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Original Research Article

Comparative evaluation of flexural strength of heat polymerized polymethylmethacrylate denture base material reinforced with silanized titanium dioxide nanoparticles and silanized aluminium oxide nanoparticles–An in vitro study

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ABSTRACT

Background: he denture base, which supports artificial teeth and rests on the oral mucosa, is commonly made from Polymethyl methacrylate (PMMA), introduced by Dr. Walter Wright in 1937. PMMA is popular for its ease of processing, low cost, light weight, and aesthetic appeal, but it has drawbacks such as low mechanical strength, brittleness, and a high coefficient of thermal expansion, leading to fractures, particularly under functional stress. Research has focused on incorporating metal oxide nanoparticles into PMMA to improve its mechanical properties and antimicrobial activity.

Aim & Objectives: To assess and compare the flexural strength of heat polymerized polymethyl methacrylate denture base material reinforced with 1% silanized aluminium oxide nanoparticles and 3% silanized titanium dioxide nanoparticles to that of heat polymerized polymethyl methacrylate denture base material.

Materials and Methods: Total 90 specimens were prepared, 30 each for PMM denture base material without reinforcement (group C), PMM reinforced with 1% Aluminium oxide nanoparticles (group A) and PMM reinforced with 3% Titanium dioxide nanoparticles (group T) respectively. Flexural strength was tested with universal testing machine at a 5mm/minute crosshead speed.

Results: Flexural strength testing on specimens showed a highly significant difference across the three groups ($p < 0.0001$). Significant differences were found between Group C and both Group A ($p < 0.000001$) and Group T ($p < 0.000001$), while no significant difference was observed between Group A and Group T ($p = 0.647$).

Conclusion: Reinforcement with nanoparticles increased the flexural strength, with 1% silanized aluminium oxide nanoparticles showing a highly significant improvement. However, no significant difference was found between specimens reinforced with 1% silanized aluminium oxide (Group A) and 3% silanized titanium dioxide nanoparticles (Group T).

1. Introduction

Denture base may be defined as that part of denture that rests on the oral mucosa and to which artificial teeth are attached. Various materials were used to fabricate it.

Dr. Walter Wright (1937) introduced Polymethyl methacrylate as a denture base material which became the major polymer to be used.[1] PMMA's appeal as a denture foundation material is due to its ease of production,

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low cost, light weight, outstanding cosmetic qualities, low water sorption and solubility, and ability to be repaired inexpensively. However, low heat conductivity, lower mechanical strength, brittleness, a high coefficient of thermal expansion, and relatively low modulus of elasticity make it more prone to failure during clinical service.[2]

According to studies, 68% of complete dentures fracture within three years.[3] Smith analyzed the real scenario in terms of denture breakage and identified two forms of failure.4 Outside the mouth, induced by impact forces (high stress rate), and inside the mouth, generally in function, which is most likely a fatigue phenomenon (low and repetitive stress rate).[4,5] Fracture of dentures has been a major worry, hence there was a need to increase the flexural strength of PMMA. Recent research have shown that properly designed metal oxide nanoparticles have strong antibacterial activity and mechanical strength, hence an attempt was made to incorporate inorganic nanoparticles into PMMA to improve its qualities.

2. Materials and Methods

Total 90 specimens were prepared, 30 each for PMM denture base material without reinforcement, PMM reinforced with 1% Aluminium oxide nanoparticles and PMM reinforced with 3% Titanium dioxide nanoparticles respectively.

2.1. Preparation of dies

Three metal dies of dimensions 65mm in length, 10mm in width, and 3mm in height were created to make molds for producing specimens of heat-polymerized polymethyl methacrylate denture base material. (ISO 1567 standard).[6,7]

2.2. PMM specimens without reinforcement

With the help of the dies, 30 specimens were prepared using the conventional heat polymerized polymethyl methacrylate denture base material as per manufacturer's instruction, where the monomer and polymer were combined in a 1:2.5 weight ratio.

2.3. PMM specimens with silanized aluminium oxide nanoparticles

30 samples were prepared using conventional heat polymerized polymethyl methacrylate denture base material reinforced with silanized aluminium oxide nanoparticles. 7.425gms of polymer, 3ml of monomer and 0.075gms of silanized aluminium oxide nanoparticles were used for fabrication of 3 specimens.[8,9]

2.4. Silanization of aluminium oxide nanoparticles

A total of 4 grams of aluminum oxide nanoparticles were introduced into 100 mL of toluene in a glass beaker and sonicated for 20 minutes at room temperature. Following sonication, a magnetic stirrer was added to the beaker, and the mixture was stirred for 30 minutes at room temperature. After this 0.2ml of silane coupling agent (3-methacryloxy propyl trimethoxy silane) was added slowly, drop by drop under rapid stirring for homogenous mixing of silane coupling agent with aluminium oxide nanoparticles and toluene

solvent.[8,10] The beaker was covered with Parafilm and left to stand for 2 days. Afterward, the toluene solvent was removed using a rotary evaporator under vacuum at 60°C and a rotation speed of 150 rpm for 30 minutes. The silanized aluminum oxide nanoparticles were dried in a vacuum oven at 60°C for 10 hours.[9]

2.5. PMMA specimens with silanized titanium dioxide nanoparticles

30 samples were prepared using conventional heat polymerized polymethyl methacrylate denture base material reinforced with silanized titanium dioxide nanoparticles. 7.275gms of polymer, 3ml of monomer and 0.225gms of slianized titanium dioxide nanoparticles were used for fabrication of 3 specimens.

2.6. Silanization of titanium dioxide nanoparticles

A 100 mL ethanol aqueous solution (70% v/v) was prepared by mixing 99.8% ethanol with de-ionized water (30% v/v), and the pH was adjusted to 4.5 using a pH meter and titrating with 99.9% acetic acid. To this solution, 0.1125 mL of a 5% (wt) silane coupling agent (3-methacryloxypropyl trimethoxysilane) was added and stirred with a magnetic stirrer. Next, 5 grams of titanium dioxide nanoparticles were incorporated into the silane solution, and the mixture was stirred for 20 minutes. This was followed by sonication using a probe sonicator for 30 minutes. The solution was then left to dry at room temperature for 14 days.[11]

2.7. Instrument Used to measure the Flexural strength

For measuring Flexural strength, a 3-point bending test was used.

Flexural strength was tested with universal testing machine at a 5mm/minute crosshead speed.[4]

A jig was present to support the specimen which was separated at a distance of 50mm.

Flexural strength (FS) was calculated with the help of the formula-

$FS= 3PI/2bd^2$

Where, FS= flexural strength

P= load

I= distance between supporting wedges (mm), b= width of the specimen (mm)

d= thickness of the specimen (mm)[7]

2.8. Statistical analysis

First the raw data were treated by the means and standard deviations and then One way ANOVA by K. Wallis test was used. For searching the strength of association between the groups and in order to search for the significance, the data was treated with Tukey's post-hoc test for independent groups.

2.9. Statistical interpretation

First the mean, median and standard deviation was calculated with the help of flexural strength score.

Table 1 shows the descriptive statistics for flexural strength. The mean score for flexural strength of Group A (M=107.59) is higher than that of Group C (M=89.18) and Group T (M=105.73). However only on the basis of means and standard deviations meaningful conclusions cannot be drawn. Hence the data were treated by One way analysis of variance (ANOVA).

Table 2 shows One-way analysis of variance for Flexural Strength across three groups. The mean flexural strength varied significantly among the three groups, as demonstrated by the p-value < 0.000 (1.4123E-14).

To identify which specific groups contributed to the overall significance, a pairwise comparison of mean strength was conducted using Tukey's HSD test.

Table 3 shows a highly significant difference in the means between Group C and Group A ($p < 0.000001$). Additionally, the difference in the means between Group C and Group T was statistically significant ($p < 0.000001$). However, the difference between Group A and Group T was statistically insignificant, as indicated by the p-value0.647 ($p > 0.05$).

3. Result

In this study flexural strength of heat polymerized polymethyl methacrylate acrylic resin reinforced with 3% silanized titanium dioxide nanoparticles and 1% silanized aluminium oxide nanoparticles was evaluated and compared with the conventional heat polymerized polymethyl methacrylate denture base resin.

Specimens of each group were subjected to flexural strength test on Universal Testing Machine at a crosshead speed of 5.0mm/min. The maximum load was determined from the chart and recorded as fracture load in N (Newton). The flexural strength was calculated in MPa.

The mean flexural strength showed a highly significant difference across the three groups, as indicated by a p-value of < 0.000001 (1.4123E-14). Specifically, a significant difference was found between the means of Group C and Group A ($p < 0.000001$), as well as between the means of Group C and Group T ($p < 0.000001$). However, the difference between Group A and Group T was not statistically significant, with a p-value of 0.647 ($p > 0.05$).

4. Discussion

Acrylic polymers were utilized as denture base materials since 1937 and by 1946, 98% of all denture bases were based on PMMA.[2] In 1942, vinyl acrylic copolymer and in 1948 polystyrene, a styrene polymer developed by Charles Dimmer, were introduced as denture base materials which had greater flexural strength and high residual stresses.[12]

Due to its low cost, biocompatibility, ease of processing, stability in the oral environment, and acceptable aesthetics, Poly(methyl methacrylate) (PMMA) is commonly used to fabricate denture bases. However, despite these advantages, PMMA is not considered an ideal denture base material due to its relatively poor physical and mechanical properties.[13]

Smith DC analysed denture fractures and identified two types of failure: one occurring outside the mouth, caused by impact forces (high stress rate), and the other inside the mouth, likely resulting from fatigue (low and repetitive stress rate). Based on these findings, he concluded that denture fractures occur due to flexural fatigue under these specific conditions.[5] Beyli et al suggested that midline fracture of a denture base is caused due to flexural fatigue failure which is a result of cyclic deformation of the base during function.[14]

These considerations led to a general conclusion that denture fracture occurs due to flexural fatigue under respective conditions and due to this reason flexural strength test was selected as most relevant to evaluate the strength of denture base resin.

Since the fracture resistance of a denture base resin is crucial, efforts have been made to enhance the mechanical properties of acrylic resin. These efforts include increasing the material's bulk in areas subjected to the highest stress through copolymerization and crosslinking, as well as reinforcing the resin with glass fibers, aluminum and sapphire whiskers, polycarbonates, carbon fibers, and metal strengtheners.

Yazdaine N and Mahood M studied the flexural strength of acrylic resin reinforced with carbon fibres and concluded that carbon fibre acrylic resin composites are stronger and stiffer than unfilled acrylic resin and strands are more efficient strengtheners than are woven mats.[15]

Berrong, Weed, and Young (1990)[16] studied the reinforcement of acrylic resin with aramid fibers, which resulted in improved fatigue resistance. However, the yellow color of the fibers was challenging to conceal within the denture, requiring the use of thick layers of acrylic resin, which significantly increased the bulk of the denture**.**

Many attempts to strengthen acrylic resin using embedded fibers or metals were unsuccessful because stress concentrations developed around the embedded materials. As a result, the overall effect of embedding fibers or metals weakened the polymer. This failure was primarily due to poor adhesion between the fibers or metal inserts and the acrylic resin matrix.[6]

Table 1: Descriptivestatistics for flexural strength

Groups	N	Mean	Median	SD	Range
Group C (Control)	30	89.18	87.625	12.058	60.43, 112.61
Group A (Al^2O^3)	30	107.59	106.155	5.716	100.76, 120.21
Group T (TiO^2)	30	105.73	104.98	4.289	99.9, 112.82

Table 2: One-way analysis of variance for Flexural Strength across three groups

Source	Degree of freedom	Sum Squares	Mean Square	F-Value	P-Value
Between Groups	2	6162.081	3081.041	47.049	0.000(1.4123E-14)
Within Groups	87	5697.317	65.486		

Table 3: Pair-wise comparison of flexural strength between groups using Tukey's post-hoc test

Comparison	p-value
Group C Vs Group A	0.000(5.1008E-9)
Group C Vs Group T	0.000 (5.1215E-9)
Group A Vs Group T	0.647

Silanization of the surface of the metal with different techniques improves the adhesion between acrylic resin and the filler particle which enhances the fracture resistance of the denture base material.[17] These considerations led to the process of silanization of the filler particle before incorporating to the acrylic resin.[18]

In his study, Solnit G concluded that silane-treated glass fibers in a loose form significantly strengthened PMMA compared to samples with untreated fibers. However, this strengthening effect was not observed when compared to the control samples. He explained that untreated fibers behave as inclusion bodies within the acrylic resin mixture, which does not strengthen the material but instead weakens it. The fibers can disrupt the homogeneous matrix, leading to reduced mechanical integrity.[19]

The increase in fracture resistance of an acrylic-fiber composite depends on the adhesion between the acrylic resin matrix and the fibers. Silane compounds can be used to enhance the adhesive properties of the fibers, thereby improving the overall strength of the composite.[18]

For a given resin/filler system, the physical-chemical properties of the silane agent—such as the chemical structure used, the orientation of the silane layer, and the extent of filler coverage—are crucial factors that influence many of the physicochemical and mechanical properties of the interphase.[20,21]

Recently, significant attention has been focused on incorporating inorganic nanoparticles into PMMA to enhance its properties. The performance of polymer nanocomposites depends on factors such as the type, size, and shape of the nanoparticles, as well as their concentration and interaction with the polymer matrix. Nanoparticles have gained increasing use in materials science due to their ability to improve wear resistance and provide anti-corrosion properties.[22]

Aluminium oxide nanoparticles possess strong ionic inter-atomic bonding, have high hardness, good thermal properties, decrease warpage, make the material radio-opaque and inhibit growth of bacteria over the denture surface.[6,13,23] Jasmin BS and Ismail IJ (2014) observed a highly significant increase in transverse strength with the addition of (Al2O3) nanoparticles to (PMMA) at the percentage of 1wt% as well as at 2wt%.[24]

Titanium dioxide is a biocompatible, non-toxic, corrosion resistant material with antimicrobial properties. It also increases the flexural and impact strength and aesthically acceptable. Several studies have explored the impact of adding titanium dioxide (TiO2) on the properties of PMMA. These studies have shown that incorporating TiO2 nanoparticles can enhance the flexural strength, fracture toughness, hardness, and thermal conductivity of PMMA.[13,25] Alwan SA and Alameer SS in 2015 concluded that addition of silanized 3% titanium dioxide nanoparticles increases the value of flexural strength of PMMA when compared with the control group.[11]

Therefore, an attempt is being made to evaluate and compare the flexural strength of heat polymerized acrylic resin denture base material reinforced with 3% silanized titanium dioxide nanoparticles and 1% silanized aluminium oxide nanoparticles with conventional heat polymerized polymethyl methacrylate denture base resin.

4.1. Scope for further studies

1. Fatigue testing of these materials under dynamic loading, using denture base configurations in simulated oral conditions with saliva or its substitutes, presents an important area for further investigation.
2. Additional research is needed to examine the effects of aging on the newly reinforced denture base material prior to its clinical implementation.

3. Further investigation could focus on other physical and mechanical properties, such as thermal diffusivity, hardness, abrasion resistance, color stability, and disinfectant effectiveness.
4. Heat-polymerized acrylic dentures can be further reinforced with nanoparticles of varying sizes, and a range of physical and mechanical properties can be evaluated.

5. Conclusion

Within the limitations of this study, it was found that reinforcement with nanoparticles significantly increased the flexural strength of denture base specimens. Specifically, 1% silanized aluminum oxide nanoparticles resulted in a highly significant improvement. However, no significant difference was observed between specimens reinforced with 1% silanized aluminum oxide nanoparticles (Group A) and those reinforced with 3% silanized titanium dioxide nanoparticles (Group T), suggesting a comparable effect on flexural strength.

6. Source of Funding

None.

7. Conflict of Interest

None.

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