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Influence of increment thickness on radiant energy and microhardness of bulk-fill resin composites

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Determining the energy transferred at the bottom of eleven bulk-fill resin composites, comparing top and bottom microhardness's and evaluating the correlation between microhardness and radiant energy were aimed. Samples were placed over the bottom sensor of a visible light transmission spectrophotometer and polymerized for 20 s. The bottom and top Knoop microhardness were measured. Paired *t*-test and correlation analysis were used for statistics ($p \leq 0.05$). In all groups, the bottom radiant energy decreased significantly with increasing thickness. For groups of Aura 2 mm, X-tra Fil 2 and 4 mm, SDR 2 and 4 mm, X-tra Base 2 mm no significant difference was found between top and bottom microhardness. For the bottom levels of Aura, X-tra Fil, Filtek Bulk-Fill Posterior, SDR, X-tra Base groups no significant difference was found between the microhardness's of 2 and 4 mm thicknesses. For X-tra Fil, Tetric Evo Ceram Bulk-Fill, Filtek Bulk-Fill Flowable and Z100 groups radiant energy affected positively the microhardness.

Keywords: Radiant energy, Microhardness, Degree of conversion, Bulk-fill resins

INTRODUCTION

Beside the ease of handling and complete control over working time of light-cured resin composites, problems related to polymerization shrinkage and depth of cure have been implicated in causing unfavorable outcomes for restorations. Due to light absorption and scattering phenomena, resin composites may be polymerized to a limited depth. In many previous studies, a maximum thickness of 2 mm has been suggested for an adequate resin polymerization[1-3]. However, this procedure is clinically time consuming and has certain disadvantages, such as the possibility of contamination, failures in bonding between resin composite layers, and void formation[4]. To overcome these problems, new types of resin composites with the possibility of being cured in increment thicknesses up to 4, 5 and 6 mm have been introduced to the dental market as "bulk-fill" resins. This new group of material has been developed based on more translucent formulations, having alternative resins and initiators[5-6] and different filler technologies[7]. However, obtaining sufficient degree of conversion (DC) at all depths[8] may still be a challenge for these bulk fill resin composites. As mentioned above, the efficiency of the curing light decreases by absorption and scattering at increasing depth of resin composite materials[9]. Also, the type of the light curing unit used[10], the translucency of the material[11], the type and shade of the resin composite[12-14], the distance of the light guide tip and the exposure time[15] are all factors that affect the overall light transmission through the resin composite. Higher DC values typically result in higher hardness, elastic modulus, and color stability of the resin composites. Moreover, the solubility, the water sorption[16] and the biocompatibility[17] of the resin material are also positively affected.

When curing a resin composite restoration, the critical issue is the total radiant energy received by the resin (J/cm^2)[18]. If a resin composite does not receive enough photons, the polymerization of the material will be inadequate. This situation becomes important especially at the bottom of the restorations. Delivery of adequate irradiance (mW/cm^2) at the correct wavelengths for an appropriate period of time and from a suitable position is required to optimize curing results. It is especially important to measure this light delivery at the deepest regions of the restoration in order to confirm that stated depths of cure beyond 2 mm for new materials are truly achievable.

The DC can be measured by microhardness tests, Fourier Transform Infrared Spectrometer (FTIR), microscopy and scraping techniques. The basis of microhardness measurements depend on the idea of 'resistance to the deformation'[19]. The deformation is usually made by a pyramidal diamond shaped indentor and the indentation depth is measured with a microscope. This is one of the most common method used to evaluate the effectiveness of the polymerization of light-cured materials today. Further, the assessment of bottom/top surface hardness ratio has conventionally been used to evaluate depth of cure, or light cure effectiveness. The threshold value of 0.8 has been used as a criteria for adequate polymerization for light cured resin composites[20,21] though there the actual clinical relevance of this value is not known.

The aims of this study were: (1) to measure the

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radiant energy transferred to the bottom level of six bulk-fill restorative resin composites and four bulk-fill resins used as base material in comparison to one conventional resin composite; (2) to compare the top and bottom microhardnesses of the same materials to confirm that they could achieve their stated depth of cure and (3) to evaluate the correlation between microhardness and radiant energy. The study tested the following hypothesis: (1) While the material gets thicker, the radiant energy decreases at the bottom level of the material; (2) There is no difference between the top and the bottom microhardness of the materials tested; (3) There is no difference between the bottom level microhardness of each group; (4) Higher radiant energy increases the bottom microhardness of the resin composites and (5) All materials exceed the threshold value of 0.8 after polymerization.

MATERIALS AND METHODS

Commercially available six bulk-fill restorative resin composites, four bulk-fill base resin composites and one conventional resin composite, all of the same shade designation, were tested. The recommended thicknesses, filler loads, compositions, and manufacturers are presented in Table 1.

Table 1 Resin composites used (information acquired from manufacturers)

Material	Code	Type	Maximum applicable thickness (mm)	Composition	Filler (%wt)	Manufacturer
Aura	AU	Bulk Fill Restorative Material	6	UDMA, Bis-EMA, Bis-GMA, Amorphous SiO_2, Barium aluminasilicate glass, prepolymerized filler	81	SDI, Bayswater, Australia
Filtek Bulk Fill Posterior	FBP		5	AUDMA, UDMA, DDDMA, zirconia/silica, ytterbium trifluoride	76.5	3M ESPE, Seefeld, Germany
SonicFill	SF		5	Ethoxylated bisphenol A dimethacrylate, bisphenol-A-dimethacrylate, triethyleneglycol dimethacrylate, barium glass, silicon dioxide	83.5	Kerr, Orange, USA
X-tra Fill	XF		4	Inorganic fillers in a methacrylate matrix, Bis-GMA, UDMA, TEGDMA	83.5	Voco, Cuxhaven, Germany
Tetric EvoCeram Bulk Fill	TEC		4	Bis-GMA, Bis-EMA, UDMA, barium glass, ytterbium trifluoride, mixed oxideand prepolymer, additives, catalysts, stabilizers, pigments	81	Ivoclar Vivadent, Schaan, Liechtenstein
Admira Fusion X-tra	AD		4	Ormocer resin, CQ, amine, BHT, SiO_2 nano particles, glass ceramics	84	Voco
Filtek Bulk Fill Flowable	FBF	Bulk Fill Base Material	4	Bis-GMA, UDMA, Bis-EMA, procrylat resin, ytterbium trifluoride, zirconia/silica	64.5	3M ESPE
SDR	SDR		4	Ba-Al-F-B silicate glass, Sr-Al-F silicate glass, modified UDMA, EBPADMA, TEGDMA, camphorquinone, photoaccelerator, BHT, UV stabilizer, titanium dioxide, iron oxide pigments, fluorescing agent	68	Dentsply, DeTrey, Konstanz Germany
X-tra Base	XB		4	Inorganic fillers in a methacrylate matrix (aliphatic dimethacrylate)	75	Voco
Venus Bulk Fill	VB		4	Multifunctional methacrylate monomers (UDMA, EBADMA), Ba-Al-F silicate glass, YbF_3, SiO_2	65	Heraeus Kulzer, Hanau, Germany
Z100	Z100	Conventional composite	2	Bis-GMA, TEGDMA, silica/zirconia	71	3M ESPE

The samples were prepared in accordance to the manufacturer's claim for curing depth for each resin composite material tested. For this reason, some of the materials used in the study like Aura, SonicFill and Filtek BulkFill Posterior are polymerized up to 5 or even 6 mm as claimed by their manufacturer's. For sample preparation, Delrin discs of 6 mm diameter and 1, 2 or 3 mm depth were stacked where needed to produce final depths of 2, 4, 5 and 6 mm. Five samples were prepared for each thickness group. A transparent Mylar strip was placed on the bottom of the molds, which were filled with the resin composite, and then a second Mylar strip and a glass slide were used to squeeze out the excess of the materials from the top.

Samples of each resin composite were placed over the bottom sensor of a visible light spectrophotometer (Marc Resin Calibrator, BlueLight Analytics, Halifax, Canada) and photo-polymerized for 20 s using an LED curing unit (SmartLite Focus, Dentsply, Milford, DE, USA) under standard curing mode with an output wavelength range of 460–490 nm. The light guide tip was positioned 1 mm above the samples. Irradiance at the bottom of the resin composites were recorded during the curing procedures and multiplied by time to determine the total radiant energy.

After curing, all the samples were stored in distilled water at 37°C for 24 h prior to Knoop microhardness measurements. For each material, microhardness was measured three times on the top and bottom of the cured samples in different locations using a Knoop diamond pyramid (Struers Duramin, Struers, Ballerup, Denmark) with a 100 g (0.98 N) load and 20 s of indentation time. The average of the microhardness values for the top and the bottom measurements was calculated and the bottom/top ratios in percentage were calculated.

Data were reported as Mean±SD. Paired sample *t*-test was used to compare two related means. The correlation analysis was used to determine whether or not two variables were correlated. SPSS version-15 (Statistical Package for the Social Sciences, SPSS, Chicago, IL, USA) was used to perform all statistical analyses ($p \leq 0.05$).

RESULTS

The mean radiant energy at the specimen bottoms of

Table 2 Mean radiant energy, standard deviations and *p* values of all groups at the bottom ($p \leq 0.05$)

Material	mm	Mean Radiant Energy (J/cm²)	±SD	*p*
AU	2	5.04	0.320	2 mm>4 mm: $p=0.000$ 2 mm>5 mm: $p=0.000$ 2 mm>6 mm: $p=0.000$ 4 mm>5 mm: $p=0.008$ 4 mm>6 mm: $p=0.000$ 5 mm>6 mm: $p=0.003$
	4	1.32	0.192	
	5	0.9	0.07	
	6	0.56	0.054	
FBP	2	5.44	0.371	2 mm>4 mm: $p=0.000$ 2 mm>5 mm: $p=0.000$ 4 mm>5 mm: $p=0.001$
	4	1.84	0.207	
	5	1.02	0.13	
SF	2	3.3	0.254	2 mm>4 mm: $p=0.000$ 2 mm>5 mm: $p=0.000$ 4 mm>5 mm: $p=0.000$
	4	0.76	0.054	
	5	0.4	0.00	
XF	2	5.88	0.268	2 mm>4 mm: $p=0.000$
	4	2.2	0.158	
TEC	2	5.88	0.334	2 mm>4 mm: $p=0.000$
	4	2.2	0.122	
AD	2	5.46	0.427	2 mm>4 mm: $p=0.000$
	4	1.98	0.13	
FBF	2	4.8	0.886	2 mm>4 mm: $p=0.001$
	4	1.98	0.13	
SDR	2	7.74	0.456	2 mm>4 mm: $p=0.000$
	4	3.4	0.264	
XB	2	8.94	0.23	2 mm>4 mm: $p=0.000$
	4	4.5	0.339	
VB	2	9.74	0.167	2 mm>4 mm: $p=0.000$
	4	5.34	0.194	
Z100	2	4.4	0.452	—

Table 3 The mean microhardness values and comparison of top and bottom microhardness for every thickness in each group ($p \leq 0.05$)

Material	mm	Mean Microhardness N/mm²	Mean Microhardness Kg/mm²	±SD	p
AU	2T	362.06	36.92	1.061	2T=2B: p=0.137
	2B	339.89	34.66	2.136	
	4T	360.49	36.76	1.718	4T>4B: p=0.045
	4B	338.32	34.5	0.484	
	5T	357.35	36.44	1.844	5T>5B: p=0.001
	5B	272.82	27.82	1.19	
	6T	350.88	35.78	0.759	6T>6B: p=0.000
	6B	243	24.78	0.349	
FBP	2T	526.02	53.64	1.15	2T>2B: p=0.001
	2B	452.67	46.16	0.676	
	4T	524.06	53.44	1.089	4T>4B: p=0.000
	4B	426.39	43.48	1.184	
	5T	531.12	54.16	1.137	5T>5B: p=0.000
	5B	407.76	41.58	0.511	
SF	2T	941.83	96.04	1.993	2T>2B: p=0.000
	2B	822.97	83.92	0.58	
	4T	947.71	96.64	2.89	4T>4B: p=0.000
	4B	703.33	71.72	2.025	
	5T	923.19	94.14	1.099	5T>5B: p=0.000
	5B	582.31	59.38	0.746	
XF	2T	737.46	75.2	0.946	2T=2B: p=0.161
	2B	723.92	73.82	1.868	
	4T	714.9	72.9	1.159	4T=4B: p=0.206
	4B	705.88	71.98	1.273	
TEC	2T	632.72	64.52	1.023	2T>2B: p=0.003
	2B	581.73	59.32	2.053	
	4T	635.86	64.84	0.835	4T>4B: p=0.000
	4B	469.54	47.88	0.712	
AD	2T	623.7	63.6	0.452	2T>2B: p=0.000
	2B	556.42	56.74	0.82	
	4T	624.09	63.64	0.702	4T>4B: p=0.000
	4B	454.04	46.3	0.418	
FBF	2T	320.48	32.68	1.874	2T>2B: p=0.000
	2B	276.93	28.24	1.372	
	4T	316.95	32.32	1.227	4T>4B: p=0.002
	4B	252.22	25.72	2.328	
SDR	2T	314.79	32.1	1.668	2T=2B: p=0.115
	2B	283.6	28.92	2.206	
	4T	314.59	32.08	1.336	4T=4B: p=0.090
	4B	283.8	28.94	3.219	
XB	2T	407.95	41.6	2.498	2T=2B: p=0.051
	2B	373.43	38.08	0.715	
	4T	392.85	40.6	1.218	4T>4B: p=0.005
	4B	352.05	35.9	1.816	
VB	2T	406.77	41.48	1.158	2T>2B: p=0.006
	2B	384.22	39.18	0.311	
	4T	405.79	41.38	0.84	4T>4B: p=0.000
	4B	329.89	33.64	0.32	
Z100	2T	1062.06	108.3	2.486	2T>2B: p=0.017
	2B	1015.77	103.58	1.037	

all groups are shown in Table 2. In bulk-fill restorative resins groups; XF (5.88±0.268 J/cm^2) and TEC (5.88±0.334 J/cm^2) and in bulk-fill base resins; VB group (9.74±0.167 J/cm^2) showed the maximum energy transfer to the bottom level at 2 mm while the control group's energy transfer was 4.40±0.452 J/cm^2. In all groups, the bottom level radiant energy decreased significantly with increasing material thicknesses ($p \leq 0.05$).

The mean microhardness values and comparison of top and bottom hardness of all groups are shown in Table 3. For groups AU 2 mm, XF 2 mm and 4 mm from the bulk-fill restorative resins; SDR 2 mm and 4 mm and XB 2 mm from the bulk-fill base resins, no significant difference was found between the top and the bottom microhardnesses. For the other groups, top microhardness values were higher than the bottom microhardness ($p \leq 0.05$).

The comparison of bottom microhardness values of each groups are shown in Table 4. For the bottom values of AU and XF groups from bulk-fill restorative resins, FBF, SDR and XB groups from bulk-fill base resins, no statistically significant difference was found between the microhardness's of 2 and 4 mm thicknesses. For the remaining groups, increasing the material thickness was found to have a significant negative effect on the bottom microhardness of the materials ($p \leq 0.05$).

The correlation of radiant energy and microhardness for all groups are shown in Table 5. For XF, TEC, FBF and Z100 groups, increase in the radiant energy affected positively the microhardness ($p \leq 0.05$). There was no correlation between the radiant energy and the microhardness for the other groups.

The bottom/top ratios in percentage of all groups are shown in Fig. 1. All of the four bulk-fill base resins (FBF, SDR, XB and VB) showed microhardness at 4 mm equal or exceeding the 80% threshold. For the bulk fill restoratives, only AU, FBP and XF materials exceeded the 80% threshold value, though the others were within 10%. None of the materials claiming greater than 4 mm depth of cure could met the threshold value, although FBP was very close.

Table 4 Comparison of bottom microhardness in each group. (Each group was evaluated separately within itself) ($p \leq 0.05$)

Material	mm	Mean Bottom Microhardness (N/mm^2)	Mean Bottom Microhardness (Kg/mm^2)	±SD	p
AU	2	339.89	34.66	2.136	2 mm=4 mm: p=0.859 2 mm>5 mm: p=0.002
	4	338.32	34.5	0.484	2 mm>6 mm: p=0.001
	5	272.82	27.82	1.19	4 mm>5 mm: p=0.000
	6	243	24.78	0.349	4 mm>6 mm: p=0.000 5 mm>6 mm: p=0.009
FBP	2	452.67	46.16	0.676	2 mm>4 mm: p=0.014
	4	426.39	43.48	1.184	2 mm>5 mm: p=0.001
	5	407.76	41.58	0.511	4 mm>5 mm: p=0.031
SF	2	822.97	83.92	0.259	2 mm>4 mm: p=0.000
	4	703.33	71.72	0.905	2 mm>5 mm: p=0.000
	5	582.31	59.38	0.333	4 mm>5 mm: p=0.000
XF	2	723.92	73.82	1.868	2 mm=4 mm: p=0.113
	4	705.88	71.98	1.273	
TEC	2	581.73	59.32	2.053	2 mm>4 mm: p=0.001
	4	469.54	47.88	0.712	
AD	2	556.42	56.74	0.82	2 mm>4 mm: p=0.000
	4	454.04	46.3	0.418	
FBF	2	276.93	28.24	1.372	2 mm=4 mm: p=0.065
	4	252.22	25.72	2.328	
SDR	2	283.6	28.92	2.206	2 mm=4 mm: p=0.992
	4	283.8	28.94	3.219	
XB	2	373.43	38.08	0.715	2 mm=4 mm: p=0.071
	4	352.05	35.9	1.816	
VB	2	384.22	39.18	0.311	2 mm>4 mm: p=0.000
	4	329.89	33.64	0.32	
Z100	2	1015.77	103.58	1.037	—

Table 5 Correlation between radiant energy and microhardness at the bottom of the samples for each resin composite group ($p \leq 0.05$)

	Material										
	AU	FBF	SF	XF	TEC	AD	FBF	SDR	XB	VB	Z100
p	0.798	0.377	0.209	0.016	0.030	0.086	0.048	0.372	0.156	0.117	0.044
r	0.160	0.512	0.677	0.942*	0.914*	0.825	0.882*	0.518	0.737	0.783	0.889*

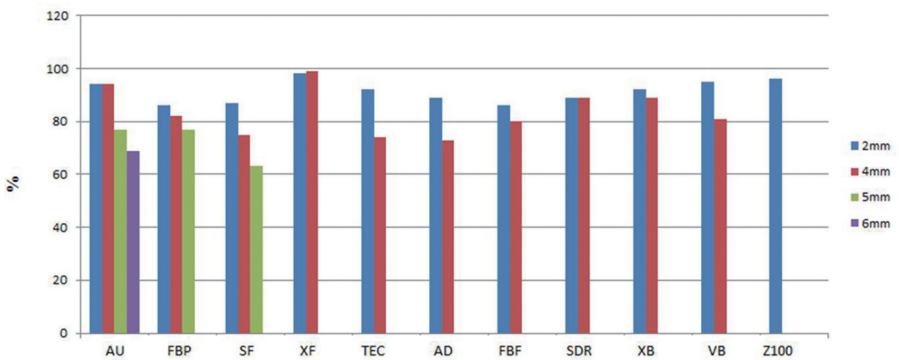

Fig. 1 The bottom-top microhardness ratios as a percentage for the evaluated resin composite groups.

DISCUSSION

In this study, six bulk-fill restorative resin composites, four bulk-fill resins used as base materials, and one conventional resin composite were evaluated in terms of light transfer through the material to the bottom layer during light curing. In general, though not always, the materials met the manufacturer's claims for depth of cure, and there was a reasonable correlation between higher radiant energy at the base of the material and higher microhardness values.

In previous studies, it has been reported that increasing resin composite thickness reduced the transmission of the polymerizing light[22,23]. In the current study, the radiant energy measured at the bottom of the samples was shown to decline with thickness, consistent with these previous studies. This was true for all materials, even those designed to provide deeper cure, *i.e.* bulk fills.

During application of bulk-fill resins in deep cavities, increasing irradiation time period or using a higher power light source would be useful in order to deliver enough energy to the bottom layers of the restoration[24]. However, there is still no consensus about the absolute energy value necessary to obtain an optimum polymerization for every resin composite. This value depends on the translucency, type and shade of the resin composite, as well as the type of photoinitiator[25]. Furthermore, the filler type used in the resin composite is one of the most important factors affecting light penetration through the material. Higher filler loading, especially with smaller particles, results in a greater number of resin matrix/filler particle interfaces that leads to increased light scattering because of the difference in refractive indices between the filler and the matrix resin[26]. The absorption of the light by the photo initiators and the pigments present in the resin composites also decreases the energy transferred to the bottom level of the restoration[27].

It has been reported that after light curing, the polymerization of resin composites continues for up to 24 h[28,29]. Therefore, microhardness measurements are usually performed after that time. Accordingly, in our study microhardness measurements were performed after 24 h's post-curing. It was expected that differences existing directly after curing would still be present when testing after 24 h, because Price *et al.*[12] found that insufficient light activation could not be compensated by waiting 24 h at 37°C. However, in their study, microhardness was not measured after 24 h. In our study, the surfaces were not polished before testing because the thickness of the samples made them difficult to handle. Moreover, avoiding polishing, ensured that the potential heat generated during polishing that may cause an increase in polymerization was also avoided. Though a study by Park *et al.*[30] found no significant difference in microhardness for polished surfaces and those cured against a Mylar strip, at least after six days.

DC is generally evaluated indirectly with microhardness tests[31]. In one study, direct measurement of the residual unreacted carbon double

bonds by FTIR was found to be less sensitive than microhardness assessments in detecting small changes in cure[32]. Also Knoop microhardness correlates well with the DC of the restorative resins[33]. Therefore, the evaluation of DC was estimated by Knoop microhardness in our study.

In previous studies, it has been shown that bottom surface microhardness levels were lower than those at the top surface in all specimens, regardless of the curing light used[34,35]. In this study, most of the composites did show reduced microhardness at the bottom of the 4 mm specimens compared to the 2 mm specimens, except for XF and AU from bulk-fill restorative resin groups and SDR, XB and FBF from the bulk-fill base resin groups. However, it should be noted that although the microhardness for all of the other composites showed a reduction at 4 mm *vs.* 2 mm, these composites were still as hard or harder at 4 mm than AU, FBF, SDR and XB. This suggests that these materials may still have clinically acceptable microhardness at greater depth, even though they showed this decline.

In a study of Flury *et al.*[36], a conventional resin composite and a bulk-fill restorative resin (Tetric EvoCeram) showed a significant decrease in microhardness at the bottom of specimens with increasing thickness, but certain bulk-fill base resins (SDR, Filtek BulkFill) remained the same. In the current study, similar findings were obtained. In groups AU and XF from the bulk-fill restorative resins, and in groups FBF, SDR and XB from the bulk-fill base resins, no significant difference was found between the bottom of 2 and 4 mm thick samples. For the other groups, increasing increment thickness reduced the bottom level microhardness.

While the depth of cure is influenced by many factors, such as the chemical structure of the monomers, filler composition, curing time and light intensity[37], in this study, standard conditions were provided for curing time and light intensity, and each group was evaluated within itself so chemical structure and filler compositions were also constant parameters. As mentioned above, in all groups, increasing increment thicknesses reduced the energy transferred to the bottom level of the samples. Thus, despite the reduced energy delivered to the bottom of the specimens, certain materials still cured sufficiently to show a consistent microhardness throughout their depth, as claimed by the manufacturers.

At the bottom of XF, TEC, FBF and Z100, a positive correlation between radiant energy and microhardness was found. In previous studies, a linear relationship between microhardness and the logarithm of energy received by resin composites[38,39], and an exponential relationship between DC and radiant energy[29] have been reported. The possible reason that certain materials showed this correlation and others did not is not obvious, but is most likely related to compositional differences.

In many studies, it was shown that the minimum value suggested for an effective light curing procedure based on bottom to top hardness ratio was 0.8[21,40,41]. In this study, AU, FBP, and XF from the bulk-fill restorative resin group and all of the bulk-fill base resin groups exceeded this threshold value at 4 mm thickness. The reason for this is likely due to the higher light transmittance within these specific resin composites. However, AU, FBP and SF claim depths of cure exceeding 4 mm, though this was not achieved in this study. It is possible that curing with a light of higher power would have allowed these materials to exceed their own claims and further studies need to be performed to evaluate these claims.

Nowadays, bulk-fill resins are often preferred because of their clinical ease of use and time savings properties. According to the results of this study, it was shown that some bulk-fill resins, and especially those used as base materials, can be used safely in clinical situations in terms of microhardness and DC. We believe that further studies performed with high power lights will give more insight into these materials.

CONCLUSIONS

Within the limitations of this study, the following conclusions may be drawn:

1. Increasing the thickness of the resin composite material reduced the energy delivered to the bottom in all groups.
2. XF and SDR resin composites showed no differences in the comparison of top and bottom microhardnesses.
3. In groups AU, XF, FBF, SDR and XB, no significant difference was found between the microhardness values of 2 and 4 mm thicknesses.
4. A positive correlation between the radiant energy and the microhardness at the bottom levels was found in XF, TEC, FBF and Z100 groups.
5. XF, FBF, SDR, XB and VB groups exceeded the threshold value for bottom to top hardness ratio of 0.8 at 4 mm, however the three resin composites claiming depth of cure exceeding 4 mm did not meet the threshold.

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