



## Influence of different shades on light transmission through different composites

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### Abstract

**Objective:** This study evaluated the influence of different composite resins shades on light transmission through the composite and the difference in percentage of the decrease in Knoop hardness between bottom and top (DKH).

**Methods:** Two restorative composites with shades A1 and A3.5 were used (Esthet X and Filtek Z350). The irradiance of the light curing unit was measured (780 mW/cm<sup>2</sup>). Twenty-five specimens were made for each shade for both composites. Light transmission test: The irradiance of light that passed through the composite was measured (n=10). Knoop Hardness test: The DKH of the same specimen was calculated (n=10). Scanning Electron Microscopy (SEM): Five specimens from either Filtek Z350 or Esthet-X composites were observed under SEM.

**Results:** The irradiance of light that passed through shade A1 composite was statistically higher than shade A3.5 for both composites (p=0.00001). For Esthet X, the DKH of shade A3.5 was statistically higher than for shade A1; however, there was no statistically significant difference for Filtek Z350 (p=0.03035). Representative areas showing the filler particles were photographed at 1,000x magnification.

**Conclusion:** Different composites influenced both light transmission and DKH. Darker shades allowed lower light transmission, but could not establish a relation between dark shades and DKH.

**Key words:** Composites; Light curing unit; Knoop hardness, Shade

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### Influência de diferentes cores na transmissão da luz através de diferentes compósitos

#### Resumo

**Objetivo:** Foi avaliar a influência de diferentes cores de resinas compostas na transmissão de luz através do compósito e a diferença percentual de diminuição da dureza Knoop entre a base e o topo (DDBT).

**Métodos:** Dois compósitos restauradores foram utilizados nas cores A1 e A3,5 (Filtek Z350 e Esthet X). A irradiância da unidade foto-ativadora foi mensurada (780 mW/cm<sup>2</sup>). Vinte e cinco espécimes foram confeccionados para ambos compósitos nas diferentes cores. Ensaio de transmissão da luz: A irradiância que passou através dos compósitos foi mensurada (n=10). Ensaio de Dureza Knoop: A DDBT do mesmo espécime foi calculada (n=10). Microscópio Eletrônico de Varredura (MEV): Cinco espécimes para cada compósito (Filtek Z350 ou Esthet-X) foram observados em MEV.

**Resultados:** A irradiância que passou através do compósito cor A1 foi estatisticamente superior à cor A3,5 para ambos os compósitos (p=0,00001). Para Esthet X, DDBT da cor A3,5 foi estatisticamente superior à cor A1; entretanto, não houve diferença estatística para Filtek Z350 (p=0,03035). Áreas representativas das partículas de cargas foram observadas com 1.000x de ampliação.

**Conclusão:** Diferentes compósitos influenciaram a transmissão de luz e DDBT. Cores escuras permitiram menor transmissão de luz, mas não foi possível estabelecer uma relação entre cores escuras e DDBT.

**Palavras-chave:** Compósitos; Unidade foto-ativadora; Dureza Knoop; Cor

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## Introduction

Dental resin composites are constituted of a three-dimensional combination of at least two chemically different materials, with a different interface separating the components. Thus, they are composed of an organic matrix, fillers (glass, quartz and/or melted silica) and a bonding agent (organic silane), enabling chemical bonding with the load particles and co-polymerization with the monomers of the organic matrix [1].

There is some theoretical and experimental evidence to support the role of filler particle-related features in the wear resistance, mechanical properties and extent of polymerization of particulate resin composites. Consequently, research efforts have been targeted at refining the microstructure of composites in terms of the arrangement, size, geometry, and volume fraction of particles [2]. The translucency of esthetic restorative materials has usually been determined by using the translucency parameter, which refers to the differences in color within a uniform thickness of a specimen observed over a white and a black background and which corresponds directly to the common visual assessment of translucency [3].

The degree of cure of visible light-activated dental resin composites was recognized as important to the clinical success of these materials soon after these materials were introduced [4]. The advantage of testing the composite by a Knoop hardness test is in the correlation between the Knoop hardness and the degree of monomer conversion (DC) [5]. DC directly influences mechanical properties of the dental resin composite [5]. Thus, after light curing, it is desirable to activate this restorative material, in order to attain the best mechanical properties, and to convert all of its monomer

into polymer. To define depth of polymerization based on top and bottom layer hardness measurements, it is common to calculate the ratio of bottom/top hardness and give this ratio a threshold value considered to represent the bottom surface as adequately cured. Values of 0.80 and 0.85 have often been used [6].

The aim of this study was to investigate the influence of different composite resins with different shades on the relation with light transmission through the composite and with the resulting Knoop hardness. The authors hypothesized that light transmission through different composite resins with different shades should influence the percentage of decrease in Knoop hardness between bottom and top (DKH).

## Methods

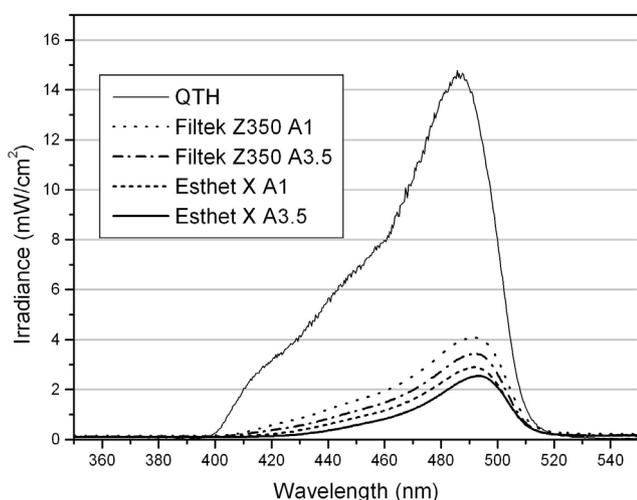
Two restorative composite resins of shades A1 and A3.5 were used in this study (Figure 1): Esthet X (Dentsply-Caulk – Milford, DE, USA) and Filtek Z350 (3M-ESPE – St. Paul, MN, USA).

Among the photo-activation units available on the market, the most traditional use a quartz tungsten halogen light [7-10]. For this reason, an XL 2500 (3M-ESPE) quartz tungsten halogen light curing unit was used, with an irradiance of 780 mw/cm<sup>2</sup>. The light curing unit (LCU) power (mW) was measured using a power meter (Ophir Optronics Inc, Danvers, MA, USA). The tip diameter was measured with a digital caliper (model CD-15C, Mitutoyo, Kanagawa, Japan) to determine the tip area. The irradiance was calculated by dividing the light power by the tip area. The spectral distributions (Figure 2) were obtained using a spectrometer (USB 2000, Ocean Optics – Dunedin, FL, USA).

Composite	Manufacturer	Organic Matrix	Filler	Shade	Batch number
Esthet X	Dentsply-Caulk, Milford, USA	BIS-GMA and TEGDMA	60% in volume; (0.6 to 0.8 $\mu$ and 0.02 to 2.5 $\mu$ - glass powder (BAFG) 10 to 20 nm - silica dioxide)	A1	061218
				A3.5	0705013
Filtek Z350	3M-ESPE, St. Paul, USA	BIS-GMA, BISEMA,UDMA and TEGDMA	59.5% in volume (clusters of 0.6 to 1.4 $\mu$ - particules of 5 to 20 nm) – Zr and Si	A1	8EK
				A3.5	6CC

BAFG = Barium aluminoflurosilicate glass.

Figure 1. Information about the composites employed according to the manufacturers.



**Figure 2.** Wavelength distributions of the light curing unit (QTH) and the light that passed through the both composite in your different shades.

Standardized cylindrical specimens were obtained by putting the composite into a circular elastomer mold (2 mm thick  $\times$  7 mm in diameter). The bottom and top surfaces were covered with a transparent polyester strip and photo-activated by LCU. The curing tip was placed close to the elastomer mold/restorative composite set. For each color, 25 specimens were made, each measuring 2 mm thick by 7 mm in diameter. For the light transmission and Knoop hardness tests, 20 specimens were photo-activated for 20 seconds (in accordance with the manufacturer's recommendation). For the Scanning Electron Microscopy, 5 specimens were photo-activated for 5 seconds.

#### Light transmission test

The resin discs were made with the same diameter as the LCU tip (7 mm). Ten specimens were randomly connected with black adhesive paper to the LCU tip. The light that passed through the composite was measured with an Ophir 10A-V2-SH (Ophir Optronics) power meter [11]. The spectral distributions were obtained using a spectrometer USB 2000 (Ocean Optics – Figure 2). The data on the irradiance of light were submitted to two-way ANOVA (shade vs. composite) and the means were compared by Tukey's test ( $\alpha = 0.05$ ).

#### Knoop hardness test

After the photo-activation procedure, 10 randomly selected specimens were dry-stored at 37°C, for 24 hours. Thereafter, both top and bottom surfaces were flattened with SiC sandpapers with #200, 400 and 600 grit (Saint-Gobain, Recife, PE, Brazil) to obtain polished and flattened surfaces.

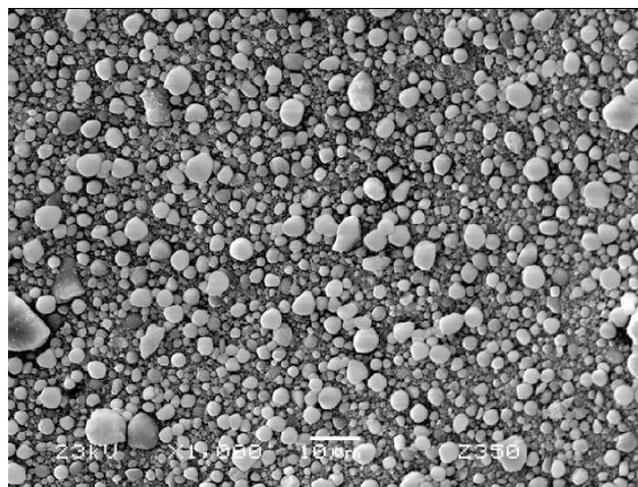
Indentations for the measurement of Knoop hardness numbers (KHN) were made in an HMV 2 (Shimadzu – Tokyo, Japan) hardness testing machine. Three readings were taken on the top and bottom surfaces, under a load of 50 gf for 15 s. The KHN for each surface was recorded as

the mean value of three indentations. Afterwards, the DKH of the same specimen was calculated [11]. The DKH data were submitted to two-way ANOVA (shade vs. composite) and the means were compared by Tukey's test ( $\alpha=0.05$ ).

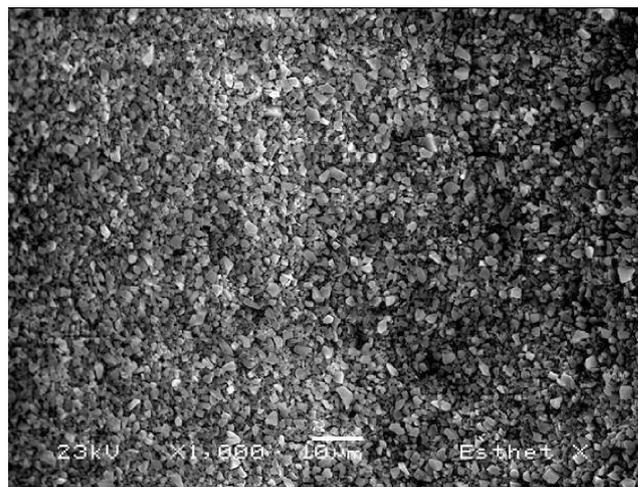
#### Scanning Electron Microscopy (SEM)

Five specimens of either Filtek Z350 or Esthet-X composite were immersed for one week in 2 mL acetone, which was changed daily [12].

Afterwards, the specimens were fixed in metallic stubs and covered with gold using MED 010 sputter coater equipment (Baltec, Balzers, Liechtenstein). Afterwards, they were observed under SEM JSM-5600 (Jeol Inc. – Peabody, MA, USA). Representative areas showing the filler particles were photographed at 1,000x magnification (Figures 3 and 4).



**Figure 3.** SEM photograph of Filtek Z350 composite resin (1,000 $\times$  magnifications). Agglomerated zirconia/silica spherical nanocluster can be noted.



**Figure 4.** SEM photograph of Esthet-X composite resin (1,000 $\times$  magnifications). Irregular filler particles can be observed.

## Results

The irradiance of light that passed through the shade A1 composite was statistically higher than the irradiance of light that passed through shade A3.5 for both composites. The irradiance that passed through the Filtek Z350 composite was statistically greater than the irradiance that passed through the Esthet X composite for both shades (Table 1,  $p=0.00001$ ).

**Table 1.** Mean values of irradiance that passes through the composite resins.

Shade	Irradiance (mW/cm <sup>2</sup> )	
	Filtek Z350	Esthet X
A1	298 (8) a, A	197 (5) b, A
A3.5	231 (6) a, B	167 (6) b, B

Mean values followed by different lowercase letters in the row and mean values followed by different capital letters in the column differed statistically by the Tukey's test at 5% level. ( ) Standard Deviation.

**Table 2.** Mean Knoop hardness number (KHN) and percentage of decrease of Knoop hardness between bottom and top (DKH).

Shade	Surface	KHN		DKH (%)	
		Filtek Z350	Esthet X	Filtek Z350	Esthet X
A1	top	59.82 (1.02)	50.23 (1.30)	16.53 (1.16) <sup>a, A</sup>	24.54 (2.00) <sup>b, A</sup>
	bottom	49.93 (0.66)	37.90 (3.22)		
A3.5	top	61.01 (1.36)	52.01 (1.64)	16.50 (1.49) <sup>a, A</sup>	28.53 (4.49) <sup>b, B</sup>
	bottom	50.94 (1.24)	37.17 (2.64)		

Mean values followed by different lowercase letters in the row and mean values followed by different capital letters in the column differed statistically by the Tukey's test at 5% level. ( ) Standard Deviation.

The DKH of shade A3.5 was statistically higher than that of shade A1 for the Esthet X composite; however, there was no statistically significant difference for Filtek Z350. The DKH of the Filtek Z350 composite was statistically higher than that of the Esthet X composite for both shades (Table 2,  $p=0.03035$ ).

Figure 2 shows the wavelength distributions of the light curing unit (QTH) and the irradiance that passed through the specimens with different shades of both composites.

## Discussion

During the photo-activation process, the light that passes through the composite resin is absorbed and scattered [13]. Thus, as the depth increases, the light's intensity is attenuated and its effectiveness is reduced [14]. Nevertheless, polymerization depth depends on light irradiance, exposure time and several other factors, such as material composition [15], resin composites shades [16] and translucency [17]. The most important limiting factor for depth of cure is light scattering, and this is maximized when the filler particle size is close to half of the wavelength emitted by the LCU [4].

For both shades (Table 1), the Filtek Z350 (spherical filler – Figure 3; shade A1-298 mW/cm<sup>2</sup> and shade 3.5-31 mW/cm<sup>2</sup>) showed greater light transmission than Esthet X (irregular filler – Figure 4; shade A1-197 mW/cm<sup>2</sup> – and shade 3.5-167 mW/cm<sup>2</sup>). Light transmission through the darker shades is diminished because of the opacity [18]. In the current study, the lighter shades (Filtek Z350 shade A1-298 mW/cm<sup>2</sup> and Esthet X shade A1-197 mW/cm<sup>2</sup>) produced statistically higher values than those of the darker shades (Filtek Z350 shade A3.5-231 mW/cm<sup>2</sup> – and Esthet X shade A3-167 mW/cm<sup>2</sup>; Table 1). This is probably due to the tendency of composites with darker shades to absorb a greater amount of light than those with lighter shades [19].

The polymerization of light-cured composite resins starts and is sustained when the rate of delivery of photons from the LCU is sufficient to maintain the photo-initiator compound (camphorquinone-CQ) in its excited or triplet state. Resin shade is a factor that can alter the efficacy of polymerization [20]. As mentioned, light transmission through the dark shades is diminished because of opacity [18]. Opaque shades decrease the capacity of light to penetrate into the bulk of the composite resin [21]. In the present study, with respect to DKH, Filtek Z350 (spherical filler – Figure 3; shade A1-16.53% and shade 3.5-16.50%) yielded a higher DKH than Esthet X (irregular filler – Figure 4; shade A1-24.54% and shade 3.5-28.55%) for both shades (Table 2). The filler volume fraction was similar for both composites tested (Figure 1) and the degree of conversion was hindered in composites whose filler particles approached the output wavelength of the curing light. This may be explained by the scattering effect of the penetrating light during photo-activation [17]. Thus, the lowest DKH was achieved in composites with irregular particle size, and it was dependent on particle shape because of the light scattering.

Table 2 shows DKH values of 24.54% (A1) and 28.53% (A3.5) for Esthet X composite. It has been suggested that a specimen of composite resin is adequately cured when there is a difference of no more than 20% between the maximum hardness at the top of the composite and the hardness at the bottom [22]. However, the ISO 10477 [23] specifies a minimum of 30%. Thus, both composites are in agreement with ISO 10477 [23]. However, insufficiently polymerized composite resin may present quite a large number of problems, such as poor color stability, greater stain uptake and risk of pulp aggression by non-polymerized monomers or portions of the material with different values of Young's modulus. It has been reported that loading well-polymerized composite layers that are placed on poorly polymerized



layers can lead the composite restoration to bend inward and become displaced, causing marginal fracture, open margins and cuspal deflection [21].

As mentioned, opaque shades decrease the capacity of the light to penetrate into the bulk of the resin composite [21]. In the current study, darker shades showed higher DKH than lighter shades for Esthet X composite (Table 2) probably due to the lower rate of light that passed through the dental composite (Figure 2). For Filtek Z350, the rate of light that passed through the dental composite was sufficient to photo-activate both shades, probably because this composite has spherical particle fillers that provide more uniform light reflection. However, none of the shades for both composites exceeded the limit stipulated by ISO 10477 [23].

For many types and shades of resin composite, a high degree of cure throughout a 2 mm thickness of light-activated resin composite is not achieved, because different types and shades of resin could promote inadequate cure. However, adequate polymerization is a crucial factor in obtaining an optimal physical mechanical performance of the dental resin composite [24]. Thus, with similar methods than other studies [11,20,25], different composite resins influenced the light transmission and the DKH, but could not establish a relation between light transmission and DKH. For this reason, the hypothesis that light transmission through different composite resins with different shades should influence the percentage of decrease in Knoop hardness between bottom and top was partially accepted.

## Conclusions

Based on the results analyzed and discussed, it may be concluded that:

1. Different composite resins influenced both light transmission and DKH.
2. Darker shades allowed lower light transmission, but could not establish a relation between dark shades and DKH.

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