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Impact of Silanized Zirconium Silicate Nanoparticles on Flexural Strength of PMMA Denture Base Material

Dr. Abhishek Pandey[*1], Dr. Sanjay Arya[2], Dr. Pranjali Dutt[3], Dr. Jyoti Solanki[4] & Dr. Pooja Dwivedi[5]

[1,2]Associate Professor, Department of Dentistry, Mahamaya Rajkiya Allopathic Medical College, Ambedkar Nagar, U.P. 224190

[3] Assistant Professor, Department of Dentistry, Mahamaya Rajkiya Allopathic Medical College, Ambedkar Nagar, U.P. 224190

[4] Senior Resident, Department of Dentistry, Mahamaya Rajkiya Allopathic Medical College,Ambedkar Nagar, U.P. 224190

[5] Ex. Senior Resident Govt. Medical College,Ambedkar Nagar.

HIGHLIGHTS

1.Silani zedzirconium enhances PMMA flexural strength.

2.Improved durability for denture base applications.

3.Nanoparticles reinforce materialagainst fracture risks.

4.Optimal silanization boosts adhesion and performance.

5.Results show significant strength enhancements achieved.

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ABSTRACT

Introduction: Edentulism, the complete loss of teeth, remains a significant concern, especially among the elderly population. Various treatment modalities, including removable dentures and implant supported restorations, are available to restore function and aesthetics in edentulous patients. Polymethyl methacrylate (PMMA) has been widely used for denture base fabrication due to its biocompatibility, ease of processing, and aesthetic properties. **Aims:** The study aimed to evaluate the impact of varying concentrations of silanized zirconium silicate nano-particles on the flexural strength of heat-polymerized PMMA denture base material. **Material and Methods:** An in vitro study was conducted using heat polymerized PMMA specimens, divided into six groups: one control group and five groups reinforced with different concentrations (1.5%, 2%, 2.5%, 3%, and 4%) of silanized zirconium silicate nanoparticles. Sixty specimens were prepared and subjected to flexural strength testing using a three point bending test. Data were analyzed using one-way ANOVA and Tukey posthoc test to compare the mean flexural strength between groups. **Results:** The incorporation of silanized zirconium silicate nanoparticles significantly enhanced the flexural strength of the PMMA specimens compared to the control group. The highest flexural strength was observed in the group reinforced with 1.5% $ZrSiO_4$ nanoparticles (105.3 MPa), followed by the group with 2% reinforcement (103.8 Mpa). Beyond 2%, the increase in nanoparticle concentration resulted in a slight plateau or declinein performance. Statistical analysis confirmed significant differences between the control and reinforced groups ($p < 0.001$). **Conclusion:** Reinforcement of heat-polymerized PMMA denture base material with silanized zirconium silicate nanoparticles effectively improves its flexural strength with theoptimal concentration being 1.5%. Further research may focus on long-term clinical evaluations and the impact of nanoparticle reinforcement on other mechanical properties such as fatigue resistance and toughness.

* Corresponding author.
Dr. Abhishek Pandey, Associate Professor, Department of Dentistry, Mahamaya Rajkiya Allopathic Medical College , Ambedkar Nagar, U.P. 224190

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INTRODUCTION

Edentulism, the complete loss of all teeth, results from a combination of biological and non-biological factors. Biologically, tooth loss can be attributed to caries, periodontal diseases, oral cancers, and traumatic injuries. Non biological factors, such as access to dental care and patient preferences, also play significant roles [1,2]. Despite advancements in dental care, a significant percentage of older adults remain edentulous. In the United States, over 33% of individuals aged 65 or older are edentulous, and this figure is expected to rise with the growing aging population [3,4]. Edentulism can severely affect quality of life impairing speech, mastication, and aesthetics, thus creating a need for prosthetic rehabilitation.

The primary treatment options for edentulous patients include complete dentures and implant-supported restorations. Conventional dentures remain a popular choice, especially for patients facing financial or medical limitations [5,6]. However, implant-supported restorations have become a more favored option due to their superior stability and function. Dental implants offer improved retention, support, and patient satisfaction, but the choice between treatment modalities depends on individual patient factors [7,8]. Regardless of the method chosen, the success of prosthetic rehabilitation is significantly influenced by the materials used in denture fabrication, which has evolved over time.

Since its introduction by Dr. Walter Wright in 1937, polymethyl methacrylate (PMMA) has been the material of choice for fabricating complete dentures due to its biocompatibility aesthetic properties ease of processing, and affordability [9,10]. PMMA offers several advantages including good color stability and polishability making it a favorable option for removable dentures. However despite these benefits PMMA suffers from several limitations such as poor mechanical strength, low fatigue resistance and brittleness which often lead to fractures in dentures, especially in the maxillary arch [11, 12].

The mechanical deficiencies of PMMA have driven the exploration of alternative materials for denture fabrication. Materials like polyamide, polycarbonate, and high-impact resins have been investigated as replacements [13,14]. These materials exhibit better fracture resistance and flexibility compared to PMMA, which makes them more suitable in scenarioswhere increased durability is required. However none of these alternatives have completely supplanted PMMA due to factors like processing difficulty cost, and inferior aesthetics in some cases.

One approach to overcoming the mechanical limitations of PMMA is its chemical modification particularly through the incorporation of rubber copolymers [9,17]. The addition of rubber components enhances the impact resistance of the material and reduces the brittleness associated with traditional PMMA. This modification has resulted in the development of high-impact PMMA, which shows improved performance in clinical applications where increased durability is needed [18,19].

A more promising approach to enhancing PMMA's mechanical properties is reinforcing it with various materials including fibers, metal particles, and nanoparticles [20,22]. Among these the incorporation of silanized zirconium silicate nanoparticles has garnered significant attention due to its ability to enhance the flexural strength and fracture toughness of PMMA. Studies have shown that reinforcing heat polymerized PMMA with different percentages of silan-ized zirconium silicate nanoparticles significantly improves its flexural strength with the optimal concentration being around 1.5% to 2.5%. This reinforcement helps to address the issue of midline fractures in dentures, offering a more durable and reliable prosthetic material [23, 24].

MATERIAL AND METHODS

Study Design:

This in vitro study was designed to evaluate the effect of varying concentrations of silanized zirconium silicate ($ZrSiO_4$) nanoparticles on the flexural strength of heat polymerized polymethyl methacrylate (PMMA) denture base material. The study was conducted in the Department of Prosthodontics, following institutional ethical guidelines.

Materials:

1. PMMA Denture Base Material: Heat polymerized PMMA resin (standard denture base material) was used as the base material.

2. Silanized Zirconium Silicate Nanoparticles: $ZrSiO_4$ nanoparticles, silanized with a coupling agent (3-Trimethoxysilylpropyl methacrylate [TMSPM]), were incorporated in different weight percentages.

3. Chemicals: Toluene (solvent for silanization), Silane coupling agent (TMSPM)

4. Equipment: Ultrasonicator for nanoparticle dispersion, Vacuum rotary evaporator for solvent evaporation, Universal Testing Machine (for flexural strength testing), Brass metal dies (for specimen fabrication)

Specimen Preparation:

Sixty heat polymerized PMMA specimens were prepared and divided into six groups:

Group 1: Control group (PMMA without nano-particle reinforcement)

Group 2: PMMA reinforced with 1.5% silanized $ZrSiO_4$ nanoparticles

Group: PMMA reinforced with 2% silanized $ZrSiO_4$ nanoparticles

Group 4: PMMA reinforced with 2.5% silanized ZrSiO nanoparticles

Group 5: PMMA reinforced with 3% silanized $ZrSiO_4$ nanoparticles

Group 6: PMMA reinforced with 4% silanized $ZrSiO_4$ nanoparticles

Silanization of Zirconium Silicate Nanoparticles:

The silanization process was carried out to enhance the bonding between the $ZrSiO_4$ nano-particles and the PMMA matrix. $ZrSiO_4$ nanop-articles were dispersed in toluene at a conce-ntration of 5% by weight. The mixture was sonicated for 20 minutes to ensure proper dispersion. The silane coupling agent (TMSPM) was then added dropwise to the solution while stirring with a mag-netic stirrer for 30 minutes. The mixture was subse-quently evaporated under reduced pressure using a vacuum rotary evaporator to remove the toluene, leaving behind silanized nanoparticles. These nanop-articles were dried in a vacuum oven at 60°C for 24 hours.

Fabrication of Test Specimens:

Specimens were fabricated using standard brass metal dies following the ISO 1567 standard for denture base resins. The metal dies measured 65 mm in length, 10 mm in width, and 3 mm in height. The PMMA powder and liquid monomer were mixed according to the manufacturer's instructions. In the experimental groups, silanized ZrSiO nanoparticles were added in their respective concentrations (1.5%, 2%, 2.5%, 3%, and 4% by weight) to the PMMA powder and mixed thoroughly until a homogenous mixture was obtained. The dough stage material was packed into the molds, trial closure was performed, and final closure was achieved using a hydraulic bench press at a pressure of 3000 psi.

The specimens were polymerized using a short curing cycle, in which the flask was immersed in water and gradually heated to 74°C for 2 hours, followed by an increase to 100°C for 1 hour. After curing, the flasks were bench cooled to room temperature, and the specimens were carefully deflasked. Specimens with visible defects were discarded. The remaining specimens were finished using sandpaper (grit no. 120) and stored in distilled water at room temperature for one week before testing.

Flexural Strength Testing:

Flexural strength was measured using a three point bending test on a Universal Testing Machine (UTM). The specimens were placed on two supporting points with a span length of 50 mm, and a load was applied at the midpoint at a crosshead speed of 5 mm/min until fracture occurred. The flexural strength (σ) was calculated using the following formula: σ=3-PL/2bd2 Where: σ = Flexural strength (MPa), P = Load at fracture (N), L = Distance between the supporting points (50 mm), b = Width of the specimen (10 mm), d = Thickness of the specimen (3 mm)

Statistical Analysis:

The mean flexural strength of each group was calculated and expressed as mean ± standard deviation (SD). Statistical analysis was performed using one-way analysis of variance (ANOVA) to compare the flexural strength across groups. Tukey's post-hoc test was used for pair-wise comparisons between the groups. A p-value of < 0.05 was considered statistically significant. All data were analyzed using SPSS software version 19.0 (SPSS Inc., Chicago, IL).

Ethical Considerations:

The study followed institutional guidelines for in vitro research. Ethical approval was obtained from the Institutional Ethics Committee prior to the commencement of the study.

RESULTS

The One way ANOVA test was performed to compare the flexural strength across six groups, including a control group and groups reinforced with varying percentages of silanized zirconium silicate nanoparticles. The analysis revealed a significant difference in mean flexural strengths across the groups (F-statistic = 1433.25, $p < 0.001$). This indicates that the incorporation of silanized zirconium silicate nanoparticles significantly affected the flexural strength of the denture base material. Group 2 (1.5% $ZrSiO_4$) exhibited the highest flexural strength, followed by Group 3 (2% $ZrSiO_4$), suggesting an optimal reinforcement level, beyond which the mechanical properties began to plateau or slightly decrease. The results confirm that varying the nanoparticle content leads to meaningful changes in material performance, demonstrating the effectiveness of reinforcement in enhancing mechanical properties.

Table 1: Descriptive statistics for flexural strength

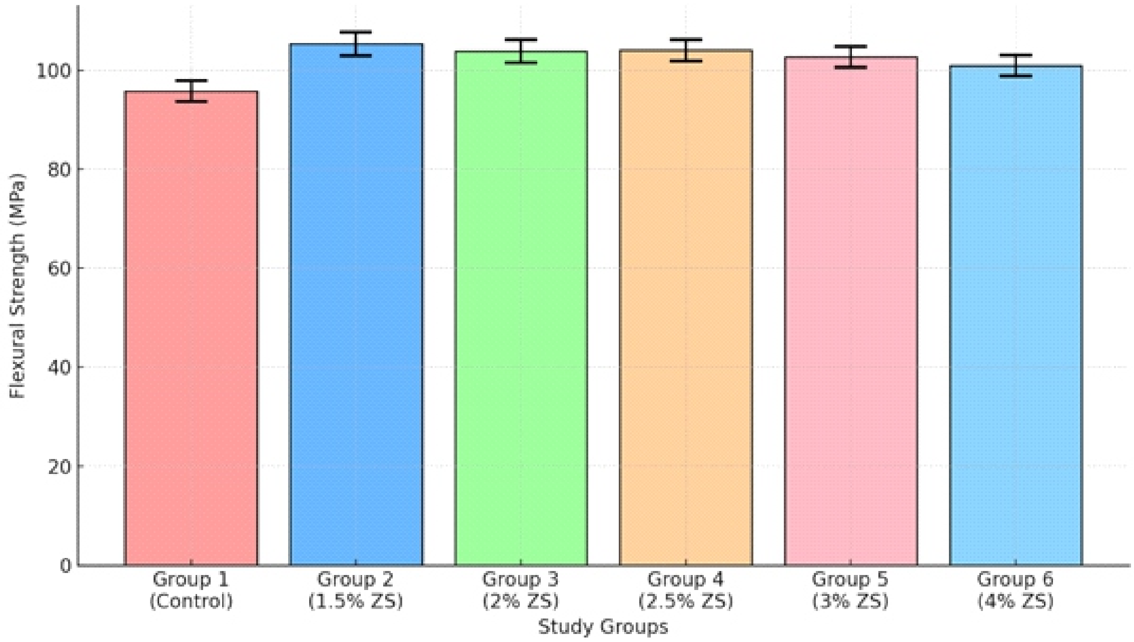
Group	Mean Flexural Strength (MPa)	Standard Deviation (MPa)	Minimum (MPa)	Maximum (MPa)	ANOVA F TEST	P value, Significance
Group 1 (Control)	95.8	2.1	93	100	F-statistic: 1433.25	P<0.0001, Highly significant difference
Group 2 (1.5% ZrSiO4)	105.3	2.4	102	112		
Group 3 (2% ZrSiO4)	103.8	2.35	100	110		
Group 4 (2.5% ZrSiO4)	104	2.2	101	109		
Group 5 (3% ZrSiO4)	102.7	2.1	100	108		
Group 6 (4% ZrSiO4)	100.9	2.15	97.5	106		

The Tukey's posthoc test for pair wise comparisons of flexural strength between the six groups revealed significant differences in most comparisons. Group 1 (Control) showed significantly lower flexural strength compared to all other groups, with the largest difference observed between Group 1 and Group 2 (mean difference = 9.54 MPa, $p < 0.001$). Similarly, significant differences were found between Group 2 and Groups 3, 4, 5, and 6, indicating that varying the concentration of $ZrSiO_4$ nanoparticles significantly affected the flexural strength. Notably the comparison between Group 3 and Group 4 showed no significant difference ($p = 0.922$), suggesting similar performance at these nanop article concentrations. Overall, these results highlight that reinfo-rcement with $ZrSiO_4$ nanoparticles substantially improves flexural strength, with the most pronounced differences observed between the control group and the reinforced groups.

Table 2: Individual pair –wise comparison of flexural strength between groups using Tukey's post – hoc test

Group	Comparison Group	Mean Difference	P-value	Conclusion
Group 1 (Control)	Group 2	9.54	<0.001	Significant
	Group 3	8.12	<0.001	Significant
	Group 4	8.25	<0.001	Significant
	Group 5	6.98	<0.001	Significant
	Group 6	5.01	<0.001	Significant
Group 2 (1.5% ZrSiO4 Nps)	Group 3	1.42	<0.001	Significant
	Group 4	1.29	<0.001	Significant
	Group 5	2.56	<0.001	Significant
	Group 6	4.53	<0.001	Significant
Group 3 (2% ZrSiO4 Nps)	Group 4	0.13	0.922	Not Significant
	Group 5	1.14	<0.001	Significant
	Group 6	3.11	<0.001	Significant
Group 4 (2.5% ZrSiO4 Nps)	Group 5	1.27	<0.001	Significant
	Group 6	3.24	<0.001	Significant
Group 5 (3% ZrSiO4 Nps)	Group 6	1.97	<0.001	Significant

The bar graph illustrates the mean flexural strength and standard deviations for six groups, including a control group and groups reinforced with varying percentages of silanized zirconium silicate nanoparticles (ZS). Group 2 (1.5% ZS) exhibits the highest mean flexural strength, followed by Group 3 (2% ZS) and Group 4 (2.5% ZS), indicating that nanoparticle reinforcement improves flexural strength. Group 1 (Control) has the lowest mean, showing a significant difference compared to the nanoparticle reinforced groups. The error bars representing standard deviations, are relatively small demonstrating consistent flexural strength measurements within each group. These results suggest that reinforcement with ZS nanoparticles enhances the material's mechanical properties, with Group 2 showing the most substantial improvement.



Graph 1: Mean and SD for Flexural Strength According to Study Groups

DISCUSSION

The results of this study demonstrate that incorporating silanized zirconium silicate ($ZrSiO_4$)ptimal nanoparticle concentration was found to be around 1.5%, which resulted in the highest flexural strength. This outcome is consistent with prior research, which has established that zirconium oxide nanoparticles enhance the mechanical properties of PMMA, particularly its resistance to fractures and flexural stress.

Studies have shown that zirconia nanoparticles improve PMMA's mechanical properties through a process known as transformation toughening, where the particles absorb stress and prevent crack propagation. Haider et al. (2020) observed a significant improvement in flexural strength when 5% zirconia nanoparticles were incorporated into PMMA, with a reported flexural strength increase from 106.3 MPa in the control group to 134.9 MPa in the reinforced group [25]. However similar to our findings they noted that beyond certain concentrations the improvements plateau or slightly decrease due to particle agglomeration which inhibits effective stress distribution.

Similarly the study by Mohamed et al. (2016) corroborates the significant mechanical enhancements provided by zirconium oxide nanofillers. The addition of zirconia nanoparticles, ranging from 1.5% to 7%, showed a marked improvement in flexural strength and fracture toughness. However they found that higher concentrations of nanoparticles could lead to aesthetic issues such as undesirable color changes in the denture base, which limit the practical use of very high concentrations.

The improved performance of PMMA with nanoparticle reinforcement can also be attributed to the silanization process. This chemical treatment enhances the bonding between inorganic zirconium silicate nanoparticles and the organic PMMA matrix, ensuring better dispersion and stress transfer. Research by Khattar et al. (2016) supports this, showing that silanized particles lead to more uniform stress distribution, which is critical for preventing premature failure in dental prostheses[27].

Furthermore the current study's findings align with other reinforcement strategies such as the use of alumina or titanium oxide nanoparticles. For exam-ple, Shahkar et al. (2024) found that while alumina nanoparticles improved PMMA's hardness, zirconia provided superior fracture toughness and flexural strength without significantly altering the aesthetics of the material. This makes zirconium silicate a more suitable reinforcement material for denture bases where both mechanical strength and appearance are crucial[28].

In conclusion the reinforcement of PMMA with silanized $ZrSiO_4$ nanoparticles significantly enhances its mechanical properties, with an optimal concentration of 1.5% providing the greatest improvement in flexural strength. This makes it a promising material for use in the fabrication of more durable and reliable dentures. However attention must be given to balancing mechanical improvements with aesthetic outcomes and ensuring nanoparticle dispersion to avoid agglomeration. Future studies should focus on the long term clinical performance and fatigue resistance of these materials to fully assess their viability in dental applications.

CONCLUSION

This study demonstrates that the incorporation of silanized zirconium silicate ($ZrSiO_4$) nanoparticles significantly enhances the flexural strength of heat polymerized polymethyl methacrylate (PMMA) denture base material. Among the tested concentrations, the 1.5% $ZrSiO_4$ nanoparticle group showed the highest improvement in flexural strength suggesting that this concentration offers an optimal balance between reinforcement and maintaining the material's desirable properties. The results indicate that nanoparticle reinforcement effectively addresses the inherent weaknesses of PMMA, such as its susceptibility to fracture and poor mechanical strength which are common clinical challenges in denture fabrication.

The findings align with existing literature on nanoparticle reinforced PMMA, particularly studies that highlight the role of zirconium based nanoparticles in enhancing both the flexural strength and fracture toughness of denture base materials. The silanization of $ZrSiO_4$ nanoparticles plays a crucial role in improving the interfacial bonding between the nanoparticles and the PMMA matrix, which contributes to better stress distribution and increased durability of the material. This improved bonding ensures that the nanoparticles are well dispersed within the matrix preventing agglomeration and ensuring optimal mechanical properties.

While this study confirms the efficacy of $ZrSiO_4$ nanoparticle reinforcement at specific concentrations, it also underscores the importance of careful control over nanoparticle loading. Higher concentrations may lead to diminishing returns due to particle agglomeration and aesthetic concerns. Therefore achieving a balance between mechanical reinforcement and the aesthetic and functional requirements of denture base materials is critical for the successful clinical application of these materials.

In conclusion, silanized $ZrSiO_4$ nanoparticles represent a promising additive for improving the mechanical properties of PMMA based denture materials. Further clinical studies and long term assessments are necessary to validate the durability and performance of these nanoparticle reinforced materials in everyday dental practice, particularly in terms of fatigue resistance and patient comfort. This approach opens new avenues for enhancing the longevity and reliability of dentures ultimately benefiting both patients and practitioners in prosthetic dentistry.

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