



Original article

Influence of polymerization time on properties of dual-curing cements in combination with high translucency monolithic zirconia



Mario Alovisi^a, Nicola Scotti^{a,b,*}, Allegra Comba^c, Elena Manzon^b, Elena Farina^{d,e}, Damiano Pasqualini^b, Riccardo Michelotto Tempesta^b, Lorenzo Breschi^c, Milena Cadenaro^{f,g}

^a Department of Surgical Sciences, University of Turin, Turin, Italy

^b School of Nanotechnology, University of Trieste, Trieste, Italy

^c Department of Biomedical and Neuromotor Sciences, University of Bologna, Bologna, Italy

^d Department of Epidemiology, ASL To3 Grugliasco, Turin, Italy

^e Associate Professor, Department of Surgical Sciences, Dental School, Turin, Italy

^f Department of Medical Sciences, University of Trieste, Trieste, Italy

^g IRCCS Burlo Garofolo, Trieste, Italy

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ABSTRACT

Purpose: The aim of this *in vitro* study was to assess conversion degree (DC), micro-hardness (MH) and bond strength of two dual-curing resin cements employed under translucent monolithic zirconia irradiated with different time protocols.

Methods: 84 square shaped samples of 1 mm thickness were prepared from high translucency zirconia blocks and divided into two groups ($n = 24$) according to the cement employed: (1) Rely-X Ultimate; (2) Panavia SA. Each group was further divided into 3 subgroups ($n = 8$) according to the irradiation time: (a) no light; (b) 20 s; (c) 120 s. Light curing was performed 60 s after the sample was placed on the diamond support of a FT-IR spectrophotometer with a high power multiLED lamp. Final DC% were calculated after 10 min. After 24 h, Vickers Test on the cement layer was performed. The same protocol was used to lute composite cylinders in order to evaluate microshear bond-strength test. ANOVA and Bonferroni tests were performed to find differences between MH and bond-strength to zirconia, while for DC% the Scheirer–Ray–Hare two-way test was used.

Results: The two cements reached higher DC% in subgroup (b) and (c). As concern MH, statistics showed an increase in curing time was able to improve MH significantly. Bond-strength was not affected by irradiation time only for Panavia SA.

Conclusions: The first null hypothesis has to be rejected since DC% and MH of the dual-cements tested were influenced by the curing time. The second null hypothesis is partially rejected since the bond strength was influenced by the curing time only for Rely-X Ultimate.

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1. Introduction

The application of a ceramic indirect restoration always requires cementation in the oral cavity. To date, dual-curing cements are preferred because they combine the light-activated polymerization with the chemical curing mode [1]. The self-curing components are able to compensate the curing light attenuation problem that results by curing through an indirect

restoration, while the light-curing components are however activated by the light and complete the entire curing process [2–4]. Several authors [5–7] reported that curing light attenuation through a restorative material is anyway present and may thus affect the degree of conversion (DC) and the hardness of dual-cured cements. A proper DC of the resin cement is essential to reach sufficient mechanical and adhesive properties and, therefore, to ensure a long-lasting restoration. Moreover, a reduced DC of the resin cement can lead to a lower bond strength to dentin and even to a reduced bond strength between resin cement and ceramic material, which can compromise the longevity of an indirect restoration [8].

* Corresponding author at: Via Nizza 230, 10100 Turin, Italy.
E-mail address: nicola.scotti@unito.it (N. Scotti).

Table 1. Chemical compositions of Rely-X Ultimate and Panavia SA.

Rely-X Ultimate (Group A)	Paste A
	<ul style="list-style-type: none"> - Methacrylate monomers - Radiopaque, silanated fillers - Initiator components - Stabilizers - Rheological additives
Panavia SA (Group B)	Paste B
	<ul style="list-style-type: none"> - Methacrylate monomers - Radiopaque alkaline (basic) fillers - Initiator components - Stabilizers - Pigments - Rheological additives - Fluorescence dye - Dual-cure activator for Single Bond Universal Adhesive
	The proportion of inorganic fillers is about 43 % by volume; the grain size (D 90 %) is about 13 μm
	Paste A
	<ul style="list-style-type: none"> - 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) – Bisphenol A diglycidylmethacrylate (Bis-GMA) - Triethyleneglycol dimethacrylate (TEGDMA) - Hydrophobic aromatic dimethacrylate - 2-Hydroxymethacrylate (HEMA) – silanated barium glass filler - Silanated colloidal silica - DL-Camphorquinone - Peroxide – catalysts – pigments
	Paste B
	<ul style="list-style-type: none"> - Hydrophobic aromatic dimethacrylate – hydrophobic aliphatic dimethacrylate – silanated barium glass filler - Surface treated sodium fluoride - Accelerators - Pigments
	The total amount of inorganic filler is approximately 40 vol%. The particle size of inorganic fillers ranges from 0.02 μm to 20 μm

The curing-light attenuation is strictly related to the characteristics of the restorative material, in particular by its thickness, shade and opacity [9–13].

Recently, the use of zirconia for fixed prosthodontics rehabilitations is increasing but, because zirconia is not etchable, the advantage of stronger adhesion using resin cements may be lost. Moreover, unlike glass ceramic, zirconia is a less translucent material [12,14] and the transmittance of the curing light through it may not always be adequate to ensure a proper degree of conversion of the resin cement. However, in recent years several studies focused on the possibility to obtain an adhesive cementation with a zirconia restoration [15]. Resin-based cements, unlike traditional cements, allow to reduce the marginal microleakage and the risk of endodontic or periodontal complications or secondary caries [16]. Moreover, they offer the possibility of a proper adhesion to dentin and enamel, which is fundamental especially when retentive features are lost. Lee et al. observed how zirconia thickness affects shear bond strength (SBS) between zirconia and dual-cure resin cement, in particular as the thickness of zirconia increases, the bond strength decreases. According to the authors, extending the light-polymerization to 40 s would be helpful to obtain a reliable bond between zirconia ceramics thicker than 2 mm and dual-cure resin cements [17].

Recently, in order to overcome the limits towards bonding procedures, a new kind of zirconia with improved translucency and optical properties has been introduced. It allowed to obtain a material with improved aesthetic properties but also with a better transmission of the light through it [7,18]. However, an appropriate curing light irradiance of dual-cured cements through zirconia is still an open challenge and it needs studies to understand the ideal curing protocol for adhesive luting procedures.

The aim of this *in vitro* study was to evaluate the effect of different irradiation times of dual-curing cements through translucent zirconia on DC, hardness and bond strength. The first null hypothesis was that different irradiation times are not able to improve DC and hardness of dual-curing cements; the second null hypothesis was that different irradiation times are not able to

affect the bond strength of the dual-curing cement to translucent zirconia.

2. Materials and methods

2.1. Specimen preparation

Monolithic zirconia (Katana UTML, Ultra Translucent Multi Layered, Standard Shade A1-D4, Kuraray Noritake Dental Inc., Tokyo, Japan) was prepared into 84 square shaped samples (20 mm \times 20 mm) of 1 mm thickness using a cutting machine (Micromet, Remet, Bologna, Italy). In its green stage, each specimen was sequentially ground to the specific thickness using silicon carbide grinding paper (600 grit, 1200 grit, 2400 grit). The precision of sample thickness was checked with a digital caliper and discs with a discrepancy of more than 0.1 mm were excluded from the study. The samples were sintered according to the manufacturer instructions and then polished with diamond polishers (Zircpol Plus and Zircoshine Plus, Diatech, Switzerland) for 60 s each, using a device that standardized a constant pressure and direction. Samples were cleaned ultrasonically in distilled water for 15 min before testing and air-dried for 1 min.

One side of each zirconia sample was airborne-particle abraded with 30 μm silica-coated Al_2O_3 particles (CoJet sand, 3M ESPE, Seefeld, Germany) for 15 s/cm² at a distance of 10 mm with an intraoral air-abrasion device (CoJet Prep, 3M ESPE). 48 specimens were used to evaluate the degree of conversion and the micro-hardness while 36 to measure the bond strength. For each test, the samples were randomly divided in two subgroups according to the dual-curing resin cement employed: Rely-X Ultimate (Group A); Panavia SA (Group B). Chemical compositions of the two dual-curing resin cements are indicated in Table 1.

2.2. Degree of conversion measurement

A layer of universal adhesive (Scotchbond Universal, 3M ESPE) was applied for 30 s using a microbrush and thinned with air for

10 s on the sandblasted surfaces of 48 samples. A 170 μm thick plastic guide with a center hole 1 mm in diameter was placed on the diamond support of an ATR FT-IR (Attenuated Total Reflectance Fourier Transformed Infrared) spectrophotometer (Thermo Scientific Nicolet IS10) to standardize a layer of dual-curing resin cement between the sample surface and the FTIR light beam. The cement was applied on the bonded surface of each specimen and then placed on the FTIR light beam. The excess cement was eliminated, thereby creating a standardized pressure until the disc contacted the plastic guide. After 1 min of self-curing phase, 3 different irradiation protocols (which defined 3 subgroups) were performed using a high power multi-LED lamp (Valo, Ultradent, South Jordan, UT, USA) at 1400 mW/cm^2 , with the curing tip contacting the center of the sample and the light beam opposite to the cement layer: no irradiation (Subgroup A); 20 s of irradiation (Subgroup B); 120 s of irradiation (Subgroup C).

The surface analysis was performed in ATR mode, in which the IR beam penetrated 1 μm into the material. The FTIR spectra of the curing process were recorded every 2 s with a range between 4000–525 cm^{-1} and a resolution of 6 cm^{-1} . The spectra recorded immediately before activation of the poly-wave LED lamp and 10 min after light exposure were fitted and used to evaluate the degree of conversion of the two tested materials at different energy density conditions. To determine the percentage of the remaining unreacted double bonds, the DC was assessed as the variation of the absorbance intensities peak height ratio of the methacrylate carbon double bond (peak 1634 cm^{-1}) related to an internal standard of aromatic carbon-carbon double bonds (peak 1608 cm^{-1}) before and after curing of the specimen, according to the following equation:

$$\text{DC}\% = \left\{ 1 - \frac{(\text{CC aliphatic}/\text{CC aromatic})_{\text{Polymer}}}{(\text{CC aliphatic}/\text{CC aromatic})_{\text{Monomer}}} \times 100 \right\}$$

2.3. Microhardness measurement

Twenty-four hours after DC evaluation, microhardness (MH) was measured using a Leica VMHT microhardness tester (Leica Microsystems S.P.A., Milano, Italy) equipped with a Vickers indenter, at exactly the same location at which DC was analyzed by the FTIR light beam. A pyramidal diamond indentation was obtained with a load of 100 g for 15 s. Four indentations were obtained for each specimen, and the mean value was considered for the statistical analyses.

2.4. Shear bond strength evaluation

Preformed polyethylene molds with a hole in the center were used to produce composite cylinders (diameter 1 mm, height 2 mm). The composite (Ceram-X Mono shade M, Dentsply Sirona, Salzburg, Austria) was placed into the mold and condensed to fill it; each cylinder was cured with a multi-LED lamp (Valo, Ultradent) at 1400 mW/cm^2 for 20 s. Then, the polyethylene mold was removed, and the composite cylinders were stored in a dark box for 7 days at 37 °C.

36 zirconia samples were randomly divided in two subgroups according to the dual-curing resin cement employed: Rely-X Ultimate (Group A); Panavia SA (Group B). Two composite cylinders were cemented, following the manufacturer instructions, on each zirconia block and, after 1 min of self-curing process, light-curing was performed by placing the lamp in contact with the opposite side of the zirconia surface in order to simulate the oral conditions. The samples of each group were randomly divided into 3 subgroups according to the irradiation time, as done for DC and MH evaluation: no light-activation, 20 s and 120 s.

Table 2. Mean degree of conversion, expressed as a percentage.

Irradiation	Degree of conversion	
	Rely-X Ultimate	Panavia SA
No light	29.7 ^A \pm 4.8	29.3 ^A \pm 10.2
20 s	63.3 ^B \pm 2.9	52.8 ^B \pm 5.7
120 s	62.9 ^B \pm 1.9	58.1 ^B \pm 5.2

Different superscript uppercase letters indicate significant differences between data within the same column ($p < 0.05$). Different subscript lowercase letters indicate significant differences between data within the same row ($p < 0.05$).

After 24 h of storage in a dark room, samples were embedded into resin stamps in order to subject them to the SBS test with a universal machine. Load was applied in shear at a constant crosshead speed of 0.5 mm/min until failure occurred. The shear bond strengths were calculated and expressed in MPa. The bonded area of the composite cylinder was measured with a digital caliber, and its radius was used to calculate the circular area bonded with zirconia surface.

Fractured specimens were observed under optical microscopy (Wild, Heerbrugg, Gaiss, Switzerland) at 40 \times magnification to establish failure modes, which were classified as (1) adhesive along the zirconia surface or (2) mixed when simultaneously exhibiting the zirconia surface and remnants of resin cement.

2.5. Statistical analysis

To evaluate the effect of dual-curing cement, irradiation time, and their interactions on MH and SBS a two-way ANOVA and Bonferroni post-hoc tests were performed, after ascertaining the normality (Shapiro-Wilk test) and homoscedastic (modified Levene test) assumptions of the data sets. As regards DC, that by construction does not have normality, the Scheirer-Ray-Hare two-way non parametric test was used. In that case, the Wilcoxon-Mann-Whitney test with Bonferroni correction was used as a post-hoc analysis. Fracture modality was analyzed using the χ^2 test.

Differences were considered statistically significant if $p < 0.05$.

Statistical analyses were performed using the Stata 13.0 (StataCorp, 4905 Lakeway Drive, College Station, TX, USA) and R 3.5.0 (R Core Team, R Foundation for Statistical Computing, Vienna, Austria).

3. Results

Mean and standard deviation values for DC, MH and SBS obtained with the two tested dual-curing cements are shown in Tables 2–4. Type of fracture (expressed as percentage) in each group is shown in Fig. 1. Scheirer-Ray-Hare test and the associated post-hoc analysis showed that the two cements reached significantly higher DC when irradiated for 20 s or 120 s with respect to no light-activation ($p < 0.05$). Further, when irradiated for 20 s Rely-X Ultimate showed higher DC than Panavia ($p < 0.05$). As concern MH, ANOVA test showed that an increase in curing time was able to significantly improve MH either for Rely-X either for Panavia SA. The bond strength was not affected by the irradiation time for Panavia SA. On the contrary, Rely-X showed greater values when no light curing was performed. The χ^2 test revealed that the two tested dual-curing cements and the different curing time did not statistically influence the type of fracture between zirconia and dual-curing cement, which were mainly adhesives.

4. Discussion

The longevity of indirect adhesive restorations depends mainly on the quality of the dental-cement-restoration interface [19,20]. To reach optimal physical and mechanical properties of

Table 3. Microhardness Vickers test results.

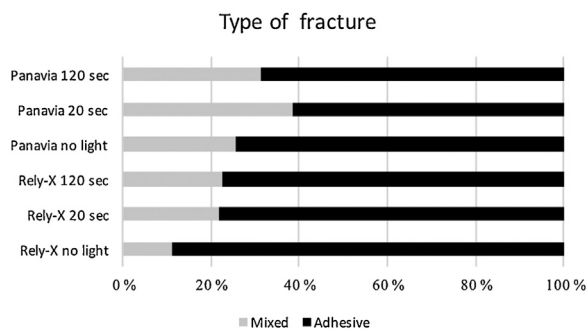
Irradiation	Microhardness	
	Rely-X Ultimate	Panavia SA
No light	17.2 ^A _a ± 2.5	9.7 ^A _b ± 3.1
20 s	53.1 ^B _a ± 7.2	23.4 ^B _b ± 5.5
120 s	79.6 ^C _a ± 14.5	26.4 ^B _b ± 3.9

Different superscript uppercase letters indicate significant differences between data within the same column ($p < 0.05$). Different subscript lowercase letters indicate significant differences between data within the same row ($p < 0.05$).

Table 4. Bond strength, expressed in MPa.

Irradiation	Bond strength	
	Rely-X Ultimate	Panavia SA
No light	17.3 ^A _a ± 2.5	8.6 ^A _b ± 3.4
20 s	10.3 ^B _a ± 1.6	7.6 ^A _a ± 1.5
120 s	10.8 ^B _a ± 1.3	8.5 ^A _a ± 4.2

Different superscript uppercase letters indicate significant differences between data within the same column ($p < 0.05$). Different subscript lowercase letters indicate significant differences between data within the same row ($p < 0.05$).

**Fig. 1.** Distribution, expressed as percentages, of different types of fracture found in the different groups.

dual-curing resin cements under ceramic restorations, the conversion rate should be as high as possible [21]. The method used in this study to assess the DC was the FTIR, a well-established technique that allows direct quantification of unreacted C=C in a resin matrix [7,22,23].

Several previous studies analyzed the influence of zirconia thickness on the curing light attenuation [6,14]. A method to overcome this evident problem, since the opacity of zirconia restorations can compromise the quality of luting cement conversion, could be the increase in curing time. The results of the present study led to accept the first null hypothesis, since either DC% either MH of the dual cements tested were not influenced by the light-curing time. Indeed, the statistical analysis showed a significant increase of the conversion degree in both dual-curing cements when they were light-cured either for 20 or 120 s, but any significant difference was highlighted between different curing time protocols in the DC%. When a dual-curing resin cement is photo-activated through a restoration, part of the light is absorbed and part is reflected on the surface of the restoration, while the part that reaches the cement is the one that is transmitted through the restoration. This portion could depend on the thickness of the restoration and on the optical characteristics of the restorative material such as opacity and shade [24]. The matter of the light attenuation through the zirconia restorations has been faced by different authors [6,7,11,14,22]. Indirect zirconia restorations could be adhesively cemented with dual-cured resin cements and, thus, a proper transmittance of the light through the restoration can increase the polymerization process of the resin

cement. Ilie and Stawarczyk [14] evaluated the amount of transmitted light through translucent and conventional zirconia and a glass ceramic of different thickness with a USB400 Spectrometer. They concluded that zirconia was less translucent than glass ceramic, but the translucency decreased more slowly with material thickness and that fewer differences were found for translucency between conventional and translucent zirconia. Similar results were achieved by Sulaiman et al. [7], who evaluated the light irradiance and the DC through various thickness and kind of zirconia. They concluded that the irradiance, radiant exposure and the degree of conversion of a resin-based cement were affected by the brand and the thickness of the zirconia sample. In the present study, we evaluated the conversion degree of two dual-curing cements which were light-cured with different time intervals through zirconia of a standard thickness of 1 mm. The statistical analysis showed that the two cements reached higher DC after 20 or 120 s of irradiance. It can be thus speculated that the light is not totally attenuated by the zirconia opacity, at least when it is 1 mm thick, but on the contrary could positively affect the conversion degree of both dual-curing cements. However there is no statistical difference between 20 s and 120 s of irradiation.

Surface microhardness of a restorative resin is one of the most important parameters for assessing physical properties of dental materials, and it is defined as the resistance of a material to indentation or penetration. In the literature, hardness of a resin is strongly related to his DC because higher is the conversion degree, greater will be the amount of cross-linked polymers and then the hardness of the material [25,26]. That is why microhardness is commonly used as a simple and reliable method for indirectly estimating the conversion degree of resin-based cements [4,26]. In the present study, the micro-hardness, which was measured in the same area of DC evaluation, was partially coherent with the DC and showed a significant improvement for both Rely-X and Panavia SA by increasing the curing time. However, absolute microhardness values should not be used to compare the DC between the different resin cements. In fact, results highlighted a significant different microhardness between the two dual-curing cements tested, which should depend to variation in filler type, shape and content and monomer composition. Rely-X Ultimate contains a greater percentage of filler and, thus, it showed a significantly higher microhardness than Panavia SA.

In the current in vitro study, the results achieved with the shear bond strength test seemed to contrast with the results of the DC. In fact, groups with no light irradiation achieved higher bond strength, and this result was statistically significant for Rely X Ultimate. Thus, the second null hypothesis was partially rejected, since different curing protocols were able to affect the bond strength of Rely-X Ultimate, and not Panavia SA, to zirconia. Based on these findings, we could hypothesize that the shear bond strength test could give results which cannot be correlated with the DC and MH findings. A recent systematic review showed that the shear bond strength test is less discriminative than a tensile test, even if it is stated that it is the most commonly applied test method for assessing bonding efficacy to zirconia. Most likely, this can be attributed to the fact that there is no need for further specimen preparation prior to the test [27]. It has been speculated that an immediate exposure to light of dual-curing cements could induce the formation of cross-linked polymers chains which could interfere with the self-curing process by entrapping polymerization promoters and unreacted monomers into the network [28]. In fact, in dual-curing cements the polymerization process is made more complex by the interaction of light and chemical polymerizing components. Moreover, when a dual-cement is light-cured, the conversion of the monomer in polymer begins and the viscosity of the material quickly increases. The increased viscosity could obstacle either the movement of the chemical components

responsible for the additional polymerization and, in some cements, either the monomers assigned to the chemical bonding with zirconia. Several studies showed that the greater amount of conversion occur during the first 5 min, as the development of a highly viscous, cross-linked polymer network would impede further propagation [3,29]. The rapid increase in viscosity due to the increase in DC of a composite during the initial stage of polymerization may hinder the diffusion of radical components to limit further monomer conversion [30]. This may apply to light-activated, dual-cured materials and light-cured materials [31,32]. A recent study [33] confirmed that Variolink II showed comparable bond strength to feldspathic ceramic with the dual or light activation modes, confirming the importance of the chemical curing process in the adhesion to ceramic materials.

5. Conclusion

Within the limitations of this *in vitro* study, the following conclusions can be made:

1. Light-curing affects the DC and the MH of dual-curing cements polymerized through high translucent zirconia, but no differences were found by increasing the curing time. It means that, clinically, the light curing process is important to obtain high DC and high MH, independently of the curing time. An extended curing time would not give a remarkable advantage in dual-curing cements properties.
2. Higher bond-strength values seem not to match with higher DC and MH values. However, based on the results, we can affirm that a proper curing process of dual-curing cement is fundamental to achieve best mechanical properties, independently by the bond strength to zirconia. Thus, an extended curing time over 120 s could be suggested to increase DC and MH, even if 20 s could be sufficient with Rely-X Unicem.

Further studies are needed to investigate the interaction between dual-curing resin cements and zirconia when depending solely on the self-curing mechanism.

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