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Laboratory Research

Resin-based Composite Light-cured Properties Assessed by Laboratory Standards and Simulated Clinical Conditions

N Ilie • H Bauer • M Draenert
R Hickel

Clinical Relevance

The irradiation technique, used to measure mechanical properties of resin-based composites according to international standards, consistently differs from clinically simulated conditions, calling into question whether laboratory findings can be unrestrictedly applied clinically, especially at short polymerization times. The study analyzes whether degree of conversion measurements at short post-polymerization time (five minutes) are able to predict the long-term material behavior.

SUMMARY

The following parameters were varied: 1) irradiation technique: top and bottom polymerization according to the ISO standard, and polymerization from only the top, simulating clinical situations; 2) polymerization time: 5, 10, 20, and 40 seconds; 3) storage conditions: 24 hours in distilled water, thermocycling followed by storage for four weeks in artificial saliva or alcohol. Flexural strength (FS), flexural modulus ($E_{flexural}$), indentation modulus (E), Vickers hardness (HV), and degree of conversion (DC) were measured.

The laboratory results were similar to those measured by mimicking clinical conditions only at high polymerization times and mild storage conditions (20 seconds and 40 seconds and storage for 24 hours in water, and 40 seconds with aging and storing in saliva). Significantly higher DC values were measured on the top than on the bottom of a 2-mm layer for all polymerization times. Overall, 5-second

*Nicoleta Ilie, PhD, Dental School of the Ludwig-Maximilians-University, Department of Restorative Dentistry, Munich, Germany

Henrik Bauer, dentist, Dental School of the Ludwig-Maximilians-University, Department of Restorative Dentistry, Munich, Germany

Miriam Draenert, DMD, Dental School of the Ludwig-Maximilians-University, Department of Restorative Dentistry, Munich, Germany

Reinhard Hickel, DMD, professor and dean of the Dental School of the Ludwig-Maximilians-University, Department of Restorative Dentistry, Munich, Germany

*Corresponding author: Goethe-Str 70, Munich 80336 Germany; e-mail: nilie@dent.med.uni-muenchen.de

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and 10-second irradiation times induced significantly lower DC values compared to the currently recommended polymerization times of 20 and 40 seconds at both the top and bottom of the samples.

The initial DC differences as a function of irradiation time are leveled at 24 hours of storage but seem to do well in predicting long-term material behavior. A minimum irradiation time of 20 seconds is necessary clinically to achieve the best mechanical properties with modern high-intensity light emitting diode (LED) units.

INTRODUCTION

Dentists' requests for a short chair time to prepare a restoration compel a continual reduction in polymerization time for curing resin-based composites (RBCs). When the first high-intensity visible light plasma arc curing (PAC) units were introduced in 1998, the manufacturer claimed to be able to fulfill these wishes, declaring a 3-second polymerization time as sufficient for an adequate polymerization. The manufacturer also claimed that curing for 3 seconds with a high-intensity PAC unit is equivalent to a 40- or 60-second exposure to a quartz-tungsten-halogen (QTH) light.[1] These statements were based on the radiant energy (dose) concept, calculated as a simple reciprocal relationship between irradiation and irradiation time, suggesting that the irradiation time can be shortened when a curing unit's irradiance is proportionally increased. Though generally valid, the radiant energy concept was put into perspective because, within a given dose, shorter exposure time at higher intensity proved to induce decreased properties (lower degree of cure, polymer cross-linking, and physical properties).[2,3] It was also shown that a polymerization of 3 seconds with the high-intensity PAC unit is too short; thus, multiple 3-second exposures are recommended for a clinically adequate performance.[4]

Due to consistent improvements in light emitting diode (LED) technology, a great increase in output power has been achieved in the second and third generation LED units,[1] again suggesting a shortening of irradiation time. While 20-second or 40-second irradiation times with modern LED units are still indicated by many manufacturers, some have recently suggested short polymerization times of up to 5 seconds.

Although time-saving, a fast cure with a high level of energy has a number of drawbacks, including increased polymerization shrinkage stress,[5,6] which is generally related to several negative clinical effects, such as less integrity of the restoration-cavity interface,[7] marginal staining, cusp fractures,[8], microleakage,[9] secondary caries, postoperative sensitivity, or pain. Also, a rise in temperature and risk of pulp damage[10] are associated with the use of curing units with very high intensity.

The quality of polymerization in RBCs is preliminarily assessed in laboratory tests. To measure macromechanical properties such as flexural strength, international standards (ISO 4049: 2009[11]) require curing 2-mm thick slabs from both sides, top and bottom. This double polymerization might generate good mechanical properties with low polymerization times, which could considerably differ from a clinical situation where curing occurs mainly from the top of a restoration. Additionally, the storage time for standard measurements is generally set at 24 hours, raising the question of the role of aging, especially in connection with low polymerization times.

Our study aimed to analyze the effect of aging on the macromechanical and micromechanical properties of a nano-hybrid RBC by varying the polymerization time, the irradiation technique, and the storage conditions. Furthermore, the variation in degree of cure as a function of polymerization time and position—top or bottom of the samples—is analyzed.

The null hypotheses tested in our study were as follows: 1) the irradiation technique—polymerization from both sides, top and bottom, according to ISO standards, and polymerization only from the top, simulating a clinical situation—would not affect the macromechanical (flexural strength and modulus) and micromechanical properties (Vickers hardness and modulus of elasticity); 2) the above mentioned mechanical properties and the degree of cure would not be influenced by the irradiation time and measuring location (top or bottom); 3) aging (24 hours in distilled water, thermocycling followed by storage for four weeks in artificial saliva or alcohol) would have no influence on the mechanical properties; and 4) the aging agent—saliva or alcohol solution—would not influence the mechanical properties.

MATERIALS AND METHODS

A nano-hybrid resin-based composite (IPS Empress Direct Dentin, Ivoclar Vivadent, Batch No. M68447, dimethacrylate resin matrix, Ba-Al-Si-glass, YbF_3,

and SiO_2/ZrO_2-mixed oxide fillers, 75 wt%, 53 vol%) was analyzed by varying the direction of irradiation (top and bottom or only top), the irradiation time (5, 10, 20, and 40 seconds), the storing conditions (24 hours in distilled water or thermocycling followed by storage for four weeks in an aging medium) and the storage medium for aging (artificial saliva or alcohol). A total of 24 combinations of the above mentioned parameters were studied.

The flexural strength (FS) and flexural modulus ($E_{flexural}$) were determined in a three-point-bending test (n=15). Therefore, 360 samples were made by compressing the composite material between two glass plates with intermediate polyacetate sheets, separated by a steel mold having an internal dimension of $2 \times 2 \times 16$ mm. Irradiation occurred in two different ways: once on the top and bottom of the specimens, as specified in ISO 4049:2009 standards,[11] and once only on top, simulating a clinical situation. When testing, the load was applied to the top surface of these specimens. The times of the light exposures were 5, 10, 20, and 40 seconds, with three light exposures, overlapping one irradiated section no more than 1 mm of the diameter of the light guide (Elipar Freelight 2, 3M ESPE, Seefeld, Germany) to prevent multiple polymerizations. The irradiance of the curing unit (1241 mW/cm^2) was measured by means of a calibrated fiber optic spectrally resolving radiometer equipped with an integrating sphere (S2000, Ocean Optics, Dunedin, FL, USA). To assess possible variations in irradiation, a calibrated spectrometer (MARC, BlueLight analytics inc, Halifax, Canada) was used at the beginning, middle, and end of a sample preparation session.

After removal from the mold, the specimens were ground with silicon carbide sand paper (grit size P 1200/4000 [Leco]) to remove disturbing edges or bulges and stored for 24 hours in distilled water at 37°C. One third of the specimens were subsequently measured and considered as references, the rest were additionally aged (thermocycling for 5000 cycles at 5°C to 55°C) before storage for four weeks at 37°C in artificial saliva or a 1:1 ethanol-water mixture. The samples were loaded until failure in a universal testing machine (Z 2.5, Zwick/Roell, Ulm, Germany) in a three-point bending test device, which was constructed according to the guidelines of NIST No. 4877 with a 12-mm distance between the supports.[12] During testing, the specimens were immersed in distilled water at room temperature. The crosshead speed was 0.5 mm/min. The universal testing machine measured the force during bending as a function of deflection of the beam. The bending modulus was calculated from the slope of the linear part of the force-deflection diagram.

Micromechanical Properties

Fragments (n=12) of the three-point bending test specimens of each group were used to determine the micromechanical properties: Vickers hardness (HV) and indentation modulus (E) according to DIN 50359-1:1997[13] by means of a universal-hardness device (Fischerscope H100C, Fischer, Sindelfingen, Germany). Prior to testing, the samples were polished with a grinding system (EXAKT 400 CS, EXAKT, Norderstedt, Germany) using silicon carbide paper (P 2500 followed by P 4000). Measurements were done on the top (n=6) and the bottom (n=6) of the slabs (10 measurements per sample per side). The test procedure was carried out with controlled force; the test load increased and decreased with a 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 and Vickers hardness was calculated and implemented in the software, such that the measurement results were indicated in the more familiar HV units. The indentation modulus E was calculated from the slope of the tangent of indentation depth-curve at maximum force.

Degree of Cure

The degree of cure (DC) was analyzed by considering two different sample geometries: one 2-mm high increment measured in a white Teflon mold measuring 2 mm in height and 3 mm in diameter and a thin 100-μm composite film. The samples were cured for 5, 10, 20, and 40 seconds by applying the curing unit directly on the sample's surface (n=5). Measurements were made in real time (five minutes, 2 spectra/s, 4 cm^{-1} resolution) with a Fourier transform infrared (FTIR) spectrometer with an attenuated total reflectance (ATR) accessory (Nexus, Thermo Nicolet, Madison, WI, USA) by applying the nonpolymerized composite paste directly on the diamond ATR crystal. The spectra were recorded in this way at the bottom of the samples.

DC was calculated as the variation in peak height ratio of the absorbance intensities of methacrylate carbon double bond peak at 1634 cm^{-1} and that of internal standard peak at 1608 cm^{-1} (aromatic

carbon double bond) during polymerization, in relation to the uncured material.

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

Statistical Analysis

The Kolmogorov-Smirnoff test was applied to verify that the data were normally distributed. The results were compared using one- and multiple-way analysis of variance (ANOVA) and Tukey honestly significant difference (HSD) post hoc-test (α=0.05). A multivariate analysis (general linear model with partial eta-squared statistics) assessed the effect of storage, irradiation time, and irradiation mode (ISO/clinical) on the considered properties. An independent *t*-test additionally analyzed the differences in mechanical properties as a function of irradiation technique (SPSS, version 19.0, SPSS Inc, Chicago, IL, USA). An additional Weibull analysis was performed for the flexural strength data.

A common empirical expression for the cumulative probability of failure P at applied stress is the Weibull model:

$$P_f(\sigma_c) = 1 - \exp\left[-\left(\frac{\sigma_c}{\sigma_0}\right)^m\right].$$

where σ_c is the measured strength, m is the Weibull modulus and σ_0 is the characteristic strength, which is defined as the uniform stress at which the probability of failure is 0.63. The double logarithm of this expression gives:

$$\ln\ln\frac{1}{1-P} = m\ln\sigma_c - m\ln\sigma_0$$

By plotting ln ln(1/(1–P)) vs ln sigma, a straight line results with the upward gradient m.

RESULTS

Post-hoc multiple pairwise comparisons with Tukey HSD test ($p<0.05$) showed a decrease in the parameters measured in the flexural strength test (FS, $E_{flexural}$, Figure 1; Table 1) after aging, with a considerable decrease in the properties after storing the samples in alcohol. Preparing the samples by mimicking clinical conditions weakened the properties for almost all polymerization times and storage conditions compared to the groups prepared according to the international standard ($p< 0.05$). Only a high polymerization time and a mild storage condition (20 seconds and 40 seconds with 24-hour water storage, and 40 seconds with aging and saliva storage) were able to equalize these differences.

Irrespective of irradiation time and storage, the difference between the micromechanical properties measured on the top and bottom of the samples was not statistically significant in the samples polymerized from both sides but was significantly lower at the bottom of the samples polymerized only from the top (Table 2).

As for the degree of cure, significantly higher values were measured for all polymerization times on the top of the samples, simulated by the use of a thin composite film, as in a depth of 2 mm ($p< 0.05$) (Figure 2; Table 3). For both conditions, 5-second and 10-second irradiation times induced significantly lower DC values compared to the currently recommended polymerization times of 20 seconds or 40 seconds.

The storage proved to have the greatest influence on all measured mechanical properties (Table 4), exercising the strongest effect on the material's reliability, expressed by the Weibull modulus, m. The effect of irradiation time was greater than the effect of irradiation technique with regard to the parameters measured in the flexural test; both effects were comparable with regard to the micromechanical properties.

The modulus of elasticity measured in both methods—the flexural test and the universal hardness test—correlated well (Pearson correlation coefficient = 0.8). There was also a good correlation between the micromechanical properties (E-HV = 0.97) and macromechanical properties (FS-$E_{flexural}$ = 0.81).

DISCUSSION

Laboratory experiments try to simulate clinical conditions as accurately as possible to obtain information that is directly applicable in daily practice. Therefore, our study compared two irradiation techniques—top and bottom, according to the ISO standard, and only top, to simulate clinical situations. The mechanical properties were assessed on a macroscopic scale, by determining the strength and the resistance the materials opposed to deformation (modulus of elasticity), and on a microscopic scale (hardness and modulus of elasticity) by means of a dynamic measuring principle, simultaneously recording the load and the corresponding penetration depth of the indenter.[13] With maximum indentation

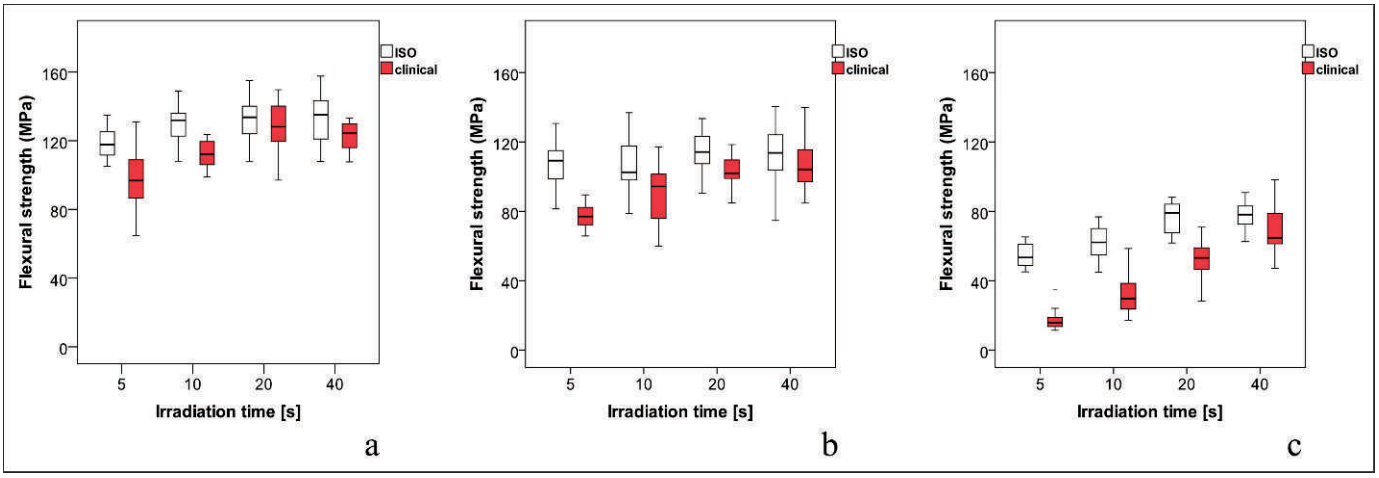

Figure 1. *Variation of flexural strength with irradiation time and irradiation technique after: 1) water storage for 24 hours; 2) thermocycling followed by storage in saliva for four weeks; and 3) thermocycling followed by storage in alcohol for four weeks.*

Table 1: *Flexural Strength (FS) and Modulus of Elasticity in Flexural Test ($E_{flexural}$) are Detailed in Mean Values and Standard Deviations (in Parentheses)*[a]

Storage	Time, s	FS, MPa		*p*	Weibull, *m*		$E_{flexural}$, GPa		*p*
		ISO	Clinical		ISO	Clinical	ISO	Clinical	
24h water	5	118.7^{de} (9.3)	98^{FG} (9.8)	< 0.001	4.09	2.81	4.7^{c} (0.7)	3.4^{C} (0.6)	< 0.001
	10	129.7^{e} (11.9)	110.6^{GH} (12.3)	< 0.001	4.28	3.23	5.6^{d} (0.6)	4.1^{D} (0.7)	< 0.001
	20	132.1^{e} (13.7)	128.5^{I} (14.5)	0.491	4.00	3.14	5.3^{cd} (0.9)	5.3^{EF} (0.7)	0.843
	40	133.1^{e} (15.3)	122.8^{HI} (8.4)	0.051	4.12	3.37	5.2^{cd} (0.8)	5.4^{F} (0.6)	0.349
Thermocycling + 4w saliva	5	105.9^{d} (15.8)	78.4^{DE} (8.9)	< 0.001	2.48	2.79	5.2^{cd} (0.6)	4.1^{D} (0.3)	< 0.001
	10	106.8^{d} (16.0)	90.4^{EF} (17.8)	0.012	2.22	2.08	5.2^{cd} (0.6)	4.7^{E} (0.4)	0.048
	20	113.4^{d} (13.0)	103.0^{FG} (10.8)	0.017	3.38	3.20	5.5^{d} (0.4)	5.3^{EF} (0.3)	0.036
	40	112.8^{d} (16.6)	106.7^{G} (14.5)	0.277	2.31	2.91	5.6^{d} (0.5)	5.7^{F} (0.4)	0.828
Thermocycling + 4w alcohol	5	53.1^{a} (8.9)	17.3^{A} (5.8)	< 0.001	1.95	0.94	2.5^{a} (0.2)	1.6^{A} (0.3)	< 0.001
	10	62.0^{ab} (9.2)	33.0^{B} (12.8)	< 0.001	2.32	0.80	2.7^{ab} (0.4)	2.3^{B} (0.3)	0.006
	20	76.3^{bc} (9.3)	51.4^{C} (10.6)	< 0.001	2.65	1.59	3.2^{ab} (0.4)	2.4^{B} (0.3)	< 0.001
	40	77.1^{c} (7.8)	69.0^{D} (13.2)	0.040	3.30	1.77	3.2^{b} (0.2)	3.2^{C} (0.2)	0.829

[a] *Superscript letters indicate statistically homogeneous subgroups within a column (Tukey HSD test, $\alpha = 0.05$). A* t*-test analyzed differences between the way of curing the samples—ISO or clinical—for each irradiation time. The Weibull parameter* m *is indicated.*

Table 2: *Micromechanical Properties—Modulus of Elasticity (E) and Vickers Hardness (HV)—Are Detailed in Mean Values and Standard Deviations (in Parentheses)*[a]

Storage	Time, s	E, GPa			HV, N/mm^2		
		ISO	Clinical Top	Clinical Bottom	ISO	Clinical Top	Clinical Bottom
24h Water	5	12.4^{e*} (0.5)	12.4^{E*} (0.4)	6.8^{b} (2.1)	66.6^{e} * (4.5)	67.5^{E*} (4.9)	28.3^{a} (13.1)
	10	13.1^{g} (0.3)	12.4 ^{E} (1.6)	11.8^{ef} (0.8)	73.5^{g} * (1.7)	71.7^{F*} (6.5)	63.3^{g} (6.8)
	20	13.1^{g*} (0.3)	13.0^{FG*} (1.2)	12.4^{fg} (0.3)	74.8^{g} * (2.6)	76.0^{G*} (6.1)	67.2^{g} (2.8)
	40	13.6^{d} (0.3)	13.3^{G} (0.5)	12.8^{g} (0.3)	76.6^{h} (2.3)	75.0^{G} (3.4)	72.0^{h} (2.4)
Thermocycling + 4w saliva	5	12.6^{ef} (0.4)	11.4^{D*} (0.6)	8.1^{c**} (0.9)	68.5^{f} (2.2)	58.4^{D*} (4.1)	33.8^{bc**} (6.8)
	10	13.0^{g} (0.4)	12.4 ^{E*} (0.4)	9.8^{d**} (1.0)	70.2^{f} (4.8)	65.5^{E*} (4.1)	44.3^{e**} (8.9)
	20	12.9^{g} (0.4)	13.0^{FG*} (0.3)	11.2^{e**} (0.4)	69.2^{f} (3.5)	72.4^{F*} (1.8)	58.0^{f**} (3.8)
	40	13.1^{g} (0.4)	12.6^{EF*} (0.5)	11.3^{e**} (0.6)	73.3^{g} (2.1)	67.4^{E*} (4.5)	57.0^{f**} (6.1)
Thermocycling + 4w alcohol	5	7.1^{a*} (0.4)	7.2^{A*} (0.4)	5.9^{a} (0.3)	38.5^{a*} (2.3)	37.9^{A*} (4.1)	31.6^{ab} (2.9)
	10	7.6^{b*} (0.3)	8.0^{B*} (0.5)	6.9^{b} (0.3)	41.5^{b*} (3.0)	43.9^{B*} (2.4)	37.0^{cd} (2.3)
	20	8.4^{c*} (0.3)	8.5^{C*} (0.4)	7.1^{b} (0.4)	48.2^{c*} (1.6)	48.7^{C*} (2.5)	40.5^{de} (2.4)
	40	9.3^{d*} (0.5)	8.3^{BC*} (0.5)	7.3^{b} (0.3)	52.7^{d*} (2.7)	45.8^{B*} (3.7)	40.4^{de} (2.9)

[a] *Superscript letters indicate statistically homogeneous subgroups within a column and asterisks(*) indicate statistically homogeneous subgroups within a line. (Tukey HSD test, $\alpha = 0.05$). For the ISO way of cure, no statistical differences between top and bottom were measured.*

depth in the range of 7-15 μm, the measurements performed at microscopic scale reflect the material properties and not the properties of the individual material components (filler and filler agglomerates or matrix and matrix-reach areas). As for the degree of cure, the measurements were done at the same depth as the measurement of micromechanical properties, by simulating the top and bottom of clinically relevant 2-mm-thick increments, allowing thus a direct comparison among the measured parameters.

The results demonstrated that when samples were stored for 24 hours, the irradiation technique significantly affected the mechanical properties at short irradiation times (5 seconds and 10 seconds), lowering FS and $E_{flexural}$ in simulated clinical conditions when compared to the ISO standard, but showed similar results for both polymerization techniques at high irradiation times (20 seconds and 40 seconds). These results support the recommended irradiation time of at least 20 seconds, as indicated by the manufacturer and also often used in ISO 4049:2009,[11] for both *in vitro* and *in vivo* use. Within the above mentioned curing and storage conditions—irradiation of at least 20 seconds and storage for 24 hours in distilled water—the results of the ISO-irradiation can be directly applied clinically, since both tested irradiation techniques produced not only similar macromechanical properties (FS, $E_{flexural}$) but also similar micromechanical properties (HV, E). After aging and storing in artificial saliva, a polymerization time of 40 seconds is necessary to ensure statistically similar properties between the ISO and the clinically simulated irradiation, whereas storing in alcohol never produced comparable results (40 seconds). These results put in perspective whether the mechanical properties measured according to the current standards are able to reflect

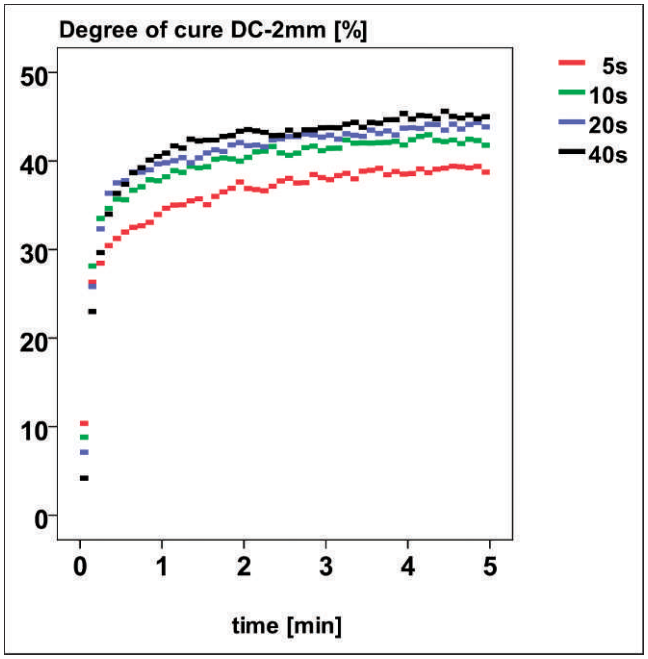

Figure 2. *Degree of cure measured at 2-mm depth in real-time for five minutes, as a function of polymerization time (mean values, n=5).*

the long-term behavior of an RBC filling and confirm the lack of correlation between the clinical behavior of restorative materials and laboratory results.[14] This statement should not, however, diminish the meaning of preliminary laboratory tests and standardized methods, since an initial selection of materials with adequate properties is of particular importance.[15]

Table 3: *Degree of Cure (DC) at 0.1 mm and 2 mm Are Detailed in Mean Values and Standard Deviations (in Parentheses)*[a].

Time, s	$DC_{0.1mm}$, %	DC_{2mm}, %	*p*
5	45.3[a] (3.5)	38.6[A] (2.8)	< 0.001
10	46.5[a] (3.0)	41.8[B] (2.3)	< 0.001
20	48.7[b] (2.4)	43.9[C] (1.5)	< 0.001
40	49.6[b] (4.2)	45.0[C] (2.7)	0.005

[a] *Superscript letters indicate statistically homogeneous subgroups within a column (Tukey HSD test, α=0.05). A t-test analyzed differences between the DC at 0.1 and 2 mm for each irradiation time.*

Table 4: *Influence of Storage, Irradiation Time and Way of Curing on Flexural Strength (FS), Modulus of Elasticity in Flexural Test ($E_{flexural}$), and Weibull parameter* m*, Vickers hardness (HV), and Modulus of Elasticity (E), as Well as Degree of Cure (DC)*[a]

Parameters	FS	$E_{flexural}$	*m*	HV	E	DC
Storage	.842	.829	.973	.825	.889	
Irradiation time	.402	.409	.776	.531	.483	.292
ISO/clinical	.342	.221	.901	.534	.490	.313

[a] *The higher the partial eta-squared values the higher is the influence of the selected variables on the measured properties.*

The study also showed that the most sensitive parameter to all of the above mentioned influences (Table 4, highest eta-squared values) was the Weibull modulus *m*, that means the reliability of the tested material, being lower in the samples cured by simulating a clinical situation compared to the groups polymerized according to the ISO standards, for all storage conditions. Furthermore, the material reliability was lowered with aging and with increased aggressiveness of the storage agent. Furthermore, not only curing time and irradiation condition but also the storage conditions and, thus, the softening effect due to aging and storage in saliva or alcohol were more strongly reflected in the reliability determined at a macroscopic scale (Weibull parameter *m*) than in the micromechanical properties E and HV. These observations highlight the importance of performing macromechanical tests with a higher number of samples to allow performance of a Weibull statistical analysis for acquiring sensitive and reliable observation on a material's behavior.

The surface of a restoration was simulated in our study by a 100-μm RBC layer used to assess the evolution of degree of cure as a function of irradiation time in real time. The measurements were done at the bottom of the film to avoid the influence of the oxygen-inhibition layer, which was shown to be less than 20 μm thick.[16] The micromechanical properties were also measured after the oxygen-inhibition layer was eliminated by grinding and polishing; thus, both tests were recorded at similar depth. Differences in DC between the 0.1-mm and 2-mm depths were statistically significant for all irradiation times. The

postpolymerization, however, seems to have leveled these differences for longer irradiation times (20 seconds, 40 seconds), as reflected in the mechanical properties (top-bottom) measured 24 hours after storage in water. However, aging again emphasizes the importance of assessing the initial degree of cure since, especially after aging and storage in alcohol solution, the difference in micromechanical properties between the top and bottom of the samples cured from only one side became evident at all polymerization times. Similar trends were found also for the flexural strength, when both irradiation techniques were compared. Since softening tests in solvents such as ethanol and water are well-established methods of assessing the cross-link density of a polymer network,[17,18] the aging and storage in ethanol performed in this study can be taken as an indicator of the effect of reduced polymerization time as well as attenuated light at the bottom of the specimens. The softening effect of solvents was shown to be generally stronger in a more linear polymer structure than in a highly cross-linked polymer,[19] emphasizing the negative effect of short curing time on the measured mechanical properties. This statement is also consolidated by the measured degree of cure.

The results of the present study also validate the literature indicating that the minimum radiant energies necessary to properly cure RBCs are 16.8 J/cm^2 for a 1-mm increment[20] and 24 J/cm^2 for 2-mm increments.[21] Under the study conditions (light intensity 1241 mW/cm^2, polymerization time 5, 10, 20, and 40 seconds; 2-mm increments), this minimum irradiation was reached only for polymerization times of 20 seconds and 40 seconds.

The statements of the study are limited by having analyzed only one RBC. Though we randomly chose a modern nano-hybrid RBC with moderate mechanical properties,[22] the large diversity of RBCs, which contain complex fillers, organic matrices, and initiator systems, make it difficult to generalize the results but gives at least a reference note for the complex impact of polymerization on material behavior.

CONCLUSIONS

All tested null hypotheses are rejected. The properties measured according to ISO standards were similar to those measured by mimicking clinical conditions only at high polymerization times and mild storage conditions (20 seconds and 40 seconds with 24-hour water storage, and 40 seconds with aging and storing in saliva). The initial (5-minute) differences in DC measurements as a function of irradiation time are leveled at 24 hours of storage but seem to be a good indicator of the long-term material behavior.

A minimum irradiation time of 20 seconds is clinically necessary to achieve the best mechanical properties, also when modern high-intensity LED units are used.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service and/or company that is presented in this article.

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