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# COMPARISON OF THE MECHANICAL PROPERTIES OF TWO NANO-FILLED COMPOSITE MATERIALS

## *Comparação das propriedades mecânicas de dois materiais nanocompósitos*

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### Abstract

**OBJECTIVE**: The purpose of this study was to quantify and compare the mechanical properties of two commercially available nanocomposite restorative materials. **MATERIAL AND METHODS**: Specimens of two nanocomposites, Z350™ and Grandio™, were polymerized with a LED light for 20 seconds and subjected to mechanical tests. Properties tested included: flexural strength, diametral tensile strength, fracture toughness and microhardness (top and bottom). **RESULTS**: Grandio exhibited significantly higher mean flexural strength values when compared to Z350 (89.1 MPa vs 61.9 MPa). Grandio exhibited significantly higher top microhardness values when compared to Z350. Additionally when microhardness for the top surfaces of each composite were compared with their corresponding bottom surfaces, the bottom surfaces demonstrated significantly lower readings. The properties of fracture toughness and diametral tensile strength were non-discerning factors in this particular study. **CONCLUSION**: Grandio has greater observed values for the properties of flexural strength and hardness when compared with Z350. There was no difference between the two materials with respect to their fracture toughness and diametral tensile strength.

**Keywords**: Dental nanocomposites. Flexural strength. Microhardness. Dental materials.

## *Resumo*

***OBJETIVO**: A finalidade deste estudo foi quantificar e comparar as propriedades mecânicas de dois materiais restauradores nanocompósitos disponíveis comercialmente. **MATERIAL E MÉTODO**: Espécimes de dois nanocompósitos, Z350™ e Grandio™, foram polimerizados com luz LED por 30 segundos e submetidos a testes mecânicos. As propriedades testadas incluíram: resistência flexural, resistência tênsil diametral, resistência a fratura e microdureza (topo e fundo). **RESULTADOS**: O Grandio apresentou valores de resistência flexural média maiores quando comparados com o Z350 (89.1 MPa vs 61.9 MPa). O Grandio demonstrou maior microdureza das superfícies de topo de cada compósito quando comparada com sua correspondente superfície de fundo. As propriedades de resistência a fratura e força tênsil diametral não foram fatores discerníveis neste estudo em particular. **CONCLUSÃO**: Grandio possui maiores valores para propriedade de resistência flexural e dureza quando comparado com o Z350. Não houve diferenças entre os dois materiais em relação à resistência a fraturas e resistência a forças tênseis diametrais.*


***Palavras-chave**: Nanocompósitos dentários; Força flexural; Microdureza; Materiais dentários.*

## INTRODUCTION

Since the inception of resin based composite materials for use as restorative dental materials, continuous research and development has occurred to improve their mechanical properties, clinical handling and performance (1). These developments have focused primarily on reducing polymerization shrinkage and stresses by manipulating resin formulation and improvement of mechanical properties such as hardness, flexural strength, fracture toughness, and compressive strength by manipulating the filler factors such as size, shape and concentration of fillers or by the development of novel filler particles. It is generally accepted that an increase in the filler concentration of resin composites is associated with an increase in certain properties such as elastic modulus, flexural strength, hardness and compressive strength (2, 3). In studies on the effect of filler loading on the mechanical properties of hybrid composites it was determined that composites with the highest filler by volume exhibited highest values of flexural strength, flexural modulus, hardness and fracture toughness (4). When characterizing composites, certain properties should be taken into consideration. These include but are not limited to, hardness, flexural strength and fracture toughness (5). Hardness is an indirect measure of the degree of conversion of the material and gives useful information on the depth of polymerization when such measurements are performed on the top and bottom surfaces of cured samples (6-8). Hardness can also give some indication of the material's polishability and abrasion resistance (9).

Flexural strength and fracture toughness are the properties that characterize the fracture behavior of composites. Flexural strength is the material property that gives an indication on the quantity of flaws within the material that may have the potential to cause catastrophic failure once subjected to loading whilst fracture toughness is a measure of the stress intensity at the tip of a flaw which may propagate in an unstable manner (10, 11). Taken together both these properties determine the bulk characteristics, as opposed to a surface characteristic, of the resin composite material (12)

Diametral tensile strength is also an important property when characterizing dental composites since many materials for intra-oral use have measurements of tensile strengths that are markedly lower than their corresponding compressive values. Low values of tensile strength may contribute to early intra-oral failure of materials (13). Since brittle dental materials, which exhibit very limited plastic deformation, cannot be subjected to traditional tests of tensile strength a compression test for tension, also referred to as, an indirect tensile test or diametral tensile test is used.

Recent research has focused on the manipulation of filler parameters in the nano-scale range in order to enhance filler loading and improve mechanical properties (14). The aim of this study was to quantify the mechanical properties of

microhardness (H), flexural strength (ä), fracture toughness ($K_{IC}$) and diametral tensile strength ($ó_t$) on two commercially available dental nanocomposites.

## MATERIALS AND METHODS

Nanocomposites, Grandio™ (Voco, 27557, Cuxhaven, Germany) and Z350™ (3M-ESPE, St. Paul, MN, USA) of shade A3 were selected for this study. Samples were polymerized using 3M Elipar Freelight™ LED light for 20 seconds (3M-ESPE, St. Paul, MN, USA).

### Microhardness

Five disk-shaped specimens were prepared for hardness testing by curing the composites in a 2 X 8 mm split brass mold. The upper and lower surfaces were covered with Mylar strips to ensure smooth surfaces and to prevent formation of an oxygen inhibited layer. The tip of the light curing unit was placed in direct contact with the Mylar strips during curing. Immediately following curing, the specimen was mounted on a hardness tester (Micromet 2130™ Microhardness Tester, Buehler, Lake Bluff, IL, USA) to assess the Vickers hardness (VHN). A 500 g load was applied through a diamond indenter for 15 seconds. Five readings, equally distributed over the surface but well away from the periphery of the sample, were taken for both the top and bottom surfaces of each specimen. The length of the diagonal of each indentation was measured using the eye-lens of the microhardness tester and microhardness for each specimen was calculated using the formula:

$$H = \frac{1854.4 \times P}{d^2} \quad (1)$$

where H is the Vickers hardness, P is the load in grams and d is the length of the diagonal in μm.

### Flexural strength

Five specimens of each composite were fabricated using a rectangular brass mold (31 X 2 X 2 mm) and Mylar strips. A mask of aluminum foil with a circular window cut to the diameter of the curing tip and the width of the specimen (2 mm) was employed to reduce the effects of overcuring. The mask was laid right up against the rectangular mold and after curing the first segment for 20 seconds the window was moved to the new location adjacent to the first section where curing was repeated. Immediately following curing the specimens were placed in a three point bending fixture on two parallel supports, 25 mm apart, in a Hounsfield H50KS ™ tensometer (Tinius Olsen Ltd, Redhill, Surrey, UK, RH1, 5DZ) and loaded at a cross head speed of 0.5 mm/min until catastrophic failure occurred. Flexural strength, ä, was calculated using the formula:

$$(ä) = \frac{3Fl}{2bh^2} \quad (2)$$

where F is the load at failure, l is the distance between supports (i.e. 25 mm), b is the width of the sample and h is the height.

### Fracture toughness

Five specimens of each composite were fabricated using a rectangular brass mold of dimensions 2 X 2 X 31 mm and a Mylar strip. The aluminum mask was employed in the same manner to cure the specimens for flexural testing. The single edge notch beam (SENB) method was used to test fracture toughness in this particular study. Immediately after curing, a diamond disc of diameter 150mm was mounted on a micro milling machine (Proxxon MicroMilling Machine MF70 ™, Proxxon, Hermann, Tonivorst, Germany) at 5000 rpm was used to notch the middle of the sample. The micro-milling machine was set to ensure the exact notch depth of 1.0 mm. The specimens were tested on the Hounsfield H50KS ™ tensometer at a crosshead speed of 0.5mm/min. Fracture toughness was calculated using the formula:

$$K_{IC} = \frac{3\,(a/W)^{1/2}[1.99 - a/W(1-a/W)\,(2.15 - 3.93a/W + 2.7(a/W)^2]\,PS}{2(1+2a/W)(1-a/W)^{3/2}\,BW^{3/2}} \quad (3)$$

where P is the load at failure, B is specimen height (2.0 mm), W is specimen width (2.0 mm), a is notch depth (1.0 mm) and S is the distance between supports (25.0 mm)

### Diametral tensile strength

Five specimens of each composite were prepared using a circular split brass mold (2.0 mm X 6.0 mm). Mylar strips were placed on both the top and bottom of the composite surface during curing to ensure smooth surfaces and the prevention of the formation of an oxygen inhibited area. Immediately following removal from the split mold diametral tensile testing was performed using a Hounsfield H50KS ™ tensometer (Tinius Olsen Ltd, Redhill, Surrey, UK, RH1 5DZ) at a crosshead speed of 0.5 mm/min along the diameter of the specimen. Diametral tensile strength was calculated using the formula:

$$\sigma_t = \frac{2P}{\pi LD} \quad (4)$$

where $\sigma_t$ is the splitting tensile strength, P is the load at failure indicated by the testing machine, L is the thickness of the specimen (2.0 mm) and D is the diameter of the specimen (6.0 mm)

### Statistical analysis

Data for each mechanical property were evaluated with a one way analysis of variance (ANOVA) followed by a post-hoc Tukey HSD at a 0.05 level of significance.

## RESULTS

The mean values for microhardness, diametral tensile strength, flexural strength and fracture toughness together with their significances are summarized in Table 1.

TABLE 1 - Mean (sd) for microhardness [H], diametral tensile strength [$\sigma_t$], flexural strength [ä] and fracture toughness [$K_{IC}$]

| Composite | H(top) | H(bottom) | $\sigma_t$ (MPa) | ä(MPa) | $K_{IC}$ ($MNm^{-3/2}$) |
|---|---|---|---|---|---|
| **Z350** | 61.8 (1.4)$^{A}$ | 59.4 (1.7)$^{B}$ | 37.1 (3.1)$^{E}$ | 61.9(5.1)$^{F}$ | 0.59 (0.1)$^{H}$ |
| **Grandio** | 75.4 (1.8)$^{C}$ | 70.0 (1.8)$^{D}$ | 32.4 (2.4)$^{E}$ | 89.1(9.6)$^{G}$ | 0.69 (0.1)$^{H}$ |
| **For each property there is no significant difference between the values with the same superscript letters** | | | | | |

## DISCUSSION

This particular study evaluated mechanical properties immediately following specimen preparation. It is common to employ a storage regimen prior to assessing mechanical properties. Generally mechanical properties of resin composite are known to be affected by the presence of water (1, 15). Specifically water storage has been shown to affect hardness of composite samples, with flexural modulus of certain composites being affected by more prolonged storage (16). In microscopic analysis of a nanofilled composite following water storage, microcracks were observed at the interface between filler particles and the resin matrix with a reduction in fracture strength even after 24 hours (1). Additionally specimens stored dry but under room light may continue to polymerize due to post-irradiation polymerization, albeit at slower rates, which may affect certain physical properties such as hardness (17). Hence in an attempt to obtain a true reflection of the material property the normally used storage regimen was not utilized in this study.

When comparing the mechanical properties of the two materials tested in this study statistically significant differences were observed with the properties of hardness and flexural strength, with the restorative Grandio exhibiting higher values for both measurements compared with Z350. The differing filler content of the two materials could account for these results. Beun et al when comparing Grandio to Z350 clearly demonstrated a significantly increased filler loading of Grandio at 84% compared with Z350 at 70% by weight (2). In this same study a significant difference in hardness values was observed when comparing Grandio with Z350 (also known as Filtex Supreme) (2). Kim et al also observed a significant effect on the hardness with increased filler loading (4).

Both composites demonstrated significant differences between the top and bottom surface values for microhardness. This could be attributed to the scattering of light by the filler particles resulting in insufficient intensity of the light in the deeper portions of the specimen causing inadequate polymertization (18). Even though the effect of such light scattering may be insignificant for individual filler particles in the nano-scale range, the agglomerated nano-clusters which closely approximate the wavelength of visible blue light would cause discernible difference. This maximizes the scattering effect thus reducing conversion at 2 mm depth as measured indirectly by microhardness.

It is accepted that flexural strength values also increase with increased filler loading of dental composites (3). Even though this statement was true in this particular study the actual values obtained varied greatly from those of Beun et al, where higher values for flexural strength were observed. This may be due to the fact that a polishing regimen prior to the actual testing of the material was not employed with a resultant large population of inherent surface flaws that could lower flexural strength values.

The property of fracture toughness was a non-discerning factor in this study with the two composites tested showing no significant difference. Fracture toughness describes the resistance of brittle materials, such as dental composites, to the catastrophic propagation of flaws under an applied load. A value of fracture toughness indicates a material's ability to resist crack propagation. It is generally accepted that filler particles with spherical shapes have improved fracture toughness measurements since they maximize filler loading and stresses concentrate in irregular points of filler particles whilst spherical particles deflect cracks (2). This result is surprising since Grandio is known to have irregular shaped filler particles (2). It is possible the property of fracture toughness in nanocomposite materials involves more complex factors than just filler loading or shape of the filler particles. The actual interaction between the resin and fillers may play an important role. Indeed Rodrigues et al postulated that with highly filled composites fracture behavior, including fracture toughness, seem not to differ between composites (5).

The values obtained for diametral tensile strength ($ó_t$) in this study were statistically similar. It can be postulated that differences in the filler loading, as measured by other researchers and values provided by manufacturers, have no effect on the property of $ó_t$. Even though diametral tensile strength is an acceptable test for newer resin composite materials because it is sufficiently brittle, as a separate property it cannot be matched with other mechanical properties (19-21). The values for diametral tensile strength observed for the nanocomposite materials in this study are within an acceptable range for resin composites (13).

## CONCLUSIONS

Within the limitations of this study, it can be concluded the restorative Grandio has greater observed values for the properties of flexural strength and hardness when compared with Z350.

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