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Water Sorption and Solubility of Luting Agents Used Under Ceramic Laminates With Different Degrees of Translucency

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Clinical Relevance

Degrees of translucency in a restorative material are important for masking tooth color alteration. Clinicians must be aware of the relationship between a decrease in translucency and loss of light penetration to avoid the clinical degradation of an improperly cured luting material.

SUMMARY

Purpose: The aim of this study was to evaluate the effect of low-thickness ceramic laminate translucency on water sorption and solubility in resin luting agents.

Methods and Materials: Ceramic slides (15×0.7 mm) were generated using lithium disilicate (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtenstein) that were A1 in color and had decreasing degrees of translucency (high, medium, and low). A slide of transparent glass of similar size was used as the control. Under each slide, 15 specimens (8×0.5 mm) of differing composite materials from the same manufacturer (3M ESPE Dental Products, St Paul, MN, USA) were prepared (n=5): light-cured resin cement (RelyX Veneer); dual-cured resin cement (RelyX ARC); and flowable composite (Z350XT Flow). To evaluate the loss or gain of mass, the specimens were dried until a constant mass was reached. Then, they were immersed in water for seven days and weighed immediately following removal from water. Subsequently, the speci-

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mens were dried again until a constant mass was obtained. The mass measurements were used to calculate the water sorption and solubility. Statistical analyses were carried out using a two-way analysis of variance and the Tukey test.

Results: Under the high-translucency ceramic slides, all of the luting agents showed similar performance regarding water sorption; the flowable composite resin and the light-cured resin cement had the lowest solubility values. Under the medium- and low-translucency surfaces, the dual-cured resin cement and the flowable composite resin showed better performance with respect to water sorption and solubility.

Conclusions: In the case of high-translucency laminates, luting agents with different activation methods might be used. However, even in thin sections, decreasing the translucency of the laminate led to significant loss of light penetration, indicating a decreased likelihood of the physical activation of the resin cement.

INTRODUCTION

The search for beautiful and harmonious smiles has increased the frequency of the use of ceramic laminates, which are an esthetic alternative that may involve minimal tooth preparation.[1,2] The cementation protocol is of fundamental importance in these minimally invasive preparations because the success of ceramic restorations will be determined in large part by obtaining a strong and durable bond among the cement, ceramic material, and dental tissues.[3]

For the cementation of ceramic laminates, light-cured (photoactivated) or dual-cured resin agents are commonly used, largely due to their ability to adhere to dental tissues and their satisfactory mechanical properties.[4] Among the light-cured resins, flowable composites have also been presented as options because their molecular composition, which is similar to a hybrid compound, allows for good mechanical resistance.[5,6]

A lower concentration of tertiary amines in flowable composites and light-cured resin cements appears to offer greater color stability, allowing better long-term esthetic results.[5,6] However, light-cured materials rely on visible light and an efficient photoinitiator system for polymerization of the materials to occur in an effective manner so that their maximum physical and mechanical properties can be achieved.[7]

With improvements in restorative techniques, higher esthetic refinements have been developed, resulting in the production of ultrafine ceramic laminates that are associated with minimum dental wear.[1] However, a significantly reduced thickness may hinder the ideal masking of color alterations in enamel and dentin. Therefore, ceramics of greater opacity become an option to allow more satisfactory esthetic results. However, increased thickness has been associated with alteration in the passage of light through the ceramic,[8] and the translucency of the laminate might also interfere with this process.

Clinically, the margins of indirect restorations are often placed close to the gingival sulcus and in contact with the sulcular fluid. Failures in these restorations can be observed due to deficiencies of polymerization and impairment of the mechanical properties of the cement related to the influence of humidity.[9,10] Most of the monomers used in dental resin materials can absorb water and chemicals from the environment and also release components into the surrounding environment.[11] Both the fluid uptake into the resin phase and the dissolution of the composite may have detrimental clinical consequences. An inappropriate polymerization might influence the degree of degradation of a composite material as well as microstructural and molecular aspects, presence of pendant hydroxyl groups capable of forming hydrogen bonds with water, degree of cross-linking of the continuous matrix, presence of residual water-attracting species, and type, dimension, volume, diffusivity, and solubility of filler particles.[10] Therefore, understanding the dynamics of diffusion in resin cements by considering the properties of water sorption and solubility is important for predicting the resins' clinical behavior, especially their stability, because these factors have direct effects on the longevity of adhesively cemented restorations.[10,11]

In view of the aforementioned considerations, there is a hypothesis that the translucency of a ceramic, even at a low thickness, will interfere with the polymerization of the cements to such a degree that it will change the water sorption and solubility of this material; similarly, the type of luting agent might also influence the outcome. Thus, the objective of this study was to evaluate the water sorption and solubility of light-cured or dual luting agents using ceramic slides of different translucencies.

Table 1: Luting Materials Used and Their Compositions	
Luting Material	**Composition**
Dual-cured resin cement, RelyX ARC (3M ESPE Dental Products, St Paul, MN, USA)	Bis-GMA and TEGDMA monomers. Particles of zirconia/silica with an average size of 1.5 μm. Paste A: pigments and tertiary amine. Paste B: benzoyl peroxide. Filler loading 68% by weight.
Light-cured resin cement RelyX Veneer (3M ESPE Dental Products, St Paul, MN, USA)	Bis-GMA and TEGDMA monomers. Particles of zirconia/silica and colloidal silica. Average particle size of 0.6 mm. Filler loading 66% by weight.
Flowable composite resin Z350XT Flow (3M ESPE Dental Products, St Paul, MN, USA)	Bis-GMA, TEGDMA, and Bis-EMA monomers. Silica nanoparticles of 75 nm, zirconia nanoparticles of 5-10 nm, zirconia/silica nano agglomerates with aggregate particle size ranging from 0.6-1.4 μm. Filler loading 65% by weight.

Abbreviations: Bis-EMA, bisphenol-A ethoxylated dimethacrylate; Bis-GMA, bisphenol-A glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate.

METHODS AND MATERIALS

Preparation of Specimens

Following the manufacturer's recommendations, ceramic slides of lithium disilicate (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtenstein) were fabricated to a 0.7-mm thickness and a 15-mm width in shade A1 and with decreasing degrees of translucency, including high (H), medium (M), and low (L). A glass slide of the same dimensions was used as a control to simulate the maximum passage of light energy to the luting agent.

Then, a matrix of polyvinyl siloxane (mA) was prepared (Elite, Zhermack, Badia Polesine, Italy) with an internal orifice, which was 0.5 mm in depth and 8 mm in diameter. This mold was used to accommodate the cement during the preparation of the specimens subjected to water sorption and solubility testing.

Another matrix of polyvinyl siloxane (mB) was prepared to adapt to the ceramic slides. The function of this device was to prevent the dissipation of light at the time of polymerization and also to prevent the interference of external light.[8]

The luting agents and their formulations are described in Table 1. Materials with similar colors (A1/light yellow) were selected. The 12 experimental groups (n=5) were formed as described in Figure 1.

To prepare each specimen, each luting agent was inserted into the matrix of polyvinyl silicone (mA), and a strip of polyester was placed over it to accommodate the material and maintain a smooth and even surface. Then, a ceramic slide and the other mold (mB) were positioned on top of this assembly. A glass slide was positioned on top of the second mold to force excess material outward for removal. No hand pressure was exerted. Finally, polymerization was performed with a light-emitting diode source (Radii Plus, SDI, Victoria, Australia) for an exposure time of 40 seconds, intensity of 1500 mW/cm^2, and peak wavelength of 470 nm (Figure 2). The light intensity of the light-emitting diode source was monitored using its built-in radiometer.

Water Sorption and Solubility Evaluation

After the removal of the matrix, the specimens were placed individually in a dark environment to prevent further polymerization until they were ready for water sorption and solubility testing based on ISO 4049: 2000 specifications.[12]

After preparation of the 60 specimens (n=5 per group), their thicknesses were measured using a digital caliper with a precision of 0.01 mm, and the measurements were used to calculate the volume of each specimen (mm^3). Both diameter and thickness were measured following a standardized method. A mean diameter for each specimen was calculated from two measures at right angles to each other. A mean thickness for each specimen was calculated from four measures performed at equally distributed points on the circumference.

Shortly thereafter, all the specimens were placed in a desiccator and transferred to an incubator at 37°C for preconditioning. After 24 hours, the speci-

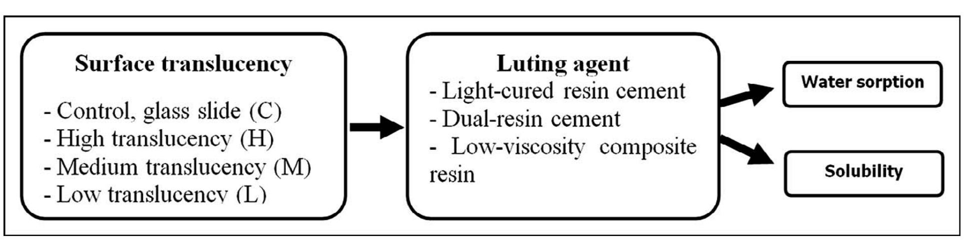

Figure 1. *Distribution of the experimental groups.*

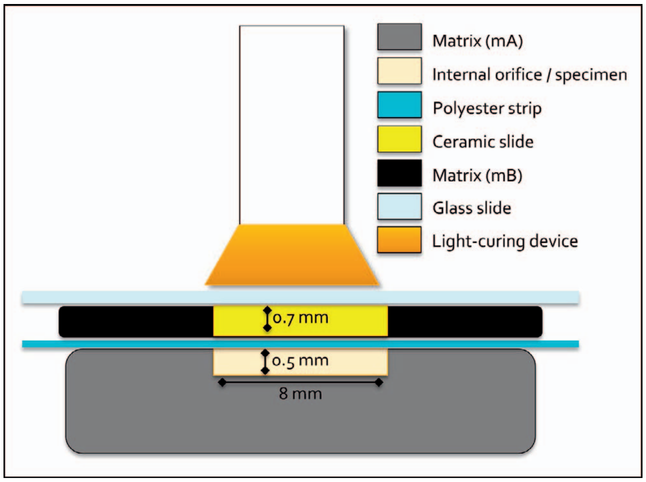

Figure 2. *Illustration of the experimental apparatus for the preparation of specimens.*

mens were weighed on an analytical balance (Analytical Plus, Ohaus Corporation, Florham Park, Switzerland) with an accuracy of a tenth of a thousandth of a gram.

The specimens were weighed repeatedly at intervals of 24 hours until a constant mass was reached (m1; less than 0.2-mg variation in a 24-hour period). After stabilization of the mass at m1, the specimens were stored individually in closed flasks containing 2 mL of distilled water (pH 7.2) in a 37°C incubator for seven days. Following the storage period, the specimens were weighed again to determine the value of m2. For this step, after removal from water, the specimens were washed in running water, and excess liquid was removed with absorbent paper until moisture could no longer be observed. The weight was annotated (m2), and the specimens were positioned in dry and open flasks and then placed in a desiccator containing silica gel in an incubator at 37°C to eliminate the absorbed water. The samples were weighed daily until reaching a constant mass (m3), as described for m1 and m2. The initial mass determined after the first desiccating process (m1) was used to calculate the mass variation during the seven days of storage in water. The water sorption (WS) and solubility (Sol) for the seven days of storage in water were calculated using the following formulas:

$$WS = (m2 - m3)/V; \qquad Sol = (m1 - m3)/V,$$

where, m1 is the mass of the sample in micrograms before immersion in distilled water, m2 is the mass of the sample in micrograms after immersion in distilled water for seven days, m3 is the mass of the sample in micrograms after being conditioned in a desiccator with silica gel, and V is the volume of the samples in millimeters cubed.[11]

Statistical Analysis

Exploratory analyses of the water sorption and solubility data (μg/mm^3) were performed to verify the parameters of the analysis of variance (ANOVA). The inferential statistical analysis was performed using a two-way ANOVA and a Tukey multiple comparison test with the statistical software SAS, version 9.1 (SAS Institute Inc, Cary, NC), with a significance level of 5%.

RESULTS

Table 2 shows the means and standard deviations of the water sorption and solubility data obtained under the experimental conditions tested in this study. The statistical analysis found that the two-way interaction between the translucency of the ceramic and the luting agent was significant for both

Table 2: *Mean Values (Standard Deviation) of Water Sorption and Solubility (μg/mm^3)[a]*

Luting Agent	Translucency of the Ceramic			
	Glass Slide (Control)	High Translucency (H)	Medium Translucency (M)	Low translucency (L)
Water sorption (WS)				
Dual-resin cement	26.22 (2.93)Ab	30.11 (1.73)Ab	27.07 (2.31)Bb	33.86 (4.77) Ba
Light-cured resin cement	27.78 (1.90)Ab	25.90 (3.93) Ab	38.40 (3.83) Aa	42.55 (5.04)Aa
Flowable composite resin	27.36 (2.11) Aa	24.87 (2.23) Aa	24.48 (1.99) Ba	28.32 (1.23) Ba
Solubility (Sol)				
Dual-resin cement	2.91 (0.12) Ab	6.86 (0.17) Aa	5.84 (0.46) Ba	7.87 (1.44) Ba
Light-cured resin cement	3.20 (0.31)Ab	3.50 (1.24) Bb	10.73 (1.63) Aa	12.60 (2.25) Aa
Flowable composite resin	2.96 (0.20)Ac	3.22 (0.20) Bc	6.29 (0.23)Bb	9.77 (0.69)Ba

[a] *Letters and symbols represent different means with statistical significance (two-way analysis of variance/Tukey test, α=5%). For each variable, capital letters compare the levels of the luting agent within each level of translucency of the ceramic. Lowercase letters compare the translucency of the ceramic within each level of the luting agent. Coefficient of variation: 10% (WS) and 16% (Sol).*

the water sorption ($p<0.001$) and the solubility ($p<0.001$) data. Thus, the interaction of each factor with the levels of the others was demonstrated, and this interaction was parsed using the Tukey test.

Controlling for the statistical interaction of the water sorption data, the luting agents showed similar patterns under the high-translucency and control (glass slide) surfaces. However, under the intermediate- and low-translucency surfaces, the light-curing resin cement showed a water sorption value that was statistically superior to the others. The data analysis also indicated the amount of influence that the type of surface had on water sorption for the dual and light-cured resin cements. Both presented higher amounts of water sorption for the low-translucency surfaces. However, the type of surface did not result in a statistically significant difference for the low-viscosity resin.

Among the solubility data, an interaction between the luting agent and the surface translucency was also observed. For the intermediate- and low-translucency ceramic specimens, the light-cured resin cement showed statistically superior values compared with the other resins. In the high-translucency ceramic, the light-cured agents showed the lowest solubility values, but in the control condition, the results were all similar. Comparison of the surfaces indicated a direct relationship between the low-translucency ceramic and the highest solubility values for all luting agents, whereas the high-translucency ceramic and the glass slide had the lowest values for the light-cured resin cement and the low-viscosity composite resin. For the dual-resin cement, only the control resulted in a lower solubility.

DISCUSSION

Inadequate polymerization of resin cements under ceramic restorations may be related to an insufficient amount of light radiation passing through the restorative material to reach and activate the monomers.[8] Among other factors, the amount of light transmitted for the conversion of resin cements may decrease depending on the optical characteristics of the ceramic materials, such as refractive index and translucency.[8,13,14]

A previous study indicated that resin cements can be light-cured under lithium disilicate–based ceramics with thicknesses of up to 2 mm without any interruption to the curing process.[15] Lee and others[13] evaluated the microhardness of light-cured and dual-resin materials under ceramics of different thicknesses and concluded that the smallest thickness exerts less influence on the properties of the materials. In the present study, ceramic slides had a thickness similar to conservative veneers (0.7 mm); thus, little interference from the lithium disilicate glass-ceramic was expected.

Nonetheless, in conservative preparations, ceramics with different degrees of translucency are commonly used to block possible interference by the color of the dental substrate with the final result of the restoration. Thus, the hypothesis of this study suggested that, even at a low thickness, more-opaque surfaces would be able to prevent complete polymerization of the luting agent, resulting in a low degree of conversion and interfering with the dynamics of diffusion (water sorption and solubility) of these materials.[5,8,16-18]

According to the results of this study, the water sorption and the solubility of the light-cured luting agents under high-translucency surfaces were similar to the control condition, indicating that this type of surface did not prevent the transmission of light and allowed proper conversion of the monomers.[5,15] In contrast, for the low- and intermediate-translucency surfaces, the light-cured resin cement showed water sorption and solubility values that were higher than those of the other resins. This finding is most likely related to the passage of light through the ceramic,[8] indicating that the degree of dilution of the resin cement components in water is higher when the compound is not properly light-cured.[19] When a light-cured material does not receive the appropriate amount of energy density, one would expect an impaired formation of free radicals to initiate polymerization and a lower degree of conversion of the polymer network.[20] Highly cross-linked polymers seem to be more resistant to dissolution, whereas linear polymers present more space and pathways for solvent molecules to diffuse within their structure.[20]

The gain of water seems to be significantly related to the material composition, content and concentration of inorganic fillers, and size and nature of the particles.[21,22] In the present study, the translucency of the surface did not significantly affect the water sorption of the flowable composite. This material differs from the light-cured resin cements because it has bisphenol-A ethoxylated dimethacrylate (Bis-EMA) associated with bisphenol-A glycidyl methacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) in the resin matrix. Bis-EMA is more hydrophobic, hampering the entry of water and, in combination with the nanoparticles of the flowable composite resins, may be associated with an improvement in the physical properties of the material.[19,23] Given that water sorption is a phenomenon in which water enters the resin matrix through direct diffusion via empty spaces that have been incorporated into the material,[21,22] it is possible that the presence of nanoparticles in the flowable composite resin filled such spaces, and the association with hydrophobicity of the resin matrix culminated in a decrease in the sorption values.

Although Archegas and others[5,6] analyzed different properties than the present study such as color stability, degree of conversion, and hardness, they observed the best results for flowable composite resins compared with other luting agents. In addition, the authors found that the opacity of the ceramic did not result in a statistically significant influence on the degree of conversion of the flowable composite resin.

In contrast, as with the light-cured resin cement, the flowable composite resin showed significantly higher solubility with the low- and medium-translucency ceramic slides. This phenomenon is characterized by the loss of resinous material components by dissolution, mainly of unreacted monomers during the polymerization of filler particles, resulting in a reduction in weight and volume.[21,24]

An explanation for this finding may be the poor quality of the polymers formed under conditions of low light passage, possibly leaving weak bonds that facilitate leaching. This hypothesis is supported by a previous study that showed that an inadequate conversion resulting from low light intensity can adversely affect the performance of the restoration.[5] Failures related to the polymerization reaction can result in the formation of a poorly structured polymer chain with easily breakable bonds.[25,26]

The dual-resin cement was used in this study as a control for the effect of the passage of light through the ceramic because chemical activation of this agent could compensate for the loss of light intensity.[8] However, according to the results of this study, even with the presence of the chemical activation system, the dual-resin cement suffered the negative effects of the limitation of the passage of light, indicating that proper photoactivation has a fundamental role in improving the physical properties of this material.

In a previous study comparing the hardness of luting agents in ceramics of different thicknesses and opacities, it was observed that even the chemical component of a dual-resin cement was not able to promote complete polymerization under an opaque surface,[15] resulting in a reduction of the mechanical properties of flexural strength, elastic modulus, and hardness. These results demonstrate the importance of light and photosensitive components in this cement.[6,7]

The light intensity is another factor that may be associated with the correct conversion of the monomers. Variations in the power of the device used will directly influence the mechanical properties of the material, demonstrating the need to work at maximum intensity.[14,17,25]

In most of the conditions tested, an association between the water sorption and solubility values was observed, corroborating a previous study.[18] Therefore, a possible directly proportional relationship between the amount of absorbed water and the amount of components leached may be assumed.

According to the American Dental Association's[27] specification No. 27, the sorption of resin materials must be less than 40 $\mu g/mm^3$ and the water solubility must be less than 7.5 $\mu g/mm^3$ for a storage period of seven days. In this study, only the light-cured resin cement showed a water sorption value significantly higher than the acceptable limit when it was under a low-translucency surface. Regarding solubility, the light-cured resin cement showed mean values higher than the acceptable limits when it was under medium- and low-translucency surfaces. In contrast, the flowable composite resin and dual-cured resin cement presented values above the acceptable limit only under the low-translucency surface. This result justifies careful attention in the cementation of ceramics that allow less passage of light.

In spite of the relevance of the reported findings, it should be considered that *in vitro* studies have some limitations. Important covariants, such as the thickness of the luting agent and changes in the pH of the oral cavity, were not simulated in this study, and thus the *in vitro* water sorption and solubility test are considered static.[28,29] Another limitation of this investigation was that materials from the same manufacturer were selected in order to standardize resin matrixes so compositional changes would not be of more importance than light exposure. Nevertheless, it should be considered that *in vitro* methods are good tools to generate clinical evidence due to the simplicity of implementation and the possibility of satisfactorily reproducing the oral environment.

In light of these findings, dentists need to be concerned with the color of the ceramic used as well as with its translucency because the latter will also interfere with the transmission of light to the luting agent, influencing the mechanical properties of these materials. Another consideration is that in spite of the strong commercial appeal of the restorative materials' industry, there are some products that might have a broader clinical application, such as the flowable restorative composites. These results are of fundamental importance; however, they do not preempt the implementation of new, long-term clinical studies.

CONCLUSIONS

Based on the observations described in the previous section, there is a relationship between ceramic translucency and luting agent water sorption and solubility for all materials tested. Therefore, for surfaces with greater translucency, the luting agents studied provide acceptable values of water sorption and solubility. In contrast, for surfaces with lower translucency, dual-cured resin cements or flowable composite resins should be preferred. These findings are preliminary, and further studies should be performed to confirm and expand our results.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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