# Effect of different light curing methods on mechanical and physical properties of resin-cements polymerized through ceramic discs

**Isil CEKIC-NAGAS[1], Gulfem ERGUN[1]**

1- DDS, PhD, Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey.

**Corresponding address:** Dr. Isil Cekic-Nagas - Address: Servi sokak 6A/7 - Kolej, Ankara, Turkey - Phone: +90 312 432 36 05 - Fax: +90 312 310 27 30 - e-mail address: isilcekic@hacettepe.edu.tr - isilcekic@gmail.com

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## ABSTRACT

Objective: The aim of this study was to compare the polimerization ability of three different light-curing units (quartz tungsten halogen, light-emitting diodes and plasma arc) and their exposure modes (high-intensity and soft-start) by determination of microhardness, water sorption and solubility, and diametral tensile strength of 5 dual-curing resin cements. Material and methods: A total of 720 disc-shaped samples (1 mm height and 5 mm diameter) were prepared from different dual-curing resin cements (Duolink, Nexus, Bifix-QM, Panavia F and RelyX Unicem). Photoactivation was performed by using quartz tungsten halogen (high-power and soft-up modes), light-emitting diode (standard and exponential modes) and plasma arc (normal and ramp-curing modes) curing units through ceramic discs. Then the samples (n=8/per group) were stored dry in the dark at 37°C for 24 h. The Vickers hardness test was performed on the resin cement layer with a microhardness tester (Shimadzu HMV). For sorption and solubility tests; the samples were stored in a desiccator at 37°C and weighed to a constant mass. The samples were weighed both before and after being immersed in deionized water for different periods of time (24 h and 7 days) and being desiccated. The diametral tensile strength of the samples was tested in a universal testing machine at a crosshead speed of 0.5 mm/min. Data were analyzed statistically by nonparametric Kruskal Wallis and Mann-Whitney U tests at 5% significance level. Results: Resin cement and light-curing unit had significant effects ($p<0.05$) on microhardness, diametral tensile strength, water solubility and sorption. However, no significant differences ($p>0.05$) were obtained with different modes of LCUs. Conclusion: The study indicates that polymerization of resin cements with different light-curing units may result in various polymer structures, and consequently different mechanical and physical properties.

**Key words:** Resin cements. Water solubility and sorption. Hardness. Tensile strength. Light-curing units.

## INTRODUCTION

High-technology processes have led to the development of glass ceramics that are increasingly attractive in restorative dentistry because of their excellent mechanical properties, aesthetics and etchability[6,27]. Long-term survival of adhesive porcelain restorations depends on the success of a reliable bond between porcelain, the luting agents and the dental substrates. Because of their brittle nature, all-ceramic restorations rely on adequate bonding[7]. For luting ceramic restorations, the use of resin cements has increased considerably in the last years[15,22]. In most clinical cases, dual-curing resin cement is used when bonding ceramic to enamel and dentin[17]. Dual polymerization, the combination of light and chemical polymerization, provides a better conversion of monomers. This is important because inadequate polymerization is usually associated with poor mechanical and biological properties of the resin cements[19]. In addition, the polymerization of resin cement might also be affected by the characteristics of the luting material, such as chemical composition, filler particle size and shade[27]. Moreover, adequate

polymerization of resin cements depends not only on resin cement but also on the light-curing unit (LCU) intensity, wavelength of the visible light and polymerization time[19].

Until recently, conventional quartz tungsten halogen (QTH) LCUs were widely used to polymerize resin cements[16]. These LCUs are susceptible to intensity output degradation with time as a result of the age of the bulb and its reflector, blistering and cracking of the filter, damage to the fiber-optic tips, because of repeated sterilization or heat generation[14]. To overcome these problems, boosted versions of QTH or high intensity LCUs such as plasma arc (PAC) and light-emitting diode (LED) LCUs that possess higher light intensity and shorter polymerization cycles than conventional LCUs have been developed[14,17]. In addition to increasing light intensity, various irradiation protocols, such as ramped and stepped intensity LCUs are marketed for their ability to "soft-start" polymerization[7]. These LCUs first use a lower intensity light followed by high-intensity light. This slower polymerization process allows the material to flow in the pregel stage, which provides some stress relief from polymerization contraction at the resin/dentin interface and reduces marginal gaps in these joints[4]. As a result, manufacturers claim that polymerization could be completed with higher intensity lights, which result in higher degree of monomer conversion, and is associated with improvements in the mechanical properties and of resin-based materials[13,20]. In this regard, modified light polymerization protocols can lead to the resulting polymer having different structures and properties, such as hardness, diametral tensile strength (DTS), and water solubility and sorption[1,11,20].

The interaction of dental resin-based materials with the aqueous oral environment may result in deterioration of their mechanical properties, dissolving and leaching of some of the components, such as unreacted monomers or fillers out of the samples leading to degradation and erosion[12,26]. In other words, sorption and solubility may act as precursors to a variety of chemical and physical processes that can lead to deleterious effects on the structure of the polymeric network, which can affect the suitability of the clinical applications of these materials[12,30]. Furthermore, surface hardness is also one of the most important physical characteristics of dental materials, and is defined as the resistance of a material to indentation or penetration[23]. Hardness testing is commonly used as a simple and reliable method to indicate the degree of conversion of resin-based cements[9]. Moreover, a basic mechanical property of resin-based materials, usually represented by manufacturers, is its strength[5]. An alternative method to test the strength of brittle resin-based materials, in which the ultimate tensile strength of a brittle material is determined through compressive testing, has become popular because of its relative simplicity and reproducibility of results[8,10]. The method is described in the literature as the diametral compression test for tension[10].

Accordingly, the following null hypotheses were tested: 1. The microhardness, water sorption and solubility, and DTS show dependence on the type of resin cements cured with different curing modes of high power LCUs; 2. High-power PAC LCUs might be considered for optimal curing efficiency of resin cements.

## MATERIAL AND METHODS

### Sample preparation

IPS Empress 2 (Ivoclar-Vivadent; shade A1, Schaan, Liechtenstein) cylindrical ceramic sample (5 mm in diameter and 2 mm in height) was fabricated by the lost wax technique and the ingot was injected to an EP 600 furnace (Ivoclar-Vivadent). The ceramic disc was ground with 220, 360, and 600 grit [Federation of European Producers of Abrasives (FEPA)] and air-abraded with 50 μm $Al_2O_3$ particles (Korox; Bego, Bremen, Germany) for 14 s from a distance of approximately 10 μm at 400 kPa with a sandblasting device (Ar-Ge Dental, Denizli, Turkey). Then the disc was cleaned in distilled water for 10 min in an ultrasonic bath (Healthsonics; Livermore, CA, USA) to ensure a contaminant-free ceramic surface.

The tested resin cements (Duolink, Bifix-QM, Nexus, RelyX Unicem and Panavia F), their manufacturers and compositions are shown in Figure 1. Seven hundred and twenty cylindrical discs (5 mm diameter and 1 mm high) were prepared by the same operator according to their manufacturers' directions. The methodology used in this study was based on the ADA Specification no. 27 and ISO standard 4049:2000[2,16].

Figure 2 shows the experimental set-up of the study. The pastes A and B of the Panavia F hand-mixing cement was mixed in a 1:1 ratio on a mixing pad for 10 s. The other self-mixing cements (Duolink, Bifix-QM, Nexus and RelyX Unicem) were mixed by activating the syringes and triturating for 5 s. After placing the resin cements in a circular polytetrafluoroethylene mold, a transparent polyethylene film was placed over the filled mold and the ceramic disc was positioned over the resin cement.

Light activation was performed through the ceramic disc using three high-power LCUs: a quartz tungsten halogen (QTH, Blue Swan Digital; Dentanet, Turkey) (high-power and soft-up modes), a light-emitting diode (LED, Elipar Freelight 2; 3M Espe, St. Paul, MN, USA) (standard and exponential modes) and a plasma arc LCU (PAC, PlasmaStar, SP-

| Trade Name | Chemical composition* (filler content by weight*) | Lot number | Manufacturer |
|---|---|---|---|
| Duolink | Base: Bis-GMA, TEGDMA, glass filler, UDMA<br>Catalyst: Bis-GMA, TEGDMA, glass filler<br>(67% wt) | 500009783 | Bisco Inc., Schaumburg, U.S.A |
| Bifix-QM | Bis-GMA, benzoylperoxide, amines, barium-aluminiumboro-silicate glass<br>(71-73% wt) | 640422 | Voco GmbH, Cuxhaven, Germany |
| Nexus | SP-345, EBPADM, Fumed silica, UDMA, TEGDMA, Silane, SrSiF6, BPO, BisGMA, UV-9, ZnSiF6, BHT<br>(68% wt) | 449569 | Kerr Co., Orange, CA |
| RelyX Unicem | Powder: glass powder, initiator, silica, substituted pyrimidine, calcium hydroxide, peroxy compound and pigment<br>Liquid: methacrylated phosphoric ester, dimethacrylate, acetate, stabilizer and initiator<br>(72% wt) | 290546 | 3M Espe AG, St. Paul, MN, USA |
| Panavia F | Paste A: BPEDMA, MDP, DMA<br>Paste B: barium, boron, silicium glass and NaF<br>(73% wt.) | Paste A: 00304A<br>Paste B: 00052A | Kuraray Medical Inc., Okayama, Japan |

*Information provided by manufacturers.

Bis-GMA: Bis-phenol-A diglycidylmethacrylate; TEGDMA: Triethyleneglycol dimethacrylate; UDMA: Urethane dimetacrylate; SP-345: barium aluminoborosilicate; EBPADM: ethoxylated Bis-phenol-A-dimethacrylate; BPO: benzoyl peroxide; BHT: Butylated hydroxy toluene; HDDMA: Hexanediol dimethacrylate; SrSiF6: strontium hexafluorsilicate; UV-9: 2-hydroxy-4-methoxybenzophene; ZnSiF6: zinc hexafluorosilicate; BHT: 2,6-di-tert-butyl-4-methylphenol; BPEDMA: bisphenol-A polyethoxydimethacrylate; MDP: 10-methacryloyloxy decyl dihydrogenphosphate; DMA: aliphatic dimethacrlyate; NaF: sodium fluoride.

**Figure 1-** Test materials and their composition according to the manufacturers

2000; Monitex Industrial Co. Ltd., Taiwan) (normal and ramp-curing modes). While high-intensity mode corresponds to high power, standard and normal modes, soft-start mode corresponds to soft-up, exponential and ramp-curing modes. The characteristics of the LCUs are shown in Figure 3. After light polymerization, the samples were stored dry in dark at 37ºC for 24 h. All of the mechanical tests were performed by the same operator.

## RESULTS

### Water solubility and sorption

For sorption and solubility measurements, 48 samples were prepared for each of the 5 test materials (n=48/*per* cement group). The thickness of the samples was measured using a digital caliper (Liaoning MEC Group; Dalian, China). All samples were placed in a desiccator containing freshly dried silica gel at 37°C 24 h and weighed to a precision of 0.0001 g using a calibrated electronic balance (AX205 DeltaRang; Mettler Toledo, Giessen, Germany). This drying cycle was repeated until a constant mass ($m_1$) for each disc was attained.

The samples were stored in distilled water at 37°C for 24 h and 7 days. Then they were weighed after being carefully wiped with an absorbent paper. When constant weight was obtained, these values were recorded as $m_2$. After these weighing procedures, the samples were returned to the desiccator at 37ºC until a constant mass was achieved ($m_3$). The volume (V) of each sample was calculated in $mm^3$.

The values for water sorption ($W_{sp}$) and solubility ($W_{sl}$), in $\mu g/mm^3$, were calculated using the following equations[26]:

$$W_{sp} = \frac{m_2 - m_3}{V}$$

$$W_{sl} = \frac{m_1 - m_3}{V}$$

### DTS

For DTS test measurements 240 samples were prepared (n=8/*per* group). The DTS of the samples (n=8/*per* group) was investigated through a diametral compression test. A compressive load was applied on the diametral surface of the samples to obtain DTS at a crosshead speed of 0.5 mm/min in a testing machine (Lloyd LRX; Lloyd Instruments,

Fareham Hants, UK) until failure. After each compressive test, the fracture load (F) in Newton (N), was recorded and the DTS (MPa) was calculated from the equation[9]:

$$DTS = \frac{2P}{\pi dt}$$

where P is the load at fracture, d is the diameter and t is the thickness.

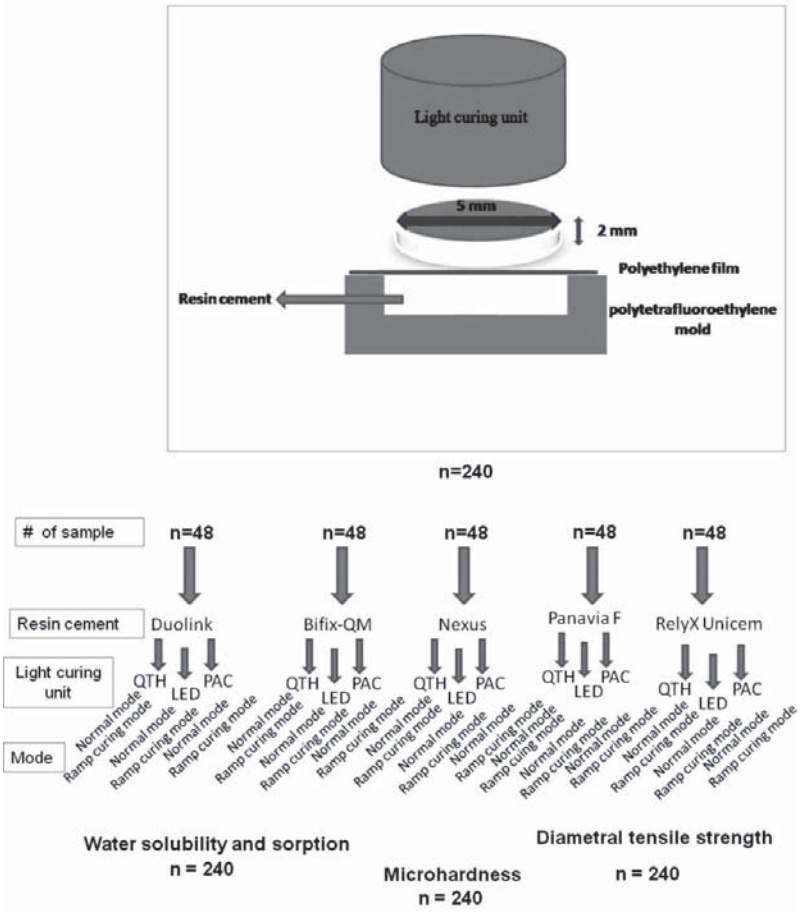


**Figure 2-** Schematic illustration of sample preparation and the experimental design

| Light curing unit | Type of light source and diameter of tip | Curing Modes | Profiles (exposure period) | Output* (mW/cm$^2$) |
|---|---|---|---|---|
| QTH | 8-11.5 mm | Soft-up mode<br>High-power Mode | The polymerization cycle of boost automatically from soft- start to high output (20 s)<br>Unit constantly generates the full polymerizing energy (20 s) | 1000 |
| PAC | 8 mm | Normal mode<br>Ramp-curing Mode | Provides full light intensity for the entire exposure period (10 s)<br>Light exposure time 6 s total, intensity as follows:<br>start 2 s at 50%, 2 s at 50%, 2 s at 100% | 2250±50 |
| LED | 8 mm | Standard mode<br>Exponential Mode | Provides full light intensity for the entire exposure period (20 s)<br>Provides light increasing to full intensity over the course of 5 s (20 s) | 1200 |

QTH=Quartz tungsten halogen
PAC=plasma arc
LED=light-emitting diode

**Figure 3-** Light-curing units and models

**Table 1-** Median values and (25th–75th) percentiles for solubility (μg/mm³) of the resin cements tested after 24 h and 7 days

| Resin cement | SOLUBILITY (μg/mm³) | | | | |
|---|---|---|---|---|---|
| | Polymerization modes | | | | |
| | | 24 h | | 7 days | |
| | LCU | High intensity | Soft-start | High intensity | Soft-start |
| **Duolink** | **QTH** | 5.09 (5.09-7.77) | 9.14 (5.73-10.19) | 1.27 (0.64-2.34) | 2.26 (1.40-2.99) |
| | **LED** | 5.09 (5.09-7.01) | 2.73 (2.28-2.96) | 1.95 (1.78-2.47) | 2.55 (1.27-4.46) |
| | **PAC** | 10.47 (9.71-11.99) | 10.19 (6.37-10.19) | 2.33 (1.54-2.80) | 4.08 (1.40-7.01) |
| **Bifix-QM** | **QTH** | 9.91 (8.39-10.19) | 5.1 (5.1-5.1) | 7.88 (6.30-8.53) | 1.66 (1.15-2.43) |
| | **LED** | 5.1 (5.1-5.91) | 5.1 (5.1-5.69) | 2.85 (2.55-3.84) | 4.33 (1.27-4.97) |
| | **PAC** | 7.64 (5.1-10.19) | 10.19 (8.88-10.19) | 5.87 (5.22-8.09) | 11.47 (10.19-12.93) |
| **Nexus** | **QTH** | 13.32 (10.67-14.3) | 10.19 (10.19-14.65) | 9.91 (8.62-10.47) | 8.66 (4.97-9.17) |
| | **LED** | 5.86 (5.1-9.55) | 10.19 (5.1-10.67) | 1.51 (1.26-2.01) | 8.13 (5.67-9.21) |
| | **PAC** | 18.59 (15.75-20.38) | 25.47 (23.75-26.31) | 17.99 (14.39-20.25) | 63.69 (59.76-70.96) |
| **RelyX Unicem** | **QTH** | 27.47 (25.48-30.38) | 35.67 (35.67-40.29) | 23.65 (21.78-28.30) | 32.33 (28.28-35.59) |
| | **LED** | 20.38 (15.29-20.86) | 17.95 (15.29-20.38) | 10,96 (8.74-13.04) | 10.5 (9.17-12.98) |
| | **PAC** | 43.07 (30.57-45.86) | 64 (61.61-67.83) | 39.03 (35.8-46.21) | 79.59 (69.38-87.91) |
| **Panavia F** | **QTH** | 33.12 (31.84-34.63) | 31.25 (29.58-33.57) | 44.78 (43.26-47.64) | 53.05 (48.49) |
| | **LED** | 29.55 (24.20-40.25) | 20.64 (19.11-28.66) | 32.22 (30.19-35.33) | 31.81 (29.01-34.03) |
| | **PAC** | 31.79 (21.91-36.93) | 77.01 (72.3-85.75) | 73.93 (73.93-76.57) | 152.14 (127.52-158.56) |

LCU=light curing unit.
QTH=quartz tungsten halogen
LED=light-emitting diode
PAC=plasma arc
The values written in italics are higher than the required values reported in ADA Specification No. 27 (7.5 μg/mm³)

**Table 2-** Median values and (25th–75th) percentiles for sorption (μg/mm³) of the resin cements tested after 24 h and 7 days

| Resin cement | SORPTION (μg/mm³) | | | | |
|---|---|---|---|---|---|
| | Polymerization modes | | | | |
| | | 24 h | | 7 days | |
| | LCU | High intensity | Soft-start | High intensity | Soft-start |
| **Duolink** | **QTH** | 10.61 (8.92-12.61) | 10.19 (10.19-10.19) | 7.13 (6.75-7.64) | 13.04 (10.23-15.54) |
| | **LED** | 10.19 (6.37-10.19) | 10.45 (10.19-10.7) | 9.68 (8.79-10.57) | 20.89 (15.8-21.91) |
| | **PAC** | 10.19 (10.19-10.19) | 10.19 (10.19-15.29) | 11.46 (7.26-15.8) | 10.70 (9.81-14.01) |
| **Bifix-QM** | **QTH** | 5.10 (5.1-6.27) | 5.10 (5.1-10.19) | 6.15 (5.1-7.53) | 4.59 (4.08-6.5) |
| | **LED** | 6.96 (5.1-7.51) | 10.19 (10.19-10.19) | 6.62 (5.22-8.97) | 7.13 (5.6-8.03) |
| | **PAC** | 5.10 (5.1-6.27) | 7.64 (5.1-10.19) | 6.88 (6.62-7.13) | 9.17 (7.77-10.06) |
| **Nexus** | **QTH** | 10.19 (10.19-14.01) | 7.64 (5.1-10.19) | 9.17 (8.79-10.38) | 8.15 (7.64-8.66) |
| | **LED** | 10.19 (5.1-10.19) | 5.10 (5.1-10.19) | 8.41 (5.1-9.68) | 8.92 (7.26-9.55) |
| | **PAC** | 10.75 (10.19-14.97) | 24.01 (16.56-25.65) | 11.97 (11.21-17.58) | 16.31 (14.27-17.70) |
| **RelyX Unicem** | **QTH** | 20.38 (16.56-25.48) | 30.57 (30.57-34.4) | 35.16 (33.63-38.6) | 26.24 (22.04-30.96) |
| | **LED** | 17.44 (15.29-20.38) | 17.83 (15.29-20.38) | 26.75 (24.97-28.41) | 16.56 (15.29-19.24) |
| | **PAC** | 30.57 (25.48-34.4) | 50.96 (45.86-59.87) | 36.18 (31.72-40) | 51.21 (48.15-55.8) |
| **Panavia F** | **QTH** | 36.69 (30.06-44.08) | 36.18 (30.64-36.6) | 36.18 (32.36-45.98) | 53.50 (46.5-56.56) |
| | **LED** | 29.55 (24.20-40.25) | 34.14 (30.45-38.47) | 32.22 (30.19-35.33) | 40.25 (35.8-44.08) |
| | **PAC** | 37.2 (35.10-38.06) | 64.33 (57.32-71.34) | 73.93 (73.93-76.57) | 106.24 (97.83-108.54) |

LCU=light curing unit.
QTH=quartz tungsten halogen
LED=light-emitting diode
PAC=plasma arc
The values written in italics higher than the required values reported in ADA Specification No.27 (40 μg/mm³)

**Table 3-** Median values and (25th–75th) percentiles for diametral tensile strength (MPa) of the resin cements

| Resin cement | LCU | Polymerization modes | |
|---|---|---|---|
| | | High intensity | Soft-start |
| Duolink | QTH | 58.26 (55.08-59.71) | 61.26 (54.4-65.26) |
| | LED | 51.80 (50.07-57.76) | 63.86 (52.40-68.44) |
| | PAC | 57.64 (50.06-62.29) | 43.58 (38.71-46.16) |
| Bifix-QM | QTH | 45.36 (37.19-50.93) | 51.21 (44.21-57.59) |
| | LED | 56.07 (51.57-63.2) | 55.86 (46.18-61.33) |
| | PAC | 46.94 (44.45-51.39) | 50.74 (46.9-58.72) |
| Nexus | QTH | 54.57 (44.96-58.09) | 54.53 (48.66-58.06) |
| | LED | 57.41 (44.99-61.02) | 63.82 (54.39-66.83) |
| | PAC | 49.65 (42.63-55.33) | 31.17 (26.92-36.14) |
| RelyX Unicem | QTH | 41.06 (38.84-45.28) | 32.87 (28.86-34.78) |
| | LED | 43.57 (38.37-47.82) | 35.57 (32.89-38.05) |
| | PAC | 34.84 (32.74-42.48) | 27.19 (24-30.24) |
| Panavia F | QTH | 36.23 (34.19-39.31) | 34.01 (30.53-37.1) |
| | LED | 44.78 (41.72-46.15) | 39.06 (34.65-41.94) |
| | PAC | 24.28 (20.20-29.82) | 27.55 (25.17-29.36) |

LCU=light curing unit.
QTH=quartz tungsten halogen
LED=light-emitting diode
PAC=plasma arc

**Table 4-** Median values and (25th–75th) percentiles for Vicker's hardness number (VHN) of the resin cements

| Resin cement | LCU | Polymerization modes | |
|---|---|---|---|
| | | High intensity | Soft-start |
| Duolink | QTH | 42.57 (40.28-43.27) | 44.03 (43.18-45.46) |
| | LED | 44.63 (43.68-45.66) | 44.13 (42.97-45.57) |
| | PAC | 36.65 (35.73-37.75) | 29.18 (28.23-30.71) |
| Bifix-QM | QTH | 52.38 (51.32-53.18) | 54.42 (54.24-57.06) |
| | LED | 54.50 (53.03-56.63) | 55.25 (52.97-56.63) |
| | PAC | 43.90 (42.51-44.38) | 33.05 (31.85-34.93) |
| Nexus | QTH | 34.55 (33.32-36.38) | 31.53 (30.77-35.43) |
| | LED | 40.63 (39.68-41.13) | 39.43 (37.59-40.84) |
| | PAC | 28.35 (27.13-29.95) | 20.15 (19.03-22.28) |
| RelyX Unicem | QTH | 47.92 (47.32-49.98) | 52.60 (50.82-53.48) |
| | LED | 52.17 (51.16-53.83) | 50.42 (48.88-51.87) |
| | PAC | 37.90 (37.16-38.52) | 21.15 (19.28-23.07) |
| Panavia F | QTH | 32.13 (31.84-34.63) | 31.25 (29.58-33.57) |
| | LED | 24.92 (24.13-27.63) | 27.57 (25.65-28.91) |
| | PAC | 15.65 (15.2-18.05) | 9.76 (8.81-11.02) |

LCU=light curing unit.
QTH=quartz tungsten halogen
LED=light-emitting diode
PAC=plasma arc

### Vicker's hardness

A total of 240 samples were polished under wet conditions with 220, 360, and 600 grit (FEPA) and placed on the platform of the tester with the surface being tested facing the diamond indenter (n=8/*per* group). The Vicker's hardness number (VHN) test was performed on the cement layer with a microhardness tester (Shimadzu HMV; Shimadzu Corporation, Tokyo, Japan) with 200 g of load application for 15 seconds. Three indentations taken for each sample were not closer than 1 mm to the margin and were averaged to determine the hardness value for each sample.

### Statistical analysis

Statistical analysis was performed using the Statistical Package for Social Sciences (SPSS) 11.5 software (SPSS Inc; Chicago, IL, USA). Whether the continuous variables were normally distributed or not was determined by using Shapiro Wilk test. Data were expressed as median ($25^{th}$-$75^{th}$) percentiles. The differences among resin cement and LCU groups were evaluated by using Kruskal Wallis test, and polymerization modes were compared by Mann Whitney U test. If the p value obtained from Kruskal Wallis test was statistically significant, multiple-comparison tests were used to determine which group differed from the others. Whether the differences between 24 h and 7 day measurements regarding solubility and sorption were statistically significant or not, was evaluated using Wilcoxon Sign Rank test ($p<0.05$). All possible subgroup analyses with Bonferroni adjustment were applied to control Type I error. A non-parametric Spearman's correlation analysis and Spearman's coefficient (rho) was used to assess the correlation between the variables.

## DISCUSSION

In this study, IPS Empresss 2 was selected as a representative ceramic material, because it has been widely used for single unit restorations as well as for three-unit fixed dental prosthesis of the anterior region extending to second premolar[6,17]. The adhesion of ceramic to dental structure with resin luting materials increases the fracture resistance of the restoration and tooth itself; it also minimizes microleakage, which may be the determining factor in the success or the failure of the treatment[6].

It is known that inadequate polymerization of resin cements might be a problem, especially under ceramic restorations[17]. Therefore, to reach maximum physical properties of resin cements; the conversion rate should be as high as possible[18]. In the present study, to simulate clinical conditions, the resin cement samples were irradiated from the top of the ceramic discs using LCUs, where the end of the light guide was in contact with the discs.

Investigations about the performance of new LCUs and polymerization modes by evaluating the physical and mechanical properties of resin-based materials are becoming increasingly available[17,27]. Under the experimental conditions of this study, the results indicated that three LCUs (QTH, LED and PAC) with different polymerization modes may point out different properties of resin cements and heterogeneity in the results.

In the literature, PAC LCUs are often discussed as an alternative to high-power LED and QTH LCUs[6,16]. However, this study indicated poor mechanical and physical properties for resin cements, polymerized with PAC LCU. The results showed that light curing with PAC had negative effects on water solubility, sorption, DTS and VHN of almost all of the resin cements, especially Panavia F, RelyX Unicem and Nexus. The special spectrum of this lamp, which has very high light intensities (2250±50 mW/$cm^2$) at certain wavelengths, might have caused this poor outcome. This might be related with the sudden polymerization shrinkage that follows curing with the PAC LCU. Additionally, high light intensity may lead the rapid development of the polymeric network in shorter molecular chains with low molecular weight and less cross-linking[9,28]. Moreover, the fact that shorter polymerization period of PAC is shorter than other LCUs might have resulted in an insufficient degree of polymerization. Besides, Uctasli, et al.[31] (2005) confirmed that PAC LCUs are so concentrated in narrow peaks (440-500 nm) that the initiator may not be decomposed in the given time.

A previous study reported that different polymerization modes can lead to resulting polymers having different structures[1]. However, in the present study, high-intensity and soft-start techniques exhibited similar values under almost all conditions, except Panavia F and RelyX Unicem in which soft-start mode of PAC LCU showed significantly higher solubility and sorption values than high-intensity mode (Tables 1 and 2). A previous study[19] reported that the two-step light-curing approach (soft-start technique) using different intensity distributions during polymerization, does not affect surface hardness. Similarly, Mehl, et al.[21] (1997) found that initial polymerization using decreased light intensity followed by high intensity has no effect on microhardness. For the DTS test performed in this study, there were no statistical differences between the polymerization modes. It could be possible that the DTS test was not sensitive to different polymerization modes, because it leads to fracture of the samples[1].

In the present investigation, the tested materials exhibited considerable differences with respect to water solubility, sorption, DTS and VHN values (Tables 1-4). The samples polymerized with PAC presented significantly more water sorption and solubility than those polymerized with LED and QTH. A hypothesis for the high solubility and sorption values for the PAC LCU could be that, a reduced polymerization time, 6 s or 10 s, is not sufficient for effective polymerization in the deepest layers of the sample, resulting in shorter chain length, thus making it more sensitive in water.

A previous study by Fleming, et al.[14] (2007) compared the water solubility, sorption and VHN of different resin composites. In that study, the authors indicated that the increased filler content

would result in reduced water sorption and solubility values. However, in the current study, Panavia F having high filler content (73% by weight) had the lowest values in almost all tests (highest values in water sorption and solubility tests). The reason for this could be that the use of hand-mixing Panavia F, unlike the other self-mixing cements tested in this study, might have resulted in mixing errors associated with different polymerization initiators and reactions. Self-mixing may help discard susceptibility to operator-induced variability[24]. Thus, unfavorable results of Panavia F could be related with the air inclusions during the mechanical mixing or consistency of the material, which might more readily accept air inclusion during the hand-mixing process[25]. Additionally, the poor results of RelyX Unicem might be due to the relatively high viscosity of the material and the limited penetration/interaction time. Higher viscosity results in lower degree of conversion increasing the mobility of molecules[7]. As the physical and mechanical properties of resin based materials are strongly influenced by the degree of conversion, Rely X Unicem showed inferior properties when compared with the other resin cements[29].

According to the ADA Specification No. 27[2], water solubility of all materials shall be less than 7.5 μg/mm$^3$ and the sorption of resin-based materials shall be less than 40 μg/mm$^3$. The median solubility values of the resin cements tested in this study were between 1.27-152.14 μg/mm$^3$ and most of them were above the required values (Table 1). In addition, the sorption values of the resin cements tested in this study were between 4.59-106.24 μg/mm$^3$ (Table 2). The sorption values except some of the values of Panavia F and RelyX Unicem indicated in Table 2, met the sorption standard's limits. Additionally, a strong positive relationship was observed between sorption and solubility values. Therefore, it is clear that the greater the amount of water absorbed, the greater the amount of components that could leach out from resin cements.

It has been shown that hardness is useful in determining the development of the mechanical properties of composites during their polymerization reaction, and that there is a direct correlation between degree of conversion and hardness development during polymerization, as a consequence of the increase in stiffness and strength of the material[27]. However, unlike degree of conversion, other important characteristics of the polymer network might affect hardness, such as the chemical structure of the monomers involved and the type and density of cross-linking. Therefore, indentation testing may give a more accurate characterization of the polymerized material than degree of conversion analysis[27]. To illustrate, a previous study by Reges, et al.[27] (2008) investigated the Knoop hardness of different shades of dual-polymerized resin cement light-activated through a ceramic restoration or not and found that hardness analysis might be more accurate than degree of conversion assessment for evaluating polymeric network structures.

On these grounds, in the current study, the hardness testing was used to evaluate the resin cement's polymerization. Furthermore, when comparing the effect of LCUs on microhardness, significant differences were observed between PAC and the other two LCUs (QTH and LED) ($p<0.05$). This finding was in accordance with a previous study that measured the hardness of luting composites and demonstrated differences between LCUs (QTH and PAC)[18]. Further on the effect of activation modes on resin cements, an investigation by Fonseca, et al.[15] (2005) related the reduced hardness of dual-curing resin cements with the partial absorption of light by the restoring esthetic materials.

As for the comparison of microhardness values of the resin cements, Duolink and Nexus showed lower microhardness values than Bifix-QM and RelyX Unicem (Table 4). This could be partially attributed to the differences in filler load, filler type, resin matrix, and formulation. The amounts of fillers used in this study were 67% and 68% by weight for Duolink and Nexus respectively, which were lower than Bifix-QM and RelyX Unicem (71-73% and 72%, respectively) (Figure 1). Moreover, Duolink and Nexus, consisting TEGDMA, presented lower values for hardness, while resin cement with Bis-GMA (Bifix-QM) showed higher values. A recent study by Moraes, et al.[22] (2010) confirmed that hardness is lower for TEGDMA-rich resin cements.

DTS is an acceptable and common test for resin based materials[8]. In a previous study, the DTS values of RelyX Unicem and Panavia F polymerized with QTH for 60 sec, were 44 MPa and 51.6 MPa, respectively[15]. In the present study, the DTS values of RelyX Unicem and Panavia F polymerized with QTH for 20 sec, were 41.06 MPa and 36.23 MPa, respectively (Table 3). These differences in DTS values might be attributed to the variation in polymerization periods.

The diametral tensile strength test might reveal different values for apparently similar materials. However, this variation could be explained by the difference between polymeric matrix, size of fillers and bond between fillers and matrix[3]. The higher the amount of TEGDMA, the more polar the organic phase, and therefore the better the interaction with the inorganic fillers, increasing the DTS[22,29]. The flexibility and low-molecular weight of TEGDMA, in addition to being responsible for its increased conversion potential, give this monomer great cross-linking capacity. Thus, high amounts of TEGDMA may enhance the 3D microstructure of

the network by increased cross-linking reactions, improving the mechanical strength[22]. Additionally, Fonseca, et al.[15] (2005) evaluated the DTS of various dual-curing resin cements and indicated that the replacement of Bis-GMA or TEGDMA by UDMA results in an increase in DTS. This result is consistent with the present study, whereby Duolink and Nexus were the compounds comprising Bis-GMA, TEGDMA and UDMA. Therefore, these cements presented higher DTS values than RelyX Unicem and Panavia F.

In the context of this study, a strong negative correlation was seen between DTS and solubility after 24 h and 7 days (rho=-0.738 and rho =-0.756, respectively, $p<0.001$), while a weak positive correlation between DTS and VHN was observed (rho=0.385, $p<0.001$). Similarly, Medeiros, et al.[20] (2007) investigated the VHN and DTS of resin composites and observed a positive correlation between these two tests. Moreover, a strong negative correlation was found between DTS and sorption after 24 h and 7 days (rho=-0.695 and rho=-0.660, respectively, $p<0.001$). Additionally, VHN was found to be strongly and negatively correlated with solubility (24 h: rho=-0.534 and 7 days: rho=-0.589, $p<0.001$) and sorption tests (24 h: rho=-0.526 and 7 days: rho=-0.539, $p<0.001$).

The present results can be compared with those obtained by previous authors only to a limited extent, as the in vitro conditions mean that a large number of different variables may affect the results of the study; that is these are not reproducible. It is worthwhile to point out that *in vitro* studies are limited in their attempt to simulate clinical conditions. Despite their limitations, *in vitro* studies are simple, repeatable and inexpensive to perform. In the present study, the first limitation was the use of water for sorption and solubility tests instead of saliva that was encountered intraorally. Another limitation concerns the fact that aging process could be useful to understand clinical behavior of these resin cements. The data obtained in this *in vitro* study should be supported by the results of clinical investigations. Further research is necessary to evaluate the effects of these LCUs on other properties of resin cements.

## CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions could be drawn:

PAC units may require considerably longer exposure times than the manufacturers' recommendations to adequately polymerize resin cements. Thereby, this may lead to better physical and mechanical properties;

High-power LED LCUs might be considered as effective as or more effective than QTH LCU for polymerization of the resin-based materials;

The results of this study do not justify the polymerization of hand-mixing Panavia F with PAC LCU;

A positive and linear correlation could be observed between DTS and VHN. Moreover, with the increase in water solubility and sorption, a decrease in DTS and VHN values could be seen.

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