ORIGINAL RESEARCH

Comparative Evaluation of Mechanical Properties of Titanium Dioxide Nanoparticle Incorporated in Composite Resin as a Core Restorative Material

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Abstract

Aim: To compare and evaluate the mechanical properties of 2.5% titanium dioxide nanoparticle (TiO_2 NP) incorporated as filler in an experimental composite resin with everX Flow and MultiCore Flow.

Materials and methods: TiO_2 was prepared and incorporated into experimental dental composite resin. The experimental and traditional composite resin was grouped as follows: Group I: The experimental composite resin with 2.5% of TiO_2 fillers, Group II: everX Flow (GC EUROPE), and Group III: MultiCore Flow (Ivoclar Vivadent). Based on International Standards Organization (ISO) 4049, the samples were prepared for the compressive, diametral tensile, and flexural test.

Results: Statistical analysis was done, and the results were tabulated. Compared to the other tested materials, the experimental composite resin had relatively high compressive strength, diametral tensile strength, and flexural strength. Compared to MultiCore Flow, the everX Flow showed strong mechanical properties.

Conclusion: Based on the result of the study, it can be concluded that the 2.5% TiO_2 NP incorporated as filler in an experimental composite resin demonstrated higher mechanical properties compared to the conventional material.

Clinical significance: The unique photoactivities of TiO_2 NP and their superior mechanical properties make them one of the ideal additives to enhance the performance of polymeric materials.

Keywords: Composite resin, Core material, Mechanical properties, Nanoparticle, Strength, Titanium dioxide.

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Introduction

For effective root canal procedure, the reconstruction of root canal-treated teeth with a permanent, definitive, post-endodontic restoration is a final step as these teeth are considered more vulnerable to fracturing. Restoration of endodontic teeth is a significant concern for the restorative dentist due to the high percentage of failures, and this high incidence of failure has led to the development of a number of restorative alternatives for endodontic teeth.[1,2]

Core buildup is a restoration that is inserted in a badly damaged tooth to preserve the bulk of the coronal portion of the tooth. It is proposed that the placement of the core is necessary if more than 50% of the coronal portion of the tooth is missing.[3] The compressive strength and tensile strength of the core materials arethought to be significant because the core typically replaces a large portion of the tooth structure and has been built to with stand multidirectional chewing forces for many years. These characteristics are important because the core structure must sustain and protect the residual tooth structure and provide an adequate form of retention and resistance for the final restoration. The effectiveness of the final restoration depends on the intact tooth structure and the good performance of the underlying structure.[4,5]

Optimal final restoration for endodontic teeth preserves esthetics, functionality, protects the residual tooth structure, and avoids microleakage. Restorative materials widely used as core materials include silver amalgam, glass ionomer cement, resin-modified glass ionomer, and light-polymerized hybrid composite resin. Most of these materials have not been explicitly designed for this purpose, but as a result of their properties, they have been found to be used in core buildup procedures.[1,4,6]

Among several nanocomposite materials, titanium dioxide (TiO_2) nanoparticles (NPs) are increasingly used due to their nontoxic, chemically inert, and low cost, high refractive index, multispectrum antibacterial characteristics, corrosion-resistant, and high hardness. Literature has also shown that nanoscale TiO_2 reinforcement agents carry new optical, electrical, and physicochemical properties with very low TiO_2 content, making polymer-TiO_2 nanocomposites a promising new class of materials.

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TiO_2 NPs also have outstanding mechanical properties; for example, the elastic modulus of TiO_2 NPs is approximately 230 GPa and is inexpensive, with titanium being the fourth most abundant metal on earth, followed by aluminum, iron, and magnesium. The unusual photoactivity of TiO_2 NPs and their superior mechanical properties make them one of the ideal additives to improve the efficiency of polymeric materials.[7,8] So, the aim of the study is to compare the mechanical properties of modified composite resin TiO_2 with the conventional composite resin used as the core material, namely everX Flow and MultiCore Flow.

Material and Methods

The study was carried out in the Nanotechnology Research Department of SRM Institute of Science and Technology.

Synthesis of Titanium Dioxide Nanoparticles

In a test tube, 7.4 mL of titanium tetra isopropoxide was measured and taken. It was then introduced to 30 mL of 1 $MHNO_3$ aqueous solution, drop by drop. To give a transparent sol containing 2.0 gmTiO_2, the mixture was then agitated for 2 hours. After diluting the colloid with 100 mL of water, the pH of the colloidal solution was then balanced to three by adding 1 M of NaOH solution. This resulted in the TiO_2 colloid being turbid. At room temperature, the suspension was agitated, centrifuged, and then washed with distilled water. The isolated TiO_2 was air-dried for 1 hour at 600°C. Then, the resulting powder was calcinated for 3 hours at 300, 350, 400, and 450°C. A total of 5 gm of TiO_2 NP was synthesized. The transmission electron microscope (TEM) images were obtained using a Philips EM400T operating at 200 kV, and the magnification is about 0.2 nanometers (nm), with a W-source and a point–point resolution of 2Å. Samples for TEM measurements were prepared by dropping a 5 gm of TiO_2 N Ponto a carbon-coated copper grid at room temperature and placed overnight.[9]

Synthesis of Experimental Composite with Titanium Dioxide as Filler

The matrix consisted of two mixed monomers: Bisphenol A-glycidyl methacrylate (bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) (all purchased fromSigma-Aldrich, St. Louis, Missouri, USA). Additionally, diketone (CQ)(Sigma-Aldrich, St. Louis, Missouri, USA) as the photoinitiator and N,N-dimethylaminoethyl methacrylate (DMAEMA) as a co-initiator (Sigma-Aldrich, St. Louis, Missouri, USA) were used for the experimental composite preparations.

Monomer Preparation

In order to allow easier handling of the material, bis-GMA was put in a glass container and preheated at 500°C for 60 minutes. To avoid accidental activation of the photoinitiator, the TEGDMA monomer was then added and stored in 500-mLamber glass bottles (Sigma-Aldrich St. Louis, Missouri, USA). Then, 0.5 wt% CQ and 0.5 wt% DMAEMA were then applied to the monomer mix using a magnetic stirrer and mixed for 60 minutes (VELP, Scientifica, Italy). The prepared mixed monomer was then packed in amber bottles and covered in aluminumfoil. Using a digital scale (0.01 gm readability), all pieces were weighted (PRECISION Advanced, OHAUS, USA).

The reinforcing fillers were silanized amorphous silica 0.8% (Evonik Industries, Essen, Germany) and silanized aluminum silicate fillers45 to 55%(Evonik Industries, Essen, Germany) and the 2.5% TiO_2. The fillers were compounded into a matrix in 50-mL glass Griffin form beakers at room temperature.

Using alkoxy-terminated silanizing agents, the amorphous silica and aluminum silicate fillers were silanized. Using a preprepared solvent mixture of 90 vol% ethanol and 10 vol% deionized water, a 1.0 vol% 3-methacryloxypropyltrimethoxysilane (Sigma-Aldrich, St. Louiscity, USA) solution was prepared. Next, the silane solution was stirred and allowed for 1 hour to hydrolyze. In a glass vessel, the filler, silanizing agent, and a ketonic solvent are taken. The material is stirred at 40 to 50°C for 5 to 8 hours, then the solvent is decanted off and the filler is dried for 2 to 3 hours at 105°C and sieved until composite usage. Fillers were applied and ultrasonically dispersed for 15 minutes. Then, at room temperature, the reaction mixture was stirred for 24 hours. The reaction mixture was filtered and rinsed with absolute ethanol after the silane grafting procedure to eliminate physically adsorbed silanes. The powder was dried at room temperature overnight and then dried for 72 hours at 60°C in an oven to improve the condensation of silanol surface molecules and to eliminate any residual solvent. X-ray photoelectron spectroscopy (Perkin-Elmer PHI 5400, Waltham, Massachusetts, USA) with MgK radiation (h_ = 1253.6 eV) studied the surface elemental compositions of TiO_2 before and after silane grafting.

The above ingredients are sequentially weighed and taken into a mortar and pestle. To obtain a composite mass, it is then mixed manually and held in the oven at 40 to 50°C overnight. It is mixed again manually in the mortar for about an hour after 24 hours of wetting at 40 to 50°C and then kept back in the oven at 40 to 50°C. This method is carried out for 5 to 7 days, or until the necessary consistency is obtained.

Preparation of Test Sample

The experimental and traditional composite resin (Table 1) was grouped as follows:

- Group I: The experimental composite resin with 2.5% of TiO_2 fillers ($N = 30$)
- Group II: everX Flow (GC EUROPE) ($N = 30$)
- Group III: MultiCore Flow (Ivoclar Vivadent) ($N = 30$)

The 30 samples from each group were subgrouped as follows:

- $n = 10$ for compressive test
- $n = 10$ for the diametral tensile strength test.
- $n = 10$ for flexural strength test

The traditional and experimental composite resin was manipulated according to manufacturers' instructions. The specimen dimensions for the compressive test, diametral tensile strength, and flexural strength test were selected according to

Table 1: Composition and percentage of filler in experimental, everX Flow, and MultiCore Flow composite resins

Sl.No.	*Material*	*Filler*
1	Experimental composite	Amorphous silica: 0.8% Aluminum silicate: 45–55% Silanized TiO2: 2.50%
2	EverX Flow	E – glassfibers (w/w): 25%Barium glass: 42–52%
3	MultiCore Flow	Barium glass fillers, Ba-Al-fluorosilicate glass, highly dispersed silicon dioxide: 54%

International Standards Organization (ISO) 4049 (ISO, 1992). All properties were measured at the end of 24 hours.

Compressive Test and Diametral Tensile Strength

Teflon molds covered with polyethylene strips were used. Twenty samples were taken from each group, comprising a total of 60 specimens (30 samples for compressive test and 30 samples for diametral tensile test) measuring 6-mm high and 4 mm in diameter. By illuminating two surfaces of the sample for 40 seconds, photopolymerization was initiated. The samples were then kept at room temperature to be placed in the molds. By using a vernier caliper, the measurements of the samples were determined. Ten test specimens in each group were vertically placed between the blotting paper disks on the plates of the universal testing machine (Instron Machine 3366, made in the USA). The specimens were then loaded at a crosshead speed of 0.5 mm/minute. The fracture load (*F*) in Newtons (N) was reported after each compressive test, and the diametral tensile strength (σ_t) (in MPa) was calculated as follows:

$\sigma_t = 2F/\pi dh$

where

d: diameter (4 mm);

h: height (6 mm) of specimens;

π: 3.1416.

Flexural Strength

For the three-point flexural strength test, according to the manufacturers' instructions and ISO4049, 30 bar-shaped specimens (10 from each group) with dimensions of 25 × 2 × 2 mm were manufactured from each composite resin. The test was performed to determine the flexural strength using a universal measuring machine. At 0.1 mm/minute, the crosshead speed was set. The maximum fracture load (*F*, in N) was registered for each specimen, and the flexural strength (σ_f) was calculated in MPa as follows:

$\sigma_f = 3Fl/2bh$

where

l: distance between the supporting rollers (20 mm);

b: specimen width (~2 mm);

h: specimen height (~2 mm).

Results

Statistical analysis was conducted for the strength of the core materials, and the mean value was measured for each material with its standard deviation (SD). The Kruskal–Wallis test (H) was used with the Statistical Package for Social Sciences (SPSS) version 11.5. The Kruskal–-Wallis test (H) was used for each material to compare the three properties. The compressive strength, diametral tensile strength, and flexural strength data, as well as the results of the statistical analysis data, are shown in Tables 2 to Table 4. The study's results demonstrate that the various core buildup materials evaluated in this analysis provide statistically significant results.

The results of the compressive strength test in Mpa are shown in Table 2. The experimental composite resin had the highest mean compressive strength of 322.61 Mpa, followed by the everX Flow with 277.44 Mpa. The MultiCore Flow had the lowest compressive strength value of 232.87 Mpa.

The mean and SD values of the diametral tensile strength test in Mpa are shown in Table 3. The experimental composite resin had a higher mean diametral tensile strength of 44.94 Mpa, which was followed by everX Flow with 28.73 Mpa. The MultiCore Flow had the lowest diametral tensile strength value of 18.53 Mpa.

Table 2: Mean and SD values of compressive strength (Mpa) of experimental, everX Flow, and MultiCore Flow composite resins

Group	*Mean*	*SD*	*Kruskal–Wallis test*	*p value*
Experimental composite	322.61	1.28	25.806	0.0001
EverX Flow	277.44	1.61		
MultiCore Flow	232.87	1.74		

Table 3: Mean and SD values of the diametral tensile strength (Mpa) of experimental, everX Flow, and MultiCore Flow composite resins

Group	*Mean*	*SD*	*Kruskal–Wallis test*	*p value*
Experimental composite	44.94	0.87	25.824	0.0001
EverX Flow	28.73	0.82		
MultiCore Flow	18.53	0.56		

Table 4: Mean and SD values of flexural strength (Mpa) of experimental, everX Flow, and MultiCore Flow composite resins

Group	*Mean*	*SD*	*Kruskal–Wallis test*	*p value*
Experimental composite	128.40	0.74	25.824	0.0001
EverX Flow	98.04	0.61		
MultiCore Flow	58.31	0.70		

Table 4 shows the effects of the flexural strength test in Mpa. The experimental composite resin was preceded by the everX Flow, which had a mean flexural strength of 128.40 Mpa. Flexural strength values were lowest in the MultiCore Flow of 58.31 Mpa.

The results of the present study show that the experimental composite resin shows improved mechanical properties than the everX Flow and the MultiCore Flow.

Discussion

Root canal-treated teeth can endure the masticatory load only if they have conservative access, no breakdown or fracture characteristics, and no resorption. However, scientific literature has advocated that the endodontically treated tooth is more prone to fracture than vital teeth.[10] Endodontic therapy alone reduces tooth stiffness by only 5%, whereas compromised tooth structure reduces tooth stiffness by 69%. An ideal post-endodontic restoration should be capable of transmitting and distributing functional stresses while maintaining an adequate coronal seal.[11]

For core material selection, strength is not just one criterion, but it is essential. Stronger core materials resist deformation and fracture better, provide more equal distributions of stress, decrease the risk of tensile or compressive failure, improve stability, and increase the likelihood of clinical success.[12]

The purpose of the study is to compare and evaluate the effect of fillers on the mechanical properties of the composite core materials. Two conventional core materials were chosen from a range of composite resin core materials available today, which were everX Flow and MultiCore Flow. The rationale for selecting the material is to compare and evaluate the effect of fiber-impregnated glass fillers (everX Flow) and the silicate glass fillers (MultiCore Flow) with experimental composite modified with TiO_2 NP fillers. The TiO_2 NPs were synthesized based on the techniques described by Venkatasubbu et al. The TiO_2 NPs were then silanized and added to an experimental composite resin.

Compressive strength is known to be a crucial performance measure because to resist masticatory and parafunctional forces,

a high compressive strength is required. Tensile strength is critical since tensile stresses from oblique or transverse loading of their complicated geometric types are exposed to dental restorations. A popular approach for calculating the tensile strength of brittle materials is diametral tensile testing since it eliminates any of the difficulties involved in direct and flexural tensile testing.[13]

All specimens were treated identically throughout this study, which was based on American Dental Association Specification No. 27. Thus, comparisons among materials were appropriately made.

Tables 2 shows the result of mean and SD values of the compressive strength test in Mpa. The experimental composite resin showed a higher mean compressive strength of 322.61 Mpa. The result of the study was in accordance with the study by Elbatanony et al. who had shown an improved compressive strength of the TiO_2-incorporated composite resin.[6] The TiO_2 NPs create micro-cross-linked hard points in the network, which gives the rigidity to the material and hence the improved compressive strength than the other materials compared.[14] The everX composite core material showed a mean compressive strength of 277.44 Mpa, which is lower than the experimental composite but higher than the MultiCore composite resin. The everX has a short composite fiber substructure that protects the composite layer of the surface particulate filler and thus prevents crack propagation in addition to distributing the stresses.[15] The MultiCore composite resin showed the least mean compressive strength of 232.87 Mpa. The lack of fibersin MultiCore compared to e-glass fibers as in everX made it to least to withstand the compressive strength.[16,17]

Table 3 shows the result of mean and SD values of the diametral tensile strength test in Mpa. Compared to the materials tested, the experimental composite resin showed ahigher mean diametral tensile strength of 44.94 Mpa. The crystalline structure of TiO_2 NP fillers gives the experimental composite resin to withstand the maximum amount of stress before fracturing under tensile strength. The everX showed a mean diametral tensile strength of 28.73 Mpa, which is lower than the experimental composite but higher than the MultiCore composite resin. The results of the study were in accordance with the study done by Garlapati et al. who had shown improved fracture resistance of the everX compared to other materials tested.[18,11] The random fiber organization is important for the optimal reinforcement of the polymers to move stresses from the polymer matrix to the fibers and hence the higher diametral tensile strength than the MultiCore. The MultiCore Flow showed the least mean diametral tensile strength value of 18.93 Mpa.[18] The crack propagated without any hindrance under the Ba-Al-fluoro-silicate glass fillers, making the material to be week under tensile strength.[11] The mechanical properties of composite core materials, such as compressive and flexural strengths, are attributed to failure of the core materials.[19]

Table 4 shows the result of mean and SD values of flexural strength test in Mpa. The experimental composite resin showed ahigher mean flexural strength of 128.40 Mpa. The improved mechanical properties of TiO_2 NP and the silanization of TiO_2 NP with the resin matrix create highly crack-resistant hard points in the final network formation, giving the material a higher flexural strength.[20] The everX showed a mean flexural strength of 98.04 Mpa, which is lower than the experimental composite but higher than the MultiCore composite resin. The short and random e-glass fiber orientation gives the material its greatest resistance to fracture. The MultiCore Flow showed the least mean flexural strength value of 58.36 Mpa. This was in accordance with the study done by Iqbal et al.[12] who had shown that MultiCore composite resin was performing the least in fracture resistance. The filler content and the percentage of filler in MultiCore Flow make the material easily fracture.[21]

The great improvement of mechanical properties of experimental composite resin ismore likely to benefit from the excellent mechanical properties and unique photoactivities of TiO_2 NPs. The TiO_2 NPs create micro-cross-linked hard points in the network, which gives rigidity to the material. When the degree of conversion reaches the maximum limit, more rigid cross-links are formed. The general mechanical properties of TiO_2 NPs as well as the final rigid cross-link network explain the reasons for the improved mechanical properties of the experimental composite resin.[17,22]

The present study compared the flowable composite resin used as core with packable experimental composite resin. The results of the study might have been changed if the experimental composite resin was compared with the packable composite resin used as core. The percentage of TiO_2 NP used in the study was 2.5%, and any change in the percentage of filler might have an influence on the result. The materials used in the study are used as core materials, and when evaluating the strength of the core materials tested, the materials were not placed as core material in the tooth for evaluation. The strength value of the materials compared might have been changed if the materials were placed inside the tooth for evaluation. Further studies need to be done to evaluate the effects of various percentages of TiO_2 NP; also, studies need to be done to evaluate the effect of bond strength between the TiO_2 NP and the fiber post.

The current research compared the flowable composite resin used as the core with the packable experimental composite resin. The outcome of the current study could have been altered if the experimental composite resin was compared with the packable composite resin instead of flowable composite resin used as the core. The percentage of TiO_2 NP used in the experiment was 2.5%, and any adjustment in the filler percentage might determine the performance. The strength of the core material was not evaluated after the placement of the material along with the post in the tooth. In order to determine the effects of the bond strength between the TiO_2 NP and the fiber post, more experiments need to be conducted on the effects of different percentages of TiO_2 NP on the mechanical properties of the core material.

Conclusion

Within the limitation of the study, it can be concluded that the experimental composite resin with 2.5% of TiO_2 NP has improved mechanical properties than the other materials compared, and the MultiCore Flow composite resin showed the least mechanical properties with the materials compared.

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