The effect of thickness and translucency of polymer-infiltrated ceramic-network material on degree of conversion of resin cements

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PURPOSE. The aim of the present study was to determine the degree of conversion of light- and dual-cured resin cements used in the cementation of all-ceramic restorations under different thicknesses of translucent (T) and high-translucent (HT) polymer-infiltrated ceramic-network (PICN) material. MATERIALS AND METHODS. T and HT PICN blocks were prepared at 0.5, 1.0, 1.5, and 2.0 mm thicknesses (n=80). Resin cement samples were prepared with a diameter of 6 mm and a thickness of 100 μm. Light-cured resin cement was polymerized for 30 seconds, and dual-cure resin cement was polymerized for 20 seconds (n=180). Fourier transform infrared spectroscopy (FTIR) was used for degree of conversion measurements. The obtained data were analyzed with ANOVA and Tukey HSD, and independent t-test. RESULTS. As a result of FTIR analysis, the degree of conversion of the light-cured resin cement prepared under 1.5- and 2.0-mm-thick T and HT ceramics was found to be lower than that of the control group. Regarding the degree of conversion of the dual-cured resin cement group, there was no significant difference from the control group. CONCLUSION. Within the limitation of present study, it can be concluded that using of dual cure resin cement can be suggested for cementation of PICN material, especially for thicknesses of 1.5 mm and above. [J Adv Prosthodont 2020;12:61-6

KEYWORDS: Hybrid ceramic; Resin cement; Degree of Conversion; Fourier transform infrared spectroscopy (FTIR)

INTRODUCTION

In recent years, development in scanning and milling systems and the increase of chair-side applications have accelerated the production of new materials adapted for computer-aided design/computer-assisted machining (CAD/CAM) technology. Aesthetic restoration materials used in CAD/CAM are mainly ceramics and composite resins. In addition, materials are developed to combine the positive properties of both materials used in permanent restora-

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Degrees of conversion of composite resins are widely studied in the dental literature and the spectroscopic methods are the most preferred method to determine the degree of conversion of composite resins.\textsuperscript{9,11-13} Fourier transform infrared spectroscopy (FTIR) measures vibration frequencies of various bonds in molecules and gives information about functional groups in molecules.\textsuperscript{14} In addition, it is an advantage of these methods to work with samples similar to the clinical applications of resin cements.

Light-cure resin cements used for cementation of all-ceramic restorations have advantages such as adequate work time and ease of cleaning extra cement after cementation.\textsuperscript{15} However, the degree of conversion varies according to the amount of light that reaches the resin.\textsuperscript{16} To solve this problem, dual-cure resin cements are used in thick and low-translucent restorations. Although the additional chemical polymerization provides an advantage in these kinds of restorations, adequate conversion cannot be obtained without photopolymerization.\textsuperscript{17} The light transmitted through ceramic restorations can affect polymerization level, and the most common factors related to restoration causing this situation are thickness and translucency.\textsuperscript{18,19} For these reasons, the aim of this study was to evaluate the degree of conversion of light- and dual-cure resin cements used for translucent and high-translucent hybrid ceramic restorations of different thicknesses. The null hypothesis was that the increase in restoration thickness and translucency of Vita Enamic would not affect the degree of conversion of resin cements.

**MATERIALS AND METHODS**

The materials used in this study are shown in Table 1. From the blocks of hybrid ceramics (Vita Enamic, VITA Zahnfabrik, Bad Säckingen, Germany), high-translucent (HT - 2M2) and translucent (T - 2M2) samples with thicknesses of 0.5, 1.0, 1.5, and 2.0 mm were prepared using a low speed precision cutting machine (IsoMet Low-speed; Buehler, IL, USA) under water coolant. Ten blocks for each shade were used for preparing the specimens. 80 specimens were obtained and divided into eight subgroups according to two resin cements groups (n = 10). The obtained samples were polished with sandpaper (Atlas Zımpara, Istanbul, Turkey) under water then cleaned ultrasonically with distilled water. Finishing and polishing were done using a two-stage laboratory finishing kit (Technical Kit, VITA Zahnfabrik, Bad Säckingen, Germany). To obtain the proper thickness of block samples, they were measured with a digital caliper. (Mitutoyo Digimatic, Mitutoyo Corp., Kanagawa, Japan).

For standardizing the size of resin cement samples, a stainless steel block of 100 µm in thickness with a hole of 6.0 mm in diameter was used as a guide placed between blocks samples, and Mylar strip was used on glass to adjust the thickness. The hole on the metal guide was filled with resin cement and was covered with another Mylar strip. Ceramics block samples were placed on the hole with light pressure, and then polymerization processes were carried out according to the manufacturer’s recommendations. After the curing of resin cement, samples were carefully removed from the metal mold. Broken, cracked samples and samples with non-regular edges were not included in the study. After checking the thickness of all samples by digital calipers, and after making sure that all samples were 100 µm (± 10 µm) in thickness, FTIR analyses were performed. In control groups, instead of ceramic block samples on resin cement, only Mylar strip and another glass were placed and polymerization was carried out (Fig. 1).

**Table 1.** The materials used in this study

| Materials           | Manufacturer                      | Color   | Composition                                                                 | Lot No.  |
|---------------------|-----------------------------------|---------|-----------------------------------------------------------------------------|----------|
| Light cure resin cement | RelyX Veneer, 3M ESPE, St. Paul, MN, USA | A1      | Bis-GMA, TEGDMA, Silica and Ceramic Particles                              | N776174  |
| Dual cure resin cement | RelyX U200, 3M ESPE, St. Paul, MN, USA | A2      | Base paste: Fiberglass, Phosphoric Acid Methacrylate Esters, TEGDMA, Silano Treated Silica and Sodium Persulfate. Catalyst paste: Fiberglass, Substitute Dimethacrylate, Silane Treated Silica, P-Toluenesulfonate Sodium and Calcium Hydroxide. | 628965   |
| Hybrid ceramic      | Vita Enamic, VITA Zahnfabrik, Bad Säckingen, Germany | HT - 2M2 | Ceramic; SiO₂, Al₂O₃, Na₂O, K₂O, B₂O₃, ZrO₂, CaO                  | 41470   |
|                     |                                   | T - 2M2 | Polymer; UDMA, TEGDMA                                                   |          |

Fig. 1. Representative image of the standardization of resin cement samples.
Polymerization was performed by applying light for 30 seconds for light-cure resin cement and 20 seconds for dual-cure resin cement in accordance with the recommendations of the manufacturers. For the polymerization process, the wide-spectrum LED light curing device (VALO Cordless, Ultradent Products, Inc., South Jordan, UT, USA) was applied in high-power mode with a light power output of 1400 mW/cm². For the control group, instead of hybrid ceramic sample, only a glass and Mylar strip were used for measuring the conversion rate of resin cements obtained as a result of direct contacting with the light curing. For the measurement of degree of conversion, 180 specimens in total were prepared from both resin cements. All resin cement specimens were stored in light-proof containers for 24 hours at room temperature before FTIR analysis.

The degree of conversion of light and dual-cure resin cement specimens was performed by FTIR (Bruker, Billerica, MA, USA). The degrees of conversion of groups in the study were analyzed using a potassium bromide (KBr) tablet test, which does not have absorption in the infrared region. Hence, prepared resin cement samples were powdered and then mixed with KBr to obtain tablets. Next, these tablets were placed in the sample reading section of the spectroscopy device and spectroscopic graphics were obtained. The measurements were done in the range of 4000-500 cm⁻¹ in wavelength and at a 4 cm⁻¹ wavenumber resolution. One single measurement was done for each of the 180 samples in total.

In the spectrum obtained with measurements, aliphatic C=C bonds were determined and recorded as 1638-cm⁻¹ -wavelength absorption pick values and aromatic C=C double bonds as 1609-cm⁻¹ -wavelength absorption pick values.

The obtained data were applied to the formula below. In this way, the conversion level of each sample was calculated in percentage.

\[
DC (\%) = 1 - \frac{[C \text{ aliphatic} / C \text{ aliphatic}]}{[U \text{ aliphatic} / U \text{ aromatic}]} \times 100
\]

Statistical analysis was done with a software package (SPSS 18, Chicago, IL, USA). For normality analyses of data, Kolmogorov-Smirnov and Shapiro-Wilk were used. According to the results, one-way analysis of variance (ANOVA) and Tukey's HSD multiple comparison test were applied. For the statistical analysis of the differences between the resin cement groups of the same thickness, an independent t-test was used. The significance level of the statistical analysis was 0.05. Additionally, the power analysis was performed by using a software package (G*Power 3.1; Universität Düsseldorf).

**RESULTS**

The conversion degrees of resin cements are shown in Table 2. According to the obtained results, when restorative materials thickness increased, the degrees of conversion of light-cure resin cements decreased. It was found that degree of conversion of light-cure resin cements under TLC 1.5 mm, TLC 2.0 mm, and HTLC 1.5 mm were lower than the control group \( (P < .05) \). It was observed that the degree of conversion of dual-cure resin cement groups varied between \%84.04 ± 1.46 and \%80.66 ± 5.79, but there was no statistically significant difference among the groups \( (P > .05) \).

When the degrees of conversion of light- and dual-cure resin cements were compared, higher conversion levels were observed in the control group and TLC 0.5 and TLC 1.0 groups. However, the difference was not found to be statistically significant \( (P > .05) \). When TDC 1.5 and TDC 2.0 were compared with the light-cure groups, significantly higher degrees of conversion were observed \( (P < .05) \).

Similarly, when the degrees of conversion of resin cement samples prepared using HT ceramics of 1.5 mm and 2.0 mm thicknesses were compared, it was found that the degree of conversion percentage of dual-cure resin cement samples was higher. The degree of conversion of resin cement in HTDC 1.0 group was higher than 1.5 mm group and the difference between these two groups was statistically significant \( (P > .05) \).

**DISCUSSION**

According to the results of the study, the null hypothesis was partially rejected. It was concluded that the translucency of hybrid ceramic has no effect on the degree of conversion of light-cure resin cement to be used. However, the increase in the thickness of hybrid ceramic investigated in the present study caused a statistically significant decrease in the degree of conversion of light-cure resin cements, especially at 1.5 mm and above \( (P < .05) \). In addition, the results show that the degree of conversion of dual-cure resin cement is not affected by ceramic thickness and translucency \( (P > .05) \).
Translucency is one of the important factors in the selection of the material to be used, especially in the restoration of esthetic zone. Translucency of dental ceramics varies depending on microstructure and crystalline volume. It is known that if the crystals in the structure of the ceramic are smaller than the wavelength of visible light, the material will be seen as more transparent. Some studies have shown that translucency decreases with increasing restoration thickness. However, in the same studies, it was reported that the materials with a high glass matrix ratio were not affected by the increase in thickness. This situation has been stated to develop as a result of the decrease in the porous structure and the prevention of light scattering. Heffernan et al. stated that the difference in translucency of ceramics of similar thickness results from a structurally different proportion of crystalline composition. The microstructure of the hybrid ceramic used in the present study is similar to that of glass ceramics, which makes the light transmittance higher than the materials having polycrystalline structure. Due to the low porous structure of the hybrid ceramic, it was concluded that translucency did not affect the degree of conversion of resin cements.

The results of the present study show that the increase in the thickness of the hybrid ceramic used in the present study affects the degree of conversion of light-cure resin cements. Runnacles et al. investigated the degree of conversion of light-cure resin cements under ceramics of different thicknesses. According to the results of their study, the degree of conversion of the resin cements prepared under 0.5- and 1.0-mm-thick ceramic samples were similar to those of the control group, but there was a statistically significant decrease in the degree of conversion at thicknesses of 1.5 mm and above. Researchers have reported that light-cure resin cements exhibit low conversion degrees, especially in different all-ceramic restorations prepared over 1.0 mm in thickness. In the present study, light-cure resin cement showed a decrease in degree of conversion with increasing thickness, and this decrease was found to be statistically significant in groups prepared similarly to those prepared by Runnacles et al. This is thought to be due to the thickness increase affecting the amount of light reaching the resin cement.

Dual-cure resin cements are used to provide secure cementation with additional chemical polymerization, especially in restorations where the light transmittance is low. However, in the absence of light activation, chemical polymerization alone is not sufficient. Kilinc et al. evaluated the degrees of conversion of dual-cure resin cements prepared using ceramics of (1 - 4 mm) different thicknesses. The degree of conversion of dual-cure resin cements prepared at thicknesses up to 3.0 mm were reported to be similar to those of the control group. Similar studies indicated that an increase in ceramic thickness by 2.0 mm does not affect the degree of conversion of dual-cure resin cement. Similar to previous studies, in the present study, increasing the thickness of the hybrid ceramic to 2.0 mm did not affect the degree of conversion of dual-cure resin cement. It has been concluded that this situation is due to dual-cure resin cements having fast developing photoactivation with light application and a slow progressing chemical polymerization process.

It has been stated that the degree of conversion of resin cements is affected by factors such as the color, transparency, and thickness of the restorative materials, the chemical structure and filler amount of the resin cement, the power output and application time of the light irradiation, the air bubbles remaining during mixing, and the ambient temperature. In the present study, samples were prepared under the standardized conditions such as ambient temperature, the power output of light source, and irradiation time. In a previous study evaluating the effect of color and translucency of restoration on the degree of conversion of resin cements, it has been pointed out that the translucency of the material was a more effective factor compared to its color. So, the present study evaluated the effect of translucency of restorative materials on degree of conversion of resin cement rather than the color. It was found that the amount of inorganic particles contained in composite resin cements was a factor affecting polymerization shrinkage and conversion degree. In our study, one light cure and one dual cure resin cement were used. The limits of our study include not using resin cement with different filler ratio.

**CONCLUSION**

Within the limitations of present study, the following conclusions can be made:

The use of both light- and dual-cure resin cements is suitable for the cementation of hybrid ceramic restorations used in the present study up to 1.0 mm in thickness, according to the preference of clinician.

The use of dual-cure resin cement for the cementation of hybrid ceramic restorations over 1.0 mm in thickness is a more favorable choice.

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