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Effect of different sizes of Nano-TiO_2 on wear resistances and surface hardness of resin-based dental composites

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ABSTRACT

Objective: The principal aim of this study was to evaluate the wear resistance and surface hardness of dental nanocomposites. Three difference sizes (5 nm, 21 nm and 80 nm) of nano-TiO_2 particles were treated with the 3-methacryloyloxy-propyl-trimethoxysilane (MPTMS). The nano-TiO_2 was then place in urethane dimethacrylate (UDMA) matrix. Methods: Nano-TiO_2 was sonically dispersed in an ethanol solution containing MPTMS. The treated particles were washed in pure ethanol and then dried. Wear resistance and surface hardness of eighteen groups of experimental dental nanocomposites were evaluated. Each group had a particle size category and was treated with three different concentrations of the silane, (MPTMS), which are 2.5, 10 and 30 wt %. For comparison, a commercially available dental resin, SR Adoro® (Ivoclar Vivadent, Cape Town, South Africa), used as the control, was reinforced with untreated and treated nano-TiO_2. Results: Statistical analysis showed that the effect of the concentrations of MPTMS on wear resistance and surface hardness of specimens was significant ($p<0.001$), which is less than 0.05. The improvement of adding treated 80 nm TiO_2 was better than that of adding treated 5 nm or 21 nm TiO_2. Conclusions: It is concluded that the most available commercial product for dental restorations could be improved by the addition of nano-TiO_2 with relatively large particles size.

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1. Introduction

A study of the modification of dental nanocomposites with nanosized fillers is presented. The incorporation of TiO_2 (titania) nanoparticles, via a silane chemical bond, to a standard dental acrylic resin matrix was explored to determine whether there was an increase in the wear resistance and surface hardness properties of the dental nanocomposites. The addition of small amounts of nanosized silica particles has been observed to improve mechanical properties, adding 1 to 2.5 wt % of nanosized fibrillar silica to a bisphenol-A glycidyl methacrylate (Bis-GMA)/triethylene glycol dimethacrylate (TEGDMA) resin improved the flexural strengths of 128 MPa and 130 MPa, compared with conventionally dental composites materials of 110 MPa and 120 MPa, respectively. This could be due to the reinforcing effect of highly separated and uniformly distributed nanofibrillar silica, whilst the formation of agglomerates of fibrillar silica may debilitate the resulting material (Tian *et al.,* 2008). Nanoparticles exhibit a more homogeneous filler distribution in low viscosity materials, such as bonding agents, which restricts 'filler settled', namely filler-rich, regions within the matrix (Wilson *et al.,* 2005; Ray, 2008). The incorporation of nanosized filler in bonding agents also produced a more homogeneous bond to the tooth/bonding agent interface as filler penetrates the dentine tubules to reinforce the hybrid zone (Matinlinna *et al.,* 2004; Breschi, 2008).

A further phenomenon that contributes to the aesthetic appearance of nanofilled composite materials was that such materials appear translucent because of the small size of the homogeneously distributed nanosized filler particles. The particle size is smaller than the wavelength of incident light (400 to 700 nm) and the scattering coefficient is decreased allowing light to pass through the composite materials without deflection at the interface between the resin matrix and inclusions, such as filler particles and porosity voids (Lee, 2007; Renaton *et al.,* 2009).

2. Materials and Methods

2.1 Materials

Three different sizes of TiO_2 nanoparticles were obtained from various suppliers as indicated in Table 1 below.

Table 1: Nano-TiO_2 particles

Particles size (nm)	Supplier	Trade name	Country of origin
5	TiPE	PC-5	China
21	Evonik	P-25	Germany
80	NELSA	Trial product	South Africa

UDMA and MPTMS were obtained from Sigma Aldrich (Johannesburg, South Africa), and were used as received. Camphorquinone (CQ) and (2-dimethylaminoethyl) methacrylate (DMAEMA), for the Photo-initiating system were obtained from Sigma Aldrich (Johannesburg, South Africa) and were used as received.

2.2 Surface treatment of nano-TiO_2

The surface treatment of nano-TiO_2 was performed by mixing MPTMS in xylene in the presence of 2 wt % by volume of n-propylamine used as catalyst. The nano-TiO_2 was then mixed with MPTMS, by adding three different concentrations of MPTMS (2.5, 10 and 30 wt %), in separate experiments, to 1 g of nano-TiO_2.

Acetic acid was added to the mixture until a pH of 3.3 was reached. This mixture was sonicated for 10 min, stirred for 1 hour with a mechanical stirrer, and then centrifuged for 10 min at 10 000 rpm to separate the silanised nano-TiO_2 from the solution. Before and after centrifuging, the mixtures was washed with ethanol, the mixture was then placed in an oven at 80°C for 12 hours to dry.

2.3 Preparation of the composite resin

The respective series of nanocomposites, namely A, B, and C (based on the particle sizes of the nano-TiO_2 5 nm, 21 nm and 80 nm), each comprises three further groups, were prepared by mixing the monomer resin matrix UDMA with 10 wt % of nano-TiO_2. The nano-TiO_2 mass ratio was determined by weight difference. The following chemicals were also added to the mixture 0.5 wt % CQ as an initiator and 0.5 wt % DMAEMA as an accelerator. The composite was prepared by mixing with a magnetic stirrer in a glass vessel. The mixture was then cast into a stainless steel templates and covered on both sides (top and bottom) with clear thin glass plates. The dimensions of the specimens were 2 mm × 4 mm × 25 mm. As a comparison with a commercial dental resin, in group D, control (SR Adoro®) was reinforced with 2.5 wt % of untreated and treated 5, 21 and 80 nm TiO_2. Specimens were then irradiated in a light curing unit for 60 minute according to the manufacturer's instructions for the control. In order to better simulate the oral cavity environment, samples were be immersed in artificial saliva (pH 5) at 37 °C for 28 days (Bruno *et al.,* 2011; Gopikrishnan *et al.,* 2015).

2.4 Wear resistance (pin-on-disk)

Wear tests were performed using a conventional pin-on-disc machine. Wear tests were performed under reciprocating dry sliding conditions according to the American Society for Testing and Materials. The rotating speed of the disc was 100 ms^{-1} under ambient *in vitro* conditions. A normal load of 15 N was applied to the test material. At the end of the sliding distances, the testing apparatus was stopped and the surfaces of the samples were cleaned with a brush to remove wear loss materials. The weight loss (in mg) was measured with an electronic balance to a precision of 10^{-5} g to determine the wear of the samples. Wear loss was computed from weight loss measurements taken after 100 m sliding distances.

2.5 Vickers hardness number (VHN)

The hardness of values of composites is required to obtain an indication of their wear resistance properties. However, the complexity of the wear process of composites has given rise to conflicting reports regarding the correlation between the hardness of a material and its wear resistance. In this study, the hardness measurements were carried out by determining the VHN. A Highwood HWDM-3 (TTS Unlimited Inc., Osaka, Japan) was used to investigate the relationship between the hardness of the dental nanocomposites and their wear resistance.

2.6 Statistical analysis

The statistical procedure was carried out using Statistical package for the social sciences SPSS 20 software. The univariate analysis of variance enabled comparisons of each group with the standard set by SR Adoro®. The level of significance for all test was = 0.05. The p-values were used to found significant difference in values in order to draw conclusions.

3. Results

3.1 Wear resistance

The effect of the concentration of MPTMS silane added to the nanocomposite containing 5, 21 and 80 nm TiO_2 is shown in Fig. 1. The group which shows less weight loss value provides more wear resistance. From the figure, it is clear that the addition of MPTMS silane increases the wear resistance. This could be attributed to the better bonding of the treated TiO_2 to the UDMA matrix. As the percentage of MPTMS silane was increased the wear resistance decreased hence the TiO_2 nanocomposite with 2.5 wt % showed highest wear resistance.

The p-value was significant, p=0.00. Therefore, the effect of the concentration of MPTMS silane on the wear resistance of TiO_2 nanocomposites was significant. Fig. 1, also shows nanocomposite containing 80 nano-TiO_2 gives higher wear resistance compared to 5 nm and 21 nm nano-TiO_2 particles.

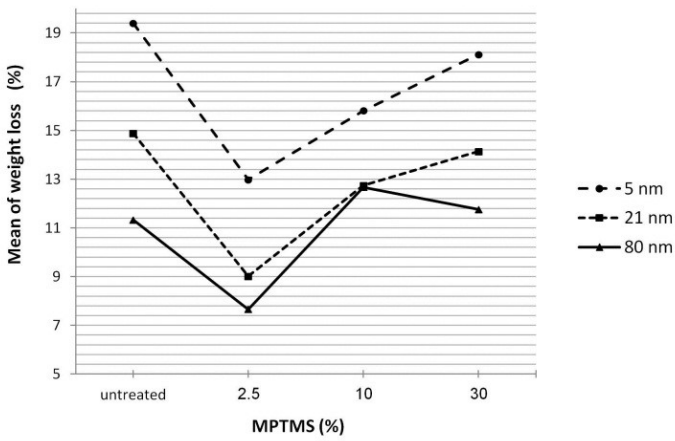

Fig. 1. Mean of wear loss value (%) of TiO_2 nanocomposites treated with 2.5, 10 and 30% of MPTMS.

In Fig. 2, SR Adoro® reinforced with treated 80 nm TiO_2 showed much better wear resistance than any of the other specimens. SR Adoro® reinforced with untreated 5 nm TiO_2 particles demo-nstrated higher wear loss values when compared to the other reinforced and unreinforced SR Adoro®.

Fig. 2. Mean of wear loss value (%) of the SR Adoro® reinforced with untreated and treated 5 nm, 21 nm and 80 nm TiO_2.

3.2 Microhardness

The mean values of the surface hardness for each group are shown in Fig.3. The surface hardness of the 80 nm TiO_2 nanocomposites showed higher surface hardness then 5 and 21 nm TiO_2 nanocomposite. Surface hardness values of the treated TiO_2 nanocomposite with 2.5 wt % of MPTMS were higher compared to those which were treated with 10 wt % and 30 wt % of MPTMS. The p-value was significant (p=0.00). However, the effect of the concentration of MPTMS silane on the surface hardness of specimens was significant. We believe that the small particles give a low surface hardness because they tend to agglomerate due to their high surface energy. We also noted that the addition of treated TiO_2 to the SR Adoro®, composite increases the surface hardness. For example, the surface hardness of the SR

Adoro® reinforced with untreated 5 nm increased when MPTMS silane was added.

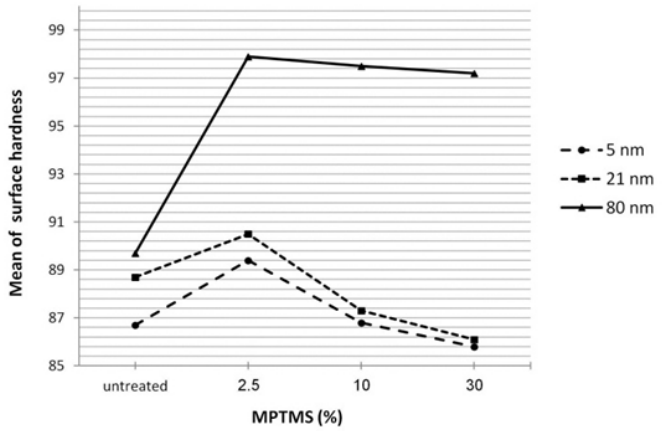

Fig. 3. Mean of surface hardness value of TiO_2 nanocomposites treated with 2.5, 10 and 30% MPTMS.

Fig. 4 shows the effect of particle size of TiO_2 and addition of MPTMS silane on the surface hardness of the SR Adoro®. When we compare the nano-TiO_2 particles of different sizes 5 nm 21 nm and 80 nm, as the size of the particles increases the surface hardness also increases.

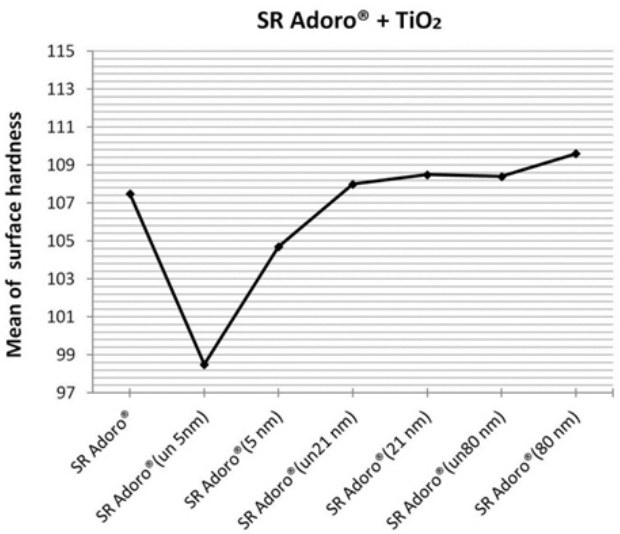

Fig. 4. Mean of surface hardness value of the SR Adoro® reinforced with untreated and treated 5 nm, 21 nm and 80 nm TiO_2, SR Adoro® reinforced with 80 nm shows highest surface hardness

4. Discussion

In this study it was found that treated TiO_2 nanocomposite exhibited significantly lower wear loss, and higher surface hardness than the untreated TiO_2 nanocomposite, this is, in agreement with literature which says that the average filler size and silane coupling agent, have a significant effect on the mechanical properties of nanocomposite materials (Manhart *et al.,* 2000; Sabzi *et al.,* 2009). The optimum concentration of MPTMS seems to be that of 2.5 wt %. Higher concentrations of MPTMS did not improve the properties of TiO_2 nanocomposites (Karabela *et al.,* 2011; Cheng *et al.,* 2014; Wong *et al.,* 2014). At lower silane concentrations (2.5 wt %) the silane molecules have a parallel orientation relative to the particle surface. At higher silane concentrations (10 and 30 wt %) silane molecules form a layer around the filler particles which now have to occupy a random, parallel and perpendicularly orientation relative to the particle surface. Wear resistance and surface hardness showed a maximum value for the TiO_2 nanocomposite contained 2.5 wt % MPTMS. Some studies have revealed a negative correlation between surface hardness values and the wear resistance of dental composite (Manhart *et al.,* 2000; Kim *et al.,* 2002; Faria *et al.,* 2007). The findings of the present study are opposed to the latter. In the present study, TiO_2 nanocomposites with higher surface hardness values showed higher wear resistance. As for the effect of particle sizes, the surface energy of the nano particles is very high, and these particles tend to agglomerate during mixing (Xia *et al.,* 2001; Ali *et al.,* 2016), the samples containing treated 80 nm TiO_2 which were less agglomerated particles showed better mechanical properties than those containing 5 nm or 21 nm TiO_2.

5. Conclusion

Addition of untreated nano-TiO_2 particles into the UDMA, resulted in their poor dispersion and resulting low mechanicals properties of dental nanocomposites. The surface treatment of nano-TiO_2 particles decreases the agglomeration between the nanoparticles and also improves their wear resistance and surface hardness in the dental nanocomposites. As for the effect of particle sizes, the samples containing smaller nano-TiO_2 which were more agglomerated particles showed low mechanical properties. However, a positive correlation between surface hardness values and the wear resistance was observed. Treated TiO_2 nanocomposites with the highest surface hardness values showed the highest wear resistance.

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