

## Effect of Light Irradiation Time on the Mechanical Properties of Two Flowable Composites with Different Initiation Systems in Bonded and Unbonded Cavities

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The aim of this study was to evaluate the regional mechanical properties of flowable composites with different initiation systems under free and constrained conditions. Forty cavities fabricated in resin blocks with or without bonding treatment were bulk-filled with Estelite Flow Quick (EFQ) or Palfique Estelite LV (ELV), followed by light irradiation for 10 or 30 seconds. Each specimen was sliced to three slabs parallel to the long axis. The middle slab was serially sliced from top to bottom to harvest three sticks for ultimate tensile strength (UTS) measurement. The remaining slabs were polished for microhardness (KHN) measurement. The results indicated that the UTS and KHN of both flowable composites decreased toward the bottom of the cavity and increased with prolonged light irradiation time. At the upper cavity region, UTS values of the bonded groups were significantly lower than those of the unbonded groups, except for the 10-second light irradiation group of EFQ. As for KHN, the values did not change significantly for both flowable composites between the unbonded and bonded groups.

Keywords: Flowable composite, Mechanical properties, Bonding

### INTRODUCTION

Current resin composites shrink during polymerization as monomer molecules are converted into a polymer network, resulting in reduced intermolecular spaces. Factors that interfere with the amount of volumetric contraction and the elastic modulus of the resin composite will influence any internal stresses<sup>1,2</sup>. When shrinkage of a resin composite occurs within a bonded cavity, a contraction stress develops at the bonded interface<sup>3</sup>. In the clinical situation, these stresses have been reported to reduce bond strength<sup>4,5</sup> and quality of marginal adaptation<sup>6,7</sup>, as well as cause enamel crack formation<sup>8</sup>. However, there have been few studies that have evaluated the effect of contraction stresses on the mechanical properties of resin composites under a constrained condition.

The polymerization velocity of resin composite affects the magnitude of internal stress. Kinomoto *et al.*<sup>9</sup> reported that auto-polymerizing composites were associated with fewer internal stresses within the resin composite than light-polymerized composites, because auto-polymerized composites require a longer polymerization time (and hence less polymerization velocity). With light-polymerized composites, contraction stress is influenced by curing characteristics that are highly dependent on the type and quantity of the initiation system<sup>10-12</sup>.

Light-polymerized composites usually employ a free radical initiation system, which commonly uses

camphorquinone (CQ) and an amine reducing agent<sup>13</sup>. CQ is inherently yellow in color, which causes problems in color matching with tooth substrates<sup>14</sup>. This is because after light polymerization, resin composites change color due to discoloration of CQ—which occurs as a result of light activation. Recently, a new initiation system has been developed that adopts radical-amplified light polymerization initiator technology (RAP technology)<sup>15</sup>. This initiation system has been reported to offer higher polymerization activity through a higher amount of radical production than a conventional CQ/amine initiation system. By reducing CQ concentration, the color change problem is thus overcome<sup>15</sup>.

Light irradiation method is an important factor that influences the polymerization activity of light-polymerized composites. Light irradiation time affects the degree of conversion and curing depth of light-polymerized composites<sup>16,17</sup>. To reflect the degree of conversion at different depths of a resin composite, the microhardness test is a simple and reliable method<sup>18,19</sup>. However, microhardness values cannot be used to compare the degree of conversion between different resin materials<sup>20</sup>. This is because filler type, size, and/or loading may affect the hardness of resin composites<sup>21</sup>. In contrast, the flexural strength test is useful for evaluating and comparing the mechanical properties of resin composites. However, comparison of the strengths of resin composites at different curing depths is difficult

because of the limitations of specimen preparation. With a view to overcoming these limitations, the microtensile test was introduced recently. It permits the measurement of regional ultimate tensile strength of resin composites at different depths<sup>29</sup>.

The aim of this study was to evaluate the effects of light irradiation time on the regional ultimate tensile strength and Knoop hardness of two flowable composites with different initiation systems under free and constrained conditions. The null hypothesis tested was that there are no differences in the ultimate tensile strength and Knoop hardness of both flowable composites at each cavity depth with regard to these factors: light irradiation time and constraint condition during polymerization (free or constrained).

## MATERIALS AND METHODS

### *Specimen preparation*

A cavity, 3 mm deep and 5 mm wide, was prepared in an acrylonitrile butadiene styrene (ABS) black resin block of  $1 \times 1 \times 1 \text{ cm}^3$ . An impression of the prepared cavity and block was made using putty and regular-type hydrophilic vinyl polysiloxane impression material (Exahiflex, GC Corp., Tokyo, Japan). Replicas of the cavity model were fabricated by filling the impression mold with Clearfil DC Core resin composite (Kuraray Medical Inc, Tokyo, Japan), followed by light polymerization for 60 seconds (Optilux 500, Demetron, Danbury, CT, USA) at a light intensity of  $600 \text{ mW/cm}^2$ . Curing was done in a laboratory light curing unit (Light II, J. Morita, Saitama, Japan) for 10 minutes to ensure complete polymerization of the resin composite. The models were then stored for one week in water before use. Table 1 lists the materials used in this study.

### *Bonding procedure*

Prior to the bonding procedure, the internal surfaces of the cavities were superficially abraded using a cylindrical abrasive point (HP 35, Shofu Inc., Kyoto, Japan) mounted in a low-speed handpiece with water spray. The external surfaces of the composite model were covered with black vinyl tape to prevent light passing through the composite walls to the bonding agent during light polymerization. Forty models were divided into four groups ( $n = 10$ ) for two filling composite materials and two irradiation times per material. For half of the models in each group, their cavity surfaces were treated with a mixture of Clearfil Photo Bond and Clearfil Porcelain Bond Activator (Kuraray Medical Inc, Tokyo, Japan), followed by light polymerization for 20 seconds (bonded cavities). The cavity surfaces of the remaining half received no surface treatment (unbonded cavities). The cavities were then bulk-filled with one of the following flowable composite materials: Estelite Flow Quick (EFQ) initiated with RAP technology or Palfique Estelite LV (ELV) with conventional initiation system (Tokuyama Dental Corp., Tokyo, Japan). Both materials were of the same shade (A3). Then, the upper surface of each specimen was covered with a plastic strip and pressed with a glass slide to squeeze out any excess resin. Light irradiation was performed for either 10 or 30 seconds for each type of composite by placing the light tip directly on the strip at the top of the cavity according to manufacturers' recommendation. The specimens were then stored in 37 °C water for 24 hours.

### *Ultimate microtensile strength test*

After 24 hours' storage, each specimen was attached to the arm of a low-speed diamond saw (Isomet,

Table 1 Resin composite materials used in this study

Material	Composition	Manufacturer
Palfique Estelite LV Medium	Silica-zirconia, silica-titania filler 68 wt%, 49 vol%, (0.08 and 0.4 $\mu\text{m}$ ), Bis-GMA, TEGDMA, Bis-MPEPP, camphorquinone	Tokuyama Dental, Tokyo, Japan
Estelite Flow Quick	Silica zirconia, silica-tetania filler 71 wt%, 53 vol%, (0.04 - 0.6 $\mu\text{m}$ ), Bis-MPEPP, TEGDMA, UDMA, camphorquinone (low concentration).	Tokuyama Dental, Tokyo, Japan
Clearfil PhotoBond	Catalyst: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, benzoyl peroxide. Universal: N,N-diethanol p-toluidine, sodium benzene sulfinate, ethyl alcohol	Kuraray Medical Inc., Tokyo, Japan
Clearfil Porcelain Bond Activator	Hydrophobic dimethacrylate, -methacryloxy propyltrimethoxy silane ( -MPS)	Kuraray Medical Inc., Tokyo, Japan
Clearfil DC Core	Catalyst: Bis-GMA, TEGDMA, silanated colloidal silica, barium glass, N,N-diethanol p-toluidine	Kuraray Medical Inc., Tokyo, Japan

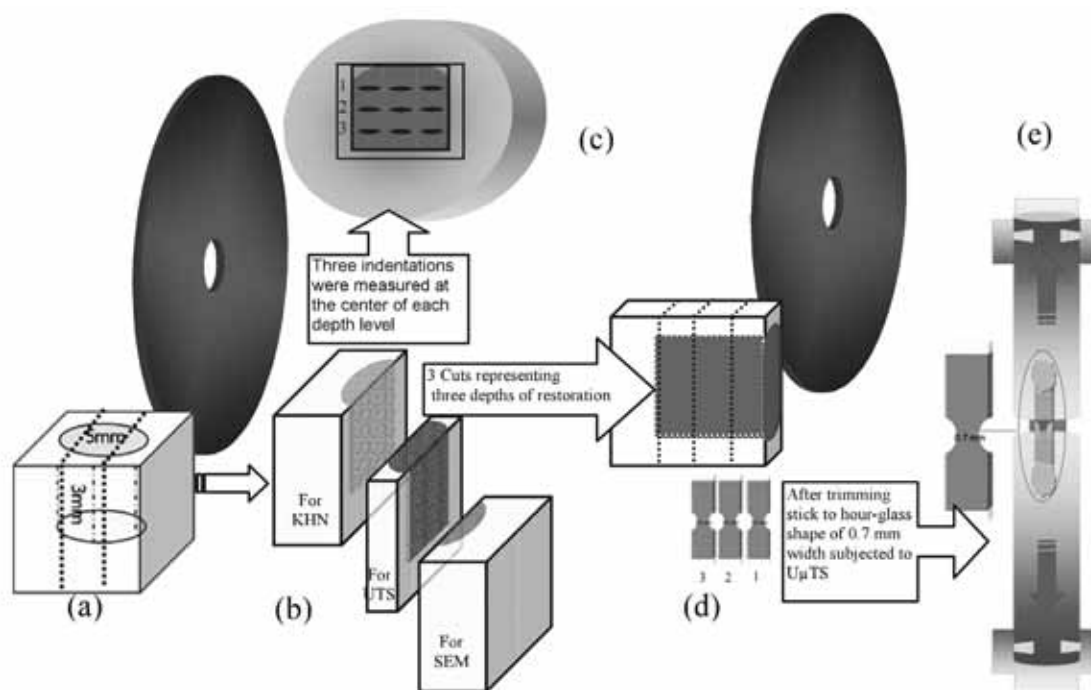


Fig. 1 Schematic diagram illustrating the experimental procedures and tests. (a) Final specimen after restoration; (b) After sectioning twice parallel to the long axis, three slabs were harvested (left, middle, and right); (c) Microhardness measurement; (d) Mid-slab was subjected to three serial cuts perpendicular to the long axis to harvest three sticks of 0.7-mm thickness; (e) Ultimate microtensile test using a universal testing machine at 1 mm/min.

Buehler, Lake Bluff, IL, USA) and sectioned twice in the middle region, parallel to the axial cavity wall, to obtain 2-mm-wide slabs (Fig. 1b). The slabs were then fixed at their bases and serially sliced, starting from the top surface of filled resin composite to harvest three sticks representing three depth levels of the restoration. The sticks were then trimmed to an hourglass shape under water cooling with a width of 0.7 mm at the narrowest region. The cross-sectional area of each beam was measured using digital calipers (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan). The ends of the hourglass-shaped specimens were glued to a testing device in a tabletop testing machine (EZ Test, Shimadzu Co., Kyoto, Japan) using cyanoacrylate glue (Zapit, Dental Venture of America, Anaheim, CA, USA) and subjected to a tensile force at a crosshead speed of 1 mm/min (Fig. 1e). Ultimate tensile strength (UTS) was calculated by dividing the fracture load by the surface area.

#### Microhardness measurement

For microhardness measurement, one of the composite slabs that remained from specimen sectioning for the UTS test was used (Fig. 1b). It was placed into an acrylic ring that was attached with an adhesive tape. The slab was placed face down on the adhesive tape and embedded in epoxy resin (Buehler Epoxycure Resin, Lake Bluff, IL, USA). After the epoxy resin

had set, the surface was polished using ascending grades of abrasive SiC papers under running water, followed by 6-, 3-, 1-, and 0.25- $\mu\text{m}$  diamond pastes (DP-Paste, Struers A\S, Denmark). Knoop hardness was measured using a microindentation tester (MVK-E hardness tester, Akashi Seisakusho Ltd., Kanagawa, Japan) with a load 0.49035 N for a dwell time of 15 seconds at three depth levels corresponding to UTS specimens. Three indentations were made at the center of each depth level and the average value was calculated (Fig. 1c). Each value of Knoop Hardness Number (KHN) was calculated using the following formula,  $\text{KHN} = 14.509F/I^2$  ( $F$  = load in N,  $I$  = length of indentation in mm).

#### Statistical analysis

The UTS and KHN data were analyzed by four-way and three-way ANOVA to test the factors of composite filling material, irradiation time, cavity region, and polymerization condition using a Statistical Package for Social Science (SPSS for Windows, Version 11.0, SPSS Inc., Chicago, IL, USA). Tukey's HSD and independent Student's *t*-test were used as *post hoc* tests for multiple comparisons. All statistical tests were performed at a 95% level of confidence.

#### SEM observation

The other remaining slabs of all specimens were used for SEM observation (Fig. 1b). The cut surface was

polished, gold sputter-coated, and placed in a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan). Bonding integrity, crack formation, and surface texture of the restorations were observed.

RESULTS

*Mechanical properties*

Tables 2 to 4 show the means and standard deviations of UTS and KHN of the flowable resin composites. Four-way ANOVA revealed that in the UTS data, there were significant differences for light irradiation time, polymerization condition, and depth ( $p < 0.0001$ ), but there were no significant differences between the materials (EFQ *versus* ELV) ( $p = 0.277$ ). In KHN data, there were significant differences for the materials, light irradiation time, and depth

( $p < 0.0001$ ), but there were no significant differences between the polymerization conditions (unbonded *versus* bonded) ( $p = 0.105$ ). With EFQ, there was a significant interaction between light irradiation time and polymerization condition in UTS ( $F = 5.8$ ;  $p < 0.0001$ ). On the other hand, no interactions were observed between these factors for ELV. As for KHN, significant interactions were observed between cavity depth and material and light irradiation time in both unbonded ( $F = 7.37$ ;  $F = 4.71$ ) and bonded groups ( $F = 7.98$ ;  $F = 5.55$ ), respectively.

Independent Student's t-test revealed that the results in the unbonded groups demonstrated that a longer light irradiation time significantly increased the UTS of EFQ at all regions of the cavity, while a significant increase for KHN value was observed only at the bottom region ( $p < 0.05$ ) (Table 2). With ELV, both UTS and KHN also increased when the duration of light irradiation was lengthened. However, a significant difference in UTS was observed only at

Table 2 UTS and KHN of unbonded groups

Depth	UTS			
	EFQ		ELV	
	10s	30s	10s	30s
1	97.9(27.7) <sup>1</sup>	128.3(12.8) <sup>A</sup>	99.7(17.6) <sup>1</sup>	112.2(22.3) <sup>A</sup>
2	77.7(15.8) <sup>1</sup>	127.0(19.4) <sup>A</sup>	94.9(5.7) <sup>1</sup>	120.4(20.5) <sup>A</sup>
3	42.6(9.2) <sup>1</sup>	102.1(16.4) <sup>A</sup>	75.8(14.4) <sup>1</sup>	96.4(25.0) <sup>A</sup>
Depth	KHN			
	EFQ		ELV	
	10s	30s	10s	30s
1	61.2(5.2) <sup>1</sup>	61.7(8.4) <sup>A</sup>	42.6(2.9) <sup>2</sup>	48.3(7.4) <sup>A</sup>
2	51.7(5.0) <sup>1</sup>	55.9(7.6) <sup>A</sup>	39.3(3.6) <sup>1</sup>	43.4(5.6) <sup>A</sup>
3	21.4(15.9) <sup>1</sup>	47.8(5.0) <sup>A</sup>	32.7(3.6) <sup>1</sup>	37.8(7.2) <sup>A</sup>

Mean(SD); n = 5

Horizontal line = Significant difference between light irradiation times ( $p < 0.05$ ); Different superscript numbers or letters in each row indicates significant differences between materials ( $p < 0.05$ ).

Table 3 UTS and KHN of bonded groups

Depth	UTS			
	EFQ		ELV	
	10s	30s	10s	30s
1	83.9(12.0) <sup>1</sup>	100.8(20.9) <sup>A</sup>	69.0(18.8) <sup>1</sup>	82.4(12.0) <sup>A</sup>
2	82.6(11.8) <sup>1</sup>	106.7(12.9) <sup>A</sup>	79.5(11.9) <sup>1</sup>	81.4(22.0) <sup>A</sup>
3	55.1(9.7) <sup>1</sup>	96.9(16.5) <sup>A</sup>	63.9(6.8) <sup>1</sup>	84.5(21.5) <sup>A</sup>
Depth	KHN			
	EFQ		ELV	
	10s	30s	10s	30s
1	64.5(4.8) <sup>1</sup>	65.4(6.6) <sup>A</sup>	44.5(6.5) <sup>2</sup>	49.5(8.4) <sup>B</sup>
2	52.4(4.9) <sup>1</sup>	57.3(4.5) <sup>A</sup>	37.7(4.0) <sup>1</sup>	43.1(4.4) <sup>A</sup>
3	30.2(3.7) <sup>1</sup>	50.4(8.5) <sup>A</sup>	32.1(2.4) <sup>1</sup>	40.6(7.0) <sup>A</sup>

Mean(SD); n = 5

Horizontal line = Significant difference between light irradiation times ( $p < 0.05$ ); Different superscript numbers or letters in each row indicates significant differences between materials ( $p < 0.05$ ).

Table 4 Comparison of UTS and KHN between bonded and unbonded groups

Depth	UTS			
	Unbonded		Bonded	
	10s	30s	10s	30s
1	97.9(27.7) <sup>a</sup>	83.9(12.0) <sup>a</sup>	128.3(12.8) <sup>a</sup>	100.8(20.9) <sup>a</sup>
2	77.7(15.8) <sup>a</sup>	82.6(11.8) <sup>a</sup>	127.0(19.4) <sup>a,b</sup>	106.7(12.9) <sup>a</sup>
3	42.6(9.2) <sup>b</sup>	55.1(9.7) <sup>b</sup>	102.1(16.4) <sup>b</sup>	96.9(16.5) <sup>a</sup>
Depth	KHN			
	Unbonded		Bonded	
	10s	30s	10s	30s
1	99.7(17.6) <sup>a</sup>	69.0(18.8) <sup>a</sup>	112.2(22.3) <sup>a</sup>	82.4(12.0) <sup>a</sup>
2	94.9(5.7) <sup>a,b</sup>	79.5(11.9) <sup>a</sup>	120.4(20.5) <sup>a</sup>	81.4(22.0) <sup>a</sup>
3	75.83(14.4) <sup>b</sup>	63.9(6.8) <sup>a</sup>	96.4(25.0) <sup>a</sup>	84.5(21.5) <sup>a</sup>

Mean(SD); n = 5

Horizontal line = Significant difference ( $p < 0.05$ ); Different superscript letters in each column indicates significant difference ( $p < 0.05$ ).

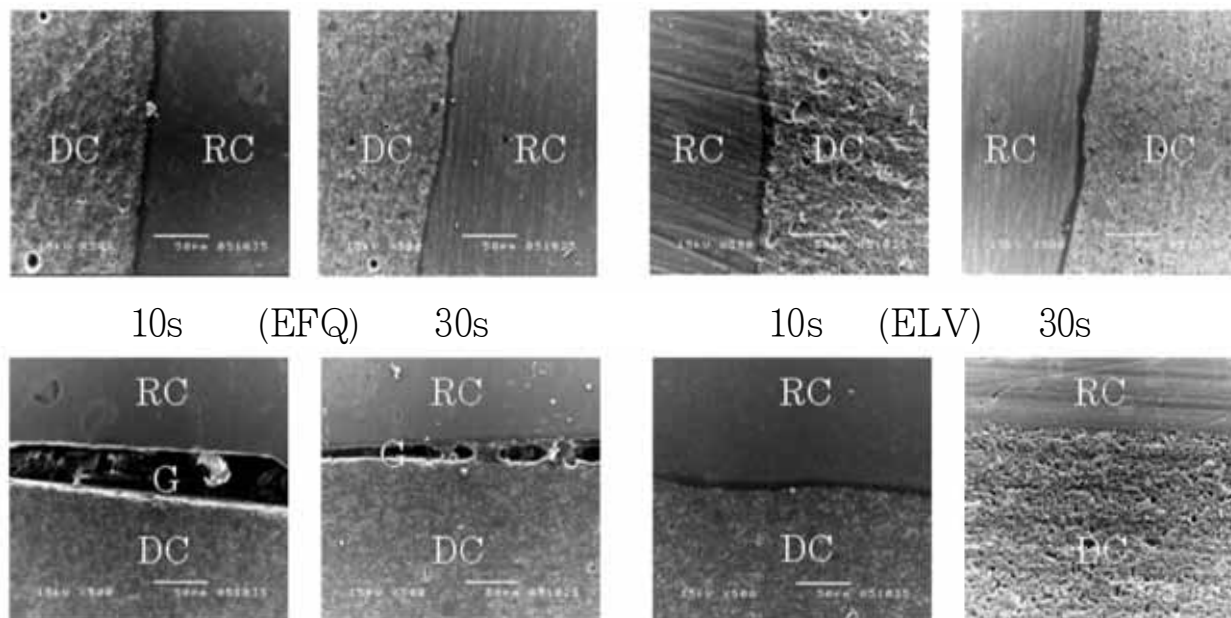


Fig. 2 Scanning electron micrographs of cut surfaces show the bond integrity at wall interface (upper row) and base interface (lower row). (DC=Dual-cure core composite, RC=Resin composite, G=Gap).

the middle region ( $p < 0.05$ ) (Table 2).

When comparing the polymerization conditions, the UTS of ELV in the bonded groups were significantly lower than those of the unbonded groups at the upper and middle regions of the cavity with both light irradiation times ( $p < 0.05$ ) (Table 4). On the other hand, with EFQ, the UTS of the bonded group was significantly reduced only at the upper region when light-irradiated for 30 seconds ( $p < 0.05$ ). For KHN, there were no significant differences at each region between the bonded and unbonded groups of both flowable composites with 10- and 30-second light irradiation times ( $p > 0.05$ ) (Table 4). Nonetheless, the UTS and KHN of both flowable composites decreased toward the bottom region, although Tukey's HSD multiple comparison test indicated that there were no significant differences in some groups (Table 4).

#### Gap formation

SEM observation revealed a clear gap around the restorations of the un-bonded specimens of both flowable composites due to polymerization shrinkage. With the bonded specimens, ELV showed an intact bond to the cavity walls and floor. On the other hand, all EFQ specimens had gaps on the cavity floor although cavity walls were intact (Fig. 2).

## DISCUSSION

Light-polymerized resin composites require proper light activation in order to achieve a relatively high degree of conversion and good mechanical properties.

The degree of conversion is dependent upon the thickness of the resin composite. This is because as the depth of the cavity increases, light intensity attenuation occurs inside the restorative material, thereby preventing complete polymerization in deeper regions<sup>20</sup>. In this study, four-way ANOVA revealed a significant effect by the cavity region factor on the UTS and KHN of both flowable resin composites. It was found that the UTS and KHN of both materials in the upper region were higher than those at the bottom region of the cavity. In the same vein, many studies have shown that the KHN of light-polymerized resin composites is affected by cavity depth. This is because KHN values reflect the degree of polymerization at different depths of a resin composite<sup>19,20,23</sup>. Similarly, regional UTS of resin composites would also be influenced by regional differences in polymerization.

Extending the light irradiation time significantly increased the regional UTS of both flowable composites. However, it did not have a similar impact on KHN at the upper and middle regions. A previous study has reported that there were few correlations between UTS and KHN of dual-cure composites, regardless of cavity depth<sup>22</sup>. KHN represents the resistance to local deformation due to loading, which is heavily affected by filler load, size, and shape of the resin composite<sup>24,26</sup>. UTS, on the other hand, is the maximum resistance to tensile force, which is mainly affected by the structure of the resin matrix and filler-matrix adhesion<sup>27-30</sup>. In other words, UTS would be more strongly influenced by the polymerization rate of the resin matrix as compared

to KHN.

The UTS and KHN of EFQ in the 10-second light irradiation group dramatically reduced toward the bottom region from the upper region, as compared with ELV. On the other hand, the reduction rate in the 30-second group of EFQ was similar to that of ELV. These results could be attributed to the different initiation systems employed by both flowable composites. EFQ utilized radical-amplified light polymerization technology (RAP technology), which offers higher polymerization activity due to a higher amount of free radical production than the conventional CQ/amine initiator system<sup>15</sup>. Therefore, the concentration of CQ in EFQ was lower than that of ELV (information obtained by personal communication).

Photoinitiation chemistry for polymerization of resin composites affects the generation of free radicals<sup>14</sup>. This is because free radical production is directly proportional to the amount of absorbed light irradiance and the quantum yield for initiation<sup>13</sup>. At the bottom region, the light intensity would be insufficient for light initiation. In our pilot study, the intensity of transmitted light through 2-mm-thick disks of EFQ and ELV was 22 - 23 mW/cm<sup>2</sup> for both 10- and 30-second light irradiation times. Therefore, polymerization at the bottom region would be mainly caused by a chain reaction from free radical production in the upper region. For EFQ, reduced mechanical properties at the bottom region in the 10-second light irradiation group might be due to fewer chain reactions which was a result of shorter light irradiation time as compared to 30-second irradiation.

Significantly reduced UTS and KHN of EFQ might also be related to the concentration of CQ. This is because CQ directly absorbs light energy in a light-transmitted area and produces free radicals. Nonetheless, further research is needed to determine the contributing factors for radical chain reactions in insufficiently light-transmitted regions. On the other hand, when light-irradiated for 30 seconds, EFQ with RAP technology could polymerize at depths of 1 - 3 mm, although EFQ had a lower CQ concentration than ELV.

The polymerization shrinkage of resin composites is due to monomer molecules being converted into a polymer network. Internal stresses within resin composite would increase when resin composite is polymerized in a restricted condition. Choi *et al.*<sup>31</sup> measured the mechanical properties of universal composites constrained within a cavity made of a rigid material. It was demonstrated that both flexural strength and elastic modulus were significantly reduced as the composites were polymerized under greater constraint. In this study, ANOVA revealed that there were significant differences in UTS for the

different polymerization conditions (bonded *vs.* unbonded condition), although there were no significant differences in KHN. It should be highlighted that UTS is affected by the structure of the resin matrix and filler-matrix adhesion. Therefore, these results might indicate that not all the polymer network was rearranged, and that its formation was interrupted during polymerization partly due to the presence of internal stresses caused by restriction of polymerization shrinkage.

Another factor associated with contraction stress is the cavity configuration (C-factor), which is the ratio of bonded to unbonded surfaces of a restoration<sup>32</sup>. The higher the C-factor, the lower is a composite's ability to flow to compensate for the reduction in volume, thereby resulting in higher stresses<sup>1</sup>. In the present study, a simulated cylindrical cavity (3 mm deep, 5 mm wide) was prepared in a resin composite block of Clearfil DC Core. Its flexural modulus (approximately 15 GPa) was similar to dentin, and whereby good adhesion to dentin is possible by using a silane coupling agent. With ELV, bonding was very good at the cavity walls and floor, and there were significant differences in UTS between the bonded and unbonded groups at the upper and middle regions. On the other hand, with EFQ, significant reduction in UTS was observed only at the upper region for 30-second light irradiation time. All EFQ specimens showed gap formation at the cavity floor under SEM observation, which was also observed by an optical microscope during KHN measurement. This might have been due to bonding failures on the cavity floor. At this juncture, it could be suggested that if there were good bonding to the cavity floor, the regional UTS of EFQ might be further reduced.

Within the limitations of this study, it was found that extension of light irradiation time significantly increased the regional UTS of both flowable composites, EFQ and ELV. However, similar effect was not observed for KHN at the upper and middle regions. The UTS of both flowable composites were lower in the bonded groups than in the unbonded groups at the upper region. These results suggested that polymerization shrinkage affected the mechanical properties of resin composites. Polymerization shrinkage, on the other hand, was affected by constraint condition (free *versus* constrained) and cavity depth.

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