Temperature Changes in Silorane-, Ormocer-, and Dimethacrylate-Based Composites and Pulp Chamber Roof during Light-Curing

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ABSTRACT

Statement of the Problem: Light-curing of resin-based composites (RBCs) is associated with temperature increase in the pulp chamber, which may have a detrimental effect on the vital pulp.

Purpose: The purpose of the study was to evaluate temperature changes of silorane-, ormocer-, and dimethacrylate-based RBCs at the bottom surface of the RBC and in the pulp chamber roof dentin (PCRD) during curing.

Materials and Methods: In part A, temperatures were measured for Filtek LS (3M ESPE, St. Paul, MN, USA), Admira (Voco GmbH, Cuxhaven, Germany), and Herculite XRV (Kerr Corp., Orange, CA, USA) with a high-power light-emitting diode (LED) unit by placing thermocouples in contact with the bottom surface of the material in standardized acrylic molds. In part B, temperature changes in PCRD were measured in extracted molars during light-curing of adhesives and RBCs in 2-mm-deep cavities with a remaining dentin thickness (RDT) of 1 mm.

Results: Filtek LS showed a different temperature curve compared with Admira and Herculite XRV. Significantly higher temperatures were recorded for Filtek LS ($p < 0.001$) than for Admira and Herculite XRV in acrylic molds. Temperature rises recorded in PCRD for adhesives and RBCs were between 4.1 and 6.4°C. No significant differences in PCRD temperatures were found between the three groups during adhesive curing and RBC curing ($p > 0.05$).

Conclusions: Filtek LS showed a different heat-generation pattern from and significantly higher temperatures than Admira and Herculite XRV when the materials were tested in acrylic molds. Similar temperatures were recorded in the PCRD during curing of adhesives and RBCs.

CLINICAL SIGNIFICANCE

Although a substantial temperature rise in the bulk material occurred during light-curing of the three resin-based composites, a remaining dentin thickness of 1 mm caused a significant reduction in pulp chamber roof dentin temperatures. Temperatures measured in the pulp chamber roof dentin corresponding to the zone occupied by the postmitotic odontoblast layer were not statistically different for the three types of resin-based composites.

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INTRODUCTION

Light-curing of resin-based materials may be a potential source of thermal stress to the vital pulp because heat is generated as a synergic combination of the light source and exothermic polymerization reaction within these materials.[1,2] The reported 5.5°C threshold for irreversible changes in the pulp tissue[3] remains controversial and questioned by other authors.[4]

Temperature changes have been studied in resin-based composites (RBCs) in experimental conditions using a deflecting disk technique with thermocouples (TCs),[1] thermistor beads,[5] pulp chamber-simulating molds, or high-resolution infrared cameras.[6]

Increase in temperature in the pulp chamber has been recorded during light-curing of different adhesives and RBCs using various light-curing units (LCUs).[2,7–10] Two main approaches have been employed in such studies, either placing TCs in the pulp chamber through resected roots[2,7,10] or using a "split-tooth" technique, which allows direct visual control of the remaining dentin thickness (RDT) and TC placement.[8,9] Temperature rise has been reported to be greater during curing of adhesives than RBCs when the same light energy output is used[8] and lower or comparable with lower source outputs.[2,10]

The effect of RBC polymerization using different LCUs on the number of living human gingival fibroblasts in a pulp chamber model revealed that the percentage of surviving fibroblasts was dependent on the chemical composition of RBCs rather than on the LCU type.[11]

Filtek LS (3M ESPE, St. Paul, MN, USA), a new RBC based on silorane resin, has been introduced to the market in an attempt to reduce polymerization shrinkage. Its siloxane–oxirane resin molecule polymerizes via a cationic ring-opening reaction, induced after the interaction between camphorquinone, iodonium salts, and electron donors. It has been recommended by the manufacturer that light-curing should be no less than 20 seconds, as this time is required for initiator activation.

Admira (Voco GmbH, Cuxhaven, Germany) is an ormocer-based RBC with an inorganic silicon dioxide backbone and dimethacrylates as "side-branches," dispersed filler particles, and a small amount of pure dimethacrylate monomers. A three-dimensional polymer network is formed via free-radical polymerization in which methacrylate groups interact with free radicals, similar to conventional dimethacrylate RBCs.

The purpose of the study was to evaluate the effect of silorane-, ormocer-, and dimethacrylate-based RBCs on temperature changes at the bottom surface of the RBCs and in dentin of the pulp chamber roof during light-curing. The null hypothesis was that there is no difference in temperature changes among the three RBCs at the bottom surface of the RBCs and in the pulp chamber roof dentin (PCRD) during light-curing.

MATERIALS AND METHODS

Temperature changes were recorded (A) at the bottom surface of RBCs and (B) in PCRD. Table 1 lists the adhesive systems and RBCs used in the present study.

Part A: Temperature Changes at the Bottom Surface of RBCs

A precision metal rod 5 mm in diameter was placed vertically in a rubber mold 1.5 × 1.5 × 6 cm, and freshly mixed white acrylic (Skillbond, Skillbond Direct Ltd., Bucks, UK) was poured around this and cured for 24 hours in an autopolymerization bath (Palamat practice EL T, Heraeus Kulzer Ltd., Newbury, UK) at temperatures between 45 and 55°C and a pressure of 2.5 bar. After curing, the metal rod was removed, leaving an acrylic block with a 5-mm-diameter internal cylinder. Eight such acrylic blocks were made. From these acrylic blocks, 30 standardized molds, 2-mm thick, were cut using an Isomet saw (Buehler, Lake Bluff, IL, USA) and randomly

TABLE 1. ADHESIVE SYSTEMS AND RESIN-BASED COMPOSITES USED IN THE PRESENT STUDY.

Material	Composition	Manufacturer
LS system adhesive self-etch primer	Phosphorylated methacrylates, Vitrebond copolymer, bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers	3M ESPE, St. Paul, MN, USA
LS system adhesive self-etch bond	Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers	
Filtek LS	Silorane resin, initiating system, quartz filler, yttrium fluoride, stabilizers, pigments	
Admira Bond	Dimethacrylates, acetone, ormocers, catalysts, auxiliaries	Voco GmbH, Cuxhaven, Germany
Admira	Ormocer, bis-GMA, UDMA, silicate filler, catalysts, stabilizers	
OptiBond Solo Plus	Etchant: 37.5% phosphoric acidAdhesive: bis-GMA, HEMA, GDMA, GPDM, ethanol, filler, initiators	Kerr Corp., Orange, CA, USA
Herculite XRV	Alkyl dimethacrylates, TEGDMA, filler, activators, stabilizers	

bis-GMA, Bisphenol A glycol dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, Urethane dimethacrylate; GDMA, glycerol dimethacrylate.

allocated to three groups. The molds were placed on a Mylar strip on a plastic stand, filled with the RBC, and covered with a Mylar strip (KerrHawe, Scafati, Italy). The bottom Mylar strip was then carefully removed, and a K-type TC (RS Components, Corby, UK) was placed in contact with the bottom side of the material in such a way that half of the TC tip was embedded inside the RBC material. A new TC was used for each specimen and was connected via a data logger (Measurement Computing Corp., Norton, MA, USA) to a computer and Tracer DAQ software (Measurement Computing Corp.). A 1-mm-thick, Teflon light guide with a diameter of 6.0 mm (i.e., just smaller than the LCU tip) was placed on the top side of the sample to maintain a standardized curing distance. The sample was then cured for 20 seconds with a high-power LED LCU (1,100 mW/cm^2; bluephase, Ivoclar Vivadent, Schaan, Liechtenstein). Light intensity was monitored using the integrated photometer before and after curing to ensure that either the low-power or high-power mode was used. The temperature was recorded at 1-second intervals throughout the curing process and afterwards until it returned to the baseline level. Figure 1 shows the experimental setup.

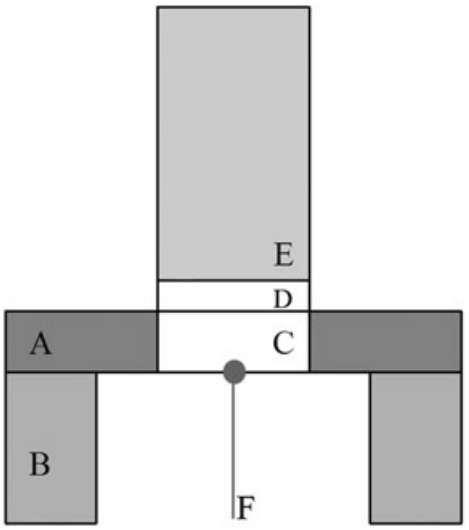

Figure 1. Experimental setup. A, acrylic mold; B, stand; C, resin-based composite; D, light guide; E, LED unit; F, thermocouple.

Additionally, temperature was recorded during 20 seconds of

irradiation through an empty mold and at the tip of the LCU using the high-power mode. Six measurements were taken; each subsequent measurement was taken 5 minutes after the temperature from the previous one had reached the baseline.

Part B: Temperature Changes in the PCRD

Thirty intact human third molars extracted for orthodontic reasons and stored in 0.02% thymol for not more than 4 months were selected for the study. Informed consent was gained for the use of these teeth for research purposes. The age range of patients was 20 to 30 years. Prior to the study, the teeth were cleaned of all debris using an ultrasonic scaler.

The previously described "split-tooth" technique was used to create standardized 2-mm-deep cavities with 1-mm RDT and to place the TC in the middle of the pulp chamber roof in direct contact with dentin.[9] The cusps were reduced to create a flat occlusal plane just into dentin using a diamond bur in a high-speed handpiece with water spray. A preliminary cavity was cut into dentin but not extending more than 1.5 mm from the occlusal plane using the same diamond bur and a Class I large Cerana diamond bur (Nordiska Dental, Angelholm, Sweden; dimensions: height, 3 mm; top diameter, 4 mm; bottom diameter, 3 mm) was used to enlarge the initial cavity. At this stage, the depth of the cavity was underprepared. The teeth were then sectioned through the prepared cavity, 1-mm off-center, along the mesial-distal plane using a diamond saw and water spray. The height of the Cerana bur was marked at 2 mm, and standard 2-mm-deep cavities with 1-mm RDT were prepared using this bur. Cavity depth and RDT were monitored using a digital caliper (Moore & Wright Europe, Maastricht, the Netherlands).

The TC was placed in contact with the dentin of the pulp chamber roof exactly in the midline of the cavity, in the larger of the two sections. The two sections were then glued together using Araldite adhesive (Bostik, Findlay, Staffordshire, UK).

The teeth were embedded up to the cementoenamel junction in an oasis (flower display sponge) and kept in a water bath until the temperature inside the pulp chamber was stable at 37 ± 1°C. Figure 2 shows the experimental setup.

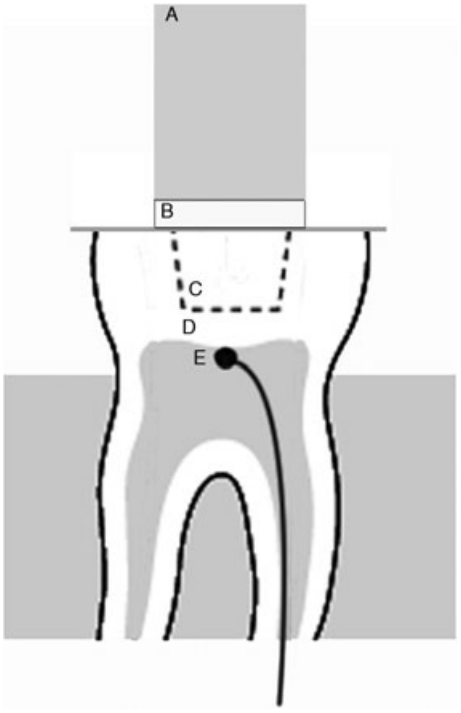

Figure 2. Prepared teeth and the thermocouple (TC) placed in contact with the pulp chamber roof. A, LED unit; B, light guide; C, 2-mm-deep cavity; D, 1-mm remaining dentin thickness; E, TC.

Before cavity restoration, 10 randomly selected teeth were irradiated using the low-power mode (650 mW/cm^2, bluephase) for 20 seconds through the light guide. Five minutes after the PCRD temperature had returned to the baseline level, the teeth were irradiated using the high-power mode (1,100 mW/cm^2, bluephase) for 20 seconds. The TC was connected via a data logger to a computer, and temperature was recorded using the software package (Tracer DAQ) at 1-second intervals throughout the experiment.

The teeth were randomly allocated to three groups:

1. Group I: Filtek LS: The primer was applied with a disposable applicator and light-cured through the light guide for 20 seconds in the low-power mode. After the PCRD temperature had returned to the baseline level, the bond was applied and light-cured in the same fashion. Similarly, when the temperature returned to the baseline level, Filtek LS was placed in the cavity, covered with a Mylar strip, and light-cured through the light guide for 20 seconds using the high-power mode of the LED LCU. The light intensity of the LED unit was monitored through its own integrated photometer. The TC was connected via a data logger to a computer, and temperature was recorded using Tracer DAQ at 1-second intervals throughout the experiment.
2. Group II: Admira: The bond and RBC were applied and light-cured as in group I.
3. Group III: Herculite XRV: OptiBond Solo Plus and Herculite XRV were applied and light-cured as in groups I and II.

Statistical Analysis

The Kolmogorov–Smirnov test was used to assess whether the data followed a normal (Gaussian) distribution, whereas Bartlett's test was used to confirm the equal variances between the groups. Paired *t*-test and one-way analysis of variance (ANOVA) with Tukey's multiple comparisons post-test at a 95% confidence level were used to assess the differences between the groups (Minitab 15, Minitab, State College, PA, USA).

RESULTS

As the Kolmogorov–Smirnov test confirmed a Gaussian distribution ($p > 0.15$) and Bartlett's test confirmed equal variances between the groups ($p > 0.05$), the two conditions for the use of parametric tests were met. Therefore, paired *t*-test and one-way ANOVA were used to assess the differences between the groups.

Part A: Temperature Changes at the Bottom Surface of RBCs

Significantly higher temperatures were recorded at the LCU tip (57.05 ± 0.48°C) than at the 3-mm distance (45.52 ± 1.05°C) when the TC was irradiated through the empty mold using the high-power mode of the LED unit ($p < 0.001$; paired *t*-test).

Filtek LS temperature curve showed a rapid increase during the first 5 seconds of light-curing with the maximum of around 72°C, ($\Delta T \sim 45$°C from the baseline). During the next 15 seconds of light-curing, the temperature started to decrease to values of around 63°C ($\Delta T \sim 37$°C from the baseline). Temperature curves for the Admira and Herculite XRV showed similar patterns, but different from Filtek LS. A rapid, but less sharp than for Filtek LS, increase in temperature was recorded in the first 5 seconds, with a plateau toward the end of the light-curing period and maximum values in the last 1 second of irradiation (Figure 3).

There were statistically significant differences between the maximum temperature and the temperature recorded at the end of light-curing for Filtek LS ($p < 0.001$).

As the mean baseline temperatures recorded in RBCs placed in the acrylic molds at room temperature

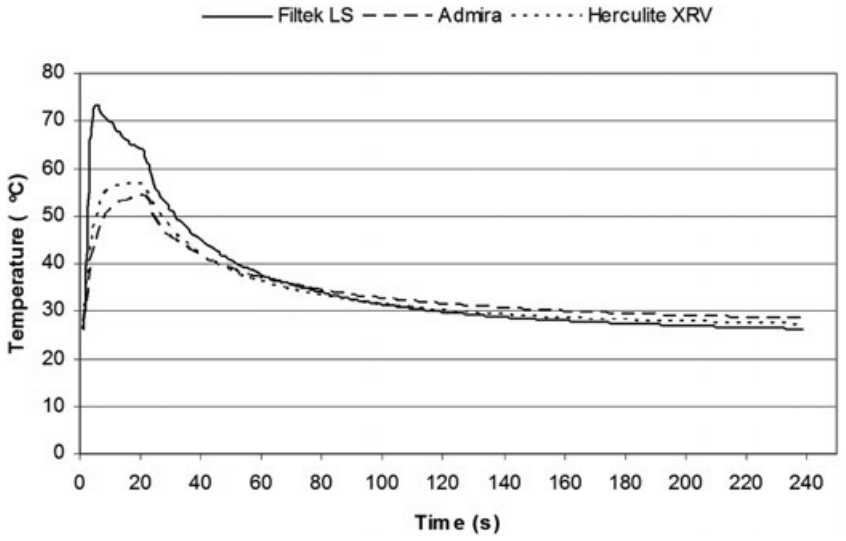

Figure 3. Representative temperature curves for the three resin-based composites (RBCs) recorded during light-curing of RBCs placed in the acrylic molds (part A of the study).

TABLE 2. MEAN AND SD VALUES FOR MAXIMUM TEMPERATURES RECORDED FOR THE THREE RBCs.

Temperatures (°C)	RBC		
	Filtek LS	Admira	Herculite XRV
Mean To (SD)	26.28 (0.61)	27.73 (0.63)	27.06 (1.20)
Mean Tmax (SD)	72.01 (2.31)	54.69 (3.45)	56.07 (2.56)
Lower 95% CI	69.87	51.50	53.70
Upper 95% CI	74.15	57.88	58.44
Mean ΔT (SD)	45.73 (1.93)	26.96 (3.04)	29.01 (2.65)

RBC = resin-based composite; CI = confidence interval; To = baseline temperature; Tmax = maximum temperature; ΔT = temperature change.

varied from 26.28 ± 0.61°C for Filtek LS to 27.73 ± 0.63°C for Admira, the mean temperature rise values were use in the statistical analysis.

The mean temperature rise for Filtek LS was significantly higher than the mean temperature rise for Admira and Herculite XRV ($p < 0.001$). No significant difference was found between the mean temperature rise for Admira and Herculite XRV ($p > 0.05$; one-way ANOVA, Tukey's post-test) (Table 2).

Part B: Temperature Changes in the PCRD

Significantly higher PCRD temperatures were recorded when irradiated through an empty cavity with the high-power (47.7 ± 0.4°C) than the low-power (42.5 ± 1.0°C) LCU mode ($p = 0.006$; paired *t*-test). The high-power mode resulted in a more than 10°C rise and the low-power mode in a 5.5°C rise compared with the baseline temperatures.

Since the baseline temperatures in part B showed no significant differences for the three groups ($p > 0.05$), the absolute recorded temperatures were used in the statistical analysis. No significant differences in maximum recorded temperature values were found between the three groups during light-curing of adhesives ($p = 0.065$) and RBCs ($p = 0.265$; one-way ANOVA with Tukey's post-test). Furthermore, neither adhesive nor RBC curing within each group produced significant temperature changes of PCRD ($p > 0.05$; paired *t*-test) (Figure 4).

Figure 5 shows the same pattern of heat generation in PCRD for the three groups. Temperatures showed a continuous rise throughout the light-curing period for both adhesives and RBCs, with maximum temperatures recorded in the last 1 second of light-curing.

DISCUSSION

The novel RBC, Filtek LS, was compared with ormocer and conventional dimethacrylate RBCs in an attempt to study the heat-generation process at the bottom surface of RBCs and subsequent

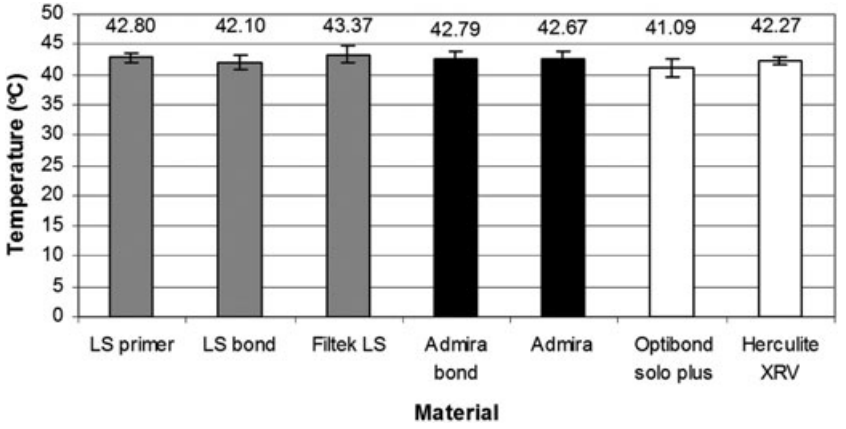

Figure 4. Mean and SD values for temperatures recorded in the pulp chamber roof dentin during light-curing of adhesive systems and resin-based composites (To = 36.8°C ± 0.3°C).

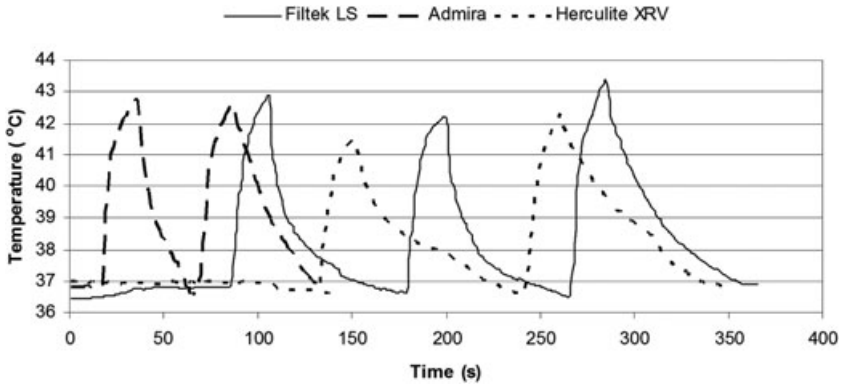

Figure 5. Representative temperature curves recorded in the pulp chamber roof dentin. The first two peaks for Filtek LS represent the adhesive system (Primer and Bond), and the third peak corresponds to the resin-based composite (RBC). In the Admira and Herculite XRV groups, the first peak corresponds to the adhesive and the second one to the RBC.

thermal changes in PCRD. In part A, acrylic molds were used, as acrylic is known to have thermal diffusivity similar to dentin.[12,13] The diameter of acrylic disks was similar to the diameter of the tooth crown, and its white color was chosen to match the color of the tooth. The thickness of the RBC sample and the distance from the LCU tip were reproduced in part B, thus allowing for the known amount of heat to be generated at the bottom surface of the RBC sample, which would then be transferred toward the pulp chamber. Such an approach was adopted as the placement of a TC at the cavity floor through a hole in the crown, used in a previous study, could result in significant loss of dentin because of the TC dimensions and possible change in heat transfer.[2] Furthermore, recording temperature profiles for the three RBCs was one of the aims of the present study, and it would be impossible to differentiate the source of heat measured by the TC, as it would contact both dentin and RBC. Embedding teeth in an oasis allowed water uptake by the spongy environment in order to maintain the constant temperature of 37°C in an attempt to replicate physiological conditions as closely as possible.

The first part of the null hypothesis is rejected, as significant differences between the three RBCs were found in acrylic mold testing, whereas the second part is substantiated, as there were no significant differences in PCRD temperatures.

The results of the part A study indicated higher temperature values and a different temperature curve for Herculite XRV compared with a previous study[1] showing that heat was being generated throughout the light-curing period with high-intensity LCUs. These differences may be attributed to differences in methodology and the lower LCU intensity used in the previous study, with exothermic heat being the major contributing factor to temperature rise during polymerization. In the present

study, the high-power LED unit was used, with the light intensity of 1,100 mW/cm^2 producing a constant temperature rise throughout the curing period. The aim of the present study was to measure temperature during light-curing without determining the contribution of the exothermic reaction and light source, as has been done in previous studies.[1,5] Irradiation of RBC samples after the initial polymerization may not reveal the exact contribution of the light source because a "secondary chemical phenomenon"[14] must be taken into account as well as the possible differences in thermal properties of RBCs in uncured and cured states.

Admira produced virtually the same "fingerprint" of heat generation as that of Herculite XRV. In both Admira and Herculite XRV, polymerizable units are dimethacrylate cross-linking monomers: Bisphenol A glycol dimethacrylate (bis-GMA) and Urethane dimethacrylate (UDMA) in Admira, and alkyl dimethacrylates and TEGDMA in Herculite XRV. Therefore, both RBCs polymerize via free radical polymerization, and this may be the reason for the similar temperature values and heat-generation dynamics in Herculite XRV and Admira.

The novel silorane-based Filtek LS showed a considerably different temperature curve and higher values compared with Admira and Herculite XRV as seen in part A of the present study.

As the light energy input during irradiation was the same for all three RBCs, a substantially different pattern of heat generation observed for Filtek LS may be related to a different polymerization reaction. Filtek LS is based on siloxanes and oxiranes, which polymerize via a cationic ring-opening reaction. This reaction occurs in the oxirane component and is induced by a photochemical event in which camphorquinone, excited by light energy, interacts with iodonium salts and electron donors to produce cations as propagating active centers. Optical pyrometry studies have shown that cationic ring-opening polymerization of oxiranes is a highly exothermic reaction, with temperatures rising from room temperatures to those above 100°C in seconds.[15] It has also been suggested that the rate of heat generated in a cationic polymerization is directly proportional to the number of photogenerated initiating species present in the system.[16] The temperature curve observed in the present study implied that the cationic ring-opening of Filtek LS has the so-called frontal behavior, with the polymerization front of silorane functional monomers showing a very steep temperature profile.[15] The rapid rise in the heat that evolved up to a maximum followed by a slower rate of decline may be associated with an increased rate of "diffusion-controlled termination reactions and reduced mobility of growing polymer chains."[17]

In light of the nature of cationic polymerization, the question of heat effect on the pulp chamber temperature arises. Heat transfer from the three RBCs toward the pulp chamber has been examined in part B of the present study. The terms "intrapulpal" or "pulp chamber" temperature were used in previous in vitro studies, although the TCs were most often in direct contact with the pulp chamber dentin[7–10] rather than positioned free in the pulp chamber.[18] Therefore, such terminology might not be suitable, and we feel that a more appropriate term "temperature of the PCRD" should be adopted, as it reflects the exact location of the measured temperature.

The limitations of this in vitro approach do not allow the recreation of in vivo mechanisms of heat dissipation such as fluid movement within dentinal tubules, blood or lymphatic circulatory mechanisms, or the cellular and intercellular matrix capacity to absorb heat and thus prevent thermal damage of the living pulp tissue. Because the temperature kinetics is unknown in the vital

pulp, the temperature was allowed to return to baseline after primer curing in the Filtek LS group and adhesive curing in all groups before curing the RBCs. This in vitro approach reduces the possibility of extrapolating data to the clinical situation but allows material comparison in standardized conditions and may indicate key points for future research.

In the present study, the PCRD directly beneath the cavity floor was chosen for temperature measurement, as it is the zone of the greatest heat transfer from dentin to the delicate layer of postmitotic odontoblast cells in vivo. A recent study has shown increased synthesis of inflammatory mediators within the pulp cell cultures exposed to temperature increases of up to 7°C.[19] The significance of produced mediators remains to be resolved but suggests the necessity to limit the exposure of the pulp tissue to procedures generating this amount of heat.

In the present study, neither curing of adhesives nor curing of RBCs in the three groups produced differences in temperatures recorded in PCRD. The low-power curing mode was used in the present study for curing of adhesives. Recorded temperatures suggested lower potential risk for heat-induced pulpal injury as the temperature rise was below 5.5°C, in contrast to a previous study when high power was used for adhesive curing.[8] Similar temperatures recorded during curing of adhesives and irradiation of empty cavities suggested that either adhesives had no insulating effect or the exothermic reaction compensated such an effect.

Temperature increases of 4.1 to 5.8°C were recorded in the PCRD during adhesive curing and 5.3 to 6.4°C during RBC curing. Temperatures reported in similar studies varied from ~1 to ~15°C, depending on the LCUs, RDT, RBCs,[7–10] or measurement methods.[2,20] The results of the present study indicated no significant differences between the investigated groups and were below or around the reported threshold value of 5.5°C, which suggested low thermal risk for the pulpal injury. However, in several cases, temperature rise recorded during curing of Filtek LS were nearly 1°C above 5.5°C. In light of the controversial nature of this threshold, the limitations of the in vitro methodology, and the statistically insignificant differences between Filtek LS and the other RBCs, no conclusive statement can be made on the effect of this finding in clinical conditions. As seen in part A of the present study, a significant amount of heat was generated at the bottom surface of all three investigated RBCs. As seen in part B, a vast proportion of this heat was absorbed not only by the 1-mm-thick dentin layer but also by the surrounding coronal dentin. An excellent insulating capability of dentin has been attributed to its low thermal diffusivity and conductivity, which have been reported to be less than half of the values for enamel.[12] Apparently, in teeth subjected to a sudden change in temperature, significantly more time is needed for dentin to reach the saturated diffusivity rate. Even an increase in temperature of more than 40°C, as produced in Filtek LS in acrylic molds, failed to induce significantly higher temperature rise in the underlying dentin of the pulp chamber roof. Temperature curves recorded in the PCRD in the present study correspond to those reported earlier for dimethacrylate-based RBCs.[7,8,18] The characteristic pattern of heat generation seen in Filtek LS RBC (part A) was not seen in the PCRD (part B) and was affected by slow heat diffusion through dentin.

CONCLUSIONS

Filtek LS showed a substantially different mode of heat generation and significantly higher temperatures at the bottom surface of 2-mm-thick samples in acrylic molds compared with Admira and Herculite XRV. There was no statistical difference in temperatures recorded in the PCRD during curing of adhesives in low-power mode and in all three RBCs in high-power mode.

CLINICAL IMPLICATIONS

Clinicians should be aware of the heat generated in RBCs during light-curing, which may be a potential source of pulpal injury. Although temperature rise in the bulk material may differ significantly for different materials, an RDT of 1 mm would seem sufficient to protect the pulp cells from exothermic heat production for the tested materials.

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