

An Investigation on Influence of Titaniananotubes on Mechanical Properties of Silica Micro-Filled Dental Composite

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Abstract

Dental resin composites are widely used to replace dental amalgam containing mercury as restorative materials. However, their mechanical properties need to be improved. The purpose of this work was to improve physical and mechanical properties of an experimental silica micro-filled dental composite with TiO₂ nanotubes (TNTs). TNTs were synthesized using an alkaline hydrothermal process and then functionalized with γ -MPS. Nanotubes were characterized by scanning and transmission electron microscopies, X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy. Experimental dental composite was made by using silica micro particles and was reinforced by varying quantities of nanotubes (0-10 wt%). Flexural strength and modulus of resulting composites were measured using three-point bending method. The degree of conversion (DC) of monomers in composites was measured by means of FTIR spectroscopy. The wear resistance of reinforced composites was determined by pin-on-disk method. Also, compressive strength and water sorption of composite were evaluated.

Microscopy and spectroscopy characterization methods revealed formation of TNTs and verified the process of functionalizing. The reinforced composite with 3 wt% TNTs exhibited significantly greater values of flexural and compressive strength in comparison with other reinforced composites. Comparison of reinforced composite with 3 wt% TNTs with unreinforced composite illustrated that wear resistance of reinforced composite considerably improved and on the other hand the water sorption decreased which leads to more composite stability.

It was concluded that TNTs are promising filler for improving mechanical properties of dental composite.

Keywords: Dental resin composite; TiO₂ nanotube (TNTs); Flexural strength; Degree of conversion (DC); Wear resistance; Water sorption

Introduction

Conventional resin based dental composite were introduced in dentistry more than 50 years ago and are widely used for the restoration of anterior and posterior carious teeth instead of dental amalgams containing mercury^[1]. Although the amalgams are superior to dental composites in longevity due to their good mechanical properties such as high compression strength, good wear resistance, hardness and shrinkage behavior but the possible release of mercury has raised concerns regarding potential adverse health effects and poor esthetic properties that causes broadly replacement from dental amalgams to dental composites^[2].

Dental composites mainly consist of four main components: a polymer matrix (Bis-GMA, TEGDMA, UDMA), inorganic filler, filler-matrix coupling agent and initiators. The inorganic filler has the most important role in improvement of the mechanical properties of dental composites, such as enhancing modulus, altering thermal expansion

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behavior, radiopacity and reducing polymerization shrinkage by reducing the resin fraction^[3]. Fillers have been traditionally obtained by grinding minerals such as quartz, glasses, barium glass, silica, zirconium oxide or sol-gel derived ceramics^[4]. Dental composites are classified according to their filler particle size to macro filled composites, micro filled composites and hybrid composites. The last one is beneficial as it contains a well distribution of filler different sizes so that smaller particles fit into the spaces between larger particles and provide more efficient packing^[5,6]. Also, different methods and silane coupling agents are used to treat reinforcing filler in order to improve the interfacial bond between the filler particles and resin matrix as well as achieve uniform distribution in the resin^[7].

The introduction of nanoscale fillers to reinforce dental composites offers high surface area to volume ratio which enhances their interfacial interaction with the resin matrix. In fact by increase the filler/resin interface area, stress transfer in the composite is enhanced which in turn causes the increasing the energy dissipation in the fracture system^[8]. Consequently, better stress transfer between fillers and resin matrix causes an increase in mechanical properties. For example, the studies related to the effects of filler size on wear behavior demonstrated that a finer particle size for the composite resulted in less inter-particle spacing, more protection of the softer resin matrix, and less filler plucking, which led to higher wear resistance of the composite^[9].

Filler shape and morphology is one of the most effective parameter in physical and mechanical properties of dental composites^[10]. Many studies have been done on the effect of different filler shapes including rod, whisker, fiber, tube and porous on composite characteristics^[11]. Recently, usage of high-strength, high-modulus nanofiber and nanotube reinforcements with a high aspect ratio^[12,13]. Moreover, the hollow structure of nanotubes allows interlocking with the matrix on internal and external surfaces of the tubes. As a result, higher filler contact can be achieved in composites, resulting in reduced polymerization shrinkage and improved mechanical properties^[14]. It is reported that the addition of 5 wt% Nylon 6 nanofibers could also lead to 36% and 26% increase in flexural strength and modulus, respectively^[15]. Addition of 20 wt% DCPA (dicalcium phosphate anhydrous) nanoparticles with silicon carbide (SiC) whiskers of average 0.9 μm in diameter and 14 μm in length enhanced the flexural strength of dental composites close to 60%^[16]. It is also reported that impregnation of small mass fractions of about 1% and 2.5% of the silanized hydroxyapatite nanotubes in Bis-GMA/TEGDMA dental resins/composites improved mechanical properties of the composite significantly^[17]. Furthermore, addition of zirconia-silica nanofibers up to 7.5 wt% exhibited significant improving on fracture toughness^[18]. Also, addition of Si_3N_4 whiskers increased wear resistance of resin based dental composite in comparison with commercial dental composite^[19].

As a candidate for inorganic filler, TiO_2 in a tubular form has many tremendous properties such as high specific surface area, biocompatibility, nontoxicity, high mechanical properties and efficient photocatalytic property^[20]. Hence, it can be considered as a suitable reinforcement in resin based dental composite. TNTs are usually synthesized via the alkaline hydrothermal method^[21]. Recently, some studies were investigated the mechanical properties of dental resins and commercial resin based composite, flow able dental composite with incorporation

of TNTs as filler^[13]. The results indicated the improvement of strength and module of composites. However, uniform dispersion of nanotubes in the resin matrix is critical as the aggregates or bundles of the nanotubes can reduce the reinforcement effects^[13,14]. In order to prevent inappropriate influences, the surface modification of TNTs is necessary^[22].

In this study, to investigate the effect of TNTs on mechanical properties of experimental resin-based hybrid dental composite, TNTs were incorporated into composite with different weight percentages (0-10%). The flexural strength, flexural modulus, compressive strength and wear resistance of composite were then tested.

Experimental

Materials

Bisphenol A-Glycidyl Methacrylate (Bis-GMA) and Tri-ethylene Glycol Dimethacrylate (TEGDMA) have been used as base resins. Camphorquinone (CQ) as photoinitiator, Dimethyl amino ethyl methacrylate (DMAEMA) as co-initiator, 3-Methacryloxypropyl-trimethoxysilane (γ -MPS), Cyclohexane, n-propylamine, NaOH, were procured from Sigma-Aldrich Chemicals (Milwaukee, USA). SiO_2 microparticles were obtained from Sibelco (Belgium). All materials used as received. TiO_2 anatase nanoparticles (US NANO) were used as the precursor of TNTs.

Synthesis of TiO_2 nanotubes (TNTs): TNTs were synthesized via hydrothermal method according previous work^[23]. Briefly, 2g TiO_2 anatase nanoparticles was dispersed in 50 mL of 10 M NaOH solutions and stirred for several minutes. The mixture was placed in a Teflon-lined stainless steel autoclave and held at 120°C for 48 h. The obtained product was washed with deionized water and 1M HCl until the pH dropped to 6-7. Finally, the resulting nanotubes were dried overnight and then calcined at 500°C for 1 h.

Surface modification of fillers: The silica microparticles and TNTs were silanized following the method of Chen and Brauer^[24]. The powders (5.0 g), Cyclohexane (100mL) as solvent, n-propylamine (0.1 g) as catalyst which accelerates the silanization of the filler particles and γ -MPS (0.5 g) as silane coupling agent were magnetically stirred for 30 minutes at room temperature and then at 60°C for an additional 30 min. The mixture was placed under a rotary evaporator at 60°C to remove the solvent and volatile byproducts. The powders were then heated at 95°C for 1 h under the same rotary evaporator. Finally, the obtained powders were dried at 80°C in a vacuum oven overnight.

Fabrication of dental composites: The resin matrix was prepared containing Bis-GMA/TEGDMA (70:30 wt/wt) were mixed and kept under sonication for 1 h to obtain a homogenous mixture. Then, CQ (0.2 wt %) as a photo initiator and DMAEMA (0.8 wt %) as a co-initiator added into the resin matrix and stirred for another 1 h to ensure uniform distribution in a light proof container. In order to produce base composite, silanized silica microparticles filler added to the resin matrix in various mass fractions (40%, 50%, 60% and 70%). After selecting the composite with optimum filler content, the TNTs were added to the base composite in 3wt%, 6wt% and 10wt%. The filler and resin matrix was mixed using laboratory stirrer (Qualtech).

TNTs and silica microparticles were distributed in the resin matrix, respectively; while between each stages, vacuum conditions were created to improve the homogeneity. The specimens were prepared by light-cure (550 mW/cm², 5 x 8 s on both sides) with a LED curing light (Mintex GT-1200). Triangular bars, Cylinder shaped specimens and disk shape were prepared for bending test, compression testing, wear test and water sorption, respectively.

Characterization of nanotubes and nanocomposites: The morphologies and structures of TNTs, silica filler and the fracture surfaces of dental composite were examined using field-emission scanning electron microscope (FE-SEM, TESCAN MIRA3 LMU, 15kV, Czech). X-ray diffractometer (XRD, Philips PW3710) (Co-K α radiation $\lambda= 1.79\text{\AA}$) at 40kV and 30mA in the 2θ range from 5° to 70° was utilized to specify TNTs crystalline phase. Functionalized surface of TNTs and silica microparticles were investigated by Fourier-transform infrared (FTIR) spectra (Bruker Vector33) using the KBr method with an attached MCT detector in the range 400-4000 cm⁻¹. In addition, the DC of monomers in composites were measured by means of subtracting the percentage of unreacted carbon-carbon double bonds (C=C) from 100%.

Evaluation of mechanical properties: Flexural strength (FS) and flexural modulus (FM) of the reinforced composites were determined by a three-point bending method, according to ISO 4049. Stainless steel mold was used for the preparation of test specimen 2 mm x 2 mm x 25 mm. The filled mold was stored in an incubator at 37 ± 1°C for 24h. The specimens were loaded to failure in a universal testing machine (SantamSTM-20) with constant cross head speed of 0.75 mm/min^[25]. The FS (σ), in MPa, and the FM (E), in GPa, were calculated by means of the following equations:

$$E=(F_1 l^3)/(4bdh^3) \quad (1)$$

$$\sigma=3Fl/(2bh^2) \quad (2)$$

where F_1 represents the load (N) extracted on the specimen, F is the failure load (N), l is the distance between supports in mm, b is the width, and h is the height of the beam in mm.

Cylinder shaped specimens in $\varnothing = 5\text{mm}$, L= 10 mm were prepared according to the D695 ASTM standard which indicates length to diameter ratio as 2:1 for compressive strength (CS) test. Examinations were done with universal testing machine (SantamSTM-20) in cross head speed of 0.75 mm/min. The compressive strength was evaluated based on formula (3):

$$\sigma=F/A \quad (3)$$

where σ (MPa) is compressive strength, F (N) is maximum failure load and A is cross sectional area of specimens.

The wear resistance of reinforced composites was determined by pin-on-disk method, which is commonly used to simulate occlusal contact wear in a two-body sliding wear test, according to the G99 ASTM standard. The disk-shaped samples were prepared with 15mm diameter and 4 mm height. The samples were tested

against a tool steel of 10 mm tip radius antagonist, with hardness of 64 Rc, roughness of 0.1 Ra in the wear apparatus under an applied vertical load of 25 N and the sliding speed at the contact between the ball and the disk was kept constant at 0.05 m/s. The number of cycle was 6000. After wear test, each specimen cleaned under running water to remove all remains. The wear measurement was reported as the volume loss in cubic millimeters. Disk volume loss was measured using following formula:

$$\Delta v=(\pi R d^3)/6r \quad (4)$$

where, R (mm) is wear track radius, d (mm) is wear track width and r (mm) is pin end radius.

Water sorption: In order to determine Water sorption in the dental composites, disk- shaped specimens were prepared with 15mm diameter and 4 mm height. Specimens were immersed in distilled water at 37°C. Water sorption was assessed by weight change over a week period. The wetting test was carried out using a CAM (Contact Angle Measuring) device. Images were taken using DFK 23U618 Color Industrial Camera with the help of a 2X lens. For contact angle measurement, open source image processing software of Image J 1.46v was used. For both pictures, LB-ADSA plugin which was developed by A. F. Stalder et al. was utilized^[26].

Statistical analyses: The acquired data were evaluated by one-way analysis of variance (ANOVA) followed by t-test, with significance denoted at P=0.05.

Results and Discussion

Characterization of silanized TiO₂ nanotube and silica: Figure 1 shows TEM image of hydrothermally synthesized TNTs. It can be seen, tubular structure of TNTs with length average of 10 nm and several hundred nanometers, respectively. The results of XRD pattern show prominent diffraction peaks at 29.3°, 44.1°, 56.3°, 64.1°, 65.2° and 74.3° which denote the anatase crystal phase of TNTs (Figure 1). Bare silica microparticles as main filler in the composite were characterized by FE-SEM. It can be seen in Figure 2 that silica microparticles have average particle size of about 0.5 μm . Since, the reinforcing fillers play an important role in mechanical properties of the composite; it has been proven that this range of particle sizes provide adequate strength with enhanced polish ability and esthetics^[1].

During silanization process the γ -MPS molecules would chemically bond to the surface of filler particles via direct condensation of -OCH₃ groups with the surface hydroxyl groups of the TNTs and silica particles, to form a covalent bond of Ti-O-Si and Si-O-Si, respectively. Also, the -OCH₃ groups on particles surface can be condensed with each other to form a polymeric siloxane film on the filler particles surface (Figure 3-a schematic). Furthermore, Figure 3-b and Figure 3-c exhibited SiO₂ microparticles and TNTs after silanization.

The investigation of FTIR spectra of silica microparticles in Figure 4 displayed the following absorption bands: (1) a broad band nearby 3500 cm⁻¹ shows the silanol groups (Si-OH), (2) an absorption band at 1627 cm⁻¹ is attributed to hydroxyl groups (OH) bending vibration of silica moisture, (3) the

strong siloxane linkages (Si-O-Si) can be observed at 1098 cm^{-1} . In the case of bare TiO_2 , the absorption broad peak at 400-800 cm^{-1} is attributed to Ti-O-Ti vibrations. The FTIR spectra of silanized particles showed the absorption bands: (1) at 2858 cm^{-1} and 2932 cm^{-1} , because of symmetric and asymmetric stretching vibrations of CH_3 and CH_2 groups of γ -MPS, (2) the absorption band at 1744 cm^{-1} is due to the free carbonyl (C=O) stretching vibrations, (3) at 1466 cm^{-1} , due to methylene group (CH_2) of γ -MPS and (4) at 1170 cm^{-1} is assigned to Ti-O-Si stretching vibrations. These spectra demonstrate that γ -MPS has been effectively grafted onto the surface of TNTs and silica microparticles.

Degree of conversion

The DC was calculated from the ratio of absorbance intensities of aliphatic carbon-carbon double bond (peak at 1637 cm^{-1}) against internal reference of aromatic carbon-carbon double bond (peak at 1580 cm^{-1}) after and before polymerization according to formula (5):

$$\text{DC} = 100 \left[1 - \frac{(A_{1637}/A_{1580})_{\text{polymer}}}{(A_{1637}/A_{1580})_{\text{monomer}}} \right] \quad (5)$$

DC is a critical factor that influence the mechanical properties, chemical stability and longevity of the composite^[27]. The DC of the specimens in this study is shown in table 1. The results indicated an increase in DC of composite from 57.45% to 58.70% and 63.15% by addition of 3 wt% and 6 wt% TNTs, respectively. By further increase of TNTs to 10 wt%, DC is dropped to 35.21%. Sun et al.^[28] added 0.5 wt% TiO_2 nanoparticles into a resin monomer without photo initiator. The DC of composite after 2 min of blue light radiation was 15.8%. This result indicates that there exist free radicals on the surface of TiO_2 nanoparticles and these radicals are more likely generated in the ideal location to complete polymerization process. Thus, according to the higher specific surface area of TNTs than TiO_2 nanoparticles, TNTs can produce more free radicals and increase DC of the composite. With increasing TNTs, due to specific surface area and higher surface energy, the tendency to agglomeration and reduce surface energy increases. The formation of the agglomerates may prevent the uniform penetration of light into the resin matrix. Thus, the DC reduced with light scattering growth. Moreover, high content of the TNTs can lead to gradually restricted mobility of monomers and prevented the cross link of monomer chains^[29].

Mechanical properties of reinforced composites

Flexural and compression strength and modulus: Flexural strength (FS), flexural modulus (FM) and compression strength (CS) of Bis-GMA/TEGDMA (mass ratio 70/30) dental composites containing different mass fraction of silanized silica microparticles were examined to optimize an experimental composite for this study. The mechanical tests results were exhibited in table 2. According to the ISO 4049 standard, minimum acceptable FS is 80 MPa, which in this study minimum acceptable FS is obtained 100 MPa. It can be seen that the experimental composites in 60 wt% silica filler show the maximum FS. Mechanical properties of composites are defined by filler content and DC of matrix. As rule of thumb, higher filler and higher DC correspond to increasing in mechanical properties. By increasing 3 wt% TNTs in the composite, resistant barriers to crack prop-

agation grow. Though, in higher percentages of filler (6 wt% and 10 wt%), agglomeration of particles may occur which leads to microvoids formation. It leads to an increase in stress concentration and crack initiation areas in the composite which is consequently associated with reduced mechanical properties^[30]. Furthermore, increase of mass fraction of TNTs from 3% to 6% leads to approximately 24% FS reduction, while DC increased 7%. It can be attributed to the greater impact of filler content compared to DC.

Table 3 shows the FS, FM, CS and CM results of dental composites containing silanized TNTs with weight contents of 3%, 6% and 10%. As explained, the control sample was the experimental dental composite with 60 wt% silica. As can be seen, the values of FS in low contents of TNTs were conducted to a significant increase, while in high filler contents the loss of FS was observed. Usage of high-strength, high-modulus TNTs in small quantities cause homogenous distribution of fillers in the matrix, therefore according to the high surface area to volume ratio, the filler/matrix interface interaction grows up significantly, which leads to a better stress transfer between fillers and matrix in the composite. Furthermore, the tubular structure of nanotubes permits mechanical interlocking between matrix and internal surface of nanotubes^[14]. Consequently, an improvement in mechanical properties is observed. On the other hand, addition of relatively high amount of nanotube in the composite results in the formation of microvoids in the matrix, due to the agglomeration of nanotubes and hence, decrease in homogeneous stress transfer and also increase in stress concentration zones in the composite which reduce mechanical properties.

The fracture surfaces from flexural strength were investigated by FE-SEM. As shown in Figure 5, fillers were completely covered by resin matrix, which confirmed appropriate function of silane to chemically bond methacrylate groups in γ -MPS and resin matrix chemically^[31]. It can be seen that the fracture surface of sample $C_{60m-3TNT}$ (Figure 5-b) are remarkably rougher than sample C_{60m} (Figure 5-a). The rougher fracture surface constitutes a high surface area, consuming more energy in the creation of new surfaces in compare with relatively flat fracture surface^[32]. The crack propagation direction during three-point bending test was from one side to the other side. High strength TNTs were deflecting the cracks and decreasing crack propagation, which rise flexural strength of the reinforced composites with TNTs. As mentioned, by increasing TNTs up to 10 wt% the formation of TNTs clusters can be observed (Figure 6-a) leading to the formation of cracks and then failure of the specimen. The elemental analysis (EDS) of fracture surface (Figure 6-b) demonstrates the presence of TNTs aggregations.

3-2- Wear resistance

Mean wear volume values of dental composite samples of C_{60m} and $C_{60m-3TNT}$ which obtained by wearing against a spherical work hardening steel are demonstrated in Figure 7. It can be seen that addition of 3 wt% TNTs to sample C_{60m} significantly decrease mean wear volume of about 0.226 mm^3 in comparison with that of the composite containing micro-silica particles (C_{60m}) which is about 0.523 mm^3 . Presence of high modulus and ductile TNTs in the composite increases strength and modulus. This is in agreement with the fact that wear rate depends on the material elastic modulus, hardness and toughness^[19]. Additionally, due to

smaller inter-particle spacing and higher surface area of TNTs in the resin matrix than microparticles can result in stronger interface with resin matrix which reduces the wear rate.

FESEM micrographs of worn surfaces of composite- $C_{60m-3TNT}$ and C_{60m} composites after the simulated 6000 cycles are displayed in Figure 8-a and 8-b, respectively. It is clear to observe that the worn surface of $C_{60m-3TNT}$ sample shows less micro ploughing and smoother surface in comparison with C_{60m} sample. Distributed TiO_2 nanotubes between the silica micro-particles with high surface area carry the load and protect the resin matrix from severe destruction. As result, increasing of the matrix wear resistance prevents debonding of the silica micro-particles entirely, which is concluded to smoother worn surface of corresponding composite.

Based on the wear analysis of the resin composites, wear process and mechanism could be explained as follows: the matrix becomes worn, fillers are exposed to the antagonist surface and then detached from the matrix resulting in the appearance of a new matrix layer. This process repeats simultaneously^[33]. Since the adhesive wear is the common form of dry wear and is related to the shear of adhesive bonding, this mechanism of wear plays important role in the wear of a polymer when it slide frequently over the same wear track^[34]. As shown in Figure 8-c and 8-d, sliding direction, ploughing and surface cracking prove abrasive wear mechanism on the surface. Also, the presence of micro cracks in the worn surface can be attributed to cyclic stresses in the fatigue wear process. Thus, the dominant wear mechanism on the worn surface can be described as adhesive wear, abrasive wear and fatigue wear.

Water sorption

The amount of water sorption influence on lifetime of dental composite, hence water sorption has a considerable role in the field of dental composites. The polymer matrix is mainly responsible for the water sorption. The existence of voids and unreacted monomer molecules in the polymer chains turns that process possible. Figure 9-displays the results of water sorption test for the C_{60m} and $C_{60m-3TNT}$ after 7 days immersion in the water. As can be seen, the amount of water sorption decreased in the $C_{60m-3TNT}$ sample due to the presence of TNTs ($p < 0.05$). Since the voids in the polymer matrix can be filled by TNTs, which have tubular structure and nanometer dimensions. Therefore, these voids can be blocked by TNTs. Besides, the DC of composite in presence of 3wt% TNTs increased which leads to reduction of residual unreacted monomer in the matrix and prevented the further water sorption in the composite^[35]. Figure 9-b and 9-c exhibit the contact angle measurements in samples C_{60m} and $C_{60m-3TNT}$ respectively. The wetting test results showed that the addition of 3wt% functionalized TNTs in resin composite increases mean contact angle measurements from 57.8° to 73.3° . It means TNTs improve hydrophobic property of resin composite which can reduce the water sorption.

Conclusion

In the present study, an experimental dental composite with silica micro particles was developed. The synthesized composite was successfully reinforced with TNTs. Flexural strength and compressive strength in the reinforced composite with 3wt%

TNTs reached to 128.9 MPa and 188.4 MPa, respectively. This indicates a growth of 14.7% and 18% in comparison with unreinforced composite, respectively. Furthermore, the results of wear test illustrated that the addition of 3 wt% TNTs reduced volume loss from 0.523 mm^3 to 0.226 mm^3 . Presence of TNTs decreased water sorption of composite from 1.23% to 1.14% that result in better performance of composite in oral environment. Therefore, it can be concluded that TiO_2 nanotube, due to its specific physical and mechanical properties, is a suitable reinforcement in dental composites.

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