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A pilot study on the influence of two reinforcing materials (glass fibre mesh and electro-spun nylon nano fibres) on the flexural strength of a heat-cured poly (methyl methacrylate) denture base resin

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Abstract

Objective: To determine the effect of a methacrylic pre-impregnated resin glass fibre mesh and electro-spun nylon nano fibres on the flexural strength of a heat-cured poly (methyl methacrylate) (PMMA) denture base resin.

Methods: A non-reinforced PMMA control group (Vertex™) and two reinforced groups, incorporating glass fibre mesh (Quartz Splint™) and electro-spun nylon nano fibres (Xantu. Layr™) were made according to ISO 20795-1:2013. Flexural strength was measured using a 3-point bend test in a universal testing machine. Statistical analysis was initially done using one-way ANOVA followed by a post-hoc Tukey HSD test ($P>0.05$) to determine the variance between groups.

Results: The PMMA glass fibre mesh reinforced group had a statistically higher mean flexural strength (99.4MPa) compared to the non-reinforced PMMA control (88.5MPa). There was no significant difference between the PMMA nylon nano fibre reinforced group (92.4MPa) and the non-reinforced PMMA control.

Conclusions: PMMA denture base resin flexural strength is improved by incorporating the glass fibre mesh, while electro-spun nylon nano fibres produced no significant increase in the flexural strength.

Introduction

Poly (methyl methacrylate) (PMMA) is extensively used in the dental industry for the manufacturing of denture base resins. This material has provided sufficient strength to resist normal mechanical failure when treating patients with complete dentures. With the introduction of implant overdentures, they are subjected to increased loads (between 162N and 341N), which can result in denture base fractures (Carlsson, 1984; Wismeijer et al., 1995; Miyaura et al., 2000). Rached et al (2011) reported that this complication affects 12% of prostheses. In an attempt to overcome this problem, Jagger et al (1999) reported on the mechanical reinforcement of denture base resins using E-glass to successfully reinforce both heat-cure and chemically-cure PMMA resins. Several authors have reported that the success of reinforcement materials lies in their ability to bond to the PMMA denture base resin. The chemical bond between the PMMA and the fibres transfers the stresses from the polymer matrix directly to the fibres because without the bond, the fibres can become stress-raising defects (Solnit, 1991; Vallittu, 1996; Kemp et al., 2004; Goguta et al., 2012). Aramid fibres, otherwise known as aromatic polyamide fibres or Kevlar™, can be used to enhance the transverse strength and impact strength of PMMA (Vallittu, 1996; Uzun et al., 1999). Likewise, polyethylene fibres have been shown to increase impact strength, modulus of elasticity and flexural strength. They also have better aesthetics, as it is almost undetectable in the PMMA resin unlike carbon and aramid fibres (Uzun et al., 1999). However, polyethylene fibres do not bond to PMMA and the surface treatment required to improve adhesion is complicated. Glass fibres on the other hand, can chemically bond to denture base resins with silane treatment but it is difficult to incorporate flexible glass cloth accurately due to the complex shape of the oral cavity (DeBoer et al., 1984; Kanie et al., 2005).

Quartz Splint™ (Recherches Techniques Dentaires, Saint Egreve, France) is a methacrylic pre-impregnated resin matrix mesh, which can chemically bond to the majority of composites and is currently recommended by the manufacturer to reinforce PMMA denture base resins. The manufacturer also claims that the product can replace standard metal frameworks, thereby avoiding corrosion and show-through. The Quartz Splint™ mesh is marketed to have a superior flexural strength as compared to polyethylene or glass fibre products[1].

Xantu.Layr™ (Xantu.Layr™, Revolution Fibres Ltd, Auckland, New Zealand) is an electro-spun un-directional mat of nano fibre nylon material, which is currently used for improving the strength of composite materials. The product is produced via a process of spinning fibres with the help of electrostatic forces known as electro-spinning. It utilizes a high voltage electric field to produce electrically charged jets from a polymer solution. The highly charged fibres are directed towards the oppositely charged collector, which can be a flat surface or a rotating drum (Thandavamoorthy et al., 2005). The application of a nano fibre to the reinforcement of

1 RTD Dental, Quartz Split Mesh (2013) http://www.rtddental.com/products/reinforcement/quartz-splint-mesh

Table 1. List of materials used in this study in terms of brand name, manufacturer, constituents and processing technique

Constituents	Processing	Product brand name	Manufacturer
Poly(methyl methacrylate)	Heat-cure	Vertex™ Rapid Simplified Shade 5	Vita Zahnfabrik, Bad Säckingen, Germany
Quartz mesh, Dimethacrylate resins (Bigma, Tegdma), light initiator, silica	LED light cure 20s/ cm^2	Quartz Splint™	Recherches Techniques Dentaires, Saint Egreve, France
Nano fibre, Electro-spun nylon	Inter-laminar processed	Xantu.Layr™	Revolution Fibres Ltd, Auckland, New Zealand

a denture base resin is novel, as the authors were not able to identify any published reports of its use in the dental literature.

The aim of this pilot study was to determine the effect of a methacrylic pre-impregnated resin glass fibre mesh (Quartz Splint™) and electro-spun nylon nano fibres (Xantu. Layr™) on the flexural strength of a heat-cured PMMA denture base resin (Vertex Rapid Simplified™).

Methods

The materials used in this study are outlined in Table 1. Eighteen specimens were prepared following manufacturer's instructions according to ISO 20795-1:2013, a non-reinforced control group (n = 6) and two reinforced groups (n = 6), incorporating a single layer of Quartz Splint™ mesh and Xantu.Layr™ electro-spun nano fibres respectively (ISO, 2013). A polished stainless steel rectangular plate (65 mm x 45 mm x 5 mm) and traditional denture processing flasks were used to create plaster moulds to fabricate the required rectangular plates for each of the experimental groups.

Vertex Rapid Cure Simplified denture base control plates were produced by following manufacturer's instructions. The dough was trial packed under 4kg/cm^2 pressure using the prepared moulds. Final packing took place under 4.5kg/cm^2 pressure. For the Quartz Splint™ reinforced specimens, Quartz Splint™ mesh was cut to fit the prepared plaster mould and cured under an LED light source (Solidilite Light Curing Unit, Shofu Incorporated, Kyoto, Japan) for 20 seconds per square centimetre, as per manufacturer's specifications. A wax spacer that occupied 1/3 of the thickness of the specimen was used to ensure the correct location of the Quartz Splint™ mesh. The rest of the cavity was filled with Vertex Rapid Cure Simplified denture base material. This was done using plastic sheets to separate the wax from the acrylic and avoid contamination, and allowed the cured Quartz Splint™ mesh to be placed at the correct level. The wax spacer was then removed and replaced with the rest of the acrylic. The plate was trial packed under 4kg/cm^2 pressure and on removal of the plastic film, evaluated to make sure that the mesh remained in the desired position. Final packing took place under 4.5kg/cm^2 pressure. The Nano-fibre plates were manufactured in a similar fashion, but because of fragility the acrylic dough was trial packed twice with the spacer in place; once for each half of the mould. The Nano-fibre was then placed between the two halves before the final pressure sequence was completed. All specimens were polymerized for 20 minutes at 100°C.

The acrylic plates were sectioned into test specimens under water irrigation, using a diamond grit blade on a low speed cutting machine (DTQ-5, Laizhou Huayin Testing Instrument Co., Ltd., Shangdong, China) then wet ground to final dimensions of 64 mm x 10 mm x 3.3 mm using 1000 grit silicon carbide abrasive paper (Struers, Denmark) and labelled accordingly. All prepared specimens were stored in a water bath at 37°C for 48 hours prior to the flexural testing. A 3-point bend flexural strength test was performed using a universal testing machine (Instron 3369, Instron, USA) and a constant displacement rate 5+/-1mm/min. The ultimate flexural strength was calculated in mega Pascals using the following equation: (ISO, 2013)

$$\sigma = \frac{3Fl}{2bh^2}$$

Where:

F = maximum load, in Newton's

l = support span in mm

b = width of specimen in mm

h = height of specimen in mm

The mean, standard deviation and statistical significance (at P=0.05) were calculated using a one-way ANOVA on groups of data and then post-hoc Tukey HSD between groups to determine significant differences. The specimens from each group showing the highest and lowest flexural strength were analysed under a scanning electron microscope (SEM) (Japan Electron Optics Laboratory Company Limited, Tokyo, Japan) to establish the mechanism of failure.

Results

The mean flexural strength (MPa) and their respective standard deviations (SD) of each specimen group are given in Figure 1 and Table 2. The mean flexural strength ranged from 88.5 (±3.6) MPa for the control group to the highest at 99.4 (±4.3) MPa for the quartz mesh reinforced group. The

Table 2: Mean, standard deviation, maximum and minimum MPa values for all three test groups

	Control (Heat cure)	Quartz mesh	Nano fibre
Mean MPa (S.D)	88.5 (±2.6)	99.4 (±4.3)	92.6 (±6.1)
Maximum MPa	91.3	105.4	101.5
Minimum MPa	85.3	95.0	86.6

one-way ANOVA results showed a statistical difference in flexural strength values between test groups (F=8.82, *P*=0.03). Post Hoc Tukey HSD analysis revealed that the difference between the control group and quartz mesh was very significant (*P*=0.002,) and that between nano fibre and quartz mesh was also significant (*P*=0.05). No statistical significance was found between the control and nano fibre group (*p*=0.295).

SEM revealed both simple and complex fracture patterns showing clear tension and compression areas. Figures 2a to 2c reveal typical fracture surfaces with tension side at the top of the image (dotted white arrows show fracture initiation sites). Figure 2a illustrates an example of a typical simple fracture initiating on the tension side (shown by dotted white arrow) with hackles propagating outwards and downwards from the fracture initiation site to a classic compression curl. Figure 2b shows a typical complex failure where the initiation site (shown by dotted white arrows in 2c) was at the quartz mesh interface and not at the top of the tension surface. Figures 2c and 3 shows typical complex failure where the initiation site (shown by dotted white arrow) was on the tension side but secondary initiation sites were observed along the plane where the nano fibres where present (Figure 3).

Figure 4 shows failure initiation of the Quartz Splint™ specimens on the tension side; then the crack tip change in direction as it goes into compression. This implies that the Quartz Splint™ mesh is sitting in tension and then changes in direction to compression when the fracture occurs. The fibres are twisted and there is a torque effect evident. The crack appeared to propagate along the Quartz Splint™ mesh with it acting as an initiation site (Figure 4). We also see evidence of brittle failure of the light-cure resin encasing the mesh fibres, indicating they are not fully bonded into this matrix (Figure 5).

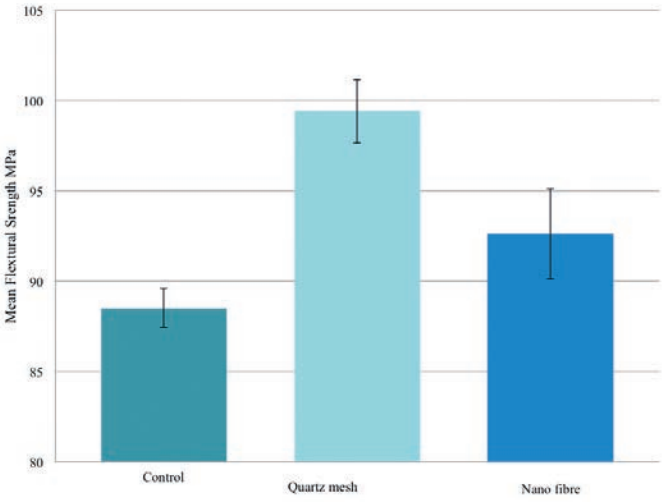

Figure 1: Mean flexural strength values in MPa for test groups (n = 6)

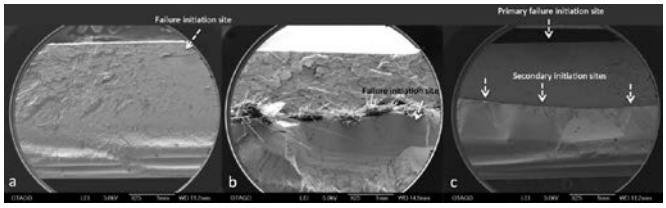

Figure 2a: SEM of PMMA control fracture surface; **2b:** SEM of PMMA fracture surface with quartz mesh; **2c:** SEM of PMMA fracture surface with Nano fibres.

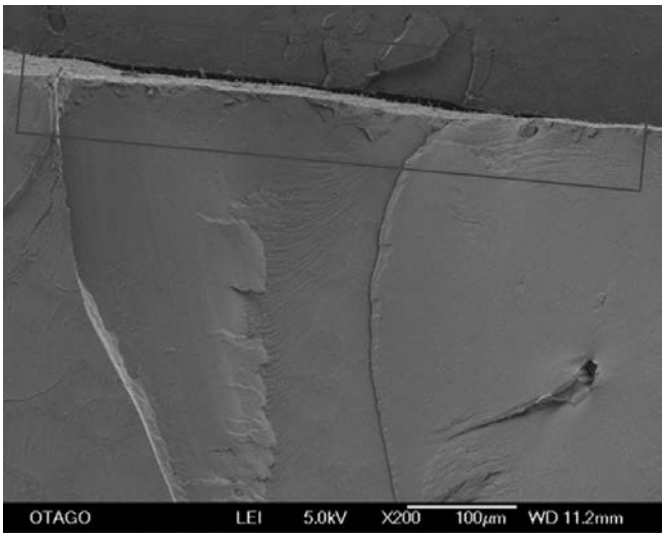

Figure 3. SEM image showing inter-laminar fracture of a Nano-fibre reinforced specimen. Note the woolly vellus-type layer (red box) which acts as a separation layer between the acrylic layers.

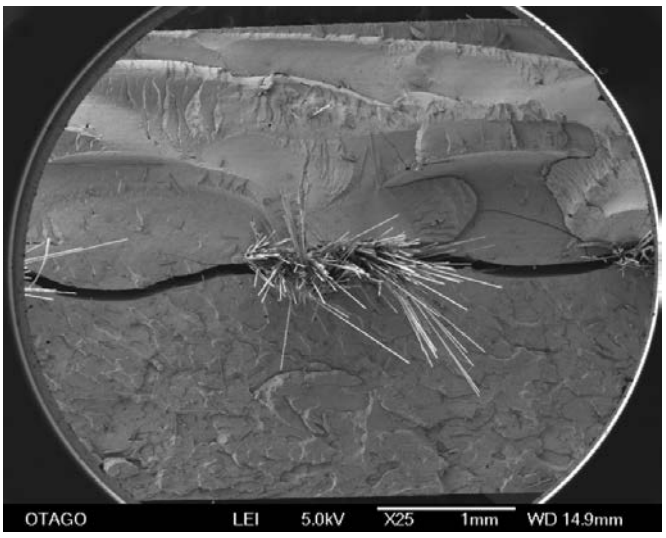

Figure 4. SEM image of PMMA acrylic with quartz mesh showing a complex fracture which has propagated along the quartz mesh and has complex multi-directional stresses evident.

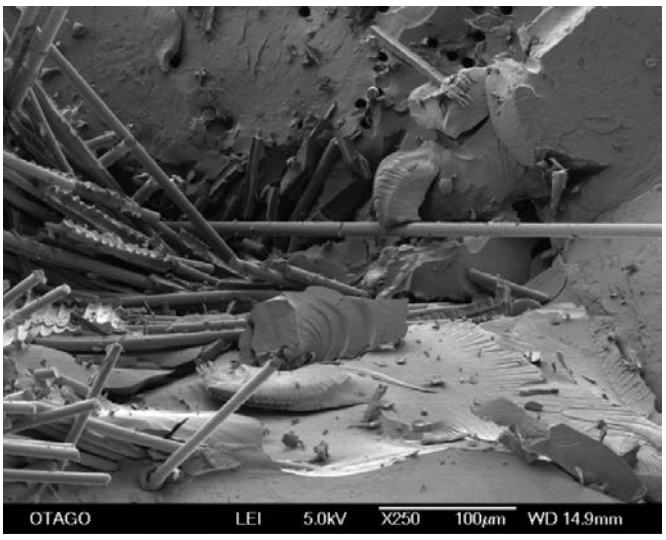

Figure 5. SEM image showing the light-cure acrylic around the quartz mesh bonded to well to the PMMA, but the fibres itself did not integrate with the light-cure matrix and that the fibres are detached, and broken due to torque effect.

Discussion

The purpose of this pilot study was to evaluate feasibility of a full scale study on the reinforcing effects of Quartz Splint™ mesh and nano fibre on PMMA. The test parameters of this study are validated by the PMMA (Vertex) manufacturers information sheet (XU162P03, XX494P02, XU49IP04) which reports the material as having a flexural strength of 85 MPa, while our study produced a mean of 88.5 MPa. It also provides a sound basis to draw a conclusion with regard to the reinforcing material's benefits.

A limitation of this study was that the specimens could not be broken under water due to a lack of appropriate facilities. This is a common limitation and most studies carried out are done in a dry environment. Ideally specimens would be broken in water as this can assist crack propagation.

To understand the results of this study it is important to recognise how the toughness of the PMMA material is dependent on the amount of stretching of polymer chains that occurs during crack growth. This plastic deformation consumes energy, and causes crack tip blunting. The addition of fillers to a PMMA resin system offers improvements in mechanical and physical properties. The toughness of a material will be increased due to the presence of fillers. The particle shape and/or size in the PMMA acrylic will increase the surface area of the filler. As a growing crack meets a filler particle, it must travel around the particle in order to continue to propagate. As the energy required for crack propagation is proportionate to the total area of new surface created, the toughness of the material is thereby increased (Darvell, 2009). When the specimens are under tension, unbonded reinforcing fibres will not prevent deformation causing a decrease in flexural strength. This was not observed in the present study. A possible explanation for this can be that the nano fibres had no positive or negative effect on the flexural strength of the material. This could be due to the diameter of the individual fibres, which might have been inadequate to create voids of sufficient size to cause a weaker matrix. Conversely it is possible that the strength benefits they provided were counteracted by the voids created.

Hamza et al. (2004) tested a variety of reinforcing fibres for PMMA and succeeded in showing that the addition of fibres increased mechanical properties of provisional resins. This increase was contributed to stresses being transferred o the fibres which have a higher tensile strength. The stronger the adhesion was between the fibres and the matrix, the greater the flexural strength. Therefore, the presence of poorly bonded fibres can be equivalent to voids. One way to increase the adhesion between the polymer matrix and the fibres is by resin impregnation of the fibres before application. This would allow the surface of each fibre to come into contact with the resin, wetting the fibres with monomer as has been commonly used, but residual monomer may impair other properties. Interpreting Figure 3 and the results of this study, it was evident that the nano reinforced fibres were not well bonded to the PMMA and there was minimal strengthening advantage by adding it. The Quartz Splint™ mesh has fibres that are encased in a light cure resin and it was evident that this resin did bond well with the PMMA, hence providing additional strength. However as shown in Figures 4 and 5, when failure did occur, the fibres were able to dislodge cleanly from the light-cure matrix and in fact the fibres were not fully bonded to the light cure resin in the first instance. Despite this there was a benefit in increased flexural strength. The reinforcing effect of the Quartz Splint™ mesh was further confirmed by the dynamic fracture and the torque effect observed in the specimens.

The orientation of the fibres plays an important role in the functionality of the reinforcing medium. Producing acrylic with correctly oriented fibres can be technically difficult and result in the fibres not being in the optimum location. Randomly dispersing fibres is more easily produced, however fibres placed perpendicular to the direction of applied stress will deliver a more reliable result (DeBoer et al., 1984; Jagger et al., 1999; Kanie et al., 2005). This seems to suggest a premade, rigid 'net' (like the mesh being tested) would work well, as the problems of both orientation and centreing are solved.

The flexural strength of the reinforced groups was higher than the control, but the nano fibre increase was not statistically significantly increased. The quartz mesh increase was very significant. The mean flexural strength of 99.4 MPa for the PMMA reinforced with quartz mesh gives it a strength of around 14 MPa greater than a non-reinforced acrylic. Quartz mesh is an effective reinforcement material for PMMA denture base material.

Conclusions

Within the limitations of this pilot study

1. PMMA reinforced with quartz mesh produced a statistically significantly higher flexural strength than non-reinforced PMMA.
2. SEM analysis showed that quartz mesh changed the fracture dynamics of the PMMA specimens.
3. PMMA reinforced with nano fibres did not result in a statistically significant increase in flexural strength and the material is very difficult to place accurately.
4. SEM analysis indicated that nano fibres did not bond to the PMMA matrix and appeared to have little involvement in the fracture dynamics of the specimens.

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News and comment

Sir John Walsh Research Institute Three Minute Thesis competition: postgraduate research

The 2017 edition of SJWRI 3MT, the three-minute thesis competition for postgraduate research students, was held on Wednesday 28 June in the Hunter Centre. As in previous years, entrants were required to present for no longer than three minutes on the topic of their thesis.

This year's winner of first prize was first year PhD student Sabarinath Prasad, of the Craniofacial Biology and Clinical Oral Physiology research programme, for his presentation 'The S.M.A.R.T. Study'. Sabarinath's project involves developing wireless devices to measure muscle activity in real time via surface electromyography. The runner up was Yasmeen Ruma, who has just begun her PhD in the Molecular Microbiology research programme, with her presentation 'Structure-directed antifungal drug discovery'.