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Effect of ceramic thicknesses and opacities on water sorption and solubility of a light-curing resin cement by different units

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Aim: This study evaluated the water sorption and solubility of a light-cured resin cement, under four thicknesses and four opacities of a lithium disilicate ceramic, also considering three light-emitting diode (LED) units. Methods: A total of 288 specimens of a resin cement (AllCem Veneer Trans -FGM) were prepared, 96 samples were light-cured by each of the three light curing units (Valo - Ultradent / Radii-Cal - SDI / Bluephase II - Ivoclar Vivadent), divided into 16 experimental conditions, according to the opacities of the ceramic: High Opacity (HO), Medium Opacity (MO), Low Translucency (LT), High Translucency (HT), and thicknesses (0.3, 0.8, 1.5, and 2.0 mm) (n = 6). The specimens were weighed at three different times: Mass M1 (after making the specimens), M2 (after 7 days of storage in water), and M3 (after dissection cycle), for calculating water sorption and solubility. Results: The higher thickness of the ceramic (2.0 mm) significantly increased the values of water sorption (44.0± 4.0) and solubility (7.8±0.6), compared to lower thicknesses. Also, the ceramic of higher opacity (HO) generated the highest values of sorption and solubility when compared to the other opacities, regardless of the thickness tested (ANOVA-3 factors / Tukey's test, α = 0.05). There was no influence of light curing units. **Conclusion:** Higher thicknesses and opacities of the ceramic increased the water sorption and solubility of the tested light-cured resin cement.

Keywords: Cementation. Ceramics. Light-curing of dental adhesives. Resin cements.

Introduction

In recent years, minimal intervention dentistry has presented, as an aesthetic restorative alternative, the use of ceramic veneers, with reduced dental preparation^{1,2}. Among the ceramic options, lithium disilicate has been widely used because it allows adhesive cementation procedures³ and is highly aesthetic, reproducing optical effects similar to those of natural teeth and enabling efficient masking of teeth with chromatic changes^{4,5}. Ceramic laminates are fixed to the teeth using adhesive systems and resin cement, which is capable of generating a strong bond between the dental substrate and the laminate ceramic^{1,5}.

Resin cement can be classified according to its polymerization: self-curing, light-curing, and dual^{5,6}. For ceramic laminate veneers, light-cured cements are the most suitable, since self-curing and dual cements present, in their composition, tertiary amines, which, when reacted with benzoyl peroxide, become responsible for the yellowing of the material⁶. Considering that ceramic laminates have a reduced thickness in many cases, this yellowing could compromise the aesthetic result of the treatment, especially in long term^{1,7}.

The ceramic material used in the restorative technique can present different thicknesses and opacities, which are associated with the depth of preparation and the darkness of the tooth². The different thicknesses and opacities of the ceramics directly affect the amount of light available to light-cure the resin cement underlying the ceramic material^{4,6}. This is because the light radiated by the light-curing unit must go through the ceramic and reach the cement, in a uniform, efficient, and satisfactory manner, to achieve a high degree of conversion of the resin monomers of the cement, ensuring its mechanical properties^{5,6}.

More translucent laminates, as well as less thick ones, tend to facilitate the passage of light through the restoration, allowing a greater amount of light to reach the resin cement below the restoration^{5,8}. However, in some clinical situations, it is necessary to mask darkened teeth, using a more opaque and thicker ceramic restorations^{1,8,9}. There are no conclusive studies in the literature associating different opacities of ceramics with different thicknesses to determine which situations could ensure the best passage of light through the ceramic restoration; or about which of the two variables – thickness or opacity – is able to compromise this passage of light more significantly^{1,5,6,10}.

In addition to the optical characteristics and thickness of the ceramic, the parameters of the light must also be considered¹¹. A high intensity of power, along with a homogeneous and collimated light beam, are required so that the light is able to pass through the ceramic, without losing so much power and without dissipating too much into the material¹². The delivery of this light energy to resin cement is also strongly influenced by the time of application of the light^{2,10}. In addition, the light must have wavelengths in the absorbance range of the photoinitiators contained in the resinous material, such as camphorquinone, considered the main photoinitiator, which shows its absorption peak around 468-470 nm^{2,6,10}. Thus, the importance of light in the light-curing process of resin cements makes the analysis of the light-curing parameters essential for the safety of clinical procedures.

When the light curing of resin cement is compromised, some negative effects can occur, such as a low degree of conversion and high water sorption and solubility of the material, which will generate poor mechanical properties, infiltration and detachment of the restoration^{5,6,11,13}. In the oral environment, fluid sorption can occur, resulting in the swelling of the resinous material at the tooth interface/restoration, causing changes in its organic matrix and impairment in the structure of this material, because of the dissociation of the inorganic charge. This decreases the resistance and the solubility of the material, reducing its mass by leaching its components and, consequently, releasing unreacted monomers, which can even cause damage to the dental pulp^{1,13-16}.

Thus, it is important to understand the influence of dental ceramic thickness and opacity both alone and in association on water sorption and solubility of resin cement, using different light-curing units. The hypothesis of this study was that the greater the thickness and opacity of the ceramic interposed, the higher the water sorption and solubility values of the resin cement, regardless of the light-curing unit used. Therefore, this study aimed to evaluate the physical properties (water sorption and solubility) of a light-cured resin cement under four thicknesses and four opacities of a lithium disilicate-based ceramic, considering three different light-emitting diode (LED) units.

Materials and methods

Preparation of Specimens

A total of 288 specimens of AllCem Veneer Trans resin cement (FGM, Dentscare LTDA, Joinville-SC, Brazil) were prepared (Table 1), being light-cured under four different thicknesses and opacities of a ceramic based on lithium disilicate (IPS e.Max Press – Ivoclar-Vivadent), also considering three different light emitting diode (LED) units. For each of the three light-curing units (Valo – Ultradent, Radii-Cal – SDI, and Bluephase II – Ivoclar Vivadent) tested, 96 specimens were obtained, divided into 16 experimental conditions: four opacities (HT – High Translucency, LT – Low Translucency, MO – Medium Opacity, and HO – High Opacity) and four thicknesses (0.3 / 0.8 / 1.5 / 2 mm) (n = 6). G1-HT/0.3mm; G2-HT/0.8mm; G3-HT/1.5 mm; G4-HT/2.0mm; G5-LT/0.3mm; G1-HO/0.8mm; G12-MO/2.0mm; G13-HO/0.3 mm; G14-HO/0.8 mm; G15-HO/1,5 mm; G16-HO/2.0mm

Resin Cement	Composition				
	Methacrylate monomers				
	(UDMA, BIS-EMA, BIS-GMA, TEGDMA)				
	Photoinitiators				
AllCem Venner Trans	(Camphorquinone, peak absorption between 400-500 nm)				
Light-curing	Coinitiators				
	Stabilizers				
	Pigments				
	Silanized barium-aluminum-silicate glass particles and silicon dioxide				

 Table 1. The composition of the light-curing resin cement AllCem Venner Trans (FGM, Dentscare LTDA, Joinville-SC, Brazil)

All specimens were made using a split stainless steel matrix with 3-mm diameter and 1-mm thickness. On this metallic matrix, filled with a single increment of the resin cement, a polyester strip was placed, followed by a glass coverslip and a weight of 500 mg, left for 30 seconds to drain the excess material. Then, the weight and the glass coverslip were removed and the specimen was light-cured for 40 s, through the polyester strip, for the control group. For the other groups, the resin cement was light-cured under a piece of ceramic material that varied its respective opacity and thickness, according to each experimental condition. This sequence was applied, in an identical manner, to the three light-curing units (Valo, Radii-Cal and Bluephase II) (Table 2).

	Operation condition	ions LED devices				
	Valo – Ultradent	Radii-Cal – SDI	Bluephase II – Ivoclar Vivadent			
Light intensity	1400 mW/cm ²	1200 mW/cm ²	1200 mW/cm ²			
Wavelength range	385-515 nm	440-480 nm	385-515 nm			

 Table 2. Description of light-curing units (LCU) used in this study, considering their respective light parameters (power intensity and wavelength)

Evaluation of water sorption and solubility

After preparation, the specimens were measured using a digital caliper with 0.01 mm precision (Mitutoyo, Suzano – SP, Brazil). Means of diameter duplicates were calculated for each specimen, to determine the radius (r), height (h), and the individual volume. Following the ISO 4049 specifications¹⁷, the specimens were placed in a desiccator and transferred to an incubator at 37°C, for preconditioning. After 24hours, the specimens were weighed repeatedly, in an interval of 24 hours, until a constant mass (M1) was reached, on an analytical scale (Shimadzu, mod. AUW220D, Barueri – SP, Brazil), with an accuracy of 0.0001 of 1g. This stabilization was verified when the variation of the values of M1 was lower than 0.2mg, in a period of 24h, for each specimen. The specimens were individually identified and stored in an incubator at 37°C (Quimis, Diadema – SP, Brazil), being kept in a container with silica gel, for desiccation, for seven consecutive days.

After stabilization of M1, the specimens were immersed in 2 ml of distilled water (pH 7.2) and again stored at 37 °C, where they remained for seven consecutive days. Afterwards, the specimens were removed from the distilled water, dried with absorbent paper (Sorella, Canoinhas-SC, Brazil), and weighed again on an analytical scale to obtain the mass (M2). After M2 registration, the specimens were stored individually in the incubator at 37°C, for desiccation.

All specimens were weighed, repeatedly, at 24h intervals, until a constant mass (M3) was reached, considering a variation lower than 0.2mg for each specimen.

After acquiring all the mass values of the specimens, water sorption (So) and solubility (Sol) were calculated using the following formulas:

$$So = m2 - m3/V$$
 (1)

Sol = m1 - m3/V

Where M1 is the constant mass, in μq , found before immersion in water; M2 is the mass, in µg, after immersion in water for 7 days; M3 is the constant mass, in µg, after desiccation; and V is the volume of the specimens in mm³.

Statistical analysis

The collected data were tabulated and evaluated for their homogeneity and normality, with Levene and Shapiro-Wilk tests being applied, respectively, with a 5% significance level, for each of the variables (Water sorption and Solubility). Considering the assumptions for the application of the parametric tests, an Analysis of Variance with 3 factors was applied: 1. Light-curing unit in 3 levels (Valo, Radii-cal, and Bluephase); 2. Ceramic thickness in 4 levels (0.3, 0.8, 1.5, and 2.0 mm); and 3. Opacity in 4 levels (HT – High Translucency, LT – Low Translucency, MO – Medium Opacity, and HO – High Opacity). Tukey's test was used as post hoc.

Results

The statistical analysis showed that the thickness and the degree of translucency of the ceramic interfered in the water sorption and solubility values of the light-cured AllCem resin cement, regardless of the light-curing unit used. As a result of water sorption (Table 3), it was found that the greater thickness of the lithium disilicate ceramic (2.0 mm) increased the water sorption values when statistically compared to the thicknesses of 0.3 mm and 0.8 mm. Similarly, medium and high opacity ceramics (MO and HO) contributed to higher values of water sorption compared to high translucency (HT) ceramics. The higher opacity lithium disilicate (HO) ceramics resulted in higher water sorption values of resin cement than the values showed by high and low translucency ceramics (HT and LT). The less thick ceramic (0.3 mm) with greater opacity (HO) showed increased water sorption values, regardless of the light-curing unit tested. The same behavior was found for high translucency (HT), thick (2.0 mm) ceramic. The type of the light-curing unit tested did not influence the water sorption values of the resin cement, with no statistically significant difference between them.

Table 4 shows the results obtained by analyzing the solubility data of the resin cement. It was found that, as with sorption, the greater thickness of the lithium disilicate ceramic (2 mm) increased the solubility values, compared to the values obtained with the thickness of 0.3 mm, regardless of the light-curing unit tested. Likewise, the difference for the other thicknesses was statistically significant with the increase in the opacity of the ceramic. The more translucent the ceramics (HT and LT), the lower the solubility values of the resin cement compared to the values obtained with the higher opacity ceramic (HO), for the three light-curing units tested. Similarly to water sorption, the type of light-curing unit, by itself, did not affect the solubility values, with no statistically significant difference between them.

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	1Ac	†30.3±3.9Abc	†33.6±3.1Ab	†41.1±3.9Aa	†29.0±3.2Ac	†31.0±2.4Abc	†32.7±2.9Ab	†44.0±4.0Aa	†26.6±3.2Ac	†30.6±3.6Ab	†33.5±3.1Ab	†43.1±4.2Aa

Different capital letters indicate difference between thicknesses in the same opacity and light curing (column). Different lowercase letters indicate difference between opacities in the same thickness and light curing (row). Different symbols (t, *, t) indicate a difference among light-curing units for the same thickness and light curing (row). Different symbols (t, *, t) indicate a difference among light-curing units for the same thickness and light curing (row). = 0.05). Table 4 - Solubility values of the light-cured resin cement considering four different thicknesses (0.3 / 0.8 / 1.5 / 2 mm) and opacities (HT – High Translucency, LT – Low Translucency, MO – Medium Opacity, and HO – High Opacity) of the ceramic lithium disilicate (IPS e.Max Press – Ivoclar-Vivadent) and three light-curing units (Valo; Radii-cal and Bluephase).

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LIGHT-CUR RADII	CERAMIC	Ц	†3.3±0.5Bbc	†3.6±0.5Bbc	†3.9±0.6Bc	†5.2±0.5Ac	nacity and lic
		노	†3.2±0.5Bc	†3.4±0.5Bc	†3.6±0.5Bc	†4.4±0.3Ad	n the same o
		우	†4.1±0.4Ca	†4.3±0.4Ca	†5.1±0.4Ba	†7.5±0.5Aa	thicknesses i
VALO		МО	†4.0±0.4Ba	†4.0±0.4Bab	†4.4±0.6Bb	†6.1±0.6Ab	nce hetween
		Ц	†3.3±0.4Cb	†3.5±0.3BCbc	†3.9±0.6Bbc	†5.3±0.5Ac	linate differen
		노	†3.1±0.4Bb	†3.4±0.4ABc	†3.4±0.4ABc	†3.9±0.5Ad	vital lattars inc
THICKNESS		0.3	0.8	1.5	2.0	Different car	

μ same thickness and light curing (row). Different symbols (t, *, t) indicate a difference among light-curing units for the same thickness and opacity (ANOVA-3 factors / Tukey's test; α cumil (countie). Different = 0.05).

Discussion

The mechanical properties of resin cement can be affected by the water sorption and solubility that occur in an aqueous environment, such as in the oral environment⁵. Studies show that increasing the thickness and opacity of the ceramic restoration reduces the passage of light during the photoactivation of cement, compromising the performance of the material^{1,8,10,18}. In this study, the experimental hypothesis was partially accepted, since the ceramics of greater thickness and opacity interposed during light-curing procedures increased the values of water sorption and solubility of the resin cement. However, this increase was not gradual as the opacity and/or thickness of the ceramic increased, with similar behaviors being observed among ceramics of different thicknesses. The tested light-curing units were not different.

In our study, thicknesses of 0.3 and 0.8 mm resulted in similar values to each other and significantly lower for water sorption and solubility, compared to the thickness of 2 mm, for all three light curing units tested. These results corroborate the data reported by Runnacles et al.⁸ (2014), who has shown that the effect of light attenuation by ceramic veneers is not significant in thicknesses up to 1.0 mm. Previous studies have also shown that the thinner the ceramic material interposed between the resin cement and the light source, the greater the degree of conversion of the resin material^{18,19}. According to Calgaro et al.²⁰ (2013), the increase in thickness is a key factor in attenuating the light emitted by the light-curing unit, since they observed that the polymerization decreases as the thickness increases.

In this study, increasing the thickness of the ceramic to 2.0 mm also significantly increased the water sorption and the solubility of the resin cement under it. Similar observations were reported in a previous study, where the resin microhardness and roughness was reduced when the thickness of the ceramic increased from 1 mm to 2 mm²¹. The explanations for this change in properties of the light-cured resin cement under thicker ceramics may be related to the reduction of the light transmitted by the light-curing unit when crossing a 2 mm thick ceramic²². According to Liebermann et al.²³ (2018), light transmittance is inversely related to the thickness of the ceramic, which will be crossed by the light beam. That is, the thicker the material, the lower the transmittance of that light^{22,23}. Transmittance can be defined as the amount of light that passes through a material, part of which is reflected or absorbed²³. If a small part of this light is scattered and most of it is transmitted through the material, higher transmittance values will be achieved^{22,23}. In a ceramic material, the light ends up being too dispersed and diffusely reflected, generating an opaque appearance^{22,23}. It should also be noted that more translucent materials show changes in light transmittance due to the variation in thickness, thus, even translucent materials, when thicker, reduce light transmittance^{22,23}. This may explain the fact that the interposition of a translucent ceramic (HT), with a thickness of 2.0 mm, also results in an increase in the values of water sorption and solubility of the light-cured cement under it in this study.

As well as the thickness, the degree of translucency of the ceramic laminate has a strong influence on the polymerization of the resin cement under the ceramic lami-

nat^{1,10}. In this study, when the HT and LT ceramics were used with the same thickness, the resin cement showed the lowest values of water sorption and solubility, for the three light units tested. This influence of the degree of translucency of the ceramic can be explained by the microstructure of the ceramic material, especially its crystalline phase, which tends to present differences in the transmittance and dispersion of light, affecting light transmission and, consequently, the light irradiance that reaches the resin cement underlying the laminate⁴. Leal et al.¹ (2016) observed that ceramics with lower translucency (more opaque) limit the passage of light emitted by the light-curing units. Likewise, Calgaro et al.²⁰ (2013), when testing different types of ceramics, observed that, among the HT, LT, and MO ceramics, the best performance in the degree of conversion of the underlying resin cement was achieved by HT ceramics. The results found in our study corroborate these observations, since the resin cement under ceramics with a low level of translucency (MO and HO) showed greater water sorption and solubility. The less translucent ceramics are often used to mask teeth with severe stains or with significant color differences between substrate and final color of the restoration^{1,10}.

When correlating the thickness and translucency variables of the ceramics tested in this study, it can be seen that, regarding thickness for ceramics up to 1.5 mm, in HT and LT opacities, no significant differences were observed for water sorption and resin cement solubility. The increase in the thickness of the ceramic piece to 2.0 mm resulted in significant increases in sorption and solubility for all degrees of translucency tested, confirming the care with measuring ceramic thickness during adhesive cementation procedures with light-curing resin cements. De Jesus et al.⁴ (2020) also showed that ceramics with thickness higher than 1.5 mm reduce the values of degree of conversion and microhardness of the resinous material below to ceramic restoration. The association of 2.0 mm thickness with higher opacities (MO and HO) increased the values of water sorption and solubility even more, probably due to the greater impairment of light transmission through the respective ceramic pieces. Concerning the opacity variable, for the HO ceramic, all thicknesses resulted in higher values of water sorption and solubility, except the minimum thickness of 0.3 mm. HO ceramics with thicknesses of 1.5 and 2.0 mm make the tested diffusion dynamics values even more critical. Higher values of water sorption and solubility of resin cements under thicker and more opaque ceramic laminates probably come from a lower degree of conversion and less microhardness of these cements, after polymerization^{4,9,22,23}. With the increase in the thickness and opacity of the ceramic, there is a decrease in the passage of light to reach the resin cement, reducing its degree of conversion and its microhardness^{4,9}.

AccordingtotheAmericanDentalAssociationspecification,thesorptionofresinousmaterials must be less than 40 lg/mm³ and the water solubility must be less than 7.5 lg/mm³, for a storage period of seven days²⁴. In this study, when the resin cement was light-cured under the 2-mm thick ceramic piece with high opacity (HO), it exceeded this acceptable water sorption limit for the three light-curing units tested: Valo (41.1 lg/mm³), Radical (44.0 lg/mm³), and Bluephase (43.1 lg/mm³). The same occurred for the solubility, with values above the limit for all LED units: Radical (7.8 lg/mm³), Bluephase (7.7 lg/mm³), and Valo (7.5 lg/mm³). Therefore, the association of high opacity with a 2.0mm ceramic tile thickness demonstrates a detrimental effect on the physical properties of diffusion dynamics of resin cement, and should be considered in clinical cementation procedures. In their study, Leal et al.¹ (2016) identified mean values higher than the acceptable limit both for water sorption and solubility of light-cured resin cement, when ceramic pieces of medium and low translucency were positioned on the resin cement. On the other hand, when highly translucent surfaces are used, values similar to the control conditions were found, indicating no significant influence to light transmission, which allowed an adequate conversion of resin monomers^{25,26}. When a light-curing material does not receive the appropriate amount of light energy, an impaired formation of free radicals that initiate polymerization and a lower degree of conversion of the polymer network are observed²⁷.

The resin cement used in this study has low viscosity, and, in its composition, high and low molecular weight monomers (Table 1). It is known that the composition of the organic matrix of resin cements can influence water sorption and solubility⁵. Lower molecular weight monomers, such as TEGDMA (triethylene glycol dimethacrylate) or UDMA (urethane dimethacrylate), are mixed with higher molecular weight monomers, such as Bis-GMA (bisphenol A-diglycidyl dimethacrylate), to promote less viscosity, changing the material handling properties and collaborating with the technical cementation procedures^{5,13}. However, monomers of lower molecular weight also have a more hydrophilic nature, which may allow greater diffusion dynamics of resin cements²⁸⁻³⁰. The hydrophilicity of these monomers can generate undesirable clinical consequences, such as microleakage, susceptibility to degradation, discoloration and decreased mechanical properties, postoperative sensitivity, and recurrent caries^{1,8,31}. It is believed that water gain is related to the composition of the material, the content, concentration and type of inorganic fillers, as well as the size and nature of the particles¹. Therefore, the water sorption and solubility values of the resin cement used in this study may also have been influenced by the hydrophilicity characteristics of the monomers incorporated in its matrix. In our study, the behavior of this cement was not compared to that of other cements, so that a more accurate assessment of the influence of this composition could be considered. Therefore, in future research, comparative investigation between different cements is recommended.

In this study, three high power light-curing units were tested, with no significant difference between them. Light intensity, irradiance, and light application time are factors that may be associated with the conversion of resin monomers from resinous material¹⁸. For some authors, variations in the power of the light emitting device used can directly affect the mechanical properties of the material, showing the need to work at maximum intensity¹. It should be noted, however, that the use of high energy densities, per se, is not directly related to higher degrees of conversion³². Other factors should be considered, such as the use of a light-curing unit with an appropriate wavelength, to sensitize photoinitiators to the resin material used³³. In view of the need for the emergence of clearer resin materials for cosmetic procedures in dentistry, alternative photoinitiators were inserted in their composition, changing the peak wavelength absorption used for camphorquinone³⁴. Therefore, some LED units were created with polywave technology, which allows them to emit a broader wavelength spectrum, photosensitizing all the photoinitiators present in the composition of the material, unlike monowave LED units, which normally only reach camphorquinone, since they have a more limited spectrum^{34,35}. In this study, two of the light-curing units tested are considered polywave (Bluephase and Valo). However, the absence of difference between them can be justified by the composition of the resin cement, which has a photoinitiator compatible with the wavelength spectrum emitted for all three light-curing units. This highlights the need for including different resinous materials, with different compositions and photoinitiators, in future studies.

In addition, the presence of a collimated light beam seems to have a considerable influence on the light-curing of resinous materials, especially when a barrier is placed between the light source and the resinous material, as in the case of ceramic restorations^{12,33}. The linear orientation of the light beams prevents this light from dispersing, which favors the delivery of energy density to the restorative material^{12,33}. Considering the exposure time factor, Archegas et al.²⁵ (2012) observed that the polymerization of resin cements for 40s through an opaque ceramic resulted in a lower degree of conversion than through a translucent ceramic. Also, a time of 120s resulted in similar degree of conversion values for opaque and more translucent ceramic requires greater exposure to irradiated light, but even so, there is a limit of thickness and opacity so that the appropriate polymerization is obtained at the best irradiation conditions. The authors emphasized that the photoactivation time should not be less than 40s. In this study, the exposure time used was standardized at 40s, for all three light-curing units tested, and there was no statistically significant difference among them.

Based on the results found in the present study, the factors opacity and thickness significantly affected the water sorption and solubility of the light-cured resin cement underlying the ceramics; therefore, they need to be evaluated by the professional. Often, ceramic pieces have variable thicknesses and need to be measured and evaluated when opting for adhesive cementation with light-curing resin cements, since the use of a ceramic restoration may not ensure effective light transmission through it, if its thickness is equal or greater to 2.0mm. Another factor that must be considered is the light-curing unit used, which must have adequate characteristics, especially regarding a high intensity of emitted power, for effective photopolymerization.

In conclusion, the light-curing resin cement under lithium disilicate ceramics with a thickness equal to or greater than 2.0 mm and/or high opacity presents high water sorption and solubility. The greater degree of opacity (HO) increased values of water sorption and solubility of resin cement in all tested thicknesses, regardless of the high-power light curing units tested.

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