

# INFLUENCE OF LIGHT SOURCE AND IMMERSION MEDIA IN SURFACE ROUGHNESS AND HARDNESS OF A NANOFILLED COMPOSITE RESIN

## ABSTRACT

The study evaluated the influence of light curing units and immersion media on superficial roughness and microhardness of the nanofilled composite resin Supreme XT (3M/ESPE). Light curing units used were: XL 3000 (3M/ESPE), Jet Lite 4000 Plus (JMorita) and Ultralume Led 5 (Ultradent) and immersion media were artificial saliva, Coke®, tea and coffee, totaling 12 experimental groups. Specimens (10mm x 2mm) were immersed in each respective solution for 5 seconds, three times a day, during 60 days and so, were submitted to measure of superficial roughness (Ra) and Vickers hardness. Data were subjected to two-way ANOVA test ( $p < 0.05$ ). Results showed that only the light source factor showed statistically difference for hardness. It was observed that the hardness of the composite resin Filtek Supreme XT (3M/ESPE) was influenced by the light source ( $p < 0.01$ ) independently of the immersion media ( $p = 0.35$ ) and the Jet Lite 4000 Plus (JMorita) was the light curing unit that presented lower values. In relation to surface roughness, it was noted no-significant statistical difference for light source ( $p = 0.84$ ), when specimens were immersed in different beverages ( $p = 0.35$ ).

**DOMINGOS**, Patrícia Aleixo dos Santos\*  
**GARCIA**, Patrícia Petromilli Nordi Sasso\*\*  
**OLIVEIRA**, Ana Luisa Botta Martins\*\*\*  
**CAMPOS**, Juliana Álvares Duarte Bonini\*\*  
**DIBB**, Regina Guenka Palma\*\*\*\*

## KEYWORDS

Composite resin. Dental technology. Beverages.

## INTRODUCTION

Despite the notable improvement in composite resins composition and characteristics, when these material are placed in the oral environment are subject to a great number of adverse conditions of pH, temperature changes, what may act as a challenge to their integrity and longevity.<sup>1</sup>

Besides this, there are several important factors that may influence the rate of polymer degradation: the type of chemical bond with the polymer chain, water sorption, the pH of the immersion media, the intensity of light unit and its correct application that allow a great degree of polymerization,<sup>2-3</sup> and oral habits.<sup>4</sup>

In relation to eating habits the consumption of beverages as coffee, tea, fruit juices, wine or soft drink may cause an impact on the properties of composites that are directly related to the amount and frequency of its intake.<sup>1</sup>

According to Wongkhantee et al.<sup>5</sup> (2006) some acidic food and drinks (Cola soft drink, drinking yogurt, orange juice, sports drink, Tom-yum soup) promoted a decrease on surface hardness of various studied substrates as enamel, dentine, universal composite, microfilled composite, conventional glass ionomer, resin-modified glass ionomer, polyacid-modified resin composite.

A study conducted by Bagheri et al.<sup>6</sup> (2005) reported that exposure to the

combined effects of food, stains and alcoholic beverages can result in surface damage as the increasing of roughness, surface hardness, flexural strength and susceptibility of staining.

Therefore, the purpose of the present study was to evaluate the effects of different light curing units on the surface roughness and hardness of a nanofilled composite resin immersed in various beverages.

## MATERIAL AND METHODS

### EXPERIMENTAL DESIGN

The surface roughness and hardness are the dependent variables and the independent variables are the four-level immersion media: Coke<sup>®</sup>, tea, coffee and artificial saliva; and three-level curing light units: two halogen units - XL 3000 (3M/ESPE, St. Paul, MN, USA - 480-530mW/cm<sup>2</sup>) and JetLite 4000 Plus (JMorita, Irvine, CA, USA - 1230mW/cm<sup>2</sup>) and one LED unit - Ultralume LED 5 (Ultradent Products, South Jordan, Utah, USA - 790mW/cm<sup>2</sup>). Twelve experimental groups were obtained from the association between variables. The number of specimens used for each experimental conditions was 10, totaling 120 test specimens.

### PREPARING THE SAMPLE SPECIMENS

The nano-composite resin Filtek Supreme XT (3M/ESPE, St. Paul, MN) (Table 1), color B1E was manipulated following the manufacturer's instructions. A stainless steel

matrix (10mm-ØX2mm-thickness) was used for specimen preparation. The resin composite was inserted into the matrix cavity in a single increment and covered with a polyester strip. In order to compact the material and prevent void and bubble formation, a microscopic slide and a 1-kg weight were placed over the resin composite/matrix ensemble for 30 seconds, thereby providing specimens with smooth, highly flat surfaces. After 30 seconds, the weight was removed and the composite resin was light cured for 40 seconds through the glass slide, being each specimen photopolymerized according to the light curing unit selected. The bottom surface of specimen was identified with a scalpel blade. There was no polishing over the specimen surface because it was considered that the major superficial smoothness was obtained with the polyester strip.<sup>7-9</sup>

The specimens of composite resin were randomly subdivided into four groups: the control group was maintained in artificial saliva and the three experimental groups were submitted to cycling in selected beverages (Table 2). The specimens were kept immersed in artificial saliva at  $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$  in the interval between cycles. The drinks were used in their usual temperature of consumption, ie, Coke  $\pm 10^{\circ}\text{C}$ , tea  $\pm 70^{\circ}\text{C}$  and coffee  $\pm 70^{\circ}\text{C}$ . The temperatures were measured with digital thermometer.

The specimens were immersed in each solution for five minutes under agitation (CT-155, Cientec - Piracicaba - SP - 13426-155 - Brazil), three times a day, with intervals of 4 hours. For the control group (specimens kept in artificial saliva), the specimens were stored at  $37^{\circ}\text{C}$ , and the artificial saliva was changed daily. These procedures were repeated for 60 days.<sup>3,9</sup>

#### EVALUATION OF SURFACE ROUGHNESS

A profilometer (Surftest SJ-401, Mitutoyo Co, Kanagawa, Japan) was used to measure arithmetic mean roughness (Ra) of the surfaces. Ra corresponds to the area created by the line of the profile above and below the central line divided by the scanned length. Each specimen was individually fixed in a clamping apparatus and the extremity of the equipment's arm. The needle was then positioned on the specimen surface and programmed to trace a 1mm course, with cutoff at 1mm/s. Three measurements were performed on each specimen. The average of these 3 values was used for statistical analysis. Roughness reading was performed after 60 days of the immersion procedures.

#### EVALUATION OF THE VICKERS HARDNESS

The Vickers hardness reading (VHN) was obtained using a pyramid-shape diamond of a digital microdurometer (Buehler, Lake

Bluff, Illinois, USA), applying a 50 gf load for 30 seconds over the surface of the specimen.<sup>10</sup>

Table 1 – Characteristics of the restorative material.

Brand	Manufacturer	Type	Resin Matrix	Filler Composition	Shade
Filtek Supreme XT	3M ESPE, St. Paul, MN, EUA	Nanofilled	Bis-GMA Bis-EMA UDMA TEGDMA	Nanoagglomerated nano silica filler (20 nm), Agglomerated Zirconia/silica nanocluster (0.6-1.4µm)	B1E

Table 2 - Tested Beverages.

Beverage (Brand)	Composition	pH	Manufacturer	Lot
Saliva Artificial	KH <sub>2</sub> PO <sub>4</sub> , K <sub>2</sub> HPO <sub>4</sub> , KCl, NaCl, MgCl <sub>2</sub> .6H <sub>2</sub> O, CaCl <sub>2</sub> .2H <sub>2</sub> O, NaF, sorbitol, nipagin, nipasol, carboxymethylcellulose (CMC), water.	7.0	Laboratory of Biochemistry of FCFRP-USP.	-
Coffee (Pilao®)	Caffeine, mineral as potassium (K), magnesium (Mg), calcium (Ca), sodium (Na), iron (Fe), manganese (Mn), rubidium (Rb), zinc (Zn), copper (Cu), strontium (Sr), chromium (Cr), vanadium (V), barium (Ba), nickel (Ni), cobalt (Co), lead (Pb), molybdenum (Mo), titanium (Ti) and cadmium (Cd), amino acids, lipids, sugars, vitamin B complex and chlorogenic acids	6.8	Pilão - Sara Lee Cafés do Brasil Ltda	302a
Yerba Mate Tea (Mate Leao®)	Leaves and stems of toasted mate ( <i>Ilex paraguariensis</i> ) also contained alkaloids (caffeine, methylxanthine, theophylline and theobromine), tannins (folic acid and caffeic), vitamins (A, B1, B2, C and E), minerals (aluminum, calcium, phosphorus, iron, magnesium, manganese and potassium), protein (essential amino acids), glucose (fructose, glucose, sucrose and raffinose), lipids (essential oils and substances CERAC), and cellulose, dextrin, saccharin and gums	6.5	Leão Junior SA	060/06
Soft Drink (Coca-Cola® - normal)	carbonated water, sugar, extract of cola nuts, caffeine, caramel color, acidulante INS 338, carbohydrates and sodium	2.62		P181207

This procedure was performed in three different places, creating three values, which resulted in a final average that was calculated for each specimen.<sup>10</sup> A device was created to

standardize the positioning of the specimens and the reading in the microdurometer. Specific coordinates were set to the north-south and east-west axis of the

microdurometer to obtain the readings at three points of the previously standardized specimens. Hardness reading was performed after 60 days of the immersion procedures.

#### STATISTICAL ANALYSIS OF DATA

The average of the surface roughness and hardness of the different groups after 60 days was calculated for the specimens.

Descriptive statistical was used. After the normality assumptions and homoscedasticity were tested and met, the

two-way analysis of variance (ANOVA) ("light source" factor and "means of immersion" factor) were carried out for the study of surface roughness and hardness of the specimens. The significance level was 5%.

#### RESULTS

The table 3 shows the averages and standard deviations of surface roughness (Ra) of the specimens according to the light source and immersion media after 60 days, and the results of Analysis of Variance.

Table 3. Surface roughness average - Ra ( $\mu\text{m}$ ) and standard deviation according to the light source and immersion media. (FOAr-UNESP, 2008).

Light source	Immersion Media*				
	Saliva	Coffee	Tea	Coke®	Total
XL 3000	1,41±0,70	0,68±0,54	1,67±1,12	1,39±0,56	1,29±0,82
Ultralume Led 5	1,49±1,77	1,15±1,06	1,12±0,70	1,04±0,78	1,20±1,13
Jet Lite 4000 Plus	1,58±0,96	1,45±0,68	1,15±0,60	1,08±0,81	1,32±0,77
Total	1,50±1,19	1,10±0,83	1,31±0,85	1,17±0,72	

\* ANOVA two-way: immersion media:  $p=0.35$ ; light source:  $p=0.84$ ; interaction:  $p=0.38$ .

It was observed that there was non-significant statistical difference in the surface roughness of the specimens according to the light source used in different immersion media.

The table 4 shows the averages and standard deviations of surface hardness of the specimens according to the light source and immersion media after 60 days, and the results of Analysis of Variance.

Table 4 - Mean and standard deviation for hardness (VHN) according to the light source and immersion media. (FOAr-UNESP, 2008).

Light source	Immersion media*				
	Saliva	Coffee	Tea	Coke®	Total
XL 3000	0,52±0,12	0,61±0,06	0,56±0,08	0,51±0,08	0,55±0,09 <sup>a</sup>
Ultralume Led 5	0,53±0,09	0,57±0,07	0,53±0,10	0,53±0,04	0,54±0,08 <sup>a</sup>
Jet Lite 4000 Plus	0,49±0,08	0,46±0,10	0,47±0,06	0,50±0,12	0,48±0,09 <sup>b</sup>
Total	0,51±0,10	0,55±0,10	0,52±0,08	0,51±0,08	

Similar letters indicate vertical statistical similarity (column).

\* ANOVA two-way: immersion media:  $p=0.35$ ; light source:  $p<0.01$ ; interaction:  $p=0.14$ .

The results showed that there was a significant statistical difference on composite resin's hardness in function of studied light sources ( $F=3,080$ ;  $p<0,01$ ), regardless of immersion media. The Jet Lite 4000 Plus (JMorita) was the curing unit that promoted lower hardness.

## DISCUSSION

This study investigated the influence of different light sources on the surface roughness and hardness of the nanofilled composite resin in different means of immersion and noted that the light source influenced the hardness of the composite studied but did not affect the surface roughness. On the other hand, the immersion media had no effect on these properties.

With regard to the light source, it was observed that the lowest hardness values were obtained in specimens polymerized with Jet Lite Plus 4000 (JMorita). Moreover, the units XL 3000 (3M/ESPE) and Ultralume Led 5 (Ultradent) presented similar hardness values and higher than the Jet Lite Plus 4000 (JMorita).

According to Kurachi et al.<sup>11</sup> (2001) one of the ways commonly used to measure the efficiency of the light source is the composite resin hardness because the polymerization of light-cured resins depends mainly on the characteristics and type of the radiation source used.

After polymerization, monomers that do not participate in reactions lead to a decrease in hardness of the inorganic fillers and that directly affects the final hardness of the material.<sup>12</sup> Some authors<sup>13</sup> suggest that depth of polymerization, and consequently hardness is not only affected by composite-related factors but also by light related factors such as light intensity, spectral distribution and exposure time.

For years, halogen lamps have been used in the polymerization of resins for presenting a well known technology. However, these devices have some disadvantages as change in light emission and degradation of the lamp, reflector, filter and fiber produced due to high temperatures, causing changes in the spectrum of light emission and decrease of output power with time of use, what could lead to a decrease in the polymerization effectiveness of the curing unit.<sup>14</sup>

Thus, the LED units came with a promise to address the shortcomings noted in previous technologies. However, the LEDs of the first and second generation of its low power density, did not get an acceptable clinical performance.<sup>15-16</sup> On the other hand, the LED light curing units of third generation, as Ultralume LED 5 (Ultradent), show a similar performance to conventional quartz-tungsten-halogen curing units (17). Cekic-Nagas et al.<sup>18</sup> (2010) also believe that high-power LED LCUs might be considered as effective than halogen

units for polymerization of the resin-based materials. In the present study, this was confirmed when Ultralume LED 5 (Ultradent) showed hardness values comparable with XL 3000, the gold standard for polymerization.

Moreover, when comparing Ultralume LED 5 (Ultradent) with a unit of high-power halogen light, it was found that the studied composite showed lower values of hardness when polymerized by Jet Lite. This may be explained by the great degree of polymerization obtained with LED units due to its spectral purity in comparison with the halogen units, as it has a narrow band of light emission with a wavelength between 450nm-490nm, with peak emission at 470nm, and this is the coincident blue light band with the absorption spectrum of most of the photoinitiators included in the composite resins, which allows full use of the light emitting diodes.<sup>19</sup>

In general, total energy – the product of light intensity and exposure time – determines the mechanical properties of the resin composite.<sup>17</sup> It is noteworthy that, as the time of light exposure during the curing were the same for all specimens (40s), regardless of light source used, the total energy received by them in different groups was proportional to the power density of the device: for the XL 3000 was 19.2 to 21.2 J/cm<sup>2</sup>; for Ultralume LED 5 (Ultradent) was 31.6 J/cm<sup>2</sup> and JET LITE Plus 4000 (JMorita) of 49.2 J/cm<sup>2</sup>. It can be

observed that the specimens that received higher activation energy were those submitted to Jet Lite Plus 4000 (JMorita). Whereas the microhardness is correlated with the degree of conversion of the material,<sup>11</sup> it was expected that these specimens showed higher hardness values. However, this was not the result observed. This happened because most of the energy generated by this high power halogen lamp, instead of promoting the complete conversion of monomers what would result in higher hardness of the material, the generated energy dissipated as heat and this was observed during development the present study through the heating of Jet Lite curing unit.

Unlike what was observed in this study with regard to the influence of light source on hardness, surface roughness did not change. Maybe this may be explained by the fact that the degree of polymerization is proportional to the amount of light that the resin is exposed, so that more light reaches the surface portions of the material, which is the one most close to the light source. Thus the resin surface is unchanged. Moreover, it might be considered that the specimens used in the present study were polymerized under a polyester matrix, which promotes greater surface smoothness of them, independently of filler concentration of composites.<sup>20</sup>

As specimens of all groups were not subjected to any finishing and polishing

procedure, regardless of the light source and the immersion media, the initial and final surface roughness possibly would present similar, since as the amount of light reaching the lower layers of the resin composite can be diminished when the distance is increased,<sup>17</sup> so the degree of conversion achieved at the top of specimen is similar for any light source<sup>21</sup> and this might explain the performance of surface roughness for the different light curing unit studied. Also in this sense, it be affirmed that immersion in saliva or beverages does not affect the roughness of the material because, although studies<sup>22-23</sup> showed that properties such as hardness and color are affected by the staining agents, surface texture is not.

With respect to the immersion media we observed in this study that they did not influence the hardness nor the surface roughness.

Similar results with respect to the hardness were observed by Yesilyurt et al.<sup>24</sup> (2009) that found that the hardness of some composites tested, including Filtek Supreme were significantly unchanged after exposure to citric acid and heptane solution. In the same way Aliping-Mckenzie et al.<sup>25</sup> (2004) found that specimens stored in soft drink with cola showed not significant changes in microhardness than those immersed in artificial saliva. However, Wongkhantee et al.<sup>5</sup> (2006) observed that cola soft drink reduced surface hardness of composite resin, enamel

and dentin. Yanikoğlu et al.<sup>23</sup> (2009) also found that tea, cola, and coffee solutions significantly affected surface hardness.

In relation to surface roughness Badra et al.<sup>1</sup> (2005) found a surface roughness change of composite resins for conservation in beverages such as coffee and Coke®. Otherwise, Yazici et al.<sup>22</sup> (2000) studying other solutions, such as citric, lactic and ethanol acid used to simulate the intake of drinks, vegetables and fruits and Oliveira et al.<sup>26</sup> (2010) that studied sodium fluoride solution at 0.05% - manipulated, Fluordent Reach, Oral B, Fluorgard found no influence on the surface roughness.

## CONCLUSION

In this context, based on the methodology and results obtained, it was concluded that independent of studied immersion media, the microhardness was influenced by light source, while roughness was not.

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