Influence of polymerization time and depth of cure of resin composites determined by Vickers hardness

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ABSTRACT

Background: Adequate polymerization of resin composites could be considered as a crucial factor in obtaining good clinical performance, particularly in stress-bearing areas. An insufficient curing degree affects the resin composite’s chemical properties. The current in vitro study evaluated the influence of polymerization time and depth of cure of six commercial resin composites by Vickers microhardness (VK).

Materials and Methods: Six resin composites were selected: Three microhybrid (Esthet.X HD, Amaris, Filtek Silorane), two nanohybrid (Grandio, Ceram.X mono), and one nanofilled (Filtek Supreme XT). The VK of the surface was determined by a microhardness tester using a Vickers diamond indenter and a 200 g load applied for 15 s. The bottom to top mean VK ratio was calculated using the formula: Hardness ratio = VK of bottom surface/VK of top surface. Vickers hardness values of test materials during exposure time of 20 and 40 s and depths of cure of 2 and 3 mm were determined and compared. Data were analyzed using analysis of variance (ANOVA) test.

Results: For all the tested materials and with all the exposure time periods, hardness ratio was higher than the minimum value indicated in literature (0.8). Exposure time and depth of cure did not affect hardness ratio values for Filtek Silorane, Grandio, and Filtek Supreme XT.

Conclusion: Among the materials tested, the nanofilled and the nanohybrid resin composites were rather insensible to thickness variations. Microhybrid composites, instead, had features different from one another.

Key Words: Depth of cure, hardness ratio, polymerization time, Vickers hardness

INTRODUCTION

The demand for esthetic restorations in dentistry has increased over the past few years. Since resin composites were first developed, many efforts have been made to improve the clinical behavior of these restorative materials.¹ Although both organic and inorganic phases might influence the material behavior, the filler particle features and rate are the most important factors affecting the mechanical properties of resin composites.²,³ Adequate polymerization could be considered a crucial factor in obtaining good clinical performance, particularly in stress-bearing areas. An insufficient curing degree affects the resin composite’s chemical properties such as water absorption, wear resistance, and strength, as well as the elution of possible irritant, allergic, or toxic components from the material.⁴,⁵ Factors such as the filler particle size, polymeric matrix, and radiant exposure generated by the light source can influence the degree of conversion of resin composites, and thereby influence their mechanical properties.

The degree of monomer conversion of resin composites can be measured using different testing techniques, either directly or indirectly. Among the indirect methods, surface hardness testing has been used in many studies because it has been shown to be a good indicator of the degree of conversion.
Surface hardness can be measured on the upper and lower surfaces of disk-shaped specimens with a given thickness. This method is used to measure the relative hardness, which is considered a good indicator for degree of conversion.

The filler particles’ features and rate are the most important factors related to improvement of the mechanical properties of resin composites.[6,7] Resin composites have been classified according to their filler particle size as hybrid (0.5–3 mm), microhybrid (0.4–1 mm), and microfilled (0.04–0.4 mm). More recently, the nanocomposites have been introduced, materials presenting high initial polishing ability combined with superior polish and gloss retention.[8,9]

The degree of cure of visible light activated dental resin composites is also strictly dependent on the characteristics of curing lights. A curing light intensity output depends on many factors (light guide, condition of the bulb, battery power); the mechanical properties of the resin composites are determined by total energy irradiation, which is clearly related to irradiation time chosen by the operator. Also, the distance of the light from the resin composite is a crucial factor.[5,10] Light-emitting diode (LED) curing lights have recently become very popular since they have a number of advantages over the conventional halogen units.

Hardness has been used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing tooth structures. To define depth of cure based on top and bottom hardness measurements, it is common to calculate the ratio of bottom/top hardness and give an arbitrary minimum value for this ratio in order to consider the bottom surface as adequately cured; values of 0.80 and 0.85 have often been used.[15,16]

Manufacturers of dental resins give recommendations about depth of cure as they relate to light activation. The most common indication is the use of a specific light intensity and exposure time which is able to cure a 2 mm thickness of the material.[12,13] Actually, in clinical practice, it is often difficult to make 2 mm regular increments, especially in cavities with irregular depth or in the presence of undercuts, where light intensity could be shielded by dental tissues.[14]

The current in vitro study focused on Vickers microhardness (VK) of six commercial resin composites: Filtek Supreme XT (3M ESPE, St. Paul, MN, USA), Amaris (Dentsply Caulk, Milford, DE, USA), Esthet.X HD (Voco, Cuxhaven, Germany), Grandio (VoK, Cuxhaven, Germany), Ceram.X mono (Dentsply Caulk, Milford, DE, USA), and Filtek Silorane (3M ESPE, St. Paul, MN, USA). Twelve samples for each material were prepared. After polymerization, the samples were stored for 24 h in complete darkness at 37°C and 100% humidity before performing the Vickers hardness test. The VK of the surface was determined.

### MATERIALS AND METHODS

Six resin composites (shade A2) were selected in accordance with their type of filler particles: Three microhybrid composites (Filtek Silorane, Esthet.X, Kalore), two nanohybrid composites (Grandio and Ceram.X mono), and a nanofilled resin composite (Filtek Supreme XT). The properties of the materials used are shown in Table 1. The chosen resin composites were light activated with an LED unit, Celalux II (Voco, Cuxhaven, Germany). A standard light polymerization mode was used for each material at 1000 mW/cm² intensity for 20 and 40 s. The hardness testing methodology used to assess the effectiveness of cure was based upon that used by Yap and Seneviratne.[12] Samples were prepared by placing the material into a stainless steel mold (Ø 7 mm, h 2-3 mm), placed on a dark opaque paper background covered with a polyester matrix strip. This arrangement minimized the possibility of artificially higher hardness due to light reflection.[15,16]

The mold was filled with the resin composite and a second polyester matrix strip was placed on the top of the filled mold. A glass slide was pressed against the upper polyester film to extrude the excess resin composite and to form a flat surface. The distal end of the light guide (diameter 10 mm) was placed against the surface of the matrix strip and positioned concentrically with the cavity in the mold; the material was then light-cured from the top. The cordless curing unit was maintained at full charge before use, and irradiance was monitored periodically using a radiometer (LED Radiometer, Kerr, Orange, CA, USA). Twelve samples for each material were prepared. After polymerization, the samples were stored for 24 h in complete darkness at 37°C and 100% humidity before performing the Vickers hardness test. The VK of the surface was determined.

### Table 1: Materials and manufacturers

| Shade  | Manufacturer          | Category    | Material     |
|--------|-----------------------|-------------|--------------|
| A2     | Dentsply Caulk, Milford, DE, USA | Microhybrid | Esthet.X HD |
| A2     | Voco, Cuxhaven, Germany          | Microhybrid | Amaris       |
| A2     | 3M ESPE, St. Paul, MN, USA       | Microhybrid | Filtek Silorane |
| A2     | Voco, Cuxhaven, Germany          | NanoHybrid  | Grandio      |
| A2     | Dentsply Caulk, Milford, DE, USA | NanoHybrid  | Ceram.X mono |
| A2     | 3M ESPE, St. Paul, MN, USA       | NanoFilled  | Filtek Supreme XT |
with a microhardness tester (Zwick-Roell durometer, ZHV1/2 Micro-Vickers, Italy) using a Vickers diamond indenter and a 200 g load applied for 15 s. Five VK readings were recorded for each sample surface (top and bottom); hardness measurements were not taken at more than 4 mm from the specimen center to avoid any possible effect of the mold on polymerization.\(^{[16]}\) For a given specimen, the five hardness values for each surface were averaged and reported as a single value. The mean Vickers hardness and hardness ratio of the specimens were calculated and tabulated using the formula: Hardness ratio = $\frac{VK_{bottom}}{VK_{top}}$ of bottom surface/VK of top surface.

### Exposure time
Mean Vickers hardness and hardness ratio of 2 mm specimens of the composite resins were evaluated after polymerization for 20 and 40 s.

### Depth of cure
Mean Vickers hardness and hardness ratio at 2 mm and 3 mm depth of the composite resins were compared choosing a polymerization time of 40 s for all the specimens.

### Statistical analysis
Differences in the averaged values amongst the groups were analyzed by analysis of variance (ANOVA) test and Tukey’s honestly significant difference (HSD) post hoc test. Statistical difference was set at $P < 0.05$.

### RESULTS

#### Exposure time
The mean Vickers hardness at 2 mm depth of top and bottom surfaces associated with the 20 and 40 s polymerization modes is shown in Table 2. Hardness ratio values for the two groups are reported in Table 3. Among the materials tested, only Grandio (Voco) showed a non-significant statistical difference ($P > 0.05$) in hardness values on both up and down sides and for 20 and 40 s polymerization time.

#### Depth of cure
The mean VK of top and bottom surfaces and hardness ratio at 2 and 3 mm depth associated with the 40 s polymerization mode is shown in Table 4. Hardness ratio values for the groups are reported in Table 5. All the materials tested provided values within the limit of 0.8.

### DISCUSSION

Resin composites are widely used in restorative dentistry and specifically in posterior restorations, putting the material under constant masticatory stresses.\(^{[17]}\) One of the most important parameters involved in resin composites’ resistance to stress is depth of cure.\(^{[18]}\)

The effectiveness of cure depends on the filler particle type, size, and quantity, and on the parameters (intensity, time, and polymerization modes) of the light source.\(^{[10,19]}\)

The optimal degree of curing throughout the bulk of a visible light-activated dental resin composite is acknowledged to be important to the clinical success of a resin composite restoration. Unfortunately, the dentist has no means of monitoring the cure of the resin surfaces not directly exposed to the curing light.\(^{[20,21]}\)

Moore et al.\(^{[22]}\) stated that one brand of composite in flowable, hybrid, and packable formulations did not achieve a 2 mm depth of cure with a 20 s light exposure. These data suggested that a 2-mm build-up layering technique may not result in adequate curing of the bottom layer for such a wide range of materials; manufacturers need to provide quantitative information about degree of conversion at specific

| Materials     | Polymerization time 40 s | Polymerization time 20 s |
|---------------|--------------------------|--------------------------|
| Top surface: Mean (SD) | Bottom surface: Mean (SD) | Top surface: Mean (SD) | Bottom surface: Mean (SD) |
| 55.6 (2.1)\(^a\) | 57.8 (1.1)\(^c\) | 45.4 (3.0)\(^a\) | 52.2 (1.5)\(^a\) |
| 39.8 (1.6)\(^a\) | 47.6 (2.7)\(^c\) | 34.8 (0.8)\(^a\) | 41.0 (1.6)\(^a\) |
| 45.0 (1.9)\(^b\) | 50.8 (0.8)\(^a\) | 45.6 (0.9)\(^b\) | 51.0 (1.6)\(^a\) |
| 98.4 (10.2)\(^a\) | 105.0 (3.2)\(^a\) | 101.0 (1.2)\(^a\) | 104.0 (5.7)\(^a\) |
| 58.2 (0.8)\(^a\) | 62.6 (0.5)\(^a\) | 44.8 (1.9)\(^a\) | 55.8 (2.4)\(^a\) |
| 79.2 (1.5)\(^b\) | 82.2 (1.9)\(^a\) | 79.8 (1.6)\(^b\) | 82.6 (1.1)\(^a\) |

\(^a\)Groups with the same letter did not show any statistically significant difference. **\(^{\text{**}}\) \(P > 0.05\)
activation time periods and light intensities for their entire range of resin materials and shades so that the dentist can devise a placement technique that will ensure adequate cure of the bulk of a restoration.

These findings are in disagreement with some studies which have shown that 2-mm increments are well polymerized.\(^{[18,23]}\)

Ferracane\(^{[2]}\) demonstrated good correlation between increasing hardness and increasing degree of conversion. Bouschlicher et al.\(^{[24]}\) concluded that the bottom-to-top surface microhardness ratios of a composite resin cannot be an accurate reflection of bottom-to-top degree of conversion; bottom-to-top microhardness and degree of conversion were independent of composite composition.

In the present study, two related aspects of resin composites’ polymerization were compared: Polymerization time of 20 and 40 s and depth of cure at 2 and 3 mm depth. In the first part of the experimentation, 2-mm-thick composite specimens were used as they ensured uniform and maximum polymerization.\(^{[11]}\) A2 shade was selected to minimize the effects of colorants on light polymerization.\(^{[19]}\)

The degree to which light-activated composites polymerize is proportional to the amount of light intensity to which they are exposed.\(^{[24]}\)

Ideally, the degree of polymerization of the composite should be the same throughout its depth and the hardness ratio should be very close or equal to 1. As light passes through the composite, the light intensity is greatly reduced due to light scattering, thus decreasing the effectiveness of cure at the bottom surface.\(^{[25,26]}\) It was suggested that hardness ratio should be greater than 0.8% for light-activated composites to be adequately polymerized.\(^{[27]}\)

Among the materials tested, only Grandio (Voco) showed a non-significant statistical difference (\(P > 0.05\)) in hardness values on both up and down sides and for 20 and 40 s polymerization time.

Comparing the two polymerization time periods, Grandio, Filtek Silorane, and Filtek Supreme XT gave no statistical difference (\(P > 0.05\)) between 20 and 40 s polymerization time periods on both sides. Amaris, Esthet.X HD, Ceram.X mono gave statistically different values (\(P < 0.05\)) between 20 and 40 s polymerization time periods on both sides. These results depend on the total amount of energy reaching the composite layer and on chemical composition of the composites.

Regarding hardness ratio, all the materials provided values within the limit of 0.8, although only for Ceram.X mono the value recorded coincided with the above-mentioned limit.

Hardness ratio values were not increased by increasing the polymerization time for Grandio, Amaris, Filtek Silorane, and Filtek Supreme XT; on the contrary, for Esthet.X HD and Ceram.X mono, hardness ratio increased by increasing the polymerization time. This means that for these materials, the mechanical properties will be affected by polymerization time.

Pires, et al.\(^{[21]}\) found that the top surface hardness of composites was less independent on light intensity than the bottom surface. The top surface is actually receiving the maximum energy from the curing light.

At the top surface it has also been established that even relatively low-intensity lights can cure the resin matrix to an extent almost equal to when high-intensity lights are used.\(^{[10]}\)

The general lack of significance between 20 and 40 s polymerization time periods in top Vickers hardness found in this study corroborates the above-mentioned studies. At the top surface, sufficient light energy reaches the photoinitiator, thus starting the polymerization reaction.

At the bottom surface, a significant difference in VK was observed, depending on their chemical composition, but hardness ratio values were not affected by these differences. Regarding the properties of the composite resins, the results were generally dependent on the material evaluated, especially with regard to filler features. Moraes, et al.\(^{[28]}\) suggested that no trend toward the size or shape of fillers affected hardness; Grandio, for instance, presented

### Table 3: Hardness ratio at 2 mm depth recorded after 20 and 40 s polymerization time periods

| Hardness ratio | Polymerization time 20 s | Polymerization time 40 s |
|----------------|-------------------------|-------------------------|
| Esthet.X HD    | 0.87                    | 0.96                    |
| Amaris         | 0.9                     | 0.89                    |
| Filtek Silorane| 0.89                    | 0.97                    |
| Grandio        | 0.90                    | 0.80                    |
| Ceram.X mono   | 0.97                    | 0.93                    |
| Filtek Supreme XT | 0.86                | 0.94                    |
the highest top and bottom VK values, probably because of its large particles and the highest filler content.\[29\] Nanofilled Filtek Supreme XT showed significantly higher hardness values than Esthet.X HD, Filtek Silorane, and Ceram.X mono, which were all conceived for posterior restorations.\[30\]

Except for Ceram.X, which was very sensible to polymerization time, the materials tested provided very good performances both at 20 and 40 s polymerization time periods.

The good values recorded for Grandio and Filtek Supreme XT are probably due to the presence of TEGDMA in the resin, which reduces the resin viscosity and increases the reactivity of the monomers.\[24\]

Analyzing the second part of the experiment, for all the materials tested, there was not a statistical difference (\(P > 0.05\)) in hardness values on the up side (irradiation side) with 2 and 3-mm thickness. A statistically significant difference (\(P < 0.001\)) was recorded on the opposite side for all the composite resins.

Despite this sharp reduction in hardness values for 3 mm thickness, the hardness ratio values were higher than 0.8 for all the materials tested.

These findings provided useful hints about clinical practice: The polymerization of a 3-mm composite resin increment for 40 s achieved a sufficient hardness ratio value for all the materials tested.

**CONCLUSION**

Among the materials tested, the nanofilled and the nanohybrid resin composites are very versatile and rather insensible to thickness variations. Microhybrid composites, instead, have features different from one another: Filtek Supreme XT showed little variation with increasing depth of cure, while Amaris and Esthet.X HD provided a sharp reduction in hardness values with increasing depth of cure.

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