Effect of Preheating on Surface Microhardness of Six Composite Resins *

Efecto del precalentamiento en la microdureza superficial de seis resinas compuestas

Efeito do pré-aquecimento na microdureza superficial de seis resinas compostas

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ABSTRACT

Background: Preheating resins has been shown to improve physical properties such as surface microhardness. Some low-cost composite resins have not been well studied and preheating could improve their properties. Objective: to evaluate if preheating increases the surface microhardness of 6 composite resins and if the microhardness of the preheated low-cost ones equals that of the other resins. Method: In this In vitro experimental investigation, 6 resins were used, EnaHr®UE2 and UD2 (Micerium, Genoa Italy) as control, Z350™ and Z250™ (3M ESPE, Minnesota United States) conventionally used composites and LUNA ® and ROK ® (SDI, Victoria Australia) as low-cost resins. Specimens without preheating (sp) and preheating at 39°C (p) were created for each resin. It was polymerized according to the manufacturers’ instructions. Three measurements were made per sample using a microhardness tester applying a 300 g load for 10 seconds. Results: Resin-enamel microhardness: LUNA ® sp 69.93 HV (95 % CI 62.02-77.85), Z350™ sp 89.9 HV (95 % CI 80.56-99.23), LUNA ®
The use of amalgam is still common when dental tissue has been lost, loss associated with dental caries, violence and accidents. When performing surgical treatments, for many years amalgam was the material of choice for posterior teeth. Due to its unique composition, amalgam presents easy handling,
adequate mechanical behavior, low cost, and long life (1, 2). However, its use is being banned worldwide due to contamination associated with mercury. In Colombia, its use will be restricted from 2023 by the Minamata treaty (3), which seeks to solve the problem of using this pollutant.

Composite resins have evolved over the years in order to improve both their aesthetic and functional properties. Some low-cost and easily accessible composite resins in Colombia have deficiencies in terms of their clinical performance: low wear resistance, incomplete polymerization, delamination, or material fracture (4). These complications compromise the integrity of the restoration and therefore the longevity of the treatment. Still, these materials should become the imminent replacement for amalgam as a direct restoration.

Among the main causes of failure of resin restorations, fracture is the most common complication, followed by secondary caries and marginal maladjustment (5). This type of failure or complications is caused not only by the patient's habits, but also by the superficial microhardness and resistance of the materials to masticatory forces. For this reason, among the most important properties to evaluate in a material exposed to changes in temperature, humidity and different types of chewing force, is the superficial microhardness.

Surface hardness and microhardness are defined as the surface resistance of a material to being penetrated and is evaluated from notch depth measurements made by a specific indenter under a static load (6). The Vickers (HV) test is the most common for polymeric materials and uses a square-based pyramidal-shaped indenter with an angle of 136 °C between its surfaces (7, 8). To perform the HV, the measurement of the diagonals formed by the indenter after removing the load is considered.

In dentistry, surface hardness and microhardness are measures of a restoration's ability to wear or be worn by opposing structures. They are related to the mechanical resistance and rigidity of the material (9). Consequently, the factors that affect the hardness and superficial microhardness of a restoration can influence its longevity by avoiding complications such as fracture of the material (10). As a method to improve this property of composite resins, the preheating of the resins before polymerization is indicated. Resin preheating is a thermal process described in the literature since the 1980s with self-curing resins (11). It is not a new process, but it has been more widely accepted in the last decade since it increases its fluidity, reduces viscosity, improves the adaptation of the material, and increases surface hardness and resistance to compressive forces (12). When they are preheated, the molecular mobility in the resins is increased, which improves the distribution of the filler molecules and facilitates polymerization, which will prevent the material from fracturing, presenting mismatches, losing polish, changing color or dislodging from restored teeth (13, 14). On the other hand, preheating the resin compounds increases the depth of cure and the hardness of the compounds (15). The effect of preheating on the superficial and deep microhardness of composite resins has shown a significant increase when compared to non-preheated resins (16).

The most used temperatures described in the literature to preheat resins are 54 °C and 60 °C, but when removing the material from the heater, the temperature drops by 50% after two minutes (17, 18). Using these temperatures generates a higher viscosity and a difficult handling of the resins to be used as direct restorative materials. Temperatures above 50 °C allow the use of preheated resins as luting material. These temperatures have shown, during in vivo placement of the composite, that the preheated resin increases the temperature from 6 °C to 8 °C with respect to room temperature, that is, it does not reach critical levels and does not affect the dentin (19). The greatest cause of intrapulpar temperature increase is attributed to curing lamps rather than preheated resin (18). It has been verified that multiple preheating exposures to a temperature not higher than 59 °C do not affect the resin that remains in the container (20).

Preheating the resin to 39 °C allows it to be manipulated and it is the temperature indicated and described by the commercial house Micerium® as the working temperature to conduct direct
restorations. The research question proposed by this study is: What is the effect of preheating on the surface hardness of conventional low-cost resins ROK®, LUNA® and resins of high commercial use Z350TM, Z250TM compared with each other and with the commercially indicated resin? to preheat EnaHri®? The purpose of this study was to determine if preheating composite resins at 39 °C increases the surface microhardness of low-cost resins in order to determine if preheating is a procedure that improves mechanical characteristics of lower-cost resins, matching them to those of greatest commercial use so that more people can access treatment, without complications such as premature wear and fracture, taking into account that composite resins are the material that should replace dental amalgam.

MATERIALS AND METHODS

An in vitro experimental study was carried out using 6 different composite resins (Table 1): two of the resins used are considered low cost and easy to access by Colombian dentists: LUNA® and ROK® (SDI, Victoria Australia). ; two resins used on a daily basis by Colombian dentists Filtek Z350 TM and Filtek Z250 XT TM (3M ESPE, Minnesota, United States), and as a control group . two EnaHri® resins (Micerium, Genoa Italy), UE2 ® (enamel) and UD2 ® (dentin) as they are indicated for use with and without preheating. The color equivalent to A2 was selected for all resins.

<table>
<thead>
<tr>
<th>Composite resins</th>
<th>Manufacturer</th>
<th>Particle size</th>
<th>Indications for use</th>
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<tbody>
<tr>
<td>EnaHri</td>
<td>Micerium</td>
<td>Microparticles</td>
<td>Anterior and posterior sector. Enamel</td>
</tr>
<tr>
<td>UE2 ® (21)</td>
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<tr>
<td>EnaHri</td>
<td>Micerium</td>
<td>Microparticles</td>
<td>Anterior and posterior sector. Dentine</td>
</tr>
<tr>
<td>UD2 ® (21)</td>
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<tr>
<td>LUNA ® (22)</td>
<td>SDI</td>
<td>Micro and nanoparticles</td>
<td>Anterior and posterior sector</td>
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<tr>
<td>ROK ® (23)</td>
<td>SDI</td>
<td>Hybrid, micro and macroparticles</td>
<td>Posterior sector</td>
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<tr>
<td>Filtek™ Z250 XT (24)</td>
<td>3M ESPE</td>
<td>Microparticles</td>
<td>Anterior and posterior sector</td>
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<tr>
<td>Filtek™ Z350 (25)</td>
<td>3M ESPE</td>
<td>Micro and nanoparticles</td>
<td>Anterior and posterior sector</td>
</tr>
</tbody>
</table>

* The scale on which the filler particles are found and the indications for use according to the manufacturer are described. Source: the authors

A silicone matrix of 5 mm in diameter by 2 mm in height was made (figure 1) considering the ISO 4049 standard (26). 10 specimens of each resin were divided into 2 groups: 5 preheating the resin at 39 °C (p) and 5 specimens without preheating (sp), at room temperature of 25 ± 1 °C. The samples were coded with letters and numbers.

Obtaining Specimens Without Preheating

The matrix was placed on a Mylar® strip, using a glass tile as a base, to obtain a flat surface. The resin was packed into the matrix, filling it with a single increment of resin. A strip of Mylar® was placed on the surface of the matrix with resin and light pressure was exerted with a glass tile to leave the surfaces flat, parallel and homogeneous, avoiding the formation of bubbles. The tile was removed and light-cured on the Mylar® strip for 20 seconds at a distance of 1 mm, according to the manufacturers' recommendations, using a Bluephase® light-curing lamp (Ivoclar Vivadent S: 050043, Schaan,
Liechtenstein) with an intensity of 800 mW/cm² and a wavelength range between 430-490 nm. This polymerization process was conducted on both bases of the resin cylinder (9).

After the initial polymerization by means of the light-curing lamp, the specimens were placed in a Binder® incubator (BD 53, 02-30792 Tuttlingen, Germany) for 24 hours, at a temperature of 37 °C, to finish the curing process. polymerization (9). Once the time in the incubator was over, the surfaces were polished and finished with polishing discs (Praxis-TDV), going through the 4 sizes of coarse, medium, fine and extra-fine grains. During this process, the measurements of each of the samples were rectified, it was observed if the surface presented irregularities or bubbles with a stereomicroscope (MOD stemi 305, 178762 New York, United States). Samples with irregularities on the surface, fissures and bubbles were excluded and performed again, working only with samples that presented the appropriate measurements, with smooth surfaces and parallel cylinder bases (figures 1A, 1B).

**Obtaining Prewarmed Specimens**

To control the temperature, the silicone matrix was placed on a glass tile that was placed on a specialized Magnetic Stirrer® (SH-2) laboratory heating plate, maintaining a temperature of 37°C to simulate body temperature. The tile temperature was kept constant and was verified with a thermocouple connected to a digital multimeter (UT33C+, Guangdong, China) throughout the process (figure 1C).

![Figure 1](image1.png)

**FIGURE 1**

A. Silicone matrix of 2 mm in diameter and 5 mm in height for the creation of the specimens. B. Sample observed under a microscope with irregularity on the surface, not suitable for the microhardness test. C. Glass tiles on a heating plate with constant temperature at 37 °C

Source: the authors

The ENA HEAT drum from Micerium® was used at a temperature of 39 °C to preheat the resins and the temperature was constantly checked with a thermocouple connected to a digital multimeter (UT33C+, Guangdong, China). The matrix was placed on a strip of Mylar® that was on the surface of the glass tile at 37 °C. The process of packing the resin inside the matrix began, taking enough material to make a single increase of the material with an FP3 spatula. The temperature of the resin was measured when it was incorporated into the matrix before being polymerized. A strip of Mylar® was placed on top of the matrix and controlled pressure was exerted with another tile at 37°C in order to give the desired thickness generated by the matrix. Both surfaces of the cylinder were cured with the Bluephase light, as described in the specimens without preheating. Before starting the packing process in the die, the temperature of the preheating drum, the syringe with the resin and the FP3 spatula were verified. The polymerization process was completed in an incubator for 24 hours at 37 °C. The same polishing protocol described was conducted with the specimens without preheating, verifying the measurements and surfaces of each of the resin cylinders.
Microhardness Tests

The Vickers microhardness (HV) tests were conducted with the equipment of the Faculty of Engineering of the Pontificia Universidad Javeriana, (digital display microhardness tester model HVS 1000A) with a load of 300 gF for 10 seconds. Considering the specifications in ISO 6507-1 and ASTM E384-22, the samples are randomly selected and 3 indentations per sample were made (figure 2). The data from the measurements of the diagonals of each indentation were analyzed. The 3 data obtained for each sample were averaged. The GraphPad Prism program was used to perform the univariate and bivariate descriptive statistical analysis. Significant differences less than 0.05 were accepted.

![Image of the indented surface where the diagonals generated by the pyramidal tip of the microdurometer can be observed](image)

Source: the authors

RESULTS

The results of the tests conducted in the microdurometer are shown in table 2. Considering the HV averages of all the composite resins without being preheated from lower to higher resistance to penetration UE2®, UD2®, LUNA®, ROK®, Z350TM, Z250TM. The HV averages of the preheated composite resins from lowest to highest microhardness, UD2, LUNA®, UE2®, Z350 TM, ROK®, Z250 TM Among all preheated and non-preheated Z250 TM resins presented the highest surface microhardness. In all preheated resins, an increase in HV was observed except in the Z350 TM which, on the contrary, shows a decrease in HV values, although this decrease is not statistically significant (p < 0.05). A decrease in the standard deviation was observed when preheating the LUNA®, ROK®, Z250 TM UE2® and UD2® resins when compared to the values obtained without preheating.

The resins that presented a lower surface hardness were the resins from the Micerium® commercial house. These 2 resins were the only ones that showed a statistically significant increase when preheated with values for UE2 ® (sp-p) p = 0.0110 and UD2 ® (sp-p) p = 0.0325.
When comparing the results of the resins divided into two groups according to their indication for use as dentin or enamel, in the posterior or anterior sector, it was found that: the resins without preheating that can be used as dentin have the following behavior: ROK® sp is equal to Z250 TM sp (p = 0.2825). ROK® sp and Z250 TM sp are greater than UD2® sp p = 0.0020 and p = 0.0032, respectively. These same preheated resins showed that ROK® p is less than Z250 TMp (p = 0.0314). ROK® p and Z250 TM p are greater than UD2®p, p < 0.000 and p = 0.0010, respectively. It is worth noting that ROK® p does not outperform Z250 TM sp (p = 0.6760). (Figure 3).

The behavior of the enamel resins was as follows: LUNA® sp lower HV than Z350 TM sp (p = 0.0019), but higher than UE2® sp (p = 0.0342). LUNA ® p lower HV than Z350 TM p (p = 0.0026) and is equal to UE2 ® p (p = 0.4649). LUNA ® p is less than Z250 TMp (p 0.0015). (Figure 4.).

Among the low-cost resins used in this study, non-preheated and preheated ROK® resin had significantly higher surface hardness than LUNA® resin with p = 0.0393 and p = 0.0018, respectively. The Z250 TM is significantly harder than the Z350 TMp = 0.0027.

**DISCUSSION**

In this experimental study it was determined that, when preheating the resins at 39 °C, a change in the viscosity of the material was identified, which allows for better handling. Additionally, it was established that when removing the resin from the preheated syringe to be packed in the matrix, there is an average temperature decrease of 5 + 1 °C of the preheated resin. This allows for greater safety when working with the resin, since an increase in intrapulpar temperature will not be generated in the case of vital teeth. The increase in temperature on the pulp tissue, as described in the literature, is more likely
with the use of an inappropriate light-curing lamp, rather than by preheating the resin to this temperature (27).

In this study, preheating to 39 °C, indicated for handling the resin and using it as a direct filling, did not generate significant increases in the surface microhardness of LUNA®, ROK®, Z350®™ and Z250™ resins, but did significantly increase the surface microhardness of ENAHRI® resins (figure 3). This can be attributed to its chemical composition, in which the presence of 1,4 butanediol dimethacrylate stands out, a molecule that provides characteristics to polymers such as greater chemical resistance, heat resistance, abrasion resistance, and water resistance (28). At a temperature of 39 °C it was determined that preheating generates a decrease in the standard deviation value of all samples except the Z350™ (Table 2). This indicates that the preheating of the resins can have an impact on the molecular organization, favoring a more constant behavior before penetration forces, which improves the capacity of resistance to indentation. This may be due to a greater molecular movement that allows a better formation of the polymer chains and a greater crosslinking, which directly optimizes the mechanical properties of the material (29-31).

Although significant changes in surface microhardness are described in the literature when resins are preheated (28,30), such changes are reported at temperatures close to 60 °C. At these temperatures, the resins completely change their viscosity, which makes it difficult to manipulate them in the process of shaping and contouring direct fillings. Viscosity is a property of matter that describes the resistance of a fluid to gradual deformation. In this case, the resins are polymeric macromolecules whose characteristics are both fluid and solid, and do not present a defined crystalline or amorphous structure (32, 33). For this reason, the viscosity at temperatures greater than 54 °C is similar to the preparation of phosphate for cementing in which filaments or threads are formed. For this reason, at these temperatures, the resins are indicated as cementing agents and not as direct sealing materials. Although in this study no statistically significant changes were observed in the low-cost composite resins, it was observed in all the composite resins that preheating changes the viscosity of the material, increasing its fluidity. This can provide a better adaptation to the walls that are part of the cavities, reducing microleakage and increasing the durability of the restorations (30, 34, 35).

Considering the information provided by each commercial house, 3M ESPE, SDI and Micerium, an analysis can be made based on the composition and fillers of each resin and try to determine the differences in HV (Figure 3). The Z250™ resin presented the highest HV values. Z250™ resin is a micro hybrid resin with a particle size between 0.01 µm and 3.5 µm with an average size of 0.6 µm. The load of inorganic filler material is 60% by volume, mostly composed of zirconium/silica (24). This resin is considered a universal resin that works for both the anterior and posterior sectors. In this study, Z250™ resin was taken as indicated for use in the posterior sector and was compared with ROK® (posterior sector resin) and EnaHri UD2® (universal dentin resin). ROK® resin has a filler material load of 67.7 % by volume with a high percentage of glass (23). When comparing ROK® resin to Z250® without preheating, although ROK® has a higher filler percentage by volume, the filler type makes this resin less hard and possibly the higher glass filler type is responsible for the lower HV values for ROK®. EnaHri UD2® presents 63% filler by volume, composed mainly of zirconium nano oxides and silicon dioxide with an average particle size of 0.04 µm (21). This means that UD2® resin presents the lowest HV value due to its high filler content with smaller particle size when compared to Z250™ and ROK®.
LUNA® resin, on the other hand, is nanohybrid. This means that it features nanometer and micrometer filler particles between 40 nm and 1.5 µm, with the majority being nanometers. Fillers make up 58% of the volume. Its filler is not mostly larger particles, which affects the microhardness of the material, but allows a better surface finish to be obtained (22). In this study, LUNA® was used as a more esthetic material for the anterior sector and was compared to Z350™ (enamel) and EnaHri UE2® resin (Figure 4). The Z350™ resin has a 20 nm nano silica filler and agglomerates of 5-20 nm zirconium/silica particles that form a nanocluster. The particle size of the aggregate oscillates within a range of 0.6 to 1.4 µm and its filler load is 63.3% by volume (21). The particle size of these two resins is similar, but the filler charge per volume is much higher in Z350™ which may be a significant factor in the higher HV strength of Z350™. EnaHri UE2® has in its composition zirconium nano oxides (12% by weight) and glass filler (silicon dioxide) to increase the refractive index (68% by weight) with an average of 0.04 µm of this particle. This makes UE2® resin highly aesthetic due to its high filler content with small particles that provide translucency. The filler load by volume is 63% with an average filler particle of 4.3 µm (21). Although the HV values without preheating are lower for UE2®, a significant increase is observed when preheating, exceeding the values for LUNA®. From the composition of Z350™ and UE2®, it can be determined that the zirconia/silica nanoclusters improve the resistance to indentation and that the high content of filler particles, which increase the translucency in UE2®, have a negative effect in terms of surface microhardness.
When comparing the resins according to their use as enamels or as dentins in the study, higher HV values were observed in the dentins than in the enamels. This may be due to the composition of these materials in which the particle size and filler content is higher in dentin than in enamel. The larger the particle size and filler content, the higher the surface microhardness (36). The filling finished with these resins with a high content of fillers and larger particles is deficient in terms of polishing and shine, so they are not indicated for use in highly aesthetic sectors (37).

CONCLUSIONS

Preheating to 39 °C does not increase the superficial microhardness of low-cost and commercial resins, neither enamel nor dentin, but it does increase in Micerium® resins that were used as a control group.

Preheating to 39 °C seems to have an impact on the molecular organization of the specimens, favoring a more constant behavior in commercial resins.

ROK® (low cost) resin microhardness is equivalent to Z350™ without preheating; preheated LUNA® (low cost) resin is different from Z350™ without preheating.

RECOMMENDATIONS

It is recommended to conduct more studies with temperatures other than 39 °C in a range that allows obtaining an adequate viscosity for the handling of the resins. This will make it possible to determine if
temperatures other than 39 °C generate changes in the superficial microhardness of composite resins for direct fillings.

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* Original research.

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