Influence of Shade and Irradiation Time on the Hardness of Composite Resins

Álvaro DELLA BONA[1]
Vinícius ROSA[2]
Dileta CECCHETTI[3]

[1]*School of Dentistry of University of Passo Fundo, Passo Fundo, RS, Brazil*
[2]*University of São Paulo, São Paulo, SP, Brazil*
[3]*Institute of Biological Sciences, University of Passo Fundo, Passo Fundo, RS, Brazil*

This study tested the following hypotheses: 1. increasing light irradiation time (IT) produces greater values of superficial hardness on different depths (0 and 3 mm); and 2. a dark shade composite (A3) needs longer IT than a light shade composite (A1) to produce similar hardness. Disk-shaped specimens (n=24 *per* shade) were fabricated using a 3-mm-thick increment of composite resin (Z100). Specimens were randomly assigned to 3 groups (n=8) according to the IT (400 mW/cm2) at the upper (U) surface: A1-10 and A3-10: 10 s; A1-20 and A3-20: 20 s; A1-40 and A3-40: 40 s. Specimens were stored in black lightproof containers at 37°C for 24 h before indentation in a hardness tester. Three Vickers indentations were performed on the U and lower (L) surfaces of each specimen. The indent diagonals were measured and the hardness value calculated. The results were analyzed statistically by ANOVA and Tukey's test ($\alpha=0.05$). Statistically significant differences were found between U and L surfaces of each composite shade-IT combination (p=0.0001) and among the ITs of same shade-surface combination (p=0.0001), except between groups A1-20U and A1-40U, confirming the study hypothesis 1 and partially rejecting the hypothesis 2.

Key Words: resin composite, shade, hardness, irradiation time.

INTRODUCTION

Composite resin has been described as an esthetic restorative material with excellent physical and mechanical properties (1). Most composites are light-activated materials, meaning that they go through polymerization in the presence of light (1). As light passes through the bulk of the composite resin, it is absorbed, dispersed and attenuated, decreasing the irradiance and curing effectiveness (3), consequently, the surface layer close to the light source is better polymerized than the layers far from it. Therefore, thick composite increments result in either partially or non-polymerized layers, which can compromise the quality of the restorations with decreased mechanical properties, low color stability and risk of pulp aggression by the non-polymerized monomers (1,2).

The conventional quartz tungsten halogen light unit consists of a tungsten filament in an iodine or bromine gas filled bulb that produces a powerful white light with the irradiance of 400-800 mW/cm² (4). As the ideal wavelength to excite a-1,2-diketone is about 468 nm (1), thus the white light emitted from this type of curing unit must be filtered at the output to a range of blue light (400-500 nm) (5,6). Despite their popularity, quartz tungsten halogen units present several shortcomings, such as the restricted depth of cure, heat generation and relatively long irradiation time (7,8).

The irradiance is related to aspects as depth of polymerization, monomer conversion, gap formation, shrinkage, infiltration and adhesion (9). The irradiance of light emission depends on the power (Watts) of the curing unit as well as the time (seconds) and the surface area (cm²) where the light is spread over (9). The energy density (irradiance x irradiation time) (10) influences the degree and depth of cure and the mechanical properties

Correspondence: Dr. Álvaro Della Bona, Faculdade de Odontologia, Universidade de Passo Fundo, Caixa Postal 611/613, BR 285, Km171, Campus I, 99001-970 Passo Fundo, RS, Brasil. Tel: +55-54-3311-5142. Fax: +55-54-3316-8403. e-mail: dbona@upf.br

of light cured resin composite (1,8,10). The degree of polymerization of these materials is basically proportional to the material thickness and irradiation time (1), which depend on some variables, such as type of material, composite shade, distance and quality of light source. For an adequate polymerization of a 2-mm-thick composite increment is recommended an energy density between 21 and 24 J/cm^2 (11), therefore, to light cure a 2-mm-thick composite increment is required a minimum irradiance of 400 mW/cm^2 for 40-60 s (12-14) and 280 mW/cm^2 for 60 s to light cure a 1-mm-thick composite increment (12). The surface near the light source can be considered well polymerized for 20 s at an irradiance as low as 200 mW/cm^2 because there is no interference with light transmission (1,14). The amount of light exposure time indicated by the composite manufacturer must be extended whenever the irradiance is reduced due to distance or any other factor.

The addition of components, such as titanium dioxide (TiO$_2$) and aluminum dioxide (AlO$_2$) in the organic matrix is a common process to produce different shades of composite resin. It has been reported that the concentration of pigments difficult considerably the light transmittance through the bulk of composite resins due to the intensification of the optical density and, consequently, there is a decrease in the depth of polymerization (15).

Hardness is a measure of a material's resistance to localized plastic deformation (16). However, the term hardness may also refer to resistance to bending, scratching, abrasion or cutting, and gives an idea of relative easiness of finishing and polishing. The usual protocol to measure hardness uses indentation methods. A composite resin may be considered well cured when the hardness of the opposing surface is equivalent to at least 80-90% of the top surface hardness (14).

The purpose of this study was to evaluate the influence of shade and irradiation time on the hardness of composite resins, testing the following hypotheses: 1. increasing irradiation time produces greater hardness on different depths (0 and 3 mm), and 2. a dark shade composite (A3) needs longer irradiation time than a light shade composite (A1) to produce a similar hardness.

MATERIAL AND METHODS

Forty-eight disk-shaped specimens (5 mm diameter x 3 mm thick) were fabricated using A1 and A3 shades of a composite resin (Z100, 3M/ESPE, St. Paul, MN, USA). Eight specimens from each shade (n=8) were irradiated for either 10 s, or 20 s or 40 s using a quartz tungsten halogen unit (Ultra Lux 200; Dabi Atlante, Ribeirão Preto, SP, Brazil) calibrated at 400 mW/cm^2, as checked with a curing radiometer (Model 100 radiometer; Demetron Research Corp., Danbury, CT, USA). The curing unit tip was kept 1 mm from the specimen upper (U) surface by means of a transparent glass blade. After the irradiation time, specimens were storage during 24 h inside black containers at 37°C.

Three Vickers indentations (load: 0.1 kg; dwell time: 15 s) were performed in both U and lower (L) surfaces of each specimen, using a hardness tester (Duramin; Struers A/S, Rodovre, Denmark) according to the ISO6507-3:1998 specifications (16). The length of the two indentation diagonals (2a) were measured, averaged and divided by 2 to obtain "a" (mm), which is the average half diagonal of the indentation. H was calculated using H= 0.5 P/a^2, where "P" is the applied load (kg).

The results were analyzed statistically by ANOVA and Tukey's test (α=0.05).

RESULTS

Hardness means ($\pm$SD) (kg/mm^2) and the statistical grouping are summarized in Table 1.

The A1U10 group showed significantly lower mean hardness than the other two groups (A1U20 and A1U40) for the U surface of this composite resin shade (A1) ($\leq$0.05). At the L surface of A1 shade a significantly higher mean hardness was found at 40 s irradiation time (A1L40) than for the A1L10 and A1L20

Table 1. Hardness means ($\pm$SD) (kg/mm^2) and statistical groupings of all experimental groups.

	A1 Shade		A3 Shade	
	Upper	Lower	Upper	Lower
40 s	100.6 ± 2.6Aa	91.6 ± 6.1Ba	103.6 ± 1.9Aa	91.3 ± 5.9Ba
20 s	99.5 ± 3.6Aa	79.0 ± 7.6Bb	99.2 ± 3.2Ab	73.2 ± 4.0Cb
10 s	94.6 ± 4.4Ab	59.7 ± 3.1Bc	93.6 ± 2.9Ac	43.9 ± 1.3Cc

Different uppercase letters indicate statistically significant difference in lines; different lowercase letters indicate statistically significant difference in columns (ANOVA and Tukey's test, α=0.05).

groups, which means that values were also statistically different (p≤0.05).

Both surfaces (U and L) of A3 shade composite revealed significantly greater mean hardness values at 40 s curing time, followed by the 20-s and the 10-s curing time groups (p≤0.05).

Statistically significant differences were found between U and L surfaces of each composite shade-IT combination (p=0.0001) and among the irradiation times of same shade-surface combination (p=0.0001), except between A1U20 and A1U40 groups.

DISCUSSION

The polymerization of light-cured composite resins starts and is sustained when the rate of delivery photons from the source of light is sufficient to maintain the photo-absorbing compound, camphoroquinone, in its excited or triplet state. In this state, camphoroquinone overreacts with an amine-reducing agent in order to form free radicals (12). Thus, it must be emphasized that, in addition to the type of restorative material, the light-curing unit plays a very important role on the polymerization process. It is responsible for both the wavelength of the emitted light and the irradiance. As mentioned, a wavelength of 468 nm results in maximum absorption coefficient of the camphoroquinone to change into an excited state (1). The irradiation time is also correlated to the light unit but controlled by the operator. Therefore, the depth of polymerization depends on several factors inherent to the composite resin such as its chemistry, shade and translucency, catalyst concentration and sort and size of filler. The latter has a fundamental role on the light scattering, which, *per se*, consists of a limiting factor in the depth of polymerization (3). The light scattering is greater when the filler particle size is about one-half of the wavelength of activating light. It may become a problem once the manufacturers produce finer particle size in commercial composite resins (3). Other factors that may determine the depth of polymerization remain on the irradiance, irradiation time, emission spectra of the light source, thickness of composite increment, distance from the light source tip to the composite resin and others (10,17).

Insufficiently polymerized composite resin may present quite a lot of problems such as follows: poor color stability, greater stain uptake, risk of pulp aggression by non-polymerized monomers and portions of the material with different values of Young's modulus (1,17). It has

been reported that loading well-polymerized composite layers that are placed on poorly polymerized layers can lead the composite restoration to bend inward and displace, causing marginal fracture, open margins and cusp deflection (17).

The results of this study showed that the superficial hardness of a composite resin is indeed influenced by the irradiation time. As expected, for both shades, increasing the irradiation time, significantly increased the mean hardness values (p<0.05) (Table 1), except between A1U20 and A1U40 groups, confirming the first study hypothesis. This was observed for both surfaces, although it was more evident in the L surfaces than in the U surfaces. Despite the statistically different mean values, a short irradiation time of 10 s was able to produce a high hardness value on the U surface, which is in agreement with other studies (12,17).

It is feasible to consider the irradiation attenuation during photopolymerization as a factor responsible for the modest hardness values measured in the lower surface. This study showed that a 10-s irradiation time produced similar mean hardness value in the U surface than a 40-s irradiation time in the L surface. Therefore, only the 40-s irradiation time was found to be effective to properly polymerize the L surface. The 10-s and 20-s irradiation times resulted in mean values lower than 80% of the mean value for the corresponding U surface. Therefore, these irradiation times are considered inadequate to produce a sufficient degree of polymerization in a 3-mm-thick increment of the composite studied.

Significant differences were found for the mean hardness values recorded at U surfaces among the tested irradiation times. However, the greatest difference was less than 10% (A3 shade - from 103.6 kg/mm^2 at 40 s to 93.6 kg/mm^2 at 10 s).

It has been reported that dark shades exhibit a lower degree of polymerization compared to light composite shades, resulting in lower mean hardness values (15,18). This rationale was not confirmed by the results of the present study in the U surfaces, but it was confirmed for the L surfaces, when IT was not 40 s (Table 1). This phenomenon suggest that whenever a satisfactory degree of conversion is obtained, the superficial hardness is not further affected by the increase of time exposure or composite shade, which is in accordance with a recent study (19) and seems to be a reasonable explanation for the results of the present study. It has also been suggested that translucency, rather than shade, may be the limiting factor regarding the depth of cure for some composite resins.

Indeed, the depth of cure is strongly associated to material opacity (20).

Considering the shades A1 and A3 at same irradiation time and same type of surface (U or L), there was no significant difference between the mean hardness values (Table 1), except for the 10-s and 20-s irradiation times in the L surface, which rejected the second study hypothesis for the U surface.

Considering the irradiation time, only the groups exposed to a 40-s curing time were able to produce adequate mean hardness values for both U and L surfaces of same specimens (p=0.74) (Table 1). Yet, significant differences were found between U and L surfaces of same specimens (p=0.0001). Therefore, to achieve an adequate hardness throughout a 3-mm thick increment of composite resin the clinician should light cure it for 40 s.

Future studies should investigate the effect of translucency, rather than shade, on the depth of cure and hardness of composite resins.

RESUMO

Esse estudo testou as hipóteses de que: 1. maior tempo de fotopolimerização (TF) produz maior dureza superficial em diferentes profundidades (0 e 3 mm) de resina composta (RC); 2. a cor escura (A3) de RC exige maior TF do que a RC clara (A1) para obter o mesmo grau de D. Foram fabricados 24 corpos-de-prova para cada cor de RC em único incremento de 3 mm (Z100), dividindo-os em 3 grupos (n=8), de acordo com os TF (400 mW/cm^2) na superfície superior (S): A1-10 e A3-10: 10 s; A1-20 e A3-20: 20 s; A1-40 e A3-40: 40 s. Os corpos-de-prova foram armazenados sem luz, a 37ºC por 24 h, antes de aferir o grau de dureza nas superfícies S e inferior (I) com um durômetro. Foram realizadas 3 penetrações Vickers em cada superfície (S e I) dos corpos-de-prova (n=24). As diagonais das penetrações foram medidas e os valores de dureza calculados. ANOVA e Tukey foram usados nas análises estatísticas (α=0,05). Ocorreram diferenças significativas entre S e I para cada combinação de cor-TF (p=0,0001) e entre os TF de mesma combinação cor-superfície (p=0,0001), exceto entre A1S20 e A1S40, confirmando a hipótese 1, mas rejeitando parcialmente a hipótese 2.

ACKNOWLEDGEMENTS

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