

# Investigation of Mechanical Properties of Experimental Bis-GMA/TEGDMA Dental Composite Resins Containing Various Mass Fractions of Silica Nanoparticles

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#### Keywords

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# Abstract

**Purpose:** Mechanical properties of dental composite resins need to be improved in order to enhance their performance for applications in direct restorations. Application of nanoparticles in this field is a recent development. The aim of this study was to investigate the mechanical properties of experimental composites containing various mass fractions of silica nanoparticles.

**Materials and Methods:** Experimental composites were composed of a visible-lightcuring monomer mixture (70 wt% Bis-GMA and 30 wt% TEGDMA) and silica nanoparticles of a size ranging from 20 nm to 50 nm modified with  $\gamma$ -methacryloxy propyl trimethoxy silane ( $\gamma$ -MPS) as reinforcing filler. The composites were classified into four groups according to their filler mass fractions ranging from 20% to 50%. Following the same preparation procedure, a conventional composite was also fabricated consisting of a mass percentage of 60% silica fillers having particle sizes ranging from 10  $\mu$ m to 40  $\mu$ m in the same organic matrix, which served as control. Ten specimens were prepared of each experimental group and also of the control. Fracture toughness was measured using single-edge notched bend (SENB) specimens. Specimen fracture surfaces were mounted on aluminum stubs with carbon cement, sputter-coated with gold and examined under scanning electron microscopy (SEM). Flexural strength was evaluated through a standard three-point bending test and Vickers microhardness test was performed to investigate the hardness of the samples.

**Results:** Filler mass fraction had a significant effect on composite properties. Fracture toughness, flexural strength, and hardness of composites at filler mass fraction of 40% of silica nanoparticles were (mean  $\pm$  SD)  $1.43 \pm 0.08$  MPa.m<sup>1/2</sup>,  $149.74 \pm 8.14$  MPa, and  $62.12 \pm 3.07$  VHN, respectively; relevant values for composites at 50% mass fraction of silica nanoparticles were  $1.38 \pm 0.07$  MPa.m<sup>1/2</sup>,  $122.83 \pm 6.13$  MPa, and  $70.69 \pm 3.67$  VHN, respectively, all of which were significantly higher than  $1.07 \pm 0.06$  MPa.m<sup>1/2</sup>,  $104.61 \pm 8.73$  MPa, and  $52.14 \pm 4.02$  VHN of the control, respectively (Tukey's multiple comparison test; family confidence coefficient = 0.95). Measured values for composites at 20% mass fraction of silica nanoparticles were  $0.94 \pm 0.06$  MPa.m<sup>1/2</sup>,  $103.41 \pm 7.62$  MPa, and  $42.87 \pm 2.61$  VHN, respectively; relevant values for composites at 30% mass fraction of silica nanoparticles were  $1.16 \pm 0.07$  MPa.m<sup>1/2</sup>,  $127.91 \pm 7.05$  MPa, and  $51.78 \pm 3.41$  VHN, respectively.

**Conclusions:** Reinforcement of dental composite resins with silica nanoparticles resulted in a significant increase in the evaluated mechanical properties in comparison with the conventional composite. The filler mass fraction played a critical role in determining the composite's mechanical properties.

Composite resin technology has continuously evolved since its introduction by Bowen.<sup>1</sup> Composite materials used in dental restorations consist of a continuous polymeric matrix, usually involving visible-light-cured bisphenol- $\alpha$ -glycidyl methacrylate (Bis-GMA) diluted with triethyleneglycol dimethacrylate (TEGDMA),<sup>2,3</sup> reinforced with a dispersed phase (barium or zinc glasses, quartz, zirconia, silica, etc.<sup>4,5</sup>). Resin composites are widely used in dentistry and have become one of the most commonly used esthetic restorative materials due to their sufficient strength, superior esthetics, moderate cost compared with ceramics, and ability to bond to tooth structure.<sup>6</sup> Furthermore, the possible health hazards associated with the release of trace amounts of Hg from amalgam restorations is an additional reason for the increase in composite resins use.<sup>7</sup>

In recent decades, increasing demands for esthetic dentistry have led to the development of resin composite materials for direct restorations with improved mechanical properties and durability. Research on the resin matrix mainly concentrates on the development of new monomers,8-11 while studies on filler content focus on particle size, loading, silanization,<sup>12</sup> and the development of new particles.<sup>13,14</sup> Such studies are of high importance considering the fact that mechanical properties of dental composites are greatly influenced by the size and mass fraction of the filler particles. The hardness and flexural strength increase with the amount of inorganic fraction.<sup>12</sup> Flexural tests vield results on deformation and fracture of the bulk specimens, while indentation tests offer important information more relevant to applications that involve localized, non-uniform deformation or point contacts such as dental occlusal contacts with surface asperities or third bodies during chewing and wear.15

One of the most important recent advances in this field is the introduction of nanofilled materials by combining nanometric particles in a conventional resin matrix. The essence of nanotechnology is in the production and manipulation of materials and structures in the range of about 0.1 nm to 100 nm by various physical or chemical methods.<sup>16</sup> Hence, new fillers with sizes ranging from 5 nm to 100 nm have been developed.<sup>17</sup>

Application of nanoparticles is useful in many fields where nanoparticles improve mechanical properties of materials, including industry, transportation, electronics, and biomedicine. In dentistry, posterior class I or II restorations require composites that exhibit high mechanical properties, while anterior restorations need composites with superior esthetics.

Therefore, nanotechnology is of great interest in resin composite research. One consequence of applying the reduced size particles is that an increase in filler loading can be achieved with the consequence of reducing polymerization shrinkage<sup>18</sup> and improving mechanical properties such as tensile strength and resistance to fracture. Furthermore, the wear rate is diminished and gloss retention is improved.<sup>19,20</sup> As a result, application of nanocomposites is recommended for both anterior and posterior restorations.

The purpose of the present study was to investigate the mechanical properties of experimental composite resins containing various mass fractions of silica nanoparticles. To investigate the influence of filler particle size and filler loading on mechanical properties of composite resins, the measured values were compared with those of a conventional composite containing silica microparticles. The mechanical properties evaluated were fracture toughness, flexural strength, and Vickers microhardness. Moreover, specimen fracture surfaces were examined under scanning electron microscopy (SEM).

# **Materials and methods**

### **Preparation of composite pastes**

The organic matrix consisted of 70 wt% Bis-GMA and 30 wt% viscosity-modifying comonomer TEGDMA obtained from Röhm (Degussa-Hüls Group, Hanau, Germany). Silica particles with sizes ranging from 20 nm to 50 nm with a mean diameter of 26 nm were used. Experimental composites were classified into four groups according to their filler mass fractions — 20%, 30%, 40%, and 50%. These groups are designated as  $G_{20}$ ,  $G_{30}$ ,  $G_{40}$ , and  $G_{50}$ , respectively.

Surface treatment of the silica particles was achieved by mixing them with 1.5% mass fraction of  $\gamma$ -methacryloxy propyl trimethoxy silane ( $\gamma$ -MPS, Fluka Chemicals, Steinheim, Germany).  $\gamma$ -MPS was prehydrolyzed for 1 hour in an aqueous solution of 70 wt% ethanol and 30 wt% double-distilled water (pH = 3 to 4).<sup>21</sup> The treated filler was dried for over 20 days at room temperature.<sup>10</sup>

Camphorquinone (Fluka Chemicals) was added at 0.5% by weight of the total monomer, followed by the addition of the same amount of N,N'-dimethyl aminoethyl methacrylate (DMAEMA, Sigma-Aldrich, Steinheim, Germany) as the photo-initiator system. The silanized filler was blended by spatulation with the monomer mixture in different amounts ranging from 20 wt% to 50 wt%, and subsequently the prepared pastes were transferred to a clean glass container stored at room temperature.

Following the same preparation procedure, a conventional composite was also fabricated, consisting of a mass percentage of 60% silica fillers having particle sizes ranging from 10  $\mu$ m to 40  $\mu$ m (Röhm) with a mean diameter of 18  $\mu$ m in the same organic matrix as described previously. The conventional composite was designated as the control. All prepared composite materials were based on 100 g of total mixture. Table 1 represents the amount of each component used in the synthesis of the composite resins.

## **Fracture toughness**

A stainless steel split mold of  $25 \times 5 \times 2.5$  mm<sup>3</sup> was slightly overfilled with composite pastes between two glass slides and

 Table 1 Composition of experimental Bis-GMA/TEGDMA composite resins (g)

Composite components	G <sub>20</sub>	G <sub>30</sub>	G <sub>40</sub>	G <sub>50</sub>	Control
Silica filler	20	30	40	50	60
Bis-GMA	55.4	48.5	41.6	34.6	27.7
TEGDMA	23.8	20.8	17.8	14.9	11.9
Camphorquinone	0.4	0.35	0.3	0.25	0.2
DMAEMA	0.4	0.35	0.3	0.25	0.2

clamped under pressure for 30 seconds to allow any excess material to escape. The mold contained a razor blade in its middle to produce a sharp notch. Removing the glass slides, specimens were subsequently polymerized using a visible light-curing unit ( $\approx$ 470 nm, Optilux 401, Demetron/Kerr, Danbury, CT, light output: 500 mW/cm<sup>2</sup>) by overlapping irradiations three times each for 40 seconds on both sides until the full surface of the specimens had been photoactivated. Therefore, single-edge notch fracture toughness (K<sub>IC</sub>) specimens, each with a notch of 2.5 mm in depth were ready for the test. The cured specimens were then stored in distilled water at 37°C for 24 hours before the test.

A three-point bending test with a span of 20 mm was performed to fracture the specimens at a crosshead speed of 0.1 mm/min and a load of 1 kN on a universal testing machine (Model 6025, Instron Corp., Canton, MA), which was calibrated prior to each testing session.

Plane-strain fracture toughness  $\left(K_{IC}\right)$  was calculated as follows:

$$K_{IC} = [PHS/(BHw^{1.5})]f(a/w), (a/w) = 0.5$$

where P is the maximum load at the point of fracture (N), S is the distance between the supports (m), a is the crack length (m), and B and w are the thickness (m) and width (m) of the specimens, respectively. f(a/w) was calculated according to the equation provided in ASTM standard E399. Ten specimens were evaluated in each group.

Specimen fracture surfaces were examined under SEM (Carl-Zeiss DSM-960A, Heidenheim, Germany). Fracture surfaces were mounted on aluminum stubs with carbon cement and sputter-coated with gold.

#### **Flexural strength**

Flexural strength was evaluated according to the ISO 4049 specification for polymer-based restorations.<sup>22</sup> Specimens of  $25 \times 2 \times 2$  mm<sup>3</sup> were prepared using a stainless steel split mold. The specimens were polymerized as described previously for the fracture toughness test. The cured specimens were stored in distilled water at 37°C for 24 hours prior to flexural testing and subsequently removed from water, blotted dry, and tested wet. A three-point bending test with a span of 20 mm was used to fracture the specimens at a crosshead speed of 1 mm/min and a load of 1 kN on the same testing machine used in the previous test. The bending data were recorded as load to failure. The flexural strength was calculated from the fracture load and specimen dimensions according to the following formula:

Flexural strength = 
$$(3PL/2wT^2)$$

where P is the load at fracture (N), L is the distance between two supports (m), w is the width (m), and T is the thickness (m) of the specimens. Ten specimens were tested in each group.

#### **Vickers microhardness**

A stainless steel split mold with a cylindrical cavity of 9 mm in diameter and 2 mm in depth was filled with composite pastes between two microscopic slides and clamped under pressure for 30 seconds to allow any excess material to escape. The polymerization procedure was the same as previous tests. The cured specimens were stored in distilled water at 37°C for 10 minutes followed by a careful polish under water using silicon carbide paper (up to 2000-grit) to ensure a metallographic finish, necessary for a sharp indentation. Microhardness measurements were carried out on the disk-shaped specimens using a Durimet microhardness tester (Leitz, Wetzlar, Germany). A load of 200 g was applied on the surface of the specimens for 40 seconds. The length of the diagonal of five square indentations on the surface of each specimen was measured directly using a graduated eye-lens. Ten specimens were tested in each group. The Vickers Hardness Number (VHN) was obtained using the following equation:

$$VHN = (1854.4 \times P)/d^2$$

where VHN is in kg/mm<sup>2</sup>, P is the load in grams, and d is the length of diagonals in  $\mu$ m.<sup>23</sup>

## **Statistical analysis**

One-way ANOVA was performed to detect significant difference in the evaluated properties. A *p*-value less than 0.05 was considered significant. Tukey's multiple comparison method was used at a family confidence coefficient of 0.95 to compare the measured values.

## Results

Mean values and standard deviations of the fracture toughness, flexural strength, and Vickers hardness of the composites are listed in Table 2. Figure 1 plots the fracture toughness of composites as a function of filler mass fraction. There is a significant difference between the fracture toughness of groups  $G_{20}$ ,  $G_{30}$ , and  $G_{40}$  (p < 0.05), which shows that filler mass fraction influenced this property; increasing the filler mass fraction from 20% to 40% resulted in a significant increase in the measured fracture toughness, while fracture toughness plateaued when the mass fraction was further increased to 50%.

SEM micrographs of fracture surfaces are shown in Figure 2 for (A)  $G_{20}$ , (B)  $G_{30}$ , (C)  $G_{40}$ , and (D)  $G_{50}$ . Silica nanoparticles are dispersed in the matrix homogeneously. Increasing the filler mass fraction increases the compaction of the specimens; therefore, the possibility of the crack propagation decreases.

The composite's flexural strength is plotted in Figure 3. There is a significant difference between the flexural strength of groups  $G_{20}$ ,  $G_{30}$ , and  $G_{40}$  (p < 0.05), which indicates that

**Table 2**Mean values (standard deviation) of fracture toughness, flexuralstrength, and Vickers hardness of composites, n = 10

Composites	Fracture toughness (MPa.m <sup>1/2</sup> )	Flexural strength (MPa)	Vickers hardness (VHN)
G <sub>20</sub>	0.94 (0.06) <sup>A</sup>	103.41 (7.62) <sup>a</sup>	42.87 (2.61)+
G <sub>30</sub>	1.16 (0.07) <sup>B</sup>	127.91 (7.05) <sup>b</sup>	51.78 (3.41)++
G <sub>40</sub>	1.43 (0.08) <sup>C</sup>	149.74 (8.14) <sup>c</sup>	62.12 (3.07)+++
G <sub>50</sub>	1.38 (0.07) <sup>C</sup>	122.83 (6.13) <sup>b</sup>	70.69 (3.67)++++
Control	1.07 (0.06) <sup>B</sup>	104.61 (8.73) <sup>a</sup>	52.14 (4.02)++

Values with different superscripts are significantly different (p < 0.05).



**Figure 1** Fracture toughness of composites as a function of filler mass fraction along with control. Each value is the mean with the error bar showing one standard deviation (SD), n = 10.

filler mass fraction influenced this property; increasing the filler mass fraction from 20% to 40% led to a significant increase in the measured flexural strength. There is a significant difference between the flexural strength of  $G_{40}$  and  $G_{50}$ ; when the mass fraction was further increased to 50%, flexural strength

decreased sharply. No significant difference was detected between flexural strength of groups  $G_{30}$  and  $G_{50}$ .

The hardness values of composites are plotted in Figure 4. There is a significant difference between the hardness of all groups (p < 0.05), showing that filler mass fraction affected this property; increasing the mass fraction from 20% to 50% results in a significant increase in the hardness values from 42.87  $\pm$  2.61 VHN to 70.69  $\pm$  3.67 VHN.

## Discussion

Fracture is one of the major causes of clinical failures in dental composite restorations.<sup>24</sup> Fracture of brittle materials, such as composites, occurs through the propagation of preexisting cracks under tensile stresses. Crack initiation is strongly influenced by the microstructural characteristics of the materials that may lead to stress concentrations and formation of microcracks.<sup>25</sup> These cracks may be produced by bubble incorporation during insertion of the material, or during polishing, or may appear due to microstructural imperfections.<sup>26</sup>

Compaction forces that occur during the bending tests are concentrated at regions of nanofiller particles. Therefore, filler



Figure 2 SEM micrographs of fracture surfaces for (A)  $G_{20}$ , (B)  $G_{30}$ , (C)  $G_{40}$ , and (D)  $G_{50}$  at 10,000 × magnification.



Figure 3 Flexural strength of composites as a function of filler mass fraction. Each value is the mean with the error bar showing one SD, n = 10.

particles initiate formation of cracks, and consequently lead to occurrence of fracture in the resin matrix.<sup>27</sup> As a result, fracture toughness is affected by the particle size and compaction of the filler particles; mechanical properties are also influenced by the particle size and particle size distribution of the filler particles. In this way, if the mass fraction of the filler particles is equal, the compaction of the specimen with the smaller filler particles in size (nanosize) should be higher. Also by increasing the compaction of the particles, the acting force will be distributed among more particles, and the possibility of crack propagation and fracture of the composites may decrease.<sup>27</sup> Increasing the mass fraction of the filler particles increases the compaction of the particles, and as a result, this can reduce the concentration of acting force on the particles and ultimately the fracture toughness will be increased; however, when the filler mass fraction was further increased to 50%, the fracture toughness plateaued (Fig 1). This is likely because the toughness increase from a higher filler mass fraction may be offset by particle entanglement and agglomeration at a high filler content, especially at 50%. It is interesting to compare the dependence of fracture toughness on filler mass fraction with that of flexural strength. Flexural strength increased with filler mass fraction up to 40%, and then decreased sharply when the mass fraction was further increased to 50% (Fig 3). In contrast, the fracture toughness plateaued and did not decrease at 50%.



**Figure 4** Hardness of composites as a function of filler mass fraction. Each datum is the mean with the error bar showing one SD, n = 10.

Composite strength is determined by the intrinsic flaws (surface and volume flaws) in the specimen, such as pores and filler agglomerates. On the other hand, fracture toughness is dependent on the material's resistance to crack propagation, often measured using notched specimens.<sup>14</sup> The notch, which was about 2.5 mm long in this study, was likely much larger than the intrinsic flaws in the specimen. Therefore, the strength of specimens without notches at filler mass fraction of 50% decreased because of strength-controlling flaws from the observed particle agglomeration at filler mass fraction of 50%. The fracture toughness at filler mass fraction of 50%, on the other hand, was not lower than that at 40% filler content, because the other specimens also had the large controlling flaw, the notch. This appears to suggest that while fracture toughness of notched specimens provides useful information, the strength of materials without artificial notches should also be characterized to obtain a more complete view, because most in vivo restorations are without notches.14

Restorations in functional areas are exposed to attrition and wear. Hence, microhardness evaluations may determine their abrasion resistance.<sup>28</sup> Nanofillers have higher contact surfaces with the organic matrix than do microparticles of conventional composites. This leads to improvement of the hardness of composites containing nanofillers<sup>29</sup> and also explains why the hardness values for G<sub>40</sub> and G<sub>50</sub> were significantly higher than that of the control. Significant differences between the hardness values of the experimental composite groups could be explained by their nanofiller content (wt%). The filler mass fraction is directly correlated to microhardness values.<sup>30</sup> Therefore, the significant increase in the hardness values of groups G<sub>20</sub> to G<sub>50</sub> is due to the increase in their filler content.

## Conclusions

Reinforcement of dental composites with silica nanoparticles of a size ranging from 20 nm to 50 nm resulted in a significant increase in the evaluated mechanical properties in comparison with a conventional composite control containing silica particles of a size ranging from 10  $\mu$ m to 40  $\mu$ m. The filler mass fraction played a critical role in determining the composite's mechanical properties. Increasing the filler mass fraction of silica nanoparticles up to 40% resulted in a significant increase in fracture toughness, flexural strength, and hardness of the experimental composites in comparison with the control. Hence, among the investigated composite groups in this study, G<sub>40</sub> seems to exhibit the most desirable performance for application in restorations under load-bearing conditions; however, further studies should be carried out to improve the knowledge of the mechanical behavior of composites containing nanofiller particles.

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