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Degree of conversion and *in vitro* temperature rise of pulp chamber during polymerization of flowable and sculptable conventional, bulk-fill and short-fibre reinforced resin composites

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ABSTRACT

Objective. Determine the degree of conversion (DC) and *in vitro* pulpal temperature (PT) rise of low-viscosity (LV) and high-viscosity (HV) conventional resin-based composites (RBC), bulk-fill and short-fibre reinforced composites (SFRC).

Methods. The occlusal surface of a mandibular molar was removed to obtain dentine thickness of 2 mm above the roof of the pulp chamber. LV and HV conventional (2 mm), bulk-fill RBCs (2–4 mm) and SFRCs (2–4 mm) were applied in a mold (6 mm inner diameter) placed on the occlusal surface. PT changes during the photo-polymerization were recorded with a thermocouple positioned in the pulp chamber. The DC at the top and bottom of the samples was measured with micro-Raman spectroscopy. ANOVA and Tukey's post-hoc test, multivariate analysis and partial eta-squared statistics were used to analyze the data ($p < 0.05$).

Results. The PT changes ranged between 5.5–11.2 °C. All LV and 4 mm RBCs exhibited higher temperature changes. Higher DC were measured at the top (63–76%) of the samples as compared to the bottom (52–72.6%) in the 2 mm HV conventional and bulk-fill RBCs and in each 4 mm LV and HV materials. The SFRCs showed higher temperature changes and DC% as compared to the other investigated RBCs. The temperature and DC were influenced by the composition of the material followed by the thickness.

Significance. Exothermic temperature rise and DC are mainly material dependent. Higher DC values are associated with a significant increase in PT. LV RBCs, 4 mm bulk-fills and SFRCs exhibited higher PTs. Bulk-fills and SFRCs applied in 4 mm showed lower DCs at the bottom.

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1. Introduction

Setting reaction of photo-polymerizable resin-based composite restorative materials (RBC) is induced by light curing units (LCU) at different irradiance levels and exposure durations. A certain level of radiant exposure ensures a given degree of monomer to polymer conversion (DC) in the RBC [1,2]. The amount of light energy transmitted to the RBC restoration is influenced by several factors, such as exposure duration, power density of the LCU, the accordance between the spectral absorption range of the photoinitiators and spectral emission profile of the curing unit, distance between the light guide tip and the restoration, composition, shade, opacity and thickness of the RBC material [1,3–6]. Even if the same number of radicals are generated, many of them do not last as long under the condition of higher incident light irradiance and shorter irradiation time as those generated under lower incident light irradiance and longer irradiation time. Thus, even at an identical radiant exposure, the rate of spectral radiant power delivery may also influence the degree of conversion of light activated RBCs [7]. Several studies confirmed that a higher degree of polymerization leads to better physical and chemical properties such as compressive strength, microhardness, wear resistance and biocompatibility [8–11]. Consequently, enhancing the DC is essential for the durability of RBC restorations [12]. An improved DC can be achieved by using a high-irradiance curing unit and/or by lengthening the exposure duration [13], however, this processes involves thermal reactions, which represent a potential hazard for the dental pulp tissue [14,15]. Despite attempts to define the threshold temperature value which would indicate a potentially irreversible pulpal damage, the multiplicity of factors involved in the heat transfer (thickness of the dental tissues, effect of blood circulation in the pulp chamber, fluid motion in the dentinal tubules and the surrounding periodontal tissues) makes it difficult to translate *in vitro* findings to clinically relevant inferences [14,16,17]. Even though the real value of the potentially harmful critical temperature rise is still controversial, it is widely accepted that the temperature rise in the pulp should be kept as low as possible during dental procedures involving the polymerization of light cured materials [18–21].

Temperature rise during polymerization of RBCs is attributed to both the exothermic reaction of polymerization and the energy absorbed during the irradiation from LCUs [22,23]. Several studies investigated this topic, addressing the effect of RBC type, shade, thickness, curing unit radiant energy and spectral characteristics on temperature rise within the pulp chamber [15,24–27]. Most of the investigations found, that compared to the low intensity curing units, the high intensity types produced a significantly higher temperature rise in the pulp chamber [17,28]. Aside from the thermal effect of the light curing unit, the exothermic reaction is the other main contributing factor to the increase in temperature during the early stages of polymerization [29]. The composition of the RBC can also influence thermal changes. The amount of C=C bonds is proportional to the exothermic heat generated during cure and the inorganic compartments affect the heat diffusion within the material by their capacity to absorb external energy [30,31]. Thus, the matrix to filler ratio, the characteristic features of the dimethacrylate monomers, filler particles and pigments also play important roles in the temperature rise of RBCs [23,29].

Numerous types of dental RBC are available on the market with different matrix-filler components, consistencies and recommended application methods. Besides the conventional high-viscosity pastes and low-viscosity flowable RBCs, applied incrementally maximum in two-millimeter-thick layers, the so-called bulk-fill RBCs are increasingly popular among clinicians, since these are applicable in a thickness of 4–5 mm without layering. Although it was reported, that these bulk-fill materials show lower polymerization shrinkage, the adequacy of the depth of cure is still contentious [32–34]. The increased thickness of the RBC layer negatively influences light transmission and DC [35]. The thermal load on the pulp tissue is also controversial. A number of researchers found that bulk-fill RBCs generated a greater increase in pulpal temperature than conventional ones [36,37]. On the contrary, some of the investigations concluded that bulk-fill RBCs, especially high-viscosity pastes can reduce thermal load on the pulp chamber [28,38]. Although, particulate RBCs are durable restorative materials, one of the most frequent failures is the fracture of the restoration [39]. To overcome this mechanical limitation and enhance their physical properties, fiber reinforcement of conventional dental composites was introduced. The reinforcement was due to the stress transfer from the matrix to the short randomly oriented fibers which occurs upon loading leading to high fracture resistance [40,41]. While sculptable and flowable short-fibre reinforced resin composites (SFRC) are increasingly used materials in clinical practice, especially in deep cavities, according to our best knowledge, no data are available in the literature addressing the thermal effect of these materials on the pulp.

The purpose of the present study was to compare *in vitro* the thermal change in the pulp chamber during the polymerization of high- and low-viscosity conventional RBCs applied in 2 mm thickness, light-cured for 20 or 40 s and high- and low-viscosity bulk-fill and short-fibre reinforced RBCs placed in 2 and 4 mm layer thickness exposed to light for 20 s through a remaining dentine thickness of 2 mm. Further aims were to determine the delivered radiant exposure and related DC of the investigated materials and to assess the influence of layer thickness and exposure time on pulpal temperature rise and DC.

2. Materials and methods

2.1. *Resin composites and radiant exposure*

During this *in vitro* study six brands of RBCs – a conventional high-viscosity (HV) sculptable microhybrid, a low-viscosity (LW) flowable nanofill, HV and LV bulk-fill and HV and LV short-fibre reinforced RBC – were analyzed. The brands, chemical compositions and manufacturers are presented in Table 1.

Materials were tested in the layer thickness recommended by the manufacturer, thus conventional high-viscosity (Conv-HV-2 mm) and low-viscosity (Conv-LV-2 mm) RBCs in 2 mm layer thickness, high-viscosity (Bulk-HV-4 mm) and low-viscosity (Bulk-LV-4 mm) bulk-fill and SFRC (SFRC-HV-4 mm

Table 1 – Materials, manufacturers and composition.

Group	Material	Manufacturer	Shade	Organic matrix	Filler	Filler loading (vol%/wt%)
High viscosity conventional RBC	Filtek Z250	3M ESPE, St. Paul, MN, USA	A2	BisGMA, UDMA, BisEMA, TEGDMA	0.01–3.5 μm (mean 0.6) Zr-silica	60/82
Low viscosity conventional RBC	Filtek Supreme Flowable Restorative	3M ESPE, St. Paul, MN, USA	A2	BisGMA, TEGDMA, Procrylat resin	20–75 nm silica, 0.6–10 μm cluster Zr-silica, 0.1–5 μm YbF_3	46/65
High viscosity bulk-fill RBC	Filtek One Bulk Fill Restorative	3M ESPE, St. Paul, MN, USA	A2	AFM, UDMA, AUDMA, DDDMA	20 nm silica, 4–11 nm zirconia, cluster Zr-silica, 0.1 μm YbF3	58.5/76.5
Low viscosity bulk-fill RBC	Surefil SDR Flow+	Dentsply, Milford, DE, USA	U	Modified UDMA,TEGDMA, di -and trimethacrylate resins	4.2 μm Ba-Al-F-B silicate glass, Sr-Al-F silica, YbF	47.4/70.5
High viscosity short-fibre reinforced RBC	EverX Posterior	GC Europe, Leuven, Belgium	U	BisGMA, TEGDMA, PMMA	0.7 μm barium glass (65.2%), 17 μmx1-2 mm short E-glass fibers (9%)	53.6/74.2
Low viscosity short-fibre reinforced RBC	EverX Flow	GC Europe, Leuven, Belgium	U	BisEMA, UDMA	0.7 μm barium glass (45%), 6 × 140 μm short E-glass fibers (25%)	48/70

Abbreviations: RBC: resin based composite; U: universal; UDMA: urethane dimethacrylate; AUDMA: aromatic urethane dimethacrylate; AFM: addition fragmentation monomer; DDDMA: 1,12-dodecane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; BisEMA: bisphenol-A polyethylene glycol diether dimethacrylate; BisGMA: bisphenol-A diglycidil ether dimethacrylate; PMMA: polymethyl methacrylate; vol.%: volume%; wt.%: weight%.

Table 2 – Mean temperature change (ΔT), time to reach the maximum temperature (Tmax), returning time to the initial temperature of investigated resin-based composites (RBC) with standard deviations (S.D.).

Group of RBCs	Investigated RBCs	Curing time/Layer thickness	Mean ΔT (°C)(S.D.)	Mean time (s) to T_{max} (S.D.)	Mean return time (s) (S.D.)
Conventional RBC	Filtek Z250 (Conv-HV)	20 s/2 mm	5.2 (0.6)	28.2 (2.6)	107.2 (4.9)
		40 s/2 mm	8.5 (0.8)	49.8 (2.9)	282.6 (5.4)
	Filtek Supreme Flowable (Conv-LV)	20 s/2 mm	6.8 (0.4)	29.8 (1.9)	95.0 (4.6)
		40 s/2 mm	11.2 (1.0)	50.8 (3.6)	220.4 (5.1)
Bulk-fill RBC	Filtek One Bulk Fill Restorative (Bulk-HV)	20 s/2 mm	4.9 (0.3)	25.2 (2.3)	101.8 (2.4)
		20 s/4 mm	5.8 (0.6)	26.0 (2.6)	341.6 (6.2)
	SDR Flow+ (Bulk-LV)	20 s/2 mm	6.0 (1.0)	27.4 (2.9)	98.8 (5.3)
		20 s/4 mm	7.5 (0.8)	31.0 (2.2)	310.4 (7.4)
Short-fibre reinforced RBC	EverX Posterior (SFRC-HV)	20 s/2 mm	5.8 (0.5)	24.8 (2.8)	122.0 (6.1)
		20 s/4 mm	7.9 (0.8)	24.8 (3.1)	404.6 (5.3)
	EverX Flow (SFRC-LV)	20 s/2 mm	7.7 (0.7)	32.8 (2.2)	112.6 (4.9)
		20 s/4 mm	9.0 (1.2)	29.2 (2.3)	384.6 (7.6)

and SFRC-LV-4 mm) RBCs in 4 mm layer thickness. In addition, the last 4 RBCs were measured in 2 mm thickness as well (Bulk-HV-2 mm; Bulk-LV-2 mm; SFRC-HV-2 mm; SFRC-LV-2 mm). All materials were irradiated with the same Light Emitting Diode (LED) curing unit (LED.D, Woodpecker, Guilin, China; average light output given by the manufacturer 850–1000 mW/cm^2; λ = 420–480 nm; 8 mm exit diameter fiberglass light guide) in standard mode for 20 s according to the manufacturer's instruction, powered by a line cord at room temperature of 23 °C ± 1 °C, controlled by an air-conditioner. Additionally, to see the influence of exposure duration, samples of conventional RBCs were also polymerized with an extended exposure time of 40 s (Conv-HV-2 mm-40 s; Conv-LV-2 mm-40 s). The radiant exitance (mW/cm^2) and exposure (J/cm^2) delivered by the LCU operating in the standard mode were measured using a checkMARC radiometer (Bluelight Analytics, Halifax, Canada). Measurements were recorded in the above mentioned twelve groups, five times for each during the preparation of the specimens (n = 60). Regarding the type of RBC, layer thickness and exposure duration the investigated groups are presented in Table 2. The radiant exitance at the tip of the LCU was determined by placing the tip directly at a distance of 0 mm from

the radiometer sensor and the radiant exposure was calculated both at an exposure duration of 20 s and 40 s. This was repeated with the interposition of a black paper with a 6 mm diameter hole on it, to represent the irradiance delivered to the top of the materials. The light attenuation of the empty cylindrical polytetrafluoroethylene (PTFE) mold with an inner diameter of 6 mm, external diameter of 12 mm and height of 2 or 4 mm was also measured by placing the tip of the LCU directly over the mold. Interposition of a black paper with a 6 mm diameter hole between the mold and the sensor prevented light transmission through the mold to the checkMARC sensor. The radiant exposure values were calculated from the recorded radiant exitance both at a curing time of 20 s and 40 s. To estimate the radiant exitance and radiant exposure transmitted through each RBC during polymerization, the mold was positioned centrally on the sensor. Polyester (Mylar) strip was used to separate the sensor from the RBC which was filled in the mold. To avoid contact with oxygen the top of the RBC was protected with a Mylar strip before the polymerization.

2.2. *Micro-Raman spectroscopy measurement*

The same RBC samples made to estimate the radiant exposure transmitted through each RBC during polymerization were used to measure the DC. The samples were then placed in an incubator (Cultura, Ivoclar Vivadent, Schaan, Liechtenstein) at 37 ± 1 °C and 90% ± 10% relative humidity and stored in dark. The 24 h post-cure DC values of the polymerized RBC samples were examined using a Labram HR 800 Confocal Raman spectrometer (HORIBA Jobin Yvon S.A.S., Longjumeau Cedex, France). The following sets of parameters were applied during the micro-Raman measurements: 20 mW He-Ne laser with 632.817 nm wavelength, spatial resolution ~15 μm, magnification ×100 (Olympus UK Ltd., London, UK). Using a spectral resolution of ~2.5 cm^{-1}, satisfactory results were obtained since the two peaks analyzed were ~30 cm^{-1} apart. Spectra were taken both on the top and bottom surfaces of the RBC specimens at three random locations (central, peripheral region and area between) and with an integration time of 10 s. Ten acquisitions were averaged for each geometrical point. Spectra of unpolymerized RBC were taken as reference. Post-processing of spectra was performed using the dedicated software LabSpec 5.0 (HORIBA Jobin Yvon S.A.S., Longjumeau Cedex, France). The ratio of double-bond content of monomer to polymer in the RBC was calculated according to the following equation:

$$DC\% = \left(1 - \left(\frac{R_{cured}}{R_{uncured}}\right)\right) \times 100$$

where R is the ratio of peak intensities at 1639 cm^{-1} and 1609 cm^{-1} associated to the aliphatic and aromatic (unconjugated and conjugated) C=C bonds in cured and uncured RBCs, respectively.

2.3. *Sample preparation for pulpal temperature measurement*

A freshly extracted, caries-free, cleaned human third molar to be used in this study was kept wet in distilled water. A single tooth model was chosen for all experimental trials to limit any

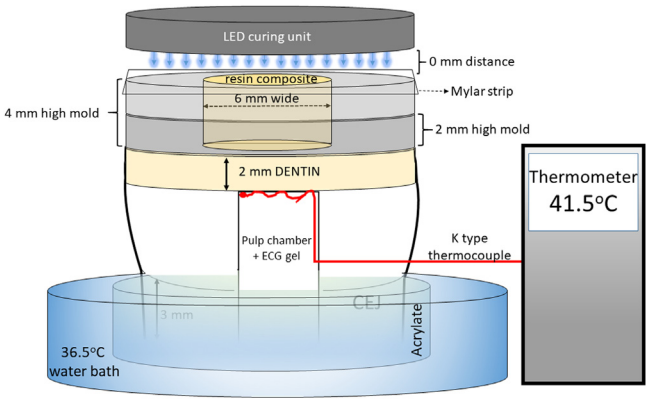

Fig. 1 – (OPTIONAL) Schematic figure of the experimental set-up for pulpal temperature measurements.

effects of structural and compositional differences in enamel and dentin. The occlusal surface was ground flat leaving a dentin thickness of two millimeters from the roof of the pulp chamber. The apices of the roots were sectioned 5 mm from the furcation to expose the root canals. All pulpal residues were removed with endodontic files. This was followed by 5.25% sodium-hypochlorite then saline irrigation and drying with paper points. A hole was created on the mesial side of the tooth with a one-millimeter diameter cylindrical diamond bur to allow the insertion of the 0.5 mm diameter Cu/CuNi thermocouple probes (K-type, TC Direct, Budapest, Hungary). The thermocouple sensor was fixed to the dentin on the top of the pulp chamber by means of a thin layer of cyanoacrylate glue (Loctite Super Glue, Loctite, Düsseldorf, Germany). The lateral hole was closed with flowable RBC (Filtek Supreme Flowable, 3M, St. Paul, MN, USA). The remaining dentin thickness and position of the thermocouple were assessed radiographically. Then to replicate the pulp tissue, the pulp chamber and root canals were injected with ECG gel (Aqua Sound Basic, Ultragel Hungary 2000, Budapest, Hungary). Flowable RBC was used to close apical orifices and the tooth was embedded in clear acrylic one millimeter below the cemento-enamel junction. The tooth was immersed in a water bath of 36.5 ± 0.5 °C.

Temperature measurements and heat registrations were recorded by a registration device (El-EnviroPad-TC, Lascar Electronics Ltd., Salisbury, UK) attached to the K-type thermocouple with a frequency of one measurement per second and resolution of 0.1 °C. The PTFE mold was stacked on top of the flat and polished occlusal surface of the tooth. First of all, the thermal effect of the light curing unit- both after a 20 and 40 s exposure- was measured through the empty 2- and 4-mm deep mold (Fig. 1). The temperature changes of each investigated RBCs were also measured: conventional RBCs applied in 2 mm thickness with an exposure time of 20 and 40 s, bulk-fill and SFRC RBCs applied in 2- and 4-mm thicknesses exposed for 20 s. As no dental adhesive system was used, the polymerized RBC could easily be removed from the mold without leaving any deposits on the dentin surface. This enabled the use of the same tooth for each measurement. Light curing was initiated when the temperature stabilized after the RBC application. Temperatures were recorded at the beginning of the irradiations and measurements continued until they returned

to the initial temperature. Temperature change was expressed as the difference between the recorded maximal temperature and the initial ambient temperature. Although, the temperature of the water bath apparatus was set to constant, the repetitive measurements found a temperature variation of 0.5 °C. Therefore, in order to reduce errors caused by environmental temperature changes, when calculating the results, the temperature change was used instead of the maximum temperature measurement.

The time to reach the maximum and the initial temperature after polymerization were also recorded. The procedure was repeated five times for each combination (n = 60).

2.4. *Statistical analysis*

The statistical analyses were performed with SPSS v. 26.0 (SPSS, Chicago, IL, USA). To test the normality of the distribution of the data the Kolmogorov–Smirnov test was applied, followed by a parametric statistical test. The differences in temperature change, time to reach the maximum, return time and in DC between the top and bottom of the investigated RBCs were compared with one-way analysis of variance (ANOVA). Tukey's post hoc adjustment was used for multiple comparison in all ANOVA models. Multivariate analysis (general linear model) and partial eta-squared statistics were used to test the influence and describe the relative effect size for *material* and *thickness* as independent factors. A two-tailed independent *t*-test was used to compare the difference between the temperature change through the empty 2 vs. 4 mm deep mold, irradiated for 20 vs. 40 s. P values below 0.05 were considered statistically significant.

3. Results

Based on measurements taken at three different locations on the radiograph (buccal-radial projection), the thickness of the remaining occlusal dentin was 2.1 ± 0.2 mm. The maximum radiant exitance of the LED LCU was 1170 ± 15 mW/cm^2. The delivered maximum incident radiant exposures with 20 and 40 s exposure duration were 23.4 ± 0.1 and 46.8 ± 0.2 J/cm^2, respectively. The radiant exitance was reduced to 930 mW/cm^2 by the 6 mm orifice, thus the radiant exposures with 20 and 40 s exposure durations were 18.6 and 37.2 J/cm^2, delivered to the top of the specimens. The 2–4 mm distance between the light guide tip and the radiometer sensor and, additionally, the limited orifice (6 mm in diameter) of the mold significantly decreased the radiant exposure. Through the empty 2 and 4 mm mold, the radiant exposure decreased by 42 % (13.5 J/cm^2 ± 0.1) and 63 % (8.7 J/cm^2 ± 0.1), respectively. The LCU increased the pulp temperature by 2.7 and 5.1 °C when the occlusal surface was irradiated through the 2 mm deep empty mold for 20 and 40 s, respectively. The difference was statistically significant ($t(8) = 11.7$, $p < 0.001$). Compared to the 2 mm deep mold (2.7 °C), the temperature increase was significantly lower ($t(8) = 4.4$, $p < 0.01$) through the 4 mm deep empty mold (1.9 °C) light cured for 20 s. The mean temperature changes of the investigated RBCs, the times it took to reach the maximum as well as return to the initial temperatures after polymerization are presented in Table 2.

Regarding the exothermic reaction, each RBC produced statistically significantly higher temperature increase in the pulp chamber than the LED unit alone. Subtracting the temperature rise caused by the LCU from the thermal change in the pulp chamber induced by the RBC gives an estimation of the heat generated by the exothermic reaction. According to this calculation – which does not account for the thermal transfer between the thermodynamic system and its environment –, the order of exothermic thermal changes through the 2 mm dentin thickness was the following: SFRC-LV-4 mm (6.3 °C) >SFRC-HV-4 mm (5.2 °C) >Bulk-LV-4 mm (4.8 °C) >Conv-LV-2 mm (4.1 °C) >Bulk-HV-4 mm (3.1 °C) >Conv-HV-2 mm (2.5 °C). The comparison of thermal change of high- and low-viscosity conventional, bulk-fill and SFRC RBCs with representative registration curves is shown in Fig. 2 (A, B).

In general, the low-viscosity RBCs showed statistically significantly ($p < 0.001$) higher mean temperature change during polymerization, irradiated for 20 s. Among the different groups of materials, the SFRC RBCs showed a significantly higher temperature change ($p < 0.001$). The flowable RBCs needed longer time to reach the maximum temperature, but it was statistically significant only for the bulk-fill RBCs. However, the time it took to return to the initial ambient temperature was significantly shorter ($p < 0.001$) for each low-viscosity RBC as compared to the condensable ones. The thermal changes of sculptable and flowable conventional RBCs irradiated for 20 and 40 s with representative temperature registration curves are reported in Fig. 3(A, B).

The extended exposure duration significantly increased the mean temperature change ($p < 0.01$) of the investigated conventional RBCs. Subtracting the thermal change caused by the LCU (5.1 °C – 40 s) alone, the exothermic temperature rise in the pulp chamber was estimated to be 3.4 °C for the Conv-HV-2 mm-40s and almost double, 6.1 °C for Conv-LV-2 mm-40 s. Although, the exposure duration was doubled, the time to reach the maximum temperature was not, however the time it took to return to baseline was more than two times longer compared to the samples polymerized for 20 s. Regarding the influence of layer thickness, a 1–2 °C higher temperature increase was measured in the case of thicker (4 mm vs. 2 mm) samples, which were significant for all types of investigated materials. The thermal changes of high and low viscosity bulk-fill and SFRC RBCs in 2- and 4-mm layer thicknesses with representative temperature registration curves are presented in Fig. 4(A–C).

The temperature values were predominantly influenced by *material* ($p < 0.001$) followed by *thickness* ($p < 0.001$) with partial eta-squared values of 0.74 and 0.49, respectively. The interaction of factors *material x thickness* did not show a statistically significant effect on the temperature change ($p = 0.16$) (partial eta-squared: 0.15). The time to reach the maximum temperature was not significant ($p > 0.05$) as a function of layer thicknesses, however, the time to return to baseline temperature was three times longer for the thicker samples, which was statistically significant for all groups of materials ($p < 0.001$). The temperature increase during 20s of polymerization was not consistent with the radiant exposures measured through the 2- and 4-mm thick samples.

The light attenuation was significant through the 2 mm (SFRC-LV-2 mm 54% <SFRC-HV-2 mm 56% <Bulk-LV-2 mm 73%

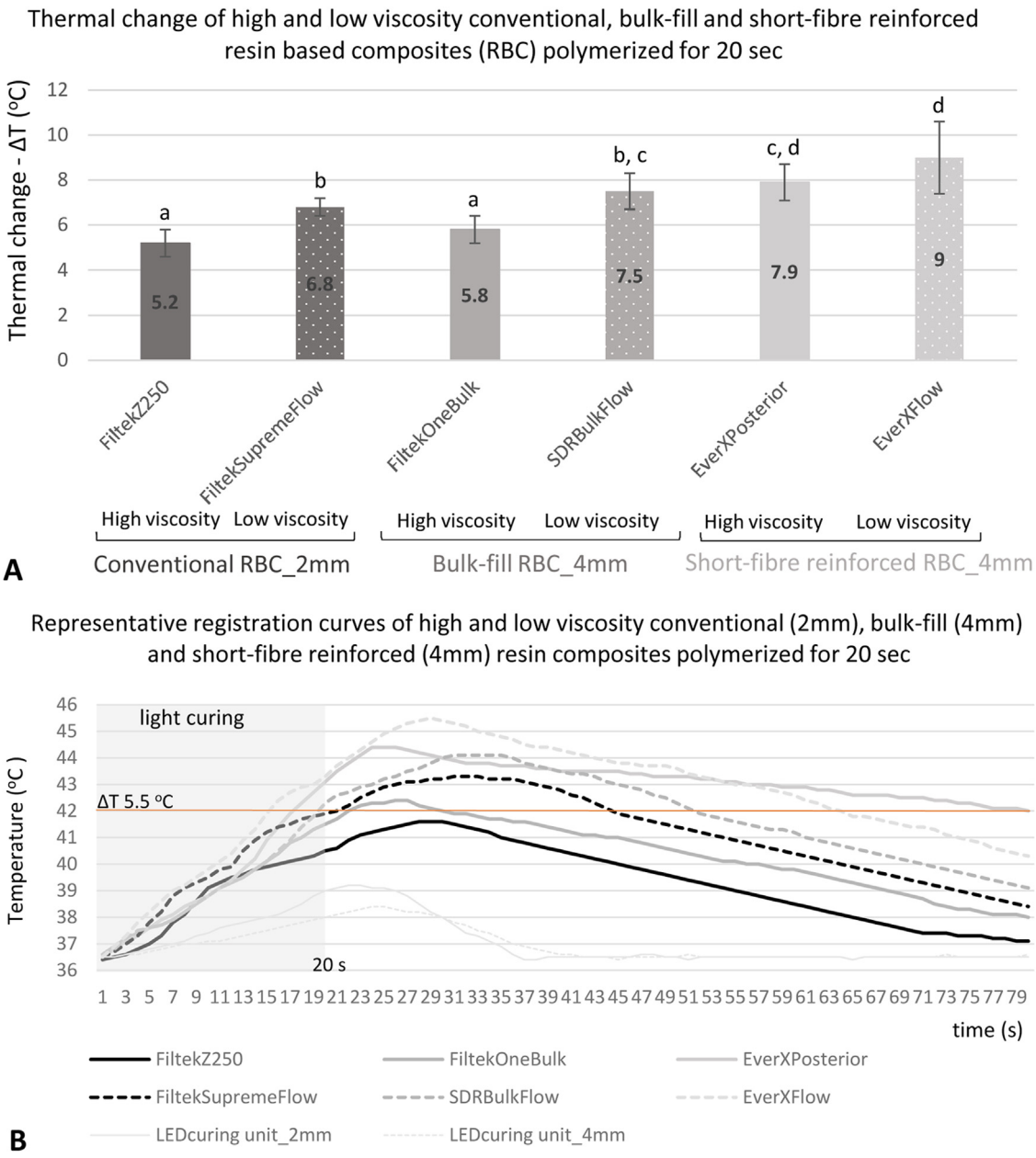

Fig. 2 – (A) Thermal change of high and low viscosity conventional, bulk-fill and short-fibre reinforced resin-based composites polymerized for 20 s. Distinct alphabetic indicates statistically significant difference between the materials. (B) Representative registration curves of high and low viscosity conventional (2 mm), bulk-fill (4 mm) and short-fibre reinforced (4 mm) resin-based composites polymerized for 20 s.

<Bulk-HV-2 mm 82% = Conv-LV-2 mm 82% <Conv-HV-2 mm 88%) and 4 mm (SFRC-LV-4 mm 68% <SFRC-HV-4 mm 71% <Bulk-LV-4 mm 89% <Bulk-HV-4 mm 92%) thick RBC samples. The mean delivered radiant exposures at the bottom of 2–4 mm thick RBC materials during polymerization are given in Table 3.

Regarding the DC at the top and bottom surfaces in samples applied in 2 mm thickness, percentages ranged between 66–75.4% and 60–72.6%, respectively. When samples were applied in a thickness of 4 mm the values ranged between 63–76% at the top and 52–69% at the bottom (Table 3). Conv-HV-2 mm, Bulk-HV-2 mm and all the 4 mm thick samples (Bulk-HV-4 mm, Bulk-LV-4 mm, SFRC-HV-4 mm, SFRC-LV-4 mm) showed statistically significant differences between the top and bottom DC values (Fig. 5 and 6).

The 2 mm thick low-viscosity RBCs (Conv-LV-2 mm, Bulk-LV-2 mm, SFRC-LV-2 mm) and SFRC-HV-2 mm provided similarly high DC values on the top as at the bottom. The highest DC at the top was achieved in the SFRC samples regardless of the sample thickness, followed by the low-viscosity conventional and bulk-fill RBCs applied in 2- and 4-mm thicknesses. The lowest degree of polymerization at the top was mea-

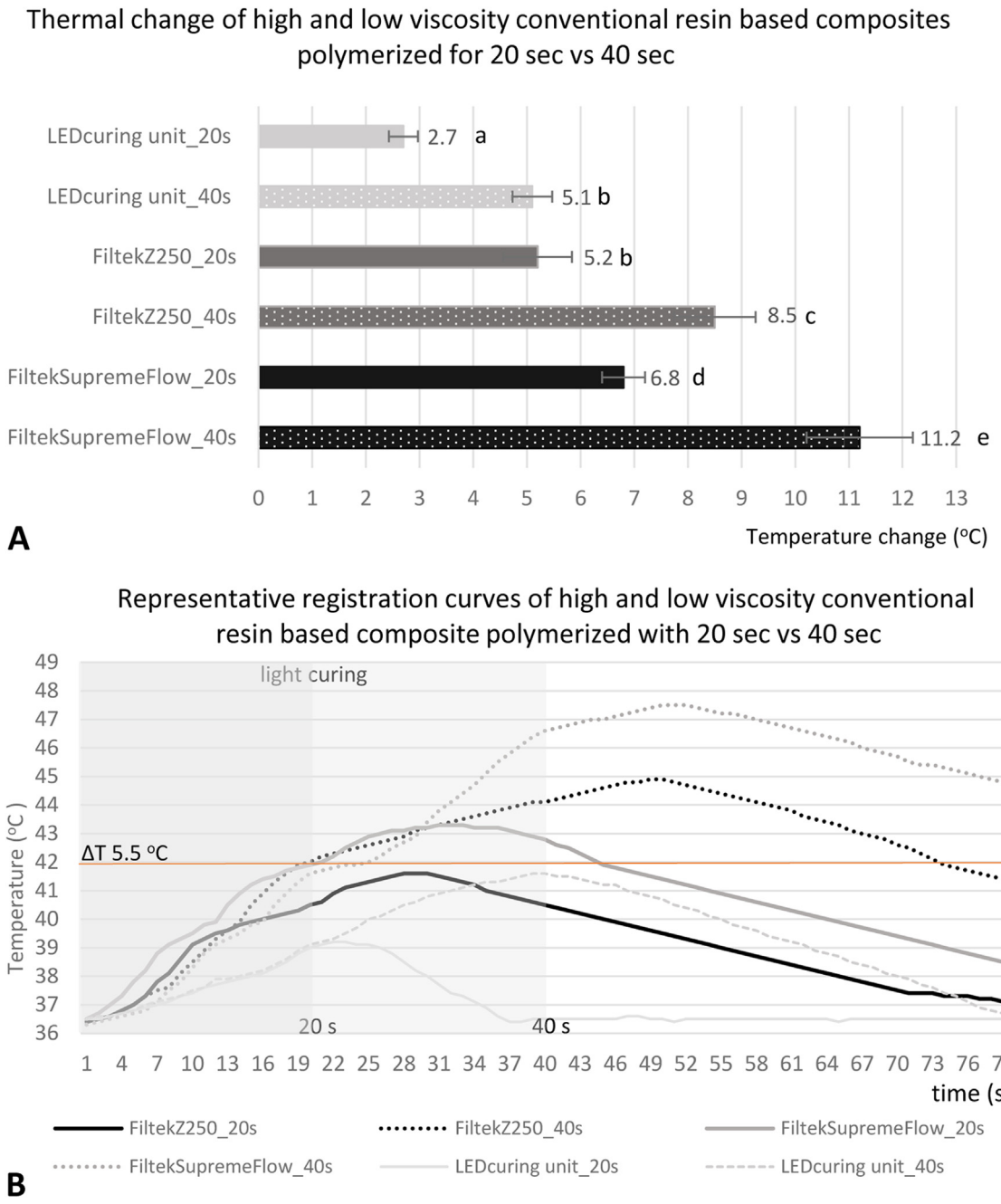

Fig. 3 – (A) Thermal change of high and low viscosity conventional resin-based composites polymerized for 20 s vs. 40 s. Distinct alphabetic indicates statistically significant difference between the materials. (B) Representative registration curves of high and low viscosity conventional resin-based composite polymerized with 20 s vs. 40 s.

sured in the high-viscosity conventional and bulk-fill RBCs. The results regarding the DC at the bottom surfaces, are consistent with the decreased radiant exposure delivered through the 4 mm thick specimens. The lowest DC was achieved by the Bulk-HV-4 mm, followed by the Conv-HV-2 mm and Bulk-HV-2 mm. Significantly higher DC values were detected in Bulk-LV-4 mm and SFRC-HV-4 mm samples. The highest DCs at the bottom were measured in all the 2 mm thick flowable materials (Conv-LV-2 mm, Bulk-LV-2 mm, SFRC-LV-2 mm) and in SFRC-LV-4 mm samples. The effect size of factor *material* is significant ($p < 0.001$) (partial eta-squared is 0.94) and higher on the DC than the effect size of factor *thickness* (partial eta square is 0.79), which was also found to be a strong and statistically significant effect ($p < 0.001$). The interaction between the two variables, *material x thickness,* significantly influenced the DC values ($p = 0.001$) with an effect size of 0.35.

4. Discussion

In this *in vitro* study the influence of the polymerization of high- and low-viscosity conventional, bulk-fill and SFRC RBCs

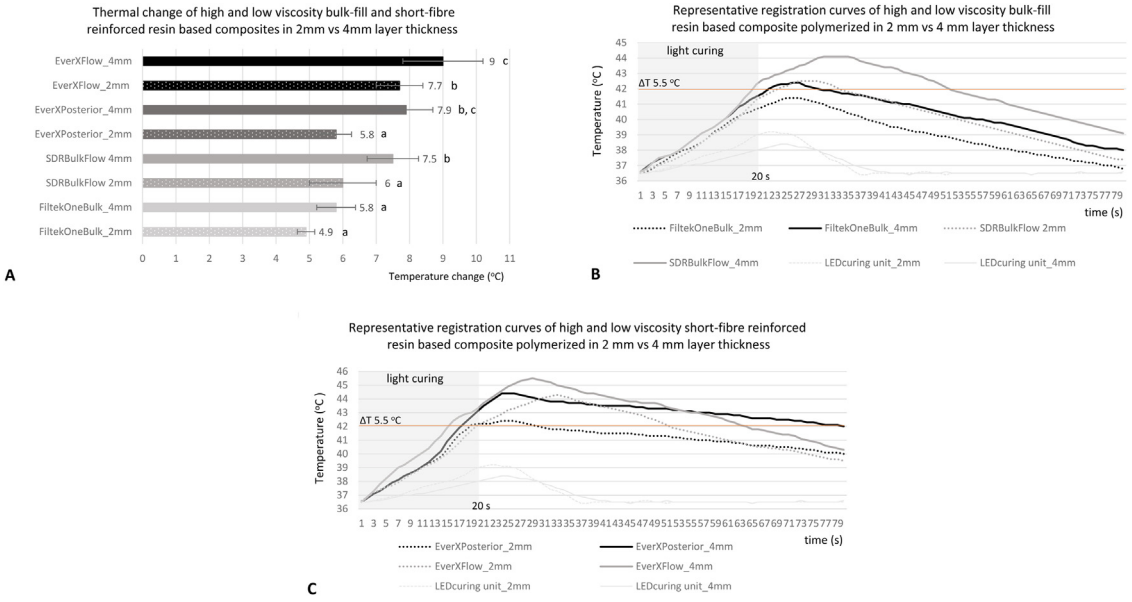

Fig. 4 – (A) Thermal change of high and low viscosity bulk-fill and short-fibre reinforced resin-based composites in 2 mm vs. 4 mm layer thickness. Distinct alphabetic indicates statistically significant difference between the materials. (B) Representative registration curves of high and low viscosity bulk-fill resin-based composite polymerized in 2 mm vs. 4 mm layer thickness.
(C) Representative registration curves of high and low viscosity short-fibre reinforced resin-based composite polymerized in 2 mm vs. 4 mm layer thickness.

Table 3 – Top and bottom degree of conversion of the investigated materials and radiant energy delivered to the bottom of 2 and 4 mm thick specimens.

Resin-based composite (RBC)	Exposure duration	Layer thickness	Mean radiant energy (J/cm^2)(S.D.)	Degree of conversion (DC) (%)					
				Top	Bottom	ΔDC	95% CI		p-value[a]
							Lower	Upper	
Filtek Z250	20 s	2 mm	1.6 (0.1)	66	60.4	5.6	−7.87	−3.72	<0.001
	40 s	2 mm	3.2 (0.2)	69	67	2	−3.36	−0.03	0.04
Filtek Supreme Flowable	20 s	2 mm	2.4 (0.06)	72	70	2	−4.09	0.98	0.19
	40 s	2 mm	4.7 (0.1)	77	74	3	−6.18	0.18	0.06
Filtek One Bulk Fill Restorative	20 s	2 mm	2.4 (0.1)	66	60	6	−8.72	−3.61	<0.001
		4 mm	0.7 (0.05)	63	52	11	−13.57	−8.47	<0.001
SDR Flow+	20 s	2 mm	3.6 (0.07)	70	69	1	−3.16	0.96	0.25
		4 mm	1.0 (0.08)	70	65	5	−5.11	−3.97	<0.001
EverX Posterior	20 s	2 mm	5.9 (0.05)	75.4	72.6	2.8	−5.88	0.29	0.07
		4 mm	2.5 (0.08)	73	68	5	−6.87	−3.16	<0.001
EverX Flow	20 s	2 mm	6.2 (0.05)	75	72	3	−4.84	0.68	0.12
		4 mm	2.8 (0.1)	76	69	7	−10.95	−3.01	<0.001

[a] One-way analysis of variance (ANOVA) and Tukey's post hoc adjustment.

on thermal change in the pulp chamber was investigated with additional measurement of the DC and delivered energy density on the top and bottom surfaces of the samples.

The study was carried out on a representative permanent molar tooth without the use of an adhesive system for all experimental groups to provide the same tooth-related conditions for all the measurements and eliminate any effect which may arise from structural and optical differences of the enamel and dentin. The remaining dentin thickness between the investigated materials and the pulp chamber in the present investigation was 2 mm. Although, dentin has a relatively low thermal conductivity, the potential for pulpal damage is expected to be greater in deep cavities, where the tubular surface area increases and the light attenuation effect is lower [42–44]. Aside from the distance between the floor of the cavity and the pulp, the perfusion rate and effect of the pulpal blood circulation, the volume and motion of the fluid in the dentinal tubules as well as the surrounding periodontal

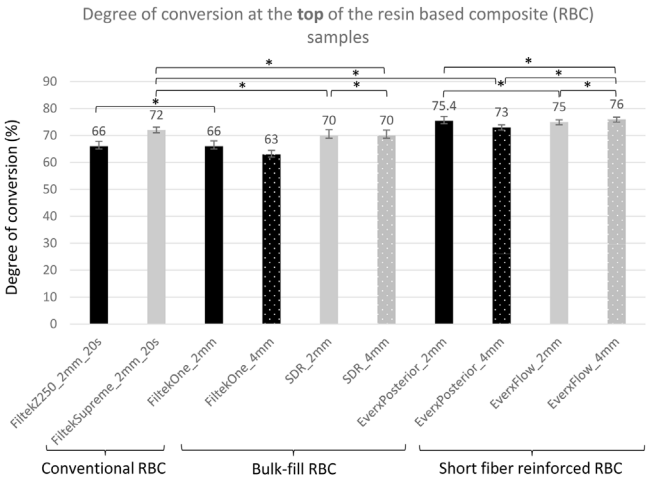

Fig. 5 – Degree of conversion at the top of the investigated materials. The * mark indicates statistically significant difference between the investigated materials.

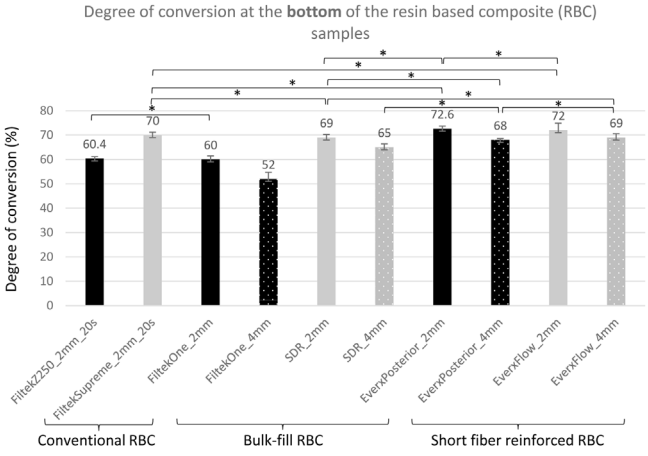

Fig. 6 – Degree of conversion at the bottom of the investigated materials. The * mark indicates statistically significant difference between the investigated materials.

tissues also play important roles in heat conduction and protection against the rise of pulpal temperature [45]. According to a simulation study on pulpal microcirculation conducted by Kodonas et al., the application of a thermal stimulus significantly influences temperature rise in the pulp chamber. Results showed a two-four times higher temperature increase in the pulp without water perfusion [46]. As a limitation of our study, the experimental design lacked a simulation of the microcirculation. Although the tooth was immersed in 36.5 ± 0.5 °C water bath and held at this temperature for the duration of the study, this physiological temperature is not able to account for all the mechanisms by which heat is dissipated *in vivo*. The reason why the authors employed such a study design was to illustrate the temperature changes which arise specifically due to the extent of the exothermic reaction occurring in the investigated materials.

In the present study, a second generation LED LCU was used to initiate polymerization with a radiant exitance of 1170 ± 15 mW/cm^2 at a wavelength range of 420–480 nm. Valid power measurements were made by a calibrated portable radiometer, namely checkMARC [47], however the beam profile and surface area of the active light tip were not analysed. This is a limitation of our experimental design, since it was demonstrated, that most of the LCUs – especially low budget LCUs, like LED.D – exhibited highly non-homogeneous light beam profiles and could have very different light output characteristics [48,49]. Although, this could affect pulpal temperature rise and influence, especially, DC – even at different locations of the top and bottom surfaces –, the same unit in standardized conditions was used to polymerize each material in the present study and thus enables the comparison of the results. The positioning of the light guide tip was also standardized, ensuring each sample received the same light beam character. Furthermore, the curing unit powered by a line cord provided a constant light output level.

The LCU increased the pulpal temperature to a different extent through the empty 2 and 4 mm deep mold (6 mm in diameter), irradiated for 20 and 40 s, however, the thermal changes (1.9–5.1 °C) did not exceed the pathological threshold considered to be 5.5 °C according to Zach and Cohen findings [14]. Pohto and Scheinin also reported that the critical temperature for reversible pulp damage was between 42 °C and 42.5 °C [50]. However, Baldissara et al. did not find an average increase of 11.2 °C to affect the pulp significantly [18]. Despite the attempts to develop reliable methodologies to simulate *in vivo* condition, a wide range from 1.5 to 23.2 °C temperature rise in the pulp chamber was reported *in vitro* during light exposure [51]. According to several investigations, the intensity and duration of the applied light seemed to be the most crucial factor in pulpal temperature rise [15,16,20,46,52,53]. Present results confirm this statement. As demonstrated on conventional RBCs an extended exposure duration (40 s) resulting in a doubled power density induced 60% higher temperature rise in the pulp chamber compared to an irradiation time of merely 20 s. In spite of the importance attributed to the curing light as a possible heat source, it is important to emphasise, that the light is attenuated with distance, and the diameter of the cavity orifice also limits the entrance of the photons which serve not just heat, but also as electromagnetic energy for the process of polymerization [51]. Thus, the strength of the light output used in photopolymerization carries a dual importance: on one hand, it should be high enough to be able to activate sufficient free radicals to ensure a clinically acceptable rate of polymerization, however not too high as to produce heat damage in the pulp. Our results showed that the radiant exposure of the LCU delivered through the 2 and 4 mm deep empty mold with 6 mm orifice diameter was decreased by 42% and 63%, respectively, with an exposure duration of 20 s. The related pulpal temperature increase through the 2 mm remaining dentin was 1.9 and 2.7 °C, respectively. However, the heat generated together with the exothermic reaction of the RBCs and conducted to the pulp chamber was high enough to reach or exceed the critical temperature at which pulpal damage begins in almost all the materials. This is in contrast to previous studies which showed that the heat produced in an irradiated empty cavity was always higher when compared to the polymerization of a 2 mm thick composite increment. The authors attributed their results to the restorative material's attenuating capacity against the exothermal effect of the

polymerization [15,54]. Other investigations however showed that the biggest risk to pulp health occurs during photopolymerization, although, the thermal effect of the curing unit and the material dependent exothermic heat production is not separated [37,53].

The maximum temperature values measured ranged between 41.4 and 47.7 °C in our study. These data are comparable to the results of other studies, where the geometry of RBCs are similar to that of our specimens [16]. In the present research, the same LED unit with 20 s duration was used to polymerize the different types of investigated materials, thus the same energy density was delivered on the top of each specimen. This study design together with the single tooth model [23] provided uniform conditions allowing to compare the exothermic temperature rise between different RBCs. Thus, the exothermic reaction is proportional to the amount of resin available for polymerization [29,55,56]. According to this statement, because of their higher resin content flowable RBCs should exhibit a higher temperature rise than non-flowable materials. In line with Baroudi et al. and Akarsu et al., our findings also confirmed that flowable RBCs polymerize with a more intense exothermic reaction [23,38]. Our results however showed higher temperature rises (5.2–11.2 °C), even with a remaining dentin thickness of 2 mm. The reason for such a difference may lie in the study design. Blood circulation was simulated in the above-mentioned studies, while is missing from our methodology. Preparing uniform 2 mm thick samples from all tested RBCs provided results which were comparable and made it possible to examine the effect of the variable composition of conventional, bulk-fill and SFRC RBCs - for the same viscosity - on pulpal temperature rise. These results showed that the detected differences were significant only between bulk-fill and SFRC RBCs in both viscosities while the conventional ones produced similar temperature changes to that of bulk and SFRC RBCs. The calculation of the partial eta-squared values pertaining to factor *material* also supported the significant influence of RBC composition on temperature change.

Regarding layer thickness, statistically significant temperature rises in 4 mm compared to 2 mm-thick samples were detected in the bulk-fill and SFRC materials. Yasa et al. also found higher temperature rise in case of the bulk-fill samples used in 4 mm, compared to the conventional RBC which was applied in two increments of 2 mm layers [37]. Exothermic differences may arise due to the presence of higher amounts of monomers in a 4 mm thick material. Although, it has been reported that increased filler content can decrease the temperature rise [57], the chemically inert fillers are capable of absorbing external energy, thus may play an indirect role in the temperature rise [58]. Moreover, heat generated within a viscous resin, becomes increasingly difficult to remove with increasing volume of the resin material [59]. Although, our results indicated that higher glass-fiber content increases temperature rise, in contrast to our investigation Ilday et al. did not find the addition of glass-fibers to influence temperature rise. [60]. Comparing the 2 mm thick conventional low viscous RBC to the 2 and 4 mm thick low viscous bulk-fill, the results showed insignificant differences compared to our results as mentioned above. The effect size of *thickness* on the thermal change of RBCs during polymerization was found to be significant, however it had a weaker effect compared to the *material* factor. This is in contrast with Par et al., who suggested that the temperature rise was mostly determined by the variations of radiant energy delivered to the bottom of the specimens, hence the curing unit type and layer thickness played more important roles than the material itself [61]. Differences may have also resulted from the different experimental conditions, like the study design for the temperature measurement, from the type of the LCU used, exposure time applied, the type and shade of the investigated RBCs, the dimensions of the specimens, the color of the Teflon mold, etc.

Not only the dentin thickness, but differences in surface-to-volume ratios of various resin materials could account for the differences in the efficiency of heat dissipation. This might be more important than the peak temperature within the resin [62].

Our results demonstrated that the temperature continues to rise almost linearly while the light is on reaching the peak 8–12 s after the exposure was finished. Other authors found the temperature rise to occur as soon as the light source was activated and the peak temperature time to be mainly dependent on the dentin thickness left above the pulp chamber [55]. Runnacles et al. observed similar dynamics in an *in vivo* study design too, where ΔT, as well as peak temperature increases were continued to be detected for approximately 10 s after teeth were exposed to the light [63]. This was explained by the heat-storing capacity of dentin [64] which resulted in a gradual dissipation of thermal energy towards the pulp, leading to a sustained temperature rise in the pulp chamber even after the curing light was shut off. Additionally, as it was discussed above, the amount of inert filler particles, especially glass fibers, could have also stored external (curing light) and internal (exothermic) heat energy [58]. This factor was supported by our present findings, since higher filler loading resulted in a longer mean time required to return to the initial pulpal temperature. In addition to the influencing effect of dentin thickness and the properties of the investigated material, the study design also plays a significant role on heat dissipation which makes it difficult to compare different research findings.

Pohto and Scheinin noted that the duration of the thermal irritation is another factor in pulpal damage, since 2 min at 46 °C can cause an arrest of blood circulation [50]. According to our measurements, the time it took for the temperature to return to the initial ranged between 95.0–404.6 s. Even though the maximum temperature detected in high-viscosity materials was lower than in low-viscosity RBCs, they took more time to cool down. The pulpal temperature however decreased below 42 °C within 80 s in all cases, even without simulation of the pulpal circulation.

Regarding the radiant energy measured at the bottom of the samples, values were 54–92% lower than the incident radiant energy measured at the top of the 2- and 4-mm thick RBC samples. The light attenuation was strongly influenced by the material composition, shade and thickness. Previous studies have shown that factors such as polymeric matrix refractive index, monomer type, filler type, loading and size can influence light transmittance in RBCs [65,66]. Higher filler content and thicker samples decreased radiant energy radically, while a more translucent shade and higher glass-fiber content pro-

vided for a higher radiant energy level at the bottom of the 4 mm thick samples as compared to the 2 mm thick, A2 shaded materials. This was in line with Garoushi et al., who also found more translucent bulk-fills and short fiber-reinforced RBCs with bulk shade (universal shade) to allow for a higher light transmission compared to the conventional resin composites [35]. Although, the filler ratio of Filtek Supreme Flowable is the lowest among the tested RBCs, the light attenuation through the 2 mm thick sample was 82%. In spite the fact that the more pigmented A2 shade of this material and the quality of the filler particles (Zr-silica clusters) may exhibit higher light distribution within the material, the monomer to polymer conversion still reached a high degree. It is well-documented, that radiant exposure undoubtedly influences the DC [1,5], however, the polymerization kinetics have been found to be highly complex, and irradiance, exposure and composition can independently affect the DC [58,67]. Bucuta and Ilie found micro hardness to be above 80% when the energy measured at the bottom of the samples was larger than 0.7 J/cm$^{2.}$ While the increase was proportional to the amount of transmitted light, they also found the filler content to have a higher effect on the results compared to the transmitted irradiance [11]. Our results demonstrated the same tendency for DC - which has a strong correlation with hardness [68] -, since the lowest DC was measured with mean radiant energy of 0.7 J/cm^2 (Filtek One Bulk RBC in 4 mm thickness) and rose with increasing light transmittance. According to Shortall et al.'s findings, individual products require different levels of radiant exposure to provide optimal properties [25].

The degree of conversion at the top and bottom surfaces of the different RBCs were assessed using micro-Raman spectroscopy which allows the direct detection of the amount of unreacted C=C in the resin matrix [58]. Raman-spectra were taken after 24 h, since significant increase in DC takes place even after the removal of the irradiation source, up to a maximum of 24 h post-irradiation [69]. Regarding conventional RBCs, the 2 mm thick flowable sample reached a high degree of conversion without significant difference between the top and bottom surfaces irrespective of whether the recommended or doubled exposure time was applied. This RBC has the lowest filler content among the investigated materials. Halvorson et al. demonstrated, that increasing the filler ratio progressively decreases conversion, because an increased amount of filler particles is an obstacle to polymer propagation [70]. Similar DC values were detected in the 2 mm thick low viscous bulk-fill and the low and high viscous SFRCs. Light penetration through the 2 mm SFRC samples was higher, which may be explained by the more translucent glass fillers and fibers. SFRC materials (EverX Posterior and Flow) are unique among the others containing randomly oriented short glass fibers in mm scale. The high translucency of the glass fibers may have increased light penetration in deeper regions resulting in a higher degree of conversion. This is in line with the findings of Garoushi et al. and Goracci et al. [35,71]. Although, the short glass fiber content increased to 25% in the flowable EverX compared to the 9% of the high viscous EverX Posterior, the DC and the light transmittance did not increase significantly. The temperature rise however was more pronounced [72]. Considering the monomer content of the investigated RBCs, it may also be assumed, that the higher DC values are attributed to the TEGDMA monomer, which is a component of all the tested low-viscosity RBCs and the high-viscosity SFRC. Besides decreasing viscosity, the low molecular weight of TEGDMA as well as the ether (C—O—C) linkage, providing a slight rotation around the bond, allows for more covalent bonds to be created during the polymerization [73].

In contrast to the results mentioned above, significant differences were found between the top and bottom DCs in cases of the 2 mm thick high viscosity conventional and bulk-fill RBCs and in all the 4 mm thick samples of high- and low viscosity bulk-fill and SFRC materials. While, the DC values at the bottom surfaces of the 4 mm thick samples failed to reach the values measured on the top, the SFRC RBCs reached or exceeded the results found at the bottom of the 2 mm thick conventional highly viscous RBCs cured for 20 s and even 40 s. The consequence of the lower rate of light attenuation in SFRCs was seen in the higher DC values, while the high grade of photon-attenuating capacity of the high viscosity conventional and bulk-fill RBCs is also clearly reflected in the DC results. The higher amount of particulate filler content of the highly viscous conventional (Filtek Z250) and bulk-fill (Filtek One Bulk Fill) RBCs explains the reduced light transmittance and the consequential lower DC at the bottom of the samples [65,69]. In addition, the incorporation of the fillers can restrict the mobility of the monomers and radicals, leading to a decreased conversion [73]. Lower conversion, may compromise the mechanical properties and the biocompatibility of the RBC, leading to lower values of hardness, early degradation of the RBC and release of free monomers, which – together with the thermal load - can further increase the hazard of pulpal damage [32,34,74]. Although, the minimum DC for a clinically satisfactory restoration has not yet been established, negative correlation with *in vivo* abrasive wear depth was found for DC values in the range of 55–65% [75,76]. As a flowable bulk-fill RBC, universal-shade of Surefil SDR Flow+ was investigated in the present study, which is an improved version of the well-known Surefil SDR Flow. It was developed to increase the mechanical properties, wear resistance and radiopacity [77]. Several researches proved the high conversion degree of the universal-shade Surefil SDR Flow, even in an eight-mm deep cavity, where the DC reached 63% after an exposure time of 20 s without a significant difference between the top and bottom surfaces [58]. However, our results demonstrated a lower DC% at the bottom in the newly developed SDR Flow+, even though the sample was irradiated from a distance of zero between the top and light guide tip. SDR Flow+ contains 2.5% more filler content with radiopacifiers, as well as a modified monomer composition, when compared to the initially developed Surefil SDR Flow. This may contribute to the lower degree of conversion.

The lowest degree of conversion (52%) was measured in the high-viscosity bulk-fill RBC (Filtek One Bulk Fill). The explanation for this cannot just be found in the higher particulate filler load and the presence of Zr-silica clusters, but may also be explained by the content of addition fragmentation monomers (AFM), incorporated into the resin matrix as a stress reliever.

Shah et al. demonstrated, that the incorporation of AFM may lead to a gradual reduction in polymerization kinetics along with a steady decrease of the reaction rate resulting in a lower conversion of the RBC [78]. The DC-reducing power of

this monomer is significant above 5 wt.%, however the exact content of AFM is a trade secret.

This study has some limitations such as the removal of the enamel and dentin walls, lack of application of an adhesive layer, and measurement of the intrapulpal temperature from a single point without simulation of the blood microcirculation. In the presence of axial enamel and dentin walls, the intrapulpal temperature increase may be more restrained due to heat dissipation. Alternatively, due to the heat-storing capacity of the dentin on top of the pulp chamber, there may be a gradual dissipation of thermal energy toward the pulp, leading to a sustained higher temperature in the pulp chamber [64], as observed in our study. Thus, the importance of the remaining dentin thickness above the pulp chamber is twofold: when it is decreased, both the insulating effect and heat-storing capacity are decreased.

In addition, the polymerized adhesive layer may serve as a barrier to heat transfer under clinical conditions. However, one must consider that during polymerization of the adhesive layer, it was found that the pulp temperature increased faster than during the photocuring of the RBC, especially with an irradiance higher than 1000 mW/cm^2 [79]. Based on this observation, an important problem may arise from the immediate application of the RBC without first allowing for heat to dissipate. Heat generated by the irradiation of the RBC could potentially compound intrapulpal thermal damage as the pulpal and dentine temperatures are already higher due to the adhesive polymerization.

Moreover, the simulation of blood circulation can decrease the intrapulpal temperature *in vitro* as it may happen *in vivo*. Although, heat dissipation is strongly influenced by blood circulation, Runnacles et al. concluded, that the pulpal temperature increase values measured *in vitro* were comparable to the *in vivo* values when clinically relevant light exposure modes were compared [17]. Bone simulation experiments investigating drilling-induced heat generation have also found blood circulation to have a lower impact on temperature accumulation [80,81].

Regarding the layer thickness, as a limitation of this study, conventional RBCs were not tested in a thickness of 4 mm. Although, it would have provided more data to estimate the effect size of the investigated variables, conventional RBCs are recommended to be applied in a maximum thickness of 2 mm- as per manufacturer's instructions- thus the authors considered testing them in 4 mm thickness not to be relevant.

Although, bulk-fill RBCs can achieve a higher depth of cure than conventional RBCs, the depth of cure is not always clinically acceptable at a thickness of 4 mm when exposed as per manufacturer's instructions (i.e. 10–20 s) [82]. As a limitation it should be mentioned that the present study investigated the DCs and PTs changes of conventional RBCs with an extended exposure time, while bulk-fill and SFRC RBCs were tested only with an exposure duration of 20 s. Several investigations demonstrated a higher DC of bulk-fills and SFRCs with an extended irradiation time [82,83]. Its effect pertaining to bulk-fills however is strongly material dependent [10,58]. Even though increased irradiation time is proven to be beneficial for mechanical properties [10], it may also increase the pulpal temperature thereby compromising the health of the pulp tissue. Further investigations are required to clarify this assumption.

Lastly, the present study did not evaluate the effects of the different, clinically more relevant cavity designs (depth, orifice diameter, remaining dentin thickness) which may also significantly impact delivered energy density, DC and pulpal temperature.

5. Conclusion

Within the limitations of this *in vitro* study, the following conclusions can be stated:

1) The temperature and DC values were predominantly influenced by the composition of the material followed by the thickness.
2) The low-viscosity RBCs and the materials used in 4 mm thickness increased the pulpal temperature by a significantly higher extent. Among the different groups of materials, the more translucent SFRC RBCs showed significantly higher temperature change.
3) Significantly higher DC levels were measured at the top of the samples as compared to the bottom in the 2 mm thick high-viscosity conventional and bulk-fill RBCs and in each 4 mm thick high- and low-viscosity materials. Among the different groups of materials, the conventional flowable and the SFRC RBCs achieved higher DC levels.
4) Doubling the exposure time increased significantly both the intrapulpal temperature as well as DC values of the investigated conventional RBCs.
5) Higher DC values are associated with significant increase in pulpal temperature, which should be considered by the clinicians when they want to achieve higher DC% for better clinical performance of the restoration.

Acknowledgments

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