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Influence of Light Source and Photopolymerization Distance on Composite Resin Microhardness

Fernanda Furtado Piras[1], Juliana Jendiroba Faraoni[1], Regina Guenka Palma-Dibb[1*]

1. Department of Restorative Dentistry, Ribeirão Preto School of Dentistry, University of São Paulo. Av. Café, s/nº - CEP: 14040-904 - Monte Alegre. Ribeirão Preto - SP, Brazil.

Abstract

The present study evaluated *in vitro* the influence of two light sources in composite resin microhardness in different distances of photopolymerization. Two light sources were used (halogen, LED) to photopolymerize the composite resin (Filtek Z250; Esthet-X) in different distances surface (1,2,3 and 4 mm), totalizing 96 specimens. Microhardness Knoop test' were made on the specimens top and bottom faces. The data were submitted to ANOVA and Fisher tests (α=5%). It was observed a significant difference ($p<0.05$) between the resins, in which Z250 presented higher values than Esthet-X ($p<0.05$). There was difference also between the halogen and LED, where the halogen showed better results with Z250 and the LED with Esthet-X. The top surface presented better value ($p<0.05$) compared to the bottom surface. It can be concluded that the light source and the photopolymerization distance influenced the composite resin microhardness, which it had lower proprieties with increase of the photopolymerization distance.

Corresponding author:
Regina Guenka Palma Dibb, Departamento de Odontologia Restauradora, Faculdade de Odontologia de Ribeirão Preto, Universidade de São Paulo, Av. Do Café, s/n CEP 14040-904, Phone: 55-16-3315-4016 Fax: 55-16-3633-0999, Email rgpalma@usp.br
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Introduction

The search for excellence in esthetic dentistry and the high demand of esthetical procedures, had proportioned to development of the new studies to evaluate composite resin behavior, besides refining equipments and photopolymerization techniques[1] as well as reducing the clinical time spent in restorations by the professional.

The availability of resin systems activated by light led to the development of a wide variety of technologies to produce the necessary light to their use, as for example: the quartz-tungsten halogen light (QTH), is mostly used by professionals[2], and the light produced by diode (LED)[3], was introduced in 1995, have a wave length between 450-490nm, with a peak of 470nm[4].

In order to obtain an appropriate photopolymerization of a composite resin, it is necessary the right amount of light intensity[5], time and adequate wavelenght[6]. High light intensity promotes temperature increase[7] and consequently high values in terms of conversion measurement besides, better physical and mechanical properties from the restoratives materials. Moreover, the increase of temperature shows significant effect on microhardness[8].

Nevertheless, the decrease on the mechanic properties of hardness is an indicative of a lesser conversion measurement by the presence of residual monomers[9]. In this aspect, a correlation has been established between the microhardness of the material and the polymerization degree[10]. From this way, the microhardness tests have been an adequate indicator regarding the conversion degree of monomers in polymers[11], allowing investigation if the light source is capable of polymerize the restoration body properly. Therefore, hardness determines the deformation capability of the restoration material and allows a comparison with enamel and dentin, with the objective of finding restoration materials with similar properties of the dental structure. Additionally, the microhardness can be a simple test to obtain information of the materials properties[12].

For the reasons shown, it is valid the importance given to the realization of analysis of the significant advantages and disadvantages characteristics in the mechanical properties of the restorative material when different light sources are used to photopolymerize composite resin as well as the distance influence of the photopolymerizer. Depth of cure and microhardness are considered essential physical properties of composite resin, concerning the clinical technique of incremental insertion and curing[13].

The results obtained with this study intend to produce new knowledge regarding the polymerization of direct composites and their mechanical properties. The definition of an ideal protocol is the aim of the research in performance of restorations, considering that an adequate polymerization seems to be a crucial factor[14] and curing distance and light source are susceptible of causing deleterious effects in the mechanical properties of the restorative materials.

The objective of the present study was to evaluate *in vitro* the influence of two light sources (halogen and LED) on the microhardness of the photopolymerized composite resins in four different distances (1mm, 2mm, 3mm, 4mm) of the material surface.

Methods and Materials

The study factors were: light sources, in two levels (Jet Lite 4000 and Ultralume LED 5), composite resin in two levels (Z250 and Esthet-X) and photopolymerization distance (in relation to the material surface), in four levels (1 mm, 2 mm, 3 mm e 4 mm). The experiment sample was composed of 120 specimens of each restoration material divided randomly into 8 groups by the combination of the levels of the factors light source and photopolymerization distance, with fifteen repetitions per each experimental group. The quantitative response variable was the difference of the values of the Knoop microhardness, measured in KHN (*Knoop Hardness Numbers*), of the superior and inferior surfaces in percentages (%). To ensure the reliability of the results, each restoration material (Z250 and Esthet-X) represents one sample, without promoting their interaction, as well as the analyzed regions (top and bottom parts).

One hundred and twenty specimens were made of each composite resin (Table 1), using split bisected cylindrical matrix of black polyurethane of 6 mm diameter and 2 mm high, individually adapted and fixed in a clamping metallic device. The composite resin was introduced into the matrix in only one increment and the surface recovered by an acetate bind in which a weight of 500g was put upon per 30 seconds to obtain a sample with a plain and smooth surface and with a standard density of 2 mm.

The photopolymerization was made with one of the two equipment presented (Table 2), by the standard time of 20 seconds. The different distances between the equipment point and the surface of the specimen were established using black polyurethane rings, with heights correspondent to the distances (1mm, 2mm, 3mm, 4mm) which were positioned over the cylindrical matrix. The light intensity of the equipment was monitored after the photopolymerization of three specimens by radiometers (Optilux Radiometer– Demetron Kerr, Orange, CA, USA- for halogen bulbs and L.E.D. Radiometer–Demetron Kerr, Orange, CA, USA- for L.E.D.).

The thickness of all specimens, after they were made, were checked with a digital caliper (Mitutoyo 500-144B, Suzano, SP, Brazil), aiming to keep the desired thickness. The specimens with higher or lower thickness were rejected and remade. The specimens were stored in distilled water at 37°C, in a dark recipient without the incidence of light. 24 hours after the photopolymerization, the microhardness test was made.

The superficial microhardness of the superior and inferior faces of the specimens were obtained by using a microhardness tester (HMV-2000, Shimadzu Corporation – Kyoto, Japan) with a Knoop indenter under a 50-g load applied for 25 seconds. The specimens were individually fixed in a prehension device in a way that the evaluated surface (top and bottom

Table 1. Technical specifications of the resin composites used.

Material	Composition	Manufacturer 3M ESPE
Microhybrid resin composite (Filtek Z250)	Bis-GMA, UDMA, Bis-EMA. Zirconic/Sylic with 82% in weight (60% in volume). The average particles' size is 0.6µm.	St Paul. MN, USA
Nanoparticules resin composite (Esthet X)	Bis-GMA Modified Urethane, Bis-EMA, e TEGDMA. Glass of Boronsilicate of Aluminum Fluoride and Barium (1µm), Coloidal Silic (0.04µm) and nanometric Silic.	DENTSPLY Petrópolis, Brazil

Bis-GMA: Bisphenol A - di-glycidyl, ether di-methacrylate; UDMA: urethane di- methacrylate; Bis-EMA: Bisphenol A - poliethylene glycol di-ether, di-methacrylate; TEGDMA: tri-ethylene glycol di-methacrylate.

Table 2. Photopolymerization equipments used and their specification.

Trademark	Manufacturer	Light Type	Wavelength	Average intensity
Ultralume LED 5	Ultradent Products, INC. South Jordan, UT 84095, USA	LED	370 to 500nm	800mW/cm^2
Jet Lite 4000 Plus	J.Morita USA INC 9 Mason Irvine, CA 92618 USA	Halogen	400 to 500nm	900mW/cm^2

parts) stayed perpendicular to the indenter. For each surface of the specimen, 3 equally distant circularly indentations were made, being one central indentation and two lateral indentation with a 2 mm space from the central indentation and 1mm space from the specimen margin, obtaining their average.

The hardness values were analyzed in relation to the different regions assessed and also in relation to the hardness percentage between the top and the bottom part of the specimen obtained from the formula:

%hardness variation = ((top part hardness - bottom part hardness)*100)/top part hardness

The data was previously analyzed in relation to the materials and it was observed that a significant difference occurred between them and therefore it was decided to analyze them separately. The different regions (*top part and bottom part*) were analyzed in the same way and a significant difference was observed between them, thus it was decided to analyze them separately. After homogeneity of variance and normal distribution had been checked, a two-way analysis of variance (light source and distance of photopolymerization) and Fisher test (≥ 0.05) was carried out. The software Origin 8.5.1 (Northampton, MA, USA) was used to perform the statistical analyses.

Results

On the data analysis it was observed a significant statistical difference between the composite resins. Fisher's test showed that Z250 resin exhibited higher microhardness values than Esthet-X resin ($p<0.05$). Comparing the different regions (top and bottom part), it was observed that the bottom part presented lower values and statistically different from the top part ($p<0.05$). This way, the data were analyzed separately.

With regard to Z250 resin, it was observed that the photopolymerization equipment influenced the material microhardness, and the halogen equipment presented statistically superior values to the LED ($p<0.05$). The distances of the curing light also has negatively affected the values independent from the analyzed region (top or bottom part), the result was similar on both regions.

Tables 3 and 4 show that on 1mm and 2mm the halogen equipment promoted better and statistically significant values than the LED, however on the 3mm and 4mm distances, both equipments showed similar results. It is observed in Figure 1 that the percentage difference between the two analyzed regions (top and bottom part) showed similar results between equipments. An increase was showed only at the distance of 4mm between the regions that were statistically significant.

As for Esthet-X resin, it was observed that the photopolymerization equipment influenced the material hardness. The LED equipment showed superior statistical values than the halogen one ($p<0.05$) and that the distances of the photopolymerization did not affect the values independently from the analyzed region (top or bottom part), the result was similar in both regions.

Table 3. Means and Standard deviation (HK) at the top surface of the Z250 resin for the different equipments and photopolymerization distances. For the column comparison, same letter statistical similarity; for the line comparison, same symbol statistical similarity.

Z 250	H (halogen)	L (LED)
1mm	75.15 (± 1.06)a♦	72.22 (± 2.17)a ♣
2mm	72.08 (± 1.76)b♥	67.75 (± 2.77)b♠
3mm	65.48 (± 1.88)c•	66.76 (± 2.63)b•
4mm	65.05 (± 2.87)c•	66.40 (± 2.61)b•

Table 4. Means and Standard deviation (HK) of the bottom surface of the Z250 resin for the different equipments and photopolymerization distances. For the column comparison, same letter statistical similarity; for the line comparison, same symbol statistical similarity.

Z 250	H (halogen)	L (LED)
1mm	65.44 (±1.48)a•	61.46 (± 2.74)a♠
2mm	61.60 (± 2.52)b♥	57.75 (± 2.12)b♣
3mm	55.78 (± 3.81)c♦	56.21 (± 2.46)c♦
4mm	52.81 (± 2.07)d♦	54.35 (± 1.96)d♦

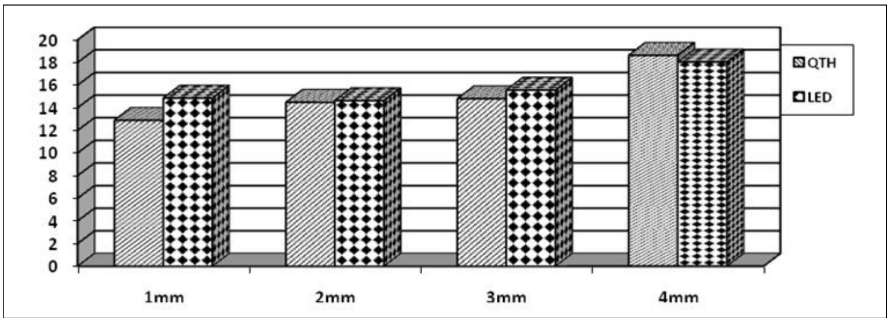

Figure 1. Means of the difference between top and bottom surface (%) resin of the resin Z250 for the equipments and photopolymerization distances.

Tables 5 and 6 show that at 1mm and 4mm the halogen equipment showed inferior and statistically significant values in relation to the LED; however, at the distances of 2mm and 3mm the equipments showed similar results in both regions. On Figure 2 it is observed that there was no difference in percentage between both analyzed regions (top and bottom part), that is, both equipments showed similar results.

Discussion

A good polymerization of resin composite is essential to obtain better mechanical properties from these materials[6,14]. Therefore, clinical longevity from adhesive restorations is linked the adoption of a proper photopolymerization protocol. The reduction of the biomechanical properties is indicators of a lower extension of conversion degrees, with excessive presence of residual monomers that can lead to deleterious effects for the restoration[11]. The composition

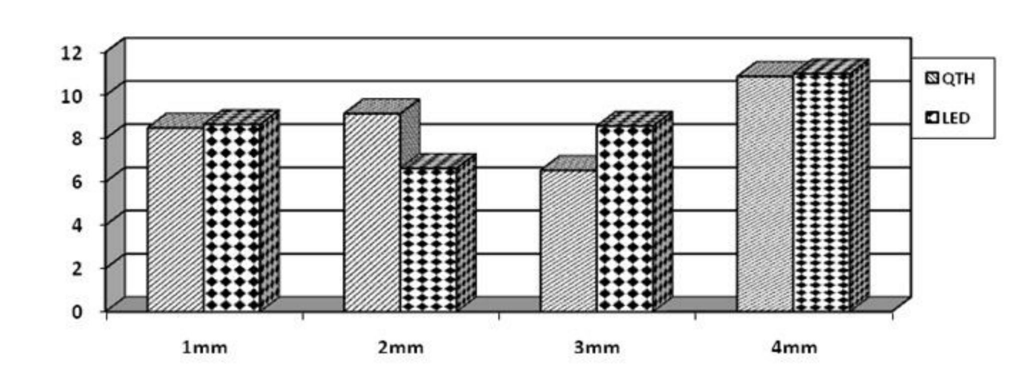

Figure 2. Means of the difference between top and bottom surface (%) of the resin Esthet-X for the equipments and photopolymerization distances.

Table 5. Means and Standard deviation (HK) of the top surface of the resin Esthet-X for the different equipments and photopolymerization distances. For the line comparison, same symbol statistical similarity.

Esthet X	H (halogen)	L (LED)
1mm	42.44 (±4.87)♦	49.46 (± 5.80)♣
2mm	40.50 (± 4.05)♥	49.35 (± 5.81)♠
3mm	43.88 (± 12.32)•	49.60 (± 5.73)•
4mm	41.81 (± 8.20)•	46.20 (± 9.72)•

Table 6. Means and Standard deviation (HK) of the bottom surface of the resin Esthet-X for the different equipments and photopolymerization distances. For the line comparison, same symbol statistical similarity.

Esthet X	H (halogen)	L (LED)
1mm	30.93 (±1.41) •	39.07 (± 5.35) ♠
2mm	30.60 (± 1.04) ♥	33.40 (± 2.67) ♥
3mm	32.08 (± 7.21) ♥	34.55 (± 3.97) ♥
4mm	31.26 (± 2.35) ♦	35.05 (± 4.06) ♠

of the composite resins associated with the characteristics of the light unit, such as sufficient radiante intensity, correct wavelength of the visible light and curing time[6], constitute important factors to obtain good materials properties.

In this present study, it was observed that the type of composite resin showed different behavior, in which it was observed that Z250 showed higher values than Esthet-X regardless of the light unit or the distance of the point in relation to the surface resin[8,15]. This fact can be explained due to the different composition of the materials, by having hybrid resins, microparticles resins and, more recently, nanotechnology resins, which imply different physical properties. The conversion degree differs between the materials, mainly by the variation found in the concentration of the weight particles, their format and disposition, as well as their organic matrix constitution[2,16,17]. Regarding the microhybrids, their weight volume is of 56 to 66% and the average size of the particles varies between 0.4 and 0.8mm. The particles size these resins is very little, allowing a high polish similar to the microparticle resins; however, their size is sufficient to a high filling, reaching therefore, a greater resistance and better mechanical properties. Moreover, there is difference in the organic and inorganic matrix of the resins[14,18], as Z250 showed particles with higher load[18], besides presenting zircon/silica, which are harder particles than the ones presented by Esthet-X resin[15].

In relation to the analyzed regions, the superficial hardness (top part) and to 2mm deep (bottom part), it was observed that the top region showed hardness values higher than the bottom part. These results are confirmed by the literature where it can be observed that the conversion degree depends on the quantity of incident light on the resin[14,20]. As a decrease in the light quantity happens in the deepest layers of the material, the conversion degree of such areas is lower and consequently the mechanical properties of the resin are negatively affected including Knoop hardness[15,22].

When the types of photopolymerizers were analyzed, it was observed that the microhardness was affected in a distinctive way to both studied resins. LED obtained the highest values for microhardness in relation to the halogen for Esthet-X, in return, Filtek Z250 resin was affected on an opposite way, that is, the LED showed lower values different from the ones observed by Correr et al.[19] where no difference between QTH and LED were observed. This fact might happen due to a high energy density equipment had been used. Adding to this, the distinctive result of the studied resins could be due to Esthet-X presents glass particles of fluorite-barium-aluminum silicate (1µm) and nanoparticles of silica (0.04 µm), and it has a uniform distribution. As LED possesses a more restrict wave length without light dispersion like it happens with the halogen light[15] it could help in a more effective polymerization. Besides, the microhardness is determined and controlled by the type and quantity of pigments employed on the composite resin[14] and as they are materials with distinctive compositions, their results are expected to be different. Corroborating with these results Beun et al.[23], also observed a proximity among the different types of resin, on their conversion degrees and on photoactivation with sources based on LED and halogen light.

As for halogen light, the bulb gradual deterioration, reflector and filter, together with the professionals negligence in maintenaning and monitorationing periodically the emitted light energy, lead them to present a frequent irradiation, many times inferior than 300mW/cm recommended average and consequently an inadequate polymerization[24]. The blue and cold light, ideal for polymerization, is generated by semiconductors dispositive of In-Ga-N (Indium-Gallium-Nitrogen) when submitted to an electrical current determined. It produces a luminous flux within the spectrum of maximum absorption of the photoinitiator molecule (camphorquinone), a component on the majority of the resins[25]. Nevertheless, studies have empathized that the first LED equipments presented reduced capability of activation due to the low power density, since there is a proportional relation between luminous intensity and cure extension[20].

Another analyzed factor was the distance from the tip of the curing light with the resin surface and it was observed that the farther it is, lower it is the hardness value, especially in relation to Z250. This could be due to a decreased light intensity according to the distance increased from the tip of curing to the resin surface[20]. Nevertheless, this factor did not happen with Esthet-X resin, which could have been explained by nanoparticle resin, which can attenuate the light spread[26] and consequently the effect of the distance it further and the light dispersion is less affected than the microhybrid resin.

Another observed factor is the difference between the hardness of the top with the bottom part that was not affected by the distance from the curing light tip except for 4mm, which allowed an increase on this percentage difference. This fact is probably explained by the decrease of energy quantity in the resin body, which is proportionally similar to the different distances.

However, data from this study can scientifically evidence an appropriate photopolymerization within the studied parameters. Nonetheless, the professional must acquire knowledge about the need of understanding different characteristics of the available commercial systems for photoactivation of resin composite, such as: wavelength, power density, presence or absence of filters, equipment life time, maintenance type, their advantages and disadvantages. This need is justified by the fact that such factors show a direct influence on the photopolymerization depth of the composite resin [27]. On the same way, the influence of the composition in the process of polymerization of resin[28] turns clear and so the basic knowledge of the latter is necessary.

Conclusion

According to the results, it is possible to conclude that:

1. The microhardness of the composite resin was influenced by the light source and by the distance of the photopolimerization, in both studied resins.
2. LED showed higher results for microhardness, in relation to halogen light, for Esthet-X, being the opposite result for Z250.
3. The polymerization depth showed difference as the polymerization distance increased, on its upper part, because on its the bottom part, the difference was only between 1 and 2mm.
4. Esthet-X showed the worst results.

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