Hardening of Dual-Cure Resin Cements and a Resin Composite Restorative Cured with QTH and LED Curing Units

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A b s t r a c t

Objective: The aim of this study was to determine the effects of light intensity and type of light unit (quartz-tungsten-halogen [QTH] or light-emitting diode [LED]) on the hardening of various resin cements and a resin composite restorative.

Methods: Disk specimens were prepared from 4 dual-cured resin cements (Variolink II, Calibra, Nexus 2 and RelyX ARC). Two QTH light-curing units (Visilux 2, at 550 mW/cm², and Optilux 501, at 1,360 mW/cm²) and a LED unit (Elipar FreeLight, at 320 mW/cm²) were used for curing. Specimens were light-cured or dual-cured for 10, 30 or 40 seconds with 1 of the 3 light units (curing applied to upper surface only) and were tested 24 hours after curing. Additional cement specimens were self-cured and tested at 15, 30 and 60 minutes and at 24 hours. Testing consisted of measurement of Knoop hardness number (KHN) for each specimen. Six KHN values were obtained for the upper surface only of the various cement specimens in each test group. Disk specimens 2.5 mm thick were also prepared from a resin composite restorative (XRV Herculite). These were light-cured as above, and KHN measurements were obtained for both the upper and the lower surfaces. Mean KHNs were determined, and data were analyzed with analysis of variance.

Results: The groups were significantly different (p < 0.05). High-intensity light curing resulted in the highest KHN values for all materials with any of the 3 light-curing times. For the cements, LED light curing (with both dual-curing and light-curing modes) resulted in hardness values similar to those achieved with conventional QTH light curing, although there were some exceptions. However, both LED and conventional QTH light curing resulted in inferior hardening of lower surfaces of the XRV Herculite specimens at the 3 curing times. For all cements except Nexus 2, self-curing resulted in significantly lower hardness values than dual curing. The self-curing mechanism of Variolink II cement needed a longer time to activate than those of the other cements.

Conclusions: High-intensity light curing and longer curing times resulted in the highest KHNs. The LED curing unit was associated with the lowest hardness values for lower surfaces of the resin composite restorative.

MeSH Key Words: composite resins/radiation effects; hardness; light; resin cements

Resin cements have been used in dentistry for more than 3 decades. Their uses include cementation of orthodontic brackets, periodontal splints, resin-bonded fixed partial dentures, porcelain veneers, posts and nonmetallic inlays, onlays, crowns and fixed partial dentures. In addition, they can be useful in certain situations for enhancing retention of restorations and fixed partial dentures.[1,2] For cementation of nonmetallic inlays and onlays, dual-cured resin cements are typically used, as they afford better control during the cementation procedure

Table 1 **Specifications for resin cements evaluated in this study**

Brand name	Code	Manufacturer and location	Shade
Variolink II	VRK	Ivoclar/Vivadent, Liechtenstein	Transparent
Calibra	CLB	Dentsply/Caulk, Milford, Del.	Translucent
Nexus 2	NXS	Kerr USA, Orange, Calif.	Translucent
RelyX ARC	RLX	3M/ESPE, St. Paul, Minn.	A1-transparent

and, in deep areas where the curing light cannot penetrate, the self-curing mechanism hardens the cement. However, a number of studies have indicated that the self-curing mechanism of some dual-cure cements is inadequate.[3–6] Other studies have reported an inverse relation between the thickness of ceramic inlays and the hardening of light-cured and dual-cured resin cements.[6–10] Furthermore, a study that investigated hardening of 3 dual-cured cements under resin composite inlays reported that with self-curing alone, hardening of the cements was insufficient when light was attenuated by tooth and restoration material.[11] Therefore, as newer versions of resin cements are introduced to the market, they must be examined to ensure that they meet the needs of dentists for their various applications.

Light curing of restorative resin composites and cements is accomplished with quartz-tungsten-halogen (QTH) light-curing units, plasma arc light-curing units or, more recently, light-emitting diode (LED) light-curing units. Although laser-based light-curing units were made available in the late 1980s they never gained popularity. Solid-state LEDs use junctions of doped semiconductors based on gallium nitride to directly emit light in the blue region of the spectrum, without the use of filters.[12] LED units have certain advantages over conventional light-curing units: many of them are wireless, and the LEDs have an estimated lifetime of about 10,000 hours (in contrast, QTH bulbs have a lifetime of 50 to 100 hours).[13,14] However, a number of newly introduced LED light-curing units have limited light output, and their effectiveness in curing resin composites and resin cement has not been fully investigated. Also, new versions of QTH light-curing units that provide high-intensity light (more than 800 mW/cm²) have recently become available. Some of these units can emit light with an intensity greater than 1,300 mW/cm² if special turbo light guides are used.

The aim of this investigation was to evaluate the hardening of a group of dual-cure resin cements cured by conventional and high-intensity QTH and LED light-curing units. Also, the hardening of a resin composite restorative was investigated to ensure efficacy of a new LED light-curing unit.

Materials and Methods

Four dual-cure resin cements were examined in this study (**Table 1**). Disk specimens measuring 2.5 mm in thickness and 4 mm in diameter were prepared from each of these cements. For 3 of the cements, 3 sets of specimens were prepared: light-cured, dual-cured and self-cured; only dual-cured and self-cured specimens were prepared from RelyX Arc transparent cement (RLX). Three light-curing units were used, 2 based on QTH (Visilux 2, 3M/ESPE, St. Paul, Minn., with 550 mW/cm² intensity; Optilux 501, Kerr USA, Orange, Calif., with 1,360 mW/cm² intensity) and 1 based on LED (Elipar Freelight, 3M/ESPE, with 320 mW/cm² intensity). The LED light unit incorporates several LEDs, and light is emitted through a regular fibre-optic light guide. The light intensity of each curing unit was measured by means of a light meter (Optilux, model 100, Kerr USA). When dual-cured or self-cured specimens were prepared, the manufacturers' instructions for proportioning and mixing of the cement were followed. Steel rings were used for specimen preparation. For each specimen, a ring was placed on a glass section lined with a Mylar polyester strip, filled with the cement, and covered with another Mylar-lined glass section; the 2 glass sections were then pressed together with 2 clamps. Light curing was applied only to the upper surface of specimens, according to the group's test conditions. The surface of the light guide was placed directly in contact with the glass section covering the upper surface of the specimen. Light-cured and dual-cured specimens were subjected to light curing for 10, 30 or 40 seconds with 1 of the 3 light-curing units. Two specimens were prepared for each test condition. All specimens were stored dry in boxes in a darkened incubator at 37°C for 24 hours before testing.

Another set of specimens was prepared from the 4 cements according to the procedure described above but without light curing. The resulting specimens were stored as above and subjected to hardness testing at 15, 30 and 60 minutes and 24 hours after mixing.

A hardness tester with a Knoop indenter and 30-g weight (Tukon 300, Acco Industries Inc., Wilson Instrument Division, Bridgeport, Conn.) was used for testing the hardness of each specimen. Six readings were obtained from the upper surface only of each cement specimen in each test group. Mean Knoop hardness numbers (KHNs) were then calculated. Data were analyzed statistically with analysis of variance (ANOVA) and Tukey's test.

To determine the efficacy of the 3 curing sources in hardening a resin composite restorative (XRV Herculite, shade A2, Kerr USA), specimens measuring 2.5 mm in

Table 2 **Knoop hardness numbers for 3 cements subjected to light curing only**

Intensity and curing time	Cement; mean Knoop hardness number ± SD		
	Variolink II transparent	Calibra translucent	Nexus 2 translucent
1,360 mW/cm²			
10 s	39.1 ± 3.4	5.1 ± 0.7	9.3 ± 0.4
30 s	46.0 ± 2.0	19.9 ± 1.9	27.6 ± 1.5
40 s	45.3 ± 1.5	34.4 ± 3.2	33.9 ± 3.6
550 mW/cm²			
10 s	30.6 ± 2.1	1.8 ± 0.3	6.0 ± 0.7
30 s	41.4 ± 1.6	8.8 ± 1.0	23.1 ± 2.8
40 s	48.3 ± 1.3	17.8 ± 0.8	26.5 ± 1.2
LED			
10 s	35.7 ± 0.7	3.7 ± 0.5	8.1 ± 1.1
30 s	36.2 ± 0.8	13.5 ± 0.5	19.9 ± 0.6
40 s	38.7 ± 2.4	20.6 ± 2.3	25.1 ± 1.4

SD = standard deviation.

Table 3 **Knoop hardness numbers for 4 cements subjected to dual curing**

Intensity and curing time	Cement; mean Knoop hardness number ± SD			
	Variolink II transparent	Calibra translucent	Nexus 2 translucent	RelyX ARC A1-transparent
1,360 mW/cm²				
10 s	51.6 ± 2.9	38.2 ± 1.5	40.9 ± 1.0	42.7 ± 3.8
30 s	52.9 ± 2.9	40.1 ± 1.9	46.6 ± 1.9	57.8 ± 2.8
40 s	58.6 ± 2.0	43.4 ± 1.9	52.0 ± 2.4	60.0 ± 2.4
550 mW/cm²				
10 s	39.8 ± 3.8	19.6 ± 1.4	24.7 ± 1.5	35.4 ± 2.0
30 s	44.4 ± 2.4	34.3 ± 1.4	44.7 ± 2.8	41.8 ± 1.5
40 s	50.5 ± 1.2	40.4 ± 2.7	46.8 ± 1.7	49.0 ± 3.6
LED				
10 s	48.3 ± 2.5	29.0 ± 2.5	29.1 ± 2.4	45.3 ± 2.3
30 s	48.3 ± 2.5	32.0 ± 2.7	34.1 ± 3.1	52.3 ± 1.7
40 s	49.0 ± 2.6	45.6 ± 1.0	43.0 ± 2.4	53.2 ± 2.7

SD = standard deviation.

thickness were prepared and light-cured (curing applied to upper surface only) for 10, 30 or 40 seconds with 1 of the 3 light-curing units. Both the upper and the lower surfaces of these specimens were subjected to Knoop hardness measurements (6 measurements for each surface under each test condition). Mean KHNs were calculated and the data analyzed with ANOVA.

Results

ANOVA indicated significant differences in mean KHN among the 3 cements that were subjected to light curing only (**Table 2**) ($p < 0.001$). For all 3 of these cements the KHN increased with increasing curing time (see **Figs. 1** to **3** at http://www.cda-adc.ca/jcda/vol-70/issue-5/323.html) and increasing light intensity. The mean KHN values for Variolink II cement (VRK) were significantly higher than those of the other 2 cements (Calibra [CLB], Nexus 2 [NXS]) under all test conditions (**Table 2**). In particular, the VRK cement achieved a greater degree of hardness with the shortest light-curing time than did the other 2 cements (**Fig. 1**), both of which needed more curing time to achieve a given level of hardness.

ANOVA revealed significant differences in mean KHN among the 4 cements when subjected to dual curing (**Table 3**) ($p < 0.001$). As with light curing, the hardness of all cements increased with increasing light-curing time (from 10 to 40 seconds; see **Figs. 4** to **6** at http://www.cda-adc.ca/jcda/vol-70/issue-5/323.html). After 10 seconds of curing, the hardness of the VRK cement was significantly higher than that of the other 3 cements, except for the RelyX Arc cement (RLX) with LED light curing (**Table 3**, **Fig. 4**). For all cements, the highest values of KHN at 10 seconds of curing were achieved with high-intensity light (1,360 mW/cm²) (**Table 3**, **Fig. 4**). After 30 seconds of curing the RLX cement had the highest values of KNH with both high-intensity and LED light curing but not with conventional light curing (550 mW/cm²) (**Table 3**, **Fig. 5**). For all cements, high-intensity light curing resulted

Table 4 **Knoop hardness numbers for the 4 cements with self-curing only**

	Cement; mean Knoop hardness number ± SD			
Time from mixing	Variolink II transparent	Calibra translucent	Nexus 2 translucent	RelyX ARC A1-transparent
15 min	–	12.1 ± 1.8	8.0 ± 0.5	3.2 ± 0.3
30 min	–	18.7 ± 2.6	8.9 ± 1.9	3.8 ± 0.2
60 min	–	26.7 ± 2.6	13.3 ± 0.9	4.3 ± 1.2
24 h	31.1 ± 2.1	35.4 ± 7.0	43.4 ± 1.3	25.3 ± 2.8

SD = standard deviation.

Table 5 **Knoop hardness numbers for XRV Herculite A2 subjected to light curing**

	Mean Knoop hardness number ± SD	
Intensity and curing time	Top surface	Bottom surface
1,360 mW/cm²		
10 s	61.9 ± 1.5	8.3 ± 0.4
30 s	65.8 ± 2.5	37.7 ± 1.3
40 s	70.2 ± 1.4	45.9 ± 2.3
550 mW/cm²		
10 s	48.6 ± 1.3	0.0
30 s	62.6 ± 1.7	16.0 ± 1.1
40 s	63.5 ± 1.6	25.4 ± 1.6
LED		
10 s	49.5 ± 2.5	0.0
30 s	53.2 ± 2.4	13.3 ± 0.3
40 s	60.8 ± 1.1	18.0 ± 1.4

SD = standard deviation.

in higher KHN values at both 10 and 40 seconds than was achieved with conventional and LED light curing (with the exception of the RLX cement at 10 seconds and CLB at 40 seconds) (**Table 3**).

ANOVA indicated significant differences in mean KHN among the 4 cements when subjected to self-curing only (**Table 4**) ($p < 0.001$). The VRK cement did not harden within the first hour after mixing (**Table 4**; see **Fig. 7** at http://www.cda-adc.ca/jcda/vol-70/issue-5/323.html). However, after 24 hours it had achieved a reasonable degree of hardness, and its hardness was greater than that of the RLX cement (**Fig. 7**). The CLB cement reached a degree of hardening after 15 minutes that was equivalent to a third of its hardness at 24 hour, whereas the NXS and RLX cements reached less that 20% of their 24-hour hardness after 15 minutes (**Table 4, Fig. 7**). At 24 hours the NXS cement had the highest mean KHN (**Table 4**).

Variability in light intensity had little effect on the upper surface of the XRV Herculite specimens but did have a significant effect on the lower surfaces (**Table 5**; see **Figs. 8** to **10 at** http://www.cda-adc.ca/jcda/vol-70/issue-5/323.html). At 10 seconds, both conventional QTH and LED light curing failed to harden the lower surface of the specimens (**Fig. 8**). However, at 30 and 40 seconds some hardening took place (**Figs. 9** and **10**).

Discussion

The hardness of resinous materials measured at different stages of the polymerization reaction can be a useful indicator of the degree of monomer conversion. Typically, the harder the material becomes during polymerization, the greater the degree of monomer conversion. One study indicated a good correlation between KHN and degree of monomer conversion for 3 unfilled dental restorative resins.[15] Similar findings were reported in another study, in which 5 commercial resin composite materials were examined.[16] However, an absolute hardness number cannot be used to predict degree of monomer conversion in comparisons of different resinous materials.[15] Therefore, the findings of the study reported here can be considered reliable indicators of the degree of monomer conversion only for the materials examined. The authors of a study that investigated the degree of monomer conversion of 4 different resin cements found no evidence to indicate that the degree of monomer conversion for a chemically induced reaction was any greater at 24 hours after mixing than at 60 minutes.[17] That conclusion does not agree with the findings of the study reported here. In the current study, the hardness of 3 of the cements continued to increase from the time of mixing up to 24 hours after mixing when subjected to self-curing (**Table 4**). However, the degree of monomer conversion was not measured directly, and it is possible that variability in the formulations of the cements might be the reason for this difference between the 2 studies, given that different cements were investigated. The relatively small standard deviations reported in **Tables 2** to **4**, which did not exceed 10% of the means, indicate the reliability and appropriateness of the hardness test used and justify the number of KHN measurements that were obtained for each material under each test condition.

Compared with nonpolymeric cements, resin cement kits are more expensive, but in a dental office, it may be more economical to have a single resin cement kit that can be used for self-curing, dual curing or light curing. The VRK cement failed to harden in the self-curing mode within 1 hour after mixing and therefore should not be used with this method of curing. The other 3 cements had various hardening patterns. After 1 hour of self-curing, the

CLB, NXS and RLX cements had mean KHN values of 26.7, 13.3 and 4.3, respectively (**Table 4**). These values represent 75.4%, 30.6% and 17.0%, respectively, of the hardness of these cements after 24 hours of self-curing (**Table 4**). Therefore, the CLB and NXS cements would perhaps be better candidates for use in the self-curing mode. These findings agree with previous reports that Nexus and Enforce cements (the original versions of the NXS and CLB cements used here) performed well in the self-curing mode.[6,18]

Nonetheless, mean KHN values for self-cured specimens at 24 hours were significantly lower than those obtained when specimens were dual cured for 40 seconds with high-intensity QTH light. This observation agrees with the findings of a recent study that determined the degree of curing of a group of orthodontic resin cements with infrared spectroscopic techniques.[19] The dual-cured cements demonstrated the highest degree of curing, whereas the self-cured ones had the lowest degree of curing.[19] In the current study, the mean KHN for CLB cement after 24 hours of self-curing was 81.6% of the value obtained with dual curing for 40 seconds with high-intensity light; the corresponding values for NXS and RLX cements were 83.5% and 42.2%, respectively. Also, the hardness values obtained with light curing only were lower than those obtained with dual curing. The mean KHN value for VRK cement after 40 seconds of high-intensity light curing was 77.3% of the corresponding value obtained with dual curing, whereas for the CLB and NXS cements, the percentages were 79.3% and 65.2%, respectively. Variability in the hardening patterns of the cements must be related to their chemical composition. The clinical significance of this finding is that dentists must be cautious about the potential for microleakage in the early hours after cementation of an indirect restoration with a resin cement when self-curing is used, if the self-curing reaction is slow or delayed. In the oral environment this may result in wash-out of the uncured cement with subsequent open margin, which could lead to postoperative sensitivity.

With the resin composite restorative, variability in light intensity had a detrimental effect on hardening of the lower surfaces of the specimens. Surprisingly, the new LED unit had the worst performance in this respect. Although 2.5 mm (the thickness of specimens in this study) might be at the upper limit of accepted thickness for a resin composite increment used for restoration, dentists do not have means to accurately determine the thickness of each resin composite increment they place into a prepared cavity. Thus, in large cavities in molars the thickness of a composite resin increment might reach this level. High-intensity QTH applied for an appropriate period of time is clearly a better option, as this curing mode will ensure sufficient hardening of the lower surface of thick increments and

hence thorough polymerization.[20–22] Rueggeberg and others[22] indicated that the incremental layer thickness of composites should not exceed 2 mm, with 1 mm being ideal. They recommended exposure time of 60 seconds with a light intensity of at least 400 mW/cm^2; however, 40 seconds of exposure was deemed sufficient. Insufficient hardening of resin composite may result in postoperative sensitivity, as well as possible accelerated wear or degradation of the restoration in the oral environment.

Johnston and others,[23] who examined hardening of 2 light-activated products using 2.5 mm thick composite specimens, suggested that depth of polymerization may be based on a relative hardness value (hardness of lower surface/hardness of upper surface × 100) and, for practical purposes, suggested a ratio of 90%.[23] Yearn[24] used 80% relative hardness as a standard for adequate depth of polymerization. However, there is no internationally recognized standard for adequate depth of polymerization as measured by the relative hardness method. The authors of the current work suggest that 80% or higher relative hardness for composite specimens 2 mm thick should be used as a standard.

Conclusions

Within the limitations of the test conditions of this in vitro investigation the following conclusions can be reached:

1. High-intensity light curing resulted in a consistently greater degree of hardness for all resin-based materials tested at the 3 curing times.

2. With some exceptions, LED light curing resulted in hardness of upper surfaces of cement specimens similar to that achieved with conventional QTH light curing.

3. For all cements except NXS, self-curing resulted in significantly lower hardness values than dual curing.

4. The self-curing mechanism of VRK cement needed more time to activate than the mechanisms of the other cements.

5. LED light curing resulted in the lowest KHN values on the lower surfaces of the composite restorative specimens. ❧

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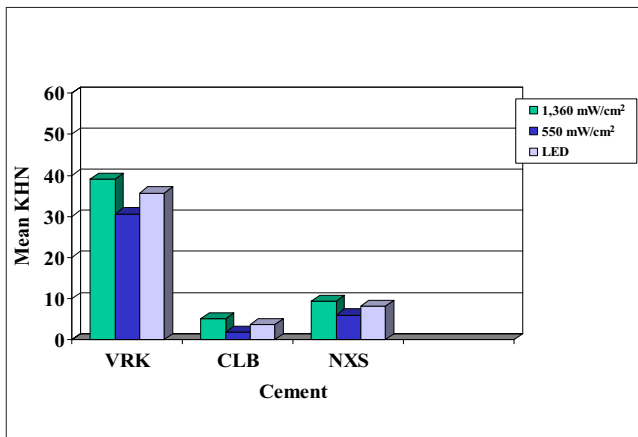
Figure 1: Mean hardness of dual-cured resin cements subjected to light curing only for 10 seconds. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2.
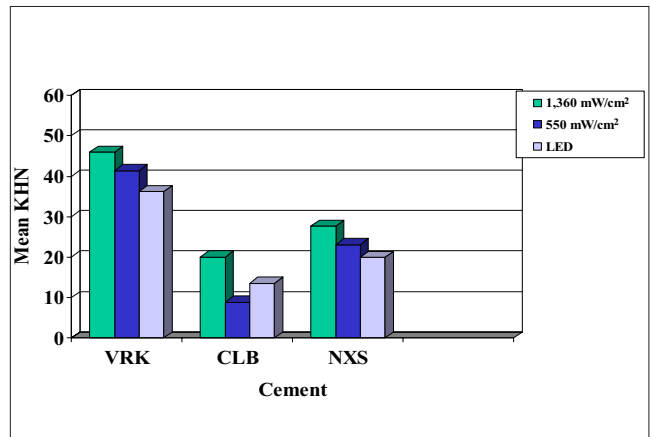

Figure 2: Mean hardness of dual-cured resin cements subjected to light curing only for 30 seconds. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2.
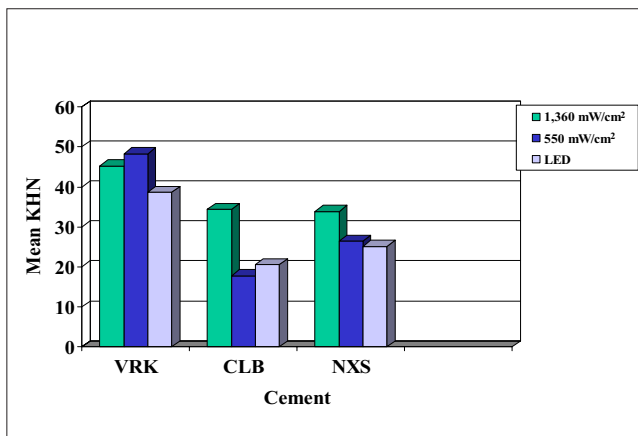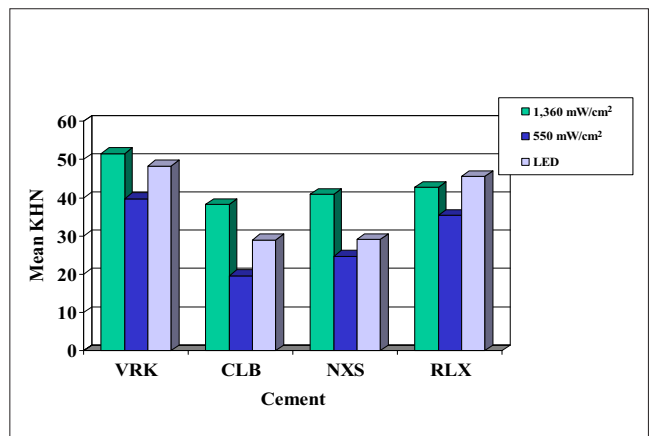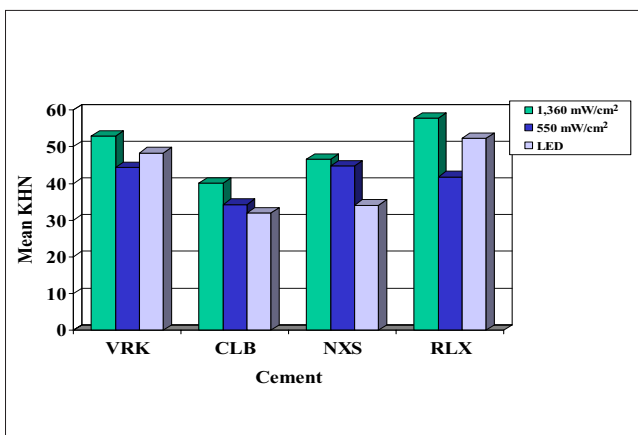

Figure 3: Mean hardness of dual-cured resin cements subjected to light curing only for 40 seconds. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2.

Figure 4: Mean hardness of dual-cured resin cements subjected to dual curing for 10 seconds. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2, RLX = RelyX Arc.
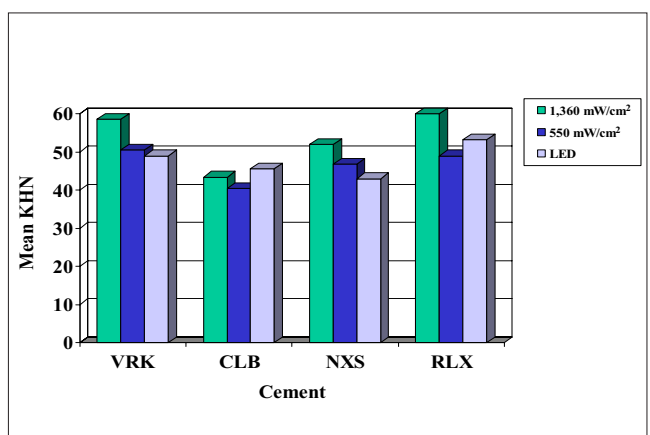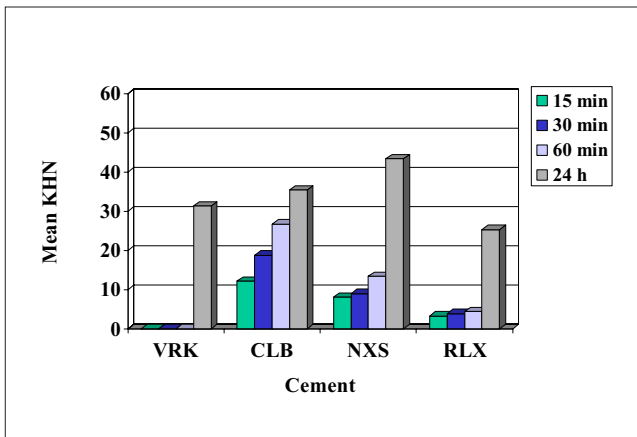

Figure 5: Mean hardness of dual-cured resin cements subjected to dual curing for 30 seconds. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2, RLX = RelyX Arc.

Figure 6: Mean hardness of dual-cured resin cements subjected to dual curing for 40 seconds. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2, RLX = RelyX Arc.

Figure 7: *Mean hardness of dual-cured resin cements with self-curing only. KHN = Knoop hardness number, VRK = Variolink II, CLB = Calibra, NXS = Nexus 2, RLX = RelyX Arc.*
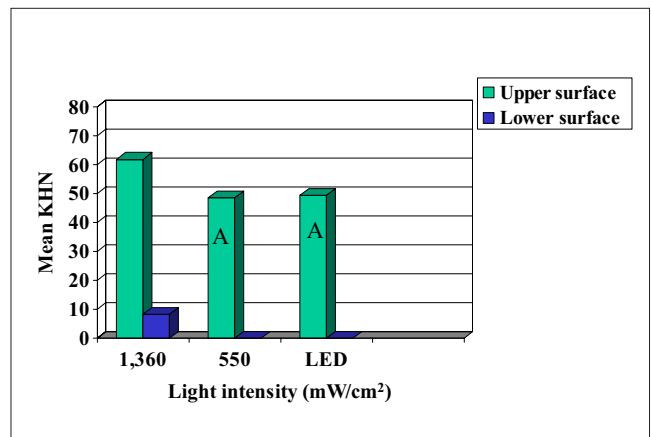

Figure 8: *Mean hardness of XRV Herculite A2 restorative subjected to light curing for 10 seconds. KHN = Knoop hardness number. A = Means are not significantly different.*
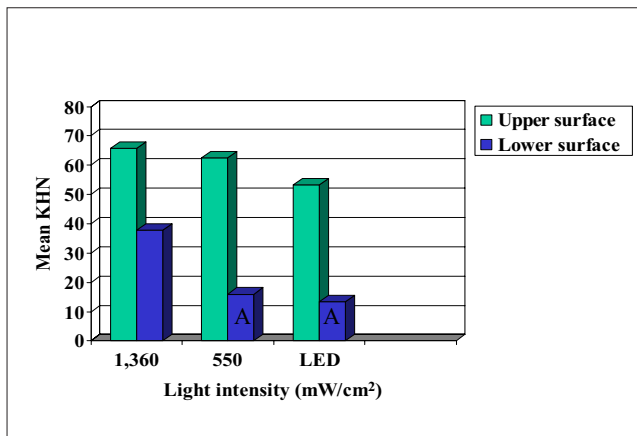

Figure 9: *Mean hardness of XRV Herculite A2 restorative subjected to light curing for 30 seconds. KHN = Knoop hardness number. A = Means are not significantly different.*
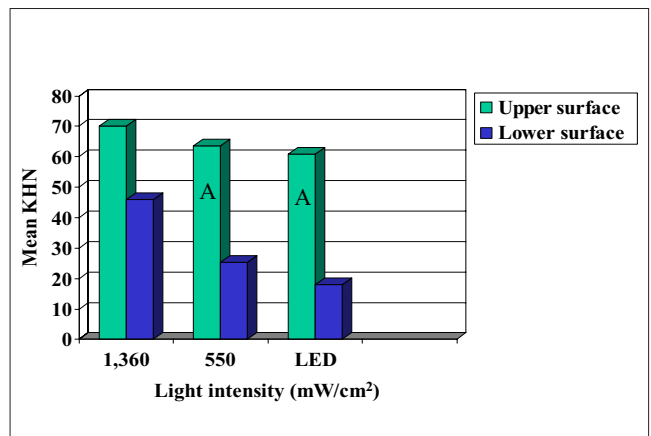

Figure 10: *Mean hardness of XRV Herculite A2 restorative subjected to light curing for 40 seconds. KHN = Knoop hardness number. A = Means are not significantly different.*