

# Evaluation of irradiance and radiant exposure on the polymerization and mechanical properties of a resin composite

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**Abstract:** The objective of the present study was to evaluate the effect of irradiance and radiant exposure on the chemical-mechanical properties of a resin composite. A micro-hybrid resin composite (Clearfil AP-X, Kuraray) was investigated under two different irradiances: low (300 mW/cm<sup>2</sup>) and high (800 mW/cm<sup>2</sup>) and radiant exposures: 8 and 16 J/cm<sup>2</sup>. Four groups, named Low 8 J/cm<sup>2</sup>, High 8 J/cm<sup>2</sup>, Low 16 J/cm<sup>2</sup>, and High 16 J/cm<sup>2</sup> were tested, and their flexural strengths, elastic moduli, depths of cure, and degrees of conversion were evaluated. Data were analyzed using two-way ANOVA and Tukey's test. A multiple linear regression model was used to correlate the irradiance and radiant exposure with dependent variables ( $\alpha = 0.05$ ). Irradiance and radiant exposure were found statistically significant for all dependent variables. The interaction between the factors was statistically significant only for the degree of conversion and elastic modulus. Group Low 16 J/cm<sup>2</sup> exhibited a significantly superior performance in all the evaluated properties. Barring the degree of conversion, no significant differences were observed among the properties evaluated between the Low 8 J/cm<sup>2</sup> and High 8 J/cm<sup>2</sup> groups. The adjusted R<sup>2</sup> values were high for the depth of cure and degree of conversion (0.58 and 0.96, respectively). Both irradiance and radiant exposure parameters play an important role in establishing the final properties of a micro-hybrid resin composite. Irradiance has a greater influence under higher radiant exposures.

**Keywords:** Composite Resins; Polymerization; Mechanical Tests; Spectroscopy, Fourier Transform Infrared; Curing Lights, Dental.

## Introduction

Resin-based composites have become widely used since their introduction in the 1960s.<sup>1</sup> The final properties of these materials are obtained via irradiation with lights of specific wavelengths, leading to their hardening through a polymerization reaction and resulting in a highly crosslinked structure.<sup>2</sup> The degree of conversion, *i.e.*, the number of monomers converted into polymers can be quantified.<sup>3</sup> An insufficient degree of conversion could affect the material biocompatibility,<sup>4</sup> color stability,<sup>5</sup>



hardness,<sup>6,7</sup> fracture toughness,<sup>8</sup> and flexural strength.<sup>9</sup> The decrease in the overall properties of the material could affect its longevity.<sup>10</sup>

The degree of conversion could be affected by various factors, primarily the characteristics of the applied light, including its irradiance, wavelength, and the type of light-curing unit (*i.e.*, light-emitting diode, quartz-tungsten-halogen lights).<sup>11</sup> Radiant exposure, defined as the irradiance (mW/cm<sup>2</sup>) multiplied by the exposure time (seconds), is an essential factor in the light-curing of resin composites.<sup>12,13</sup> The “reciprocity law” establishes that comparable properties in a material can be obtained, while the radiant exposure remains constant, regardless of how it is obtained among different combinations of irradiances and exposure times.<sup>14</sup> Based on this law, attempts have been made to reduce clinical times by introducing high-power light-curing units in the market.<sup>11</sup>

Some studies have established that to achieve an adequate degree of conversion, a radiant exposure between 16–20 J/cm<sup>2</sup> is required.<sup>11,15</sup> According to the reciprocity law, irradiances of 400 and 800 mW/cm<sup>2</sup> must be delivered for 40 and 20 s, respectively. Since ISO 10650-1 and 10650-2 neither specify the irradiance that light-curing units must emit nor the potential of the light-curing unit, there is a lack of standardization in terms of irradiance of contemporary light-curing units. The standards are primarily concerned with the patient safety by setting limits to prevent harmful exposure to ultraviolet (UV) light and heat, thereby merely stating that the irradiance must match the manufacturer’s specifications.<sup>12</sup>

In this context, some studies supported the use of light-curing units with high irradiance and short curing time,<sup>11,15</sup> suggesting the importance of a constant radiant exposure.<sup>16</sup> However, the reciprocity law is not satisfied in all cases,<sup>9</sup> and to date, its role is not

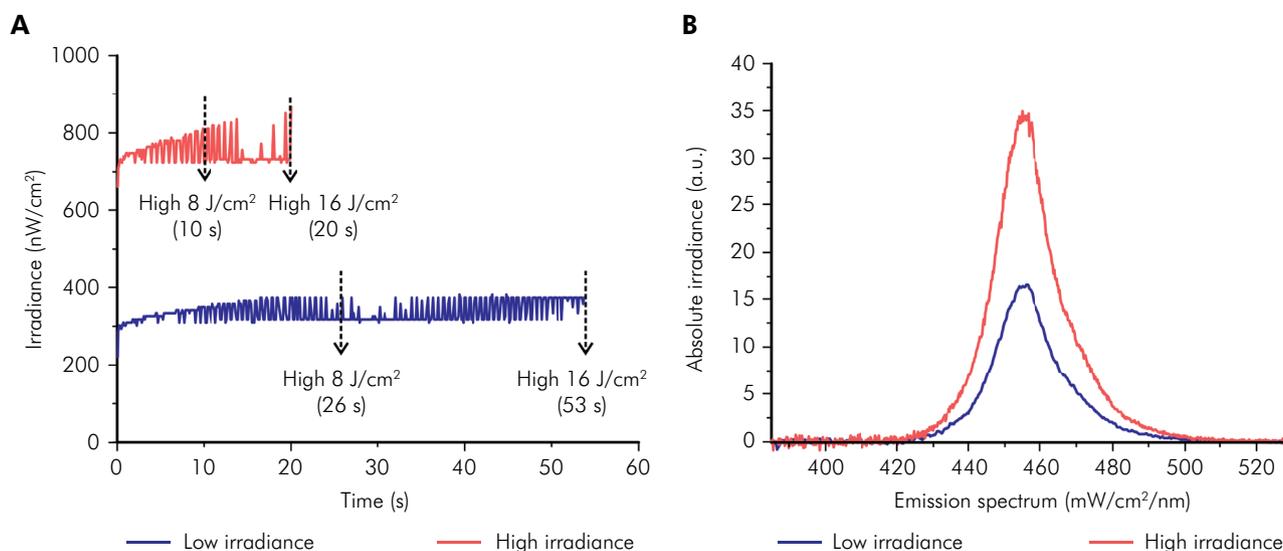
clear when determining the final material properties. Considering this, the objective of the present study was to evaluate the effect of irradiance and radiant exposure on the chemical-mechanical properties of a resin composite. We hypothesize that both radiant exposure and irradiance have a significant effect on the chemical-mechanical performance of a resin composite for dental use.

## Methodology

In this study, the chemical and mechanical characterizations of a micro-hybrid composite (Clearfil AP-X, Kuraray; Tokyo, Japan. Batch # BV0016) were performed according to the following factors: a) irradiance at two levels: low - 300 mW/cm<sup>2</sup> and high - 800 mW/cm<sup>2</sup>; b) radiant exposure at two levels: 8 J/cm<sup>2</sup> and 16 J/cm<sup>2</sup>. Following the manufacturer’s instructions, the group consisting of a radiant exposure of 16 J/cm<sup>2</sup> with an irradiance of 800 mW/cm<sup>2</sup> (H16) was considered as the control.<sup>17</sup> Resin composites were photopolymerized using an LED light-curing unit (Radii-cal, SDI, Bayswater, Australia). Table 1 describes the study design. The light-curing unit irradiance was tested following the ISO 10605-2 directions.<sup>18</sup> The irradiance was controlled by adjusting the distance between the light tip and the sensor, controlled by plastic spacers (Delrin, DuPont) with a standardized thickness (1 mm), and the light-curing unit was fixed with a clamp. The distance necessary to achieve different testing irradiances was determined by the bottom sensor of the MARC Resin Calibrator spectrophotometer (BlueLight Analytics Inc; Halifax, Canada). The irradiance (mW/cm<sup>2</sup>) and emission spectrum (mW/cm<sup>2</sup>/nm) of the light-curing unit are described in Figure 1. Each condition was measured in triplicate to ensure reproducibility.

**Table 1.** Experimental groups evaluated.

Group	Radiant exposure (J/cm <sup>2</sup> )	Irradiance (mW/cm <sup>2</sup> )	Exposure time (seconds)	Distance (mm)
Low 8 J/cm <sup>2</sup> (L8)	8	300	26	9
High 8 J/cm <sup>2</sup> (H8)		800	10	4
Low 16 J/cm <sup>2</sup> (L16)	16	300	53	9
High 16 J/cm <sup>2</sup> (H16)		800	20	4



**Figure 1.** [A] Irradiance variation (mW/cm<sup>2</sup>) and [B] Emission spectrum (mW/cm<sup>2</sup>/nm) of the photopolymerization unit used in this study.

The primary response variable was flexural strength ( $n = 10$ ). The specimen size was estimated based on a previous study<sup>19</sup> evaluating the effect of the applied radiant exposure on the mechanical properties of a resin composite in a comparative study design with four groups, a minimum detectable difference in means of 26.3, standard deviation of 14.6, power of 0.8, and  $\alpha = 0.05$ . Secondary response variables related to the material characterization were elastic modulus ( $n = 10$ ), depth of cure ( $n = 3$ ),<sup>20</sup> degree of conversion, and polymerization rate ( $n = 3$ ).<sup>21</sup>

Flexural strength of different groups was evaluated based on the specifications provided by the International Standard ISO 4049,<sup>22</sup> except for the specimen dimensions.<sup>23</sup> Bar-specimens (10 mm × 2 mm × 2 mm) were prepared by filling stainless steel molds (ODEME Dental Research; Luzerna, Brazil) with uncured materials ( $n = 10$ ). Then, the specimens were activated on both sides using the curing protocol described above (a radiant exposure of 8 J/cm<sup>2</sup> using an irradiance of 300 or 800 mW/cm<sup>2</sup> and a radiant exposure of 16 J/cm<sup>2</sup> using an irradiance of 300 or 800 mW/cm<sup>2</sup>). After removing specimens from the mold, their dimensions were measured using a digital caliper (Mitutoyo; Kawasaki, Kanagawa, Japan). Specimens were evaluated after 24 h of storage in distilled water at 37°C. For the

three-point bending test, the specimens were placed in a universal mechanical testing machine (EMIC DL 500; São José dos Pinhais, Brazil) on two cylindrical supports of 2.0 mm diameter, parallel to each other with 8.0 mm between their centers. The mechanical test was performed using a load cell of 1 kN at a crosshead speed of 1 mm/min. Flexural strength ( $\sigma$ ) and elastic modulus ( $E$ ) were calculated using the formulas provided by international standards:<sup>22,24</sup>

$$\sigma = \frac{3Fl}{2bh^2} \quad E = \frac{F_1 l^3}{4bh^3 d}$$

where  $\sigma$  = flexural strength [MPa];  $F$  = maximum load at the fracture point [N];  $l$  = distance between the supports [12 mm];  $b$  = width of the specimen [mm];  $h$  = height [mm].  $E$  = elastic modulus [MPa];  $F_1$  = force recorded when the deformation stops are directly proportional to the force registered in the graph [N];  $d$  = deflection [mm] at  $F_1$ .

The scraping test methodology was performed according to the methods described in ISO 4049.<sup>22</sup> Stainless steel mold (8.0 mm height × 4.0 mm diameter) was placed over a Mylar strip and a glass microscope slide. Afterwards, the mold was filled, and a second Mylar strip film was placed on top, followed by a second microscope slide for compressing the material within the mold to avoid bubble entrapment.

Specimens were activated on top using the curing protocol described above (a radiant exposure of 8 J/cm<sup>2</sup> using an irradiance of 300 or 800 mW/cm<sup>2</sup> and that of 16 J/cm<sup>2</sup> using an irradiance of 300 or 800 mW/cm<sup>2</sup>). After polymerization, the specimens were removed, and the unpolymerized material was withdrawn from the bottom with a plastic spatula. The length of the cured material cylinder was measured using a digital micrometer (Mitutoyo; Kawasaki, Kanagawa, Japan) and divided by two.

The degree of double bond conversion and polymerization rate of the experimental groups was evaluated via Fourier transform mid-infrared spectroscopy using a Fourier transform-infrared spectrometer (Shimadzu Prestige 21 spectrometer, Shimadzu; Kyoto, Japan) coupled to an attenuated total reflectance device with a diamond cell. The spectrophotometer software package was used in the monitoring scan mode in the range of 1500–1800 cm<sup>-1</sup>, a resolution of 4 cm<sup>-1</sup>, and a mirror speed of 2.8 mm/s. With this configuration, a scan was acquired every 1 s during photoactivation. All measurements were performed under a controlled room temperature of 23°C (± 2°C) and 60% (± 5%) relative humidity. A silicon mold (3.0 mm diameter and 2.0 mm height) was placed at the center of the attenuated total reflection unit diamond cell window. The mold was filled with the resin composite and a Mylar strip, and a microscope glass was used to standardize the surface and material height. Then, the specimens (n = 3) were activated on top using the curing protocol described above. The spectrum was recorded before and immediately after the polymerization process. The degree of double bond conversion was obtained considering the aliphatic C=C bond absorbance located at 1636 cm<sup>-1</sup> and aromatic C=C bond located at 1609 cm<sup>-1</sup> using the following equation:<sup>3</sup>

$$DC (\%) = \left( 1 - \frac{\frac{h_{1636}}{h_{1609} \text{ pol}}}{\frac{h_{1636}}{h_{1609} \text{ non-pol}}} \right) \times 100\%$$

Where  $h_{1636}$  and  $h_{1609}$  are the heights of the bands at 1,636 and 1,609 cm<sup>-1</sup>, respectively. The term “non-pol” corresponds to the spectrum of the unpolymerized

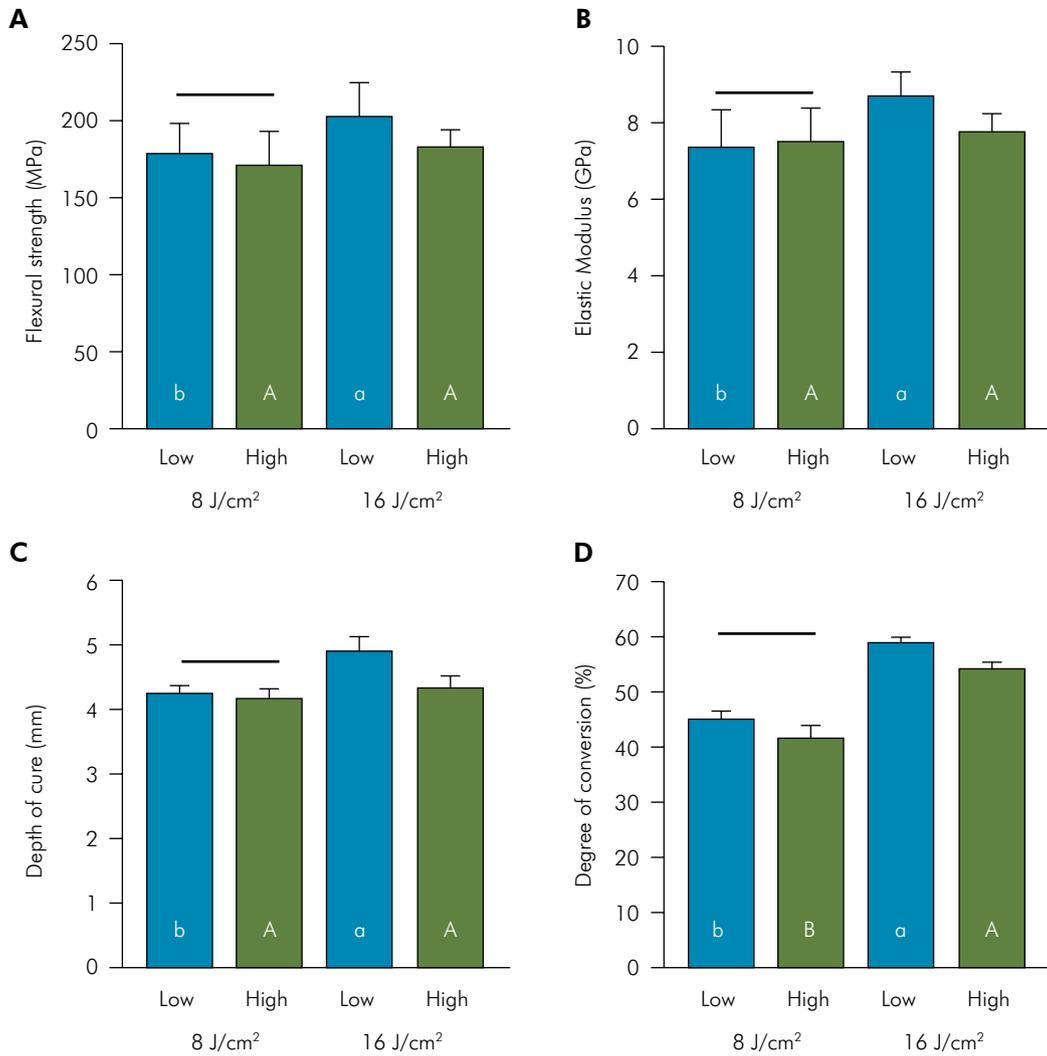
monomer mixture, and the term “pol” refers to the spectrum of the material after 30 s of photoactivation.

The degree of double bond conversion vs. polymerization reaction time data was plotted, and Hill’s 1 three-parameter nonlinear regression was performed for curve fitting.<sup>26</sup> The rate of polymerization (Rp) was calculated considering these data/fitted plots.

The statistical analyses were performed using the SPSS Statistics 26.0 software (IBM; Armonk, USA). The data were analyzed to test the assumption of normal distribution and homogeneity of variance. Two-way ANOVA analysis was used to evaluate the effects of irradiance and radiant exposure on dependent variables. Multiple comparisons were performed using the Tukey’s post-hoc test. A multiple linear regression model was used to investigate the linear dependence of the outcomes (flexural strength, elastic modulus, depth of cure, and degree of conversion) on the predictors (irradiance and radiant exposure). In all cases, the significance level was set to a < 0.05.

Figure 2 shows the results of (A) flexural strength (MPa), (B) elastic modulus (GPa), (C) depth of cure (mm), and (D) degree of conversion (%) of the different groups. Flexural strength was significantly influenced by both irradiance and radiant exposure factors (p < 0.05); however, the interaction between these factors was not statistically significant (p = 0.32). The effect of radiant exposure was statistically significant when low irradiance was used (p = 0.007); however, this effect could not be observed for high irradiance (p = 0.16). Irradiance was statistically significant only when the materials were polymerized using a radiant exposure of 16 J/cm<sup>2</sup> (p = 0.03).

Regarding the elastic modulus, the radiant exposure and interaction between radiant exposure and irradiance were statistically significant (p = 0.002 and p = 0.03, respectively). Radiant exposure was statistically significant when low irradiance was used (p < 0.001), but insignificant under high irradiance (p = 0.459). At a radiant exposure of 8 J/cm<sup>2</sup>, the irradiance was not statistically significant (p = 0.67). Conversely, for the radiant exposure of 16 J/cm<sup>2</sup>, the irradiance of 300 mW/cm<sup>2</sup> was statistically significant higher (p = 0.011).



**Figure 2.** Flexural Strength (A), elastic modulus (B), depth of cure (C), and degree of conversion (D) of the micro-hybrid composite as the functions of irradiance and radiant exposure are represented in bar charts. For each chart, bars connected by the same horizontal line represent no differences between the groups (irradiance for each radiant exposure). Different lowercase and capital letters indicate the differences between the blue and red charts, respectively.

Depth of cure was significantly influenced by both irradiance and radiant exposure ( $p < 0.05$ ), and a significant interaction between these two variables was also observed ( $p = 0.03$ ). Radiant exposure was statistically significant only when low irradiance was used ( $p = 0.002$ ). In contrast, regarding irradiance, statistically significant differences were observed only for the radiant exposure of  $16 \text{ J/cm}^2$  ( $p = 0.004$ ).

The degree of conversion was significantly influenced by both irradiance and radiant exposure ( $p < 0.001$ ); however, the interaction between these

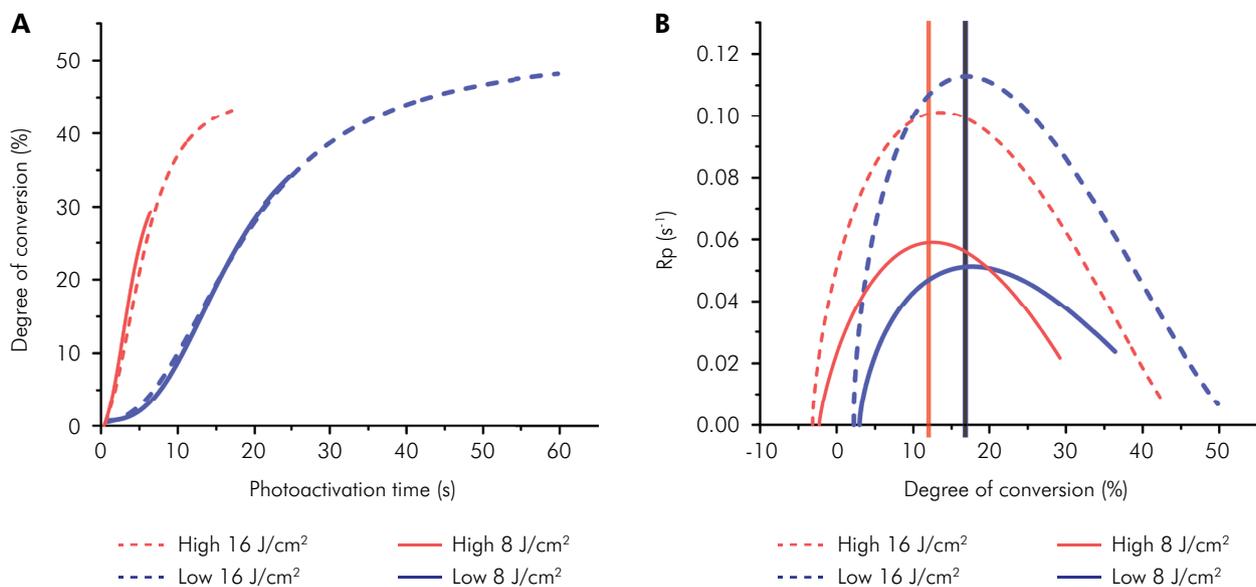
factors was not statistically significant ( $p = 0.53$ ). Radiant exposure was statistically significant for both high and low irradiances ( $p < 0.001$ ). Similarly, statistically significant differences were also observed when analyzing the irradiance for both radiant exposure levels ( $p = 0.006$  for  $16 \text{ J/cm}^2$  and  $p = 0.02$  for  $8 \text{ J/cm}^2$ ).

Figure 3A shows the degree of conversion as a function of time. The material polymerized with a radiant exposure of  $16 \text{ J/cm}^2$  under an irradiance of  $300 \text{ mW/cm}^2$  exhibited the highest degree of conversion. Conversely, the material polymerized

with a radiant exposure of 8 J/cm<sup>2</sup> and an irradiance of 800 mW/cm<sup>2</sup> displayed the lowest conversion degree. Figure 3B shows the degree of conversion against the polymerization rate. Regardless of the radiant exposure, the materials polymerized using a high irradiance (800 mW/cm<sup>2</sup>) vitrified at the earliest stages of polymerization.

The result summary of the multiple linear regression model is depicted in Table 2. The multiple linear regression model indicates that the variabilities in radiant exposure and irradiance significantly influence the variability in all the

properties evaluated ( $p \leq 0.008$ ). The adjusted R<sup>2</sup> values were high for the depth of cure and degree of conversion (0.58 and 0.96, respectively). The multiple linear regression model also indicated that an increase in the radiant exposure was associated with a significant increase in all response variables, being a highly influential parameter for depth of cure and degree of conversion ( $b_i = 0.63$  and  $0.94$ , respectively). In contrast, an increasing irradiance was associated with a significant decrease in all dependent variables, being a highly influential parameter only for the depth of cure ( $b_i = -0.51$ ).



**Figure 3.** Degree of conversion (A) and polymerization rate as the functions of degree of conversion (B) of the different irradiance and radiant exposure parameters. Vertical lines at B show the point in conversion where Rp approaches the maximum and approximately corresponds to the material vitrification.

**Table 2.** Summary of the multiple linear regression model.

Dependent variable	Explanatory variable	Unstandardized coefficients (95%CI)	Standardized coefficient ( $b_i$ )	Adjusted R <sup>2</sup>	p-value
Flexural strength	Radiant exposure	2.28 (0.76–3.81)	0.42	0.24	0.002
	Irradiance	- 0.27 (-0.05– -0.003)	-0.32		
Elastic modulus	Radiant exposure	99.42 (34.66–164.17)	0.44	0.21	0.006
	Irradiance	- 0.780 (-1.82–0.26)	-0.22		
Depth of cure	Radiant exposure	0.05 (0.02–0.09)	0.63	0.58	0.008
	Irradiance	- 0.001 (-0.001–0)	-0.51		
Degree of conversion	Radiant exposure	1.16 (1.42–1.9)	0.94	0.96	< 0.001
	Irradiance	-0.008 (-0.01– -0.004)	-0.29		

## Discussion

The present study analyzed the chemical-mechanical properties of a micro-hybrid resin composite under different irradiances (high and low) and radiant exposures (8 and 16 J/cm<sup>2</sup>). The irradiance of 300 mW/cm<sup>2</sup> was included in the experimental design considering that the delivery of this irradiance could occur when polymerizing increments at the bottom of deep preparations, as in the proximal box of a Class II preparation, wherein the distance between the tip of the light-curing unit and the material could imply a low irradiance, thus reducing the total energy delivered. Results showed the superior depth of cure, degree of polymerization, flexural strength, and elastic modulus as the radiant exposure was increased. Conversely, an increase in the irradiance was associated with a decrease in the dependent variables. Therefore, the results confirmed the hypothesis stating that both radiant exposure and irradiance have a significant effect on the chemical-mechanical performance of a resin composite.

Several studies have shown that sufficient radiant exposure must be provided to resin composites to achieve sufficient degree of conversion and mechanical properties.<sup>26-29</sup> According to the reciprocity law, irradiance should not interfere with mechanical properties for a constant radiant exposure.<sup>14</sup> However, according to our results, irradiance was a key factor in obtaining enhanced mechanical properties for group Low 16 J/cm<sup>2</sup>. This behavior was expected since this group achieved a statistically significantly higher degree of conversion values. Reportedly, a high degree of conversion is related to improved resin composite physical, chemical, and biological properties.<sup>29,30</sup> The better performance of the Low (300 mW/cm<sup>2</sup>) 16 J/cm<sup>2</sup> group could be related to the longer exposure, directly impacting the reaction kinetics and improving the polymer network development, and consequently, the mechanical properties of the material.<sup>31</sup> In contrast, when the radiant exposure of 8 J/cm<sup>2</sup> was delivered, the reciprocity law was fulfilled.

Literature describes several methods to evaluate the depth of cure,<sup>32,33</sup> making it difficult to compare our results with other studies; however, our data still agreed with the reported literature.<sup>20,32</sup> The results of

this study showed a statistically significant difference for group Low 16 J/cm<sup>2</sup>, when compared to other groups (Figure 1). Additionally, all groups complied with the requirements specified in international regulations<sup>22</sup> with values ranging from 4.21 to 4.94 mm. In comparison, one study<sup>32</sup> showed that a conventional resin reached up to 5-mm depth of cure by applying 73 J/cm<sup>2</sup>, a radiant exposure much higher than the 16 J/cm<sup>2</sup> used in the present study. *In-vitro* studies suggest that a radiant exposure of 16–20 J/cm<sup>2</sup> is needed to polymerize a 2.0-mm thick layer.<sup>11,15</sup> However, this work verified that this value could be overestimated, and a radiant exposure of 8 J/cm<sup>2</sup> was sufficient to achieve adequate properties in terms of depth of cure. However, depth of cure is a material-dependent variable<sup>32-34</sup> suggesting that intrinsic factors, such as the resin color and translucency could affect the light scattering during the photoactivation time.<sup>35,36</sup>

Irradiance and the duration of exposure are important factors in resin composite polymerization.<sup>34</sup> Reciprocity law assumes that the degree of conversion will be similar as long as the same radiant exposure is delivered to the material.<sup>11</sup> In the present study, results showed that the reciprocity law was not fulfilled when the radiant exposure was 16 J/cm<sup>2</sup> as the Low 16 J/cm<sup>2</sup> group presented a higher degree of conversion (Figure 1). This could be because when a low irradiance is used, the polymer vitrification is delayed, thereby facilitating the polymer chain formation with higher molecular weight and crosslinking degree.<sup>11,30</sup> This was confirmed by plotting the degree of conversion versus the polymerization rate (Figure 3B), where the vitrification point observed for groups polymerized with an irradiance of 300 mW/cm<sup>2</sup> is reached at a higher degree of conversion values. This effect may explain the lower degree of conversion observed for an irradiance of 800 mW/cm<sup>2</sup>. Also, the RP<sub>max</sub> for the groups with an irradiance of 800 mW/cm<sup>2</sup> occurred early in the polymerization, associated with the molecular mobility restrictions and limiting conversion.<sup>37</sup> A major consequence of the early vitrification is the entrapment of free radicals and other active species (*i.e.*, pendant double bonds, free monomers, and photoinitiators) in the polymer

network, influencing the mechanical properties of the material,<sup>14</sup> as observed in the study.

The results of the present work suggest that the radiant exposure of 16 J/cm<sup>2</sup> at low irradiance results in better polymerization and final properties. This takes clinical relevance due to the relationship between the chemical–mechanical properties of the material and its clinical performance.<sup>38</sup> Alternatively, low irradiance can imply a longer polymerization time, negatively impacting the total clinical time invested in performing a resin composite restoration.<sup>32</sup> Importantly, under the *in-vitro* circumstances used in this study, the differences observed in the chemical–mechanical properties may not be clinically relevant; thus, it is advisable to apply a radiant exposure of 16 J/cm<sup>2</sup> using an irradiance of 800 mW/cm<sup>2</sup> as a justifiable option when analyzing the cost/benefit. Moreover, considering that current light-curing units work using high irradiance, clinicians must be aware that the increased irradiance may affect some material properties.<sup>39</sup>

Limitations of the present study include its *in-vitro* design, the evaluation of two irradiances and radiant exposures only, the use of one composite resin brand and one light-curing unit only. Higher irradiances must be considered for future studies. Furthermore, the material properties were assessed using specimens polymerized with the so-called “hotspots” of the light-curing unit for which the highest and uniform irradiance is achieved. Further studies are required to understand if the irradiance and radiant exposure have any effect on the material properties after aging. Additionally, further studies must be

conducted to evaluate other material properties, such as polymerization shrinkage and contraction stress. Clinical studies are also required to evaluate the effect of irradiance and radiant exposure on clinical longevity.

Considering the current trend of increasing the irradiance of light-curing units to decrease the clinical times, it is important to highlight that the use of higher irradiances could further decrease the resin composite properties. Further research is required into the effect of higher intensities and their long-term effects considering different resin-based materials.

## Conclusion

Despite the limitations of the present study, we conclude that irradiance and radiant exposure influence the chemical–mechanical properties of a micro-hybrid resin composite. The present findings suggest that a radiant exposure of 8 J/cm<sup>2</sup> could compromise the material’s mechanical performance. The material under the radiant exposure of 16 J/cm<sup>2</sup> achieved better mechanical properties and under this polymerization condition, irradiance played a significant role.

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