intens and Solitaire II) types formed two groups with similar characteristics, with the Charisma F being the exception (Table 5;

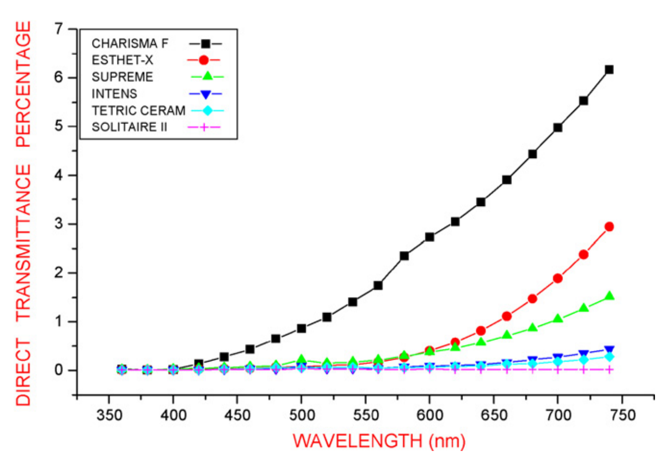

Fig. 2 – Light transmittance spectral distributions of dentin shades of composite resins.

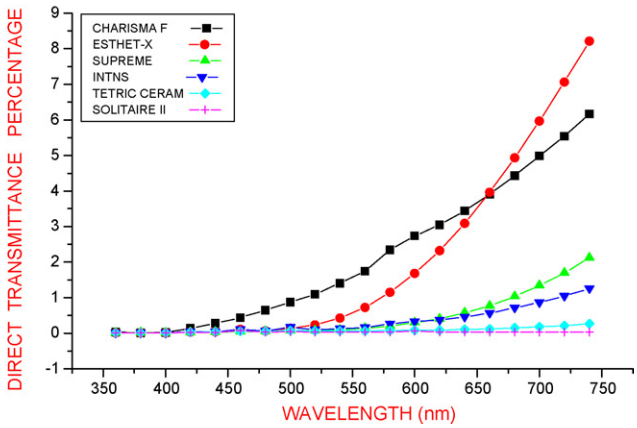

Fig. 3 – Light transmittance spectral distribution of translucent shades of composite resins.

Fig. 2). Whereas for the composite resins of the translucent shade, the particle distribution of the load does not appear to be a factor that allows the materials to be grouped (Table 6; Fig. 3).

The relation between the factors referring to material composition may have a great influence on the results of incident light transmittance, its effect being somewhat ambiguous, up to a certain point.

It is important to emphasize that the information given up until now can be analyzed from the practical point of view, in spite of the caution necessary when transposing it to clinical dentistry. An example of this is the behavior of incident light blockage shown by the composite resin Solitaire II (Figs. 2 and 3).

Therefore, from the results obtained in this study, the initially formulated hypothesis with regard to the higher percentages of direct transmittance for the translucent shade in comparison with the dentin shade could not be proved for all the composite resins. However, the second hypothesis was proved, as the composition of the material did influence the direct transmittance percentages.

Thus it is suggested that further studies be conducted to investigate the relation between direct and total transmittance and the modulator factors of this relation, with the present studying serving only as a starting point for this investigation.

5. Conclusions

The composite resins Exthet-X, Intens, Solitaire II and Tetric Ceram obtained higher direct transmittance in the translucent shade in comparison to the dentin shade. The composite resin Filtek Supreme had similar direct transmittance for dentin and translucent shades. The composite resin Charisma F had higher direct transmittance for the dentin shade in comparison to the translucent shade.

The composition of the composite resin influenced the direct transmittance of the material, and the direct transmittance is not directly related with the composite resin shade.

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