

Flexural properties of electrospun polymethyl methacrylate microfiber-reinforced BisGMA for dental post prefabrication

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ABSTRACT

Introduction: A dental post is a restoration to preserve the remaining tooth structure thus can be functioned normally. Many researchers suggested a fiber dental post due to its biomechanical properties that are similar to dentin structure. This study aims to analyse the flexural properties of electrospun polymethyl methacrylate microfiber-reinforced BisGMA for dental post prefabrication. **Methods:** The sample used was following the ADA guideline, as well as for the number of samples. The sample size was 25×2×2mm, which is close to the average dental post size. PMMA microfibers were prepared by dissolving heat cure PMMA powder with 99% acetone, then electrospinning with a rotary collector. Acquired PMMA microfibers were immersed into the resin matrix containing BisGMA, camphorquinone, and 2-dimethylaminoethyl methacrylate (DMAEMA) as a monomer, initiator, and co-initiator, respectively, to prepare the dental posts. **Results:** PMMA microfibers structure and surface fracture of dental posts were confirmed by Scanning Electron Microscopy (SEM). PMMA microfibers show unaligned fiber morphology with an approximate diameter size of 1-5 µm. A universal testing machine was used to measure the dental post's flexural properties (flexural strength and flexural modulus). Dental posts with PMMA fibers showed higher flexural strength (83.5 ± 10.7 MPa) compared to the dental post without PMMA fibers (61.7 ± 3.03 MPa) with a p-value <0.05. On the other hand, PMMA fibers' addition did not significantly increase the dental post's flexural modulus. **Conclusion:** The PMMA microfibers can intimately adhere to the BisGMA mixture as the resin matrix. Therefore, the PMMA microfiber significantly improves the flexural strength of the BisGMA for dental post prefabrication.

Keywords: Dental post, flexural properties, polymethyl methacrylate, BisGMA

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INTRODUCTION

A dental post is a treatment to rebuild a tooth structure that lost a massive portion of the tooth after endodontic treatment. It can function as a solid restorative unit together with the dental crown.¹⁻³ Nowadays, fiber dental posts were introduced to the market, and it is known as less stiff and possesses acceptable stress distribution in remaining dentin structure, thus hindering fracture incident.^{4,5} Fiber dental post is one type of dental post that contains a high volume percentage of continuous fibers in a methacrylate-based resin, for instance, BisGMA. This kind of dental post is more aesthetic than metal post and ceramic post. Therefore, it is more applicable to be used for anterior crown restoration.² However, despite the advantages of fiber as a dental post material, its ability to enhance the mechanical properties of the dental post is still controversial. Some studies show positive results while others do not.¹

PMMA is a type of material that can be used as a reinforcing material for methacrylate-based resins because both are known to be chemically bonded.⁶ PMMA fibers have the following characteristics, such as biocompatible, transparent, colorless, easy to handle, good processing ability, low cost, and high surface area to volume ratio.^{7,8,9,10} However, the utilization of PMMA fiber as a reinforcing material for methacrylate-based resins has not been widely found

It is well known that there is a relationship between the enhancing mechanical properties and characteristics of fiber beneath the resin matrix. The characteristics are type, size, integrity, density, distribution, configuration, and the bond between the fibers and the matrix.^{11,12} Studies show that characteristics play a role in determining the flexural strengths of the fiber dental posts. The flexural strength represents the fracture resistance of the specimens toward flexural stresses. It is determined by the highest load the specimen can retain and related to the specimen configuration.^{13,14} The selection of materials that can produce interfacial bonds needs to be considered because the discontinuity in the interface and the existence of the void within the resin may ruin the mechanical properties of fiber

dental posts.^{11,15} In this study, the author proposes to synthesis the PMMA fiber in micron size using the electrospinning method. This method has several advantages such as simple procedure, easy to control the size, and alignment of the fibers.^{16,17} Upon synthesis of the PMMA microfiber, it is used to be a reinforcing material of BISGMA for the dental post prefabrication then analyze its flexural properties via flexural strength and modulus value and interface evaluation.

METHODS

Polymethyl methacrylate (PMMA) heat cured (Triplex Hot) was purchased from a local dental store in Bandung, Indonesia. Bisphenol-A-Glycidyl Methacrylate (BisGMA), champhorquinone (CQ), 2-(dimethylamino)ethyl methacrylate (DMAEMA) were purchased from Sigma-Aldrich, Singapore. PMMA 0.6 g were dissolved into 20 ml acetone 99% (concentration of pmma in the solvent was 38.5%). The solution was stirred for 30 minutes by a magnetic stirrer until PMMA dissolved. The solution was then electrospun with a rotary collector and aluminum foil sheet on it. A flow rate of 0.8 mL/h set electrospun, 13.8 kV applied voltage, 12 cm distance between needle and collector, and 45% chamber moisture.

Fabrication of Electrospun PMMA microfibers reinforced BisGMA as dental post prefabrication

The BisGMA (98.5 wt%) was mechanically mixed with initiator CQ (0.5% wt) and co-initiator DMAEMA (1 wt%) at 70 °C for 1 hour. Microfibers were cut into pieces with 25 mm × 2 mm size for a three-point bending test. BisGMA mixture was poured into Teflon mold of 25 mm × 2mm × 2 mm until a half mold was filled, then laid up PMMA microfibers on top of the mixture. BisGMA and PMMA microfibers were mixed mechanically by spatula, then continued by adding the BisGMA mixture on top until throughout mold was fulfilled. Put mylar strip over the mold, press with a glass slide, then light-cured using LED (SmarLite Focus, Dentsply Sirona, Charlotte, USA) for 20 seconds. Withdraw the glass slide and mylar strip, continue light-cured for another 20 seconds. The specimens were then removed from the mold then stored at 37 °C for 24 hours. Dental posts prefabrication containing BisGMA mixture without PMMA

microfiber were also prepared with the same steps as the control. The number of test specimens and control specimens is 6 samples each.

Morphology of PMMA microfibers

The PMMA microfibers were characterized by Scanning Electron Microscopy (SEM), with specifications as the follows: Hitachi S-4700, Tokyo, Japan, to identify the morphologies and particle size of the PMMA microfibers. The fracture surface was also observed by SEM characterization to see interfacial bonding between PMMA microfibers with resin BisGMA.

Functional group evaluation by Fourier Transform Infrared Spectroscopy (FTIR)

The PMMA microfibers specimen were ground to obtain fine powder for FTIR analysis. A ratio of 1:200 (specimen and KBr) were mixed well and compacted in stainless steel die. A spectrometer (Spectrum 100, PerkinElmer Inc., Shelton, USA) uses a spectral resolution of 4 cm⁻¹ and a range of wavenumber from 400-4000 cm⁻¹.

Flexural properties evaluation using Universal Testing Machine

The specimens and controls with n = 6 were tested in three-point bending by a universal testing machine (Lloyd Instrument, Fareham, Hampshire, UK) for flexural strength test (according to ISO 10477 standard) with a span of 20 mm and crosshead speed 1 mm/min.

The following formula was used to calculate the flexural strength (1) and modulus (2):

$$F_s = 3F_l/2bh^2 \dots\dots\dots (1)$$

$$F_y = l^3F_1/4fbh^3 \dots\dots\dots (2)$$

F is the applied load at the highest point (N); l is the span length (20 mm); b is the width of specimens; h is the thickness of the specimens; F₁ is the load at the convenient point in the straight-line portion of the trace; f is the deflection of the specimens at load F₁.

Data analysis

Statistical analysis was carried out using an unpaired t-test by KaleidaGraph 4.1 (Synergy Software, PA, USA) to ensure the statistical significance noted experimentally.

RESULTS

Morphology view of PMMA microfibers

The SEM image in Figure 1 exhibits the characteristic morphology of PMMA fibers. It was shown unaligned fiber morphology with a relatively homogeneous size. The diameter size of PMMA fibers was ranging from 1- 5 microns.

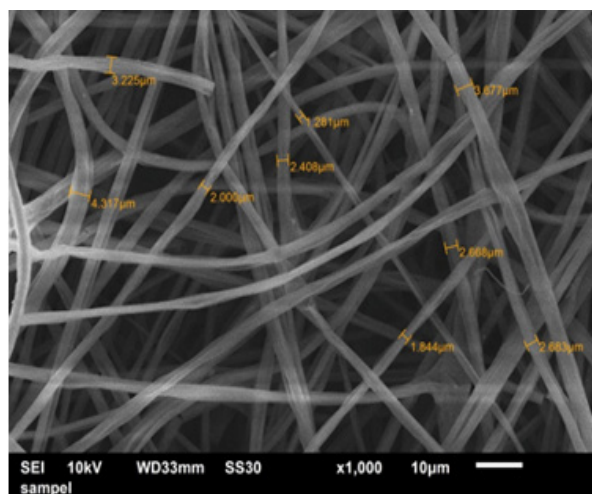


Figure 1. SEM image of the electrospun PMMA microfiber

Functional group detection of PMMA microfibers

Figure 2 shows the spectra of the PMMA microfibers by infrared spectroscopy. The peak at 2950cm⁻¹ corresponds to the stretching of the methacryloyl group (CH₃ and CH₂). The typical PMMA is shown by the presence of carbonyl group in 1722 cm⁻¹ with a strong peak. The peaks correspond to the C-O and CH₂ vibrates at 1142 and 749 cm⁻¹. At the same time, the peak of 985 cm⁻¹ attributed to the C-C group.

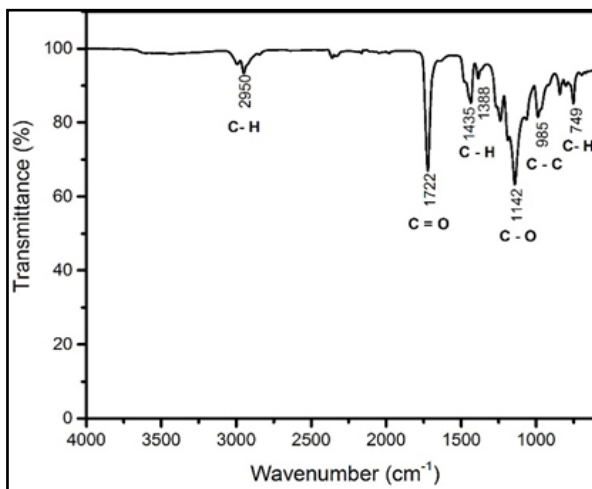


Figure 2. FTIR spectra of PMMA microfibers

Flexural properties of the dental posts with and without PMMA microfibers

Table 1 shows the data normality test of flexural strength and flexural modulus values in the dental

post fabrication group with and without PMMA. The results of the normality test show that both groups have data values that are normally distributed ($p > 0.05$) in both tests using Saphiro-Wilk Test.

Table 1. Test the normality of the two data groups on the flexural strength and flexural modulus value using the Shapiro-Wilk test

Groups	Flexural strength (MPa)*	Flexural Modulus (GPa)*
Without PMMA (n=6)	0.271	0.206
With PMMA (n=6)	0.432	0.380

Notes: * $p > 0.05$

Table 2 summarizes the flexural strength and flexural modulus of the dental post prefabrication with and without PMMA microfibers. Dental posts with PMMA microfibers showed higher flexural strength compared with the dental post without

PMMA microfibers. The PMMA microfibers increase the flexural strength of dental posts by 35.3%. However, the addition of PMMA microfibers did not show increasing in flexural modulus in the dental post.

Table 2. The flexural strength and modulus value of the sample without and with PMMA. The statistical analysis was done by unpaired t-test

Groups	Flexural strength (MPa)*	Flexural Modulus (GPa)*
Without PMMA (n=10)	61.7 ± 3.03	3.1 ± 1.0
With PMMA (n=10)	83.5 ± 10.7	5.0 ± 2.9

Notes: * $p > 0.05$

Interface evaluation of dental post with and without PMMA microfibers

Figure 3 shows the dental post's fracture surface without PMMA (a) and with PMMA microfibers (b). Dental post prefabrication without PMMA microfiber presents a smooth surface and few rough spots after fracturing. On the contrary, the

fracture surface for a dental post prefabrication with PMMA microfibers looks very rough than another group.

Although the shape in the image looks like the fiber is not very visible in the image below, this shows that the formation of chemical bonds between PMMA and BisGMA fibers.

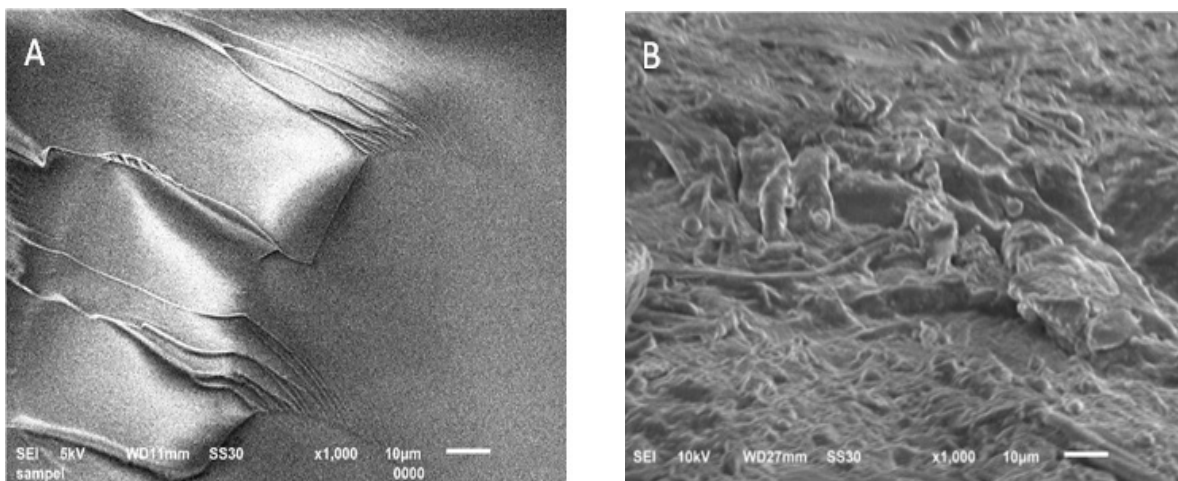


Figure 3. SEM images of the dental post's fracture surface; (A) without PMMA microfibers and (B) with PMMA microfibers

DISCUSSION

Electrospinning is commonly used to produce nano-and micron-sized fibers. This method not only controls the size but also the alignment. Ura et al. tailored 3.6 μm PMMA microfibers using a 30% PMMA concentration.¹⁰ This is similar to the concentration used in this study which is 38% then maybe that is why the size was also similar which is 1-5 μm (Fig 1). The support theory remarks that PMMA concentration affects the formation of fiber, for instance, lower concentration ($\leq 12\%$) tends to obtain nano-sized fiber, and higher concentration ($\geq 30\%$) tends to obtain micron size fiber.¹⁰

Subsequently, the surface of acquired PMMA fibers is smooth with unaligned orientation. The unaligned fiber occurs due to fluid instability during the electrospinning process. It is known as splaying or branching, which leads the jet to undergo splitting into multiple jets. This would change the shape of the jet and charge per unit area as compensation for the jet elongation and evaporation. As a result, the unstable jet will eject the smaller one from the surface of the primary jet.¹⁸

BisGMA reinforced with PMMA microfibers improved the flexural strength but not the modulus elasticity (Table 2). The elevation of flexural strength is related to the effect of the fibers. The fibers provide a high ratio of the specific area to resist flexural stress, impeding the formation of cracks. On the contrary, the flexural modulus values for both groups are similar. There are no statistical differences between those groups. It is due to flexural strength mainly relying on the fibers' strength; however, the flexural modulus mainly relies on the matrix.¹⁹ Previous studies stated that the ultrahigh specific nanofiber area is risky to decrease the flexural properties. It will create inferior impregnation, leading to air entrapment formation.

Therefore, increasing the mass fraction of fibers to further improvement will be difficult. Hinder the defect is the critical point to elevate the flexural properties. Flexural strength tends to decrease with defects increasing.¹³ The interfacial adhesion between the microfibers and the matrix was identified in Fig. 3. The SEM image shows there are no microfibers that were maintaining their configuration in the resin matrix. Although not

entirely true, the authors suspect that the PMMA fibers were partly dissolved into the resin matrix. This phenomenon is similar to the study of Cheng et al. where the PMMA-containing fiber exhibited a borderless image between the fiber and the resin matrix.¹⁴ This is due to the favorable interaction between BisGMA and PMMA by methacryloyl groups (CH_3 and CH_2) on each of their chains. The FTIR result (Fig. 2) shows the methacryloyl groups belonging to PMMA microfibers are identified at 2950cm^{-1} .

These groups play the role of forming a semi-IPN structure with resin matrix after curing.^{14,20} Based on the things above, the authors suspect the formation of a good interfacial bond between the PMMA microfiber and the resin matrix, thereby increasing the value of the flexural strength of the dental post prefabrication. Since the microfibers are strongly bonded to the resin matrix, the frictional force arises following stress transfer across the matrix, increasing the flexural strength.^{13,14,20}

This study shows that the type of fiber and fiber-matrix interfacial bond, PMMA, can increase the flexural strength of dental posts prefabrication, but the effect of size and configuration of PMMA fiber cannot be clearly identified because further research is needed. The results of the study show that PMMA polymer has the potential as a material used as a reinforcement material for dental posts prefabrication.

CONCLUSIONS

The PMMA microfibers can intimately adhere to the BisGMA mixture thus significantly improving the flexural strength of the dental post prefabrication. However, PMMA microfibers do not increase the flexural modulus where this property is more influenced by the resin matrix used.

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