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Article

The Effects of Exposure Time on the Surface Microhardness of Three Dual-Cured Dental Resin Cements

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Abstract: This study evaluated the exposure time of light-curing of the polymers used for cementation on microhardness test in different storage times. The polymers (specifically called resin cements) were RelyX ARC, RelyX U100, and SET. Five specimens of each group were prepared and photo-polymerized with exposure times of 20 s and 180 s, using a LED polymerization unit with wavelength of 440 ~ 480 nm and light output was consistently 1,500 mW/cm^2. The Vickers hardness test was performed in a MMT-3 Microhardness Tester. Data were submitted to ANOVA and Tukey's test ($\alpha = 0.05$). The values of RelyX ARC showed statistically significant difference to groups with light exposure when considering only chemical cure ($p < 0.05$). The groups with light exposure (20 s and 180 s) showed no significant difference between them ($p > 0.05$). The RelyX

U100 cured only chemically showed statistically significant difference between 48 h and 7 days ($p < 0.05$). The SET resin cement showed no significant difference to groups without light exposure for all storage times ($p > 0.05$). The values of hardening of the dual-cured resin cements improved after setting by light and chemical activation demonstrating the importance of light curing.

Keywords: polymers; photo-responsive polymers; hardness

PACs: 82.35.-x; 62.20.Qp; 46.55.+d

1. Introduction

Dental polymers, specifically resin cements, have been used considerably in recent years due to a larger application of dental adhesive procedures [1,2].

There are a variety of currently available cements on the market, though none is ideal for all clinical situations. Therefore, the choice of the luting agent must rely on its physical, biological and handling properties being allied to the characteristics of the prepared tooth and prostheses [3].

Factors, such as exposure time of light-curing and type of light-curing unit can influence the characteristic of the resin cement, changing the final quality of the restorations [4].

The dental resin cements can be classified based on the type of their cure such as chemical-, light- or dual-cure [5].

The chemical-cure polymerizes exclusively by chemical activation. The dual-cured resin cement is formulated to not depend only on light activation; the cure is started by photo-and chemical-initiators. These systems can be used locally where light transmission is limited. The light-cure resin cement is exclusively initiated by light, which offer the clinical advantages of extended working time, setting on demand, and improved color stability [6,7].

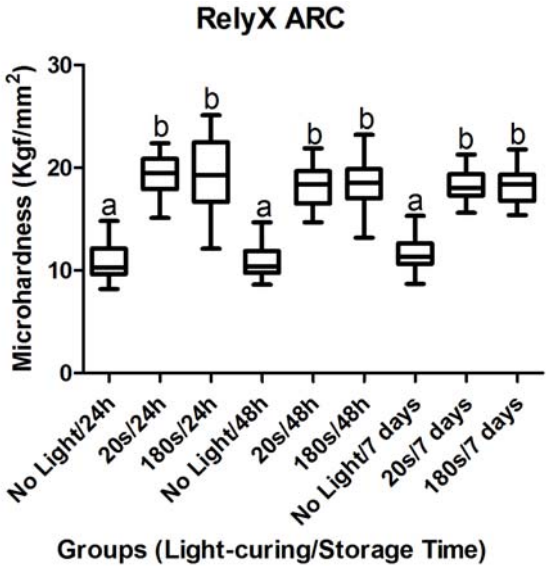
Hardness testing is commonly used as a simple and reliable method to indicate the degree of conversion [8].

The purpose of this study was to investigate the exposure time of light-curing of the dual-cured resin cements on microhardness test at different storage times.

2. Results and Discussion

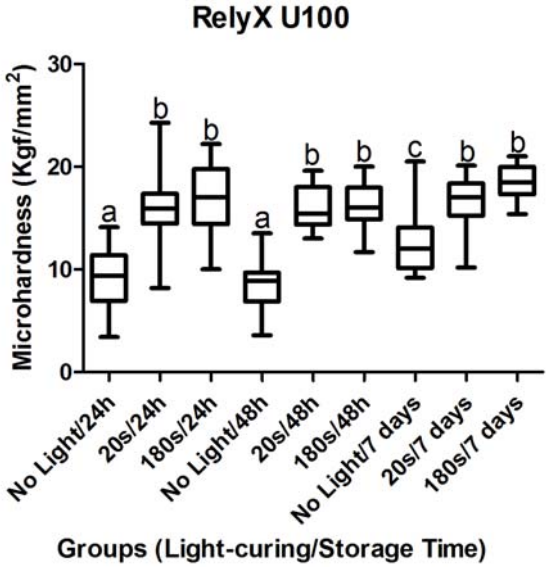
The data were analyzed separately for each dental material. The objective was to compare different exposure and storage times. Figure 1 shows the mean values (Kgf/mm^2) for RelyX ARC. The values of RelyX ARC showed statistically significant difference to groups with light exposure when considering only chemical cure ($p < 0.05$). The groups with light exposure (20 s and 180 s) showed no significant difference between them ($p > 0.05$).

Figure 1. The mean values (Kgf/mm^2) were analyzed according to exposure time of light-curing and storage time for RelyX ARC. Different letters (A–B) indicate statistically significant difference at 5% level (Tukey's test; $P < 0.05$).



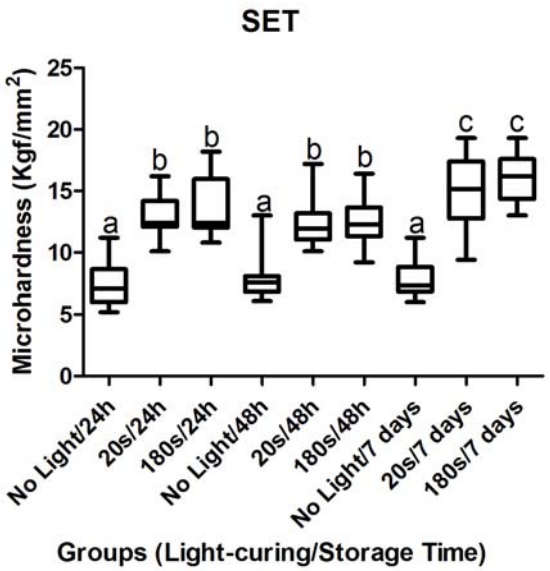
The RelyX U100 cured only chemically, showed statistical significance between 48 h and 7 days ($p < 0.05$). Others storage times showed no difference when compared with same light exposure (Figure 2).

Figure 2. The mean values (Kgf/mm^2) were analyzed according to exposure time of light-curing and storage time for RelyX U100. Different letters (A–C) indicate statistically significant difference at 5% level (Tukey's test; $P < 0.05$).



The SET resin cement (Figure 3) showed no significant difference to groups without light exposure for all storage times ($p > 0.05$). The cement analyzed 7 days after curing showed statistically significant difference for 24 and 48 h ($p < 0.05$).

Figure 3. The mean values (Kgf/mm^2) were analyzed according to exposure time of light-curing and storage time for SET resin cement. Different letters (A–C) indicate statistically significant difference at 5% level (Tukey's test; $P < 0.05$).



The dental resin materials depend on many factors such as the properties of the monomer, polymer and the filler used, the photo-curing process, their concentration, the curing-light unit and their power density and exposure time [9-12].

Furthermore, physical properties of dental materials provide an indication of how the material will function under stress in the oral environment [13]. Microhardness test is widely used to examine dental resin material, analyzing their polymerization, the efficiency of the light units [14,15] and also related to its wear resistance and ability to maintain form stability [16]. This study was analyzed by microhardness test to eliminate any variation that could change the results, because the results of analyzing were only applied for cement. Some studies indicate that the RelyX Unicem (Self-adhesive, such as RelyX U100) contains phosphoric acid esters, which require wet surfaces for ionization and subsequent interaction with dentin and enamel and the values are better if pressure is maintained throughout the cementation procedure to include the self-curing period [17-19].

Resin cements setting by light and chemical activation (dual cured) resulted in the highest hardness values, even compared to resin cement setting only by chemical activation. This outcome might be attributed to the fact that the all resin cements studied are dual cure system, for which the photo-initiator was not sensitized. The chemical mode started the polymerization reaction, characterized by cross-linking formation, which could have reduced monomer mobility in the bulk mass, hindering the completion of cure [20].

Hardness of dental resin cements can be used as indicator of the degree of conversion [21,22], for which there is a correlation between the degree of conversion and hardness test [23]. In this study, the hardness value should not be compared between the dental cements used, because there is a variation in monomers composition, filler content and type.

The SET resin cement showed statistically significant difference 24 h after curing and after 7 days. Others resin cements were similar between them. The larger change of hardness probably happened within 24 h initial.

The mean values and standard deviations are shown in Table 1.

Table 1. The mean values (Kgf/mm^2) and standard deviation (SD) of all groups in this study.

Storage time	Dental Materials					
	RelyX ARC		RelyX U100		SET	
	Mean	SD	Mean	SD	Mean	SD
No light/24 h	10.86	1.88	9.17	2.94	7.68	3.50
20 s/24 h	19.99	3.18	16.18	3.41	12.93	2.88
180 s/24 h	19.35	3.72	16.83	4.60	13.33	2.61
No light/48 h	11.49	3.00	8.37	2.42	7.40	1.87
20 s/48 h	18.33	2.01	13.54	2.93	11.64	1.06
180 s/48 h	18.25	3.38	16.17	2.02	11.62	2.52
No light/7 days	11.64	1.46	11.88	4.35	7.75	1.55
20 s/7 days	18.96	2.66	15.92	2.74	14.32	3.75
180 s/7 days	17.60	1.61	18.51	1.62	16.00	4.77

3. Experimental Section

The cement materials used were RelyX ARC, RelyX U100, and SET (Tables 1 and 2). Precapsulated materials (SET, SDI) were prepared using a mixing unit (Ultramat 2, SDI) and the materials (RelyX ARC and RelyX U100) were dispensed, manually mixed, placed in molds. The metallic mold (fabricated in stainless steel) with central orifice (2 mm in diameter and 1 mm in thickness) was used. Five specimens of each group were prepared for each exposure time of light-curing and storage time evaluated. The resin cement was packed into the mold and a polyethylene film covered each side of the sample and a glass slide was placed on the top surface of the samples. The top surface was standardized with a circular weight (1 kg). The light tip of the light-curing unit was positioned on the glass slide, leaving the 0.55 mm of the material. Dual-cure samples were then photopolymerized with exposure times of 20 s and 180 s, using a LED polymerization unit with wavelength of 440 ~ 480 nm and Light output was consistently 1,500 mW/cm^2 (Radii Plus, SDI) (Figure 4). The power density were checked using a powermeter (Fieldmaster, Coherent Commercial Products Division, model number FM, set n° WX65, part number 33-0506 in USA) and the irradiance were calculated with this formula:

$$I = P/A$$

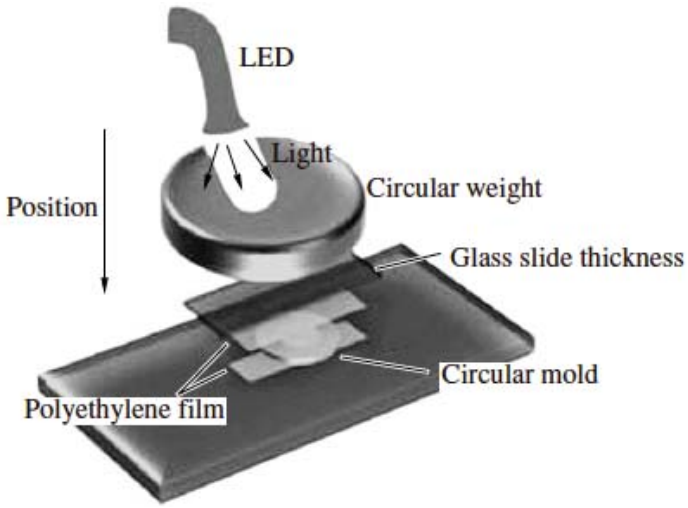
where: P is power in mW; A is area of the light tip in cm^2.

All samples were stored in darkness to prevent ambient light from causing additional light-curing polymerization. Specimens were stored in a 98 ± 2% humidity environment in a humidor at 37 ± 2 °C until being tested at 24 h, 48 h, and 7 days.

Table 2. Dental materials used in the study.

Materials	Manufacturer	Composition	Shade	Recommended Light-curing Duration
RelyX ARC	3M Dental products Div., St Paul, MN, USA	Paste A: Bis-GMA, tri-ethylene glycol dimethacrylate, zircon/silica filler, photoinitiators, amine, pigments Paste B: Bis-GMA, tri-ethylene glycol dimethacrylate, benzoic peroxide, zircon/silica filler	A2	20 s
RelyX U100	3M Dental products Div., St Paul, MN, USA	Paste A: Glass powder, silica calcium hydroxide, pigment, substituted pyrimidine, peroxy compound, initiator Paste B: Methacrylated, phosphoric esters, Dimethacrylates, Acetate, Stabilizers, Self-cure initiators, Light-cure initiators	A2	20 s
SET	SDI Limited. Bayswater, WA, AU	Methacrylated phosphoric esters, UDMA, photoinitiator, Fluoroaluminosilicate glass, pyrogenic silica	A2	20 s

Figure 4. Schematic drawing of the specimen's preparation for microhardness test.



The Vickers hardness test was performed in a hardness testing machine, MMT-3 Microhardness Tester (Buehler Lake Bluff, Ilinois USA) equipped with Vickers diamond (VHN), which has a format of pyramid of 136° where the two diagonals are measured [8,9] using load of 50 gf (gram force) for 30 s. The surface was divided in quadrant and the diamond took place an impression for quadrant. The hardness data were submitted to the Analysis of Variance with two fixed criteria (two-way-ANOVA). The tests were performed at the level of 5%.

4. Conclusions

The values of hardening of the dual-cured resin cements improved after setting by light and chemical activation. The use of light-curing of dual-cured resin cements became important, within limitations of this study. However, further research is necessary.

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