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ORIGINAL ARTICLE

Depth of cure and mechanical properties of nano-hybrid resin-based composites with novel and conventional matrix formulation

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Abstract
Objectives This study's purpose was to evaluate the depth of cure (DOC) and the variation of mechanical properties with depth of two nano-hybrid resin-based composites (RBCs) containing a novel monomer composition based on dimer-acid derivatives (h-Da) or rather tricyclodecane–urethane structure (TCD-urethane) compared to three conventionally formulated nano-hybrid RBCs based on hardness-profile measurements.
Materials and methods Specimens were produced through different layering techniques (bulk, incremental) and curing times (10, 20, and 40 s). Mechanical properties (Vickers hardness (HV), modulus of elasticity (E)) were evaluated every 100 μm longitudinally throughout the bisected samples using an automatic micro-hardness indenter. DOC was determined as the depth at which the 80% hardness cutoff value in relation to the surface hardness was reached. Results were compared using one- and multiple-way ANOVA, Tukey HSD post-hoc test (α=0.05) and partial eta-squared statistic.
Results Increasing curing time resulted in a significant increase in DOC. Generally, the novel-formulated materials showed higher DOC values. "Curing time" and "material" showed the strongest effect on DOC. Starting in 4 mm depth, significantly higher HV and E was reached for incremental compared to bulk-curing technique. Values in 0.1 and 2 mm depth (bulk, incremental) as well as in 4 mm depth (incremental) were independent from curing time, while in greater depths, values generally increased with curing time. "Filling technique" and "material" performed the strongest influence on mechanical properties.
Conclusions Within the limits of this study, the novel-formulated RBCs showed better performance concerning DOC compared to conventional materials.
Clinical relevance For cavities deeper than 3 mm, all tested materials should be placed incrementally to ensure adequate polymerization. In large cavities (≥6 mm), the lowest increment should be cured at least 40 s. The novel-formulated RBCs might be cured in comparatively bigger increments.

Keywords Depth of cure · Nano-hybrid resin-based composites · Dimer-acid derivatives · Tricyclodecane-urethane

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Introduction

The incorporation of nanotechnology may be regarded as one of the most recent advancements in the development of resin-based composites (RBCs). Compared to conventional micro-hybrid RBCs, the application of nano-scaled filler particles in RBCs leads to an increased filler volume which may result in a reduction of polymerization shrinkage [1] as well as in improved mechanical properties [1–3]. Furthermore, the nano-filler particles contribute to better polishability [4] as well as higher aesthetical performance since the human's eye is unable to detect particles which are smaller in size than the wavelength of visible light [2]. Compared to micro-hybrid RBCs, nano-hybrid materials are also suggested to generally imply higher translucency [5]. As a consequence, modern nano-hybrid RBCs are proposed as universal restorative dental materials in a wide range of applications.

The successful usage of RBCs requires high mechanical performance, particularly in stress-bearing sites of the posterior tooth area, where frequently appearing high masticatory forces [6] are summarized to an exceeding load for dental RBC materials over the years [7]. In this context, adequate polymerization throughout the whole depth of the cavity represents a key factor for long-time stability. In turn, a low polymerization depth in extended cavities may be responsible for low mechanical performance [8], clinically inducing the occurrence of fracture, gaps or cracks, secondary caries, postoperative hypersensitivity or pain, even possibly leading to the failure of the restorations [9].

Adequate resin polymerization is influenced by material formulation [10] as well as placement and polymerization strategy [11–13]. Concerning material composition, research is not only based on enhancing the inorganic filler components of RBCs but even more on the development of novel matrix formulations. In this context, the application of high molecular weight monomers in modern RBCs recently became popular to dental composite development [14, 15]. These monomers possess a lower initial double bond concentration compared to conventional dimethacrylate monomers which is supposed to lead to high final double-bond conversion in conjunction with low polymerization shrinkage [15]. Examples of these novel formulations contain a high molecular weight monomer derived from a core structure based on hydrogenated dimer acid (h-Da, Fig. 1) [16] or rather a tricyclodecane–urethane dimethacrylate composition (Fig. 2) which is said to dispense without diluents and may thus avert high polymerization shrinkage [17, 18].

For dental filling therapy, incremental layering techniques are recommended not only to reduce shrinkage but also to ensure an adequate polymerization by applying the RBC in multiple steps with suitable layer thickness [13]. The dimension of this suitable incremental thickness can be assessed by different approaches such as degree of conversion measurements [19], penetrometer or scraping tests [20]. However, hardness measurements seem to belong to the most sensitive methods for determining this adequate layer thickness [19]. According to a well-accepted definition, a specimen of resin-based composite may be regarded adequately cured if the hardness at the bottom of the sample is at least 80% of the maximum hardness measured at the top of it [21, 22]. This range indicates the adequate layer thickness, also referred to as depth of cure (DOC).

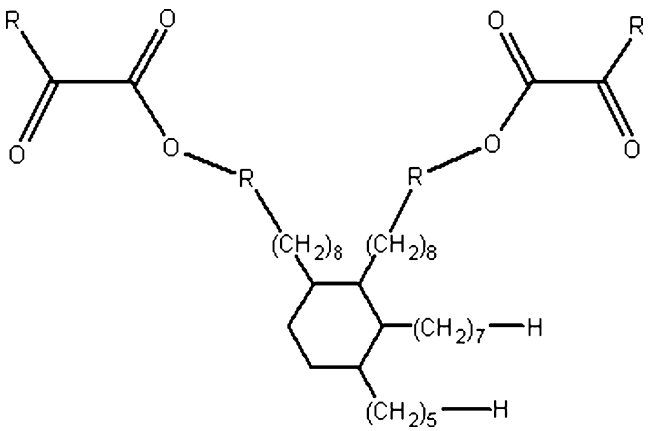

Fig. 1 Chemical structure of h-DA monomer [16]

The purpose of this study was to evaluate the DOC of nano-hybrid RBCs with novel monomer composition in comparison to well-established, conventional materials by assessing the variation of Vickers hardness (HV) as a function of polymerization time and depth. In addition to HV, the modulus of elasticity (E) was evaluated as a second parameter of mechanical properties. Moreover, differences between bulk and incremental filling techniques were examined.

The following research hypotheses were tested: (a) the two novel materials would show no differences in DOC and mechanical properties compared to the conventional RBCs; (b) the measured properties would not be influenced by curing time; (c) there would be no differences in measured properties concerning bulk and incremental placement techniques.

Materials and methods

Five dimethacrylate-based nano-hybrid RBCs were investigated in this study—two materials containing novel monomer matrix composition and three conventionally formulated products. The materials were selected from various manufacturers based on differences in their matrix and filler composition (Table 1).

Specimens were produced in an opaque mold of 6 mm height with a diameter of 4 mm. The mold was filled either in bulk or by applying a horizontal incremental layering technique, with three consecutive 2-mm increments being separately cured. The light-curing unit (Freelight 2, 3M ESPE, Germany; 1,241 mW/cm^2) was applied directly on the upper mold surface for 10, 20, or 40 s. An amount of six specimens was produced for each combination of filling technique and curing time, resulting in a total of 36 specimens for each of the tested materials. Following storage in distilled water for 24 h at 37°C, the samples were fixed on a plate and grinded and polished (EXAKT 400CS, Norderstedt, Germany; Abrasive paper for EXAKT grinding machines, 800–1,600 grit, Norderstedt, Germany) longitudinally until the specimen's center was reached.

The variation of HV and E with increasing depth was evaluated by using an automatic hardness indenter (Fischerscope H100C, Fischer, Germany). The test procedure was carried out from the top to the bottom of the samples in 100-μm steps, starting 0.1 mm under the surface (Fig. 3a). The test load increased and subsequently decreased at

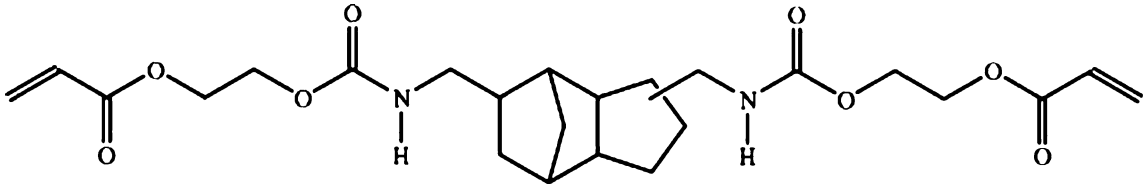

Fig. 2 Chemical structure of tricyclodecane–urethane dimethacrylate monomer (TCD-di-HEA) [18]

constant speed between 0.4 and 500 mN, while load and penetration depth of the diamond indenter were continuously measured during this load–unload hysteresis. Universal hardness is defined as the applied test force divided by the apparent area of indentation. A conversion factor between universal hardness and Vickers hardness was calculated and implemented into the software from a multiplicity of measurements stored in a database supplied by the manufacturer. In doing so, the results were documented in the more familiar Vickers hardness units. The indentation modulus (E) was calculated from the slope of the tangent to the indentation–depth curve at maximum force. The DOC was assessed at the samples cured in bulk technique as the depth at which the 80% hardness cutoff value in relation to the surface hardness was reached.

Statistical analysis

Results were compared using one- and multiple-way ANOVA and Tukey HSD post-hoc test (α=0.05) and partial eta-squared statistic (SPSS 18.0, Chicago, IL, USA). The results for HV and E were compared within each depth and filling technique as well as within each material and curing time, respectively. DOC values were also statistically compared within the different materials as well as curing times. In the multivariate analysis, the influence of the parameters "material," "curing time," "filling technique," "filler volume," and "filler weight" on HV, E, and DOC were analyzed. The partial eta-squared statistic reports the practical significance of each term, based upon 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.

Results

Results are listed in Tables 2, 3, 4, and 5 and illustrated in Fig. 3a–c. For all tested materials, increasing curing time resulted in a significant increase in DOC. Exceptions

Table 1 Summary of the dental nano-hybrid resin-based composites compared in this study

Brand name Batch no. (LOT)	Manufacturer	Composition	Shade; dosage
Miris 2 191818	Coltène/Whaledent AG, Altstätten, Switzerland	Matrix: methacrylate Filler: silanized barium glass, amorphous silica (hydrophobed); (range of particle size: 0.02–2.5 μm, av. particle size: 0.6 μm) [80 wt.%, 65 vol.%]	S2[a]
N'Durance 080609A	Septodont, Louisville, CO, USA	Matrix: Bis-GMA, UDMA, dicarbamate dimethacrylate dimer acid Filler: ytterbium fluoride (silanated), barium glass (silanated), silica; (range of particle size: 0.01–0.5 μm) [80 wt.%, 65 vol.%]	A3[b]
Premise 3123777, 3120178	Kerr, Orange, CA, USA	Matrix: Bis-EMA,TEGDMA Filler: barium glass, silica filler, pre-polymerized filler; (range of particle size: 0.02–50 μm) [84 wt.%, 69 vol.%]	A3[a]
Simile 180254	Pentron Clinical, Orange, CA, USA	Matrix: PCBisGMA, BisGMA, UDMA, HDDMA Filler: barium boro-silicate glass, silica filler, zirconium silicate; (range of particle size: 0.02–0.7 μm) [75 wt.%, 68 vol.%]	A3[b]
Venus Diamond 10029	Heraeus Kulzer GmbH, Hanau, Germany	Matrix: TCD-DI-HEA, UDMA Filler: barium aluminum fluoride glass, highly discrete nanoparticles; (range of particle size: 0.005–20 μm) [82 wt.%, 64 vol.%]	A3[b]

[a] Unidose, approx. 0.25 g

[b] Syringe, approx. 4.5 g

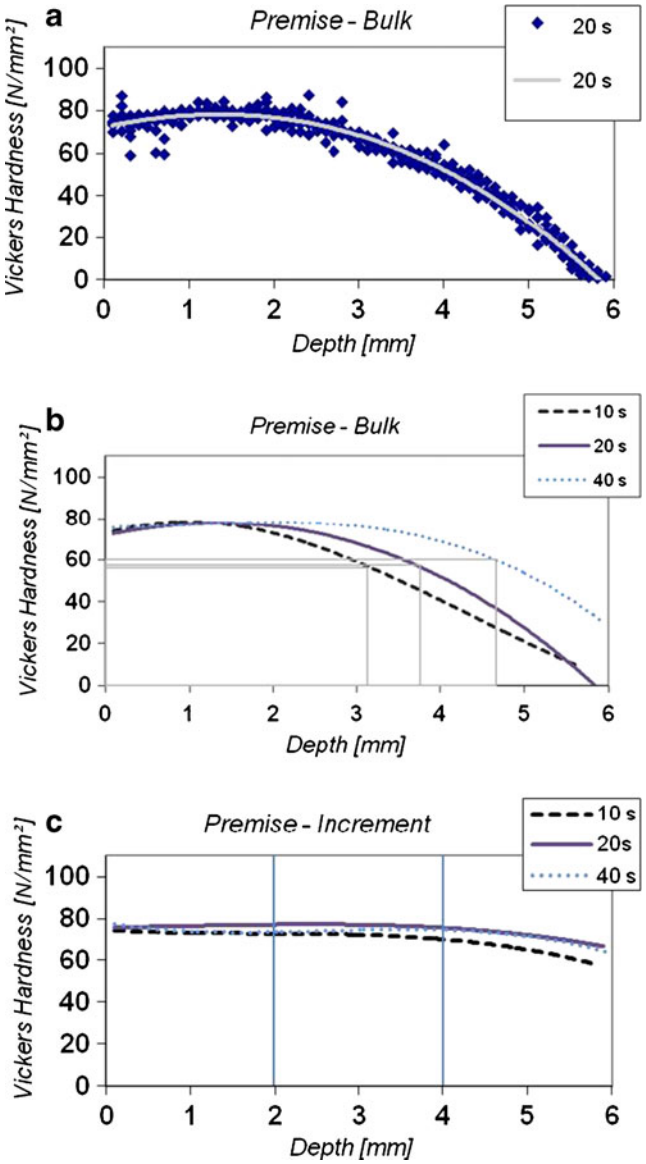

Fig. 3 **a** Variation of Vickers hardness (Premise; bulk technique, 20 s) with depth. Measurements were carried out each 100 μm throughout the middle of every test specimen (n=6). The obtained values are visualized by the *dots* (360) in the diagram. Mean values were calculated subsequently and are shown by the centrically situated graph. **b** Influence of polymerization time on the variation of HV with depth in bulk technique (Premise; 10, 20, and 40 s). The *gray rectangles* represent the 80% Vickers hardness cutoff value calculated from the maximum hardness measured at the top, leading to its corresponding depths of cure (DOC). **c** Influence of polymerization time on the variation of HV with depth in incremental technique (Premise; 10, 20, and 40 s). The *blue vertical lines* represent the 2-mm width of the increment layers which were cured separately

concern N'Durance for which no significant difference was found between curing times of 10 and 20 s as well as for Venus Diamond which showed no increase of values between 20 and 40 s. The lowest values at each curing time achieved Premise, whereas the highest values were measured for N'Durance and Venus Diamond at 10-s polymerization time and for Miris 2, N'Durance, and Venus Diamond at 40-s polymerization time, respectively. For 20-s irradiation time, all materials, except Premise, performed statistically similar. Measurements delivered an overall range of values between 3.10 and 5.55 mm.

Within one material generally no significant difference in HV and E was found at depths of 0.1 mm as well as 2 mm between bulk and incremental technique. Regarding polymerization time within one material, the duration of light curing also showed, in general, no influence on HV and E at these depths. Starting with a depth of 4 mm, significantly higher HV and E values were measured for the incremental technique compared to bulk curing. Within one material, values in 0.1 and 2 mm depth (incremental and bulk curing) and 4 mm depth (incremental curing) were generally similar to each other, irrespective from curing time. Comparing the values of 4 mm depth at bulk curing to 6 mm depth at incremental curing at the same material, there were mostly no significant differences within the same curing time. The overall lowest values were reached for bulk curing at 6 mm depth. In the bulk-cured samples, values for HV and E generally increased with longer curing time starting at 4 mm depth. At 6 mm depth, the highest mechanical properties in bulk-cured samples were reached by Venus Diamond and N'Durance, followed by Miris 2, Premise, and Simile.

The influence of the parameters: "material," "filling technique," "curing time," "filler volume," and "filler weight" on HV, E, and DOC, analyzed in an ANOVA multivariate test (Table 5), was significant. The strongest influence on HV and E was performed by "filling technique" (higher eta-squared values) followed by "material" and "curing time." "Filler volume" and "filler weight" showed only a low effect on HV but influenced E even slightly stronger than "curing time." DOC was strongly affected by "curing time" followed by "material"—a considerably lower effect was performed by "filler volume" and "filler weight." "Filler weight" influenced the measured properties slightly stronger than "filler volume."

Exemplified for the tested material Premise, Fig. 3a, b illustrates the decrease of HV with increasing depth in a bulk placement technique for different curing times. In contrast, Fig. 3c shows the same material applied in a 2-mm incremental layering technique illustrating that HV remains more constant throughout the specimen depth. The graph only shows a slight decrease for the lowest of the three 2-mm high increments.

Discussion

In daily clinical practice, assured knowledge about the maximum incremental layer thickness is inevitable for adequate polymerization and so for predictable successful treatment with RBC fillings. Most of the manufacturers recommend

Table 2 Depth of cure evaluated from the samples, which were cured for 10, 20, or 40 s by using bulk technique

Time [s] Material	10	20	40
Miris 2	3.80a,b *(0.44)	4.92B **(0.27)	5.55γ ***(0.16)
N'Durance	4.75c *(0.44)	4.80B *(0.35)	5.42β,γ **(0.24)
Premise	3.10a *(0.21)	3.80A **(0.45)	4.67α ***(0.48)
Simile	3.42a,b *(0.18)	4.42B **(0.26)	4.87α,β ***(0.27)
Venus Diamond	4.12b,c *(0.69)	4.93B **(0.38)	5.35β.γ **(0.38)

Calculation delivered the depth, at which the 80% Vickers hardness cutoff value in relation to the surface hardness was reached. Statistical analysis was made respectively within one curing time (shown by the different uppercase/lowercase/greek letters in the table), as well as within one material (shown by the number of asterisks in the table fields)

increments of about 2 mm thickness for the application of their dental RBCs. In our study, overall test results delivered values for DOC in a range between 3.10 and 5.55 mm. Thus, a layer thickness of 3 mm and a curing time of just 10 s might be generally recommended for the evaluated materials if clinical conditions are comparable to those in our study setup. Taking a closer look to Premise, its manufacturer specifies a DOC of 4.3 mm on the product's homepage. Under the ideal conditions of our test, however, Premise reached this DOC only after having been irradiated for 40 s. For shorter curing times, DOC was remarkably below 4 mm. Regarding Simile in this context, this material may also be layered in increments up to 4.3 mm and with an irradiation time of only 10 s according to the manufacturer's information. In our measurements, Simile reached DOC values beyond 4 mm only after curing times of 20 or 40 s. In this context, it has to be considered that several inaccuracies in clinical application, as being caused by difficult access to the oral cavity, for instance, may probably result in more inconsistent DOC values. Concerning Premise and Simile, the manufacturer's predictions thus cannot be confirmed by means of our study's measurements. This asks for critical consideration of the information given by manufacturers and supports the ongoing need for independent scientific research on new developed materials. As a conclusion, it can be stated that for a layer thickness of 2 mm, every material in this evaluation showed adequate polymerization already after a curing time of 10 s. In comparison to the three conventionally formulated materials, N'Durance and Venus Diamond, as representing the novel-formulated materials, both together reached the best results for DOC. These results were also in accordance to our previous study (journal article in publication process) comparing the degree of conversion among the same sample of tested materials; N'Durance and Venus Diamond showed the lowest decrease for the degree of conversion with increasing depth. The research hypothesis referred to as (a) can thus be rejected. In accordance to this study's findings, the two novel-formulated materials might be recommended for the application in comparatively bigger increments.

There are various factors influencing the depth of cure of resin-based composite materials. The results from this study showed that the material (η^2=0.601) and the curing time (η^2=0.718) perform the strongest effect on DOC among the tested parameters. The research hypothesis referred to as (b) may thus be rejected. Referring to Premise as being one of the tested materials in this study, Fig. 3c shows the progression of Vickers Hardness throughout the 6 mm-test-specimen which was cured using incremental layering technique. The graph shows a certain diminishment of HV for the lowest increment—a finding that was also noticed for all other RBCs in this study—which became, however, less significant with prolonged curing time. In incremental layering technique, all 2-mm high layers were consecutively placed and cured in the same manner, but the light tip—resin distance varied between the upper, the middle and the bottom increment as this was preset by the shape of the mold, representing the clinical situation of an oral cavity. As it has already been demonstrated in former tests that increasing the distance between the light tip and the resin composite surface leads to a diminishment in DOC [23] due to the fact that a certain amount of light energy is absorbed while light passes through air [24], the increased irradiation distance may be given as a reason for the finding of lower DOC values in the lowest increments. As a consequence for clinical treatment of large dental cavities, the placement of comparatively smaller increments or the use of prolonged curing time for the lowest layers at the bottom of the cavity might be taken into consideration in order to ensure adequate resin polymerization.

DOC may also be influenced by the composition of the material [25] as matrix components [26] as well as filler [27] and color particles [28] affect translucency and cause light intensity diminishment throughout the material differently. It has been shown that the opalescence of dental materials is mainly influenced by the difference in the refractive indices

Table 3 Vickers hardness evaluated for all specimens by micro-hardness indentation

Material	Time [s]	HV $_{0.1\text{-mm bulk}}$	HV $_{0.1\text{-mm incr.}}$	HV $_{2\text{-mm bulk}}$	HV $_{2\text{-mm incr.}}$	HV $_{4\text{-mm bulk}}$	HV $_{4\text{-mm incr.}}$	HV $_{6\text{-mm bulk}}$	HV $_{6\text{-mm incr.}}$
Miris 2	10	94.68c,d,e [3,4] (2.03)	91.81C [3] (3.87)	104.31d [4] (10.07)	98.18C [3,4] (6.89)	71.50e,f [2] (4.58)	96.17C,D [3,4] (2.69)	2.25a [1] (2.05)	72.65C,D,E [2] (9.73)
	20	87.12b [3,4] (6.58)	92.52C [4,5] (2.99)	92.12c,d [5] (3.48)	95.73C [5] (7.81)	80.19g,h [2] (3.88)	95.77C,D [5] (4.06)	12.04a [1] (4.91)	83.23E,F,G,H [2,3] (8.14)
	40	90.76b,c,d [2,3] (3.64)	91.45C [2,3] (4.60)	118.20e [4] (11.11)	96.57C [3] (3.19)	91.59i,j [2,3] (3.09)	92.94C [2,3] (3.10)	49.34d,e [1] (15.03)	87.66F,G,H [2] (6.59)
N'Durance	10	73.68a [3] (5.78)	79.99A,B [3] (3.19)	77.85a,b [3] (2.74)	74.86A,B [3] (10.23)	60.80c,d [2] (2.05)	73.51A,B [3] (6.50)	28.44b [1] (10.23)	62.94A,B,C [2] (8.27)
	20	75.44a [3,4] (4.84)	77.75A,B [4] (4.13)	79.69b [4] (1.13)	80.33B [4] (4.43)	67.24d,e [2] (2.79)	77.56B [4] (4.40)	28.33b [1] (8.95)	70.40B,C,D,E [2,3] (7.71)
	40	78.71a [2] (4.76)	80.80B [2] (6.40)	81.25b [2] (1.97)	79.90B [2] (2.71)	76.17f,g [2] (2.33)	77.87B [2] (2.33)	57.55e,f [1] (9.79)	76.54D,E,F [2] (2.85)
Premise	10	73.53a [4] (3.06)	72.48A [4] (5.18)	73.63a [4] (5.10)	71.87A [4] (11.10)	41.23a [2] (6.32)	71.02A [4] (3.51)	13.62a [1] (11.68)	57.61A,B [3] (8.38)
	20	76.15a [4,5] (5.01)	73.36A [4] (4.61)	76.30a,b [4,5] (2.65)	79.12A,B [5] (2.34)	52.77b [2] (4.25)	74.44A,B [4,5] (3.84)	2.85a [1] (1.80)	46.99A,B,C,D [3] (5.28)
	40	73.31a [3] (6.00)	70.47A [3] (6.99)	75.61a,b [3] (8.18)	76.36A,B [3] (6.99)	67.57e [2,3] (5.86)	73.44A,B [3] (5.24)	29.52b,c [1] (13.19)	55.38A [2] (19.80)
Simile	10	88.96b,c [4] (3.11)	95.40C [5,6] (3.45)	91.04c [4,5] (2.39)	102.21C [6] (6.85)	55.17b,c [2] (6.75)	95.15C,D [5,6] (2.47)	2.51a [1] (1.42)	80.67E,F,G [3] (7.12)
	20	91.21b,c,d [3] (5.23)	89.43C [3] (7.52)	94.10c,d [3] (1.80)	95.65C [3] (7.52)	74.32f,g [2] (3.78)	92.41C [3] (2.04)	3.47a [1] (2.08)	77.79D,E,F [2] (11.99)
	40	94.88c,d,e [3,4] (2.83)	92.41C [2,3] (4.74)	98.85d [4] (5.04)	95.74C [3,4] (2.20)	85.24h,i [2] (1.82)	93.26C [3,4] (2.17)	43.29c,d [1] (11.57)	86.48F,G,H [2] (5.80)
Venus Diamond	10	100.57e [4] (5.45)	89.43C [3,4] (7.52)	99.30d [4] (5.16)	99.10C [4] (5.19)	87.02i [2,3] (7.40)	98.54C,D [4] (3.91)	15.80a,b [1] (10.00)	80.55E,F,G [2] (12.68)
	20	97.96d,e [2] (8.88)	97.44C [2] (10.60)	99.35d [2] (4.45)	102.28C [2] (4.27)	95.46j [2] (6.56)	94.11C [2] (9.56)	58.82e,f [1] (16.41)	93.48G,H [2] (3.17)
	40	93.66b,c,d,e [2,3] (4.95)	94.55C [2,3,4] (5.79)	99.50d [2,3,4] (4.07)	102.40C [4] (4.70)	91.78i,j [2] (4.37)	100.53D [3,4] (4.10)	68.06f [1] (12.15)	94.17H [2,3,4] (8.14)

Statistical analysis was made respectively within one depth and filling method (bulk, incremental; in different uppercase / lowercase letters) as well as for each material and irradiation time throughout the different depths with filling methods (numbers in brackets). For each category, the statistical analysis was made separately

Table 4 Modulus of elasticity (E) evaluated for all specimens by micro-hardness indentation

Material	Time	E $_{0.1\text{-mm bulk}}$	E $_{0.1\text{-mm incr.}}$	E $_{2\text{-mm bulk}}$	E $_{2\text{-mm incr.}}$	E $_{4\text{-mm bulk}}$	E $_{4\text{-mm incr.}}$	E $_{6\text{-mm bulk}}$	E $_{6\text{-mm incr.}}$
Miris 2	10s	14.99e,f [3.4] (0.83)	14.59E,F [3] (0.87)	16.72e [4] (0.96)	15.86C [4] (0.92)	13.23f,g [2] (0.98)	15.94E [4] (0.69)	0.92a,b,c [1] (0.45)	12.49D,E [2] (1.61)
	20s	13.90d,e [2] (1.14)	14.69E,F [2.3] (0.98)	15.29e [3.4] (0.79)	15.59C [4] (1.28)	14.09g,h [2] (0.54)	15.95E [4] (0.87)	2.89b,c,d [1] (0.82)	14.22E,F [2] (1.21)
	40s	13.54d [2] (0.71)	14.22D,E [2.3] (1.12)	16.85e [4] (0.83)	15.67C [3.4] (0.80)	14.94h [2,3,4] (0.95)	15.64E [3,4] (0.40)	9.08f [1] (2.23)	13.57E,F [2] (1.69)
N'Durance	10s	10.20a [2,3] (0.96)	11.18A,B [3,4] (0.57)	11.97a,b [4] (0.24)	11.86A [4] (0.87)	9.69b,c [2] (0.76)	11.56A,B [4] (0.43)	4.70d,e [1] (1.75)	9.71A,B,C [2] (1.07)
	20s	10.46a,b [2] (0.75)	11.18A,B [2,3] (0.76)	12.08a,b [3] (0.12)	12.07A [3] (1.19)	10.50c,d [2] (0.70)	11.87A,B,C [3] (0.39)	4.66d,e [1] (1.38)	10.19A,B,C [2] (1.08)
	40s	11.68b,c [2,3] (1.12)	11.20A,B [2] (1.06)	12.80b,c [3] (0.62)	12.31A [2,3] (0.80)	12.28e,f [2,3] (0.51)	11.28A [2] (0.75)	9.67f,g [1] (1.43)	9.52A,B,C [1] (1.78)
Premise	10s	11.00a,b [4] (0.80)	11.24A,B [4] (1.01)	11.68a [4] (0.62)	11.92A [4] (1.55)	7.36a [2] (1.33)	11.85A,B,C [4] (0.64)	2.11a,b,c [1] (1.87)	9.22A,B [3] (1.22)
	20s	11.56a,b,c [4,5] (0.73)	11.05A,B [3,4] (1.09)	12.31a,b,c [5,6] (0.26)	12.77A [6] (0.60)	9.25b,c [2] (0.40)	12.48C [6] (0.67)	0.72a,b [1] (0.26)	10.26A,B,C [3] (0.88)
	40s	11.17a,b,c [3] (0.93)	10.83A [3] (1.69)	12.19a,b [3] (0.65)	12.28A [3] (0.72)	11.35d,e [3] (0.92)	12.21B,C [3] (0.63)	4.90d,e [1] (2.37)	8.69A [2] (2.52)
Simile	10s	11.73b,c [3] (0.82)	12.51B,C [3,4] (1.41)	13.03c [4,5] (0.62)	13.99B [5] (0.46)	8.62a,b [2] (1.78)	13.53D [4,5] (0.78)	0.55a [1] (0.33)	11.19B,C,D [3] (1.21)
	20s	12.54c,d [3,4] (0.72)	13.03C,D [4,5] (0.80)	14.33d [6] (0.32)	13.94B [5,6] (0.44)	12.00e,f [2,3] (0.44)	13.83D [5,6] (0.34)	0.80a,b [1] (0.37)	11.39C,D [2] (1.51)
	40s	11.76b,c [2] (2.02)	13.33C,D,E [3,4,5] (0.54)	14.37d [5] (0.47)	14.24B [4,5] (0.22)	12.60e,f [2,3] (0.55)	14.26D [4,5] (0.22)	6.49e [1] (1.46)	13.09D,E [3,4] (0.85)
Venus Diamond	10s	15.53f,g [2,3] (1.02)	16.35G [3,4] (1.20)	17.61f [4,5] (0.85)	18.75D [5] (0.63)	15.21h [2] (2.16)	18.64G [5] (0.39)	3.03c,d [1] (1.19)	15.46F,G [2,3] (1.62)
	20s	16.44g [2] (1.06)	16.58G [2,3] (1.09)	18.07f [3,4] (0.82)	18.74D [4] (0.55)	17.52i [2,3,4] (0.72)	17.62F [2,3,4] (0.81)	11.51g [1] (2.69)	16.54G [2,3] (1.04)
	40s	16.89g [2,3] (0.70)	16.11F,G [2] (1.70)	18.15f [3,4] (0.70)	18.84D [4] (0.38)	17.60i [3,4] (0.36)	18.64G [4] (0.89)	13.76h [1] (2.04)	16.79G [2,3] (0.99)

Statistical analysis was made respectively within one depth and curing method (bulk, incremental; in different uppercase / lowercase letters) as well as for each material and irradiation time throughout the different depths with curing methods (numbers in brackets). For each category, the statistical analysis was made separately

Table 5 Influence of the parameters material, curing time, filling technique, filler volume, and filler weight on Vickers hardness (HV), modulus of elasticity (E), and depth of cure (DOC) (partial eta-squared)

	Material	Curing time	Filling technique	Filler volume	Filler weight
HV	0.570	0.264	0.861	0.131	0.154
E	0.758	0.257	0.848	0.259	0.308
DOC	0.601	0.718	–	0.269	0.275

between the filler particles and the resin matrix [29, 30] which leads to light scattering within the material [31]. Translucency may thus correlate with similar refractive indices of the components of a RBC as it could have already been demonstrated in further tests that examined experimental RBCs [26]. N'Durance and Venus Diamond generally showed the highest results for DOC in this study. As in the case of N'Durance which contains Bis-GMA and silica filler particles with similar refractive indices [26], this aspect might contribute for its high DOC values. It was also figured out that translucency generally decreases linearly with an increasing amount of filler particles [27]. As these two materials contain lower filler fractions compared to the other materials in this test, this aspect may also be given as a reason to explain this finding. Premise delivered the lowest results for DOC in this study. One reason for this behavior may be regarded in the higher fraction of filler volume and weight of this material when compared to the other RBCs which were tested. A former study showed that the small nano-particles that are contained in nano-hybrid RBCs do not contribute to light scattering within the material because their small dimension only amounts a fraction of the polymerization light's wavelength [5]. Possibly, a comparably big fraction of these nano-fillers in the composition of N'Durance and Venus Diamond might contribute to the high DOC values of these materials. Darker color shades, in comparison to lighter shades, are by trend associated with shallower depth of cure [12, 28, 32] since their color pigments may absorb the light stronger and reduce its penetration throughout the composite material, consequently inducing lower translucency and DOC values [33]. Although similar colors were chosen for the materials tested in this study (Color A3 or equal), inevitable differences concerning the color particles might also have taken an effect on the measured test results. Additionally, aspects of particle geometry or particle shape might possibly have a certain effect in terms of light scattering within RBC materials—this, however, asks for further experimental investigation. Today's literature provides only few data concerning the comparison of DOC values of RBC materials. In a formerly published study in which the DOC of a micro-hybrid RBC was evaluated, considerably lower values were shown (1.92 and 2.31 mm) in comparison to the results of our study. As already mentioned above, it has been demonstrated that nano-fillers, as they are contained to a certain amount in the nano-hybrid RBCs, investigated in our study, do not affect light scattering within the composite material [5]. Thus, it can be suggested that despite of a commonly higher amount of filler particles in nano-hybrid RBCs in comparison to micro-hybrid RBCs [34], nano-hybrid materials may still generally reach higher results in terms of DOC.

There may be discussed various advantages for applying incremental layering techniques in clinical practice, such as easier handling, the possibility of multiple-shade application leading to more aesthetic results [35], the higher quality of the tooth-composite bonding [36] and, at the first and foremost, adequate material polymerization throughout the whole depth of the dental filling. The question if incremental techniques induce a positive effect in terms of lower polymerization shrinkage stress on the dental cavity is still controversially discussed [37–39]. While former tests found out that incremental filling techniques may reduce shrinkage and cuspal deflection from polymerization shrinkage [40], other investigations showed that incremental layering yields higher shrinkage and tensile stress concentrations at the restoration-enamel interface when compared to bulk filling [38]. However, going beyond the aspect of polymerization shrinkage stress, the benefit of incremental layering techniques by means of adequate polymerization should be emphasized even for the usage of low-shrinkage materials [41]. With the highest measured DOC values in this study being underneath 6 mm, adequate polymerization could not be reached throughout the whole depth of the 6 mm bulk-filled test specimens at any time. In addition, the results from mechanical measurements point out, too, that there are remarkable differences concerning bulk placement in comparison to incremental layering technique. The research hypothesis referred to as (c) may thus be rejected. Venus Diamond as being one of the materials with the highest results for DOC in this study and also with a comparably low polymerization shrinkage according to the results of former evaluations [17], might also be used in bulk technique in cavities of up to 5 mm depth (with at least 40 s of irradiation time) under ideal clinical conditions. Although the usage of incremental filling techniques is a contentious issue, low-shrinkage materials should be generally preferred in any case irrespective of aspects concerning the placement technique.

The restoration of large cavities in the posterior tooth area by the use of RBC materials requires high mechanical performance of the applied material by means of fracture toughness, fatigue resistance, and good wear resistance [6, 7]. Mechanical properties are strongly determined by the filler volume and weight fraction of dental RBCs but are also influenced by the structure, viscosity, and composition of the monomer components [42]. In general, high values of E are corresponding with a high resistance to deformation [43] under masticatory forces, while high values of HV are associated with a high wear resistance and a high resistance to abrasion [44]. Since mechanical properties evaluated at micro-scale correspond with the mechanical properties of the same material measured at macro-scale (flexural strength, modulus of elasticity in flexural test) [45], the results from this study may allow, to a certain extent, to infer to macro-mechanical behavior of our tested devices, too. In our study, the highest values concerning mechanical properties were reached by Venus Diamond. As E generally increases with filler weight [46] the high filler fraction of Venus Diamond (82 wt.%) may be given as one reason for this finding. In former evaluations among the same sample of materials as examined in this study, Venus Diamond reached the highest flexural strength and flexural modulus after either 24-h storage in distilled water or thermocycling and subsequent 4-week storage in distilled water, artificial saliva, or ethanol [47]. Even though all materials showed a significant decrease in mechanical properties after having been stored in ethanol for 4 weeks, Venus Diamond, however, showed the lowest decline in this experiment followed by N'Durance [47]. In our study, mechanical properties of N'Durance were comparatively low, which is in accordance to formerly published data [17, 47], although this specific material possesses the same amount of filler volume and weight fraction as Miris 2, which showed significantly higher mechanical properties, thus supporting the aspect that mechanical properties are not only determined by the filler fraction alone. Experimental materials containing dimer-acid derivates, as they are also included in N'Durance, also performed just moderately in terms of mechanical properties [16], so again suggesting the existence of a certain influence of the organic matrix components to the mechanical properties of a corresponding material. Premise showed the lowest results for mechanical properties which were in accordance with a formerly published study [48], although having the highest filler volume and weight fraction among the tested RBC materials of this experiment. The fraction of pre-polymerized filler particles in this specific RBC might be given as a reason for this finding, as former tests could demonstrate that the addition of pre-polymerized fillers leads to decreasing strength and toughness of conventional RBC materials [49].

Literature provides extensive documentation that filler content is the main responsible for mechanical properties of RBC materials [50, 51]. However, the findings from our test which demonstrated only a moderate influence of "filler volume" and also "filler weight" and the measured properties (DOC, HV, and E; range of η^2=0.131–0.308), give support to the aforementioned aspect, that mechanical properties are not only influenced by the filler components alone (Table 5). As the RBC materials that were evaluated in this experiment were all chosen from the same material category, the relatively slight differences in their general composition of filler components may be given as a reason, that our experiment was able to identify parameters other than filler volume and filler weight with a comparatively stronger effect on the measured properties. In this context, also the characteristics of the matrix components may be taken into account for a certain influence on the overall mechanical performance of a RBC material.

Conclusion

As to transfer the experimental findings of this trial to clinical application, all of the tested materials need to be placed incrementally in cases of a cavity that is deeper than 3 mm in order to ensure adequate polymerization. For cavities exceeding a depth of 6 mm, the lowest increment should be cured for at least 40 s. Concerning the novel-formulated resin-based composites, N'Durance and Venus Diamond, showed the highest values for DOC and might thus also be cured in comparatively bigger increments. In this context, mechanical properties are not only influenced by the filler fraction alone, but also by the matrix components. Inadequate filling technique or inadequate curing time, however, cannot compensate for ideal material characteristics.

Conflict of interest The authors declare that they have no conflict of interest.

References

1. Moszner N, Salz U (2001) New developments of polymeric dental composites. Prog Polym Sci 26:535–576
2. Mitra SB, Wu D, Holmes BN (2003) An application of nanotechnology in advanced dental materials. J Am Dent Assoc 134:1382–1390
3. Lohbauer U, Frankenberger R, Kramer N, Petschelt A (2006) Strength and fatigue performance versus filler fraction of different types of direct dental restoratives. J Biomed Mater Res B Appl Biomater 76:114–120. doi:10.1002/jbm.b.30338
4. Endo T, Finger WJ, Kanehira M, Utterodt A, Komatsu M (2010) Surface texture and roughness of polished nanofill and nanohybrid resin composites. Dent Mater J 29:213–223

5. Kim JJ, Moon HJ, Lim BS, Lee YK, Rhee SH, Yang HC (2007) The effect of nanofiller on the opacity of experimental composites. J Biomed Mater Res B Appl Biomater 80:332–338. doi:10.1002/jbm.b.30601
6. Dejak B, Mlotkowski A, Romanowicz M (2003) Finite element analysis of stresses in molars during clenching and mastication. J Prosthet Dent 90:591–597. doi:10.1016/S0022391303005900
7. Papadogiannis Y, Lakes RS, Palaghias G, Helvatjoglu-Antoniades M, Papadogiannis D (2007) Fatigue of packable dental composites. Dent Mater 23:235–242. doi:10.1016/j.dental.2006.01.015
8. Ferracane JL, Greener EH (1986) The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins. J Biomed Mater Res 20:121–131. doi:10.1002/jbm.820200111
9. Hickel R, Manhart J (2001) Longevity of restorations in posterior teeth and reasons for failure. J Adhes Dent 3:45–64
10. Soh MS, Yap AU, Siow KS (2003) Effectiveness of composite cure associated with different curing modes of LED lights. Oper Dent 28:371–377
11. Francci C, Loguercio AD, Reis A, Carrilho MR (2002) A novel filling technique for packable composite resin in class II restorations. J Esthet Restor Dent 14:149–157
12. Koupis NS, Martens LC, Verbeeck RM (2006) Relative curing degree of polyacid-modified and conventional resin composites determined by surface Knoop hardness. Dent Mater 22:1045–1050. doi:10.1016/j.dental.2005.08.008
13. Deliperi S, Bardwell DN (2002) An alternative method to reduce polymerization shrinkage in direct posterior composite restorations. J Am Dent Assoc 133:1387–1398
14. Stansbury JW, Trujillo-Lemon M, Lu H, Ding X, Lin Y, Ge J (2005) Conversion-dependent shrinkage stress and strain in dental resins and composites. Dent Mater 21:56–67. doi:10.1016/j.dental.2004.10.006
15. Ge J, Trujillo M, Stansbury J (2005) Synthesis and photopolymerization of low shrinkage methacrylate monomers containing bulky substituent groups. Dent Mater 21:1163–1169. doi:10.1016/j.dental.2005.02.002
16. Trujillo-Lemon M, Ge J, Lu H, Tanaka J, Stansbury JW (2006) Dimethacrylate derivatives of dimer acid. J Polymer Sci Polymer Chem 44:3921–3929. doi:10.1002/pola.21493
17. Boaro LC, Goncalves F, Guimaraes TC, Ferracane JL, Versluis A, Braga RR (2010) Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites. Dent Mater 26:1144–1150. doi:10.1016/j.dental.2010.08.003
18. Utterodt A (2008) Heraeus Kulzer GmbH (Hanau/Germany). Dental composites with Tricyclo[5.2.02.6] decane derivatives. Europe Patent
19. DeWald JP, Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites. J Dent Res 66:727–730
20. Koupis NS, Vercruysse CW, Marks LA, Martens LC, Verbeeck RM (2004) Curing depth of (polyacid-modified) composite resins determined by scraping and a penetrometer. Dent Mater 20:908–914. doi:10.1016/j.dental.2004.01.001
21. Watts DC, Amer O, Combe EC (1984) Characteristics of visible-light-activated composite systems. Br Dent J 156:209–215
22. Leonard DL, Charlton DG, Roberts HW, Cohen ME (2002) Polymerization efficiency of LED curing lights. J Esthet Restor Dent 14:286–295
23. Lindberg A, Peutzfeldt A, van Dijken JW (2005) Effect of power density of curing unit, exposure duration, and light guide distance on composite depth of cure. Clin Oral Investig 9:71–76. doi:10.1007/s00784-005-0312-9
24. Meyer GR, Ernst CP, Willershausen B (2002) Decrease in power output of new light-emitting diode (LED) curing devices with increasing distance to filling surface. J Adhes Dent 4:197–204
25. Atmadja G, Bryant RW (1990) Some factors influencing the depth of cure of visible light-activated composite resins. Aust Dent J 35:213–218
26. Azzopardi N, Moharamzadeh K, Wood DJ, Martin N, van Noort R (2009) Effect of resin matrix composition on the translucency of experimental dental composite resins. Dent Mater 25:1564–1568. doi:10.1016/j.dental.2009.07.011
27. Lee YK (2008) Influence of filler on the difference between the transmitted and reflected colors of experimental resin composites. Dent Mater 24:1243–1247. doi:10.1016/j.dental.2008.01.014
28. Shortall AC, Wilson HJ, Harrington E (1995) Depth of cure of radiation-activated composite restoratives–influence of shade and opacity. J Oral Rehabil 22:337–342
29. Primus CM, Chu CC, Shelby JE, Buldrini E, Heckle CE (2002) Opalescence of dental porcelain enamels. Quintessence Int 33:439–449
30. Shortall AC, Palin WM, Burtscher P (2008) Refractive index mismatch and monomer reactivity influence composite curing depth. J Dent Res 87:84–88
31. Lee YK, Lu H, Powers JM (2005) Measurement of opalescence of resin composites. Dent Mater 21:1068–1074. doi:10.1016/j.dental.2005.03.015
32. Tarle Z, Meniga A, Ristic M, Sutalo J, Pichler G (1995) Polymerization of composites using pulsed laser. Eur J Oral Sci 103:394–398
33. Leloup G, Holvoet PE, Bebelman S, Devaux J (2002) Raman scattering determination of the depth of cure of light-activated composites: influence of different clinically relevant parameters. J Oral Rehabil 29:510–515
34. Beun S, Glorieux T, Devaux J, Vreven J, Leloup G (2007) Characterization of nanofilled compared to universal and microfilled composites. Dent Mater 23:51–59. doi:10.1016/j.dental.2005.12.003
35. Chi HH (2006) A posterior composite case utilizing the incremental and stratified layering technique. Oper Dent 31:512–516
36. Amaral CM, de Castro AK, Pimenta LA, Ambrosano GM (2002) Influence of resin composite polymerization techniques on microleakage and microhardness. Quintessence Int 33:685–689
37. Kuijs RH, Fennis WM, Kreulen CM, Barink M, Verdonschot N (2003) Does layering minimize shrinkage stresses in composite restorations? J Dent Res 82:967–971
38. Versluis A, Douglas WH, Cross M, Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? J Dent Res 75:871–878
39. Loguercio AD, Reis A, Ballester RY (2004) Polymerization shrinkage: effects of constraint and filling technique in composite restorations. Dent Mater 20:236–243. doi:10.1016/S0109-5641(03)00098-8
40. Park J, Chang J, Ferracane J, Lee IB (2008) How should composite be layered to reduce shrinkage stress: incremental or bulk filling? Dent Mater 24:1501–1505
41. Van Ende A, De Munck J, Mine A, Lambrechts P, Van Meerbeek B (2010) Does a low-shrinking composite induce less stress at the adhesive interface? Dent Mater 26:215–222. doi:10.1016/j.dental.2009.10.003
42. Asmussen E, Peutzfeldt A (1998) Influence of UEDMA BisGMA and TEGDMA on selected mechanical properties of experimental resin composites. Dent Mater 14:51–56. doi:10.1016/S0109-5641(98)00009-8
43. Park C, Robertson RE (1998) Mechanical properties of resin composites with filler particles aligned by an electric field. Dent Mater 14:385–393
44. Draughn RA, Harrison A (1978) Relationship between abrasive wear and microstructure of composite resins. J Prosthet Dent 40:220–224
45. Ilie N, Hickel R (2009) Macro-, micro- and nano-mechanical investigations on silorane and methacrylate-based composites. Dent Mater 25:810–819. doi:10.1016/j.dental.2009.02.005

46. Ilie N, Hickel R (2009) Investigations on mechanical behaviour of dental composites. Clin Oral Investig 13:427–438. doi:10.1007/s00784-009-0258-4
47. Schmidt C, Ilie N (2011) The mechanical stability of nano-hybrid composites with new methacrylate monomers for matrix compositions. Dent Mater, doi:10.1016/j.dental.2011.11.007
48. Blackham JT, Vandewalle KS, Lien W (2009) Properties of hybrid resin composite systems containing prepolymerized filler particles. Oper Dent 34:697–702
49. Ruddell DE, Maloney MM, Thompson JY (2002) Effect of novel filler particles on the mechanical and wear properties of dental composites. Dent Mater 18:72–80
50. Kim KH, Ong JL, Okuno O (2002) The effect of filler loading and morphology on the mechanical properties of contemporary composites. J Prosthet Dent 87:642–649
51. Chung KH, Greener EH (1990) Correlation between degree of conversion, filler concentration and mechanical properties of posterior composite resins. J Oral Rehabil 17:487–494