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Effect of Incorporation of Zirconium Oxide Nanoparticles on Thermal Conductivity and Microhardness of PMMA Heat Polymerized Acrylic Denture Base Material - An Invitro Study

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Abstract: ***Introduction: Poly (methyl methacrylate) (PMMA) is widely used in denture base fabrication due to its favourable properties. However, limitations such as low thermal conductivity, poor mechanical strength, and susceptibility to microbial adhesion necessitate modifications to improve its performance. Zirconium oxide (ZrO_2) nanoparticles have shown promise in enhancing the mechanical and thermal properties of PMMA - based denture materials. Objective: This study aimed to evaluate the effect of incorporating different concentrations of zirconium oxide nanoparticles (3%, 7%, and 10%) on the thermal conductivity and microhardness of heat - cured PMMA denture base resin. Materials And Methods: Total 80 Heat - cured PMMA samples were fabricated using stainless steel molds, following standard processing techniques. The samples were reinforced with ZrO_2 nanoparticles at varying concentrations and divided into four experimental groups. Thermal conductivity was measured using the Cussons Thermal Conductivity Apparatus, while microhardness was assessed using a Vickers Hardness Tester. Data were analyzed using one - way ANOVA and Tukey's post hoc test, with statistical significance set at $p < 0.05$. Results: The incorporation of zirconium oxide nanoparticles significantly improved the thermal conductivity and microhardness of PMMA resin. The 10% ZrO_2 group exhibited the highest microhardness and thermal conductivity values compared to the lower concentrations. However, a trade - off was observed, where increasing ZrO_2 concentration beyond 7% the rate of improvement may slow as the material percentage approaches 10% this is due to higher concentrations nanoparticles tend to agglomerate leading to uneven distribution with in the resin matrix This hinders the formation of continuous thermal pathways and reduced processability of the resin. Conclusion: Zirconium oxide nanoparticle reinforcement enhances the thermal and mechanical properties of PMMA denture base materials. The 7% ZrO_2 concentration demonstrated the optimal balance between improved properties and material workability, making it a suitable form for clinical applications. Further studies should evaluate long - term durability and biocompatibility in oral conditions.***

Keywords: PMMA, zirconium oxide nanoparticles, denture base resin, thermal conductivity, microhardness

1. Introduction

Dentures remain a widely used prosthetic solution for edentulous patients, offering high success rates. Complete dentures are commonly fabricated using polymers, precious metal alloys, and base metal alloys. Among these, Poly (methyl methacrylate) (PMMA) resins are the most preferred due to their favorable working characteristics, accurate fit, stability in the oral environment, superior aesthetics, and ease of processing with inexpensive equipment [1]. However, PMMA has limitations such as low thermal conductivity, susceptibility to mechanical fatigue, and microbial adhesion leading to denture stomatitis [2]. Researchers have explored the incorporation of various fillers like metallic particles, fibers, and nanoparticles to

enhance PMMA's mechanical properties with varying degrees of success [3]. Attempts to copolymerize PMMA with rubber materials improved impact strength but failed to prevent microbial adhesion [4]. Antifungal agents, delivered in the form of drops, lozenges, creams, or mouthwashes, have shown limited effectiveness due to rapid loss in saliva, uneven distribution, and resistance development [5].

Nanoparticle incorporation has gained attention for its antimicrobial benefits. Among various nanoparticles, silver nanoparticles (AgNPs) have demonstrated broad - spectrum antimicrobial activity. Studies have confirmed that denture base materials integrated with AgNPs exhibit antimicrobial properties against Candida albicans and Streptococcus mutans, particularly at lower concentrations [6].

A denture base with high thermal conductivity enhances patient comfort by improving taste perception and reducing the sensation of a foreign body. To enhance PMMA's thermal conductivity, materials like silver, aluminum, and copper powders have been added, but high - volume additions (25%) significantly reduce tensile strength [7]. Thermally conductive ceramics, including silicon carbide (SiC) and aluminum nitride (AlN), have emerged as superior alternatives due to their comparable thermal conductivities to metals, lightweight nature, and excellent biocompatibility [8].

Nanoparticles are widely used in material science for their wear resistance and anti - corrosion properties. Their high surface - to - volume ratios enhance mechanical properties when used as fillers. Various nanoscale fillers, including silica, calcium carbonate, alumina, zirconia (ZrO_2), titanium dioxide, zinc oxide, and hydroxyapatite nanoparticles, have been incorporated into dental polymers to improve properties [9].

Among these, zirconia (ZrO_2) nanoparticles have shown particular promise. Studies have demonstrated that ZrO_2 nanoparticle reinforcement improves the mechanical properties of PMMA denture bases, including increased flexural strength, hardness, and fracture resistance [10]. Additionally, ZrO_2 exhibits excellent biocompatibility, corrosion resistance, and thermal stability, making it a suitable additive for prosthetic applications [11]. Silicon carbide (SiC) has also been explored due to its superior thermal conductivity, hardness, and cytocompatibility. Compared to oxide ceramics, SiC is more durable due to its high covalent bonding, making it ideal for dental prostheses [12]. Similarly, alumina has been incorporated into PMMA due to its exceptional hardness, dielectric properties, heat resistance, and thermal stability [13].

This study aims to evaluate the reinforcement effect of nano - ZrO_2 on the microhardness and thermal conductivity of heat - cured acrylic denture base materials. By improving PMMA's mechanical and thermal properties through nanoparticle incorporation, this research contributes to the development of more durable and comfortable prosthetic solutions.

2. Materials and Methods

In this invitro study, Total 80 samples were prepared using denture base resin. To fabricate the acrylic samples a cylindrical stainless steel shape mould (38 mm length × 25 mm diameter) and a stainless-steel rectangular shape mould (30 mm × 10 mm × 2.5 mm) was used to create a mould space. (FIGURE 1)

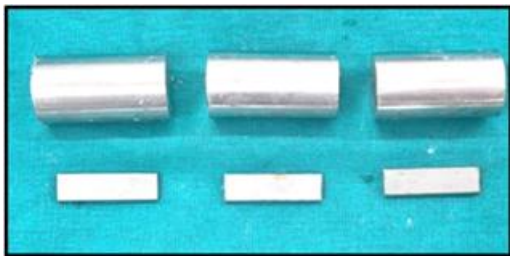

Figure 1: Stainless Steel Metal Dies

Two pour technique, was used for the flasking of wax blocks. After 15 - 20 minutes when the gypsum was completely set, it was placed in the dewaxing unit at 100°C for 5 - 7 minutes. Flask was carefully opened and clean boiling water was poured over it to completely eliminate the wax. A brush and soap solution was used to clean any traces of wax. It was allowed to cool for 10 minutes and then two layers of cold mold seal (DPI - The Bombay Burmah Trading Corporation Ltd. Cold Mold seal) was applied all over the set gypsum. Gypsum moulds were thus, obtained. Total 80 samples divided in to two groups containing 40 samples each group, Group A - thermal conductivity, Group B - microhardness with 4 subgroups in each group (10 samples in each sub group respectively)

Preparation of thermal conductivity samples as follows –

Group A1 - control group - 23 gms of poymer DPI heat cure (the bombay burmah trading corporation ltd, india) mixed with 7ml of monomer,

GROUP A2 - 3% zirconia np: 0.69 g zirconium oxide was mixed with 7 ml monomer, followed by incorporation into 22.31 g PMMA powder.

GROUP A3 - 7% zirconia np: 1.61 g zirconium oxide was mixed with 7 ml monomer, followed by incorporation into 21.39 g PMMA powder.

GROUP A4 - 10% zirconia np: 2.3 g zirconium oxide was mixed with 7ml monomer, followed by incorporation into 20.7 g PMMA powder.

Preparation of microhardness as follows

GROUP B1 - Control group - 1.785 gm of polymer DPI Heat Cure (The Bombay Burmah Trading Corporation Ltd, India) mixed with 2 ml of monomer

GROUP B2 - 3% Zirconia np: 0.05 g zirconium oxide was mixed with 2 ml monomer and 1.78 g PMMA powder.

GROUP B3 - 7% Zirconia np: 0.12 g zirconium oxide was mixed with 2 ml monomer and 1.665 g PMMA powder.

GROUP B4 - 10% Zirconia np: 0.17 g zirconium oxide was mixed with 2 ml monomer and 1.615g PMMApowder

Curing of the samples:
The flask was immersed in an acryliser at room temperature. The temperature was raised to 73°C, held for 1 ½ hours, then to 100°C and this temperature was maintained for half an hour. After the curing cycle, the flask was removed from the acryliser water - bath and bench cooled for 30 minutes, immersed in cool tap water for 15 minutes preceding the deflasking.

Finishing and polishing of samples:

The acrylic specimens were then retrieved, finished and polished. The dimension and quality of specimens were verified for any porosity, visible impurities and dimensional deformity. finally, 80 samples were prepared for testing.

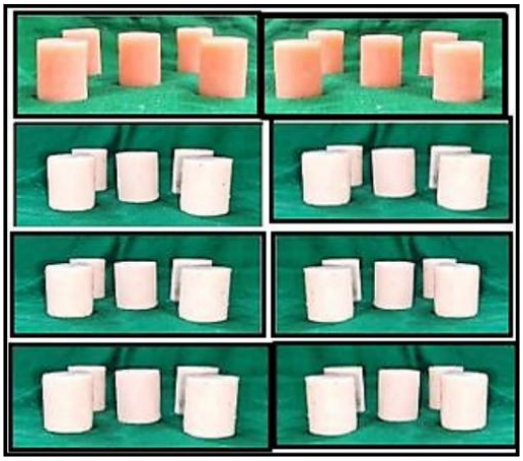

Figure 2: Finishing and Polishing of Thermal Conductivity Samples

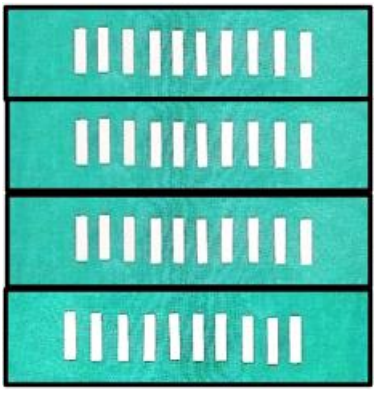

Figure 3: Finishing and Polishing of Microhardness Samples

Evaluation of Samples for thermal conductivity
Two holes were drilled at opposite ends (6.5 mm apart) of sample and thermocouples were inserted into these holes placed in cussons thermal conductivity apparatus and water flow was maintained across the sample to measure temperature differences and calculated by below formula;

K=J×L× (T2 - T1)
A× t× (T2 - T3)

where J = the 0º
mechanical equivalent of heat = 0.186 J/kcal
M = mass of water
L = the sample length
A = the surface area
T = the time of flow
T2 = the temperature of the outflowing water
T1 = the temperature of the inflowing water
t2 = the temperature of the cold end
t3 = the temperature of the warm end

Evaluation of Samples for microhardness

Microhardness was evaluated using a Vickers Hardness Tester with a diamond indenter and a 20X objective lens. Five indentations were made using a 200 g load for 10 seconds, and the average microhardness value was calculated.

Statistical Analysis

Descriptive statistics were used to summarize data. One - way ANOVA was performed to determine differences between groups, followed by post hoc analysis using the Tukey HSD test for multiple comparisons. Statistical significance was set at $p < 0.05$.

3. Results

This study evaluated the influence of different material compositions (Control, 3%, 7%, and 10% Groups) on micro hardness and thermal conductivity. The findings demonstrate a progressive increase in both parameters with increasing concentration. Statistical analysis using ANOVA and Tukey post hoc tests confirmed significant differences across the groups ($p \leq 0.001$), indicating that material modifications substantially impact mechanical and thermal properties. The descriptive statistics further support these trends, with narrow confidence intervals ensuring precision. The mean micro hardness increased across the groups, from 20.97 (Control) to 27.96 (10% Group), indicating a significant enhancement with increasing percentage. Standard deviation values suggest consistent data distribution, with a narrow confidence interval (CI) confirming the precision of measurements. Thermal conductivity followed a similar trend, rising from 2.65 (Control) to 5.66 (10% Group). Standard errors were relatively low, ensuring measurement reliability, and the 95% CI values further support statistical accuracy. Both parameters exhibit a progressive increase across the groups, highlighting a strong correlation between material composition and mechanical/thermal properties in table 1 and 2.

Table 1: Descriptive Statistics for Micro Hardness Across Different Groups

Statistic	Control Group (n=10)	3% Group (n=10)	7% Group (n=10)	10% Group (n=10)
Mean	20.9710	24.4530	26.3120	27.96
Std. Deviation	0.15044	0.21292	0.26832	0.247
Std. Error	0.04757	0.06733	0.08485	0.078
95% CI (Lower Bound)	20.8634	24.3007	26.1201	27.78

95% CI (Upper Bound)	21.0786	24.6053	26.5039	28.13
Minimum	20.77	24.20	25.90	27.57
Maximum	21.23	24.77	26.67	28.37

Table 2: Descriptive Statistics of Thermal Conductivity (W/mK) Across Groups

Parameter	Control Group	3% Group	7% Group	10% Group
Mean	2.650	3.620	4.350	5.660
Standard Deviation	0.1581	0.1932	0.2068	0.1713
Standard Error	0.0500	0.0611	0.0654	0.0542
95% CI (Lower Bound)	2.537	3.482	4.202	5.537
95% CI (Upper Bound)	2.763	3.758	4.498	5.783
Minimum	2.4	3.3	4.0	5.4
Maximum	2.9	3.9	4.6	5.9

The table 3 presents the One - Way ANOVA results for micro hardness and thermal conductivity, comparing the four groups. The statistically significant p - values (≤0.001) indicate a strong influence of material composition on both properties. The high F - values (1789.908 for micro hardness and 478.552 for thermal conductivity) suggest a substantial variation between groups, confirming that increasing material concentration significantly enhances mechanical and thermal characteristics.

Table 3: One - Way ANOVA for Micro Hardness and Thermal Conductivity (W/mK)

Source of Variation	Sum of Squares	df	Mean Square	F - Value	p - value
Micro Hardness					
Between Groups	270.040	3	90.013	1789.908	≤0.001*
Within Groups	1.810	36	0.050		
Total	271.851	39			
Thermal Conductivity (W/mK)					
Between Groups	48.254	3	16.085	478.552	≤0.001*
Within Groups	1.210	36	0.034		
Total	49.464	39			

Table 4 shows the Tukey HSD test results indicate significant differences in micro hardness between all groups ($p \leq 0.001$). The mean differences show a progressive increase in micro hardness as material concentration increases. The highest difference is observed between the Control vs.10% Group (- 6.900, $p \leq 0.001$), confirming that higher material concentration enhances hardness.

Table 4: Post Hoc Comparisons (Tukey HSD Test) for microhardness

Group Comparison (I – J)	(I - J)	Std. Error	p - value	95% CI (Lower – Upper)
Control vs 3% Group	- 3.480*	0.10029	≤0.001 *	- 3.7521 to - 3.2119
Control vs 7% Group	- 5.340*	0.10029	≤0.001 *	- 5.6111 to - 5.0709
Control vs 10% Group	- 6.900*	0.10029	≤0.001 *	- 7.2611 to - 6.7209
3% Group vs Control	3.4820*	0.10029	≤0.001 *	3.2119 to 3.7521
3% Group vs 7% Group	- 1.850*	0.10029	≤0.001 *	- 2.1291 to - 1.5889
3% Group vs 10% Group	- 3.500*	0.10029	≤0.001 *	- 3.7791 to - 3.2389
7% Group vs Control	5.340*	0.10029	≤0.001 *	5.0709 to 5.6111
7% Group vs 3% Group	1.850*	0.10029	≤0.001 *	1.5889 to 2.1291
7% Group vs 10% Group	- 1.650*	0.10029	≤0.001 *	- 1.9201 to - 1.3799
10% Group vs Control	6.900*	0.10029	≤0.001 *	6.7209 to 7.2611
10% Group vs 3% Group	3.50*	0.10029	≤0.001 *	3.2389 to 3.7791
10% Group vs 7% Group	1.650*	0.10029	≤0.001 *	1.3799 to 1.9201

Table 5 showed the post hoc comparisons for thermal conductivity show statistically significant differences ($p \leq 0.001$) between all groups. The mean thermal conductivity increases with material concentration, with the largest difference between the Control vs.10% Group (- 3.010, $p \leq 0.001$), suggesting a strong positive correlation between material percentage and thermal conductivity. These findings reinforce that increasing material concentration significantly improves both micro hardness and thermal properties of the material.

Table 5: Multiple Comparisons Across Different Groups (Tukey HSD Test) for thermal conductivity

Comparison (I - J)	(I - J)	Std. Error	Sig.	95% Confidence Interval
Control vs 3% Group	- 0.9700*	0.0820	0.000	- 1.199 to - 0.741
Control vs 7% Group	- 1.7000*	0.0820	0.000	- 1.929 to - 1.471
Control vs 10% Group	- 3.0100*	0.0820	0.000	- 3.239 to - 2.781
3% Group vs Control	0.9700*	0.0820	0.000	0.741 to 1.199
3% Group vs 7% Group	- 0.7300*	0.0820	0.000	- 0.959 to - 0.501
3% Group vs 10% Group	- 2.0400*	0.0820	0.000	- 2.269 to - 1.811
7% Group vs Control	1.7000*	0.0820	0.000	1.471 to 1.929
7% Group vs 3% Group	0.7300*	0.0820	0.000	0.501 to 0.959
7% Group vs 10% Group	- 1.3100*	0.0820	0.000	- 1.539 to - 1.081
10% Group vs Control	3.0100*	0.0820	0.000	2.781 to 3.239

10% Group vs 3% Group	2.0400*	0.0820	0.000	1.811 to 2.269
10% Group vs 7% Group	1.3100*	0.0820	0.000	1.081 to 1.539

Table 6 presents homogeneous subsets for micro hardness and thermal conductivity across different material concentrations. The results indicate distinct progressive subsets, showing a significant increase in micro hardness as material concentration rises. The control group has the lowest hardness (20.9710), while the 10% group has the highest (27.9620), reinforcing the trend that increasing material concentration enhances hardness. Similarly, the thermal conductivity values form separate homogeneous subsets, indicating significant differences between each group. The control group has the lowest conductivity (2.650 W/mK), whereas the 10% group has the highest (5.660 W/mK), suggesting a direct relationship between material concentration and thermal conductivity. These findings confirm that both micro hardnessthermal conductivity increase significantly with higher material concentrations, supporting the material's enhanced mechanical and thermal properties.

Table 6: Homogeneous Subsets for Micro Hardness & Thermal Conductivity (Tukey HSD Test)

Group	N	Micro Hardness (Subset for α = 0.05)	Mean Thermal Conductivity (W/mK) (Subset for α = 0.05)
Control	10	20.9710	2.650 (Subset 1)
3% Group	10	24.4530	3.620 (Subset 2)
7% Group	10	26.3120	4.350 (Subset 3)
10% Group	10	27.9620	5.660 (Subset 4)

Note: *The mean difference is significant at the 0.05 level.*

4. Discussion

Polymethyl methacrylate (PMMA) remains the most commonly used denture base material due to its favorable properties. However, its inherent limitations, such as low thermal conductivity, insufficient wear resistance, and susceptibility to microbial adhesion, have prompted extensive research into material modifications. Various studies have explored the incorporation of fillers such as metallic particles, fibers, and nanoparticles, with varying degrees of success [2]. Additionally, efforts have been made to copolymerize PMMA with rubber materials to improve impact strength. While these modifications have shown promise, microbial adhesion remains a significant concern, contributing to the development of denture stomatitis [15].

Among the various antimicrobial agents explored, silver nanoparticles (AgNPs) have received particular attention due to their broad - spectrum antimicrobial activity [44]. evaluated the antimicrobial effects and flexural strength of heat - cure denture base materials incorporated with different concentrations of AgNPs. Their findings indicated that AgNPs effectively inhibited *Candida albicans* and *Streptococcus mutans*, especially at lower concentrations. However, the effect of AgNP incorporation on the mechanical properties of PMMA - based denture base materials remain inconclusive [15].

Surface hardness is a crucial mechanical property, as it determines the material's resistance to abrasion during mastication and denture cleaning. Poor wear resistance leads to surface roughness, facilitating food and debris accumulation, which may contribute to microbial colonization and denture stomatitis [16]. Therefore, enhancing the hardness of PMMA - based materials is necessary to improve the longevity and hygiene of denture prostheses.

Another significant limitation of acrylic dentures is their low thermal conductivity. Despite several attempts to reinforce PMMA, only a few studies have examined the effects of such modifications on thermal conductivity. An ideal denture base prosthesis should exhibit maximum resistance to abrasion while maintaining adequate heat transfer properties under varying masticatory loads. Conventional cleansing agents may also weaken denture base materials, increasing the risk of fracture. PMMA - based materials, being inherently weak against abrasion, develop surface irregularities that encourage microbial adhesion and increase the risk of stomatitis. [17]

To address these issues, the present study incorporated zirconium dioxide (ZrO_2) into PMMA in different concentrations to evaluate its effect on thermal conductivity and microhardness. the specimens were fabricated using a metal die of respective measurements [14]. Enhancing the thermal conductivity of denture base materials could significantly improve patient comfort, while increased microhardness would enhance resistance to surface wear and microbial accumulation [5].

This study contributes to the growing body of research on denture base modifications and highlights the need for further investigations into optimal filler concentrations to balance antimicrobial effectiveness, mechanical strength, and thermal conductivity. Future studies should focus on long - term clinical outcomes, including wear resistance, patient comfort, and the durability of ZrO_2 np induced PMMA denture bases in the oral environment [9].

5. Limitations

Limitations include the risk of increased brittleness at higher zirconia concentrations and challenges in achieving uniform nanoparticle distribution. The study lacks long - term clinical evaluation, and cost implications may limit widespread adoption. Higher concentrations of zirconium dioxide may increase the cost of production due to additional processing requirements. While ZrO2 nanoparticles can enhance hardness, excessive incorporation may make the denture base more brittle and prone to fractures. Nanoparticles can increase surface roughness, which may promote plaque accumulation and bacterial adhesion. The increased hardness due to nanoparticles can make polishing more difficult, leading to a rougher surface. .

6. Future Scope

Newer methodologies are needed to ensure better dispersion and homogeneity of the nanoparticle in the polymer matrix and to improve mechanical properties of resins. Studying

how zirconia - reinforced dentures influence bacterial adhesion, biofilm formation, and fungal growth, especially in denture - wearing patients prone to infections. Evaluating the long - term stability of ZrO2 - modified denture bases in terms of water absorption, color stability, durability biocompatibility and resistance to wear and tear. Exploring advanced finishing and polishing methods to maintain a smooth surface while retaining the benefits of ZrO2.

7. Conclusion

The study concluded the incorporation of zirconia (ZrO_2) nanoparticles at different concentrations influenced the mechanical and thermal properties of PMMA. At 3% ZrO_2, there was a moderate improvement in microhardness and thermal conductivity without significantly affecting the material's workability, making it a viable option for enhancing denture base properties while maintaining flexibility. The 7% ZrO_2 concentration provided the most significant enhancement, improving both strength and thermal performance, making it the optimal concentration for reinforcement. However, at 10% ZrO_2, while microhardness and thermal conductivity further increased, the material became more brittle, potentially compromising its long - term durability and resistance to fracture. Thus, 7% ZrO_2 emerged as the most effective concentration, offering a balance between improved properties and material integrity.

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