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MICROHARDNESS OF COMPOSITE RESINS AT DIFFERENT DEPTHS VARYING THE POST-IRRADIATION TIME

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ABSTRACT

Objective: The purpose of this study was to assess the microhardness of posterior composite resins at different depths varying the post-irradiation time. Materials and methods: For each composite resin [Solitaire 2 (SO) – Heraus Kulzer, P60 (P) - 3M, Prodigy Condesable (PC) - Kerr, Surefil (S) - Dentsply and Alert (A) - Pentron], 6 specimens (3 mm in diameter; 4mm high) were prepared using a black polyurethane cylindrical matrix. The resins were inserted in a bulk increment and light cured for 40 seconds. Microhardness was analyzed at different depths (top, 0.4 mm, 1.0 mm, 2.0mm, 3.0 mm and 4.0 mm) and at two moments (20 minutes and 24 hours after light-curing). Data were analyzed by ANOVA and Tukey's test ($p<0.05$). Results: Overall, microhardness means decreased significantly with the increase of depth, being lower in the first moment tested. P, S and PC showed the highest microhardness means. Conclusion: It may be concluded that the tested composite resins presented a gradual decrease of microhardness as depth increased and this drop was more accentuated for depths beyond 2 mm. For all materials, higher microhardness means were recorded 24 hours after light activation. P60 yielded the best results at the different depths evaluated.

Uniterms: Microhardness; Composite resins; Light-curing units; Depth; Post-irradiation time.

INTRODUCTION

Because of their remarkable evolution and improved physical and chemical properties, the use of composite resins for rehabilitation of posterior teeth has increased considerably[14]. The improvements in the currently available composite materials include the increase of filler content, variations in size, type[14] and morphology of the particles, in addition to changes in the organic matrix[20]. Together, these changes have conferred higher mechanical strength and modulus of elasticity to these materials[24], therefore allowing them to be used in areas subjected to great masticatory efforts[14]. However, it is common sense that incomplete polymerization of composite restorations is one of the major clinical problems to be overcome[16] because since inadequate resin activation compromises the restoration both mechanically[19] and biologically[10]. The non-polymerized components may influence the material's chemical stability, increasing its susceptibility to degradation and leading to release of by products, such as formaldehyde[17] and acid metacrylates[29], which increases the possibility of pulpal adverse reactions and decreases the wear resistance and color stability[8].

Polymerization of the core of the restoration is directly related to the material's chemical composition, the organic (type of matrix) or inorganic portion, type, morphology and filler contents[11]. Moreover, it is influenced by the thickness of the increment inserted into the cavity[10], intensity and irradiation time, light spectrum[23] and distance of the tip of the light-curing unit to the material to be activated[7].

A wide array of composite resins for posterior teeth is currently available, with different chemical compositions and different physical and chemical characteristics. This leads to the need of studies that assess the real properties of such composites and determine the thickness resin increments to be used for posterior fillings. Therefore, the purpose of this study was to assess the microhardness of posterior composite resins at different depths varying the post-irradiation time. The test hypothesis is that there will be no difference among the materials, the post-irradiation time and the microhardness at different depths.

MATERIAL AND METHODS

The tested materials and their composition, specifications and manufacturer information are displayed on Table 1.

Thirty specimens were prepared using a black polyurethane hemi-cylindrical matrix with 6 mm in diameter and 4 mm in height. The matrix was attached in a metallic clamping device and a stainless steel sheet was used to bisect the cavity diameter and provide a smooth and flat composite surface for Vickers microhardness measurement. The matrix cavity was filled with a single increment of the following composite resins: Solitaire 2 (SO) - Heraus Kulzer; P60 (P) - 3M; Prodigy Condensable (PC) - Kerr; Surefil (S) - Dentsply and Alert (A) - Pentron. The composite resin increment was covered with a clear polyester matrix strip and a 1-mm-thick glass slide, which was gently pressed under a load of 200 gf during 1 minute. The material was light cured during 40 s using a visible light-curing unit with 450 mW/cm^2 output (XL3000, 3M/ESPE, St Paul, MN, USA).

The matrix was thereafter removed from the clamping device, thus providing a hemi-cylinder with the same dimensions of the bisected cavity (3 mm diameter; 4 mm height). Six specimens *per* material were fabricated and stored in a lightproof receptacle with distilled water at 37°C.

Vickers hardness was measured on the surface in contact with the stainless steel sheet using a micro-indentation tester (Shimadzu Micro Hardness Testers HMV-2, Shimadzu Corporation, Kyoto, Japan) with a 100 gf load applied during 45 sec[18] at two moments: 20 minutes and 24 hours after light curing.

The specimens were individually fixed in a holder and positioned in such a way that the test surface was kept perpendicular to the indentator tip. Measurements were made at the top surface and at depths of 0.4 mm, 1.0 mm, 2.0 mm, 3.0 mm and 4.0 mm from the upper surface. In each specimen, 3 indentations equally spaced over a circle and not closer than 1 mm to the adjacent indentation or the margin of the specimen were made at all predetermined depths, means were calculated[18]. For all tested materials, microhardness means were calculated for all evaluated depths. Data were analyzed statistically by one way ANOVA for analysis of the materials and two-way ANOVA for

TABLE 1- Description of the composite resins

Material	Composition	Particles size	Percent
Solitaire 2 Heraus Kulzer #030225 Gruner Weg 11, D-63450 / Hanau	UDMA, Bis-GMA, TEGDMA and tetrafunctional monomers, bariumboroaluminosilicate glass, silicon dioxide, fluoraluminosilicate glass.	2 a 20µm	65 %/weight
P60 3M/ESPE #9AY St Paul, MN 55144-1000/ USA	Bis-GMA, UDMA and Bis-EMA; silica/ zirconia	0.01 a 3.5µm – 0.6µm	83 %/weight
Alert Pentron #8358 53 North Plains Industrial Road, Wallingford, CT-06492	Functional dimethacrylates of ethoxylated bisphenol A policarbonate resin, photoinitiator, amine accelerator, UV absorber, silane treated bariumboroaluminosilicate glass, silica and inorganic pigments	0.7µm	84%/weight
Prodigy Condensable Kerr #906433 1717 West Collins Orange, CA-92667	Bis-GMA, RCA Aluminoborosilicate, colloidal silica	0.6µm	80%/weight
SureFilDentsply #000418 Caulk-Milford, DE-19963-0359	Bis-GMA, TEGDMA Bariumboroaluminosilicate glass	0.8µm	84%/weight

analysis of depths and post-irradiation time. Tukey's test was used for multiple comparisons of the means at a 0.05 significance level.

RESULTS

Vickers microhardness means (±SD) recorded at the different depths as a function of the post-irradiation time for all tested materials are displayed on Table 2.

There was statistically significant difference ($p<0.05$) among the materials. P60 (67.58 Hv) was statistically different from the other composite resins and yielded the highest microhardness means ($p<0.05$), while Solitaire 2 (24.73 Hv) presented the lowest microhardness means. Surefil, Prodigy and Alert had statistically similar microhardness means to each other.

Comparing the depths and post-irradiation times, it was observed that, regardless of the post-irradiation time, microhardness means on the top was statistically different ($p<0.05$) from that of the other depths for all materials. There was a significant decrease in the means with depth, the bottom surface presenting the lowest means. Regarding the post-irradiation time, in general, for all materials, microhardness means recorded after 24 hours were higher and statistically different ($p<0.05$) from those measured 20 minutes after light curing.

Regarding the time x depth interaction, it was observed that all materials showed a significant decrease in microhardness with the increase of curing depth, mainly for depths beyond 1.0 mm. For Alert, Surefil and Solitaire, curing did not occur at the bottom the specimens (4.0 mm). For Solitaire in particular, resin was not cured beyond the depth of 3 mm. In addition, for Prodigy, Surefill and Solitaire there was no statistically significant difference between the post-irradiation time for the same depth.

DISCUSSION

Passage of light through the bulk of the restoration is limited by the dental structure and by the characteristics of the restorative material undergoing light activation[11,14]. This fact demands that resin materials are inserted into the cavity in increments[10] because polymerization at the top surface may be different from that at greater depths. Therefore, the effectiveness of composite resin curing may be assessed directly and indirectly. Direct methods that assess the degree of conversion are very complex, expensive and time-consuming[16]. Indirect methods include visual, scraping and microhardness testing. Incremental surface hardness has been shown to be an indicator of the degree of conversion[1].

Measuring the material's hardness at specific depths is one of the most used methods for assessing *in vitro* depth of polymerization. As a rule, high hardness means indicate an adequate polymerization[1]. In the present study, light source and intensity were standardized in order to relate the polymerization depth strictly to the material's composition.

It was observed that for all resins microhardness decreased gradually with the increase of the depth, as published elsewhere[4]. This may probably be attributed to the fact that light intensity was greatly reduced while passing through the bulk of the composite resin[3,5] due to light scattering and absorption, decreasing polymerization effectiveness[27]. This may possibly be ascribed to the optical properties of resins (optical transmission coefficient)[10], which vary with the material composition (particle type/contents, size and morphology)[10,11].

These findings are consistent with the outcomes of the present study, in which the tested composite resins exhibited

TABLE 2- Microhardness means and standard deviations of the materials

Solitaire 2	*0 h*	*24 h*
Top	44.50 (3.43) b	57.58 (8.79) a
0.4 mm	36.10 (6.97) bc	36.03 (0.92) bc
1.0 mm	34.30 (7.35) cd	35.22 (6.40) bcd
2.0 mm	27.45 (8.55) cd	25.62 (2.27) d
3.0 mm	0 (0) e	0 (0) e
4.0 mm	0 (0) e	0 (0) e
P60	*0 h*	*24 h*
Top	93.10 (3.57) b	109.78 (16.25) a
0.4 mm	81.87 (6.66) bc	91.30 (6.02) b
1.0 mm	78.58 (9.84) cd	92.28 (8.42) b
2.0 mm	66.23 (12.10) d	72.47 (9.14) cd
3.0 mm	44.92 (12.09) e	48.42 (11.56) e
4.0 mm	15.83 (12.10) f	16.14 (13.06) f
Surefil	*0 h*	*24 h*
Top	94.23 (4.96) a	97.10 (9.33) a
0.4 mm	79.70 (9.09) b	84.77 (20.69) ab
1.0 mm	61.78 (11.92) cd	74.52 (18.38) bc
2.0 mm	42.35 (10.95) e	53.07 (18.89) de
3.0 mm	17.32 (6.56) f	25.70 (13.26) f
4.0 mm	0 (0) g	0(0) g
Alert	*0 h*	*24 h*
Top	83.40 (7.27) a	89.92 (5.60) a
0.4 mm	61.03 (18.86) c	71,73 (17.58) b
1.0 mm	59.12 (15.28) c	62.43 (15.37) c
2.0 mm	35.12 (15.80) e	46.85 (16.58) d
3.0 mm	14.03 (6.65) f	21.92 (10.10) f
4.0 mm	0 (0) g	0 (0) g
Prodigy	*0 h*	*24 h*
Top	65.57 (5.16)bc	74.60 (6.86)ab
0.4 mm	72.10 (2.14)ab	80.38 (7.23)a
1.0 mm	62.50 (4.64)c	69.00 (4.12)bc
2.0 mm	47.75 (5.61)d	51.82 (5.37)d
3.0 mm	21.72 (6.32)ef	23.9 (6.77)e
4.0 mm	11.13 (4.52)g	15.59 (4.67)fg

different behaviors. However, all materials showed a significant decrease in microhardness for depths beyond 2 mm. Regarding polymerization depth, it was noticed that the tested materials behaved differently after light activation. P60 and Prodigy yielded the highest microhardness means for all depths, whereas Solitaire, Alert and Surefill exhibited a greater decrease of hardness with increase of depths. These results are in contrast with the manufacturers' instructions, which suggest insertion of greater amounts of composite. Alert's manufacturer (Pentron), for example, advises increments of up to 5 mm thick. These differences are mainly due to the filler content and optic modifiers present in the composition of the resins. Nevertheless, the manufacturers do not specify filler particle morphology or the type of optical modifiers in the resins, hindering a further analysis of the differences in the behavior of the tested materials.

If polymerization was effective (i.e. maximum cure of the specimens were achieved), an ideal 1:1 ratio should be reached and top surface hardness would be similar to that of the other depths. Nevertheless, it has been suggested that the hardness gradient should not exceed 10% to 20% (hardness ratio greater than 0.8) to adequately photo-activate composite resins[30]. In this study, the hardness ratio obtained for P60 and Prodigy was 0.8 up to 1-mm depth. For the other composite resins, the same value was found up to the 0.4-mm depth.

An important finding of this study was that 3 of the tested materials (Surefil, Alert and Solitare) were not polymerized at 4-mm depth. Solitaire was not polymerized beyond 3-mm depth, as previously reported[21,25]. This fact can compromise the success of the restorative treatment with posterior composites because the existence of unpolymerized resin in the bulk of the restoration may have deleterious effects, increasing the risk of secondary caries underneath the material, hypersensitivity[2], discoloration or even fracture of the restoration[13].

As regards the materials' composition, a positive relationship between hardness and inorganic particle contents has been observed[22], as an increase in filler content results in higher hardness means[24]. These findings are in agreement with those of the present study because the composite resin with the lowest filler content (Solitaire 2) had the lowest Vickers microhardness. Nevertheless, Prodigy Condensable exhibited intermediate hardness values in spite of its high load content. A possible explanation for this is the fact that hardness depends also on other factors, such as the type and size of filler particles, and the tested methodology[26]. In addition, other characteristics of the material may have contributed to these results, among which the organic matrix composition, as the polymerization level varies according to the amount of monomers and oligomonomers present in the composite resins[20].

Another issue addressed in the present study was the relationship between microhardness and post-irradiation time after photo-activation. It was observed that all tested materials presented higher hardness 24 hours after light curing, as published elsewhere[15]. A suitable explanation for this is that irradiation of the materials by visible light (over 470 nm wavelength) produces photo excitation of camphorquinone molecules, which react with amine, resulting in free radicals that start the polymerization reaction[12]. However, a significant amount of free radicals remains in the bulk of the restoration after irradiation ceases, allowing formation of polymer chains for up to 24 hours, which increases the microhardness means[28].

As a result from this, the polymerization reaction of the composite resins goes on for a certain time after photo-activation. Consequently, the accomplishment of finishing and polishing procedures immediately after light curing may undermine the mechanical properties of the restorative materials because after activation the resin exhibits great difference in microhardness values between the organic and inorganic components, which can result in loss of matrix and release of filler particles[6]. In addition, immediate polishing may influence adversely the formation of marginal cracks along the restoration. There are reports that indicate a direct relationship between delayed polishing and less formation of marginal cracks[9]. This suggests that polishing should be performed at least 24 hours after placement of the restoration in an attempt to preserve the mechanical characteristics of the restorative material.

In view of the findings of the present study and the literature[14,18], posterior composite resins should preferably be placed in increments no thicker than 2-mm in order to improve the mechanical characteristics of the material. These results confront the manufacturers' instructions for these materials because one of the advertised advantages of these resins is the use of increments thicker than 2 mm. Nevertheless, clinical studies are required to assess the ultimate performance of posterior composite resins used according to the manufacturers´ instructions.

CONCLUSIONS

Within the limitations of an *in* vitro study and under the evaluated conditions, the following conclusions may be drawn: 1. the tested composite resins presented a gradual decrease of microhardness with the increase of depth and this drop was more accentuated for depths beyond 2 mm. 2. For all materials, higher hardness means were recorded 24 hours after light activation; 3. P60 yielded the best results, regardless of depth and post-irradiation time. The test hypothesis was rejected.

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