Journal of Applied Polymer Science

Polymerization efficiency of bulk-fill dental resin composites with different curing modes

Dimitrios Dionysopoulos, Kosmas Tolidis, Paris Gerasimou
Department of Operative Dentistry, School of Dentistry, Aristotle University of Thessaloniki, Thessaloniki, Greece
Correspondence to: D. Dionysopoulos (E-mail: ddiondent@gmail.com)

ABSTRACT: The aim of this study was to investigate the effect of four different curing modes on the polymerization efficiency of eight bulk-fill composites. Five specimens for each material were prepared for Vickers hardness measurements. The measurements were performed at 0 and 2 or 4 mm from the top of the surface of the specimens 24 h after photopolymerization. Statistical analysis was performed with one-way analysis of variance and Tukey *post hoc* tests at a level of significance of $a = 0.05$ where a is the the level of significance. The light-curing mode affected the microhardness in all depths, but this influence was material-dependent ($p_{mat} < 0.001$), where p_{mat} is the probability to be affected by the material. The Vickers hardness numbers of the tested composites at 0 mm ranged from 9.32 ± 0.87 to 72.58 ± 6.52 and those of the tested composites at 4 mm ranged from 5.48 ± 0.32 to 54.34 ± 2.27. The clinician has to be aware of the technical characteristics of the materials and light-curing units (LCUs) to use the most appropriate combination of LCU, composite material, and application technique. © 2016 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2016**, *133*, 43392.

KEYWORDS: composites; irradiation; photopolymerization

Received 12 September 2015; accepted 27 December 2015
DOI: 10.1002/app.43392

INTRODUCTION

The depth of cure of resin-based composites (RBCs) is crucial in determining the maximum increment thickness in the restoration of tooth cavities. In restorations with conventional RBCs, the thickness of each increment is defined as up to 2 mm.[1] This limitation is related to the degree of monomer conversion of RBCs; this determines their mechanical and physical properties, such as the strength, modulus, hardness, solubility,[2] and biocompatibility.[3] Although the critical threshold for the degree of conversion of RBCs has not yet been established, it has been accepted that the degree of monomer conversion below 55% is not appropriate for adequate clinical performance.[4]

There are many factors that influence the depth of cure of RBCs. These include the composition (monomers, fillers, photoinitiators, silane-coupling agents),[5–7] shade, and translucency[8] of the composite and characteristics of the light-curing unit (LCU), such as the light intensity, thermal emission, wavelength range, diameter of the tip, and curing mode.[9] Other factors, such as the exposure time[10] and distance of the restorative from the tip of the LCU[11] also affect the depth of cure of these materials.

The incremental technique for composite restorations is associated with various weaknesses; these include the risk of incorporating air bubbles or contaminants between composite layers, failure in bonding between the increments, and an extended treatment time.[12,13] To overcome these problems, bulk-fill resin composites have been recently introduced to the market; they enable increments of up to 4 mm to be adequately cured.[14] To achieve this extended depth of cure, bulk-fill materials have acquired certain modifications in their composition to increase the penetration of visible light through materials; these modifications include an increased filler size[15] and novel photoinitiators.[16]

It has been reported that a reduction in the intermolecular distance from 0.3–0.4 to 0.15 nm may occur during the polymerization of RBCs[17]; this generates stresses because of the contraction of the material and may lead to bonding failure and an increase in the microleakage of the restoration. Previous studies have reported that bulk-fill composites may exhibit better polymerization shrinkage stress kinetics[18] and reduced cuspal deflection[19] than conventional RBCs. However, the improved behavior of these newly introduced materials with respect to marginal adaptation to dental tissues remains unconfirmed.[19,20]

It has been suggested that the use of altered light application methods could enhance the degree of conversion and reduce the effects of the polymerization shrinkage of RBCs.[21,22] These

methods include soft-start and pulse-delay curing modes, which start with a lower light intensity followed by high-intensity light; this reduces the curing speed. As a result, this slower polymerization process allows the composite to flow in the pregel stage and leads to a lower level of shrinkage stress. Feitosa *et al.*[21] found that soft-start and pulse-delay step-curing approaches may provide less shrinkage outcomes that are appropriate for high-configuration factors (c factors) class I and II RBC restorations. Additionally, plasma arc LCUs provide reduced curing times because of their higher light intensity emission. However, an adequate degree of conversion in 4 mm of bulk-fill RBCs when a plasma arc is used for polymerization has not been sufficiently investigated.

Surface microhardness measurements of RBCs are an effective method for indirectly determining the degree of monomer conversion.[13] In this study, Vickers hardness measurements were performed to evaluate the curing efficiency of various bulk-fill RBCs. To achieve an acceptable curing efficiency, bulk-fill materials have to meet the requirement of having a bottom/top surface microhardness of 80% or greater.[13]

The aim of this study was to investigate the effect of four different light-curing modes on the polymerization efficiency of four highly viscous and four flowable bulk-fill RBCs at three depths (0, 2, and 4 mm) and to determine if they achieved the curing efficiency requirement. The novelty of the study was that some of the tested materials had never been investigated before with respect to this property, and their polymerization efficiency was studied with four different light-curing modes for the first time so that they could compared with each other.

The first null hypothesis of the study was that there was no difference in the Vickers hardness values of the materials light-cured with the same curing mode at the same depth. The second null hypothesis of the study was that there was no difference between the Vickers hardness values of the same material when it was polymerized with different curing modes at the same depth.

EXPERIMENTAL

Materials

Four high-viscosity bulk-fill RBCs [X-tra Fil (XF), EverX Posterior (EXP), Tetric EvoCeram Bulk Fill (TEB), and Beautifil-Bulk Restorative (BBR)] and four flowable bulk-fill RBCs [X-tra Base (XB), Beautifil Bulk Flowable (BBF), Filtek Bulk Fill (FB), and Venus Bulk Fill (VB)] were investigated in this study, and a nanohybrid conventional composite [Filtek Z550 (FZ)] was used as a control (Table I). The shade of the bulk-fill RBCs was Universal, and the shade of FZ was A2. In addition, three light-emitting diode (LED) LCUs (Table II) were used for the photopolymerization of the composite materials. The settings of each curing mode were made according to the manufacturer's recommendations. A radiometer (Demetron L.E.D. Radiometer, Kerr Corp.) was used to verify the output irradiance of each LCU.

Preparation of the Specimens

Four experimental groups for each composite material were tested according to the curing mode of the LED unit used for photopolymerization. The curing modes of each LED unit are presented in Table II. The specimens with standardized dimensions (5 mm wide, 5 mm long, and 2 or 4 mm high) were prepared for surface microhardness measurements with a reusable and custom-made stainless steel mold. The composite was inserted in the mold in one increment 2 or 4 mm in thickness. Polyester strips 0.05 mm in thickness were placed on both sides of the mold, and glass microscope slides were placed over the polyester strips and clamped to produce a standardized smooth surface and to remove excess material. Subsequently, the top of each specimen was irradiated for the designated time according to the manufacturer's instructions. There was no distance between the light tip of the LED and the top surface of the composite specimen. Five specimens were prepared for each combination of the parameters (resin composite and curing mode); this resulted in 72 experimental groups and a total of 360 specimens.

Surface Microhardness Measurements

Immediately after photopolymerization, the composite specimens were removed from the mold and stored in the dark for 24 h at 37 °C. After 24 h, the specimens were placed under a microhardness indentation device (HMV-2000, Shimadzu, Tokyo, Japan). A fixed load of 200 g was applied for 10 s (Vickers pyramid, a diamond right pyramid with a square base and an angle of $a = 136°$ between the opposite faces at the vertex, where a is the angle of the pyramidal diamond intender). Vickers hardness measurements were performed on the top and bottom surfaces of the specimens (0 and 2 or 4 mm depth). Four indentations were made for each specimen's surface, one in every quadrant (>100 μm from each other), and these were independently averaged and reported as the Vickers hardness number (VHN). Because that the surfaces were in direct contact with a polyester film and provided a uniform surface luster, no polishing was performed.

Statistical Analysis

Having preliminarily checked that data distribution was normal in each material and that the group variances were homogeneous (with Kolmogorov–Smirnov test and Levene test, respectively), we applied a one-way analysis of variance to verify the existence of statistically significant differences; we then performed the Tukey test for *post hoc* comparisons (Bonferroni corrected). The previous analysis was followed for each combination of depth and light-curing mode ($3 \times 4 = 12$). Additionally, the data were analyzed by a two-way analysis of variance to define how the material and light-curing mode affected the VHN level. In all of the analyses, the level of significance was set at $a = 0.05$. The relationship between the mean VHN and the filler load (vol %) of the materials at the 4 mm depth was evaluated by linear regression. For composite microhardness measurements, the bottom surface hardness of the specimens should be 80% of the top surface hardness. Therefore, in this study, the reduction of the composite microhardness with depth, expressed in percentage VHN of the top surface of the specimens, was also calculated, and we noted when it was recorded as less than 80%.

Table I. Investigated Resin Composites

Material	Type	Organic matrix	Filler load (wt %, vol %)	Filler type (size)	Photoinitiator system	Manufacturer	Lot number
TEB	Nanohybrid bulk fill	Bis-GMA, UDMA, Bis-EMA	80, 61	Barium glass (0.04–3 μm)	CQ, TPO, Ivocerin	Ivoclar Vivadent, Schaan, Lichtenstein	R56348
BBR	Giomer bulk fill	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	87, 74.5	Fluoroborosilicate glass	CQ	Shofu, Inc. (Kyoto, Japan)	01131701
XF	Bulk fill	Bis-GMA, UDMA, TEGDMA	86, 70.1	Ba–B–Al silicate glass (0.05–10 μm)	N/A	Voco GmbH (Cuxhaven, Germany)	1311472
EXP	Fiber-reinforced bulk fill	Bis-GMA, PMMA, TEGDMA	74.2, 53.6	E-glass fiber (1–2 mm), barium borosilicate glass (0.1–2.2 μm)	CQ, DMAEMA	GC Corp. (Tokyo, Japan)	1308222
VB	Flowable bulk fill	UDMA, EBADMA	65, 38	Ba–Al–F silicate glass, YbF_3, SiO_2 (0.02–5 μm)	TPO	Heraeus Kulzer (Hanau, Germany)	010032
BBF	Flowable giomer bulk fill	Bis-GMA, UDMA, Bis-MPEPP, TEGDMA	72.5, 62	Fluoroborosilicate glass	CQ	Shofu, Inc. (Kyoto, Japan)	09121301
XB	Flowable bulk fill	UDMA, Bis-EMA	75, 58	N/A	N/A	Voco GmbH (Cuxhaven, Germany)	1310371
FB	Flowable bulk fill	Bis-GMA, UDMA, TEGDMA	64.5, 42.5	YbF_3 (0.1–5 μm), zirconia silica (0.01–3.5 μm)	CQ, EDMAB	3M ESPE (St. Paul, MN)	N421893
FZ	Nanohybrid	Bis-GMA, UDMA, Bis-EMA, PEGDMA, TEGDMA	82, 68	Zirconia silica ($\leq$3 μm)	CQ	3M ESPE (St. Paul, MN)	N407730

Bis-GMA, bisphenol A-glycidyl methacrylate; UDMA, urethane dimethacrylate; Bis-EMA, bisphenol A-ethyl methacrylate); TPO, 2,4,6-trimethyl benzoyl diphenyl phosphine oxide; Bis-MPEPP, 2,2-bis[(4-methacryloxypolyethoxy)phenyl] propane; TEGDMA, triethylene glycol dimethacrylate; N/A, not available; PMMA, poly(methyl methacrylate); DMAEMA, *N,N*-dimethyl aminoethyl methacrylate; EBADMA, ethoxylated bisphenol A dimethacrylate; EDMAB, ethyl-4-*N,N*-dimethyl aminobenzoate; PEGDMA, poly(ethylene glycol dimethacrylate).

Table II. Technical Characteristics of the Investigated LCUs According to the Manufacturers

LED	Curing mode	Exposure time	Output irradiance	Energy density (top)	Wavelength range	Diameter of the light tip	Manufacturer
Demi Plus	Pulse	20 s	1100–1330 mW/cm^2	~24 J/cm^2	450–470 nm	8 mm	Kerr Corp. (Orange, CA)
Bluephase C8	Soft start	15 s	650–800 mW/cm^2	11.25 J/cm^2	385–515 nm	10 mm	Ivoclar Vivadent (Schaan, Lichtenstein)
Valo	Standard	20 s	1000 mW/cm^2	20 J/cm^2	395–480 nm	10 mm	Ultradent (South Jordan, UT)
	Plasma	3 s	3200 mW/cm^2	9.6 J/cm^2			

RESULTS

The Vickers hardness measurements for the experimental groups in this study are presented graphically in Figure 1(a–d). The Vickers hardness values at the top surface of the specimens ranged from 9.32 ± 0.87 to 72.58 ± 6.52 VHN at 2 mm, from 7.50 ± 0.61 to 56.36 ± 2.36 VHN at 4 mm, and from 5.48 ± 0.32 to 54.34 ± 2.27 VHN. In almost all of the experimental groups, the microhardness values decreased as the measurement depth increased. At 0 mm, the highest VHN was observed in control group (FZ), whereas at 2 and 4 mm, the high-viscosity bulk-fill composites exhibited higher values than the control group ($p < 0.05$). Generally, the highest microhardness values were presented by XF followed by EXP, and the lowest were presented by VB followed by FB. The flowable bulk-fill composites exhibited significantly lower VHNs (almost the half) than the highly viscous bulk-fill composites at all depths, regardless of the light-curing mode applied ($p < 0.001$).

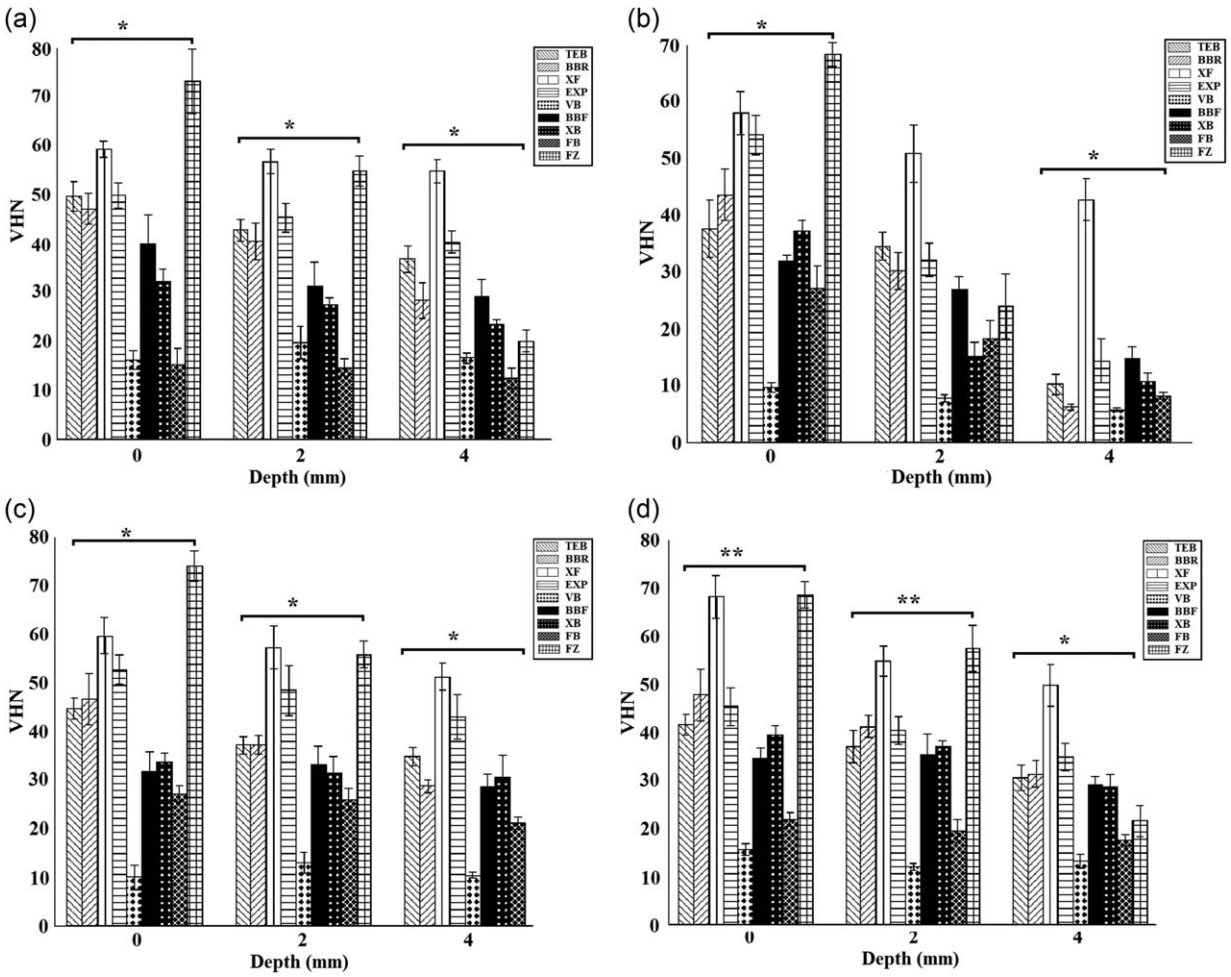

Figure 1. Mean values and standard deviations of the microhardness (VHN) of the experimental groups light-cured at three depths (0, 2, and 4 mm) with (a) Valo (*$p < 0.001$), (b) Valo plasma (*$p < 0.001$), (c) Bluephase (*$p < 0.001$), and (d) Demi Plus (*$p < 0.05$, **$p < 0.001$).

Table III. Reduction of the Microhardness at 2 and 4 mm Depths for Each Experimental Group Expressed in the Percentage of VHN of the Top Surface of the Specimens

Materials	Depth	Valo	Valo Plasma	Bluephase	Demi Plus
XF	2 mm	95.88%	87.66%	96.00%	80.27%
	4 mm	92.45%	73.62%[a]	85.92%	73.00%[a]
EXP	2 mm	91.05%	59.21%[a]	91.95%	88.79%
	4 mm	80.97%	26.40%[a]	81.55%	76.68%[a]
BBR	2 mm	85.75%	69.30%[a]	79.89%[a]	86.22%
	4 mm	60.13%[a]	14.29%[a]	61.72%[a]	65.54%[a]
TEB	2 mm	85.98%	91.58%	83.00%	89.02%
	4 mm	74.33%[a]	26.94%[a]	77.98%[a]	73.24%[a]
XB	2 mm	85.69%	40.64%[a]	93.50%	93.77%
	4 mm	73.32%[a]	28.77%[a]	90.50%	72.77%[a]
BBF	2 mm	78.32%[a]	83.92%	104.68%	102.03%
	4 mm	72.62%[a]	45.79%[a]	90.19%	83.79%
FB	2 mm	95.26%	67.40%[a]	95.38%	88.78%
	4 mm	80.52%	30.01%[a]	77.80%[a]	79.55%[a]
VB	2 mm	121.17%	80.47%	129.46%	76.32%[a]
	4 mm	101.99%	58.80%[a]	104.15%	83.73%
FZ	2 mm	74.78%[a]	35.02%[a]	75.38%[a]	83.62%
	4 mm	27.39%[a]	N/A	25.21%[a]	31.32%[a]

N/A, not applicable.
[a] VHN < 80% of the top surface.

The two-way analysis of variance revealed that both the material and light-curing mode affected the VHN level individually ($p_{mat} < 0.001$, $p_{led} < 0.001$), where p_{mat} is probability affected by material factor and p_{led} is the probability affected by curing-mode factor, but there was no evidence of a synergistic (interaction) effect of the two ($p_{mat*led} = 0.064$), where $p_{mat*led}$ is probability of interaction between material and curing mode. The light-curing mode affected the microhardness at all depths, but this influence was dependent on the material. The plasma experimental groups exhibited significantly lower microhardness values than the other light-curing modes at 2 and 4 mm depths ($p < 0.05$), except for XF at the 2 mm depth ($p = 0.449$). Moreover, at 4-mm depth, data for FZ (control) was not applicable (N/A) for plasma curing mode. The standard curing mode showed the highest mean VHN at 0 mm (FZ: 72.58 ± 6.52 VHN), 2 mm (XF: 56.36 ± 2.36 VHN), and also 4 mm (XF: 54.34 ± 2.27 VHN).

The reduction of the composite microhardness with depth for each experimental group expressed in percentage VHN of the top surface of the specimens is presented in Table III. At the 2 mm depth, all of the materials investigated met or were very close to the threshold value of the requirement for microhardness (≥80% VHN of the top), except for the plasma data, which were dependent on the material. At the 4 mm depth, there was a further reduction in the mean VHN for all of the materials evaluated in comparison with that at the 2 mm depth. However, the tested composites again met or were very close to the threshold value of the requirement, except for BBR (14.29–65.54%) and FZ (not applicable to 31.32%). The conventional resin composite FZ presented a significantly higher reduction in microhardness than all of the bulk-fill composites investigated at the 4 mm depth, although it did not exhibit the lowest VHN. Furthermore, neither of the composites light-cured with the plasma curing mode met the Vickers hardness requirement at 4 mm.

Regression analyses between the mean VHN and filler loading (volume percentage) of the materials for each light-curing mode at the 4 mm depth are illustrated in Figure 2(a–d). Linear regression confirmed a poor positive correlation between the mean VHN and the filler content of the composites for all of the curing modes (standard $r^2 = 0.29$, plasma $r^2 = 0.08$, soft start $r^2 = 0.08$, and pulse $r^2 = 0.44$), where r^2 is the coefficient of determination. A statistical method that explains how much of the variability of a factor can be caused or explained by its relationship to another factor.

Coefficient of determination is used in trend analysis. It is computed as a value between 0 (0 percent) and 1 (100 percent). The higher the value, the better the fit. Coefficient of determination is symbolized by r^2 because it is square of the coefficient of correlation symbolized by r. The coefficient of determination is an important tool in determining the degree of linear-correlation of variables ('goodness of fit') in regression analysis.

DISCUSSION

The results obtained from this study demand rejection of the first null hypothesis, that there is no difference in the Vickers hardness of materials light-cured with the same curing mode at the same depth. The tested bulk-fill RBCs varied markedly with respect to their Vickers hardness profiles at all measurement

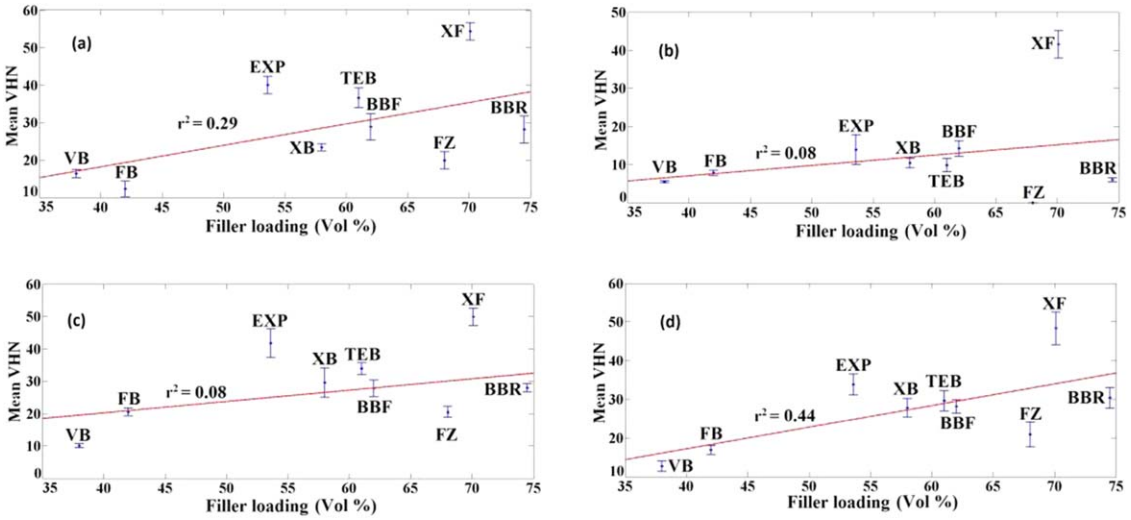

Figure 2. Regression analysis between the mean VHN and the filler loading (vol %) of the materials for (a) Valo (standard), (b) Valo (plasma), (c) Bluephase (soft start), and (d) Demi Plus (pulse) at a 4 mm depth. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

depths. These variations in microhardness may have been due to the different compositions of the materials with respect to filler particles, monomers, and photoinitiator systems. In addition, the curing efficiency of the bulk-fill materials at 4 mm was better compared to the conventional RBC, which was used as a control. This discrepancy may have been the result of an increased translucency of bulk-fill composites because their composition did not consistently differ from that of the conventional resin composite.[14]

In particular, the fillers of the bulk-fill RBCs increased in size to achieve a lower filler–matrix interface; this led to decreased light scattering and, as a result, to an enhanced light transmittance through the materials.[15] Another interesting issue with respect to the filler size of the resin composites was that as the filler size approached the output wavelength of the LCU used for polymerization, the light-transmitting ability was reduced because of the scattering of light on fillers of this size.[23] It has also been postulated that differences in the refractive indices between inorganic fillers and organic matrixes affect the translucency of the composites.[24] As a result, the increasing filler size of the bulk-fill RBCs improved their translucency. Conversely, it was found that with increasing filler content, the translucency of a composite material is reduced.[5] In contrast, the filler shape and type are not related to changes in the curing efficiency of resin composites.[23]

The variations in the monomer composition among resin composites may influence their degree of polymerization.[25] The initial viscosity and flexibility of monomers and their ultimate degree of polymerization crucially affect the polymerization efficiency of the composite materials.[26] Nevertheless, in this study, the materials that we investigated did not present main differences in the monomer composition to explain the results.

Furthermore, the degree of polymerization of a resin composite may be affected by the light absorbance of the photoinitiator system[27] and the included pigments.[28] In this study, the shade of all of the bulk-fill RBCs tested was Universal, whereas the photoinitiator system was camphorquinone (CQ), except for TEB, which contained an additional photoinitiator system called *Ivocerin*. This recently introduced photoinitiator, which is a dibenzoyl germanium compound, has higher photocuring activity than CQ; this leads to an enhanced degree of conversion in deeper layers of the material.[16] However, in our study, TEB showed lower Vickers hardness values than XF and EXP, which contained CQ as a photoinitiator, at all depths. This may have been due to the smaller filler size of TEB, which resembled conventional RBCs and led to lower translucency.[29]

The results of this study demonstrate that XF and EXP exhibited the highest microhardnesses at 2 and 4 mm. This evidence could be explained by the fact that larger fillers (>20 μm) were incorporated into the resin matrix of XF than the other composites; this led, as mentioned previously, to an increased translucency.[15] Also, EXP contained E-glass fibers, which provided better mechanical properties.[30] This was in agreement with similar reported results of previous studies.[15,31] Despite the high microhardness values of XF, in previous investigations, it has been reported that XF did not show reductions in shrinkage stress like the other bulk-fill RBCs that were tested compared to conventional composites.[18,31] BBR exhibited the lowest Vickers hardness values among the high-viscosity bulk-fill RBCs, perhaps because it presented the highest filler content; this led to the lower translucency.

The flowable bulk-fill composites tested in this study presented significantly lower microhardness values than the highly viscous bulk-fill composites, regardless of the polymerization conditions. These results coincide with the results of previous investigations.[31,32] Because of the lower mechanical properties of the flowable bulk-fill RBCs, the finishing of restorations with a layer of a high-viscosity bulk-fill RBC is recommended. To increase the curing efficiency of bulk-fill flowable composites in comparison with conventional ones, their filler size was increased,

whereas their filler load decreased.[29] In this investigation, XB and BBF exhibited higher Vickers hardness values than FB and VB. Perhaps the difference in the filler content was the main reason for this evidence. This finding supported the work of Ilie *et al.*,[29] who found that FB and VB presented very low hardness values. It, therefore, seemed of interest to verify to what extent the hardness of the composite restoratives was adequate for clinical use.

Linear regression analyses revealed a poor positive correlation between the Vickers hardness and filler load of the materials for all curing modes at 4 mm ($r^2 = 0.08–0.44$). This finding was expected because the variations in the microhardness of the materials could not be explained by only the differences in their filler load. Thus, other factors, such as the filler size or refractive indices between the fillers and matrix, may have been more important for the curing efficiency of these materials. This was in agreement with other recent studies.[18,29,31]

In this investigation, four different LED irradiation modes were evaluated for the curing efficiency of the bulk-fill composites. The results indicate that the curing mode affected the microhardness of the tested materials to different extents. Consequently, the second null hypothesis of the study, which states that the curing mode does not influence Vickers hardness of the materials, was rejected. Among the curing modes, the Vickers hardness values on the surfaces of the composite specimens were similar. However, at the 2 and 4 mm depths, the plasma curing mode exhibited a significantly lower VHN in all of the materials tested. This evidence has been reported in several previous studies.[15,33] Moreover, it has been found that soft-start and pulse-curing modes may present lower hardness values in composites at 2 mm compared with the standard curing mode, but they may reduce the crosslinking density of the composite polymeric network.[21] In addition, Neo *et al.*[22] reported the increased curing effectiveness of a resin composite at 2 mm by soft-start and turbopolymerization regimens. Nevertheless, the results of this study do not support these findings, and it might be argued that the differences observed between these curing modes were more material-dependent.

Furthermore, the different technical characteristics of the LEDs, such as the light intensity, wavelength range, exposure time, and diameter of the light tip, may have affected, to some extent, the results of our study. In this study, the energy density produced on the top surface of the specimens by the light-curing modes evaluated varied markedly (9.6–24 J/cm^2), and may have been the main reason for the differences in the Vickers hardness values among the group specimens of the same material light-cured with different curing modes.[14,34]

As a matter of fact, we postulated that the degree of conversion at a certain depth of a composite depends on the light energy delivered. Consequently, the energy density at 4 mm was the crucial parameter for the sufficient light curing of a bulk-fill RBC; this was directly related to the translucency[35] of the material and the technical characteristics of the LCU.[14] As we concluded from the results of our research, short exposure times, such as in the plasma curing mode (3 s), provided low energy density at the 4 mm depth, and this led to insufficient polymerization of the bulk-fill composites.[33]

In this study, almost all of the tested bulk-fill materials met or were close to the requirement of 80% VHN of the top at 4 mm when light-cured with three out of four curing modes (except plasma). Some bulk-fill materials presented values below the threshold value of the microhardness requirement, but this finding was reported previously in comparative studies.[36] Perhaps the filler composition of the materials reduced their translucency, and this led to lower energy density in the deeper layers. Moreover, it was reported[37] that the coefficient factor (k) was much lower for the bottom surfaces compared with the top surfaces of the composite specimens. This reduction in the value of k was more severe for the material with a higher concentration of photoinitiator and a higher percentage of glass filler particles in the wavelength range affecting the photopolymerization. It was argued that the relationship between k and the irradiation intensity could be used to quantify the decay of irradiated light with its penetration into the resin composites.

CONCLUSIONS

Within the limitations of this *in vitro* study, we concluded that the tested bulk-fill RBCs were adequately polymerized at the 4 mm thickness. However, the efficiency of polymerization depended on the light-curing mode and the composition of the material. Short exposure times, such as the plasma curing mode, provided low energy density at deeper layers, and this led to insufficient polymerization of the bulk-fill composites. As a result, it is not recommended for the light curing of this category of composites. In contrast, the other tested protocols of curing modes (standard, soft-start, and pulse) provided adequate polymerization at the 4 mm depth. A poor positive correlation between the Vickers hardness and filler load of the materials was revealed. The clinical significance of the study was that the dental practitioner has to be aware of the technical characteristics of the composite material to use the appropriate LCU and to choose the right application technique. Further clinical and laboratory studies regarding the physical and mechanical properties of bulk-fill RBCs are necessary to estimate their clinical performance.

ACKNOWLEDGMENTS

The authors thank Dr. Stavros Dimitriadis for his contribution to the statistical analysis of the data of this study.

AUTHOR CONTRIBUTIONS

Dimitrios Dionysopoulos contributed to the idea, hypothesis, experimental design, performance of the experiment, and writing of the manuscript. Kosmas Tolidis contributed to the idea, experimental design, and proofreading of the manuscript. Paris Gerasimou contributed to the proofreading of the manuscript and substantially to the discussion.

REFERENCES

1. Sakaguchi, R. L.; Douglas, W. H.; Peters, M. C. *J. Dent.* **1992**, *20*, 183.

2. Ferracane, J. L.; Mitchem, J. C.; Condon, J. R.; Todd, R. *J. Dent. Res.* **1997**, *76*, 1508.
3. Sideridou, I. D.; Achilias, D. S. *J. Biomed. Mater. Res. B* **2005**, *74*, 617.
4. Silikas, N.; Eliades, G.; Watts, D. *Dent. Mater.* **2000**, *16*, 292.
5. Amirouche-Korichi, A.; Mouzali, M.; Watts, D. C. *Dent. Mater.* **2009**, *25*, 1411.
6. Sideridou, I. D.; Karabela, M. M. *J. Appl. Polym. Sci.* **2008**, *110*, 507.
7. Lizymol, P. P.; Kalliyana Krishnan, V. *J. Appl. Polym. Sci.* **2007**, *107*, 3337.
8. Rueggeberg, F. A.; Caughman, W. F.; Curtis, J. W., Jr.; Davis, H. C. *Am. J. Dent.* **1993**, *6*, 91.
9. Torno, V.; Soares, P.; Martin, J. M.; Mazur, R. F.; Souza, E. M.; Vieira, S. *J. Biomed. Mater. Res. B* **2008**, *85*, 166.
10. Okulus, Z.; Buchwald, T.; Voelkel, A. *J. Appl. Polym. Sci.* **2015**, *132*, 42812.
11. Catelan, A.; Mainardi, M. D.; Soares, G. P.; De Lima, A. F.; Ambrosano, G. M.; Lima, D. A.; Marchi, G. M.; Aguiar, F. H. *Acta Odontol. Scand.* **2014**, *72*, 898.
12. Flury, S.; Peutzfeldt, A.; Lussi, A. *Dent. Mater.* **2014**, *30*, 1104.
13. Flury, S.; Hayoz, S.; Peutzfeldt, A.; Husler, J.; Lussi, A. *Dent. Mater.* **2012**, *28*, 521.
14. Ilie, N.; Keßler, A.; Durner, J. *J. Dent.* **2013**, *41*, 695.
15. Ilie, N.; Stark, K. *J. Dent.* **2014**, *42*, 977.
16. Moszner, N.; Fischer, U. K.; Ganster, B.; Liska, R.; Rheinberger, V. *Dent. Mater.* **2008**, *24*, 901.
17. Jenson, M. E.; Chan, D. C. N. In Posterior Composite Resin Dental Restorative Materials; Vanherle, G., Smith, D. C., Eds.; Peter Szulc: Utrecht, The Netherlands, **1985**; p 243.
18. El-Damanhoury, H.; Platt, J. *Oper. Dent.* **2013**, *39*, 374.
19. Moorthy, A.; Hogg, C. H.; Dowling, A. H.; Grufferty, B. F.; Benetti, A. R.; Fleming, G. J. *J. Dent.* **2012**, *40*, 500.
20. Roggendorf, M. J.; Kramer, N.; Appelt, A.; Naumann, M.; Frankenberger, R. *J. Dent.* **2011**, *39*, 643.
21. Feitosa, V. P.; Fugolin, A. P. P.; Correr, A. B.; Correr-Sorbinho, L.; Consani, S.; Watson, T. F.; Sinhoreti, M. A.; Sauro, S. *J. Dent.* **2012**, *40*, 802.
22. Neo, B. J.; Soh, M. S.; Teo, J. W.; Yap, A. U. *Oper. Dent.* **2005**, *30*, 671.
23. Turssi, C.; Ferracane, J.; Vogel, K. *Biomaterials* **2005**, *26*, 4932.
24. Azzopardi, N.; Moharamzadeh, K.; Wood, D. J.; Martin, N.; van Noort, R. *Dent. Mater.* **2009**, *25*, 1564.
25. He, J.; Liu, F.; Vallittu, K. P.; Lassila, L. V. G. *J. Polym. Res.* **2012**, *19*, 9932.
26. Sideridou, I.; Tserki, V.; Papanastasiou, G. *Biomaterials* **2002**, *23*, 1819.
27. Schneider, L. F.; Cavalcante, L. M.; Prahl, S. A.; Pfeifer, C. S.; Ferracane, J. L. *Dent. Mater.* **2012**, *28*, 392.
28. Shortall, A. C. *J. Oral Rehabil.* **2005**, *32*, 906.
29. Ilie, N.; Bucuta, S.; Dreaenert, M. *Oper. Dent.* **2013**, *38*, 618.
30. Garoushi, S.; Vallittu, P. K.; Watts, D. C.; Lassila, L. V. *J. Dent. Mater.* **2008**, *24*, 211.
31. Leprince, J. G.; Palin, W. M.; Vanacker, J.; Sabbagh, J.; Devaux, J.; Leloup, G. *J. Dent.* **2014**, *42*, 993.
32. Arlahlah, A.; Silikas, N.; Watts, D. C. *Dent. Mater.* **2014**, *30*, 149.
33. Lohbauer, U.; Rahiotis, C.; Kramer, N.; Petschelt, A.; Eliades, G. *Dent. Mater.* **2005**, *21*, 608.
34. Dewaele, M.; Asmussen, E.; Peutzfeldt, A.; Munksgaard, E. C.; Benetti, R.; Finne, G.; Leloup, G.; Devaux, J. *Dent. Mater.* **2009**, *25*, 1576.
35. Anfe, T. E.; Caneppele, T. M.; Agra, C. M.; Vieira, G. F. *Braz. Oral Res.* **2008**, *22*, 358.
36. Garcia, D.; Yaman, P.; Dennison, J.; Neiva, G. *Oper. Dent.* **2014**, *39*, 441.
37. Abedian, B.; Garcia-Godoy, F.; Garcia-Godoy, F. *J. Appl. Polym. Sci.* **2005**, *97*, 426.