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ORIGINAL ARTICLE

Vickers microhardness comparison of 4 composite resins with different types of filler.

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Abstract: Composite resins are the material of choice to restore minimal invasive cavities; conversely, it is important to explore the mechanical properties of commercially available dental materials. Objective: To compare the Vickers microhardness (VHN) of four available commercial composite resins using standardized samples and methods. Methodology: Composite cylinders were manufactured in a Teflon mould. We used the follow composite resins (n=4/gp): Microhybrid resins [Feeling Lux (Viarden) and Amelogen Plus (Ultradent)], Hybrid resin [Te-Econom Plus (Ivoclar)] and Nanohybrid resin [Filtek Z350 (3M ESPE)]. All samples were incubated in distilled water at 37ºC for five days. The test was carried out with microhardness indenter at 10 N, and a dwelling time of 10 s for 9 indentations across the specimens resulting in a total of 36 indentations for each group. Data were subjected to Kolmogorov-Smirnov normality test and ANOVA (post-hoc) Tukey test. Results: The VHN mean values ranged from harder to softer as follows: Filtek Z350 (71.96±6.44) ($p<0.01$)> Amelogen Plus (59.90±4.40) ($p<0.05$)> Feeling lux (53.52±5.72)> Te-Econom Plus (53.26±5.19). Conclusion: According to our results, the microhardness of the evaluated conventional composite resins can withstand the masticatory forces; however nanohybrid composite resins showed better Vickers microhardness and therefore are a more clinically suitable option for minimal invasion treatments.

Keywords: *Vickers microhardness, composite resin, in vitro.*
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INTRODUCTION.

The recent technological trends have changed the view of dentistry in the 21th century since the Black postulates. Nowadays, minimal invasive dentistry is a new paradigm that is supported by evidence-based dentistry. Approaches of oral health care require the judicious integration of clinically relevant evidence, the patient's dental and medical condition and records, with the dentist's clinical expertise and the patient's treatment needs and preferences using minimal interventions[1,2].

Composite resins are one of the most suitable dental materials to make minimal invasive treatments due to their aesthetics, easy handling, biocompatibility and adhesive properties; however, easy discoloration during their long term in the oral cavity and poor marginal sealing are the main disadvantages of their use and directly related to their composition and mechanical properties such as microhardness[3,4]. On the other hand, the oral environment is under constant pH and temperature cycles that can alter the organic and inorganic matrix of composite resins particles resulting in filtration and reducing their durability in the mouth. While physical and mecha-

nical properties of these materials may be significantly altered by the effects of solvent uptake and component elution, the greatest concerns are the short-term release of unreacted components and the long-term elution of degradation products in the oral cavity, both of which should be strongly considered during restorative material development[5,6].

Composite resins are formed by four main elements: organic monomers or polymers, filler particles, bonding agents, and activator agents. Composite resins can be classified according to the size of their filler particles as follows: macrofill, microfill, hybrid and nanofill. Monomers correspond to Bisphenol A-Glycidyl Methacrylate (Bis-GMA) and Triethylene glicol Dimethacrylate (TEGMA)[7-9]. Filler particles enhance the hardness, manipulation and reduce the dimensional changes. The most common fillers are barium oxide silicates, strontium, zinc, aluminum and zirconium[8,10].

The choice of an appropriate resin composite for a restoration requires the evaluation of its functional properties, including the enhanced longevity of the restorations because of their excellent mechanical properties such as high strength, fracture toughness, surface hardness, optimized modulus of elasticity, low wear, low water sorption and solubility, low polymerization shrinkage, low fatigue and degradation, high radiopacity, better detection during removal of a composite restoration as well as adequate systemic and local biocompatibility without postoperative pain or hypersensitivity, not causing fractures or cracks and with caries preventing properties (bioactive), good color matching, stability, optimum polishability, long-term surface gloss, absence of marginal or surface staining and a good long-term anatomical form[11,12]. However, the predominant clinical failure of dental composites is secondary caries (39.2-54.1%), oclusal wear (21.6%), and material fracture (18.9-35.7%) in large cavities[13-15].

The hardness of composite resins is directly related to the conversion rate of polymerization depending on polymerization time, distance of polymerization light[16], irradiation power[17], and the type of material at the tip of the energy source[18]. However, a very powerful energy irradiation source can alter the polymerization contraction resulting in a poor marginal sealing and microfiltration[3]. Stratification layers higher than 2mm can partially polymerized, affecting the hardness of the material and increasing the risk for fracture[16,18]. In the present work, we compare the Vickers microhardness (VHN) of four available commercial composite resins using standardized samples and methods according to the guidelines for reporting pre-clinical *in vitro* studies on dental materials[19].

MATERIALS AND METHODS.

Samples

Composite cylinders (23x2mm, n=4/gp) were manufactured in a Teflon mold at the Laboratorio de Investigación Interdisciplinaria, Biomateriales Dentales, ENES, Unidad León, UNAM. Table 1 shows the composite resin groups used in the study.

Briefly, the resins were set in a mold and then covered with glass, to provide a flat and smooth surface. Polymerization time was 20 seconds with LED lamp (Light-Emitting Diode, Gnatus, Optilight, Brazil, wavelength of 420-480nm, energy source of 1200mW/cm^2) at about 1mm distance from tip to cover glass.

The curing process corresponded to five different places starting at the center of each sample and in the four cardinal points to achieve uniform polymerization. Total polymerization time was 100 seconds for each sample. Four samples were performed for each group resulting in a total of 16 samples (Figure 1).

Vickers Microhardness

The test was carried out at the Dental and Advanced Studies Research Center "Dr. Keisaburo Miyata", School of Dentistry, UAEMex. All samples were incubated in distilled water at 37ºC for five days. The samples were divided by group and randomly tested with microhardness indenter (DongGuanSinowon precision instruments, Nancheng, China). The Vickers microhardness was performed as reported by Scougall-Vilchis *et al.*[20]: a diamond indenter was applied to the composite surface at 10 N, and a dwelling time of 10s was used for 9 indentations across the

Table 1. Composite resins used in the study.

Resin	Filler	Composition	Manufacture
Feeling Lux	Microhybrid	Not available.	Viarden, D.F, Mexico
Amelogen Plus	Microhybrid	Matrix: Bis-GMA. Fillers: silicone dioxide, silicone, silicate 76%w, 61% v). Mean size: 0.7µm.	Ultradent Inc, South Jordan, UT, USA
Filtek Z350	Nanohybrid	Matrix: Bis-GMA, UDMA with small amounts of TEGMA. Fillers: nanosilica (20nm), zirconia/silica nano clusters (5-20nm). Mean size: 0-6 to 1.4µm (78.5%).	3M ESPE, St. Paul, USA
Te-Econom Plus	Hybrid	Matrix: Dimethacrylate and TEGMA (22wt%). Fillers: barium glass, ytterbium trifluoride, silicon dioxide and mixed oxide (76wt% or 60%vol). Mean size: 0.04 and 7µm, mean 850nm.	Ivoclar, Vivadent, Schaan, Liechtenstein

Figure 1. Schematic representation. The distance between indentations of Vickers microhardness was about 4mm. We calculated 9 indentation for each sample.

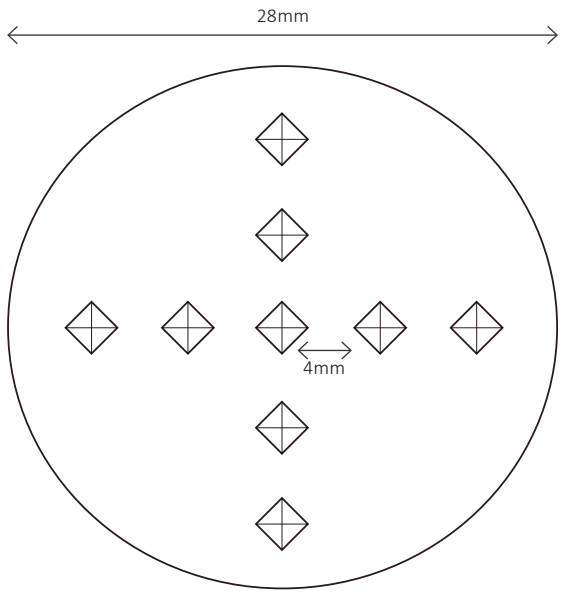
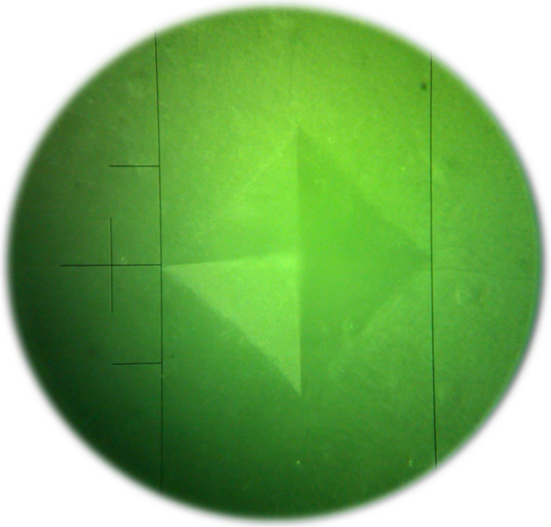


Figure 2. Representative image of Vickers microhardness. The image was obtained after indentation of the diamond pyramid of VHN test in a composite resin.



specimens resulting in a total of 36 indentations for each group. The microhardness indenter stared in the center of the sample and three indentations linear to the four cardinal points with a distance of 4mm between each other (Figure 2).

For the calculation of Vickers microhardness (VHN), the lengths of the two diagonals of each indentation were measured and VHN was calculated using the following formula:

$$VHN = 1.854F/d2$$

Where F is the load applied in Newtons and d is the mean length of the two diagonals of each indentation.

Statistical analysis

The mean value and standard deviation were calculated. VHN data were subject to Kolmogorov-Smirnov (Lillierfors) normality test and analyzed using ANOVA with post-hoc Tukey test. All data were analyzed with SPSS (Version 18; SPSS, Inc., Chicago, IL, USA). Significant differences were considered at $p<0.05$. All the statistical analysis was performed by a masked researcher.

RESULTS.

In this experimental and comparative *in vitro* study, all data showed normal distribution detected by Kolmogorov-Smirnov test. The mean values of Vickers microhardness are summarized in Table 2. The Filtek Z350 (71.96±6.44 VHN) exhibit the highest VHN (Harder) followed by Amelogen Plus (59.90±4.40 VHN), Feeling Lux (53.52±5.72 VHN), and Te-Econom Plus (53.26±5.19 VHN).

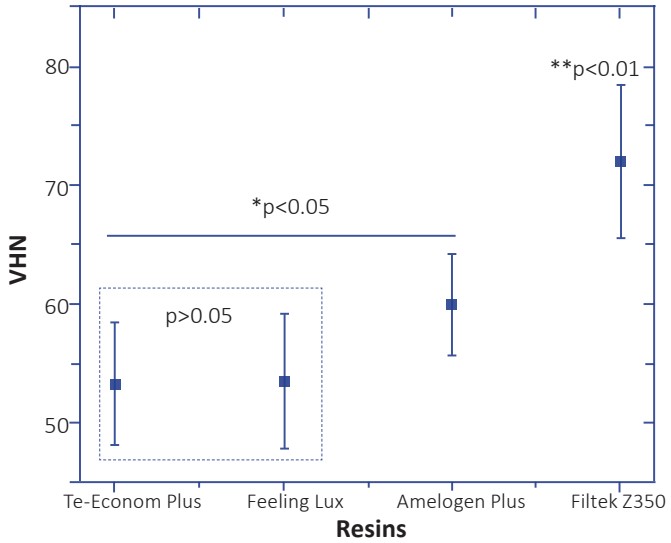
Filtek Z350 resulted in higher ($p<0.01$) microhardness than others composites. Amelogen Plus resulted in higher ($p<0.05$) microhardness than Feeling Lux and Te-Econom Plus. The comparison between Feeling Lux and Te-Econom Plus did not show significant differences ($p>0.05$), as seen in Figure 3. It must be mentioned that, in all cases, the size of the indentions was larger than the filler particles when compare with the size of the filler reported by the manufacturer.

Table 2. Vickers microhardness (VHN) of composite resins (n=36).

Resin	VHN (mean±S.D)	p-value	95% Confidence interval
Feeling Lux	53.52±5.72	A*	51.66;55.37
Amelogen Plus	59.90±4.40	B*	55.48;58.33
Filtek Z350	71.96±6.44	C**	69.87;74.05
Te-Econom Plus	53.26±5.19	A*	51.57;54.97

Mean values for each resin group with the same capital letter are not significantly different, while mean values with different letters are significantly different.

Figure 3. Vickers microhardness (VHN) values of different composite resins.
All samples (n=4/gp) were incubated in distilled water at 37ºC for five days. The samples were divided by group and the test was carried out randomly with Vickers microhardness indenter at 10 N, and a dwelling time of 10s. We use 9 indentations per sample resulting in n=36 by group. **p<0.05, **p<0.01 from ANOVA (post-hoc) Tukey test.



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DISCUSSION.

The Filtek Z350 composite resins showed the highest mean values of VHN (71.96). Probably their increased microhardness is related to the nanoparticle size of inorganic filling, because Filtek Z350 is considered as nanofill and nanohybrid resin that can display better mechanical properties than the others resins tested. Previous reports have revealed that the Vickers microhardness values of Filtek Z350 are from 74.9 to 97.68 VHN[21,22]. One of the factors that influence the decrease of composite hardness is the depth of cure of resins, it can be affected by several factors associated with the source of light polymerization, including the spectral emission (wavelength distribution), light intensity, exposure period, and irradiation distance[16,17,23].Among these factors, the irradiant rate of light given out by different light-curing units and the light-curing times. Depth of cure for light-activated dental composites has often been evaluated by the measurement of the hardness of the material at specific depths[23].

In this study, we found that specimens showed significantly different microhardness values according to the different composition and filler particles of composite resins. P90 Bulk fill resin (3M ESPE) and Z250 (3M ESPE) showing values from 54.1 to 67.8 VHN and 73.9 to 86.1 VHN[21], respectively. These results are similar and comparable to the data obtained here. In the same study, a linear correlation was observed between microhardness and specimen depth of polymerization (R^2=0.975-0.995) regardless of the composite. This research concludes that the energy of lamps is essential for successful curing of all the composite resins. It is similar to the correlation between the degree of conversion and depth[21]. In general, a higher degree of conversion correlates with greater hardness[24-26].

Some researchers suggest that the light-curing time recommended by the manufacturers is 20s, and that doubling or tripling the light-curing time did not increase the hardness values regardless of the type of composite resin[17,22,27,28] Similarly, one study suggested that bulk layers of resin less than 4mm are recommended to cure dental composite resins with a 1.226 mW/cm² power source[29].

The Amelogen Plus Vickers microhardness here tested (59.90 VHN) is comparable to other commercial resins such as Xenius (GC, Europe, Leuven, Belgium), Tetric Evo Celam Bulk Fill (Ivoclar-Vivadent, Schaan, Liechtenstein), X-tra Base (Voco, Cuexhaven, Germany), with microhardness values of 52.3, 47.7 and 47.0 VHN[30,31], respectively. These results suggest an acceptable VHN when compared with the microhardness of human dentin teeth (30-55.5 VHN)[32].

It is difficult to distinguish the effect of filler size and shape on the mechanical properties of commercial composites, and the filler load is the main factor for determining elastic modulus properties, while filler size and shape should be considered as secondary "fine-tuning" factors for altering material properties. Nevertheless, the different indentation force such as lower VHN values can be attributed to variations in test parameters as per reported by other authors[33]. We could not find any statistical difference between Te-Econom Plus and the Feeling Lux. Interestingly, Feeling Lux, a microhybrid composite resin, showed acceptable VHN values compared to dentin, however, neither of them included manufacturer or scientific data.

According to our results, nanohybrid composite resins possess better Vickers microhardness than hybrid and microhybrid resins. The incorporation of nano size filler particles enhance their mechanical properties. However, it is necessary to study not only microhardness but also other properties under ideal laboratory conditions such as higher irradiance and adequate curing time. We obtained satisfactory *in vitro* results; nevertheless, in practice this is totally different. It is important to mention that the hardness values obtained depend on nanofiller particles. Nanofiller composites should show more resistance to fracture and wear, therefore showing a better clinical performance for restorations. Filler particles integrated in the resin matrix and filler size of commercial dental composites has continuously decreased in the past years from microcomposite to nanocomposite materials.

Fillers not only regulate the mechanical properties of composite materials but also lead the reduction in monomer content and consequently the polymerization shrinkage, optimizing wear, translucency, opalescence,

radiopacity, intrinsic surface roughness and thus polishability, as well as enhancing aesthetics and improving handling properties and long term endurance in the mouth[34]. The smaller the particles, the better the polish and gloss, but the reduction of filler size and subsequent increase in surface area to volume ratio has limited the achievable filler loading, resulting in decreased handling and reduced mechanical properties[11,28]. Significant negative correlation (R^2=0.67) between the percentage of hardness increase after 24 hrs of dry storage and the initial microhardness values has been established[30,31]. This finding can be explained by the effect of mobility of reactive molecules in initial polymer network. On the other hand, the filler particle fraction is correlated to Vickers microhardness (R^2>0.8)[30].

One of the limitations of the present study was to test the hardness at different time intervals after immersion in water in order to evaluate the hardness in the time-line after the specimens were incubated in a wet environment such as lactic acid, in order to determinate the changes suffered in different ambient solutions, as well as evaluating the grade of polymerization from the top and bottom of the samples and determine how the cure irradiation altered the curing of conventional composite resins.

CONCLUSION.

According to our results, the Vickers microhardness of conventional composite resins could withstand the masticatory forces in the clinical context.

However, nanohybrid composite resins showed better microhardness properties becoming a more clinically suitable option for minimal invasive treatments.

Comparación de la Microdureza Vickers de 4 resinas compuestas con diferente tipo de relleno

Resumen: Las resinas son los materiales de elección para restaurar cavidades mínimamente invasivas, sin embargo, es importante conocer sus propiedades mecánicas de los materiales dentales disponibles comerciales. Objetivo: Comparar la microdureza Vickers (VHN) de cuatro resinas compuestas comerciales disponibles con muestras y métodos estandarizados. Metodología: Cilindros de resina compuesta fueron conformados en un molde de Teflón. Se utilizaron las siguientes resinas compuestas (n=4/gp): resina microhíbrida [Feeling Lux (Viarden) y Amelogen Plus (Ultradent)], resina híbrida [Te-Econom Plus (Ivoclar)] y resina nanohíbrida [Filtek Z350 (3M ESPE)]. Todas las muestras fueron incubadas en agua destilada a 37°C durante cinco días. El análisis fue realizado con un indentador de microdureza a 10 N y 10s de presión sobre la muestra, nueve indentaciones fueron realizadas a lo largo de la muestra resultando un total de 36 indentaciones por cada grupo. Los datos fueron sometidos a prueba de normalidad de Kolmogorov-Smirnov y un análisis de ANOVA (post-hoc) de Tukey. Resultados: Los valores de VHN correspondieron de la resina más dura a la más suave como se muestra a continuación: Filtek Z350 (71.96±6.44) (p<0.01)> Amelogen Plus (59.90±4.40) (p<0.05)> Feeling lux (53.52±5.72)> Te-Econom Plus (53.26±5.19). Conclusiones: De acuerdo con nuestros resultados, la dureza de las resinas compuestas convencionales evaluadas puede soportar las fuerzas masticatorias pero las resinas compuestas nanohíbridas mostraron una mejor microdureza Vickers que clínicamente puede resultar como una adecuada opción para restaurar tratamientos mínimamente invasivos.

Palabras clave: *Microdureza Vickers, Resina compuesta, In vitro.*

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