



Evaluation of the Light Energy Transmission and Bottom/Top Rate in Silorane and Methacrylate-based Composites with Different Photoactivation Protocols

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ABSTRACT

Aim: This study investigated the influence of different composite resin organic matrix (methacrylate – Filtek Z350 XT and silorane – Filtek P90) on light energy transmission through the composite and bottom/top rate.

Materials and methods: A light-emitting diode (New Blue Phase), light-curing unit was used with different photoactivation protocols (high-continuous mode – HCM, 1400 mW/cm² for 20 seconds; low-continuous mode – LCM, 700 mW/cm² for 40 seconds; and soft-start mode – SSM, 140 mW/cm² for 5s followed by 39 seconds for 700 mW/cm²). Twenty specimens were prepared for each composite. The light energy transmission through the composite was calculated (n=10). The bottom/top rate of the same specimen was calculated (n=10). The data were compared by Tukey's test in different tests (light energy transmission through the composite and bottom/top rate).

Results: The light energy transmission through the Filtek Z350 XT composite (HCM – 576 mW/cm², LCM – 238 mW/cm², SSM – 232 mW/cm²) did not show statistical difference when compared with Filtek P90 composite (HCM – 572 mW/cm², LCM – 233 mW/cm², SSM – 230 mW/cm²). The bottom/top rate of the Filtek Z350 XT composite (HCM – 88.98%, LCM – 90.94%, SSM – 89.92%) was statistically higher than that of the Filtek P90 composite (HCM–77.29%, LCM–77.51%, SSM–77.79%).

Conclusion: Light energy transmission through the composite was not influenced by the use of different dental composite restoratives. However, the bottom/top rate of the composites was influenced by the use of different dental composite restoratives.

Clinical significance: Insufficiently polymerized composite resin may present a large number of problems. For this reason, dental composite resins should have the similar deep surface polymerization as the top surface in dental restorations.

Keywords: Photoactivation, Dental composite resins, Low-shrinkage composite.

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INTRODUCTION

Light-cured composite resins are widely used in dental restoration^{1,2} because of their ease in handling, esthetic appearance and minimal invasion of healthy tooth tissue.² Basically, dental composite resins (DCR) are composed of an organic matrix, load particles (glass, quartz, and/or melted silica) and a bonding agent, and, usually, an organic silane with a dual characteristic enabling chemical bonding with the load particle and copolymerization with the monomers of the organic matrix.³ The activation mode used for the curing of these materials is an indispensable factor in maintaining the effectiveness of the functionality of these direct restorations.⁴ Light-cured composite resins are activated using light-curing units (LCUs) that emit a narrow wavelength between 400 and 500 nm.⁵ The emitted blue light excites the photoinitiator present in the organic formulation of the composites, unleashing the polymerization DCR. The visible light photosensitizer camphorquinone (CQ) is widely used in the majority of DCR formulations.⁶

The light-emitting diode (LED) was developed to minimize the heat generated by the halogen light during photoactivation.⁷ The first light-emitting diodes (LEDs) emitted wavelengths from 455 to 486 nm, related to the

rate of absorption of the spectrum of camphorquinone.⁸ Nowadays, LEDs emit wavelengths wider than the first LEDs. Among the available LCUs, the LED is in common use in dentistry for the photoinitiated polymerization of DCR.⁴ LEDs are made of semiconductor materials that determine the type of emitted light.⁴ Each semiconductor material presents a range of energy that determines the spectrum of light emission, characterizing the emitted color. LEDs are designed to emit blue light for the photoactivation of dental composites.⁹

The rate of monomer conversion is dependent on the irradiance,⁴ and the shrinkage stress is due to the polymerization reaction speed.¹⁰ As the irradiance increases, the monomer conversion accelerates but results in higher stress generation.¹¹ Thus, the operator can minimize shrinkage stress and gap formation at the bonding interface (between tooth structure and the restoration) during photoactivation using different photoactivation methods¹² or incremental techniques.¹³ To decrease the speed of the polymerization reaction and, consequently, the shrinkage stress, techniques that suggest low irradiance during the initial period of photoactivation have been proposed.¹²⁻¹⁴ Polymerization using lower irradiance can reduce the stress, but the light exposure time must be extended to maintain the radiant exposure similar to that used in conventional methods.¹⁵ The soft-start polymerization technique adopts an initially low light intensity followed by a final cure with high light intensity.¹⁶ A slower rate of monomer-to-polymer conversion allows for better flow of the material, which, in turn, decreases contraction stresses, leading to better marginal adaptation.¹⁴

Vinyl cyclopropane derivatives, such as radical curing ring-opening monomers, are also able to copolymerize with common methacrylate-based resins.¹⁷ A different chemical approach was utilized for this reaction, focusing on the cationic ring-opening of spiro orthocarbonates, especially in combination with epoxy monomers.¹⁸ Silorane, a low-shrinkage, tooth-colored restorative material (as claimed by the manufacturer, 3M ESPE, St Paul, MN, USA), has been introduced in the dental market. Silorane was named to indicate a hybrid compound (siloxane and oxirane functional moieties).¹⁹ The cycloaliphatic oxirane functional groups

are responsible for lower shrinkage when compared to methacrylate-based composites, and the siloxane determines the highly hydrophobic nature of the siloranes. Oxiranes, which are cyclic ethers, polymerize by a cationic ring-opening mechanism, while methacrylates polymerize via a free-radical mechanism.¹⁸

The aim of the study was to investigate the influence of different composite resin organic matrix on light energy transmission through the composite and bottom/top rate. The null hypothesis tested was that there is no difference in the bottom/top rate between silorane and methacrylate-based resin composites.

MATERIALS AND METHODS

Resin Composites

Restorative resin composites, Filtek P90 (shade A2, 3M-ESPE) and Filtek Z350 XT (shade body A2, 3M-ESPE), were used in this present study (Table 1).

Light-curing Unit (LCU)

A LED (New Blue Phase, Ivoclar-Vivadent, Schaan, Liechtenstein) light-curing unit was used with different photoactivation protocols (high-continuous mode – HCM, low-continuous mode – LCM, soft-start mode – SSM). The LCU output (mW) was measured with a power meter (Ophir Optronics; Har-Hotzvim, Jerusalem, Israel). The tip diameter (9 mm) was measured with a digital caliper (model CD-15C; Mitutoyo, Japan) to determine the tip areas. The irradiance was calculated by dividing the light power by the tip area and photoactivated according to different photoactivation protocols (HCM – 1400 mW/cm² for 20 seconds, LCM – 700 mW/cm² for 40 seconds, SSM – 140 mW/cm² for 5 seconds followed by 39 seconds for 700 mW/cm²). Thus, the energy density was standardized at 28 J/cm². The spectral distributions (Figs 1 to 3) were obtained using a spectrometer (USB 2000; Ocean Optics, Dunedin, FL, USA).

Specimens' Preparation

Standardized cylindrical specimens were obtained by placing the composite into a circular elastomer mold (2 mm thick × 9 mm in diameter). The bottom and top surfaces

Table 1: Information about the composites employed according to the manufacturer

Composite	Organic matrix	Filler	Photoinitiator	Batch number
Filtek P90	Silorane resin	76% by weight (0.1 to 2.0 µm) – Quarz and Yttrium fluoride	Camphorquinone, iodonium salt and electron donor	N183458
Filtek Z350 XT	Bis-GMA, UDMA, TEGDMA, PEGDMA and Bis-EMA	78.5% by weight (silica – 20 nm, zirconia – 4 to 11 nm and zirconia/silica clusters of 0.6 to 1.0 µm)	Camphorquinone	N173043

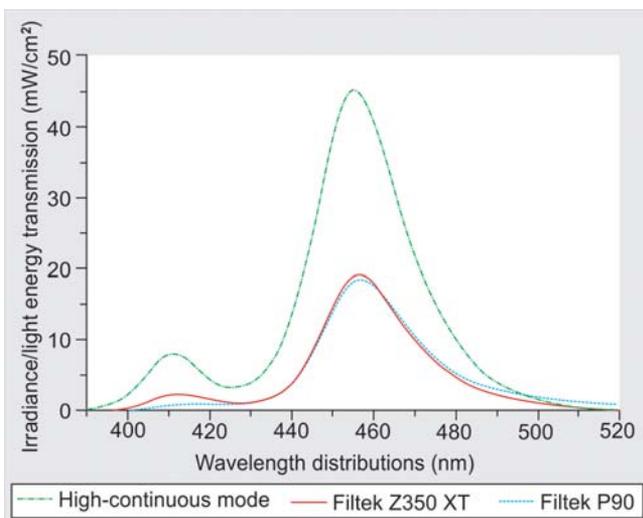


Fig. 1: Wavelength distributions of the irradiance in high-continuous mode and the light energy transmission that passed through the both composite restoratives

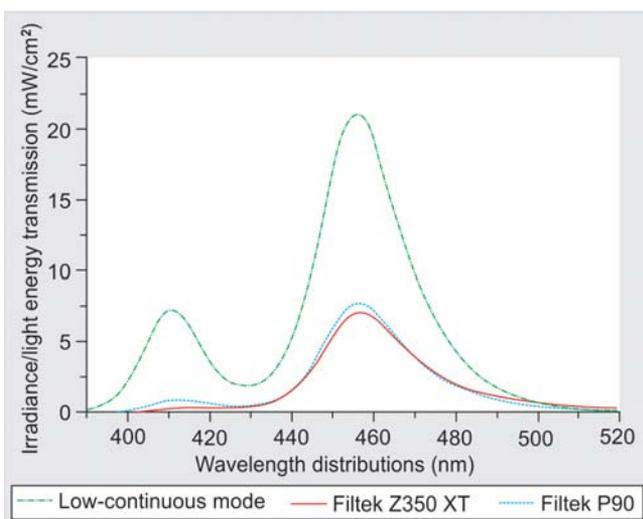


Fig. 2: Wavelength distributions of the irradiance in low-continuous mode and the light energy transmission that passed through the both composite restoratives

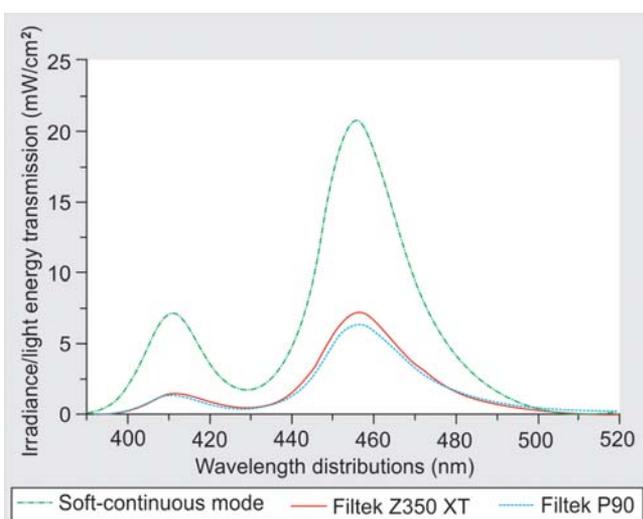


Fig. 3: Wavelength distributions of the irradiance in soft-start mode and the light energy transmission that passed through the both composite restoratives

were covered with a transparent polyester strip and photoactivated by LCU. Photoactivation was performed in accordance with the manufacturer's recommendations. For photoactivation, the curing tip was positioned close to the elastomer mold/restorative composite set. For each composite resin, 20 specimens were prepared, measuring 2 mm thick and 9 mm in diameter.

Light Energy Transmission Test

Randomly, ten specimens were attached to the LCU tip with black adhesive paper. The light energy transmission that passed through the composite was measured with a power meter (Ophir Optronics; Har-Hotzvim).^{20,21} The light energy transmission that passed through the composite data was submitted to ANOVA and the means were compared by Tukey's test ($\alpha = 0.05$). The spectral distributions of light energy transmission that passed through the composite were measured with a spectrometer (USB 2000; Ocean Optics, Dunedin, FL, USA).

Bottom/top Rate Test

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

Indentations for the Knoop hardness number (KHN) measurements were sequentially performed in a hardness testing machine (HMV 2; Shimadzu, Tokyo, Japan). Three readings were taken on the top and bottom surfaces under a load of 50 gf for 15 seconds each. The KHN for each surface was recorded as the mean of the three indentations. The KHN data were subjected to ANOVA and the means were compared by Tukey's test ($\alpha = 0.05$).

After this, the bottom/top rate was calculated for each specimen. The bottom/top rate data were subjected to ANOVA and the means were compared by Tukey's test ($\alpha = 0.05$).

RESULTS

Light Energy Transmission

As shown in Table 2, the light energy transmission that passed through the Filtek Z350 XT resin composite was not statistically greater than that which passed through the Filtek P90 resin composite in all photoactivation protocols ($p = 0.97$). The spectral distributions of light energy transmission that passed through the composite (Figs 1 to 3) were obtained using a spectrometer.

Table 2: Mean light energy transmission that passed through Filtek Z350 XT and Filtek P90 composites

Light energy transmission	Resin composite	
	Filtek Z350 XT	Filtek P90
High-continuous mode	576 (12.0) A	572 (18.3) A
Low-continuous mode	238 (13.5) A	233 (14.7) A
Soft-start mode	232 (15.0) A	230 (10.5) A

Mean values followed by different uppercase letters in rows differ statistically by Tukey's test at 5% level of significance. Standard deviations are in parentheses

Table 4: Mean bottom/top rate (%) of Filtek Z350 XT and Filtek P90 composites

Photoactivation protocols	Bottom/top rate (%)	
	Filtek Z350 XT	Filtek P90
High-continuous mode	88.98 (2.77) Aa	77.29 (6.45) Ba
Low-continuous mode	90.94 (13.42) Aa	77.51 (7.34) Ba
Soft-start mode	89.92 (4.35) Aa	77.79 (5.77) Ba

Mean values followed by different uppercase letters in rows and lowercase letters in columns differ statistically by Tukey's test at 5% level of significance. Standard deviations are in parentheses

Bottom/top Rate

The low-continuous mode showed higher values for the Knoop hardness ($p = 0.00062$) than soft-start mode in different surface (Table 3).

Table 4 indicates that the bottom/top rate ($p = 0.00001$) of the Filtek P90 resin composite was statistically lower than that of the Filtek Z350 XT resin composite for all photoactivation protocols. There was no statistical significance among the different photoactivation protocols.

DISCUSSION

Composite formulations have continued to evolve since Bis-GMA was first introduced to dentistry in 1962 by Bowen.²² It was modified in various ways based on different properties, such as viscosity or polarity. These changes were produced by variations of functional groups. Different comonomers, such as triethylene glycol dimethacrylate (TEGDMA) or urethane dimethacrylate (UDMA), were developed over time and contributed, in very different ways, to the product profile of composites.¹⁸ Several attempts to reduce shrinkage by changing the nature of the DCR have been made by universities and manufacturers.¹⁸ The silorane resin has been introduced in the dental market.¹⁹ The tested null hypothesis was rejected; differences were found between the bottom/top rate of the silorane and methacrylate-based resin composites.

During the photoactivation process, the light that passes through the DRC is absorbed and scattered.²³ Thus, the light intensity is attenuated and its effectiveness is reduced as the depth increases.²⁴ Nevertheless, the polymerization depth depends on light intensity, exposure time and several other factors, such as material composition,²⁵ composite

shade²⁶ and translucency.²⁷ All of these factors influence the amount of light intensity that reaches the deepest layers of the material. If the amount of light is not sufficient, the degree of conversion in these areas is low and the mechanical properties of the material, including Knoop hardness, are negatively affected. Other optical phenomena are important in understanding light transmission through a resin composite layer. A considerable part of the irradiation light that illuminates the resin composite surface is reflected. The part that penetrates has the function of exciting the photosensitizer to start the polymerization process as deeply as possible.²³ However, the extent of this light's depth depends on the absorption coefficients of the resin composite's component parts.²⁸ A previous study indicated that factors, such as filler and polymeric matrix refractive index, monomer type, filler type, and filler content can influence the light transmittance of resin composites.²⁹ In the present study, the wavelength distributions of light energy transmission (Figs 1 to 3) that passed through the resin were similar between the resin composites (Table 2), even when using composites with different matrix and loads (Table 1).

The generation of radical species for methacrylate composite curing is produced using a two-component system consisting of CQ, which is the actual photoinitiator, and an amine, responsible for the hydrogen transfer reaction.¹⁸ This system decomposes immediately due to exposure to light with a wavelength between 410 and 500 nm, generating the radical species to start the polymerization process.³⁰ The development of a photoactivated silorane composite occurs with a three-component initiating system composed of CQ, iodonium salt and an electron donor. CQ was chosen as a photoinitiator to match the emission spectra

Table 3: Mean values of the Knoop hardness number (KHN) in different surfaces for Filtek Z350 XT and Filtek P90 composites

Photoactivation protocols	Filtek Z350 XT (KHN)		Filtek P90 (KHN)	
	Top	Bottom	Top	Bottom
High-continuous mode	88.09 (10.97) ab	78.44 (10.62) ab	84.23 (8.51) a	64.71 (2.96) ab
Low-continuous mode	93.91 (4.47) a	85.36 (4.38) a	87.75 (9.01) a	67.59 (4.76) a
Soft-start mode	84.08 (7.37) b	75.57 (7.11) b	75.22 (7.91) b	58.31 (5.41) b

Mean values followed by different lowercase letters in columns differ statistically by Tukey's test at 5% level of significance. Standard deviations are in parentheses

of the currently used dental lamps. In this reaction path, the electron donor acts in a redox process and decomposes the iodonium salt into an acidic cation, which starts the ring-opening polymerization process.¹⁸ It is beneficial to use noncoordinative counter-anions A^- such as SbF_6^- or $B[(C_6F_5)_4]^-$ to enhance the reactivity. The three-component system provides the optimal balance between high polymerization reactivity and light stability.¹⁸ Thus, adequate polymerization is a crucial factor in obtaining an optimal physical mechanical performance of dental resin composites.³¹ Moreover, hardness evaluation was used as an indirect method to verify the degree of conversion of resin composites.³²

For the soft-start method, the activation of some free radicals during the first photoactivation cycle occurs and initiates the polymerization reaction.¹⁶ Immediately after the first polymerization cycle, the specimens are photoactivated using the irradiance higher than first polymerization cycle.¹⁶ The degree of conversion of composite resins depends on the energy density supplied during photoactivation.³⁰ Soft-start mode uses 0.7 J/cm^2 in the first polymerization cycle and 27.3 J/cm^2 in the second polymerization cycle. This amount of energy may have been insufficient to generate an adequate amount of free radicals and to polymerize adequately the composite resin during the first cycle. Thus, most of the polymerization may have occurred in the second photoactivation cycle that uses an energy density above that of the first polymerization cycle. However, this second polymerization cycle was not enough to the soft-start mode shows similar values of the Knoop hardness than that low-continuous mode (Table 3).

The difference found when comparing the top and bottom hardness numbers can be explained by the reason that the amount of light available to excite the photoinitiator dramatically decreases from the top surface inward as a result of light absorption and scattering by the composite restorative itself.^{33,34} Thus, the hardness values decrease continuously with increasing depth.³⁵ Deeper in the composite restorative, light attenuation results in lower excited photoinitiator molecules.^{34,36} The mobility of the developing polymer chains becomes progressively more restricted as a consequence of the increase in viscosity, reduction in free volume, formation of microgels and entanglement.^{34,37} Whereas, the hardness depends on the extent of the reaction and the degree of crosslinking produced during the monomer curing process.³⁵ The network becomes rigid and the chains become essentially immobile, and the propagation reaction is limited in diffusion; thus, the overall conversion rate decreases.^{34,38} To define the depth of polymerization, based on top and

bottom hardness measurements, it is common to calculate the bottom/top ratio hardness and give this ratio an arbitrary minimum value to consider the bottom surface adequately cured. Values of 0.80 and 0.85 have often been used.³⁹ According to current study,³⁹ Filtek P90 did not present efficient polymerization in deeper layers (Table 4). However, the ISO 10477⁴⁰ gives minimum in 0.70. Thus, both composites are in agreement with ISO 10477.⁴⁰ On the contrary, insufficiently polymerized composite resin may present a large number of problems, such as poor color stability, greater staining and a risk of pulp aggression by nonpolymerized monomers and portions of the material having different values of Young's modulus.⁴¹ It has been reported that loading well-polymerized composite layers on poorly-polymerized layers can cause the composite restoration to bend inward and displace, causing marginal fracture, open margins and cusp deflection.⁴¹ For this reason, DCR should have the similar deep surface polymerization as the top surface in dental restorations.

CONCLUSION

Considering the limitations of study, the null hypothesis was not accepted.

Light energy transmission through the composite was not influenced by the use of different dental composite. The composite Knoop hardness was influenced by different photoactivation protocols. The bottom/top rate of the composites was influenced by the use of different dental composite.

CLINICAL SIGNIFICANCE

Insufficiently polymerized composite resin may present a large number of problems. For this reason, dental composite resins should have the similar deep surface polymerization as the top surface in dental restorations.

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