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IP Annals of Prosthodontics and Restorative Dentistry

Journal homepage: https://www.aprd.in/

Original Research Article

Comparative evaluation of flexural strength of heat polymerized polymethyl methacrylate (PMMA) denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles – An in vitro study

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ARTICLE INFO

Article history:
Received 30-05-2024
Accepted 18-06-2024
Available online 16-08-2024

Keywords:
Denture base resin
Denture reinforcement
Nonoparticles

ABSTRACT

Introduction: PMMA has been the material most frequently utilized to create complete dentures. Due to its biocompatibility, stability in the oral environment, favourable operating properties, processing ease, it has become the material of choice. However, it is still far from ideal in terms of meeting the mechanical criteria of the denture base material due to some limitations such weak mechanical strength, low fatigue strength, brittleness, etc. Zirconia has a physical characteristic called as transformation toughening, which accounts for its high flexural strength and fracture toughness. Zirconia's biocompatibility has also been thoroughly investigated. Inorganic nanoparticles being added to PMMA to enhance its characteristics has received a lot of interest.

Aim: To evaluate and compare the flexural strength of heat polymerized polymethyl methacrylate (PMMA) denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles.

Materials and Methods : Metal dies were fabricated to prepare moulds for the fabrication of heat polymerized PMMA denture base material specimens. Three brass metal dies of dimension 65 mm in length, 10 mm in width, and 3 mm in height (65×10×3) were fabricated. These specimens were reinforced with different percentages of silanized zirconium silicate nanoparticles. The polymerized specimens were carefully removed and specimens with defects were discarded. Finishing of the specimens was done using sandpaper and the finished specimens were stored in distilled water for 1 week at room temperature.

Results: When flexural strength was compared between Group 1 (control) and Group 2 (1.5%) (silanized zirconium silicate nanoparticle) ($p = 0.004$), there was a significant statistical difference ($p < 0.05$). Additionally, Group 2 (1.5% silanized zirconium silicate nanoparticle) and Group 6 (4% silanized zirconium silicate nanoparticle) showed a statistically significant difference ($p < 0.05$) ($p = 0.029$). However, there was no discernible statistically significant difference ($p > 0.05$) between the remaining Groups.

Conclusion: Specimens with reinforcement increased the flexural strength. Reinforcement with 1.5% of silanized zirconium silicate nanoparticles showed statistically significant increase in flexural strength.

1. Introduction

Edentulism is the result of loss of all permanent teeth. Tooth loss is regarded as mutilating, terminal outcome of a multifactorial process involving biologic processes (caries, periodontal diseases, pulpal pathology, trauma, oral cancer) as well as nonbiological factors related to dental procedures (access to care, patient's preferences, treatment options etc.).[1]

Even though over a past few decades, there has been a steady decline in the rates of tooth loss, more than one-third (33.1 %) of those aged ≥65 years are edentulous.[2] The need

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https://doi.org/10.18231/j.aprd.2024.045

and demand for complete dentures will increase over the next 2 decades as this generation matures into the upper age groups.[3]

As the missing tooth structure is replaced by artificial materials it is very important that there has to be continuous research and development in the field of dental materials.[4] Dentures are believed to have surfaced as a mode of treatment for replacing missing teeth since around 2500 BC.[5]

Since Dr. Walter Wright first introduced polymethyl methacrylate (PMMA) in 1937, PMMA has been the most popular material for making full dentures.[6] Because of its biocompatibility, stability in the oral environment, excellent aesthetics, advantageous properties, ease of processing, precise fit, and low equipment requirements for the fabrication process, it has been the material of choice.[7,8] However, because of certain drawbacks like poor mechanical strength, low fatigue strength, brittleness, poor thermal conduction and low hardness, it is still far from ideal in fulfilling the mechanical requirements of the denture base material.[9] Studies have shown that 68 % of the complete dentures fabricated, fractured within the first three years.[10] The midline fracture of a maxillary denture is most common and is often the result of flexural fatigue and deep incisal notching at the labial frenum.[11] Smith[7,12] after researching the real-world scenario surrounding denture fractures, he came to the conclusion that there are two different kinds of failures. I. Impact forces outside the mouth, such as when a denture is inadvertently dropped during cleaning, insertion, or removal. II. Inside the mouth, typically in function; this is most likely the result of a low-repeated stressor, or tiredness phenomenon.[13]

Much attention has been directed towards the incorporation of inorganic nanoparticles into PMMA to improve its properties.[14] In nature, zirconia does not occur in a pure state. It can be found in conjunction with silicate oxide with the mineral name Zircon ($ZrO2 \times SiO2$)/ Zirconium silicate or as a free oxide ($ZrO2$). Zirconia possesses strong ionic inter-atomic bonding, giving rise to its desirable material characteristics. The high flexural strength and fracture toughness of zirconia is because of a physical property known as transformation toughening. The biocompatibility of zirconia has also been extensively studied.[15] Silanes have the capacity to form bonds between inorganic particles and organic matrix, which enhances bonding, mixing, and matrix strength.[16] Due to a lack of information in the literature regarding the effects of varying percentages of silanized zirconium silicate nanoparticles on the flexural strength of heat polymerized PMMA, the purpose of this study is to compare the flexural strength of heat polymerized PMMA denture base materials reinforced with these different percentages of nanoparticles.

2. Aim

To evaluate and compare the flexural strength of heat polymerized PMMA denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles.

3. Objectives

1. To assess the flexural strength of the heat-polymerized PMMA denture base material without reinforcement;
2. To assess the flexural strength of the heat-polymerized PMMA denture base material reinforced with 1.5% silanized zirconium silicate nanoparticles;
3. To assess the flexural strength of the heat-polymerized PMMA denture base material reinforced with 2% silanized zirconium silicate nanoparticles;
4. To assess the flexural strength of the heat-polymerized PMMA denture base material reinforced with 2.5% silanized zirconium silicate nanoparticles;
5. To assess the flexural strength of the heat-polymerized PMMA denture base material reinforced with 3% silanized zirconium silicate nanoparticles;
6. To assess the flexural strength of the heat-polymerized PMMA denture base material reinforcing
7. To compare, with and without reinforcement, the flexural strength of heat-polymerized PMMA denture base material enhanced with different concentrations of silanized zirconium silicate nanoparticles.

4. Materials and Methods

Ninety specimens in all were prepared, with fifteen specimens in each group. The following groups comprised the specimens: -

a) Die preparation: Metal dies were fabricated to prepare moulds for the fabrication of heat polymerized PMMA denture base material specimens. Three brass metal dies of dimension 65 mm in length, 10 mm in width, and 3 mm in height ($65 \times 10 \times 3$) were fabricated. (ISO 1567 standard). These fabricated metal dies had a threaded hole at the centre. These holes were 5 mm in diameter and 3 mm in depth.[8] Screws were used to engage these threaded holes to facilitate easy removal of dies from the stone mold.

b) Silanization of zirconium silicate nanoparticles Zirconium silicate nanoparticles ($ZrSiO4$) weighing 25 gms were added to 175 ml of pure toluene solvent, which was then sonicated for 20 minutes using an ultrasonic probe. Then the beaker contained a magnetic stirrer. Then, 1.25 ml of silane (3-Trimethoxypropylsilyl methacrylate TMPSM) (5% wt to nano-filler) was added dropwise using a sterile syringe while being rapidly stirred. The slurry was left for two days with the beaker covered with parafilm. To remove the toluene solvent, the slurry was heated to 60°C and rotated at 150 rpm for 30 minutes. The silanated nanoparticles were then dried in a vacuum oven for 20 hours

Table 1:

Group 1	Heat-polymerized PMMA denture base specimens without reinforcement comprised the control group. (15 in total)
Group 2	PMMA denture base specimens that have been heat polymerized and augmented with 1.5% silanized zirconium silicate nanoparticles (n = 15)
Group 3	PMMA denture base specimens that have been heat polymerized and augmented with 2% silanized zirconium silicate nanoparticles (n = 15)
Group 4	Base specimens for dentures made of heat-polymerized PMMA augmented with 2.5 percent silanized zirconium silicate nanoparticles (n = 15)
Group 5	PMMA denture base specimens that have been heat polymerized and augmented with 3% silanized zirconium silicate nanoparticles (n = 15)
Group 6	PMMA denture base specimens that have been heat polymerized and augmented with 4% silanized zirconium silicate nanoparticles (n = 15)

at 60 oC, and they were then kept at room temperature until they were needed.[17] Preparation of gypsum mould for fabrication of specimens Preformed brass metal dies were used to prepare gypsum moulds.

For all samples, Trial closing was carried out after packing, which was completed during the dough stage. According to the manufacturer, final closure was carried out under a hydraulic bench press for three minutes at a pressure of 3000 psi. For one hour, the flask was kept under pressure using a clamp.[18] After that, it was submerged in room-temperature water in an acrylizer. The temperature was raised slowly up to 74^0C and was held for 2 hours. The temperature was then raised to 100^0C and was maintained for 1 hour.[19] After completion of this short curing cycle, the flask was removed from the water bath and allowed to bench cool at room temperature prior to deflasking.[19]

The polymerized specimens were carefully removed and specimens with defects were discarded. Finishing of the specimens was done using sandpaper (No. 120). The finished specimens were stored in distilled water for 1 week at room temperature.[7,20]

4.1. Testing of specimens

Flexural strength was assessed on each group's specimens. Because it replicates the kind of stress that is placed on the denture during mastication, the flexural three-point bending test is helpful for comparing the flexural strength of denture base materials.

Using the formula, flexural strength (FS) was determined. where P = load at fracture (N), I = distance between the supporting wedges (mm), b = specimen width (mm), d = specimen thickness (mm), and FS = flexural strength (N/mm2).[20]

FS = 3PI / 2bd2.

5. Results

Group 1's strength ranged from 82.16 to 105.43 MPa, with a mean of 95.7 MPa.

1. The range of the mean flexural strength for Group 2 was 88.87 to 118.54 MPa.
2. The range of the mean flexural strength in Group 3 was 95.58 to 118.70 MPa.
3. The range of the mean flexural strength for Group 4 was 77.58 to 117.58 MPa.
4. The range of the mean flexural strength in Group 5 was 85.45 to 113.50 MPa.
5. The flexural strength for Group 6 ranged from 86.54 to 111.08 MPa, with a mean of 97.49 MPa.

A graphical visualization of mean strength along with error bar is given in Graph 1. Table 2 reveals that using ANOVA F test, the mean flexural strength across groups differed significantly across six groups, as indicated by p-value =0.002 (p 0.05) was observed between rest of all groups. In order to determine, which groups contributed to overall significance, a pairwise comparison of mean strength was performed using Tukey's HSD test. Table 3 displays the individual pair-wise Tukey's post-hoc test comparison of flexural strength between groups. The comparison of flexural strength between Group 1 (control) and Group 2 (1.5%) silanized zirconium silicate nanoparticles reveals a significant statistical difference (p <0.05) (p = 0.004). Moreover, Group 2 (1.5% silanized zirconium silicate nanoparticle) and Group 6 (4% silanized zirconium silicate nanoparticle) differed statistically significantly (p 0.05) (p = 0.029). However, there was no statistically significant difference (p >0.05) found between the remaining Groups.

6. Discussion

The denture base material's chemical deterioration and wear and tear cause the acrylic resin denture base to break.[9] Any factor that exacerbates deformation of the base or alters its stress distribution will predispose the denture to fracture.[11] According to studies, heat polymerizing acrylic resins have average flexural strengths that are close to 78-92 MPa.[11] There are three ways to improve the properties of PMMA: development of an alternative material to PMMA; the chemical modification of PMMA such as by the addition of a rubber graft copolymer and the reinforcement of PMMA with other materials such as carbon fibres, glass fibres and ultra-high modulus polyethylene.[9]

Table 2:

S.No.	Specimen Preparation	Method
1.	Preparation of heat polymerized PMMA denture base specimens without reinforcement (n=15)	To prepare three specimens, 7.5 grammes of polymer powder and 3 millilitres of monomer were employed.
2.	Preparation of heat polymerized PMMA denture base specimens reinforced with 1.5% silanized zirconium silicate nanoparticles (n=15)	3 millilitres of monomer, 0.112 grammes of silanized zirconium silicate nanoparticles, and 7.388 grammes of polymer powder were used to create the three specimens.
3.	Preparation of heat polymerized PMMA denture base specimens reinforced with 2% silanized zirconium silicate nanoparticles (n=15)	Three different specimens were made using 7.35 grammes of polymer powder, 3 millilitres of monomer, and 0.15 grammes of silanized zirconium silicate nanoparticles.
4.	Preparation of heat polymerized PMMA denture base specimens reinforced with 2.5% silanized zirconium silicate nanoparticles (n=15)	Three millilitres of monomer, 0.187 grammes of silanized zirconium silicate nanoparticles, and 7.313 grammes of polymer powder were used to create the three specimens.
5.	Preparation of heat polymerized PMMA denture base specimens reinforced with 3% silanized zirconium silicate nanoparticles (n=15)	3.25 millilitres of monomer, 0.225 grammes of silanized zirconium silicate nanoparticles, and 7.275 grammes of polymer powder were used to create the three specimens.
6.	Preparation of heat polymerized PMMA denture base specimens reinforced with 4% silanized zirconium silicate nanoparticles (n=15)	Three millilitres of monomer, 0.2 grammes of silanized zirconium silicate nanoparticles, and 7.2 grammes of polymer powder were used to create the three specimens.

In 1959, Feynman introduced the concept of nanotechnology. Since that time, nanotechnology has found widespread application in a variety of fields, including the medical sciences where it is crucial for diagnosis, therapy, and regenerative medicine. An object is considered a nanomaterial if at least one of its dimensions is on the nanometer scale (approximately 1 to 100 nm).

Recent development of composite materials of great strength and low mass have made significant contributions in the field of dental material science. The incorporation of the ceramic nano-filler into the more flexible and lower thermal resistance polymer improves its stiffness and thermal stability.[21] Ceramic fillers were used for reinforcements as opposed to metal fillers because of its lower filler density.[7]

In nature Zirconia does not occur in a pure state. It has been found in conjunction with silicate oxide with the mineral name Zircon ($ZrO2 \times SiO2$)/ Zirconium silicate or as a free oxide (ZrO2) with the mineral name Baddeleyite. The inclusion of 1.5% wt zirconium silicate nano-filler resulted in a significantly substantial increase in the impact strength, transverse strength, and surface hardness, according to Kareem S.[22] A non-significant improvement in impact strength, a sizable rise in transverse strength, and a highly significant improvement in surface hardness were the effects of the addition of 1% weight ZrSiO4 nano-filler. With ZrSiO4 nano-filler at both 1% and 1.5% weight, surface roughness was seen to rise significantly. When compared to the control group, 1.5% wt ZrSiO4 nano-filler caused a highly significant decrease in water sorption and solubility, while 1% wt ZrSiO4 nano-filler caused a non-significant decrease. This new compound (ZrSiO4+PMMA), which not only amplifies the internal resistance but also significantly affects the compound's stress-strain behaviour due to the particle size and bonding interaction, was responsible for the highly significant increase in impact strength when PMMA was reinforced with zirconium silicate nanoparticles. Additionally, as forces are applied, they are passed to the nanoparticles, strengthening the impact.

Nanoparticle addition fills the empty spaces between the chains and pulls resin molecules, causing polymer chains to form more intricate network chains during the curing process, improving the transverse strength. The intrinsic hardness characteristic of the ZrSiO4 nanoparticles, which have a tetragonal crystal structure that appears like small prism-shaped structures separated or may give the impression of double pyramids connected from the bottom, may be to blame for the increase in hardness of PMMA reinforced with zirconium silicate nanoparticles. This results in very hard and heavy properties of the polymer nanocomposite. The effective distribution of nanoparticles in the resin matrix may also contribute to the increase in hardness. Nanofiller made of zirconium silicate has the feature of being insoluble in water. It decreased the water molecule's diffusivity when it was introduced into the PMMA resin matrix, which in turn reduced water sorption and solubility.[22]

Sehjpal and Sood (1989)[13] stated that reinforced PMMA with metal oxide fillers like silver, copper, aluminium not only increases the strength but also provides radiopacity to the heat polymerized denture base material. According

to a study by Ihab et al.,[23] a good dispersion of the extremely fine size of the nanoparticles caused an increase in the transverse strength with the addition of 2-5wt% ZrO2 nanoparticles. However, due to nano-ZrO2 agglomeration, raising the percentage of modified nano-ZrO2 to 7wt% decreased the impact strength and transverse strength. Therefore, the percentages of 1.5%, 2%, 2.5%, 3%, and 4% were chosen for this investigation.

The hydrophilic ionic nature of the inorganic filler particles typically causes them to exhibit high surface energy. The filler surface must be altered for better surface wetting and dispersion, which will enhance the composites' physical characteristics. Therefore, in this investigation, trimethoxysilylpropyl methacrylate (TMSPM) was used to increase the adherence of zirconium silicate nanoparticles to the resin matrix.[23] This study demonstrates that adding 1.5% of zirconium silicate nanoparticles to PMMA for reinforcement led to the greatest gain in flexural strength. These findings are in line with those of a study by Kareem S,[23] which found that the use of a 1.5% wt zirconium silicate nano-filler significantly increased the impact strength, transverse strength, and surface hardness.

Vojdani's[24] results showed that adding 2.5 weight percent Al2O3 powder significantly increased the flexural strength and hardness of heat-polymerized acrylic resin. When compared to the control group, the addition of 5–20 wt% Al2O3 considerably decreased flexural strength in their pilot investigation. A decrease in the cross-section of the load-bearing matrix; stress concentration due to too many filler particles; an increase in fillers may also change the resin's modulus of elasticity and the way cracks spread through the specimen; and trapped air and moisture may cause voids to form and incomplete wetting of the fillers by the resin are all potential explanations for a decrease in strength with an increase in percentage.

7. Conclusion

The following conclusions, given the constraints of the investigation, were reached:

1. The flexural strength of specimens with reinforcement improved;
2. The flexural strength of heat-polymerized PMMA denture base specimens reinforced with 2% (Group 3), 2.5% (Group 4), 3% (Group 5), 4% (Group 6) silanized zirconium silicate nanoparticles did not increase statistically significantly when compared to unreinforced specimens.
3. Reinforcement with 1.5% of silanized zirconium silicate nanoparticles showed statistically significant increase in flexural strength.

8. Source of Finding

None.

9. Conflict of Interest

None.

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Cite this article: Chavan R, Deshpande S, Radke U, Pande N. Comparative evaluation of flexural strength of heat polymerized polymethyl methacrylate (PMMA) denture base material reinforced with different percentages of silanized zirconium silicate nanoparticles – An in vitro study. *IP Ann Prosthodont Restor Dent* 2024;10(3):234-239.