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ORIGINAL ARTICLE

Influence of the interaction of light- and self-polymerization on subsurface hardening of a dual-cured core build-up resin composite

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Abstract

Objective. To investigate the influence of time delay and duration of photo-activation on subsurface microhardness of a dual-cured resin composite. ***Material and methods.*** A commercially available dual-cured core build-up resin composite (Rebilda DC) was filled in cavities (diameter: 4.0 mm; height: 6.0 mm) of polystyrene molds and light-cured for 20 or 60 s either immediately after the filling procedure (time delay 0 s) or after a time delay of 30, 90, 180 or 300 s. Non-irradiated self-cured specimens served as a control group (n = 15). Specimens were stored in complete darkness and at 100% relative humidity at 37°C for 2 weeks and cross-sectioned. Knoop Hardness Numbers (KHNs) were measured six times per depth and averaged at distances of 0.25, 0.50, 1.00, 2.00, 3.50 and 5.50 mm from the light-exposed surface. Data were statistically analyzed using one- and two-way ANOVA followed by Scheffé's post-hoc test at a level of significance of 0.05. ***Results.*** Mean hardness values in all experimental groups ranged between 54.3 ± 2.1 and 58.1 ± 2.3 KHN. Light-curing did not significantly increase composite KHN at any depth measured. Delaying light exposure had no influence on KHN, irrespective of depth. A longer light-exposure time (60 versus 20 s) resulted in significantly higher KHN only at depths of 3.50 and 5.50 mm. ***Conclusion.*** Photo-activation of the tested dual-cured resin composite provided no clinically relevant benefit compared to self-curing regarding the degree of hardening.

Key Words: *Dual-cured resin composite, light polymerization, microhardness*

Introduction

Dual-cured resin composites are widely used in modern adhesive restorative dentistry as both core build-up and luting materials. Self- or light-cured materials seem to be appropriate for these applications as well but have their limitations. While self-cured materials do not allow the clinician to adjust the setting time individually, light-cured resin composites cannot ensure adequate polymerization in areas with limited access for the curing light. Thus, attenuation of light at an increased composite depth leads to potential gradation of the extent of conversion at subsurface levels beyond 2 mm [1]. Even the use of light-transmitting posts cannot compensate for the limited depth of cure, resulting in incomplete polymerization of light-cured resin composites in the apical region of root canals [2,3]. When luting indirect tooth-colored restorations, a combination of scattering, reflection and absorption phenomena reduces light irradiance [4] and, as a result, compromises polymerization of the luting material depending on the thickness and shade of the intervening material [5]. Inadequate polymerization has been associated with inferior mechanical properties [6], postoperative sensitivity, microleakage, recurrent caries [7] and pulp irritation caused by residual monomers [8].

Dual-cured resin composites have been developed in an attempt to overcome these limitations, and include a redox initiator system in addition to

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photoinitiators. While superficial areas mainly polymerize through photo-activation, which is responsible for set-on-command capability and initial stabilization of the restoration, the chemical setting modality is expected to ensure complete polymerization even in deep portions of the material that have received an insufficient light intensity. However, the incorporation of self- and light-curing modes in the same material has not been shown to guarantee uniform maximal curing. The self-curing option is not only slower [9–11] but also less effective in terms of monomer conversion than when photo-activation is used as a supplement [9,12–15]. Owing to incomplete compensation for deficient light activation, lower hardness values of dual-cured resin composites have been observed with increased cavity depth [16,17], which may weaken the restoration against occlusal loads. However, studies on the curing potential of the two activation modes mainly focused on the initial 24 h of the polymerization reaction. Research into mechanical properties of dual-cured materials after longer observation periods is needed [18].

Despite their independent onset, both curing modes initiate free radical formation and monomer conversion, which overlap each other during the curing process. The exact mechanism of interaction of self- and light-curing is as yet unknown. One approach to optimize the extent of polymerization and consequently the structural integrity of the material might be the application of modified light-curing protocols. The duration of light irradiation affects the polymerization depth [19], degree of conversion [20] and hardness [21] of resin composites. Recently, it has been speculated that a delay of photo-activation would be beneficial in enhancing the degree of conversion of dual-cured materials, as immediate exposure to light could interfere with the self-curing mechanism [22].

Based on these considerations, the purpose of the present *in vitro* study was to systematically investigate the influence of time delay and duration of photo-activation on the extent of polymerization at different depths of a dual-cured core build-up resin composite. Microhardness (as an indirect measure of degree of conversion) was determined 2 weeks after the initiation of polymerization in order to assure ultimate double-bond conversion and consequently to gain an insight into the maximum curing potential of each protocol tested.

Material and methods

Specimen preparation

The dual-curing core build-up resin composite Rebilda DC (VOCO, Cuxhaven, Germany) was used. Details of the tested material are listed in Table I. For fabrication of the composite specimens, molds with through holes (diameter: 4 mm; height: 6 mm) were prepared from white polystyrene. The core build-up material was filled in the cavities of the molds using QuickMix syringes in combination with the corresponding mixing tips type 11 and thin application tips type 4 (VOCO). A 1-mm thick glass plate (Schott, Mainz, Germany) with minimum 90% permeability to light of wavelengths between 380 and 2400 nm was placed on each mold, squeezing out any excess material. The duration of the filling procedure of each cavity and positioning of the glass plate was measured with a stopwatch and did not exceed 15 s, otherwise the specimens were discarded.

Specimens were light-cured for 20 or 60 s through the glass plate at a standardized distance of 1 mm from the test material either immediately after the filling procedure (time delay 0 s) or after a time delay of 30, 90, 180 or 300 s. Non-irradiated self-cured specimens served as controls. The corresponding experimental groups (n = 15) are described in Table II. Light intensity (high-intensity mode) of the light-curing unit (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein) was controlled periodically during the experiment with a radiometer (Optilux Model 100; SDS Kerr, Danbury, CT), ensuring a constant output intensity of 950 mW/cm^2. Specimens were prepared at ambient room temperature of 28°C ± 1°C. Immediately after their preparation the composite specimens were covered with light-proof adhesive tape and stored for 2 weeks in 100% relative humidity at 37°C.

Table I. Manufacturer's information about the tested composite material Rebilda DC.

Composition (wt.%)	Filler size (μm)	Shade	Batch no.	Manufacturer
Base: bariumborosilicate glass (63.6), fumed silica (6.0), Bis-GMA (5.0), UDMA (19.0), DDDMA (5.0), CQ (0.2), DABE (0.4), N,N-Bis (0.6), BHT (0.2)	1.5	White	0806461	VOCO (Cuxhaven, Germany)
Catalyst: bariumborosilicate glass (64.4), fumed silica (6.0), Bis-GMA (5.0), UDMA (19.0), DDDMA (5.0), BPO (0.4), BHT (0.2)				

Bis-GMA = bisphenol-A-glycidyldimethacrylate; UDMA = urethane dimethacrylate; DDDMA = dodecanediol dimethacrylate; CQ = camphorquinone; DABE = dimethylaminoethylbenzoate; N,N-Bis = N,N-Bis-hydroxyethyl-*p*-toluidine; BHT = butylated hydroxytoluene; BPO = benzoyl peroxide.

Table II. Details of the experimental groups.

Group	Time delay (s)	Light-exposure time (s)
1 (control)	–	0
2	0	20
3	30	20
4	90	20
5	180	20
6	300	20
7	0	60
8	30	60
9	90	60
10	180	60
11	300	60

Determination of Knoop hardness

In order to achieve coplanar centerpieces for determination of microhardness, the molds containing the composite specimens were cross-sectioned with a water-cooled diamond saw (Isomet; Buehler, Lake Bluff, IL) and the cut surfaces were polished with 4000 FEPA P SiC paper. Knoop Hardness Numbers (KHNs) were determined using a digital microhardness tester (Model No. 1600–6106; Buehler). A load of 50 g was applied, with a dwell time of 20 s. Hardness measurements were performed at the following distances from the light-exposed surface: 0.25, 0.50, 1.00, 2.00, 3.50 and 5.50 mm. For each specimen, six measurements were made at each depth. Across all the groups, a total of 5940 hardness measurements were performed.

Statistical analysis

Based on preliminary data with six specimens in each group, a power analysis was performed to determine the number of specimens required in each experimental group. According to this analysis, 15 specimens per group were used to gain a power of 94%.

The mean KHN of each depth was calculated from six measurements for each specimen. Data were statistically analyzed using one-way ANOVA for comparison of all groups at the same depth and between different depths within each group. In addition, one-way ANOVA was performed between light-cured groups (Groups 2–6 and 7–11) and the self-cured control group (Group 1) for each depth. Two-way ANOVA was conducted for the factors time delay and light-exposure time. Scheffé's post-hoc test was used for multiple comparisons. In all analyses, the level of significance was set at 0.05. Calculations were performed using the SPSS 14.0 software for Windows (SPSS Inc., Chicago, IL).

Results

Mean KHNs and standard deviations (SDs) of all experimental groups at the respective depths ranged between 54.3 ± 2.1 and 58.1 ± 2.3 KHN, as presented in Table III. Comparisons between groups at the same depth revealed that light-curing did not significantly increase composite KHN at any depth measured ($P > 0.05$). In addition, within each experimental group no significant differences in KHN were detected between different depths ($P > 0.05$). Two-way ANOVA for the factors time delay and light-exposure time showed that the delay of light exposure as well as the interaction between the two factors had

Table III. Mean (SD) KHN of the experimental groups at the respective depths of the Rebilda DC test material ($n = 15$)[a].

			KHN at different depths (mm)					
Group	TD (s)	LET (s)	0.25	0.50	1.00	2.00	3.50	5.50
1 (control)	–	0	55.8 (2.3)	56.0 (2.5)	57.2 (2.6)	56.8 (2.3)	56.6 (2.2)	54.9 (2.5)
2	0	20	55.1 (2.6)	55.8 (2.8)	55.7 (2.5)	56.8 (2.4)	56.4 (1.8)	55.9 (2.4)
3	30	20	56.5 (2.4)	56.2 (1.9)	56.8 (2.5)	57.1 (2.4)	57.3 (1.9)	57.2 (2.7)
4	90	20	56.6 (2.7)	57.1 (2.2)	58.1 (2.3)	57.8 (2.3)	57.5 (2.3)	56.0 (1.9)
5	180	20	56.5 (2.7)	56.5 (2.7)	56.5 (1.7)	56.5 (2.6)	56.4 (3.0)	54.3 (2.1)
6	300	20	55.1 (2.4)	55.8 (2.0)	57.0 (2.1)	57.6 (2.3)	57.0 (2.0)	55.4 (2.7)
7	0	60	56.5 (2.7)	55.9 (2.4)	56.6 (2.2)	57.5 (2.5)	57.9 (2.1)	57.1 (3.0)
8	30	60	56.2 (3.1)	56.6 (3.3)	57.1 (2.9)	56.9 (2.8)	57.4 (2.1)	57.0 (1.9)
9	90	60	56.1 (3.8)	56.3 (3.5)	56.9 (2.3)	57.7 (2.7)	57.7 (2.3)	56.3 (2.6)
10	180	60	55.1 (1.7)	55.6 (1.6)	56.2 (1.6)	57.3 (3.8)	57.8 (3.1)	56.6 (3.1)
11	300	60	55.7 (3.7)	56.4 (3.2)	56.8 (3.7)	56.7 (1.9)	57.7 (2.4)	56.6 (1.9)

[a]None of the differences are statistically significant at the 0.05 level.
TD = time delay before light exposure; LET = light-exposure time.

no significant influence on KHN, irrespective of depth ($P > 0.05$). Figure 1 illustrates the influence of light-exposure time on KHN at the respective depths. A longer light-exposure time (60 versus 20 s) resulted in significantly higher KHN only in the deepest composite layers, at 3.50 mm ($P = 0.038$) and 5.50 mm ($P = 0.018$). However, one-way ANOVA between 20-s light-cured groups (Groups 2–6) and controls (Group 1) and between 60-s light-cured groups (Groups 7–11) and controls (Group 1) revealed no significant differences in KHN at any depth measured ($P > 0.05$).

Discussion

Adequate polymerization is a prerequisite for the overall clinical success, longevity and biocompatibility of resin composite restorations. The effectiveness of polymerization may be assessed either directly or indirectly. Direct methods measuring the degree of conversion, such as Fourier transform infrared spectroscopy (FT-IR) [23] or laser Raman spectroscopy [24], are the most sensitive techniques, but are time-consuming and complex. To simplify the measurements, various indirect methods have been described in the literature. These include changes in optical translucency [25], scraping [26], resin leaching [27] and hardness measurements [28]. Knoop hardness has been shown to correlate well with FT-IR [27,29,30] and was therefore used in the present study to reflect monomer conversion at different depths of the tested dual-cured resin composite. However, the prediction of an absolute value of degree of conversion by means of an absolute hardness value is not valid, because, in addition to the degree of conversion, other factors such as filler load, size and type, as well as monomer composition and the density of network cross-linking, affect the microhardness of resin composites [31–33].

According to Meredith et al. [34], dentin hardness ranges from 50 to 70 KHN, depending on the distance from the amelodentinal junction. The mean hardness value of 56.6 KHN measured for Rebilda DC in the present study therefore predicts dentin-like mechanical properties. This observation renders the material suitable for build-ups later prepared for taken-up crowns or bridges, since the cutting behavior is similar to that of dentin. However, the microhardness of resin composites may be affected by the molds in which specimens are prepared [35]. White polystyrene was used as mold material due to the fact that its light reflection ranges between that of dentin on the one hand and that of metal on the other. This imitates reflection phenomena in cavities that are surrounded by both dental hard tissue and the metal matrix commonly used when placing core build-ups.

Temperature is known to have a significant influence on final conversion values of dimethacrylate-based materials by affecting monomer mobility and thus the onset of autodeceleration of the polymerization reaction [36,37]. According to Plasmans et al. [38], rubber dam application, as recommended for adhesive restorations, results in virtually the same relative humidity and temperature intra-orally as in the dental surgery. A preliminary study was performed in order to gain insight into tooth temperature during the filling procedure. Tooth 16 of a volunteer was isolated by means of a rubber dam at a constant room temperature of 20°C. Measurements were taken

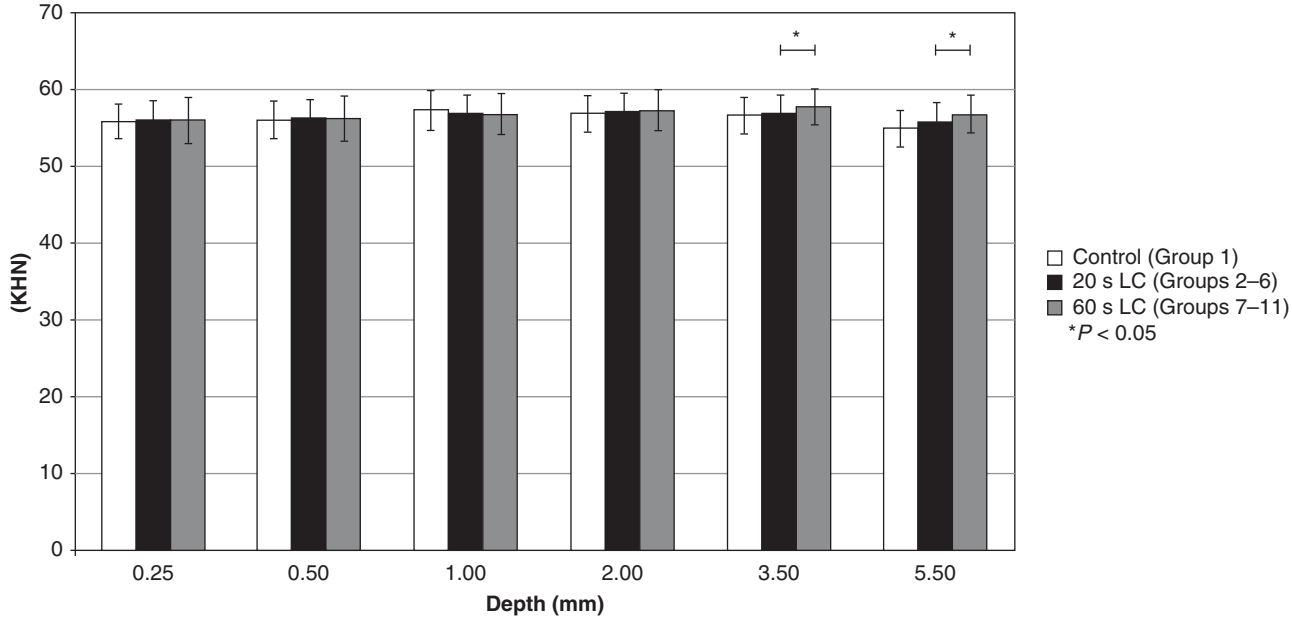

Figure 1. Mean (SD) KHN of non-irradiated controls (Group 1) and of all the groups light-cured (LC) for 20 s (Groups 2–6) or 60 s (Groups 7–11) at the respective depths of the Rebilda DC test material ($n = 15$).

15 min after application of the rubber dam using a thermocouple (TES-1303; TES, Taipei, Taiwan) and revealed a tooth-surface temperature of 28°C. This observation is consistent with results published by Pogrel et al. [39], who found that tooth temperature after rubber-dam application varied from 27.5°C to 29.2°C. Therefore, composite specimens in the present study were prepared at ambient room temperature of 28°C ± 1°C prior to storage for 2 weeks in 100% relative humidity at 37°C.

During hardness measurements, light had to be applied to the specimens. In a second preliminary study it was therefore evaluated whether exposure to light after 2 weeks of dark storage influences the microhardness of the resin composite. For that purpose, four specimens of Rebilda DC were allowed to self-cure for 2 weeks, whereas two of the specimens were subsequently light-irradiated for 120 s with the light-curing unit in high-intensity mode. No differences in hardness values at the top surface were determined between the light-irradiated and non-irradiated groups, indicating that the restricted polymer network that developed during 2 weeks of self-curing did not allow any additional mobility of the polymer chains, which justified light application during hardness determination.

The present study demonstrated that light irradiation does not affect the microhardness of the tested resin composite (Table III, Figure 1), indicating a similar extent of polymerization in self- and dual-curing modes. This finding suggests a different trend to that observed in previous reports, in which photo-activation of dual-cured materials resulted in a higher degree of conversion compared with chemical activation alone [9,12–15]. El-Mowafy et al. [7] found that for three of eight examined dual-cured resin cements, self-curing produced hardness values <50% of those obtained when dual-curing was used. They concluded that the self-curing option is not appropriate to achieve sufficient hardening, even after 1 week of storage. However, it has also been reported that the polymerization behavior of dual-cured resin composites is strongly material-related and can vary as a function of composition [40]. Therefore, conclusions regarding the curing mechanism of a specific composite material may not be transferred to other products. According to Hasegawa et al. [41], dual-cured materials differ markedly in terms of the relative contents of light- and chemically activated catalysts. Differences in the degree of conversion among materials when subjected to various curing protocols may consequently be attributed to variations in catalyst systems. Because in the present investigation similar hardness values were observed irrespective of composite depth (Table III), it might be inferred that the tested material exhibits high levels of chemical-curing activator compensating for attenuation of light energy in the deep part of the restoration. The equal degree of polymerization within the core material may support a uniform distribution of stress along tooth–material interfaces under load. Even though the results of the present study may be limited to the specific resin composite tested, the evaluation of only a single core build-up material is justified due to the large number of measurements that were performed in order to gain insight into the exact polymerization mechanism in different subsurface composite layers. This research project particularly intended to thoroughly investigate the influence of the moment of photo-activation on the interaction between the self- and light-curing modes, a parameter occasionally overlooked in studies on the polymerization behavior of dual-cured materials. Rebilda DC was chosen as the test material since the exact quantitative compositions of both the base and catalyst paste were revealed by the manufacturer. In general, such information is not available for commercial products, only for model materials.

A previous study directly measured C=C conversion of Rebilda DC at 15 min post-mix and found that monomer conversion was significantly lower in self-curing mode compared with dual-curing [12]. The similar extent of polymerization of the same material in the presence or absence of light 2 weeks after initiation of polymerization, as observed in our study, indicates an increased impact of the self-curing mode with time and may be explained by the difference in velocity between the two curing modes. Lee et al. [10] reported up to ≈320 times slower curing speeds by chemically induced cure than by additional light irradiation, suggesting that very early measurements may severely underestimate the progressive hardening potential of the self-curing mode. Microhardness is generally determined 24 h after initiation of polymerization. However, a substantial increase in the degree of cure following the first day post-mix has previously been described for dual-cured resin cements subjected to self-curing only [42]. Consequently, setting times in the present study were extended to 2 weeks, in order to ensure ultimate double-bond conversion and hence to allow an assessment of the maximum hardening potential of the various polymerization scenarios tested.

Recently, Meng et al. [43] observed that light irradiation shortly after composite application inhibits the self-curing mechanism of dual-cured materials, probably by entrapping polymerization promoters and unreacted monomers in the polymer network. In order to avoid premature interaction between the two curing modes, it has been recommended to delay photo-activation of dual-cured resin composites to the maximum time clinically possible [22]. The present study revealed that the moment of photo-activation does not affect the microhardness of the tested core build-up material. It is therefore supposed that immediate photo-activation, despite causing a rapid

increase in the viscosity of the polymer matrix, does not hinder migration of activated free radicals responsible for further chemically induced polymerization. Moreover, delayed photo-activation does not seem to cause synergic effects of the curing modes given that neither short (30 s) nor medium (90 s, 180 s) nor long (300 s) delay periods were able to increase the extent of polymerization. Thus, the structure of the polymer network at the start of light polymerization does not influence the curing potential of the tested material. In accordance with our results, Moraes et al. [13] reported that delayed photo-activation does not affect the degree of conversion of dual-cured materials. On the other hand, recent literature demonstrates that delaying light irradiation of dual-cured resin composites reduces microleakage in Class II restorations [44]. Therefore, delayed photo-activation procedures, allowing for some initial conversion by the self-curing mode, might be beneficial in reducing the shrinkage stress of dual-cured materials. This speculation warrants investigation in future studies.

Evaluation of the duration of photo-activation revealed that a longer exposure time (60 versus 20 s) results in significantly higher hardness values in the deepest composite layers, at 3.50 and 5.50 mm (Figure 1). Although statistically significant, differences in microhardness between groups light-cured for 20 or 60 s were <2% at both depths and therefore extremely small in absolute terms. Thus, the effect of longer photo-activation seems not to be clinically relevant for the tested material.

The self-curing mechanism of dual-cured resin composites is usually based on a redox reaction of benzoyl peroxide with aromatic tertiary amines, generating free radicals that will break the aliphatic carbon double bonds to start the polymerization process. Both peroxides and amines are organic compounds with limited storage stability. Dentists should be aware that deterioration of these components may compromise the effectiveness of the self-curing mechanism even before the expiration date, particularly at increased storage temperature. For the present study, fresh material was provided by the manufacturer, which was stored at a cool temperature when not in use. These conditions may have contributed to the good performance of the chemically induced setting reaction.

Based on the results of the present *in vitro* study, it can be concluded that photo-activation of the tested core build-up resin composite provides no clinically relevant benefit compared to self-curing regarding the degree of hardening achieved 2 weeks after initiation of polymerization. The dominant self-curing mode allows application of the tested material in areas that are inaccessible to the curing light. Further research into the effect of different curing protocols on the extent of polymerization is warranted with other dual-cured core build-up resin composites.

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