# Changes in Degree of Conversion and Microhardness of Dental Resin Cements

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### Clinical Relevance

Chemical- and dual-cured resin cements, as well as light-cured resin cements, appear to be cured within the first 24 hours post-mix or post-light activation with no further significant changes in the degree of conversion or microhardness.

# **SUMMARY**

There are few studies available on the post-light activation or post-mix polymerization of dental

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resin cements as a function of time. This *in vitro* study evaluated the successive changes in the degree of conversion (DC) and microhardness during polymerization of six commercial resin cements (light-cured [Choice 2, RelyX Veneer], chemical-cured [Multilink, C&B Cement] and dual-cured [Calibra, RelyX ARC]) within the first 24 hours and up to seven days.

Resin specimens were prepared for Fourier transform infrared (FTIR) spectroscopy and microhardness testing to determine the DC and Vickers hardness (VH), respectively. The light-cured materials or mixed pastes of the dual-cured materials were irradiated with a light-curing unit (Elipar TriLight) through a precured composite overlay for 40 seconds. The FTIR spectra and microhardness readings were taken at specified times: 1, 2, 5, 10, 15, 30 and 60 minutes; 24 hours and after two days and seven days.

According to the FTIR study, most of the curing reaction of Choice 2 and RelyX Veneer occurred within 10 and 30 minutes, respectively. Multilink, C&B Cement and Calibra exhibited gradual increases in the DC up to 24 hours, with no further statistically significant increase (p>0.05). RelyX ARC attained a DC value within five minutes, similar to that at seven days (p>0.05). Choice 2 and RelyXARC showed gradual increases in the VH, up to 15 minutes, with no further significant change over the remaining observation time (p>0.05). For RelyX Veneer, Multilink, C&B Cement and Calibra, there were no significant increases in the VH value after 24 hours (p>0.05). The light-cured materials produced significantly higher DC values than the chemicalcured materials (p<0.05). The DC values of the two dual-cured resin cements were significantly different from each other (p<0.001). The results suggest that the significant polymerization reaction was finished within 24 hours post-mix or post-light activation for all resin cements tested.

### INTRODUCTION

Dental resin cements are generally composites, which provide one or more polymerization modes (light, chemical or dual) to meet different clinical requirements. 1-2 Visible light-cured systems are appropriate where light can penetrate through the restoration, since adequate light is needed to ensure optimal polymerization. 3 Meanwhile, chemical-cured systems are able to uniformly set even at the bottom of deep cavities, where access for light-curing is limited. 4 Dual-cure versions were developed in an attempt to combine the most desirable properties of the light- and chemical-cured systems. 5

Light-cured resin cements can polymerize both during and after light activation. Although post-irradiation polymerization continues for 24 hours, most polymer-

ization occurs within the first 10 to 15 minutes after light exposure.6-7 Even though an increased amount of an amine in the base (accelerator) can expedite the setting speed of the mixed material,8 chemical-cured materials set at much slower rates than light-cured materials.9-10 dual-cured resin cements, the chemical polymerization reaction starts as soon as the base and catalyst are mixed; whereas, the system is light-curable at any time during the chemical polymerization period. Several studies have suggested, though, that the majority of dual-cured resin cements should be light-cured to produce higher rates of conversion and hardness values. <sup>11-12</sup> In a previous study by Rueggeberg and Caughman, <sup>13</sup> the cure that observed 10 minutes post-mix in several dual-cured resin cements was almost equivalent to the cure after 24 hours.

There seems to be a common belief that chemical-cured and even light-activated dual-cured resin cements take longer (more than 24 hours) to finish their polymerization reaction than light-cured resin cements. Nonetheless, most studies for polymer-based dental materials employ a 24-hour (water) storage condition after the start of mixing (chemical-cured materials) or irradiation (light- or dual-cured materials).<sup>2,14</sup> Although several studies have examined post-irradiation polymerization using commercial or experimental composites, <sup>7,13,15-16</sup> few research papers have reported the post-mix or post-irradiation polymerization of chemical- or dual-cured resin cements as a function of time.

Accordingly, the current study evaluated successive changes in the degree of conversion (DC) and microhardness of six commercial resin cements (light-, chemical- and dual-cured) within the first 24 hours and up to seven days.

# **METHODS AND MATERIALS**

Two light-cured, two chemical-cured and two dual-cured resin cements were investigated in this study (Table 1). To polymerize the light- or dual-cured resin cements under a simulated restoration, a composite disk was prepared using a composite (Esthet-X, shade A2, Dentsply Caulk, Milford, DE, USA) 1.5 mm thick and 5 mm in diameter.<sup>16-17</sup>

Material	Curing Method	Manufacturer	Batch # (shade)
Choice 2	Light-cured	BISCO, Inc Schaumburg, IL, USA	0700005973 (Translucent)
RelyX Veneer		3M ESPE St Paul, MN, USA	8FP (Translucent)
Multilink	Chemical-cured	Ivoclar Vivadent, Schaan, Liechtenstein	K06460 (Yellow)
C&B Cement		BISCO, Inc Schaumburg, IL, USA	Base: 0800011982 (Natural); Catalyst: 0800013105
Calibra	Dual-cured	Dentsply Caulk Milford, DE, USA	Base: 080410 (Translucent); Catalyst: 060202
RelyX ARC		3M ESPE St Paul, MN, USA	FU8HT (Transparent [A1])

The DC of the materials was determined using a Fourier transform infrared (FTIR) spectroscope (IRPrestige-21, Shimadzu Corp, Kyoto, Japan) with an attenuated total reflectance (ATR) unit (MIRacle, Pike Technologies Inc, Madison, WI, USA). A piece of 50 umthick adhesive tape containing a circular hole (5 mm in diameter) was placed on a polyester strip over a glass slide. A small amount of each light-cured material was placed into the hole, covered with another polyester strip and gently pressed to expel the excess material. The resin cement was then irradiated for 40 seconds by placing the end of the light guide of a light-curing unit (Elipar TriLight, 3M ESPE, Seefeld, Germany; standard mode) onto the top of the composite disk, so that the light could transmit through the disk and polyester strip over the material. The output intensity of 750 mW/cm<sup>2</sup> was constantly monitored during the experiment by a built-in radiometer. For the chemical- or dual-cured materials, equal amounts of base and catalyst were mixed into a uniform paste for 10 seconds, then transferred into the prepared hole over the polyester strip and glass slide. The material underwent chemical curing without irradiation or dual-curing with irradiation as described above. Fifteen specimens were prepared for each material. The top surface of each thin resin film was pressed against an ATR prism. The absorbance spectrum was acquired by scanning the specimens 10 times over a 1670–1550 cm<sup>-1</sup> range with a resolution of 4 cm<sup>-1</sup>. The DC was determined from the aliphatic C=C peak at 1638 cm<sup>-1</sup>, while the aromatic C=C peak at 1608 cm<sup>-1</sup> was used as the internal calibration for calculating the final value. The DC was then calculated by comparing the height of the peaks for the methacrylate vinyl group in the cured material against that in the uncured material, using the following for-

$$DC(\%) = (1 - C/U) \times 100$$

where C and U are the absorption peaks for the cured and uncured materials, respectively.

For the microhardness test, 15 specimens per resin cement were prepared by placing the material into a round Teflon mold (5 mm in diameter, 2.5 mm in depth) and covered with a polyester strip. The curing protocol was the same as that conducted for the DC measurement. Using a Vickers hardness tester (HMV-2, Shimadzu Corp), three indentations were made on the top surface of each specimen along a middle line with each indentation separated by approximately 0.5 mm. To make the indentations, a 10 second dwell time and a 100 g load were chosen. The Vickers hardness (VH) of each specimen was recorded as the average of the three readings.

The FTIR spectra for the DC calculation and microhardness readings of the specimens were taken at specified times (post-irradiation for light-cured materials and post-mix for chemical- or dual-cured materials): 1, 2, 5, 10, 15, 30 and 60 minutes; 24 hours and after two days and seven days. Between each observation, the specimens were stored in dark, dry conditions at 37°C. <sup>17</sup>

All the data were statistically analyzed by non-parametric methods at  $\alpha$ =0.05. Since the data were not normally distributed, the difference between the DC or microhardness value at each specified time and the final value at seven days within the same resin cement was analyzed using the Wilcoxon signed rank test. In the microhardness test, the specimens to be observed at one and two minutes were prepared separately, because there was a problem in attempting to match the start of reading with the time required for the specimen preparation. In those cases, the Mann-Whitney test was adopted to analyze the difference between the microhardness value at one and two minutes and the final value at seven days. The DC data after seven days post-irradiation or post-mix among the resin cements tested were also analyzed by the Mann-Whitney test. The significance levels were adjusted using the Sidak correction for multiple testing. All the statistical analyses were performed using SPSS 14.0 for Windows (SPSS Inc, Chicago, IL, USA).

## **RESULTS**

The rates of change in the DC between the two neighboring observation times are presented in Figure 1. Choice 2 (BISCO, Inc, Schaumburg, IL, USA) produced a similar, yet slightly faster curing reaction to RelyX Veneer (3M ESPE, St Paul, MN, USA). Multilink (Ivoclar Vivadent, Schaan, Liechtenstein) and C&B Cement (BISCO, Inc) exhibited small changes in the DC up to two minutes, then marked increases at 5 and

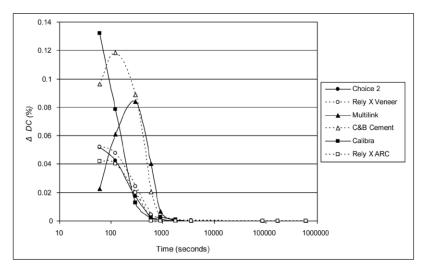


Figure 1: Rate of change in degree of conversion (DC) between two neighboring survey times. The x-axis of the plot is scaled logarithmically.

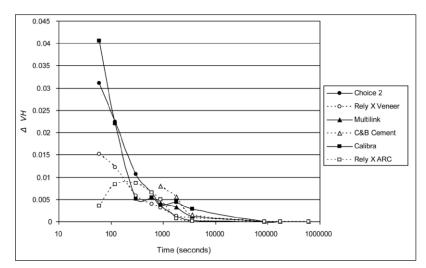


Figure 2: Rate of change in Vickers hardness (VH) between two neighboring survey times. The x-axis of the plot is scaled logarithmically. For Multilink and C&B Cement, no measurable data were obtained from 1 to 10 minutes.

10 minutes. Calibra (Dentsply Caulk) and RelyX ARC (3M ESPE), both dual-cured materials, showed a difference in the rate of change from each other during the early observation period. In terms of DC, all the materials reached a plateau at approximately 30 minutes.

Figure 2 shows the rates of change in the microhardness values between two neighboring observation times. As for microhardness, all the materials attained a plateau at 24 hours.

The DC values of the six resin cements after the specified post-irradiation or post-mix times are presented in Table 2. Choice 2 and RelyX Veneer showed no further significant increases in value after 10 and 30 minutes, respectively. when compared to the values at seven days, according to the Wilcoxon signed rank test (p>0.05). Multilink and C&B Cement both exhibited gradual increases in the DC up to 24 hours, after which there were no further significant increases for seven days (p>0.05). Statistical changes in the DC values for Calibra were similar to those for the two chemicalcured materials (p>0.05), whereas RelyX ARC attained a DC value within five minutes, which was similar to that at seven days (p>0.05).

Table 3 presents the microhardness values of the resin cements for a period of seven days. For Multilink and C&B Cement, both chemical-cured materials, the surface of the specimens from 1 to 10 minutes showed no measur-

able microhardness. The light-cured material Choice 2 and dual-cured material RelyX ARC showed gradual increases in microhardness up to 15 minutes, with no further statistical change throughout the remaining observation time (p>0.05). For RelyX Veneer, Multilink, C&B Cement and Calibra, there were no significant increases in value after 24 hours (p>0.05).

The final DC values of the resin cements after seven days post-irradiation or post-mix are summarized in Figure 3. The light-cured materials produced signifi-

Table 2: Degree of Conversion (DC) (%) of Resin Cements After Specified Post-irradiation or Post-mix Times										
Material	1 Minute	2 Minutes	5 Minutes	10 Minutes	15 Minutes	30 Minutes	60 Minutes	24 Hours	2 Days	7 Days
Choice 2	46.72 (2.77)*	49.83 (2.97)	55.72 (4.12)	56.28 (2.99)	56.98 (2.66)	57.10 (3.03)	57.62 (2.18)	58.53 (1.97)	58.92 (2.52)	58.97 (3.32)
RelyX Veneer	39.97 (3.43)	43.10 (4.25)	50.95 (2.24)	52.33 (2.58)	53.61 (2.94)	54.82 (2.67)	55.84 (2.67)	57.31 (2.31)	57.72 (2.76)	58.01 (2.85)
Multilink	0.85 (0.35)	2.22 (0.34)	20.12 (5.59)	40.71 (3.49)	44.35 (2.50)	45.35 (1.59)	45.59 (1.97)	49.79 (2.78)	51.20 (5.14)	51.08 (5.55)
C&B Cement	1.41 (0.33)	7.18 (1.66)	32.45 (2.72)	43.71 (1.86)	44.63 (2.77)	45.41 (2.39)	45.84 (2.11)	49.44 (2.50)	49.63 (4.70)	52.01 (2.47)
Calibra	29.80 (2.18)	37.72 (1.02)	42.23 (3.46)	42.36 (3.64)	43.56 (2.60)	44.62 (1.61)	45.18 (1.61)	47.36 (1.57)	47.79 (2.92)	48.15 (2.28)
RelyX ARC	55.16 (2.77)	57.67 (1.98)	64.62 (3.37)	64.83 (1.93)	64.60 (2.83)	65.14 (2.95)	64.76 (2.89)	65.74 (2.30)	66.14 (1.91)	66.71 (2.23)
,	55.16 (2.77)	57.67 (1.98)	64.62 (3.37)	64.83 (1.93)	64.60 (2.83)	65.14 (2.95)	64.76 (2.89)	65.74 (2.30)	66.14 (1.91)	66.7

N=15, mean (standard deviation)

t: Within a row, each underlined value is significantly different from the value at seven days according to Wilcoxon signed rank test at lpha=0.05.

Table 3: V	Table 3: Vickers Hardness (VH) of Resin Cements After Specified Post-irradiation or Post-mix Times									
Material	1 Minute	2 Minutes	5 Minutes	10 Minutes	15 Minutes	30 Minutes	60 Minutes	24 Hours	2 Days	7 Days
Choice 2	33.39 (3.54)*	<u>35.26 (4.21)</u>	<u>37.73 (3.97)</u> #	40.01 (3.66)	41.65 (4.27)	43.52 (4.61)	44.51 (5.43)	46.19 (6.69)	46.26 (3.06)	46.50 (6.66)
RelyX Veneer	<u>17.85 (2.05)</u>	<u>18.76 (1.22)</u>	20.41 (2.18)	21.15 (1.63)	22.77 (1.32)	23.58 (1.88)	26.75 (2.97)	31.81 (3.62)	31.49 (3.23)	32.67 (2.86)
Multilink	**	**	**	**	6.96 (0.62)	10.69 (1.03)	15.00 (0.64)	19.85 (2.10)	21.55 (1.74)	21.64 (3.77)
C&B Cement	**	**	**	**	8.66 (1.06)	15.77 (1.05)	21.34 (1.13)	27.39 (2.10)	29.17 (1.52)	29.11 (2.98)
Calibra	9.52 (3.13)	<u>11.96 (1.98)</u>	12.55 (3.37)	14.61 (3.04)	15.77 (3.33)	18.81 (3.00)	28.64 (3.30)	34.62 (4.26)	35.06 (3.80)	35.89 (4.59)
RelyX ARC	31.30 (2.99)	31.51 (3.43)	33.90 (2.95)	35.14 (2.23)	37.81 (3.08)	38.75 (2.00)	38.40 (3.17)	42.00 (2.86)	42.25 (5.14)	43.00 (4.00)

N=15 (three readings per specimen), mean (standard deviation)

<sup>\*:</sup> Within a row, each double-underlined value is significantly different from the value at seven days according to the Mann-Whitney test at α=0.05.

<sup>#:</sup> Within a row, each singe-underlined value is significantly different from the value at seven days according to the Wilcoxon signed rank test at \alpha=0.05

<sup>\*:</sup> no measurable data.

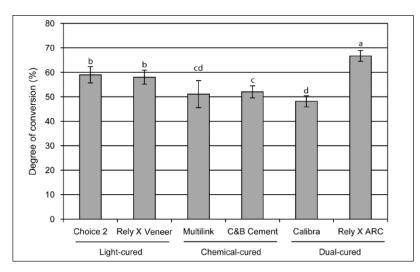


Figure 3: Degree of conversion (DC) of resin cements after seven days post-light activation or post-mix. The vertical bar indicates standard deviation. Bars with the same lower case letter are not statistically different according to the Mann-Whitney test at  $\alpha$ =0.05.

cantly higher DC values than the chemical-cured materials (p<0.05). The DC values of the two dual-cured materials were significantly different from each other (p<0.001).

# DISCUSSION

The monomer conversion of a composite is measured in a number of ways, typically FTIR spectroscopy. The microhardness test can also provide useful information on the conversion as an indirect method. For a single material, a rate of monomer conversion correlates with its microhardness. However, since the microhardness value is greatly influenced by factors, such as filler loading of the composite, cross-comparison between the different brands is limited. There may be a difference in the sensitivity to detect small changes in monomer conversion of a composite between the two techniques. Therefore, the current study employed both the FTIR spectroscopy and microhardness test to evaluate changes in monomer conversion within the same resin cement as a function of time.

The current FTIR study showed that most of the curing reaction for the light-cured material Choice 2 occurred during the early period after light exposure (within the first 10 minutes) (Table 2). This finding is comparable to the results of previous studies that showed most of the post-irradiation reaction occurs within 10 to 15 minutes. From RelyX Veneer, a slightly longer time was required to finish the majority of the curing reaction when compared to Choice 2. This difference could partly be explained by the faster polymerization reaction for Choice 2 when compared to RelyX Veneer during the early period (Figure 1). The extent of monomer reaction of a composite can be limited due to steric hindrance during polymerization. Steric hindrance during polymerization.

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. 13,15 This may apply to light-activated, dual-cured materials and lightcured materials.13 The curing reaction of RelyX ARC, although a dual-cured material, was the fastest among the materials tested. This material produced higher DC values than the two light-cured materials during the initial one and two minutes. The rapid increase in DC for RelyX ARC could have impeded the diffusion of radicals for further chemically-induced postirradiation polymerization.<sup>13</sup> Nonetheless, among the resin cements tested, RelyX ARC yielded the highest DC value at seven days post-mix. In contrast, Calibra, also a dualcured material, produced the lowest DC value at seven days, while the curing reaction seemed to continue up to a minimum of 24 hours. Thus, in the current study, the final DC

values at seven days for the light- or dual-cured materials polymerized by light activation do not always seem to be consistent with the duration of post-irradiation polymerization. Instead, the last DC values of a composite appear to be more related to the composite's initial conversion caused by light exposure. In this FTIR evaluation of light- and dual-cured materials, the DC value after one minute decreased in the following order: RelyX ARC > Choice 2 > RelyX Veneer > Calibra. This order did not change, even at seven days. Therefore, the level of initial conversion obtained from light exposure seems to be a highly influential factor in the final curing of light- or dual-cured materials. <sup>13</sup>

The chemical-cured materials exhibited slower setting speed and lower final DC values than light- or dual-cured materials, except for Calibra. In the current study, light activation was performed through a 1.5 mm-thick composite overlay, because the intensity of the light reaching the cement might not be sufficient in all clinical situations.<sup>17</sup> Despite such attenuation in light irradiance, in the current study, light-cured materials produced higher DC values than chemical-cured materials by seven days. It has been thought that the DC of chemical-cured composites might be lower than light-cured composites, partly because the presence of oxygen in voids incorporated during mixing could inhibit polymerization.<sup>9-10</sup> However, a lower DC is not necessarily an inherent characteristic of chemicalcured resins, as shown in a study by Feng and Suh,9 using a single dual-cured composite in which chemicaland light-cured specimens produced a similar DC to each other. The lower DC values frequently found in commercial chemical-cured resin cements might be partly attributable to the suboptimal concentration of

inhibitors added to the materials.13 Inhibitors are added to dental resins to allow for increased clinical working time to manipulate the materials and/or to promote shelf life.25 Excessive amounts of inhibitor, however, can reduce the cure rate. 13 Although light-cured materials also contain inhibitors, the concentration is relatively low. In some dual-cured products, the light curing reaction responding to light activation would compensate for lower conversion.8 A good balance is essential between shelf life and cure speed and between concentration of the initiator and the inhibitor.25 Thus, adequate, but not overextended working time is critical, not only for clinical use of a resin, but also for optimal polymerization. The current FTIR observations showed slight, yet gradual increases in DC values of the two chemical-cured materials during the induction period. Therefore, it is recommended that even chemical-cured resin cements should be applied soon after mixing and the restoration is seated in place. In addition, it should be noted that the chemical-cure reaction is accelerated when ambient temperature is high.<sup>10</sup>

The current microhardness test results (Table 3) showed a slightly different tendency as compared to FTIR assessment. Choice 2, RelyX Veneer and RelyX ARC attained DC values that had no further significant increases within 5 and 30 minutes; whereas, the microhardness values still increased even up to 24 hours (Figure 2). These findings imply that the microhardness test might be more sensitive than the FTIR technique at detecting post-irradiation or post-mix polymerization.<sup>22</sup> However, during the early observation period for the two chemical-cured materials (from 1 to 10 minutes), the current microhardness test could not be performed, because of a lack of detectable polymerization. Therefore, although the microhardness test might be more powerful than the FTIR technique for evaluating the rate of monomer conversion, the former should be applied to detecting the surface hardness of polymer-based materials hardened to a certain degree after the network has been crosslinked.7,22 Longer than 24 hours post-irradiation or post-mix, however, showed no further changes in both DC and microhardness values.

For light-cured materials, stiffening takes place within a short time after light exposure, especially by a high-intensity curing light.<sup>8</sup> Although the materials appear hard and fully cured immediately after exposure to a curing light, it should be noted that the curing reaction continues for a period of 24 hours. This is especially indicated in the microhardness test results of RelyX Veneer. Therefore, careful and sometimes delayed finishing and polishing is recommended for a restoration luted by a light-cured resin cement after light exposure.<sup>7-8</sup>

The current FTIR and microhardness test results showed that the polymerization reaction of dual-cured materials could be material-specific. Calibra produced a similar change in DC and VH to chemical-cured materials; whereas, RelyX ARC showed a faster polymerization reaction similar to typical light-cured materials. By seven days, the dual-cured material Calibra attained a significantly lower DC than the chemical-cured material C&B Cement (Figure 3). Thus, careful selection and manipulation is needed when using dual-cured resin cements, as the relative contributions of light-cure and chemical-cure mechanisms seem to differ, depending on the brands. 11-12

Although this in vitro study attempted to mimic a clinical situation with the use of a composite overlay during light activation, certain limitations to the experimental design were present. In the current study, it was necessary to irradiate the dual-cured materials immediately after mixing the paste in order to obtain similar time intervals as the light-cured materials. The light activation technique may be slightly different from that used in a clinical situation. Different timing of light exposure to dual-cured materials could result in different DC or microhardness results. In the current study, moreover, only one type of onlay (Esthet-X composite shade A2 [Dentsply Caulk]) was applied over the resin cements tested. When a light- or dual-cured resin cement is irradiated through an indirect restoration, the amount of light attenuation and, as a result, DC of the resin cement, would vary according to the material (for example, composite or ceramic), density, shade or thickness of the restoration.<sup>26</sup> More extensive research on the polymerization of dual-cured resin cements is necessary regarding light exposure conditions (delay, removal, duration or intensity) and their sensitivity to light and chemical activation. 11-13 In addition, the specimen storage condition employed in the current study (dark, dry conditions at 37°C) was not the same as simulating an intraoral condition (water storage), although influence on the DC and microhardness data did not seem to be significant during the test period for this study.27-28 Finally, only six commercially available resin cement products were tested in this study, and more need to be examined.

Care must be taken not to attribute clinical success to DC or microhardness values alone, as these values are not the only factor determining clinical performance. Although microhardness is one of the mechanical properties, it does not reflect the behavior of the bulk material. The clinical success of resin cements will depend on many other properties, such as handling properties, bonding strength with tooth and restoration, color stability and wear resistance. Nevertheless, obtaining a higher rate of monomer conversion for a composite is the basic step to maximizing the chemical and physical properties within the mate-

rial. Adequate polymerization of resin cements is also desirable to improve a material's biocompatibility by reducing the amount of residual monomers leached into the oral environment.<sup>30</sup>

In the current study, although the curing reaction of irradiated light- or dual-cured resin cements might have been faster than that of chemical-cured cements, the significant polymerization reaction was finished within 24 hours for all resin cements. Further research is needed to clarify the polymerization reaction of resin cements as a function of time, using various methods for testing mechanical and physical properties and rate of monomer conversion.

# **CONCLUSIONS**

The microhardness test was more sensitive than FTIR spectroscopy in detecting small changes in monomer conversion after the resin material hardened to a certain degree. Regardless of the curing modes (light, chemical or dual), a significant curing reaction appeared to be finished within 24 hours in terms of DC and microhardness values. Therefore, prior to that time, careful handling must be exercised, including finishing and polishing procedures of restoratives luted with resin cement. For primary evaluation of mechanical and chemical properties or bonding behavior of dental resin cements, a 24-hour storage condition may be sufficient, irrespective of the curing modes. However, long-term simulation of a resin cement or resin-bonded restoration in the oral environment would require a longer period of observation.

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