Original Articles

SUSCEPTIBILITY OF RBC TO VARIOUS CLINICAL RELEVANT CURING CONDITIONS

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Introduction: The study aims to quantify the impact of various curing conditions on the micro-mechanical properties of methacrylate and silorane resin-based composites (RBCs) in order to determine the threshold for sufficient polymerization.
Methodology: The analyzed RBCs have either a similar filler volume amount (55%) but a different monomer matrix composition (methacrylate or silorane) or a similar monomer matrix but a different filler volume amount (63.3% vs. 55%). Twenty-four different curing conditions were simulated. A blue-violet LED curing unit was applied in different curing modes, exposure times and distances (0-mm and 7-mm). Measurements (Vickers hardness, HV, and Indentation modulus, E) were performed after 24 h of storage in distilled water at 37°C at the top and bottom of 2-mm thick specimens (360 specimens in total).
One and multiple-way ANOVA and Tukey HSD post hoc-test (α =0.05) was used. A multivariate analysis (general linear model) assessed the effect strength of the parameters exposure time, location of measurement (top-bottom), incident irradiance, radiant exposure (ranging from 1.0 to 47.0 J/cm^2) and exposure distance on HV and E.
Results: In all materials, the highest effect on HV and E was exerted by the exposure time and location of measurement. Susceptibility to various curing conditions is material dependent, while less filled methacrylate-based as well as the silorane micro-hybrid are more robust to these variations. Fast polymerization (3s) with high irradiance is not recommended.
Conclusions: The best micro-mechanical properties at the top and bottom of 2-mm thick specimens are generated with a curing time of at least 20s at moderate irradiance.
Keywords: Resin-based composites; Hardness; Modulus of elasticity; Light curing unit; Radiant exposure.

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1. Introduction

Incremental layering technique is accepted as a golden standard for the placement of regular resin-based composite (RBC) restorations [1]. The increments are limited to a thickness of 2 mm to allow for adequate polymerization in a clinically reasonable time. In spite of decades of expert knowledge in light curing, the amount of light needed to adequately cure a regular 2-mm thick RBC increment is still debatable. This is justified in the observation of a material-dependent susceptibility to variation in radiant emittance (= radiant flux emitted by a surface per unit area) under simulated clinical conditions [2,3]. In addition, calculations based on the total energy delivered to guide irradiation protocols were shown to be invalid and not to recognize product behavior [2]. Nonetheless, there is a high demand to set a general limit for adequate curing, in order to support clinicians choosing a proper curing strategy. Literature data, based on the direct evaluation of the curing quality in various modern RBCs, indicate radiant exposure values in the range of (21-24) J/cm^2 [4,5] as sufficient for adequate polymerization. These values are often not met in a clinical situation, due to a number of factors that are related either to the technique sensitivity of the polymerization process [6,7] or to erroneously chosen curing strategies. Particularly in posterior cavities that are difficult to access, where the light curing unit (LCU) may not be placed perpendicularly to the restoration, the exposure distance may vary from an ideal position, the exposure time may be chosen too short or the LCU be contaminated with resins. The total amount of light received by the material may be further reduced by using curing strategies involving a pre-curing of the lowest increment when incrementally reconstructing a deep cavity, based on the wrong estimate [8] that the lowest increment will receive enough light when curing the upper increments or when curing through the tooth structure [9].

Table 1. Resin composite brand, type, chemical composition of matrix and filler as well as filler content by weight (wt.) and volume (vol.) %.

RBCs	RBC-Type	Batch	Shade	Resin Matrix	Filler	Filler wt%/vol%
Filtek™ Supremee XTE	Nano	N229448	A3 Dentin	Bis-GMA, Bis-EMA, UDMA, TEGDMA, PEGDMA	ZrO_2, SiO_2, ZrO_2/SiO_2	78.5/63.3
Filtek™ Supremee XTE flow	flowable Nano	N236527	A3	Bis-GMA, Bis-EMA, TEGDMA, PEGDMA	ZrO_2, SiO_2, ZrO_2/SiO_2	65/55
Filtek™ Silorane	Microhybrid	N225426	A3	3,4-Epoxycyclo-hexylethylcyclopolymethylsiloxane Bis-3,4-epoxycyclo-hexylethylphenyl-methylsilane	SiO_2, YF_3	76/55

Abbreviations: Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; Bis-EMA, ethoxylated Bisphenol-A-dimethacrylate; UDMA, Urethane dimethacrylate; TEGDMA, Triethyleneglycol dimethacrylate, PEGDMA, polyethylene glycol dimethacrylate. Data are provided by manufacturer (3M) in the instruction leaflet.

A very low transmitted irradiance was identified through 2-mm thick layers of various RBCs as well as through the tooth structure [8,9]. In a nano RBC (Filtek Supreme XTE), the transmitted light through 2-mm thick increments amounted only 1.4% to 2.1% of the incident light and was merely slightly higher (4.2% to 7.4%) in the more translucent, low viscous version of the same material (flowable nano RBC Filtek Supreme XTE flow) [8].

The same applies for RBCs with a non-methacrylate polymer matrix, such as the micro-hybrid Filtek Silorane (4.2% to 6.5%) [8]. Note that a variety of clinical relevant irradiances (= radiant flux or power received by a surface per unit area; 656.4 mW/cm² to 3361.5 mW/cm²), radiant exposures (46.96 to 0.99 J/cm²) and exposure distances (0 to 7 mm) were considered in these calculations.

The indicated % limits for the light transmittance through the various materials are related to the irradiance while the lower the irradiance, the higher the % transmitted light in the above indicated interval [8]. Based on these calculations, it was assumed that the low light transmittance will be directly reflected in the mechanical properties of the RBCs [8].

When transferring the transmitted irradiance to the more clinically relevant term of radiant exposure, the transmitted light at the bottom of 2-mm thick increments is quantified as to not exceed 4 J/cm² at an ideal exposure distance. This involved the application of the LCU in close contact and perpendicularly to the material. The transmitted light is lowered to < 2 J/cm² when the exposure distance is elevated at 7 mm [8].

The values shown above are far below the radiant exposure values indicated in the literature for adequate polymerization (21-24) J/cm² [4,5]. An initially improper cured lower increment will consequently receive low light during curing the upper ones, which is insufficient to compensate the initial deficits [8]. The aim of this study was to evaluate the effect of simulating clinically relevant curing conditions on the micro-mechanical properties of RBCs with various fillers content and similar chemical composition of the organic matrix (regular vs. flowable methacrylate-based RBCs), as well as similar volume filler content but different chemical composition of the organic matrix (methacrylate vs. silorane monomers). Therefore, 24 different radiant exposures were simulated by varying the irradiance (656.4 mW/cm² to 3361.5 mW/cm²), the radiant exposure (0.99 and 47.0 J/cm²) and the exposure distance (0 to 7 mm) [8]. Moreover, the study aims to determine the bandwidth for adequate curing in response to the application of light.

The tested null hypothesis were: i) the impact of the curing conditions would be similar in all materials; ii) there would be no difference within one material among the assessed curing conditions; iii) there would be no difference in the mechanical properties among the analysed materials.

2. Material and Methods

The micro-mechanical properties of three regular RBCs (Table 1) were analyzed under different curing conditions at a specimen thickness of 2 mm. The violet-blue LED LCU VALO (Ultradent, South Jordan, USA, serial number VO 7710) was used in three different exposure modes (Standard, High Power and Plasma Emulation), at various exposure times (5s, 10s, 15s, 20s and 40s (=2x20s with no delay in-between exposures) the Standard mode; 1s, 2s, 3s, 4s and 12s (=3x4s with no delay in-between exposures) in the High Power mode and 3s and 6s (=2x3s with no delay in-between exposures) in the Plasma Emulation mode) and exposure distances (0 mm and 7 mm). This resulted in 24 different curing conditions, that have been quantified previously [8].

2.1. Micro-mechanical properties

The micro-mechanical properties (Vickers Hardness HV and Indentation modulus, E) were assessed on cylindrical specimens (diameter 6 mm, thickness 2 mm, n = 5) according to DIN 50359-1:1997-10 [10]. For the specimen preparation, a white Teflon mould was used. Immediately after curing, the specimens were removed from the mould and stored in distilled water for 24 hours at 37°C. Thereafter, the specimens were ground and polished under water with diamond abrasive paper (mean grain sizes: 20 µm, 13 µm, 6 µm) in a grinding system (EXAKT 400CS, Exakt, Norderstedt, Germany) and transfered to an automatic universal testing device (Fischerscope H100C, Fischer, Sindelfingen, Germany). Measurements (n=6 per specimen and side) were performed at the top and bottom of each specimen. The test procedure was carried out force-controlled, where the test load increased (within 20s) and decreased (within 20s) with constant speed between 0.4 mN and 500 mN. The load and penetration depth of the indenter (Vickers pyramid: diamond right pyramid with a square base and an angle of α = 136° between the opposite faces at the vertex) were continuously measured during the load-unload hysteresis. Universal hardness is defined as the test force divided by the apparent area of indentation under the applied test force.

From a multiplicity of measurements stored in a database supplied by the manufacturer, a conversion factor (0.0945) between Universal hardness and HV was calculated by the manufacturer and entered into the software, so that the measurement results were indicated in the more familiar HV units. E was calculated from the slope of the tangent adapted at the beginning (at maximum force) of the non-linear indentation depth curve upon unloading.

2.2. Statistical Analysis

A Shapiro–Wilk test verified the normal distribution of the data. The results were compared using one and multiple-way ANOVA and Tukey HSD post hoc-test (α = 0.05). A multivariate analysis (general linear model) assessed the effect of the parameters exposure time, incident irradiance, incident radiant exposure (varying among 0.99 and 47.0 J/cm^2 [8]), exposure distance (0 mm and 7 mm) and measuring position (top-bottom) on HV and E.

The partial eta-squared statistical data report the practical significance of each term, based on the ratio of the variation accounted for by the effect. Larger values of partial eta-squared indicate a greater amount of variation accounted for by the model effect, to a maximum of 1. Correlation analyses (Pearson correlation) between HV and E were performed within each RBC. In all statistical tests, p-values < 0.05 were considered statistically significant when using SPSS Inc. (Version 24.0, Chicago, IL, USA).

3. Results

The exposure time ($p<0.05$, ηP^2 = 0.899 for E and 0.872 for HV) and the location of the measurement (top-bottom; $p<0.05$, ηP^2 = 0.849 for E and 0.867 for HV) strongly influenced the micro-mechanical properties measured in the Filtek Supreme XTE flow specimens. With a lower, but significant influence, there followed the factor incident irradiance ($p<0.05$, ηP^2 = 0.399 for E and 0.251 for HV), while the impact of exposure distance was very low ($p<0.05$, ηP^2 = 0.038 for E and 0.030 for HV). The impact of the incident radiant exposure was significant as well ($p<0.05$, η_p^2 = 0.607 for E and 0.498 for HV), but lower compared to the individual effect of exposure time.

The impact of the above-mentioned factors was even stronger in the higher filled methacrylate-based nano RBC Filtek Supreme XTE. It follows the sequence: exposure time ($p<0.05$, ηP^2 = 0.972 for E and 0.973 for HV), location of measurement (top-bottom) ($p<0.05$, ηP^2 = 0.993 for E and 0.996 for HV), irradiance ($p<0.05$, ηP^2 = 0.126 for E and 0.128 for HV) and exposure distance ($p<0.05$, ηP^2 = 0.037 for E and 0.031 for HV). The impact of the incident radiant exposure was significant as well ($p<0.05$, η_p^2 = 0.248 for E and 0.182 for HV).

In Filtek Silorane, the impact of the above-mentioned factors follow the same sequence as above: exposure time ($p<0.05$, ηP^2 = 0.932 for E and 0.903 for HV), location of measurement (top-bottom) ($p<0.05$, ηP^2 = 0.903 for E and 0.932 for HV), incident irradiance ($p<0.05$, ηP^2 = 0.760 for E and 0.637 for HV) and exposure distance ($p<0.05$, ηP^2 = 0.033 for E and 0.041 for HV). The impact of the incident radiant exposure was significant as well ($p<0.05$, η_p^2 = 0.476 for E and 0.401 for HV).

Considering all curing conditions and both specimen sides (both top and bottom), the micro-mechanical parameters, HV and E, were lower in Filtek Supreme Flow ($p<0.05$; E = 8.09 GPa, HV = 54.34 N/mm^2), while Filtek Silorane and Filtek Supreme showed statistically similar values (p = 0.138 for E (11.18 GPa and 10.47 GPa) and 0.283 for HV (75.34 N/mm^2 and 79.95 N/mm^2). Considering the specimen surfaces individually, the material sequence for the top surface was, in statistically significant decreasing order of the measured micro-mechanical properties (Filtek Supreme > Filtek Silorane > Filtek Supreme Flow, with E = 15.11 GPa; 13.22 GPa; 9.19 GPa; and HV = 124.03 N/mm^2; 93.71 N/mm^2; 64.59 N/mm^2). For the bottom surface, significant higher values were identified in Filtek Silorane specimens ($p<0.05$), while both methacrylate RBCs performed statistically similarly (p = 0.078 for E and 0.065 for HV). The values measured at the bottom of the specimens in the sequence Filtek Silorane, Filtek Supreme Flow and Filtek Supreme were E = 9.15 GPa; 7.00 GPa and 5.83 GPa; HV = 56.98 N/mm^2; 44.08 N/mm^2; 35.87 N/mm^2). The individual values for all materials and curing conditions are summarized in Table 2 and illustrated in Fig 1.

An excellent correlation was calculated between HV and E within each material (Pearson correlation coefficient = 0.985 for Filtek Supreme XTE, 0.977 in Filtek Silorane and 0.973 in Filtek Supreme XTE Flow).

4. Discussion

Although constantly improvement in the last decades, the demand for shortening the exposure time for curing dental RBCs, to allow for faster and more economical clinical work, is still valid nowadays. For this purpose manufacturers do not stray from the development of LCUs with continuously

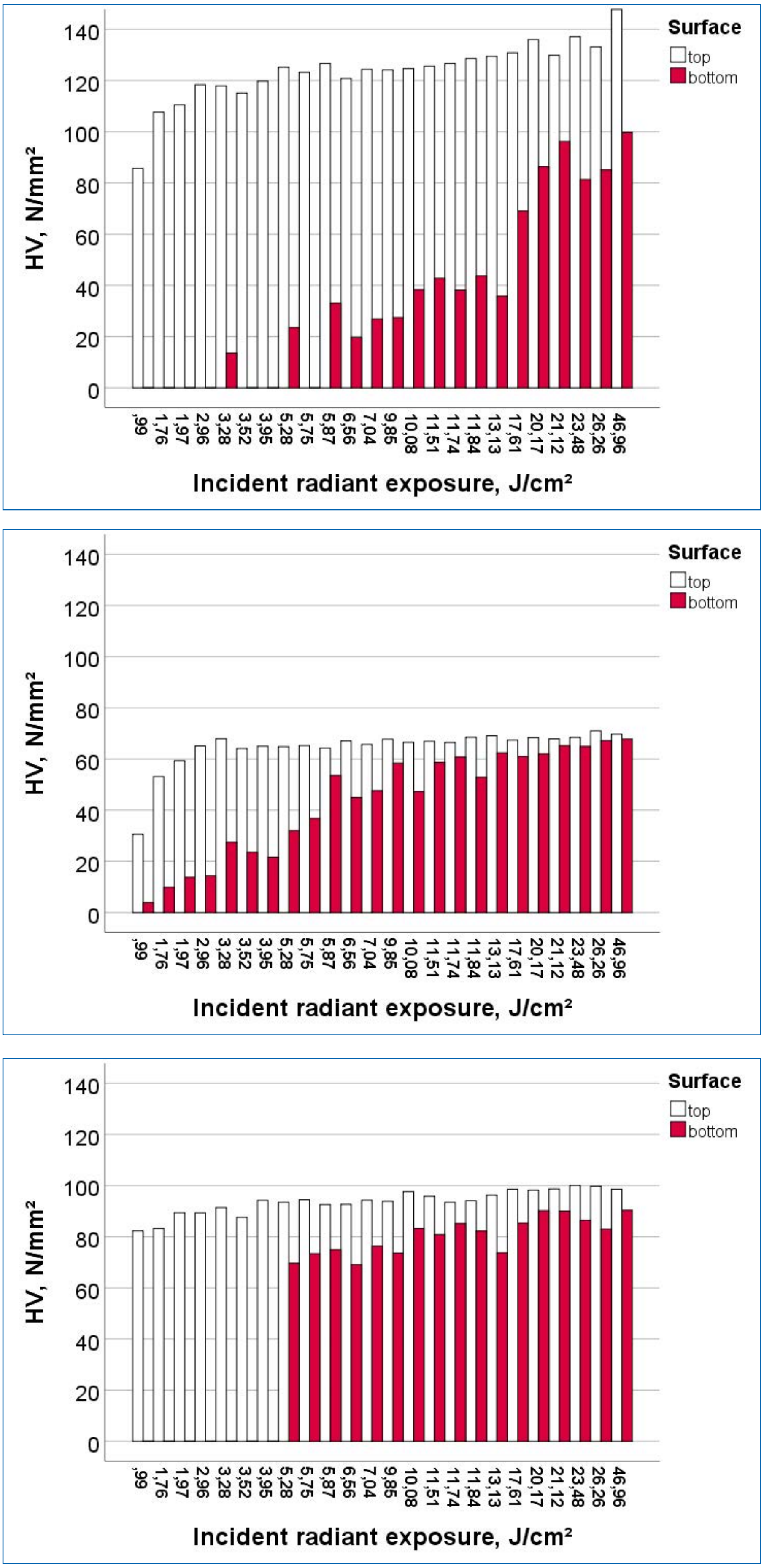

Figure 1. Variation in Vickers Hardness as a function of radiant exposure measured on top and bottom of 2-mm thick specimens made out by the RBC: a) Filtek Supreme XTE; b) Filtek Supreme XTE flow; c) Filtek Silorane; corresponding irradiance, exposure time and distance are presented in Table 2.

higher radiant emittance. This trend was motivated in the concept of "exposure reciprocity" that is based on the opinion that a RBC needs a certain amount of energy to be adequately cured, while the way in which photons are supplied is supposed to be of minor importance. The term radiant exposure (J/cm^2) characterizes the amount of energy supplied by the LCU and is calculated as the product of the radiant emittance (mW/cm^2) and exposure time (s). The "exposure reciprocity" concept assumes a similar effect when exposing the RBC to a given radiant exposure, irrespective if the radiant emittance is enhanced to the detriment of the exposure time or vice versa.

This simple construct sounds useful in a clinical situation but it is not universally valid [2]. Musanje et al. [2] identified no lower limit of radiant emittance for an effective polymerization, up to 25 mW/cm^2, but the very long exposure time needed for appropriate curing is no longer relevant for practical use. Numerous studies in recent years have clearly shown that efficient polymerization, especially in depth, is achieved with LCUs of a radiant emittance about 1200 mW/cm^2 and exposure times of at least 20s [11]. The radiant exposure levels required for a RBC to be adequately polymerized depend to a large extent on the composition of the RBC [2] and consequently each product must be individually analyzed.

The present study attaches particular attention to the effect of the different composition of the organic matrix (silorane vs. methacrylate) at a comparable filler volume amount (55%).

With the limitation of using commercially available materials with a small difference in their organic matrix (Table 1), the study also analyzes the effect of the filler volume amount at a given composition of the organic matrix. Confirming the data of Musanje et al. [2,3], the analyzed RBCs reacted differently to the variation in radiant exposure. The analyzed flowable RBC (Filtek Supreme XTE flow) showed only minor differences in micro-mechanical properties at a sharp variation of radiant exposure levels, while the higher viscous RBCs (Filtek Supreme XTE and Filtek Silorane) reacted with a higher variation in the measured properties.

The present study also identified that the exposure time and the irradiance itself, and not only their connection as radiant exposure (time x irradiance), has an influence on the polymerization of the analyzed RBC, thus confirming again previous studies [2,3]. Moreover, the study identified that in all analyzed materials, it was the exposure time showing the highest impact higher partial eta-squared values) on the measured properties, while irradiance played a minor role. In general, the best results were identified in all RBCs at the longest exposure time (40 s) and lowest LCU's radiant emittance (1174 mW/cm^2). Note, however, that the lowest irradiance used in the present study has a comparatively high value and involves a modern, well-functioning LCU.

The highest radiant emittance employed in the present study was delivered by the program "plasma", with a value of 3361.5 mW/cm^2. A 3s curing in this mode, based on the data of the present study must be considered as insufficient for all analyzed materials and should be avoided clinically. When doubling the exposure time (6-s) in the same curing mode, the properties improved, but to a different extent. The silorane-based material showed properties that were statistically similar to a 20s or 40s exposure in the standard mode, and thus similar to the highest achievable properties in this material.

For the methacrylate-based RBCs, the 6-s exposure might be considered as sufficient only for the top surface. At the bottom surface, the recorded values were comparable to a 20-s exposure in the standard mode but significant lower as a 40-s exposure in the same mode. The differentiation among materials accentuates even more when the clinically relevant exposure distance of 7 mm is additionally considered. The silorane-based material still performed well at both top and bottom sides, the flowable methacrylate-based RBC reached comparable values with a 20-s polymerization in the standard mode, but the values measured at the bottom of the regular methacrylate-based material are very low, indicating insufficient polymerization.

Under these conditions, a fast, 6-s polymerization at the highest analyzed radiant emittance can be unrestrictedly recommended only for the silorane-based RBC and unconditionally banned for the analyzed regular methacrylate-based RBCs. The results confirm the failure recorded earlier with the $P_{(plasma)}A_{(arc)}C_{(curing)}$-LCUs, which were advocated for a rapid, 3-second curing, as being equivalent to that of a 40 or 60-s s exposure from a QTH (Quartz Tungsten Halogen) light [12].

Numerous studies attested insufficient polymerization [12,13], emphasizing the need for multiple 3-s exposures to achieve a clinically adequate performance [12,14]. Enhancing LCU's radiant emittance alone is thus insufficient in curing currently available RBCs. Modern attempts conjunct a fast curing (3-s) at high radiant emittance (> 3000 mW/cm^2) with altering the polymerization mechanism of methacrylate-based RBCs. The latest one involves a reversible addition-fragmentation chain transfer (RAFT) polymerization and has been so far incorporated in two commercial available bulk-fill RBCs [15,16].

The proof for the clinical success of a 3-s curing of materials with a RAFT polymerization is missing for the moment. This is not least due to the recent launch (2019) of the products. Note, however, that curing with high radiant emittance increases the risk to over-heat the pulp or to injure the soft tissues. Besides, curing fast at high irradiances will leave no room to relieve internal stresses accumulated during shrinkage [17].

To simulate clinically relevant curing conditions, the LCU was placed either directly on or at a distance of 7 mm from the specimen's surface. This distance was chosen based on the observation of Price at al.[18] that the distance between the LCU and the cavity bottom is 6.3 mm (standard deviation 0.7) in a standard class II cavity.

Note that the irradiance in the simulated curing conditions of the present study amounted at an exposure distance of 7 mm only 43% to 49% of the values at a direct contact to the surface [8]. It should also be noted that the curing times recommended by the manufacturer are mainly based on the assumption

Table 2. Vickers hardness [N/mm^2] and Indentation modulus (GPa) measured on specimen's top and bottom at 0 mm and 7 mm exposure distance. Superscripts indicate statistically homogeneous subgroups within a column (Tukey's HSD test, α = 0.05).

a) Filtek™ Supreme XTE

Vickers hardness HV [N/mm^2]

Curing mode	Exposure time [s]	0mm- exposure distance						7mm-exposure distance					
		top			bottom			top			bottom		
Standard	5	126.6	CD	(2.1)	33.1	CD	(2.7)	117.9	c	(1.2)	0.0	a	(0.0)
	10	126.6	CD	(3.5)	38.1	D	(2.5)	120.8	cdef	(2.5)	19.7	b	(2.7)
	15	130.8	DEF	(2.6)	69.0	E	(5.7)	124.1	efg	(2.1)	27.4	c	(2.7)
	20	137.2	F	(2.8)	81.4	F	(5.0)	129.4	hi	(2.4)	35.9	d	(2.3)
	40	155.9	G	(3.8)	99.8	G	(2.9)	133.1	i	(1.1)	85.1	f	(4.5)
High Power	1	107.7	A	(3.7)	0.0	A	(0.0)	85.7	a	(3.5)	0.0	a	(0.0)
	2	115.1	B	(4.5)	0.0	A	(0.0)	110.5	b	(3.6)	0.0	a	(0.0)
	3	125.2	CD	(3.4)	23.5	B	(2.7)	118.4	cd	(2.2)	0.0	a	(0.0)
	4	124.4	C	(2.1)	26.9	BC	(4.0)	119.7	cde	(3.2)	0.0	a	(0.0)
	12	129.8	CDE	(4.6)	96.2	G	(5.6)	128.6	ghi	(2.1)	43.7	e	(1.1)
Plasma Emulation	3	124.7	CD	(1.8)	38.3	D	(3.4)	123.2	def	(2.9)	0.0	a	(0.0)
	6	135.9	EF	(3.2)	86.4	F	(4.9)	125.5	fgh	(2.8)	42.8	e	(2.1)

0 = not measurable, material was not cured

Indentation Modulus, E [GPa]

Curing mode	Exposure time [s]	0mm- exposure distance						7mm-exposure distance					
		top			bottom			top			bottom		
Standard	5	14.6	BC	(0.7)	6.2	BC	(0.3)	14.3	c	(0.5)	0.0	a	(0,3)
	10	15.0	BC	(0.2)	6.4	BC	(0.5)	14.9	cde	(0.6)	4.8	b	(0,3)
	15	16.0	DEF	(0.4)	10.5	D	(0.6)	15.8	def	(0.5)	5.4	c	(0,5)
	20	16.7	F	(0.3)	12.1	E	(0.4)	15.8	ef	(0.4)	6.4	d	(0,2)
	40	17.7	G	(0.3)	13.4	F	(0.5)	16.2	f	(0.5)	11.9	g	(0,5)
High Power	1	12.9	A	(0.4)	0.0	A	(0.0)	10.3	a	(0.3)	0.0	a	(0,0)
	2	14.3	B	(0.4)	0.0	A	(0.0)	13.0	b	(0.6)	0.0	a	(0,0)
	3	14.8	BC	(0.2)	5.6	B	(0.5)	14.9	cd	(0.4)	0.0	a	(0,0)
	4	15.3	CD	(0.2)	6.3	BC	(0.6)	15.2	cde	(0.4)	0.0	a	(0,0)
	12	16.2	EF	(0.4)	12.9	EF	(0.4)	15.8	def	(0.2)	8.2	e	(0,1)
Plasma Emulation	3	15.8	DE	(0.3)	6.5	C	(0.2)	15.1	cde	(0.7)	0.0	a	(0,0)
	6	16.4	EF	(0.2)	12.2	E	(0.6)	15.4	def	(0.4)	7.3	f	(0,4)

0 = not measurable, material was not cured

b) Filtek™ Supreme XTE flow

Vickers hardness, HV [N/mm²]

Curing mode	Exposure time [s]	0mm- exposure distance						7mm-exposure distance					
		top			bottom			top			bottom		
Standard	5	64.3	B	(4.6)	53.6	E	(2.8)	67.9	cd	(1,1)	27.5	d	(3.2)
	10	66.5	B	(2.6)	60.9	F	(2.1)	67.0	cd	(1.9)	44.9	f	(3.2)
	15	67.4	B	(1.0)	61.0	F	(2.1)	67.7	cd	(2.0)	58.4	h	(1.5)
	20	68.4	B	(1.5)	64.9	F,G	(1.5)	69.1	cd	(2.1)	62.4	hi	(0.9)
	40	69.7	B	(2.0)	67.8	G	(1.2)	71.0	d	(1.6)	67.2	i	(2.8)
High Power	1	53.1	A	(2.3)	9.8	A	(1.9)	30.6	a	(1.5)	3.9	a	(0.5)
	2	64.1	B	(2.9)	23.5	B	(1.4)	59.3	b	(3.2)	13.8	b	(1.4)
	3	64.8	B	(4.9)	32.0	C	(3.2)	65.1	c	(4.0)	14.4	b	(2.5)
	4	65.7	B	(3.8)	47.7	D	(3.5)	65.0	c	(3.8)	21.6	c	(1.2)
	12	67.9	B	(1.6)	65.2	F,G	(0.1)	68.5	cd	(1.6)	52.9	g	(3.1)
Plasma Emulation	3	66.5	B	(3.2)	47.3	D	(2.4)	65.2	c	(1.3)	36.8	e	(4.2)
	6	68.3	B	(2.2)	62.0	F	(2.8)	66.9	cd	(1.5)	58.7	h	(2.5)

Indentation Modulus, E [GPa]

Curing mode	Exposure time [s]	0mm- exposure distance						7mm-exposure distance					
		top			bottom			top			bottom		
Standard	5	9.6	CD	(0.3)	8.5	DE	(0.5)	9.0	cd	(0.1)	5.9	d	(0.5)
	10	9.7	CD	(0.4)	8.7	EF	(0.2)	9.3	cde	(0.2)	7.6	f	(0.3)
	15	9.8	CD	(0.3)	9.3	FG	(0.3)	9.5	def	(0.3)	8.9	gh	(0.2)
	20	9.9	D	(0.1)	9.3	FG	(0.3)	9.8	ef	(0.1)	8.8	gh	(0.3)
	40	10.1	D	(0.2)	9.9	G	(0.3)	9.9	f	(0.3)	9.4	h	(0.2)
High Power	1	7.3	A	(0.2)	2.2	A	(0.3)	5.0	a	(0.2)	1.0	a	(0.1)
	2	8.5	B	(0.6)	4.6	B	(0.3)	8.2	b	(0.6)	2.8	b	(0.1)
	3	9.1	BC	(0.7)	5.9	C	(0.6)	8.8	bc	(0.4)	3.3	b	(0.3)
	4	9.4	CD	(0.4)	7.8	D	(0.4)	9.2	cd	(0.3)	4.5	c	(0.1)
	12	9.7	CD	(0.5)	8.7	EF	(0.2)	9.5	def	(0.3)	8.4	g	(0.3)
Plasma Emulation	3	9.7	CD	(0.2)	7.9	D	(0.5)	9.3	cde	(0.2)	6.6	e	(0.4)
	6	10.1	D	(0.3)	9.4	FG	(0.4)	9.5	def	(0.3)	9.0	h	(0.4)

c) Filtek™ Silorane

Vickers hardness, HV [N/mm²]

Curing mode	Exposure time [s]	0mm- exposure distance		7mm-exposure distance	
		top	bottom	top	bottom
Standard	5	92.5 BC (4.3)	74.9 BC (1.9)	91.4 bc (2.9)	0.0 a (0.0)
	10	93.4 BCD (2.7)	85.1 DE (6.3)	92.6 bc (3.2)	69.0 b (4.8)
	15	98.5 DE (2.2)	85.3 DE (3.0)	93.9 bc (1.7)	73.6 b (2.3)
	20	98.5 DE (2.2)	86.5 DE (1.4)	96.2 cd (2.5)	73.8 b (3.3)
	40	100.0 E (3.4)	90.3 E (1.4)	99.7 d (1.0)	82.9 c (1.8)
High Power	1	83.2 A (4.0)	0.0 A (0.0)	82.3 a (5.7)	0.0 a (0.0)
	2	87.6 AB (4.3)	0.0 A (0.0)	89.4 b (2.2)	0.0 a (0.0)
	3	93.4 BCD (2.1)	69.7 B (4.8)	89.4 b (2.1)	0.0 a (0.0)
	4	94.3 CD (2.7)	76.4 C (1.9)	94.1 bc (2.3)	0.0 a (0.0)
	12	98.7 DE (2.4)	90.0 E (3.5)	94.2 bcd (2.4)	82.3 c (3.1)
Plasma Emulation	3	97.6 CDE (2.2)	83.3 D (2.8)	95.8 bcd (3.6)	73.4 b (4.0)
	6	98.2 CDE (2.4)	90.2 E (3.0)	95.8 cd (3.6)	80.9 c (4.1)

0 = not measurable, material was not cured

Indentation Modulus, E [GPa]

Curing mode	Exposure time [s]	0mm- exposure distance		7mm-exposure distance	
		top	bottom	top	bottom
Standard	5	12.3 AB (0.5)	12.0 B (0.2)	13.1 cde (0.3)	0.0 a (0.0)
	10	13.7 CDE (0.4)	12.3 B (0.8)	13.0 bcd (0.2)	12.5 b (0.7)
	15	14.1 DE (0.5)	12.4 B (0.3)	13.5 def (0.1)	13.1 bc (0.3)
	20	14.1 DE (0.5)	13.5 C (0.4)	13.5 def (0.3)	13.0 bc (0.2)
	40	14.3 E (0.5)	13.8 C (0.5)	14.3 f (0.5)	13.5 c (0.3)
High Power	1	11.7 A (0.6)	0.0 A (0.0)	11.2 a (1.2)	0.0 a (0.0)
	2	13.2 BCD (0.2)	0.0 A (0.0)	12.1 abc (1.0)	0.0 a (0.0)
	3	13.2 BCD (0.2)	11.8 B (0.9)	12.0 ab (0.5)	0.0 a (0.0)
	4	13.6 CD (0.4)	12.1 B (0.2)	13.4 de (0.3)	0.0 a (0.0)
	12	13.7 CDE (0.8)	13.8 C (0.5)	14.2 ef (0.3)	12.7 b (0.8)
Plasma Emulation	3	12.9 BC (0.5)	13.3 C (0.3)	13.2 def (0.7)	12.8 b (0.4)
	6	13.5 CDE (0.4)	13.5 C (0.2)	13.6 def (0.4)	13.5 c (0.2)

0 = not measurable, material was not cured

that the LCU is placed directly and perpendicularly on the restoration, which would be considered an ideal situation. Compared to the effect of the exposure time or location of measurement (top-bottom) the effect of the exposure distance was low in all materials.

This effect corroborates well with the identified low effect of the exposure distance on the transmitted irradiance through the same materials and specimen geometries [8]. Moreover, the influence of the exposure distance was lower at the surface than at the bottom, and larger in the higher filled RBC Filtek ™ Supreme XTE, which is also related to the attenuation of light [8].

A further aspect analyzed in the present study is the effect of the chemical composition of the organic matrix at a given filler amount or the viscosity of the material at a given chemical composition of the organic matrix, on the material's susceptibility to different curing conditions. To analyze this aspect, all

RBCs were chosen in the same shade, A3. This supposes that differences in the material reaction to different curing conditions are related to the chemical composition of the individual ingredients, the filler size and the proportional relation between filler and organic matrix.
A further component that may have an influence on the amount of light transmitted in the depth of the specimen is the initiator. While there is less information about the amount and exact composition of the initiator system, it is likely that both methacrylate-based RBCs are based on the same initiator, a camphorquinone/amine system. As for the silorane material, the initiator is camphorquinone as well, but it contains in addition an iodonium salt and aromatic amines [19] that may have an effect in light transmission as well.
The lower filler amount in the flowable RBC lead to the significant highest light transmission at the bottom of the specimens [8] when compared to the higher filled methacrylate-based RBC. This fact is clearly reflected in the present study in a lower susceptibility of the flowable material to variations in light exposure. A difference of 8.3 vol. % in filler amount resulted in significantly lower light transmittance and a ca. 50% higher absorbance in the higher filled methacrylate-based RBC [8].
This is directly related to the identified insufficient polymerization at the bottom of the 2-mm thick specimens of the last-mentioned material, also at high exposure times.
At a given filler amount, 55 vol-%, the silorane-based RBCs was less translucent than the flowable methacrylate-based RBCs, but the difference in the light transmittance and absorbance characteristics of both materials was lower compared to the differences related to the higher filled methacrylate-based RBC [8].
The reasons for the different light transmittance at a similar filler volume amount is related, besides the abovementioned differences in the type of initiator, to the monomer reactivity and differences in refractive index between fillers and organic matrix as well as to the filler size and chemical composition [20,21]. It must also be emphasized that the micro-mechanical properties measured in the silorane material were superior to the values measured in the flowable methacrylate-based flowable material at a similar filler volume content. This may also be related, besides the filler type, to the high crosslinking density of the final silorane-polymer, as the silorane monomer, which was obtained from the reaction of oxirane and siloxane molecules, is a four-branched monomer [19].
The presented data allow rejecting all specified null hypothesis.

5. Conclusions

The analyzed RBCs react differently to the analyzed curing conditions depending on their structure and composition. It can therefore be concluded that:

- The susceptibility to various curing conditions is material-dependent, while less filled methacrylate-based as well as the silorane micro-hybrid seems to be more robust to these variations.
- The indentation modulus, E, reacts more sensitively to variations in curing conditions then the hardness, HV, thus being a better indicator in the characterization of changes in polymerization.
- Within the bounds of this study, exposure time has a significant stronger effect on the micro-mechanical properties than irradiance. The influence of radiant exposure, which is the product of exposure time and irradiance, was lower compared to the effect of exposure time. The effect of exposure distance was by comparison much lower.
- Fast polymerization (3s) with high irradiance is not recommended for the analyzed materials. The doubled exposure time (6s) at high irradiance proved to be insufficient for the methacrylate based RBCs, while acceptable for the silorane material.
- On the basis of these findings, under comparable clinical conditions, a curing time of at least 20s at moderate irradiance is likely to be recommended for the majority of the tested materials, providing clinical conditions are comparable to those in the described study.

Author Contributions

NI: designed the study, provided the infrastructure (devices, materials), developed the measurement methods, supervised the experiment, performed statistics, and wrote the manuscript. EP: performed the experiments and recorded the data.

References

1. Park J, Chang J, Ferracane J, Lee IB. How should composite be layered to reduce shrinkage stress: incremental or bulk filling? *Dent Mater.* 2008;24(11):1501-1505.
[Full text links] [CrossRef] [PubMed] Google Scholar
2. Musanje L, Darvell BW. Polymerization of resin composite restorative materials: exposure reciprocity. *Dent Mater.* 2003;19(6):531-541.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
3. Musanje L, Darvell BW. Curing-light attenuation in filled-resin restorative materials. *Dent Mater.* 2006;22(9):804-817.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
4. Rueggeberg FA, Caughman WF, Curtis JW, Jr. Effect of light intensity and exposure duration on cure of resin composite. *Oper Dent.* 1994;19(1):26-32.
[PubMed] Google Scholar Scopus
5. Sobrinho LC, Goes MF, Consani S, et al. Correlation between light intensity and exposure time on the hardness of composite resin. *J Mater Sci Mater Med.* 2000;11(6):361-364.
[PubMed] Google Scholar
6. Ilie N, Hilton TJ, Heintze SD, et al. Academy of Dental Materials guidance-Resin composites: Part I-Mechanical properties. *Dent Mater.* 2017;33(8):880-894.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
7. Ferracane JL, Hilton TJ, Stansbury JW, et al. Academy of Dental Materials guidance-Resin composites: Part II-Technique sensitivity (handling, polymerization, dimensional changes). *Dent Mater.* 2017;33(11):1171-1191.
[Full text links] [CrossRef] [PubMed] Google Scholar
8. Ilie N, Plenk E. Light transmission through resin composites. *Stoma Edu J.* 2018;5(3):148-154. [CrossRef]

9. Ilie N, Furtos G. A comparative study of light transmission through various dental restorative materials and the tooth structure. *Oper Dent* - in revision. 2019.
10. *DIN-50359-1*. Testing of metallic materials - Universal hardness test - Part 1: Test method. 1997.
11. Ilie N, Bauer H, Draenert M, Hickel R. Resin-based composite light-cured properties assessed by laboratory standards and simulated clinical conditions. *Oper Dent*. 2013;38(2):159-167.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
12. Rueggeberg FA. State-of-the-art: dental photocuring--a review. *Dent Mater.* 2011;27(1):39-52.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
13. Deb S, Sehmi H. A comparative study of the properties of dental resin composites polymerized with plasma and halogen light. *Dent Mater.* 2003;19(6):517-522.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
14. Kim JW, Jang KT, Lee SH, et al. Effect of curing method and curing time on the microhardness and wear of pit and fissure sealants. *Dent Mater.* 2002;18(2):120-127.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
15. Joly GD, Abuelyaman AS, Fornof AR, et al. Dental compositions comprising addition-fragmentation agents. *US Patent* 9414996B2. 2016.
16. Ilie N. Sufficiency of curing in high-viscosity bulk-fill resin composites with enhanced opacity. *Clin Oral Investig.* 2019;23(2):747-755.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
17. Ilie N, Jelen E, Hickel R. Is the soft-start polymerisation concept still relevant for modern curing units? *Clin Oral Investig.* 2011;15(1):21-29.
[Full text links] [CrossRef] [PubMed] Google Scholar
18. Price RB, Derand T, Sedarous M, et al. Effect of distance on the power density from two light guides. *J Esthet Dent.* 2000;12(6):320-327.
[CrossRef] [PubMed] Google Scholar Scopus
19. Weinmann W, Thalacker C, Guggenberger R. Siloranes in dental composites. *Dent Mater.* 2005;21(1):68-74.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
20. Palin WM, Leprince JG, Hadis MA. Shining a light on high volume photocurable materials. *Dent Mater*. 2018;34(5):695-710.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus
21. Shortall AC, Palin WM, Burtscher P. Refractive index mismatch and monomer reactivity influence composite curing depth. *J Dent Res.* 2008;87(1):84-88.
[Full text links] [CrossRef] [PubMed] Google Scholar Scopus

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Original Articles

Questions

1. The highest influence on the micro-mechanical properties of a light cured resin-based composite (RBC) is exerted by:

❑a. Exposure time;
❑b. Exposure distance;
❑c. Incident irradiance;
❑d. LCU's irradiance.

2. When curing a RBC, following in valid:

❑a. Fast polymerization (3s) with high irradiance is recommended, to speed up the restoration procedure;
❑b. A curing time of at least 20 s at moderate irradiance;
❑c. A lower increment can only be pre-cured for few seconds, since it will receive sufficient light while curing the upper increments;
❑d. All RBCs react similar to variations in radiant exposure.

3. What are "Silorane"?

❑a. Resin-based composites with particular glass ionomer filler;
❑b. Resin-based composites designed to release ions like F;
❑c. Four branched monomer obtained from the reaction of oxirane and siloxane molecules;
❑d. A sort of ceramic.

4. At a given composition of the filler and matrix in a RBC, an enhanced filler amount will result in:

❑a. Higher mechanical properties;
❑b. Higher light transmittance;
❑c. Lower susceptibility to variation in irradiation;
❑d. Better polymerization at the bottom of the 2 mm increments.