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Original Article

Dentistry Section

Comparison of Flexural Strength and Surface Hardness of Polymethyl Methacrylate Resin Reinforced with Silanised Aluminium Oxide Nanoparticles- An In-vitro Study

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ABSTRACT

Introduction: In complete denture fabrication, the common denture base material used is heat activated Polymethyl Methacrylate (PMMA). Considering various advantages, still there are some disadvantages like poor flexural strength and poor wear resistance. The flexural strength of any material reflects its potential to resist catastrophic fracture under a flexural load. Another property that influences the surface characteristics of acrylic resins is the surface hardness, which indicates the ease of finishing a material and its resistance to in-service scratching during cleaning procedures and exposure to various oral fluids. Thus an ideal denture base material should exhibit greater flexural strength and high surface hardness for the longevity of the dentures.

Aim: To evaluate the effects of adding different percentages of silanised aluminium oxide (Al_2O_3) nanoparticles on the flexural strength and surface hardness of a conventional heat-polymerised acrylic resin.

Materials and Methods: The in-vitro experimental study was conducted between October 2020 to Janaury 2021 at Drs. Sudha and Nageswara Rao Siddhartha Institute of Dental Sciences, Vijayawada, Andhra Pradesh, India. A total of 120 samples were fabricated and were grouped into four groups coded A to D (n=30). Group A was the control group (without adding Al_2O_3). Specimens in the other three groups (B to D) were reinforced with silanised Al_2O_3 at loadings of 1%, 2.5% and 5% w/w. Flexural strength was assessed with a three-point bending test using a universal testing machine. Surface hardness test was conducted using a Vickers Hardness (VH) tester. Data was analysed using Analysis of Variance (ANOVA) and Tukey's post-hoc test.

Results: Among all the reinforced groups highest flexural strength value was seen in Group C- PMMA+2.5% w/w silanised aluminium oxide nanoparticles reinforced group (88.33 Mpa) and highest surface hardness value was seen in the Group D- PMMA+5% w/w silanised Aluminium oxide nanoparticles reinforced group (29.44 VH).

Conclusion: Reinforcement of the conventional heat cured acrylic resin with 2.5% w/w silanised Al_2O_3 nanoparticles significantly increased its flexural strength and hardness.

Keywords: Complete denture prosthodontics, Denture base resins, Mechanical properties, Silane coupling agent

INTRODUCTION

Complete denture is still one of the best solution to replace the missing natural teeth and their surrounding structures in the oral cavity. Dentures enhance masticatory efficiency along with aesthetics and phonetics, thereby improving the quality of life [1]. Dentures are fabricated using heat cure acrylics, light cure acrylics, microwave acrylics and Computer Aided Design and Computer Aided Manufacturing (CAD/CAM) acrylics [2]. The most widely used denture base resin is heat-activated Polymethyl Methacrylate (PMMA), because of its properties like lightweight, ease of fabrication, aesthetical properties, and being less economical. Less strength and wear resistance are drawbacks of this material [3].

To enhance these properties like less strength and wear resistance of PMMA, various techniques have been used, like different curing techniques, incorporating different nanoparticles (sizes 1-100 nm range) and nano fibres [4]. Of all these, the most promising method recently used is the incorporation of nanoparticles into the PMMA that act as reinforcing material to enhance its strength and wear resistance [5]. According to the results of a study, the properties of the polymer nanocomposites have proven to be dependent on the nanoparticle. The type of incorporated nanoparticles, shape, size, concentration and interaction of the nanoparticles with the polymer matrix determine the properties of the polymer nanocomposite [6]. The commonly used nanoparticle material is Aluminium oxide (Al_2O_3) due to its high hardness and excellent thermal properties. Being white in colour; it is less likely to hinder the esthetic properties [7]. The mechanical properties of polymer nanocomposites depend on the dispersion and adhesion of the fillers between the filler and matrix [8]. Surface treatment with silane coupling agents is applied to the fillers to increase this compatibility between the filler and the matrix [9].

With limited amount of data available in the literature regarding the strength and wear resistance of Silanised Aluminium oxide nanoparticles (Si Al_2O_3) with PMMA resin, the aim of this study was to evaluate the flexural strength and surface hardness of PMMA resin reinforced with Si Al_2O_3 nanoparticle material.

MATERIALS AND METHODS

This in-vitro experimental study was conducted at Drs. Sudha and Nageswara Rao Siddhartha Institute of Dental Sciences, Gannavaram, Vijayawada, Andhra Pradesh, India, between October 2020 to January 2021. Ethical clearance was obtained from Institutional Ethical Committee (Certificate OC no:/IEC/03/2018 Dated on 08/12/2018).

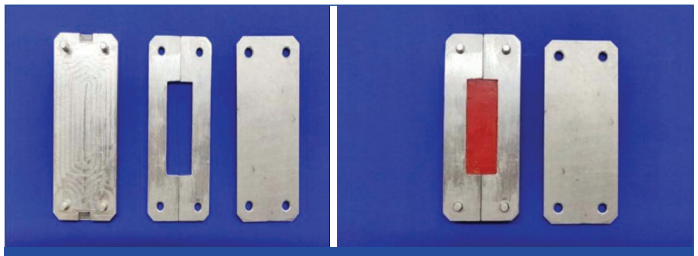
A total of 120 samples were fabricated and were grouped into four groups coded A to D (n=30). Each group of 30 samples were further categorised into two subgroups of 15 samples each for measuring flexural strength and 15 samples to evaluate surface hardness.

Sample Preparation

Metal mould for preparation of samples: A metal mould of size 65×10×3 mm was constructed using substractive technology

in aluminium for the fabrication of heat cure acrylic denture base resin samples number- 120 using wax (according to International Organisation for Standardisation (ISO) 1567 standard) [10,11]. Later they were processed in acrylic [Table/Fig-1]. The moulds had three removable plates with reorientation grooves in upper, middle and lower plates and a split middle plate for easy reorientation and aids in easy removal of the plates without distorting the desired wax pattern.

Fabrication of wax patterns: Modelling wax (Hindustan Pvt., Ltd.,) was melted using a wax pot at a controlled temperature of 58°C [12]. Then the molten wax was carefully poured into the aluminium metal mould for the fabrication of the wax samples. The modelling wax was allowed to set till it attains room temperature (around 20 min) and the mould was dearticulated carefully for the removal of wax sample [Table/Fig-2].



[Table/Fig-1]: Metal mould fabricated in aluminium showing three layer removable plates.
[Table/Fig-2]: Dearticulated first compartment showing intact wax sample at room temperature. (Images from left to right)

Flasking of samples: At a time two wax samples were invested in a varsity dental flask (Classic & Co.) using type II gypsum product-plaster of paris (Neelkanth Pvt., Ltd.,) in a two pour technique [7]. After the final set of gypsum, the flasks were placed in dewaxing unit at a temperature of 100°C for a period of 5 minutes [13]. The flasks were then opened and dewaxed to remove any traces of wax to obtain the mould space [Table/Fig-3].

Preparation of PMMA Reinforced with $SiAl_20_3$

a) Silanisation of aluminium oxide nanoparticle materials before addition to PMMA: Aluminium oxide nanoparticle materials (>50 nm size- Nano research laboratory)- 17 gm without any purification, were dispersed into the solution consisting of 3-methacryloxypropyltrimethoxysilane (MPS) (3 Macquira Pvt., Ltd.,)- 5 mL and manually triturated until the silane gets evaporated completely. This silanised aluminium oxide nanopartiles were then left to dry at room temperature for 14 days before use [2] [Table/Fig-4].

[Table/Fig-3]: Clean mould space obtained after dewaxing.
[Table/Fig-4]: Dried Silanised aluminium oxide nanoparticles. (Images from left to right)

These silanised aluminium oxide nanoparticle materials were now mixed with PMMA resin [Table/Fig-5]. To evenly distribute the silanised aluminium oxide nanoparticle materials within the PMMA matrix, the Si Al_20_3 powder and PMMA powder was mixed thoroughly using a mortar and pestle for initial mixing and blending, followed by hand tumbling in a plastic jar until a uniform colour was achieved [14].

b) Processing of samples: The PMMA resin (Trevalon - Dentsply India Pvt., Ltd.,) powder and liquid were mixed in 2:1% w/w according to the specification of the manufacturer in a ceramic crucible with a lid [15,16]. In the late stages of stringy and early stages of dough, the mixture was packed into the mould space and the flask was closed

Groups (n=30)	Si Al_20_3 (gm)	PMMA-Polymer (gm)	PMMA-Monomer (mL)
Group A- Unmodified PMMA (without any Si Al_20_3)	-	200 gm	100 mL
Group B- PMMA+1% w/w Si Al_20_3	2 gm	198 gm	100 mL
Group C- PMMA+2.5% w/w Si Al_20_3	5 gm	195 gm	100 mL
Group D- PMMA+5% w/w Si Al_20_3	10 gm	190 gm	100 mL

[Table/Fig-5]: Weight measurement of PMMA for the preparation of samples-Groups division.

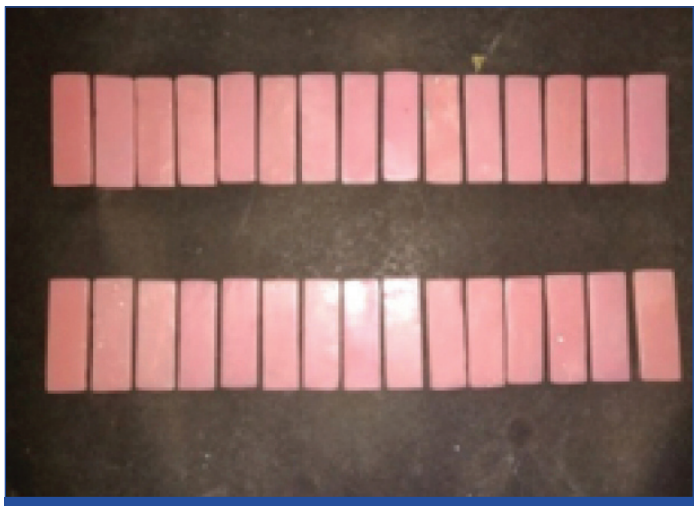
under 100 psi pressure using a bench press unit and left for about 30 minutes for bench curing [17,18].

At room temperature the flasks were kept in the acryliser and a short curing cycle was followed as 74°C for 2 hours followed by 99°C for 1 hour [19].

After the water cooled to room temperature, deflasking was carried out, and the excess material was trimmed using a tungsten carbide bur (Waldent acrylic trimming bur H6.) in a low speed rotary instrument. The samples were then finished and polished using 120 grit sandpaper for 1 minute followed by pumice polish for 1 minute on both surfaces of the samples [20]. The finished and polished samples were then placed in incubator, in distilled water medium for three days at a temperature of 37°C before testing to stimulate the oral environment [21,22].

Division of Samples

A total of 120 samples were fabricated and were grouped into 4 groups coded A to D [Table/Fig-6].



[Table/Fig-6]: Division of prepared samples.

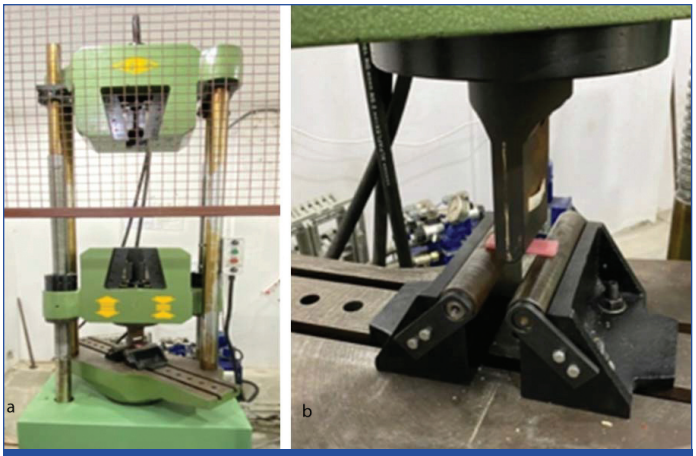
Each group of 30 samples was further categorised into two subgroups of 15 samples each for measuring flexural strength and 15 samples to evaluate surface hardness.

Measurement of Flexural Strength of Samples

Flexural strength was measured by using universal testing machine (Instron 3366), with three-point bending test [23,24]. Samples were placed in a position where the load was applied in the center of the specimen from an upper side and its two edges are supported from the lower side (three-point bending) [Table/Fig-7]. The distance between the two supports of the machine was 50 mm and the test was carried out using a cross head speed of 5 mm/min [25,26]. The specimens were then subjected to load up to its fracture point.

The flexural strength was then measured by using the following equation:

$$S=\frac{3pl}{2bd^2}$$



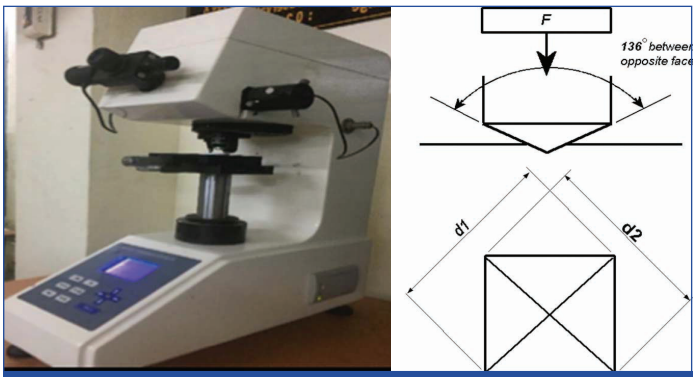
[Table/Fig-7]: a) Universal testing machine- instument to measure flexural strength; b) Sample in position and application of three-point bending test.

in which S is the flexural strength, p is the force at the fracture point, l is the distance between the two parts of the machine's base, b is the specimen's width, and d is the specimen's thickness. Flexural strength test was conducted in accordance to the common ISO-20795-1 standard [27].

Measurement of Surface Hardness of Samples

The Micro VH test was performed using a calibrated VH Tester FV (Future-Tech) to determine the surface hardness [Table/Fig-8].

The Vickers Hardness (VH) involves the use of diamond pyramid indenter. The indenter is in the form of a right pyramid with a base of square shape and an angle of 136° between the opposing faces [Table/Fig-9]. The sample was subjected to a load(p) of (50 gm) and for the time duration of 10 seconds [28].



[Table/Fig-8]: Vickers Hardness (VH) Tester- instrument to measure surface hardness.
[Table/Fig-9]: Schematic representation of Vickers Hardness (VH) testing. (Images from left to right)

The test specimen was held firmly in position and lens were arranged to get the image clearly at its focal length, then the indentation was made using set parameters. The two diagonals of the indentation left behind on the surface of the material after the removal of the load were measured and their average value was calculated (d).

VH was calculated using the equation as stated in ASTM standard [28]:

$$VH = \frac{1:8544\ P}{d^2}$$

load (p)

Average value of the two diagonals of the indentation (d).

The mean value of 10 indentations on surface of specimens was recorded and used to determine the VH according to ASTM E 384-89 standard [29].

STATISTICAL ANALYSIS

Descriptive statistics was conducted from the collected data using one-way Analysis of Variance (ANOVA) test to compare the flexural strength and surface hardness between the four groups. Tukey's post-hoc test was used to determine whether there were significant differences in flexural strength and surface hardness among the four experimental groups (intergroup comparison). The p-value <0.05 was taken as the level of significance.

RESULTS

Maximum flexural strength mean value (88.33 Mpa) was seen in Group C- PMMA + 2.5% w/w silanised Aluminium oxide and minimum flexural strength mean value (73.17 Mpa) is seen in Group A- Control group - unmodified PMMA [Table/Fig-10].

Group	Mean (MPa)	SD	p-value
Group A	73.17	1.81	p<0.001*
Group B	83.67	1.42	
Group C	88.33	1.55	
Group D	84.16	1.52	

[Table/Fig-10]: Comparison of the groups on the basis of flexural strength. *Statistically significant; SD: Standard deviation; ANOVA test applied

[Table/Fig-11] All the other groups (Group B, C, D) show a significant increase in the flexural strength compared to the control group-Group-A. Group-B shows a significant increase in the flexural strength compared to Group A, whereas compared to Group C, it shows a significantly low value. There is no statistically significant change between the flexural strengths of Group B and Group D. Group C shows a statistically significant increase in the flexural strength value compared to all the other groups (Group A, B, D). Group D shows a significant increase in the flexural strength compared to group A, whereas compared to Group C, it shows a significantly low value. There is no statistically significant change between the flexural strengths of Group D and Group B.

(I) Group	(J) Group	Mean difference (I-J)	Std. Error	p-value
Group A	Group B	-10.50333*	0.57915	p<0.001*
	Group C	-15.16067*	0.57915	p<0.001*
	Group D	-10.98733*	0.57915	p<0.001*
Group B	Group A	10.50333*	0.57915	p<0.001*
	Group C	-4.65733*	0.57915	p<0.001*
	Group D	-0.48400	0.57915	0.837
Group C	Group A	15.16067*	0.57915	p<0.001*
	Group B	4.65733*	0.57915	p<0.001*
	Group D	4.17333*	0.57915	p<0.001*
Group D	Group A	10.98733*	0.57915	p<0.001*
	Group B	0.48400	0.57915	0.837
	Group C	-4.17333*	0.57915	p<0.001*

[Table/Fig-11]: Intergroup comparison on the basis of flexural strength. Post-hoc Tukey applied, *Statistically significant

Maximum surface hardness mean value (29.44 VH) was obtained in Group D- PMMA + 5% w/w silanised Aluminium oxide and minimum surface hardness mean value (21.58 VH) was obtained in Group A- Control group - unmodified PMMA. There shows an increase in surface hardness from Group A to Group D [Table/Fig-12].

Group	Mean (VH)	SD	p-value
Group A	21.58	1.47	p<0.001*
Group B	24.12	1.44	
Group C	27.24	1.28	
Group D	29.44	0.73	

[Table/Fig-12]: Comparison of the groups on the basis of surface hardness. *Statistically significant, ANOVA applied

[Table/Fig-13] All the other groups (Group B, C, D) show a significant increase in the Surface hardness compared to the control group-

Group-A. Group B shows a significant increase in the surface hardness compared to group A, whereas compared to Group C and Group D, it shows a significantly low value. Group C shows a statistically significant increase in the surface hardness value compared to Group A and Group B, whereas compared to Group D, it shows a significantly low value. Group D shows a significant increase in the flexural strength compared to all the other groups (Group A, B, C).

(I) Group	(J) Group	Mean difference (I-J)	Std. Error	p-value
Group A	Group B	-2.54000*	0.46445	p<0.001*
	Group C	-5.66000*	0.46445	p<0.001*
	Group D	-7.86000*	0.46445	p<0.001*
Group B	Group A	2.54000*	0.46445	p<0.001*
	Group C	-3.12000*	0.46445	p<0.001*
	Group D	-5.32000*	0.46445	p<0.001*
Group C	Group A	5.66000*	0.46445	p<0.001*
	Group B	3.12000*	0.46445	p<0.001*
	Group D	-2.20000*	0.46445	p<0.001*
Group D	Group A	7.86000*	0.46445	p<0.001*
	Group B	5.32000*	0.46445	p<0.001*
	Group C	2.20000*	0.46445	p<0.001*

[Table/Fig-13]: Intergroup comparison on the basis of surface hardness. *Statistically significant, Tukey's post-hoc test applied

DISCUSSION

In complete denture fabrication, the common denture base material used is heat activated PMMA. Considering various advantages, still there are some disadvantages like poor flexural strength and poor wear resistance [1,30-32]. The flexural strength of any material reflects its potential to resist catastrophic fracture under a flexural load. As a foundation, the acrylic resin materials should exhibit a greater flexural strength to resist plastic deformation under cyclic load, and also should resist fracture during improper handling like sudden drop. Another property that influences the surface characteristics of acrylic resins is the surface hardness, which indicates the ease of finishing a material and its resistance to in-service scratching during cleaning procedures and exposure to various oral fluids. Thus an ideal denture base material should exhibit greater flexural strength and high surface hardness for longetivity of the dentures [6].

Various materials have been used to enhance the properties like flexural strength and wear resistance of PMMA. These include addition of metal wires and cast metal plates. The primary problem with using metal wire is poor adhesion between the wire and resin. Although metal plates increase the strength, they may be expensive and prone to corrosion and difficult to adapt and polish [3,33]. Reinforcement of acrylics with fibers also produced encouraging results. However, this method has various problems including tissue irritation, difficulties in manipultion, the need for precise orientation, and placement or bonding of the fibers within the resin [6,34].

With the emergence in nanotechnology, addition of different nanoparticles from sizes 1-100 nm range is the most promising method recently used [4]. Nanoparticle materials like quartz, colloidal silica, zirconia, titanium and aluminium oxide have been used in dental materials as different types of inorganic fillers. The type of incorporated nanoparticles along with their size, concentration determine the properties of the PMMA [6]. Aluminium oxide (Al_2O_3) nanoparticles have high surface hardness and excellent thermal properties due to which it is the most widely used nanoparticle, being white in colour; it is less likely to alter the esthetics [6,35]. Considering the advantages mentioned above, in this study Aluminium oxide nanoparticles are taken as filler particles, which are added to heat activated PMMA resin in different concentrations (i.e., 1%, 2.5% and 5%).

Since there is no chemical bonding like that of nanofibers, to achieve good bonding between Aluminium oxide nanoparticles and PMMA, modification of Aluminium oxide nanoparticles surface is necessary [7]. Surface treatment with silane coupling agent is applied to the Aluminium oxide nanoparticles to increase this compatibility between the Aluminium oxide nanoparticles and PMMA thus getting better bond [5,36].

These silane coupling agents consist of two terminal groups; organofunctional group, which establish a bond with an organic PMMA resin, and hydrolysable groups, which establish a bond with inorganic Aluminium oxide nanoparticle materials [2]. Thus, the addition of silane coupling agents increases the bonding between Aluminium oxide nanoparticles and the PMMA resin matrix. With paucity in the literature regarding the strength and wear resistance of Si Al_2O_3 with PMMA resin, the present study was undertaken to evaluate the flexural strength and surface hardness of PMMA resin reinforced with Si Al_2O_3 nanoparticle material.

Flexural strength was measured for all the prepared samples by using universal testing machine (Instron 3366), with three-point bending test. The results for flexural strength test showed that all the three groups that are reinforced with silanised aluminium oxide nanoparticles showed an increase in the flexural strength compared to the unreinforced group. This increase in flexural strength can be explained on the basis of transformation toughening. The Al_2O_3 exists in various crystalline phases, and all the Al_2O_3 particles revert to the most stable hexagonal alpha phase at increased temperatures [6]. When sufficient stress develops and microcracks begin to propagate, the transformation phenomenon occurs, which depletes energy for crack propagation. Therefore, proper distribution of the Aluminium oxide nanoparticles within the PMMA resin matrix can stop or deflect cracks thus increasing its flexural strength [6,37].

The Micro VH test was performed using a calibrated VH Tester FV (Future-Tech) to determine the surface hardness. The results for Surface hardness test showed that all the 3 groups that were reinforced with silanised aluminium oxide nanoparticles showed an increase in the flexural strength compared to the unreinforced group. This increase in hardness may have been due to inherent characteristics like high hardness of the Al_2O_3 particles. The most stable hexagonal alpha phase Al_2O_3 is the strongest and stiffest of the metal oxide nanoparticles [29]. Therefore, it is expected that when Al_2O_3 particles disperse in a PMMA resin matrix, they increase its hardness and strength. Various studies comparing the physical properties of PMMA reinforced with different materials have been compared in [Table/Fig-14] [27-34,37].

Authors name and year	Place of study	Sample size	Particles used for reinforcement of PMMA	Parameters assessed	Conclusion
Farina AP et al., 2010 [27]	São Paulo, Brazil.	120	Glass Fibre Reinforcement (GFR)	Vickers Hardness (VH) testing	GFR increased the Vickers Hardness (VH) of resins
Kamble VD et al., 2012 [28]	Nagpur, India.	45	Polyethylene and glass fibers	Flexural strength	Of two fiber reinforcement methods evaluated, glass fiber reinforcement for the PMMA resin and bis-acryl composite resin materials produced highest flexural strength.
Alhareb AO et al., 2016 [29]	Penang, Malaysia	180	Nitrile Butadiene Rubber (NBR) with two types of ceramic fillers (Al_2O_3 and Yttria Stabilised Zirconia, respectively)	Impact strength, fracture toughness and hardness	The reinforced PMMA denture bases are significantly different in IS and KIC between study groups. However, the Vickers Hardness (VH) is statistically not significantly different

Protopapa P et al., 2011 [30]	Macedonia, Greece	96	Nnanodiamonds	Fracture toughness, impact strength and the dynamic thermomechanical properties	reinforcing PMMA with ND nanoparticles especially at low concentrations may increase the overall performance of fixed interim prostheses.
Sodagar A et al., 2012 [31]	Kargar Tehran, Iran.	90	Silver nano particles (AgNps)	Flexural strength	The effect of AgNps on flexural strength of PMMA depends on several factors including the type of acrylics and the concentrations of nano particles.
Vikram S and Chander NG, 2020 [32]	Chennai, India.	60	Zinc oxide (ZnO) nanoparticles.	Flexural strength	The addition of ZnO nanoparticles in all concentrations increased the flexural strength of acrylic resin when compared to the control group.
Ladha K and Shah D 2011 [33]	Ghaziabad, Uttar Pradesh, India	160	Novel pre-impregnated glass fiber and nylon fiber reinforcement	Flexural strength	The reinforcement of denture base resin with preimpregnated glass fibers may be a useful means of strengthening denture bases.
Bangera MK et al., 2020 [34]	Mangalore, Karnataka, India	Systematic review	Titanium dioxide	Flexural strength	There is no precise conformity on the ideal titanium dioxide nanoparticle concentration required to improve the flexural strength of the polymer.
Ergun G et al., 2016 [37]	Turkey	160	Zirconium oxide	Transverse strength, modulus of elasticity, surface roughness, hardness, and water sorption/solubility	Increased hardness values, implying that the addition of nano-ZrO_2 would contribute positively to some mechanical properties of PMMA denture base material when nano-ZrO_2 was homogeneously distributed in PMMA.
Present study	Vijayawada, Karnataka, India	120	Silanised Aluminium oxide nanoparticles	Flexural strength and Surface hardness	Reinforcement of the conventional heat-cured acrylic resin with 2.5% w/w silanised Al_2O_3 nanoparticles significantly increased its flexural strength and hardness.

[Table/Fig-14]: Brief details of some previous studies and the present study [27-33,36,37].

Limitation(s)

Use of uniform thickness specimens instead of more complex denture shapes, thickness and design is one of the limitations of this study. Quantitative analysis like bonding between Aluminium oxide nanoparticles and PMMA has to be verified at molecular levels. Further research is needed to examine other physical and mechanical properties of PMMA reinforced with Aluminium oxide nanoparticles with denture teeth. In-vitro studies are limited in their ability to predict the success of a material or technique in a clinical situation based on its exposure to various oral environmental conditions (patient deleterious habits).

CONCLUSION(S)

Flexural strength of conventional heat cure PMMA resin significantly increased when reinforced with Silanised Aluminium oxide nanoparticles in different concentrations (i.e., 1%, 2.5%, 5%). Among all the reinforced groups highest flexural strength value was seen in the Group C-PMMA+2.5% w/w silanised Aluminium oxide nanoparticles reinforced group. Surface hardness of conventional heat cure PMMA resin significantly increased when reinforced with Silanised Aluminium oxide nanoparticles in different concentrations (i.e., 1%, 2.5%, 5% w/w). Among all the reinforced groups highest Surface hardness value was seen in the Group D- PMMA+5% w/w silanised Aluminium oxide nanoparticles reinforced group.

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