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Influence of light-exposure methods and depths of cavity on the microhardness of dual-cured core build-up resin composites

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ABSTRACT

Objective: The purpose of this study was to evaluate the Knoop hardness number (KHN) of dual-cured core build-up resin composites (DCBRCs) at 6 depths of cavity after 3 post-irradiation times by 4 light-exposure methods. Material and Methods: Five specimens each of DCBRCs (Clearfil DC Core Plus [DCP] and Unifil Core EM [UCE]) were filled in acrylic resin blocks with a semi-cylindrical cavity and light-cured using an LED light unit (power density: 1,000 mW/cm²) at the top surface by irradiation for 20 seconds (20 s), 40 seconds (40 s), bonding agent plus 20 seconds (B+20 s), or 40 seconds plus light irradiation of both sides of each acrylic resin block for 40 seconds each (120 s). KHN was measured at depths of 0.5, 2.0, 4.0, 6.0, 8.0, and 10.0 mm at 0.5 hours, 24 hours, and 7 days post-irradiation. Statistical analysis was performed using repeated measures ANOVA and Tukey’s compromise post-hoc test with a significance level of p<0.05. Results: For both DCBRCs, at 0.5 hours post-irradiation, the 20 s and 40 s methods showed the highest KHN at depth of 0.5 mm. The 40 s method showed significantly higher KHN than the 20 s method at all depths of cavity and post-irradiation times, except UCe at depth of 0.5 mm (p<0.05). The 120 s method did not result in significantly different KHN at all depths of cavity and post-irradiation times (p>0.05). In DCP, and not UCe, at 24 hours and 7 days post-irradiation, the B+20 s method showed significantly higher KHN at all depths of cavity, except the depth of 0.5 mm (p<0.05). Conclusion: KHN depends on the light-exposure method, use of bonding agent, depth of cavity, post-irradiation time, and material brand. Based on the microhardness behavior, DCBRCs are preferably prepared by the effective exposure method, when used for a greater depth of cavity.

Keywords: Composite resins. Exposure time. Hardness.

INTRODUCTION

Core build-up materials are often required to reconstruct and provide an ideal morphology to severely damaged teeth prior to their preparation for indirect foundation restorations. Despite substantial documented evidence of the long-term success of large amalgam restorations, resin composites, since the early days of self-cured materials, have also been used for this purpose. More recently, light-cured core build-up materials that are more convenient to use than chemically cured composites have been widely used. Both these materials, however, have their disadvantages. While chemically cured materials do not allow clinicians to adjust the setting time individually, light-cured resin composites do not ensure adequate polymerization in areas with limited access to the curing light.

However, resin-based composites are associated with polymerization shrinkage, causing stress development under confined conditions. Several strategies have been used to overcome the limitations of polymerization shrinkage. Various layering techniques have been suggested to minimize shrinkage stress. Nevertheless, time limitations when placing core build-up materials...
restrict clinicians from using elaborate multilayering techniques. Therefore, dual-cured resin composites, in which polymerization is chemically initiated in the deeper portions of the canal or preparations, have been developed for use as core build-up materials; this has allowed clinicians to build extended foundation restorations quickly, and in bulk.

At the coronal region, dual-cured core build-up resin composites are mainly polymerized through photo-initiated reactions, whereas, in the apical region, polymerization is chemically initiated. However, the incorporation of self- and light-curing modes in the same material does not ensure maximal curing of the material. Due to incomplete compensation for deficient light activation, lower hardness values of dual-cured core build-up resin composites have been observed with increased depth of cavity. It has been speculated that a delay in light activation would be beneficial in enhancing the degree of conversion of dual-cured resin cements, since immediate exposure to light could interfere with the chemical-curing mechanism. On the other hand, it has been reported that time delay and duration of light exposure does not increase microhardness at different depths of a dual-cured core build-up resin composite 2 weeks after light irradiation. Moreover, light-activation delayed by 5 minutes after seating the fiber-reinforced post did not affect the microhardness of dual-cured resin cements at 3 regions of the root after 3 months of storage in water. Although dual-cured core build-up resin composites have been recently used to prepare prefabricated posts and core or coronal-radicular build-ups, their hardness behavior at greater depths of cavity is unknown. Moreover, no information is available in the literature regarding the effect of applying a bonding agent on the cavity wall on polymerization of the dual-cured core build-up resin composite.

Based on these considerations, the purpose of the present study was to investigate the influence of light-exposure durations (20, 40, and 120 seconds) and application of a bonding agent on the extent of polymerization by measuring the microhardness of 2 dual-cured core build-up resin composites at different depths of cavity without prefabricated posts and various post-irradiation times. The following null hypotheses were tested: (1) an increase in light-exposure time results in no difference in hardness, regardless of the depth of cavity and type of dual-cured resin composite; (2) hardness is not affected by depth of cavity and post-irradiation time; and (3) application of the bonding agent does not improve hardness.

**MATERIAL AND METHODS**

**Specimen preparation**

Forty semi-cylindrical cavities with a diameter of 3 mm and a depth of 11 mm were prepared in 5x10x16 mm acrylic resin blocks (Figure 1). Two acrylic resin blocks, with or without a semi-cylindrical cavity, were placed in a silicon impression material mold (15x15x20 mm). Either 1 of 2 dual-cured core build-up resin composite pastes [Clearfil DC Core Plus (DCP) or Unifil Core EM (UCE) (Figure 2)] was filled directly in the cavities using auto-mixing tips, being sure to avoid entrapment of air, according to the manufacturer’s instructions. The upper surface in the resin composite material was covered with a plastic strip and pressed with a thin cover glass to remove any excess resin. Light-irradiation was provided by placing the tip of the LED light unit (power density: 1,000 mW/cm²; Pencure, J. Morita MFG Corp., Kyoto, Japan) on the plastic strip. Power output was verified with a curing radiometer (Cure Lite; Dentsply Caulk, Milford, CT, USA) immediately before every light-activation throughout the study. The core build-up resin composites were light-cured for different durations using 1 of the 4 following light-exposure methods: (1) light-irradiation for 20 seconds on the plastic strip after filling the core build-up resin composite (20 s method); (2) light-irradiation for 40 seconds (40 s); (3) application of the bonding agent on the cavity wall with a brush, followed by air-drying, and light-irradiation for 10 seconds on the top of the cavity before the 20 s method (B+20 s method); (4) light-irradiation for 40 seconds on the plastic strip, followed by removal of the acrylic blocks from the silicon impression material mold, and irradiation of both sides of each acrylic resin block for 40 seconds each (120 s method). After irradiation of all the specimens, the acrylic resin blocks were removed from the silicon mold and separated. Five specimens of each dual-cured core build-up resin composite were irradiated by each of the 4 methods.

**Hardness measurements**

Hardness was measured at the following depths from the light-irradiated surface of the cavity: 0.5, 2.0, 4.0, 6.0, 8.0, and 10.0 mm. For each specimen, the Knoop hardness number (KHN) was measured 5 times at each depth using a microhardness tester (FM-700: Future-Tech Corp., Kawasaki, Japan) at 0.5 hours, 24 hours, and 7 days post-irradiation. A Knoop diamond indenter was applied under a load of 25 g for a dwell time of 15 seconds; the load was then removed, and the long diagonal of the indentation was measured under 400x magnification. KHN, which is inversely proportional to the square of the long diagonal,
### Materials

| Core material | Manufacturer | Composition |
|---------------|--------------|-------------|
| Clearfil DC Core Plus (DCP) (Batch # 0002AA) | Kuraray Noritake Products Corp., Tokyo, Japan | A paste: Bis-GMA, hydrophilic aliphatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silanized barium glass filler, silanized colloidal silica, colloidal silica, chemical-initiator, photo-initiator, pigments. B paste: TEGDMA, hydrophilic aliphatic dimethacrylate, hydrophobic aromatic dimethacrylate, silanized barium glass filler, silanized colloidal silica, aluminum oxide filler, photo-accelerator, chemical-accelerator. |
| Clearfil S3 Bond Plus (Batch # 00024B) | Kuraray Noritake Products Corp., Tokyo, Japan | Bis-GMA, HEMA, MDP, hydrophilic dimethacrylate, hydrophobic dimethacrylate, colloidal silica, ethanol, water, photo-initiators, photo- and chemical-accelerator, sodium fluoride. |
| Unifil Core EM (UCE) (Batch # 1107011) | GC Corp., Tokyo, Japan | Base: UDMA, dimethacrylates, fluoro-alumino-silicate glass, silicon dioxide, photo-initiator, accelerator. Catalyst: UDMA, dimethacrylates, fluoro-alumino-silicate glass, silicon dioxide, chemical-initiator, pigment. |

| Bonding material | Manufacturer | Composition |
|------------------|--------------|-------------|
| Self-etching bond A (Batch # 1205181) | GC Corp., Tokyo, Japan | dimethacrylates, 4-META, silicon dioxide, ethanol, water, photo-initiator. |
| Self-etching bond B (Batch # 1206011) | GC Corp., Tokyo, Japan | ethanol, accelerator. |

Bis-GMA: bis-phenol-A-glycidyldimethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; MDP: 10-methacyrloyloxydecyl dihydrogen phosphate; 4-META: 4-methacyrloyloxyethyl trimellitate anhydride.

**Figure 1** - Schematic illustration of the preparation of specimens for measurement of Knoop hardness: a: acrylic resin blocks (5x10x16 mm) with or without semi-cylindrical cavity (diameter of 3 mm and depth of 11 mm); b: two acrylic resin blocks joined; c: two acrylic resin blocks placed in a silicon impression material mold (15x15x20 mm); d: dual-cured core build-up resin composite filled in the cavity and irradiated on the upper surface using LED light unit; e: two acrylic resin blocks separated; f: Knoop hardness measurement for the surface of resin composite filled in the semi-cylindrical cavity.

**Figure 2** - Dual-cured core build-up resin composites used in this study.
was thus calculated. All specimens were stored under dry and dark conditions in a box, which was placed in a biochemical incubator at 37°C to avoid exposure to light, and was accessed only to obtain measurements.

Statistical analysis

The KHN data were statistically analyzed by the repeated measures three-way ANOVA test. The independent variables analyzed were depths of cavity and post-irradiation times for within-subject analysis, and light-exposure methods and type of dual-cured core build-up resin composite for between-subject analysis. A one-way ANOVA with the post-hoc Tukey’s compromise test was used to establish specific differences in KHN values between the groups (α=0.05).

RESULTS

The results of this study are summarized in Tables 1 and 2, which show the mean KHN and standard deviation of all the experimental groups of dual-cured core build-up resin composite for each exposure method and post-irradiation times.

Table 1- Mean KHN±SD for Clearfil DC Core Plus (DCP) at 6 depths of cavity after 3 post-irradiation times by 4 light-exposure methods

The same lower-case superscript Greek characters indicate no statistically significant differences between exposure methods at same post-irradiation time in the same depth of cavity; the same upper-case superscript letters indicate no statistically significant differences between depths of cavity at same post-irradiation time in the same exposure method; the same upper-case superscript letters indicate no statistically significant differences between post-irradiation times at same depth of cavity in the same exposure method (p>0.05).

KHN=Knoop hardness number
**DISCUSSION**

The results of this study indicate that the KHNs of dual-cured core build-up resin composites depend on the light-exposure method, including irradiation duration, use of bonding agent, depth of cavity, post-irradiation time, and material brand. Therefore, the research hypotheses formulated for this study must be rejected.

KHN has been shown to be a good indicator of the degree of conversion/polymerization based on its good correlation with infrared spectroscopy,10,31. However, the prediction of an absolute value of degree of conversion by means of an absolute hardness value is not achievable, since other factors such as type and size of filler, filler load, monomer composition, quantity of initiators, and the ratio of chemical- and light-cured components strongly influence the final quantity of reacted monomers.2,5,14. Microhardness data from the same resin cement only should be compared according to the depth of the root canal or time elapsed since luting. KHNs could be used to reflect the degree of conversion at different depths of a resin composite. Therefore, KHNs were measured in the present study to reflect monomer conversion at different depths of a resin composite.32 Therefore, KHNs were measured in the present study to reflect monomer conversion at different depths of a resin composite.32

For DCP, with the B+20 s method, no significant differences in the KHN values were observed at 0.5 hours post-irradiation at all depths of cavity, except 0.5 mm (p>0.05). However, at 24 hours and 7 days post-irradiation, the B+20 s method resulted in significantly higher KHN values than the 20 s method at all depths of cavity, except 0.5 mm (p<0.05). On the other hand, for UCE, at 0.5 hours post-irradiation, the B+20 s method showed significantly lower KHN values than the 20 s method up to the depth of 6.0 mm; however, the KHN values were significantly higher at the depths of 8.0 mm and 10.0 mm (p<0.05). At 24 hours and 7 days post-irradiation, no significant differences in KHN values were observed between the B+20 s and 20 s method at all depths of cavity (p>0.05).

For all light-exposure methods, except the 120 s method, the UCE exhibited significantly higher KHN values at all depths of cavity, except at 4.0 mm in the 40 s method, at 7 days post-irradiation than at 0.5 hours and 24 hours post-irradiation (p<0.05). It was found that the KHN values of DCP were significantly higher at those of UCE at all depths of cavity and post-irradiation times, regardless of the exposure method (p<0.05).

**Table 2- Mean KHN±SD for KHN for Unifil Core EM (UCE) at 6 depths of cavity after 3 post-irradiation times by 4 light-exposure methods**

The same lower-case superscript Greek characters indicate no statistically significant differences between exposure methods at same post-irradiation time in the same depth of cavity; the same lower-case superscript letters indicate no statistically significant differences between depths of cavity at same post-irradiation time in the same exposure method; the same upper-case superscript letters indicate no statistically significant differences between post-irradiation times at same depth of cavity in the same exposure method (p>0.05).

| Exposure method | Post-irradiation time | Depth of cavity (mm) |
|-----------------|-----------------------|----------------------|
|                 | 0.5 hours | 2 | 4 | 6 | 8 | 10 |
| Exposure time of 20 s (20 s) | 0.5 hours | 36.26±1.31α | 34.18±0.97α | 32.99±0.64α | 31.69±0.64α | 29.10±0.49α | 27.31±0.39α |
|                 | 24 hours  | 41.22±0.81β | 38.42±1.14β | 38.35±1.07β | 38.42±1.19β | 37.42±1.33β | 36.87±1.50β |
|                 | 7 days  | 42.86±0.80α | 40.52±1.04α | 39.79±1.20α | 39.56±1.15α | 38.37±1.01α | 38.08±1.23α |
| Bonding + exposure time of 20 s (B+20 s) | 0.5 hours | 32.23±0.97α | 30.78±0.79α | 30.58±0.65α | 30.54±0.47α | 30.10±0.93α | 29.49±0.79α |
|                 | 24 hours  | 40.12±0.76α | 37.97±1.08α | 37.56±0.25α | 37.50±0.11α | 37.39±0.21α | 36.79±0.42α |
|                 | 7 days  | 40.06±0.40α | 40.21±0.28α | 39.08±0.24α | 39.19±0.26α | 38.82±0.26α | 38.09±0.43α |
| Exposure time of 40 s (40 s) | 0.5 hours | 37.19±0.24α | 36.50±0.37α | 35.28±1.15α | 34.14±0.61α | 32.22±1.77α | 30.36±0.13α |
|                 | 24 hours  | 42.31±0.38α | 40.93±0.53α | 40.90±0.61α | 40.62±0.49α | 39.80±1.10α | 39.51±0.87α |
|                 | 7 days  | 43.07±0.15α | 42.12±0.45α | 41.87±0.45α | 41.97±0.47α | 40.76±0.61α | 40.77±0.69α |
| Exposure time of 120 s (120 s) | 0.5 hours | 37.35±0.32α | 37.60±0.35α | 37.46±0.25α | 37.59±0.18α | 37.36±0.21α | 37.32±0.25α |
|                 | 24 hours  | 42.58±0.30α | 42.67±0.62α | 42.60±0.46α | 42.59±0.46α | 42.62±0.55α | 42.74±0.44α |
|                 | 7 days  | 42.78±0.40α | 42.98±0.77α | 42.97±0.86α | 42.81±0.45α | 43.00±0.85α | 43.06±0.60α |
be simply attributed to the direction of photo-
initiation. Light irradiation was focused on the top
surface of the cavity. Therefore, polymerization
of the resin composites, by means of photo-
activated free radicals, may occur immediately
at the shallow depths of cavity. This finding, that
is, KHN of light- and dual-cured resin composites
are affected by depth of cavity, has been reported
previously4,6-8. The chemical-curing mechanism of
dual-cured resin composites is usually based on a
redox reaction of benzoyl peroxide with aromatic
tertiary amines, which generates free radicals that
break the aliphatic carbon double bonds to initiate
the polymerization process. It is supposed that
immediate photo-activation after light irradiation,
despite causing a rapid increase in the viscosity of
the polymer matrix, does not hinder migration of
the activated free radicals responsible for further
chemically induced polymerization. Although the
photo-activated free radicals at shallow depths of
cavity could induce chain propagation of the resin
polymer in the downward direction, the exact polymerization mechanism of dual-cured
core build-up resin composite at greater depths
of cavity remains unknown. It is difficult to
distinguish clearly between the depths of cavity
at which polymerization of the resin composite
occurs through photo-initiation and those at which
polymerization occurs by means of chemical
initiation alone.

Evaluation of the duration of photo-activation
revealed that a longer exposure time of 40 seconds
resulted in significantly higher KHN values than
did a shorter exposure time of 20 seconds at all
depths of cavity and post-irradiation times for
both resin composites; however, this trend was
not observed in UCE at the depth of 0.5 mm.
Thus, our study showed that prolonged irradiation
durations resulted in increased hardness values.
Light-curing units with lower power density
needed longer light-exposure times to produce a
similar microhardness value of resin composite
as that of light sources with high power density11.
Therefore, with longer light-exposure durations,
which result in higher energy densities at a given
irradiance, more photo-sensitizer molecules are
activated, which in turn increase the free radical
concentration and consequently the conversion of
double bonds. However, at the depth of 0.5 mm,
no significant differences were observed in the KHN
values between the light-irradiation durations of
40 seconds and 120 seconds (40 seconds each on
the top and either side of the cavity) for both resin
composites. This finding might be attributed to the
fact that the polymer network developed during
light exposure for 40 seconds does not allow any
additional mobility of the polymer chains in order
to increase monomer conversion, indicating that
the light-irradiation duration of 40 seconds alone
is sufficient.

Dentin hardness ranges from KHN values of
50 to 70, depending on the distance from the
amelodentinal junction21. The mean microhardness
(KHN) value of 52.9 for DCP with 120 s method at
the depth of 10.0 mm at 7 days post-irradiation
therefore predicts similar mechanical properties to
that of dentin. The equal degree of polymerization
within the core material may support a uniform
distribution of stress along the tooth-material
interface under load. Longer light-exposure
 durations result in superior microhardness, but,
at the same time, contribute to an increase of
shrinkage and contraction stress of the dual-
cured core build-up resin composite28. In cases of
significant coronal destruction, it is necessary to
replace the lost tooth structure with a core build-
up material to attain full-coverage restoration. The
cast post and core, prefabricated post and core
materials, and coronal-radicular build-ups are the
available options for this purpose. The fracture
resistance and survival probability of post and core
restorations depend on several factors such as the
post material, luting agent, amount and condition of
residual tooth structure, core material, preparation
of the tooth for restorative procedures, and, finally,
the fixed restoration16,22,25,34. Therefore, when using
dual-cured core build-up resin composites, it is
preferable to prepare the composites by using the
effective exposure method.

Before core build-up resin composites are used,
a bonding agent must be applied on the cavity
wall. The effectiveness of the bonding agent on the
KHN as an indirect method of monomer conversion
differed between the 2 resin composites. For DCP,
at 24 hours and 7 days post-irradiation, application
of the bonding agent resulted in significantly higher
KHNs than the 20 s method at all depths of cavity;
however, this was not observed with UCE. The
initiator and/or accelerator present in the bonding
agent would promote monomer conversion of the
dual-cured core build-up resin composites. On the
other hand, the acidic monomer in the bonding
agent inhibits the amine co-initiator in the dual-
cured materials16, which in turn adversely affects
polymerization of dual-cured core build-up resin
composites. To prevent this inhibitory effect,
artificial sulfinic acid sodium salts are sometimes
added to the bonding agent15,37. The differences in
compositions between the 2 core build-up resin
composites might be responsible for the difference
in their KHN behaviors.

In the present study, both resin composites at
all depths of cavity had a post-curing effect 7 days
after irradiation, showing statistically higher KHN
values than those at 0.5 hours post-irradiation
for all light-exposure methods. These results are
consistent with those of a previous study\(^3\), but not with those of another study\(^4\), which did not find changes in microhardness values 24 hours after irradiation. However, the polymerization reaction of the dual-cured materials might be specific\(^9\), and these previous studies used resin cements, whereas dual-cured core build-up resin composites were used in the present study. In fact, in the former study, neither did the authors evaluate dual-cured core build-up resin composites nor did they evaluate hardness behavior of dual-cured core build-up resin composite in simulated depths of cavity.

Dual-cured resin composites have been introduced for use as both luting and core build-up materials. Superior physical properties are important for a successful restoration. In this study, it was found that the KHNs of DCP were superior to those of UCE, regardless of exposure methods. Various factors can influence the microhardness of a resin composite, such as filler load, type, or size, or resin matrix type\(^{17,18}\). Increasing the monomer viscosity decreased the hardness\(^27\). In this study, the association between KHN values of the resin composite and composition of filler and matrix resin was not evaluated. On the other hand, the differences between the KHNs at the depths of 0.5 mm and 10.0 mm in UCE were lower than those in DCP, regardless of the light-exposure method. Dual-cured materials differ markedly in terms of the relative content of light- and self-activated catalysts\(^{13}\). Differences in the degree of conversion among materials when subjected to various curing protocols may consequently be attributed to variations in catalyst systems. In the present study, since similar KHNs were observed irrespective of the light-exposure method at greater depths of cavity in UCE rather than DCP, it might be inferred that the former exhibits high levels of chemically curing activators compared with the latter, compensating for attenuation of light energy at greater depths of cavity. Indeed, the polymerization behavior of dual-cured resin composites is strongly related to the material and can vary as a function of composition\(^35\). The speed of the polymerization reaction is strongly influenced by inhibitor concentration in the unfilled light-cured methacrylate-based systems\(^{30}\). Therefore, the curing mechanism of a specific composite material may not be applicable to other materials.

**CONCLUSIONS**

Within the limitations of the present study, the following conclusions can be drawn:

1. The microhardness of the dual-cured core build-up resin composites vary depending on the light-exposure method, including irradiation duration, use of bonding agent, depth of cavity, post-irradiation time, and material brand.

2. For both resin composites, irradiation for 120 seconds does not result in significant differences in KHNs among all 6 depths of cavity at all post-irradiation times.

3. Based on the physical property of microhardness behavior, dual-cured core build-up resin composites are preferably prepared by the effective exposure method.

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