

POLYMERISATION KINETICS IN A FIBRE REINFORCED RESIN-BASED COMPOSITENicoleta Ilie^{1a*}¹Department of Operative Dentistry and Periodontology, University Hospital, Ludwig-Maximilians-Universität München, DE-80336 Munich, Germany^aDipl. Eng, PhD, ProfessorReceived: July 14, 2017
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Cite this article:Ilie N. Polymerisation kinetics in a fibre reinforced resin-based composite. *Stoma Edu J.* 2017;4(3):164-170.**ABSTRACT**

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Objective: The study aimed to evaluate the potential of a commercial fiber reinforced resin composite (FRC) to be cured adequately also in increments larger than 2-mm.**Material and methods:** One FRC (EverX Posterior, GC) was investigated by assessing in real-time the degree of conversion (DC) and polymerisation kinetic at increasing depths (100- μ m, 2-mm 4-mm and 6-mm). In addition, a battery of mechanical properties - flexural strength, flexural modulus, Vickers hardness, indentation modulus, creep - and the characteristics of the used curing light were determined.**Results:** One-way ANOVA revealed no significant difference in DC measured 300 s post-irradiation in a depth of 100- μ m and 2-mm ($p = 0.281$). Similarly, no significant difference was identified between DC measured at 2-mm and 4-mm ($p = 0.724$), while the DC measured at 6-mm depth was significantly lowest ($p < 0.001$). The polymerisation kinetic was well described ($R^2 > 0.95$) by a double exponential sum function, distinguishing between the gel and the glass phase of the polymerisation process. It allowed identifying a slower start of polymerization in depth, associated with a lower maximal rate of C-C double bond conversion. The mechanical properties amounted (128.30 \pm 8.38) MPa (flexural strength), (8.38 \pm 0.87) GPa (flexural modulus), (92.00 \pm 15.86) N/mm² (Vickers hardness), (17.82 \pm 1.82) GPa (indentation modulus) and (3.35 \pm 0.84) % for creep.**Conclusions:** While DC recorded in 2-mm and 4-mm depths were statistically similar, there is evidence that the quality of curing in a depth of 4-mm is lower compared to the top of the specimen. The mechanical properties were within the range of high viscosity bulk-fill resin-composites.**Keywords:** fiber reinforced resin-based composite, degree of cure, polymerisation kinetics, bulk-fill, mechanical properties.**1. Introduction**

Fiber-reinforced resin-based dental composites (FRC) are advanced restoratives, particularly designed to be placed in load bearing areas. The denomination FRC implies that the material is composed of dissimilar constituents, involving a homogeneous polymer matrix that is reinforced by a stronger and stiffer fibrous constituent. The shape, dimension, orientation and volume amount of fibers are used to modulate the mechanical properties of the FRC.

The shape of fibers is usually described by its aspect ratio R , and is defined as the proportional relationship between their length and width. To achieve a high material strength, a high aspect ratio is sought. In dental materials, an optimum was estimated at ca. 5.2,¹ thus a fiber must be 5.2 time longer as it is wide. In addition to the aspect ratio, the strength of a FRC is directly related to the length of the reinforcing fibers. The critical fiber length in dental FRC was shown to lie between 0.5

mm and 1.6 mm,² while fibers below these values are inducing a lower reinforcement effect and are considered to act similarly to fillers present in particulate micro hybrid resin-composites.³ A third aspect influencing the strength of a FRC is the fiber orientation that can be unidirectional or randomly distributed. In continuous, unidirectional fiber composites, high strength and stiffness is found in the fiber longitudinal axis with very minor changes from the matrix properties in the transverse direction. In contrast, the material behaves macroscopically homogenous and isotropic in randomly orientated, discontinuous FRC.⁴ It must however be considered that, as demonstrated by Karbhari and Strassler, a dental FRC having the highest strength does not necessarily have the highest flexural stiffness or the greatest capacity for energy absorption,⁵ thus the mechanical behavior of this particular material category requires a battery of different tests to be understood.

Beside improvements in mechanical properties

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by integrating fibers as a reinforcing phase in resin-composites, their use as direct restorative materials underlie modern requests for fast and save application. This implies that a cavity may be restored in larger increments (> 2 mm), helping thus to reduce both the chair time and the risk to insert defects or contaminants between layers, in comparison to an incremental placement technique with conventional resin composites.⁵

Currently, a FRC that is indicated to restore large posterior cavities, owing to its enhanced mechanical properties, is also conferred by the manufacturer right to use in larger increments (up to 4 or 5 mm) similarly to a bulk-fill resin composite. Whether a resin-composite is adequately cured in its deeper layers is essentially influenced by the amount of photons reaching these areas during polymerization, and consequently by the translucency of the material. It has been shown that bulk-fill resin composites may become either progressively opaquer or transparent during polymerisation, allowing hence for less or more light to reach deeper areas in relation to the light transmitted at the beginning of irradiation.⁷ The change in light transmission vs. time is determined by the increasing or decreasing mismatch between the refractive indexes of monomer and filler, as the resin polymerizes.⁸ It has been evidenced in commercially available bulk-fill resin composites, however, that the mode of light transmission does not alter the degree of conversion or polymerisation kinetics in depths up to 4 mm,⁷ providing that the radiant exposure specified by the manufacturer was applied properly.

The aim of the present study was therefore to evaluate the potential of a commercial FRC to be adequately cured in increments larger than 2 mm, and to describe the kinetic of polymerization as a function of incremental thickness. In addition, several mechanical properties, measured at micro and macro scale, were evaluated, to allow a comparison of the FRC with several particulate high viscosity bulk-fill resin composites that were measured under identical conditions and published previously.⁹

The null hypotheses tested were that: a) the depth (100- μ m to 6-mm) has no effect on polymerisation kinetic parameters and degree of conversion (DC) measured 300s post-irradiation

2. Materials and Methods

One FRC (EverX Posterior, GC, Lot 1310242) was investigated by assessing the degree of conversion (DC) and the polymerisation kinetic at various depths (100- μ m, 2-mm 4-mm and 6 mm), as well as the macro (flexural strength and flexural modulus) and micro-mechanical properties (Vickers hardness, HV, indentation modulus, Y_{HU} and creep, Cr). In addition, the curing characteristics of the light curing unit (LCU) which was used in all tests for polymerisation (LED-LCU Bluephase 20i, Ivoclar Vivadent, Schaan, Liechtenstein, High power mode) were considered.

As indicated by the manufacturer, the organic

matrix of the analysed FRC is methacrylate-based (Bis-GMA, TEGDMA, PMMA), while the inorganic fillers (77 % by weight) consist of E-glass fibers (5-15 wt%) with an average length between 1-2 mm, barium borosilicate glass filler (60-70 wt%) with an average size between 0.1-2.2 μ m and silicon dioxide (1-5 wt%) (<http://www.dibateb.com/wp-content/uploads/2016/01/FAQ-everx-posterior.pdf>).

2.1. Degree of conversion (DC) and polymerisation kinetic.

DC was measured in a real-time profile (5 minutes, with 2 spectra/s) with an FTIR-Spectrometer with an attenuated total reflectance (ATR) accessory (Nexus, Thermo Nicolet, Madison, USA). Four different specimen geometries were considered. Therefore 100 μ m and 2-mm, 4-mm and 6-mm high molds (4 mm diameter) were filled in bulk. Specimens were cured by applying the curing unit for 20 s directly on the top (0 mm exposure distance) of the mold, covered by a transparent polyacetate sheet. For each thickness, six specimens were measured (n = 6). The non-polymerized composite paste was applied directly on the diamond ATR crystal in the mold as described above. DC was measured on the bottom of the specimens and was calculated by assessing the variation in peak height ratio of the absorbance intensities of methacrylate carbon carbon (C-C) double bond (peak at 1634 cm^{-1}) and that of an internal standard (aromatic C-C double bond, peak at 1608 cm^{-1}) during polymerization, in relation to the uncured material.

$$DC_{\text{Peak\%}} = \left[1 - \frac{(1634\text{cm}^{-1}/1608\text{cm}^{-1})_{\text{Peak height after curing}}}{(1634\text{cm}^{-1}/1608\text{cm}^{-1})_{\text{Peak height before curing}}} \right] \times 100$$

The DC at the end of the observation time (300 seconds) is indicated, while the increase in DC (= decrease of the C-C double bonds) until 300 seconds post-irradiation was described by the superposition of two exponential functions as:

$$y = y_0 + a \cdot (1 - e^{-bx}) + c \cdot (1 - e^{-dx})$$

The term y_0 presents the y-intercept, depending e.g. on the thickness of the specimen and the composition of the material. The parameters: a, b, c, d are modulation factors of the exponential function to optimize the exponential sum function on the measured curve.

In addition, the change in carbon-carbon double bond conversion with respect to time was calculated and plotted against DC. The maximal rate of C-C double bond conversion (Rate_{max}) and the corresponding DC are indicated.

2.2. Macro mechanical properties - Flexural strength (FS) and flexural modulus (FM)

The flexural strength (FS) and flexural modulus (FM) were determined in a three-point-bending test (n = 20) in analogy to ISO/DIN 4049:1998.¹⁰ The specimens were made by compressing the composite material between two glass plates with intermediate polyacetate sheets, separated by a steel mould having an internal dimension of 2 x

2 x 16 mm. Irradiation occurred with the above described LCU on top and bottom of the specimens, with three light exposures of 20 seconds per side, overlapping one irradiated section no more than 1 mm of the diameter of the light guide, in order to prevent multiple polymerization. After removal from the mould the specimens were grinded with silicon carbide sand paper (grit size P 1200/4000 (Leco)) in order to remove excess material. All specimens were then stored in distilled water at 37°C for 24 h prior to testing. The specimens were loaded until failure using a universal testing machine (Z 2.5, Zwick/ Roell, Ulm, Germany) in a three-point bending test device, constructed according to the guidelines of NIST No. 4877 with 12 mm distance between the supports. Specimens were immersed in distilled water at room temperature during testing. The crosshead speed was 0.5 mm/min. The universal testing machine measured the force during bending as function of deflection of the beam. The bending modulus was calculated from the slope of the linear part of the force-deflection diagram.

2.3. Micro-mechanical properties

Fragments ($n = 10$) of the three-point-bending test specimens were used to determine the micro-mechanical properties - Vickers hardness (HV), indentation modulus (Y_{HI}) and Creep (Cr) - according to DIN 50359-1:1997-10 by means of a universal hardness device (Fischerscope H100C, Fischer, Sindelfingen, Germany). Prior to testing, the specimens were polished with a diamond suspension (mean grain size: 1 μm). Measurements were done on the first polymerised side of the specimens (6 measurements per specimens, 60 measurements in total). The test procedure was carried out force controlled; the test load increased and decreased with constant speed between 0.4 mN and 500 mN. The load and the penetration depth of the indenter were continuously measured during the load-unload-hysteresis. The Universal hardness is defined as the test force divided by the apparent area of the indentation under the applied test force. From a multiplicity of measurements, a conversion factor between Universal hardness (Martens Hardness) and Vickers hardness was calculated and implemented in the software, so that the measurement results were indicated in the more familiar Vickers hardness (HV) units. The indentation modulus (Y_{HI}) was calculated from the slope of the

tangent of indentation depth-curve at maximum force. By measuring the change in indentation depth with constant test force, a relative change in the indentation depth can be calculated. This is a value for the creep of the materials.

2.4. Curing unit characteristics

The analysis of the variation in irradiance delivered at distances up to 10 mm away from the light tip was performed in 1 mm steps ($n = 3$) on a laboratory-grade NIST-referenced USB4000 Spectrometer (MARC (Managing Accurate Resin Curing) System, Bluelight Analytics Inc., Halifax, Canada). The miniature fiber optic USB4000 Spectrometer uses a 3648-element Toshiba linear CCD array detector and high-speed electronics. The spectrometer has been spectro-radiometrically calibrated using Ocean Optics' NIST-traceable light source (300-1050 nm). The system uses a CC3-UV Cosine Corrector to collect radiation over 180° field of view thus mitigating the effects of optical interference associated with light collection sampling geometry.

2.5. Statistical analysis

Statistical analysis were performed using statistical software (SPSS Inc. 23.0, Chicago, IL, USA). A Shapiro-Wilk test verified the normal distribution of the data. Descriptive statistics and 95% confidence interval are indicated. DC and irradiance results were statistically compared using one-way ANOVA followed up with Tukey's post hoc test. In all statistical tests, p -values < 0.05 were considered statistically significant.

3. Results

3.1. Degree of conversion (DC) and polymerisation kinetic

The degree of conversion as function of increment thickness measured 300 s after initiating the polymerisation as well as the kinetic parameters are summarised in Table 1.

One way ANOVA revealed no significant difference in DC measured 300 s post irradiation in a depth of 100- μm and 2-mm ($p = 0.281$). Similarly, no significant difference was identified between DC measured at 2-mm and 4-mm ($p = 0.724$), while the DC measured at 6-mm depth was significantly lowest ($p < 0.001$).

The polymerisation kinetic (Fig 1, Table 1) was well described ($R^2 > 0.95$) by an exponential sum function. Parameters "a" and "b" are describing the gel phase of the polymerisation process.

Table 1. DC (%) measured 300 s post-irradiation (mean and standard deviation in parenthesis; superscript letters indicate statistically homogeneous subgroups as function of specimen's thickness, Tukey's HSD test, $\alpha = 0.05$), maximal rate of polymerisation, Rate_{max} and parameters of the exponential sum function (95% confidence interval).

| Depth | DC | Rate _{max} | y_0 | a | b | c | d |
|-------------------|----------------|---------------------|--------------------|------------------|------------------|------------------|------------------|
| 100 μm | 58.91 (1.35)A | 25.5 \div 27.4 | -15.9 \div -14.6 | 66.1 \div 67.3 | 0.40 \div 0.42 | 7.30 \div 7.60 | 0.02 \div 0.02 |
| 2 mm | 57.43 (1.88)AB | 20.0 \div 22.1 | -11.7 \div -10.6 | 60.5 \div 61.6 | 0.27 \div 0.28 | 7.46 \div 7.77 | 0.01 \div 0.01 |
| 4 mm | 56.58 (0.62)B | 13.5 \div 15.7 | -9.6 \div -8.8 | 57.6 \div 58.4 | 0.20 \div 0.21 | 7.35 \div 7.60 | 0.01 \div 0.01 |
| 6 mm | 52.40 (1.43)C | 6.7 \div 8.2 | -3.1 \div -2.6 | 46.6 \div 47.1 | 0.17 \div 0.17 | 8.53 \div 8.72 | 0.01 \div 0.01 |

Considering the indicated 95% confidence interval, no difference was identified for "a" at 100- μ m and 2-mm depths, while "a" decreased subsequently with depth. In contrast, the parameter "b" decreased with increased specimen thickness already in thin layers. Parameters "c" and "d"

characterise the glass phase of the polymerisation process and varied comparatively less with the depth. While parameter "d" was not dependent on specimen thickness, parameter "c" showed a slightly increase, however only at a depth of 6-mm. The maximal rate of polymerisation, Rate_{max}' was,

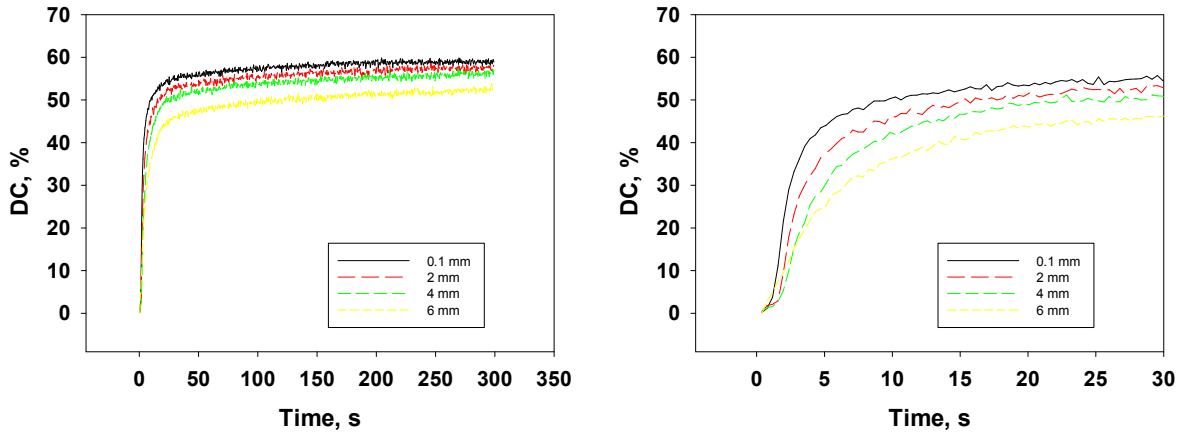


Figure 1. Variation in DC in time measured at the bottom of 100- μ m, 2-mm, 4-mm and 6-mm increments (mean value of n = 6), left: 300 s recordings; right a close up view of the first 30 s allowing identifying the different start of the C-C double bond conversion as function of specimen thickness).

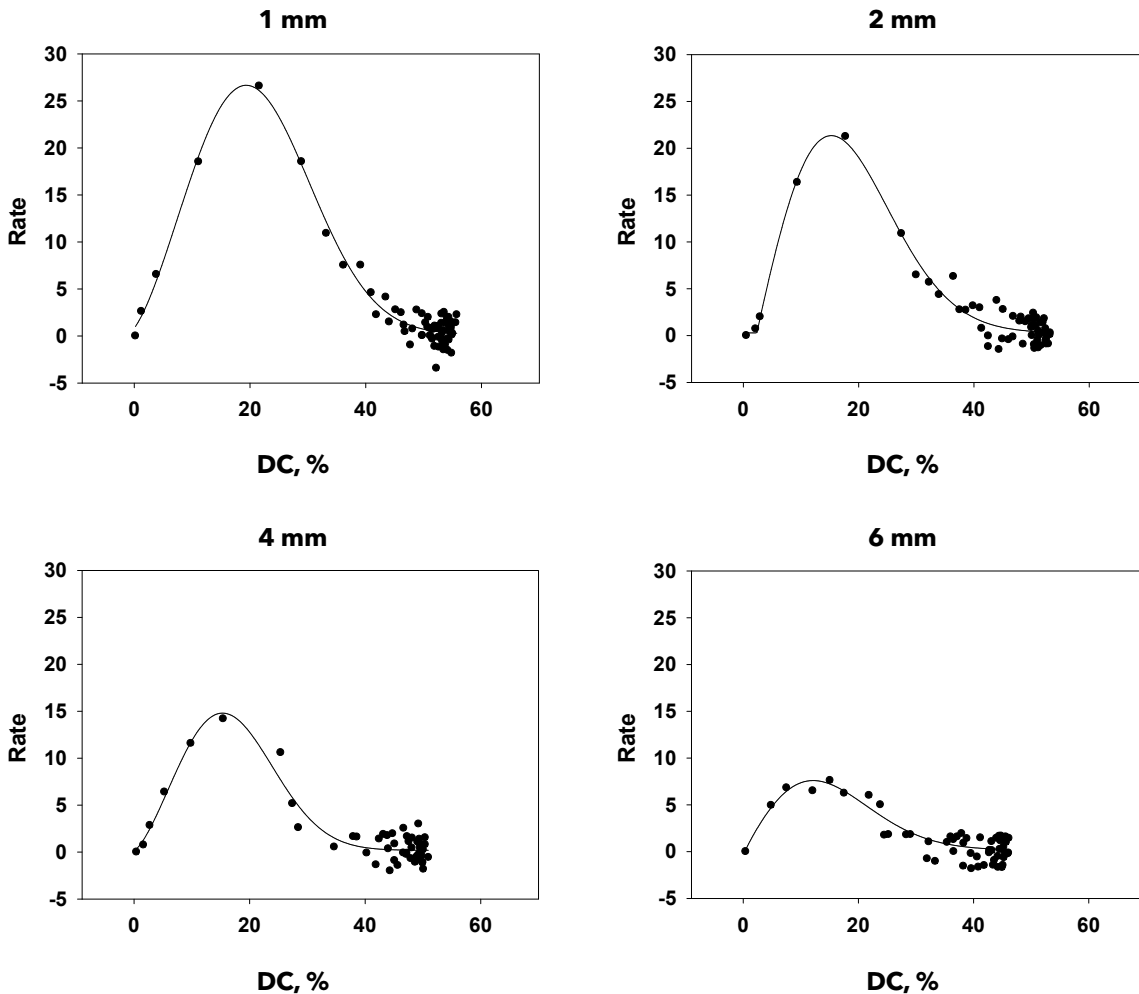


Figure 2. Rate of C-C double bond conversion versus degree of cure (DC) as function of increment thickness.

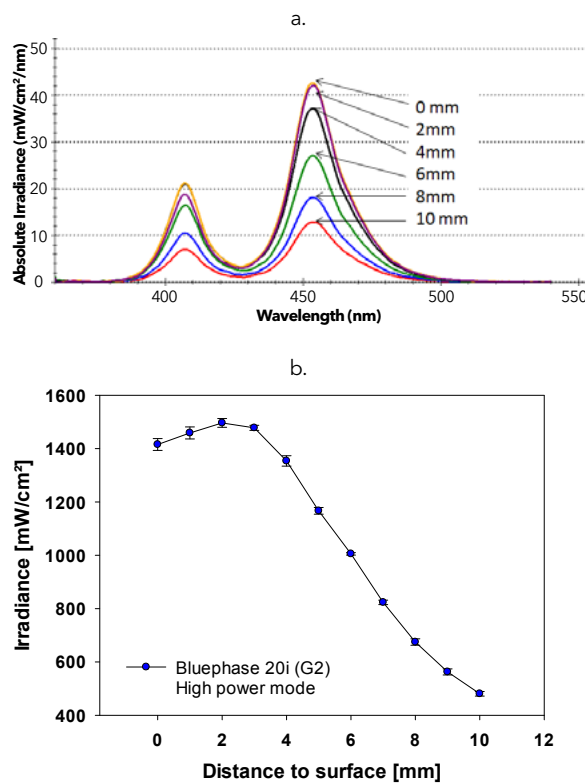


Figure 3. a) Spectral distribution (exposure distances 0, 2, 4, 6, 8 and 10 mm) and b) Variation in irradiance delivered at exposure distances up to 10 mm (Bluephase 20i (G2), High power mode, $n=3$).

in contrast, strongly dependent on specimen thickness, being about 4 times lower in 6-mm thick increments in comparison to the thin, 100- μm increments (Fig. 2 and Table 1).

As for the mechanical properties, the flexural strength (mean \pm standard deviation) amounted (128.30 ± 8.38) MPa, while the flexural modulus (8.38 ± 0.87) GPa. The properties measured at microscopic scale amounted (92.00 ± 15.86) N/mm² for HV, (17.82 ± 1.82) GPa for Y_{H_U} and (3.35 ± 0.84) % for Cr.

3.2. Curing unit characteristics

The spectral distribution identified the used light curing unit as a violet-blue LED LCU, with two distinct peaks at 407 nm (violet) and 454 nm (blue). The spectral distribution measured at exposure distances of 0, 2, 4, 6, 8 and 10 mm is indicated in Fig. 3a. The incident irradiance (Fig. 3b) was identified as (1415.3 ± 22.2) mW/cm². The irradiance increases significantly up to an exposure distance of 2 mm, when it reaches a value of (1496.3 ± 16.3) mW/cm². Subsequently, the irradiance decreases exponentially with the exposure distance, amounting 78% of the maximal irradiance at an exposure distance of 5 mm, while only 32% at an exposure distance of 10 mm.

4. Discussion

This study analyzed the curing performance of a FRC for direct restorations, indicated to be used particularly in large posterior cavities, including cavities with 3 surfaces or more as well as deep cavities or such with missing cusps. Although the

FRC it is not specifically advertised as a bulk-fill resin composite, the manufacturer specifies that the material may be cured in larger increments. This statement can only partially be confirmed by the present study. While the DC measured at the end of the observation period (5 minutes) revealed statistical similar values at the bottom of 2 mm and 4 mm thick increments, the situation changes when including in this comparison the top of a restoration. In this latter case, which was simulated in the present study by evaluating a 100- μm thin increment, a decrease in DC was identified in 4-mm depths, while values measured in 2-mm depths were similar to the top. The attenuation in polymerization degree with depth is even clearly evidenced when involving the calculated polymerization kinetic parameters and the maximal rate of polymerization, as it will be discussed below.

The analyzed material is a methacrylate-based dental restorative, containing monomers with two carbon-carbon (C-C) double bonds, namely the Bowen monomer bisphenol-A-glycidyl dimethacrylate (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA), both able to build a three-dimensional polymer network. As generally known, the light-induced polymerization process is a radical one that initiates when photons emitted by the LCU activates the photo-initiator system. Applied exposure time and radiant exposure are both in line with manufacturer recommendations. The curing conditions involved in the present study an exposure time of 20s by using a modern LED LCU with an irradiance of (1415.3 ± 22.2) mW/cm², which was placed directly and perpendicularly to the specimen's surface. The radiant exposure (= incident irradiance \times exposure time) reaching the specimen's surface was accordingly high, and amounted 28.3 J/cm². These curing conditions are comparable or even superior to radiant exposure values identified for adequate polymerisation in conventional (21-24 J/cm²)^{11,12} or bulk-fill resin-based composites (20 J/cm²).^{13,14}

The second stage of the polymerization process, the propagation, is directed by the radical attack on Bis-GMA and TEGDMA monomers, leading to larger molecules (chain growth) by preserving the free radical.¹⁵ This stage of polymerization was intended to be monitored in the present study by following the development of the DC over time. It must however be noted that the presented DC measurements evaluated the functional group conversion (C-C double bond) and not the monomer conversion. This stage of the polymerization process is described as a sequence of three distinguished phases: the quasistatic process, the gel phase, and the glass phase. The quasistatic process is described by a normal chain growth, while the number of high molecular weight chains increases and, consequently, also the viscosity of the composite. Next, the reaction rate increases during the gel phase while at a

further progress of the polymerization process, the organic matrix becomes a gelatinous state, and the reaction rate decreases (glass effect).¹⁵

The variation of DC during irradiation of the FRC was well described by the superposition of two exponential functions. The first exponential function, characterizing the gel phase, is described by the parameters "a" and "b", while the second exponential function is characterizing the glass phase and is described by the parameters "c" and "d". The experimental data reveal that parameter "a" decreases with increased specimen thickness, but only in increments thicker than 2 mm, while parameter "b" decreases with increased specimen thickness already starting from thin layers. Both effects demonstrate that the decrease of the C-C double bonds in the gel phase is slower in thicker layers compared to the top, due to the exponential decrease (Lambert's Law) of light transmission with the composite's thickness.¹⁶ It has been previously shown that merely 24% ÷ 44% of the incident blue light and 9 ÷ 14% of the incident violet light of a LCU is transmitted through 2 mm commercially available bulk-fill resin composite increments. These values are yet consistently reduced in 4 mm increments (9 ÷ 24% and 3 ÷ 9%, respectively)⁷ and may further change to the detriment of curing quality when LCUs with low irradiances are used or, as usual in clinical practice, when the LCU cannot be applied perpendicularly and directly on the restoration surface.

While the effect of light attenuation was not directly reflected in DC, as evidenced by the statistically similar DC values measured in 2 and 4 mm increments, the polymerization kinetic describes a slower polymerization (lower parameter a and b) as well as a lower maximal rate of polymerization ($\text{Rate}_{\text{max}} = 20.0 \div 22.1$ and $13.5 \div 15.7$, respectively). The lower amount of photons reaching 4-mm layers compared to 2-mm will activate less efficiently the photo-initiator, inducing less nuclei of polymerization and therefore longer polymer chains and a lower cross-linking. This can have as a result a lower modulus of elasticity, although the amount of C-C double bond conversion (DC) is similar. This effect might be in accordance with data presented by Omran et al. by evaluating the bond strength of various resin composites to dentin, revealing that EverX Posterior can be safely applied in bulks of 4-mm increments, similarly to other analysed bulk-fill composites, but its performance was better in 2-mm thick increments.¹⁷

In addition to that, parameters "c" and "d" describing the glass phase reveal that "c" is not

altered up to 4-mm thick increments, while "d" has a very low value and is of less relevance. The low variation of parameters "c" and "d" demonstrates that the reaction kinetic is less thickness-dependent in the glass phase compared to the gel phase.

Since the analyzed material is indicated to be used in large posterior cavities in optional larger incremental thickness compared to conventional resin-composites layered in 2 mm increments, a direct comparison with the material category of high-viscosity bulk-fill resin-composites seems pertinent. This comparison is possible owing to identical specimen geometry, test type and test parameters as well as specimen storage conditions. Accordingly, EverX Posterior may be ranged for the flexural strength (128.30 ± 8.38) MPa in the middle of the above specified material category, characterised by values varying among 99.9 ± 10.7 MPa (Admira Fusion x-tra) and 142.8 ± 12.9 MPa (SonicFill). Similar considerations apply to the Vickers hardness (92.00 ± 15.86 N/mm²) when compared to the high-viscosity bulk-fill resin-composite category, delimited by the values 77.1 ± 5.6 N/mm² (Admira Fusion x-tra) and 133.5 ± 32.0 N/mm² (X-tra Fil). The modulus of elasticity measured in either macro and micro scale was rather interrelated to the upper values of the material category (8.38 ± 0.87) GPa vs 4.5 ± 0.8 (Tetric EvoCeram Bulk Fill) to 9.5 ± 0.6 (X-tra Fil) and for Y_{HU} (17.82 ± 1.82) GPa vs 13.4 ± 0.8 GPa (Tetric EvoCeram Bulk Fill) to 22.2 ± 1.7 GPa (X-tra Fil).⁹

5. Conclusions

In the present study and considering the tested FRC, the polymerization kinetic allowed identifying a slower start of the polymerization process depending on material's depth. This was associated with a lower maximal rate of C-C double bond conversion, although DC values recorded in 2 and 4 mm depth were statistically similar. There is, however, evidence that the quality of curing at 4-mm depth is lower compared to the top of the material. The null hypothesis must therefore be rejected.

The analyzed FRC revealed mechanical properties situated within the range of the high viscosity bulk-fill resin-composites category, with a high modulus of elasticity (upper range of the mentioned material categories).

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Questions**Which of the following fiber characteristics are considered an optimum in dental fiber reinforced resin composite (FRC)?**

- a. An aspect ratio (= proportional relationship between the length and width of a fiber) about 5.2;
- b. An aspect ratio < 1;
- c. Fiber length > 2 mm;
- d. Fiber length < 0.5 mm.

Which parameters do not influence the amount of light reaching deeper areas of a resin composite restoration?

- a. The translucency of the material;
- b. The mismatch between the refractive indexes of the monomer and filler;
- c. The wavelength of the incident light;
- d. The placement of a resin composite in a molar or a premolar of similar cavity size.

What is defined as radiant exposure?

- a. The product of incident irradiance and exposure time;
- b. The amount of photons emitted by a curing light;
- c. The amount of photons reaching the specimen's surface;
- d. The distance a curing unit is applied to the specimen's surface.

The degree of conversion measured by an FTIR-Spectrometer assesses:

- a. The conversion of the functional groups (e.g. C-C double bonds in methacrylates);
- b. The conversion of monomers (e.g. bis-GMA, TEGDMA);
- c. The cross-link density of polymer chains;
- d. The amount of unreacted monomers.