



Medical Applications of Poly Methyl Methacrylate Nanocomposites

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Abstract

Hybrid materials, which consist of organic–inorganic materials, are of profound interest owing to their unexpected synergistically derived properties. Aluminium oxide (Al_2O_3) nanoparticles/polymer composites have been produced using a one-system polymer synthesis. The linear polymer, poly (methyl methacrylate) (PMMA, MW = 15,000 g/mol) and polymethacrylic acid (PMAA) are applied for the stabilization of Al_2O_3 nanoparticles. The Fourier transfer infrared (FT-IR) analysis data and scanning electron microscopy (SEM) image reveal that the core shell structure of Al_2O_3 /PMMA or Al_2O_3 /PMMA/PMAA nanocomposites have been synthesized. The ratio of concentration of the capping polymer material to the concentration of the Al_2O_3 precursor could control the size of Al_2O_3 nanoparticles. With specific concentration of the reductant, the core–shell nanostructure could be fluctuated in order.

Keywords: Poly methyl methacrylate; Poly methacrylic acid; Aluminium oxide nanoparticles; Denture materials

Introduction

Denture stomatitis is a common form of oral candidiasis, which is associated with the adherence of *Candida albicans* to denture base surfaces [1-4]. *Candida* is a commensal organism that is frequently present in healthy individuals. Introduction of predisposing factors such as systemic disease, immunosuppressive drugs, xerostomia, or dentures result in fungal infections [5,6]. Candidiasis has been associated with increased numbers of *C. albicans* particularly on the tissue fitting surface of maxillary complete dentures. Maxillary denture wearers are more susceptible to *Candida* infections because the denture base serves as an effective reservoir harboring microorganisms. Low salivary flow rates, low buffering capacities, and low pH values under dentures contribute to colonization of the oral mucosa and denture surfaces by *Candida* [7-12]. Development of pathogenesis is preceded by the initial attachment of *Candida* on the palatal mucosa and mucosal surface of the denture.

Surface characteristics resulting from chemistry are significant in the initial adherence of *Candida* to the denture resin and offer an opportunity for further bonding and colonization [13-15]. Understanding the effect of electrostatic interaction in the adhesion of *C. albicans* to poly(methyl methacrylate) (PMMA), our previous research supported the hypothesis that negatively charged denture base materials can prevent adhesion of *C. albicans* and reduce the development of denture-induced stomatitis [16].

PMMA is the resin of choice for fabrication of denture bases in clinical dentistry. It has excellent physical properties and a clearly defined polymerization process that is easy for modification. Many attempts have been made to modify PMMA taking advantage of the broad scope of modification available in polymer chemistry. In a previous study [17], the experimental resin had synthesized by copolymerization of methacrylic acid to methyl methacrylate (MMA). Results showed that the adhesion of *C. albicans* significantly decreased as the ratio of methacrylic acid increased *in vitro*. A significant decrease in candida adhesion to the resin samples existed when the methacrylic acid was present at 10 % of the modified PMMA. An optimized resin material should exhibit a positive biologic response while maintaining the desired physical properties. Physical and mechanical properties of polymers are crucial in achieving clinical success and longevity of complete dentures fabricated. Important physical properties include the following: compressive and tensile strengths, elongation, hardness, thermal characteristics, molding properties, polymerization shrinkage, solubility, dimensional stability, and dimensional accuracy [18]. One of the most crucial characteristics of a denture base resin is strength. The denture base must be able to withstand high impact forces in addition to normal masticatory forces. The main aim of this study was to investigate a new surface-modified PMMA in terms of transverse strength, transverse deflection, flexural strength, and modulus of elasticity for its application as a denture base [19].

Methods

Figure 1 illustrates the micrographs of the obtained composites revealing that their production was successfully achieved yielding materials with particles well dispersed within the matrices. Results show the micrograph of virgin polymers and it can be seen that the distribution of size is not uniform and the particles size varies. They range from 3 to 12 μm in size and their chain formation is clearly visible from the micrograph. The virgin polymer also exhibits porous nature while the pores disappear in the composite structure. This result illustrates that the nanoparticles are intercalated into the structure of the polymer. In Figure 1, the SEM image shows Al_2O_3 nanoparticles on the surface of the copolymer surface.

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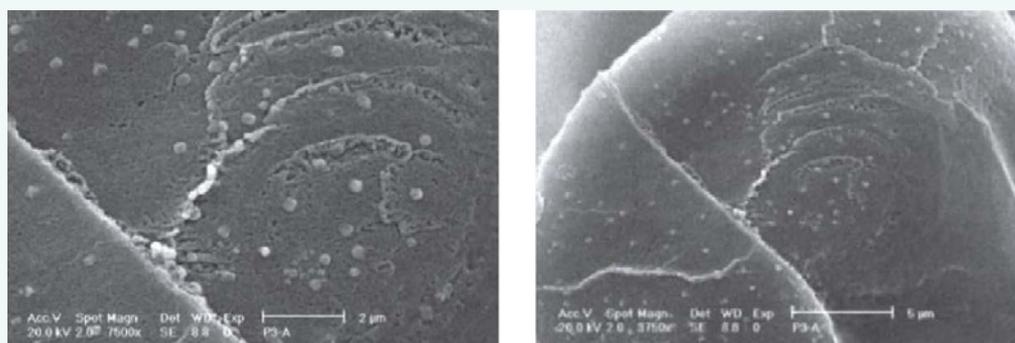


Figure 1 SEM of PMMA/ Al₂O₃ nanocomposites.

Additional evidence for the particle coating was provided by FT-IR analysis. After washing with ethanol to remove most of the free PMMA polymer, a small amount of associated PMMA might remain. Figure 2 shows a FT-IR spectrum of a nanocomposite that was prepared from Al₂O₃ and PMMA isolated. The vibration bond of carbonyl ($\nu_{C=O}$) at 1780 /cm is characteristic of the PMMA branches.

Figure 3 illustrates the diffractograms of PMMA and PMMA/Al₂O₃ nanocomposites in the 2 θ range between 5° and 90°, which are similar and without any sharp diffraction peaks confirming their non-crystalline nature. PMMA is known to be an amorphous polymer and shows three broad peaks at 2 θ values of 21°, 27°, and 29° (d spacing around 4Å, 2.94 Å, and 2.79 Å), with their intensity decreasing systematically.

A representation of the difference in mean transverse strength is shown in Figure 4. The PMMA with 5 % Al₂O₃ nanoparticles group showed the highest mean force required to fracture the specimens. A comparison of mean transverse strength revealed no significant difference between the control group and the PMMA with 5 % Al₂O₃ nanoparticles group.

The PMMA with 20 % Al₂O₃ nanoparticles group showed a decrease in transverse strength that was statistically significant compared with the PMMA with 5 % Al₂O₃ nanoparticles group. The transverse deflection measurements and the mean values are shown in Figure 5. The higher the reflection of the specimen was, the farther the crosshead needed to travel to fracture the specimen. In materials with similar transverse strength, the material with higher transverse deflection is more flexible. Results showed that as the amount of MMA increased, the transverse deflection decreased, indicating a decrease in its flexibility. A comparison of mean transverse deflection revealed significant differences between the control group and all groups except the PMMA with 5 % Al₂O₃ nanoparticles.

Figure 6 shows the mean and standard deviation values for flexible strength for each experimental group. The higher the load or force required to fracture the specimens, the higher the fracture resistance. As the ratio of MMA/Al₂O₃ nanoparticles increased, the transverse deflection decreased, indicating a decrease in its flexibility. A comparison of mean transverse deflection revealed significant differences between the control

group and all groups except the 5% PMMA group.

Figure 7 shows the mean and standard deviation values for Young's modulus of elasticity for each of the experimental groups. The elastic modulus is a measure of the stiffness of the material. The higher the elastic modulus is, the more the material will exhibit a lower elastic deformation per unit of stress applied. A comparison between the mean modulus of elasticity of the control group and the 5% PMMA group revealed no significant difference.

The 20 % PMMA group exhibited the lowest modulus of elasticity, which was significantly lower than both the 5 % group and the commercially available dental resin group. After drying at 37°C for 48 h, the mean diameter of the dried nanoparticles was determined by a sieving method using USP standard sieves. Observations are recorded (Table 1).

The hydrophilic Al₂O₃ nanoparticles on the surface of the copolymer, hydrophilic due to the hydroxyl groups on the Al₂O₃ combined with inherent surface roughness impart hydrophilic nature, according to Cassie's equation. During the reaction, the hydrophilic Al₂O₃ particles migrated to the polymer water interface due to Van der Waal's attraction.

The micrograph shows a distribution of two groups of approximately 1-2 μm and 0.5 μm Al₂O₃ particles, which are spherical in shape. The crystallinity of the formed nanocomposites was followed with XRD as a function of wt% Al₂O₃ nanoparticles added. The XRD data of composite containing variable amounts of Al₂O₃ nanoparticles percentage are shown in Figure 3. It was observed that the PMMA component was semi crystalline, whereas the Al₂O₃ nanoparticles phase was, with no discernable peaks, amorphous. It appeared that even at very low Al₂O₃ nanoparticles additions, a slight decrease in the degree of PMMA crystallinity occurred. The interlayer spacing of the system was determined by the diffraction peak in the X-ray method, using the Bragg equation:

$$\lambda = 2d \sin \theta$$

where d is the spacing between diffractive lattice planes, θ the diffraction position, and λ the wavelength of the X-ray (1.5405 Å). The shape of the first most intense peak reflects the ordered

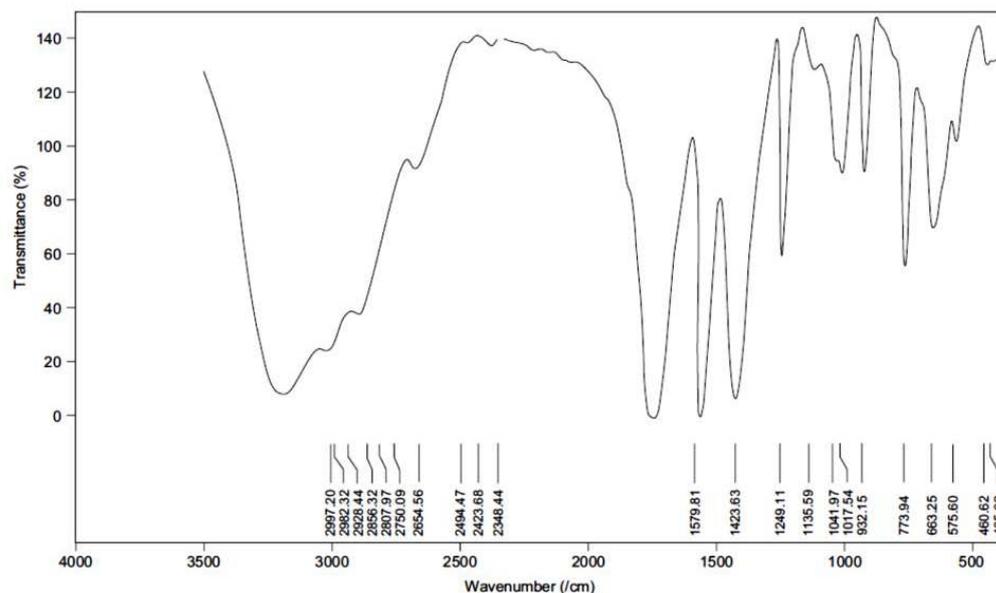


Figure 2 FT-IR spectra of PMMA/ Al₂O₃ nanocomposites.

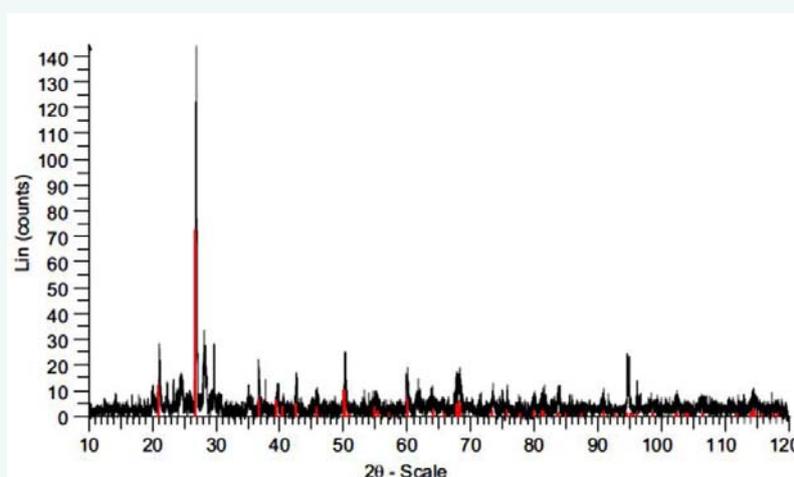
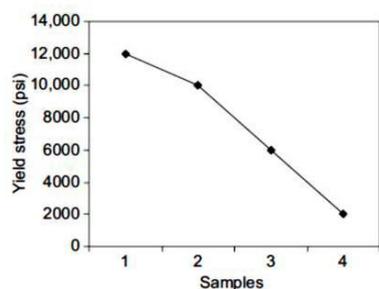


Figure 3 XRD pattern of (A) PMMA (red), (B) PMMA- Al₂O₃ nanocomposite (black).

packing of polymer chains, whereas the second peak denotes the ordering inside the main chains. The addition of Al₂O₃ nanoparticles does not induce any crystallinity in these polymers. This also explains the homogeneous nature of these samples.

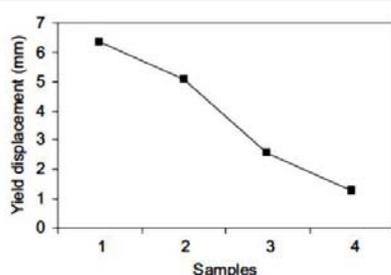
It is clear from the comparison of this spectrum with the free PMMA spectrum that PMMA is present in appreciable quantity of the composite material. The interaction between PMMA and the Al₂O₃ surface is probably due to a hydrophobic interaction. The PMMA polymer exhibits hydrophobic characteristics. Polymers allow good interactions both with the Al₂O₃ surface and the Al₂O₃ precursor for obtaining stable colloids. In the present study, in the absence of PMMA/PMAA copolymer, precipitation occurs immediately or shortly.

In the present study, the greatest decrease in transverse and flexural strengths occurred when the ratio of Al₂O₃ nanoparticles content was increased from 5 % to 10 %. Interestingly, it was also between these two groups that the most significant reduction in adhesion of *C. albicans* to resin surfaces decreased; however, the physical properties declined as a consequence. The PMMA with 5% Al₂O₃ nanoparticles was comparable to the control (dental resin) group and did not exhibit any significant difference in any parameter tested. The PMMA with 5% Al₂O₃ nanoparticles group produced a higher transverse strength and modulus of elasticity than the dental resin group; however, it was not statistically significant. This could be attributed to the method of fabrication of the modified resin samples. The experimental resins were not optimized for dental use, whereas the dental



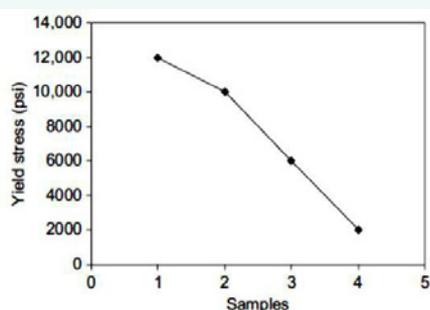
Group	Group 1 (control)	Group 2 (5% PMMA)	Group 3 (10% PMMA)	Group 4 (20% PMMA)
Mean	12.00	10.03	5.96	1.98
SD	2.94	2.65	2.31	1.40

Figure 4 The bar graph represents the mean and standard deviation values for transverse strength of force at fraction for each of the experimental groups.



Group	Group 1 (control)	Group 2 (5% PMMA)	Group 3 (10% PMMA)	Group 4 (20% PMMA)
Mean	6.38	5.32	2.30	1.63
SD	2.94	2.65	2.31	1.40

Figure 5 The mean and standard deviation values for transverse deflection for each of the experimental groups.



Group	Group 1 (control)	Group 2 (5% PMMA)	Group 3 (10% PMMA)	Group 4 (20% PMMA)
Mean	12,000.3	10,546.2	6012.5	2431.8
SD	2.94	2.65	2.31	1.40

Figure 6 Representation of mean and standard deviation values for flexible strength for each of the experimental groups.

resin group has been produced specifically to enhance these physical characteristics. In the present study, polymerization of MMA with Al_2O_3 nanoparticles produced a copolymer. Further modifications might be needed for the modified resins to improve its physical properties while still exhibiting its beneficial antifungal characteristics. A range of methods have been reported for improving the strength of resin through chemical modification of PMMA and through incorporation of fibers, such as carbon, glass, and polyethylene [20-23]. High-impact acrylic is produced from the incorporation of butadiene styrene rubber into the beads during polymerization. Rubber graft copolymers obtained from this process can improve the impact strength of the denture base by as much as 50% [24]. These resins use a monomer that contains little to no crosslinking agent. Normally, crosslinkers are said to provide the craze resistance in a denture base. Fiber reinforcement has also been shown to be effective in improving flexural strength of PMMA. Effective fiber reinforcement is dependent on many variables including the fiber type, number, distribution, and orientation. However, concerns about the possible increased adherence of *C. albicans* to fiber-reinforced denture resin bases have been raised. Studies suggest that exposed fibers can increase surface roughness and provide mechanical retention *in vivo* [25-28].

- (i) We have studied that a nanometer PMMA copolymer network could be formed by Al_2O_3 nanoparticles as a template system. The present study is significant for several reassembly of Al_2O_3 nanoparticles within the self-assembly of block PMMA copolymers in organic solvent;
- (ii) assemblies of nanoparticles within a polymer matrix, with spatial confinement at the nanometer scale; and
- (iii) employment of the *in situ* synthesis strategy for the synthesis of organic-inorganic hybrid nanonetwork structure.

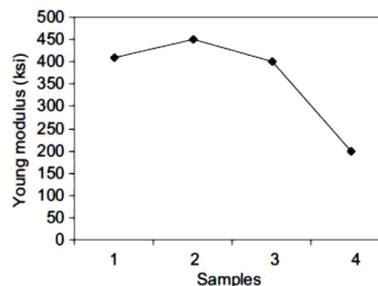
Controlling the surface properties of nanomaterials is a major technological research area encompassing studies in the pharmaceutical, mining, semiconductor, biological, and medical fields. This study demonstrates a method to generate network structures and represents a powerful and general strategy for highly functional materials. In the other hand, Hybrid materials, which consist of organic-inorganic materials, are of profound interest owing to their unexpected synergistically derived properties. Aluminium oxide (Al_2O_3) nanoparticles/polymer composites have been produced using a one-system polymer synthesis. The linear polymer, poly (methyl methacrylate) (PMMA, MW = 15,000g/mol) and poly methacrylic acid (PMAA) are applied for the stabilization of Al_2O_3 nanoparticles. The Fourier transfer infrared (FT-IR) analysis data and scanning electron microscopy (SEM) image reveal that the core shell structure of Al_2O_3 /PMMA/PMAA nanocomposites have been synthesized. The ratio of concentration of the capping polymer material to the concentration of the Al_2O_3 precursor could control the size of Al_2O_3 nanoparticles. With specific concentration of the reductant, the core-shell nanostructure could be fluctuated in order.



PMMA is the resin of choice for fabrication of denture bases on clinical dentistry. It has excellent physical properties and clearly defined polymerization process that is easy for modification. Many attempts have been made to modify PMMA taking advantage of the broad scope of modification available in polymer chemistry. In the previous study, the experimental resin had synthesized by copolymerization of methacrylic acid to methyl methacrylate. Results showed that the adhesion of *C. albicans* decreased significantly as the ratio of methacrylic acid increased in vitro. A significant decrease in Candidal adhesion to the resin samples existed when the methacrylic acid was present at 10% of the modified PMMA. An optimized resin material should exhibit a positive biologic response while maintaining the desired physical properties. Physical and mechanical properties of polymers are crucial in achieving clinical success and longevity of complete dentures fabricated. Important physical properties include the following: compressive and tensile strengths, elongation, hardness, thermal characteristics, molding properties, polymerization shrinkage, solubility, dimensional stability, and dimensional accuracy. One of the most critical characteristics of a denture base resin is strength. The denture base must be able to withstand high impact forces in addition to normal masticatory forces. The main aim of this study was to investigate a new surface-modified PMMA in terms of transverse strength, transverse deflection, flexural strength, and modulus of elasticity for its application as denture base.

In this study, we have attempted a novel approach to overcome the limitations of existing methods. We have used two-stage sonication process in order to complete free radical polymerization and hybridization. The sonication process using power ultrasonic wave was employed to enhance nano-scale dispersion during melt mixing of monomer, polymer and Al_2O_3 . It is known that ultrasonic can initiate the radical polymerization of vinyl monomers and also it is able to carry out the controlled degradation of polymer molecules in solution. With regard to the origin of such effects, it is generally recognized that sonochemical reactions in liquid proceed by cavitation collapse of a bubble that is primarily induced by the medium. According to the ultrasonic irradiation, the present method was expected to be efficient for the breakup of the Al_2O_3 agglomerates and exfoliation of the Al_2O_3 layers to yield a useful polymer- Al_2O_3 nanocomposites. Figure 8 shows XRD patterns of nanocomposites based on PMMA/PMAA and Al_2O_3 nanoparticles. In case of specimen obtained by simple mixing without sonication, the peak position for sample was found at $2\theta = 26.84$ (corresponding d-spacing of 3.3260). However, it was also found that weak peaks appeared at higher angles than those of neat Al_2O_3 . They are not considered as the d reflection of the intercalated Al_2O_3 , since the position of peak does not correspond to two times of that for the first peak. Instead, they are considered as a shift of the characteristic peak of neat Al_2O_3 , which is mainly due to its structural instability of Al_2O_3 .

The thermal stability of each Al_2O_3 was evaluated by TGA and results are given in Figure 9. As can be expected from the figure, there is a possibility that the organic molecules may be degraded or exuded out of the gallery during melt mixing at the processing temperature. In addition, prolonged shear may also be active in



Group	Group 1 (control)	Group 2 (5% PMMA)	Group 3 (10% PMMA)	Group 4 (20% PMMA)
Mean	403.6	450.8	400.2	210.0
SD	2.94	2.65	2.31	1.40

Figure 7 The mean and standard deviation values for Young's modulus of elasticity for each of the experimental groups.

Table 1: Characterization of PMMA/ Al_2O_3 nanocomposite.

Nanocomposite Formulation (wt% of polymer)	Mean diameter (nm)	Zeta potential (Mv)
Group 1	76±10.4	34.6±2.6
Group 2	82.4±23.2	30.2±0.7
Group 3	93.7±42.1	24.3±3.0
Group 4	153±42.3	19.6±2.7

such consequence. By this reason, decrease of interlayer distance becomes inevitable. During intercalation, the polymer chains that are initially in an unconstrained environment must enter the constrained environment of the narrow Al_2O_3 interlayer, whereas the organic chains gain configurational freedom as the interlayer distance increases.

Scanning electron microscopy (SEM) was performed to examine morphology development of composites. Figure 10 shows SEM micrographs of PMMA/PMAA copolymer containing 3 wt. % Al_2O_3 nanoparticles. The dark lines in the figure correspond to the Al_2O_3 layers in the polymer matrix (bright). As shown in Figure 10, it is evident that a very fine dispersion of individual platelets was promoted during the two-stage sonication process. In this figure, SEM picture shows Al_2O_3 nanoparticles on the surface of the copolymer surface. Stress relaxation time was measured by using stress relaxation mode of ARES system.

As shown in Figure 11, relaxation times of nanocomposites were higher than those of the neat polymer, which is mainly ascribed to the phase structure of nanocomposites. Al_2O_3 layers which are uniformly dispersed in the matrix can retard the relaxation of polymer molecules.

Dynamic mechanical properties of the PMMA/PMAA copolymer containing Al_2O_3 nanoparticles are shown in Figure 12. The storage moduli were increased by the incorporation of the Al_2O_3 , and further improvement was promoted by ultrasonic irradiations, compared to those of the neat polymer. Although

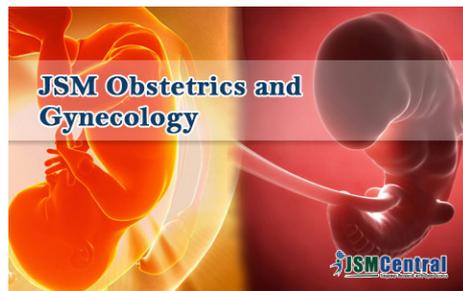
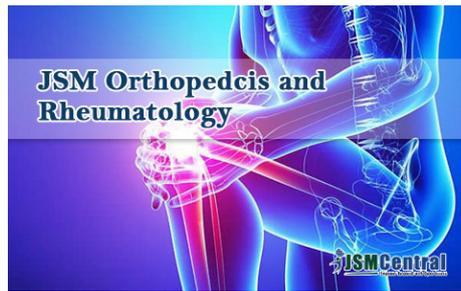


PMAA copolymer, precipitation occurs immediately or shortly. The PMMA/PMAA copolymer with 3% Al₂O₃ nanoparticles was comparable to the control (dental resin) and did not exhibit any significant difference in any parameter tested. This may be attributed to the method of fabrication of the modified resin samples. The experimental resin was not optimized for dental use, whereas the dental resin has been produced specifically to enhance these physical characteristics. In the present study, polymerization of MMA/MAA with Al₂O₃ nanoparticles produced a copolymer.

Further modifications may be needed for the modified resins to improve its physical and rheological properties while still exhibiting its beneficial antifungal characteristics. A range of methods have been reported for improving the strength of resin through chemical modification of PMMA/PMAA and through incorporation of fibers, such as carbon, glass, and polyethylene. High impact acrylic is produced from the incorporation of butadiene styrene rubber into the beads during polymerization. Rubber graft copolymers obtained from this process can improve the impact strength of the denture base by as much as 50%. These resins use a monomer that contains little to no cross-linking agent. Normally, crosslinkers are said to provide the craze resistance in a denture base. Fiber reinforcement has also been shown to be effective in improving flexural strength of PMMA/PMAA. Effective fiber reinforcement is dependent on many variables including the fiber type, number, distribution, and orientation. However, concerns about the possible increased adherence of *C. albicans* to fiber-reinforced denture resin bases have been raised. Studies suggest that exposed fibers may increase surface roughness and provide mechanical retention *in vivo*.

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