



Research article

Flexural strength and surface hardness of nanocomposite denture base resins

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ABSTRACT

Purpose: Higher bending forces during chewing and occlusal loading can lead to the deformation of denture bases. Roughness and microbial adhesion can be the result of improper care of the denture. Many attempts have been made to improve the properties of denture bases through the addition of different materials. The present study aimed to evaluate the surface hardness and flexural strength (FS) of newly formulated nanocomposite denture base resin made by adding zinc oxide (ZnO) and titanium dioxide (TiO₂) nanoparticles in heat polymerized polymethyl methacrylate resin in concentrations of 1 % and 2 %.

Methods: Rectangular metal master dies of dimension 65mm × 10mm × 3.3 mm for flexural strength and 30mm × 10mm × 3 mm for surface hardness were made. These dies were duplicated in 120 acrylic resin samples. These samples were divided into five groups in which group I is control group samples in conventional resin and group II,III, IV & V contained 1 % and 2 % concentrations of ZnO & TiO₂ nanoparticles in heat cure acrylic resin. The processing and finishing of the models were done. Flexural strength was measured using a universal testing machine and surface hardness using a Rockwell hardness testing machine.

Results: The minimum SH reported was 101.7 HRM while FS was 81.1 MPa and maximum was 118.7 HRM and 131.8 MPa respectively. The results showed that group IV containing 1 % TiO₂ nanoparticles showed the highest surface hardness values whereas the flexural strength was highest in group II containing 1 % ZnO nanoparticles. The analysis of variance showed a *p* value of <0.001 which was statistically highly significant.

Conclusion: Nanocomposite denture base resins modified with ZnO & TiO₂ nanoparticles have more flexural strength and surface hardness than conventional denture base resin.

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Clinical implication: The hardness of a denture base material can be increased by adding these nanoparticles for long term use in oral cavity and in cases prone to denture fracture.

1. Introduction

Nano-dentistry is the perfect example of using nanoscale structures to maintain and improve oral health with different diagnostic and treatment approaches. Nanorobots and nanocapsules can be installed into the gingiva of the patient and for drug delivery. Nanoparticles are used in bone treatment and can be incorporated into vinylpolysiloxanes to enhance model pouring with precision and fewer marginal voids [1] or even in the denture bases to modify the properties [2]. Recent experiments have shown that nanoscale reinforcements are bringing a new era in improving the properties of Polymethylmethacrylate (PMMA). Nanoparticles are structures which are roughly the size of 2 or 3 atoms (10^{-9} m). These nanostructures can be developed with the help of “top-down” or “bottom-up” techniques. The “top-down” technique involves the assembly of these structures at a micron-scale. The “bottom-up” technique helps in fabricating these structures. The various structures developed by this method are: (1) nanotubes (2) nanopore (3) nanoshells (4) quantum dots [2,3]. These can be used in dentistry to enhance the quality of the dental material and provide enhanced prosthesis for rehabilitation.

One of the most common treatment modalities in the rehabilitation of completely edentulous patients are dentures. But these conventional dentures are prone to fracture easily. It was discovered that over the first three years, almost 68 % of complete dentures are susceptible to fracture. This could be endorsed by either masticatory force or dropping a denture [4]. To prevent fracture of the dentures, the thickness of acrylic resin in susceptible regions was increased or reinforced [5].

Many methods have been suggested to increase the mechanical properties of the material for the denture base either with the use of high-impact resins or with chemical modifications and polymerization cycle changes. The most reasonable and economical method to reinforce the resin is the addition of fillers [6]. Current dental research attempts are focused on the horizons of nanotechnology, and extending to examining its potential applications, and figuring out how to use it for future therapeutic gains. By altering the size of the filler to the nanometer level, a new material known as a nanocomposite is produced, and this led to the idea of utilizing nanoscale reinforcing agents in conventional materials, thus resulting in enhanced material with advanced favourable physical and mechanical properties [3,5].

Development focuses mainly on nanoparticles (NPs) as they exhibit excellent characteristics like shape, size, and composition and can enhance existing properties of resins and polymers [2]. Because of their ability to withstand fatigue and rupture, biocompatibility, and anti-corrosion properties, nanoparticles have recently found increased application. Because of their larger surface-to-volume ratios and higher percentage of atoms at the grain boundary, nanoparticles are preferred over macroscopic materials. The nanoparticles reduce the filler size increase the materials compaction and improve the mechanical properties [7,8]. The mechanical effects of denture base acrylic resins mixed with nanoparticles such as silicon oxide (SiO_2), aluminium oxide (Al_2O_3), copper oxide (CuO), iron oxide (Fe_2O_3), and zinc oxide (ZnO) have been extensively investigated [9–11]. These investigations suggest that the fracture resistance and endurance of materials may be influenced by the size, shape, and concentration of nanoparticles [10]. These nanoparticles are incorporated in the resin matrix.

Recently researchers are more attracted towards two nanoparticles that have made a strong impression - zinc oxide and titanium dioxide. In order to improve denture base mechanical and physical qualities, TiO_2 is a widely utilized and researched nanoparticle. Alternatively, the incorporation of any NP as an additive has the potential to change the substrate's physical characteristics. To prevent any negative effects, it is advised to assess the impact of the additive on the mechanical properties of acrylic resin.

It has been noted that adding TiO_2 nanoparticles can alter the optical, chemical, and physical properties of a denture base polymer. Moreover, the antimicrobial qualities and photocatalytic potential of this substance encourage its integration into biomaterials. They offer endurance, great biocompatibility, and an attractive color [12]. Zinc oxide can be made to have different morphological, mechanical, and photochemical properties by altering its manufacturing technique or particle size. PMMA's color stability and flexion characteristics have been considerably enhanced by the addition of ZnO nanoparticles [9].

Recently few advanced research utilized X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR), to examine the structural and phase analyses in denture bases modified with nanoparticles. Energy-dispersive spectroscopy and scanning electron microscopy were also used to study microhardness, water absorption analyses, and thermogravimetric analysis/differential scanning calorimetry. Zinc oxide (ZnO) nanoparticles are added to heat-cured acrylic resin as antibacterial fillers to reduce the adherence of *Streptococcus mutans* (*S. mutans*) and mucin. These features were investigated, according to the most recent review of the TiO_2 -based PMMA nanocomposite for denture base, which included physical and mechanical tests such as sorption, solubility, and color stability as well as flexural, impact, and tensile testing [13].

Based on different studies, it can be inferred that the addition of filler materials can help in improving the physical & mechanical properties of the heat cure resins. These fillers need to be incorporated in various concentrations to produce a homogenous mix [10]. The filler materials and the resin matrix should have good contact points and interlocking for a superior bond. For this, coupling agents can be used [11]. However, studies conducted by Kmonkhantikul K et al., have recommended the use of low concentrations as very high concentrations lead to a decrease in properties of the matrix [14].

Polymethylmethacrylate (PMMA) is most commonly used as a denture material because of its biocompatibility, esthetics, accurate fit, intraoral stability, ease of fabrication, low cost, and the possibility of repair. However, it has a few limitations like polymerization

shrinkage, reduced impact strength, low fatigue resistance and flexural strength. Ever-changing trends in dentistry require a material which shows optimal mechanical performance. The material should be easy to finish, polish and should not cause scratching of the surface [15]. During chewing, the denture is subjected to bending forces. This can be due to high occlusal loading or the result of an uneven pattern of bone resorption, which can cause the denture base to become unsupported with varied unbalanced vectors of forces [16]. Flexural strength is a measure of this deformation. Another important property is surface hardness which relates to the wear of material that can take place over time due to chemical and mechanical wear of the denture caused by brushing, cleaning or poor denture care. This causes roughness and encourages microbial adhesion [12]. Along with all positive effect the nanoparticles have some undesirable properties and can cause toxicity, like the pulmonary toxicity, nanotoxicity of liver, kidney and spleen is largely dependent on physicochemical properties of nanomaterials such as particle size, surface charge, morphology, (sphere, rod, and spindle-shape) and aggregation potential. The effects of inhaled nanoparticles in the body may also include lung inflammation and heart problems [17].

Various previous studies had examined at various nanoparticles; however, there haven't been any comparison studies between these two (ZnO and TiO₂) nanoparticles in one investigation. Moreover, the surface hardness and strength of modified nanocomposite denture bases are not extensively researched. However, it is crucial to comprehend the behaviour of these newly conceptualized nanocomposite denture bases under various conditions. Thus, the present study was planned to evaluate the surface hardness and flexural strength of newly formulated nanocomposite denture base resin made by adding ZnO & TiO₂ nanoparticles in heat polymerized polymethyl methacrylate resin in concentrations of 1 % and 2 %. The null hypothesis formulated that there would be no difference in the surface hardness and flexural strength of the modified denture base material from conventional material.

2. Material and methods

This cross-sectional in-vitro study was carried out in the Department of Prosthodontics and Crown & Bridge, Institute of Dental Sciences, Bareilly. The study was carried out in pre-decided steps.

One of the commonly used commercial brands of heat-cured acrylic resin Acrypol R (Ruthinium group, Dental Manufacturing, Plot no. 204, New GIDC, Gundlav, Gujarat 396035, India) was used in the study. 10 nm size of ZnO and TiO₂ nanoparticles (Nano Research Lab, Jamshedpur, Jharkhand, India) were incorporated as reinforcing agents. These ZnO and TiO₂ nanoparticles were added in concentrations of 1 % and 2 % by weight.

2.1. Sample size-

G*power software version 3.0.1(Franz Faul universitat, Kiel, Germany) was used for power analysis. To yield 80 % power for determining significant differences a sample size of 100 samples was considered, with an effect size of 0.4468 [15] and a significance level of 0.05.

[Effect size $f = 0.4468$; α err prob = 0.05; Power (1- β err prob) = 0.95; Number of Groups = 5.
Output: Non-centrality parameter $\lambda = 19.9630240$; Critical F = 2.4674936; Numerator df = 4.
Denominator df = 95; Total sample size = 100; Actual power = 0.9545310].

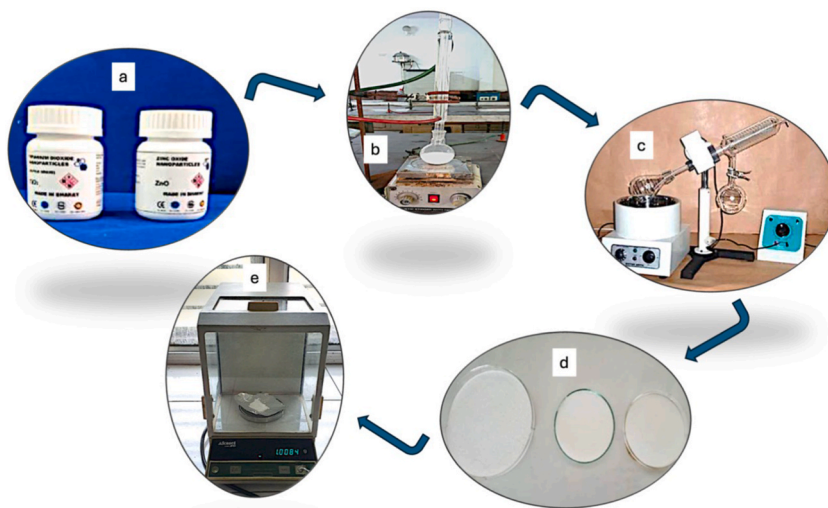


Fig. 1. Silanization of nanoparticles – a- Nanoparticles – ZnO and TiO₂; b- Magnetic stirrer; c- Rotary Evaporator; d- Silanized Nanoparticles; e- Weighing of Nanoparticles.

2.2. Silanization of nanoparticles

Initially, to enhance the ZnO and TiO₂ nanoparticle's wettability with the resin matrix through the formation of a reactive group on their surface, the silanization process was carried out individually for each type of nanoparticles. The nanoparticle powder of 99.9 % purity, with $8 \pm 3 \text{ m}^2/\text{g}$ surface area and an average particle size of 10 nm, (Nano Research Lab, Jamshedpur, Jharkhand, India) was saline-treated using the saline coupling agent 3-trimethoxysilyl propylmethacrylate (Sigma Aldrich, U.S.A). This procedure was accomplished by mixing 25 g of ZnO and TiO₂ nanoparticles separately with Methoxytrimethylsilane/acetone solution (Sigma Aldrich, U.S.A) respectively. 0.25–3 g of Methoxytrimethylsilane dissolved in 100 mL of acetone for 1 h with the help of a magnetic stirrer (SSU, LabPro, India) at 300 rpm for 60 min. Subsequently, the solvent was removed using a rotary evaporator under vacuum for 30 min at 60 °C and 150 rpm, followed by heating for 2 h at 120 °C. Next, the treated powder was allowed to cool to 28 °C. (Fig. 1- a-e).

2.3. Preparation of heat-cured acrylic resin and nanoparticle mixture

The silanized ZnO and TiO₂ nanoparticles and heat-cured acrylic resin Acrypol R (Ruthinium group, Dental Manufacturing, Plot no. 204, New GIDC, Gundlav, Gujarat 396035, India) were pre-weighed using an electronic balance (Afcoset, Fine Electronic Systems, India) with an accuracy of 0.0001 gm so that the Nano-filler concentrations were 1 % and 2 % by weight. Pre-weighed Nano-fillers powder with two concentrations (1 % and 2 %) were added individually to the heat-cured acrylic resin powder and thoroughly mixed using an electric mixer to produce a uniform blend and ensure that the homogeneous mix [18,19].

2.4. Sample fabrication and group division-

A conventional heat-cure acrylic was used as main component of the matrix and TiO₂ and ZnO nanoparticles were used as agents for reinforcing the matrix. 120 samples were made for the study. These were allotted into five groups (n = 20/group) based on the concentration and type of the nanoparticles. Group I- Heat cure conventional acrylic resin as a control group; Group II- Modified heat cure nanocomposite acrylic resin containing 1 wt% ZnO; Group III- Modified heat cure nanocomposite acrylic resin containing 2 wt% ZnO; Group IV- Modified heat cure nanocomposite acrylic resin containing 1 wt% TiO₂; Group V- Modified heat cure nanocomposite acrylic resin containing 2 wt% TiO₂.

For the fabrication of samples, a metal master die (MMD) was made. To obtain a specific size metal master Computer numerical control (CNC) manufacturing method was used. Initially, the designing of the die was done with the help of advanced software (Meshmixer Software -version 3.5, Autodesk, Inc./USA) a virtual die was designed with a specific dimension 65mm × 10mm × 3.3 mm (ADA specification No. 12) for flexural strength (ISO 20795–1:2013) and 30mm × 10mm × 3 mm for surface hardness (ISO/TS 19278:2019) in length, width, and thickness, respectively [20]. The STL files of the designed die were transferred to a CNC-enabled computer. CNC is a manufacturing technique that uses computer software that has been preprogrammed and incorporated into the equipment to automate the control, movement, and precision of machine tools. CNC is commonly used in manufacturing for machining metal parts. The milling of the metal master die was done in a cobalt-chromium alloy disc (98 × 10mm) (Maxidon Dental - 800 Enterprise Dr. Suite 101, Oakbrook, IL, 60523). Four dies were made of each type, of the same dimensions to speed up the work. The accuracy of the dimensions of the dies was verified with a micrometre at three locations for each dimension to within a 0.05 mm tolerance for width, height, and thickness. These MMDs were duplicated in modified acrylic resin. For duplication, the MMDs were

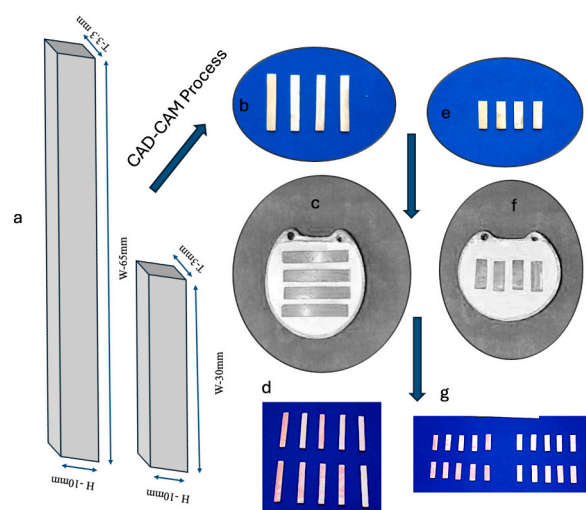


Fig. 2. Representative flow chart showing fabrication of samples-a- CAD process of designing metal master dies; b and e milled metal dies; c and f-Flasking of metal dies; d and g-acrylic resin samples with modified with ZnO and TiO₂. [b-c-d- Samples for Flexural Strength; e-f-g- Samples for Surface Hardness] [H- height; W-width; T-thickness].

invested in a flask with dental stone (Neelkanth dental plaster class-2 Pvt. Ltd Mumbai, India) in Hanau flask (Hanau Eng. Co. Buffalo, NY, USA) using the conventional compressive mould technique. The MMDs were invested in the flask after complete setting, following the cooling of the flask segments were separated and the metal master dies were removed using air pressure. The conventional and modified heat cure nanocomposite acrylic resin were used for packing and sample fabrication. The powder and liquid were mixed in ratios of 3:1 and packed in the dough stage in the mould. Once the dough stage was reached, the mix was packed into a stone mould coated with separating media. The flask's upper and lower members were closed together so that metal-to-metal contact was achieved. The hydraulic bench press was used to apply the pressure slowly to allow the uniform and complete spread of the dough in the mould space. The flask was held under a Hydraulic press (Dental Hydraulic Press-Sirio Italy- Unident Instruments INDIA PVT LTD) under a 1250kgf load for 5 min and then transferred to a water bath. The hydraulic press was initially tightened to the surface of flask and then the pressure was increased to maintain the 1250 kgf load. Following this, the flasks were placed for curing the specimens in an acrylizer (Unident pvt ltd Cemented Road, Anand Parbat Industrial Area, New Delhi, India) at 74 °C for 2 h and then temperature was increased to 100 °C for 1 h. After the curing cycle was over, the flask was cooled for 30 min as bench curing, before deflasking, and the specimens were retrieved from the mould (Fig. 2a–g).

2.5. Finishing and polishing

After the retrieval of the samples, each strip was trimmed with acrylic bur taking care that the samples prepared so precisely were not damaged. The use of trimming stone and burs were limited to removal of flakes of acrylic (Acrylic Contouring & Finishing Kit -SHOFU DENTAL INDIA PVT. LTD) was used. The procedure implemented was unidirectional trimming with average speed 5000–7000 rpm. The main smoothing was done with the 120-grain size sandpaper with continuous water cooling. For polishing, the lathe was fitted with bristle brush, later it was coated with pumice and glossy shiny surface was obtained by a wool brush with polishing agent by using lathe at a low speed (1500 rpm), taking care that heat was not generated, by using cool water to avoid distortions of samples. After this the samples were remeasured to check their dimensions; if any discrepancy was found, the specimens were discarded, and the process was repeated. The entire process was performed by a trained technician who was blinded from the research under the guidance of the primary researcher. Each sample was soaked in distilled water for 48 h at 37⁰ C, before their testing.

2.6. Flexural strength test

The specimens were placed on the universal testing machine (UTM- AMT-50SC, New Delhi, India) with a 50 mm distance between the 2 supports. The 3-point bending test was used to determine the flexural strength by applying a load of 50 kg-force (kgf) at 5 mm/min, in the midpoint of the sample until the specimen fractured. The load which causes the fracture of sample was recorded for every specimen. (Figure -3-a).

It was calculated by the formula:

$$\text{Flexural Strength} = 3WL/2bd^2$$

where,

W is the load, L is the length of support, b is width of specimen and d is thickness of specimen.

2.7. Surface hardness test

The Rockwell Hardness Testing Machine (RHT S-8, VMC R01, Delhi) was used to conduct this test. Rockwell Hardness Number was

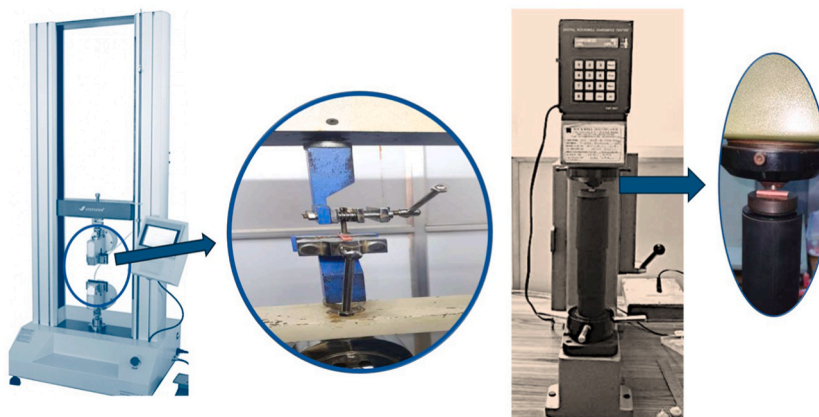


Fig. 3. Testing of samples-a- Universal Testing Machine for Flexural Strength – insight showing sample position.; c- Rockwell Hardness Testing Machine – insight showing sample position.

obtained digitally by applying a load of 60 g-force (gf) by using diamond pyramid for 20 s of indentation time.

All the data was entered on a MS EXCEL spreadsheet and subjected to statistical analysis using SPSS Software 21 (Figure -3-b).

2.8. Statistical analysis

All the data was entered on a MS EXCEL spreadsheet and subjected to statistical analysis using SPSS Software 21. The results were presented as mean and standard deviation. One-way ANOVA and Turkey Post hoc LSD was used to find significant difference in between the groups. A P-value of less than 0.05 was taken as statistically significant and that less than 0.001 as highly statistically significant.

3. Results

The Mean Surface Hardness for all the groups were statistically analyzed and tabulated (Table 1 and Fig. 4). The mean hardness for Group IV was maximum (114.93 ± 2.34) HRM ranging from (112.2–118.7). It was followed by Group II (112.42 ± 1.77) HRM, then Group I (108.93 ± 1.42) HRM and then Group V (105.54 ± 1.07) HRM. The lowest value was seen in Group III where the mean was (103.58 ± 1.32) HRM. The statistical analysis of the surface hardness between all the groups by One-Way ANOVA was done. The results showed highly significant differences in all the tested groups with a P value of less than 0.001. The mean values had statistical highly significance. The results were in order of Group IV > Group II > Group I > Group V > Group III.

The values for Mean Flexural Strength were tabulated (Table 2 and Fig. 5). The statistical analysis showed mean flexural strength for Group II was maximum (130.44 ± 1.85) MPa in the range of (125.5–131.8), followed by Group V (113.46 ± 1.37) MPa, then Group IV (113.42 ± 0.96) MPa and Group I (92.02 ± 0.80) MPa. The lowest value was seen in Group III (82.56 ± 1.19) MPa. The statistical analysis of the flexural strength between all the groups by One-Way ANOVA was done. The results had significant difference in all the tested groups with a P value of less than 0.001. The mean values had highly statistically significant. The results were in order of Group IV > Group II > Group I > Group V > Group III.

The statistical analysis of the flexural strength between all the groups by One-Way ANOVA was done. The highest mean was shown by Group II with a value of 130.44 MPa. The lowest mean was of Group III with the value of 82.566 MPa. The results had significant differences in all the tested groups with a P value of less than the mean values had high statistical significance. The statistical analysis among the various groups done by One-Way ANOVA showed a mean of 3630.441 when compared between all the groups. The comparison within groups showed a mean of 1.660. The P-value showed high statistical significance between the tested groups.

Post Hoc LSD test was done for multiple comparisons of mean surface hardness in between the different groups. The mean difference between Group IV and Group III was the highest. The P value of all groups was <0.001 which was highly statistically significant except for between the Group III and Group V which showed no statistically significant difference (Table 3). The mean difference for Flexural strength between Group II and Group III was the highest (47.8800). The P value of all groups was <0.001 which was highly statistically significant except for between Group IV and Group V which showed no statistically significant difference (Table 4).

4. Discussion

The longevity of the prosthesis is directly related to the properties of denture base materials. The material used for fabrication of denture should be biocompatible and stable in the oral cavity. It should be able to bear the masticatory load when in function and parafunction, should have good bonding with the available denture teeth and must be able to resist fracture on the drop of the prosthesis. Titanium dioxide and ZnO nanoparticles are nowadays recommended to be added in denture base resins to enhance these properties. ZnO and TiO₂ exhibit excellent biocompatibility and durability. Many studies have demonstrated zinc oxide nanoparticle incorporation has improved the mechanical traits of PMMA resin matrix [9].

Table 1

Descriptive statistics of surface hardness and flexural strength among various groups.

GROUP	N	Mean	Std. Deviation	Std. Error	95 % Confidence Interval for Mean		Minimum	Maximum
					Lower Bound	Upper Bound		
SURFACE HARDNESS (in HRM)								
GROUP I	10	108.93	1.42	0.451	107.9	109.95	106.2	110.6
GROUP II	10	112.42	1.77	0.56	111.15	113.68	107.8	114
GROUP III	10	103.58	1.32	0.418	102.63	104.52	101.7	105.7
GROUP IV	10	114.93	2.34	0.741	113.25	116.6	112.2	118.7
GROUP V	10	105.54	1.07	0.339	104.77	106.3	103.9	107.2
FLEXURAL STRENGTH (in MPa)								
GROUP I	10	92.02	0.8	0.254	91.44	92.59	90.8	93.5
GROUP II	10	130.44	1.85	0.585	129.12	131.77	125.5	131.8
GROUP III	10	82.56	1.19	0.378	81.71	83.42	81.1	84.6
GROUP IV	10	113.42	0.96	0.304	112.73	114.1	112	115
GROUP V	10	113.46	1.37	0.433	112.48	114.44	111.6	115.8

*P < 0.001 statistically highly significant.

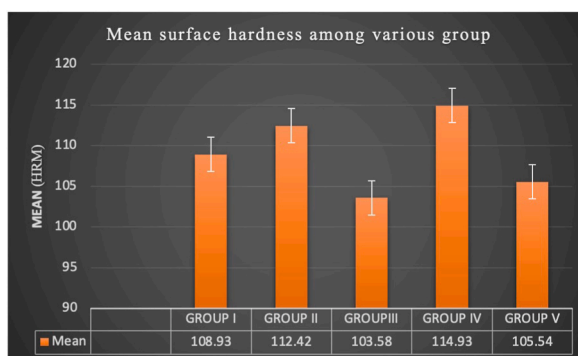


Fig. 4. Mean surface hardness among various group.

Table –2

Comparison based on surface hardness and flexural strength among various groups by one way ANOVA.

Surface Hardness (in HRM)					
	Sum Of Squares	Df	Mean Square	F-Value	P-Value
Between Groups	881.822	4	220.456	81.104	0.000*
Within Groups	122.318	45	2.718		
Total	1004.14	49			
Flexural Strength (in MPa)					
	Sum Of Squares	Df	Mean Square	F-Value	P-Value
Between Groups	14521.765	4	3630.441	2186.706	0.000*
Within Groups	74.71	45	1.66		
Total	14596.476	49			

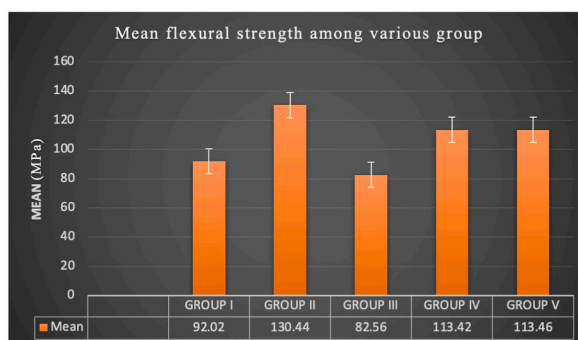


Fig. 5. Mean flexural strength among various group.

High concentrations typically had a detrimental effect while low amounts had positive effect [11,21]. Furthermore, it was noted that nanoparticle concentrations higher than 7 % may result in a significant alteration in the color and strength of the nanocomposite [21,22]. Thus, 1 % and 2 % were chosen to relatively represent adequate concentrations without affecting other properties.

In the present work, the efforts were taken to determine the effect of adding these nanoparticles on the surface hardness and flexural strength of the nanocomposite denture base resin and compared it with the conventional resin. The study's results rejected the null hypothesis as it was revealed that the surface hardness and flexural strength of the modified nanocomposite denture base resin were more than the conventional resin. There were statistically significant differences in values of surface hardness and flexural strength between the groups.

Flexural fatigue occurs in a denture due to continuous bending and flexing of the material. Here the load applied is so small that it does not cause any changes in a single application but gradually subjecting the material to such load leads to creating micro-cracks on the surface. These cracks then continue to grow and weaken the structure from the inside. After a certain limit when the final loading cycle exceeds the mechanical capacity of the sound portion, fracture of the material takes place. Midline fractures are a perfect example of these types of flexural forces. These can also occur due to sudden blow to the denture or accidental dropping while coughing or sneezing [23,24]. In these circumstances, increasing the bulk is not an option as leads to gagging and dislodgement of the denture. It can also hinder in the movements of the mandible and cause problems in speech. Factors like these have led to the need of developing

Table 3
Intergroup comparison based on surface hardness among various groups by Post hoc test.

Dependent Variable		Mean Difference (Variable 1- Variable 2)	Std. Error	P-VALUE	95 % Confidence Interval	
Variable 1	Variable 2				Lower Bound	Upper Bound
GROUP I	GROUP II	-3.4900 ^a	0.7373	0.000	-4.975	-2.005
GROUP I	GROUP III	5.3500 ^a	0.7373	0.000	3.865	6.835
GROUP I	GROUP IV	-6.0000 ^a	0.7373	0.000	-7.485	-4.515
GROUP I	GROUP V	3.3900 ^a	0.7373	0.000	1.905	4.875
GROUP II	GROUP V	8.8400 ^a	0.7373	0.000	7.355	10.325
GROUP II	GROUP IV	-2.5100 ^a	0.7373	0.000	-3.995	-1.025
GROUP II	GROUP V	6.8800 ^a	0.7373	0.000	5.395	8.365
GROUP III	GROUP IV	-11.3500 ^a	0.7373	0.000	-12.835	-9.865
GROUP III	GROUP V	-1.9600	0.7373	0.011	-3.445	-0.475
GROUP IV	GROUP III	11.3500 ^a	0.7373	0.000	9.865	12.835
GROUP IV	GROUP V	9.3900 ^a	0.7373	0.000	7.905	10.875

^a P < 0.001 statistically highly significant.

Table 4
Intergroup comparison based on flexural strength among various groups by Post hoc test.

Dependent Variable		Mean Difference (Variable 1- Variable 2)	Std. Error	P-VALUE	95 % Confidence Interval	
Variable 1	Variable 2				Lower Bound	Upper Bound
GROUP I	GROUP II	-38.4260 ^a	0.5762	0.000**	-39.587	-37.265
GROUP I	GROUP III	9.4540 ^a	0.5762	0.000**	8.293	10.615
GROUP I	GROUP IV	-21.4000 ^a	0.5762	0.000**	-22.561	-20.239
GROUP I	GROUP V	-21.4400 ^a	0.5762	0.000**	-22.601	-20.279
GROUP II	GROUP III	47.8800 ^a	0.5762	0.000**	46.719	49.041
GROUP II	GROUP IV	17.0260 ^a	0.5762	0.000**	15.865	18.187
GROUP II	GROUP V	16.9860 ^a	0.5762	0.000**	15.825	18.147
GROUP III	GROUP IV	-30.8540 ^a	0.5762	0.000**	-32.015	-29.693
GROUP III	GROUP V	-30.8940 ^a	0.5762	0.000**	-32.055	-29.733
GROUP IV	GROUP V	-0.0400	0.5762	0.945 ^b	-1.201	1.121

^a P < 0.001 statistically highly significant.

^b P > 0.05 statistically not significant.

materials with higher strengths which can in the long run [25].

Another important property of denture base material is the surface hardness, which helps in its durability. It's the measure of a material's resistance to various permanent shape changes after application of compressive force. It helps in finishing and polishing the denture bases. It also provides resistance to scratching during the cleaning of the prosthesis. The roughness and irregularities on the surface lead to the accumulation of plaque and stains which negatively affect the esthetics and biological traits of the material over time [6].

In the current study, the values of surface hardness were obtained using a Rockwell Hardness Tester. It is a scale based on the hardness of the material after indentation. It measures the depth of penetration made by a major load and compares it to a minor preload. It uses a diamond or a ball indenter. The hardness number is the distance between baseline and final depth measurements. The scale for testing acrylic material uses a 6.35 mm ball indenter. The main advantage of Rockwell Hardness Testing is its ability to display hardness values directly and the speed of testing. The values are denoted according to the scale HRM in which HR stands for Rockwell hardness and M denotes thermoplastic material.

The longevity of the prosthesis is directly related to the properties of denture base materials. PMMA is most used as denture base material due to its various advantages which include biocompatibility, light weight, good esthetics and inexpensiveness. However, it has few drawbacks like weak flexural strength, low impact and fatigue resistance and polymerization shrinkage. To overcome these disadvantages, different methods have been suggested such as Chemical modification by addition of copolymers or cross-linking agents and Incorporating fibres or metal inserts to act as fillers. The filler materials can be coated with chemicals to enhance the interfacial bonding and prevent the matrix and reinforcements from degradation. The promising filler particles at present are nanoparticles, which requires uniform dispersion in the polymer matrix. Many methods have been given in literature like melt mixing and in situ polymerization. Rong M et al. [26], used monomers of styrene to surround the particles. They used isotactic polypropylene as the matrix and added silicon dioxide particles which were first heated to remove any residual water. Another method by Yang F et al., used the in-situ polymerization in which the mixture was first dispersed in the monomer and then polymerized. The polymerization was done at a high temperature under nitrogen [27]. Hafizah N et al., used two techniques namely sonication & solution casting to develop the nanoparticle-resin matrix which resulted in superior physical properties. Strong interfacial interactions between the polymer and filler were observed which restricted the mobility of polymer chain and resulted in higher modulus [28].

In this study, [3-Methoxytrimethyl silane], was used to coat the nanoparticles by the process of silanization. The silanization of nanoparticles was carried out beforehand as it improved the bond between the filler and the resin [14]. This was like the study by

Alzayyat S et al., in which they silanized the silanized nano-SiO₂ and combined it with PMMA resulting in increased flexural strength and elastic modulus. They also help in controlling the polymerization shrinkage [29].

In the present study also, the mean flexural strength increased with addition of salinaized nanoparticles of ZnO and TiO₂. The highest value was recorded in Group II (1 % ZnO) 130.44 MPa. These reason for this can be attributed to the homogenous dispersion of the nanofillers in the matrix. The silanization created a high contact area which increased the load transfer and cross-linking. The lower concentrations helped in non-agglomerated nanoparticles which increased mechanical interlocking and thus the flexural properties [10]. These values were in correlation with the study conducted by Vikram S et al., in which the flexural strength was significantly increased (91.31 ± 1.15 MPa) in the group with 1.4 % nanoparticle addition in comparison to the control group in which a mean value of 61.36 ± 4.91 MPa was observed [9].

The lowest values were obtained in the group containing 2 % zinc oxide nanoparticles with mean as 82.56 MPa. The values were less in comparison to the control group. This was caused due to aggregation of the fillers when the concentration was increased which led to incomplete distribution because of higher density of zinc oxide nanoparticles. This decrease could also be because of high amount of residual unreacted monomer as the nanoparticles act as impurities and interfere with the polymerization [11]. The results contrasted with the study done by Harini P et al., which showed increase in mean flexural strength of denture base material with increasing concentration of TiO₂ nanoparticles (1 %, 2 % and 5 %) [30]. Another study conducted by Nazirkar G et al., stated that flexural values increased by adding 0.5 % titanium dioxide nanoparticles but with the increase in concentration to 1 % the results showed a downward trend [31]. 1 % TiO₂ and 2 % TiO₂ showed lesser mean strength than the group containing 1 % ZnO. 1 % titanium dioxide has better strength values as compared to samples containing 2 % TiO₂ but it showed a statistically insignificant difference between the two when intergroup comparison was done. This may be due to their higher pre-existing nanomechanical properties. It has greater cross-linking with the polymer which leaves less residual monomer. So even if the concentration is increased, the surface area remains the same [16,32]. The increase in the strength of 1 % ZnO when compared with 1 % TiO₂ could be because of a lack of adhesion between the metal oxides of titanium and matrix.

Surface hardness was tested using a Rockwell Hardness Testing machine in which a load of 60 g force was applied at a 20-s interval indentation time. The highest values were seen in Group IV (1 % TiO₂) with the mean of 114.93 ± 2.34 HRM in comparison to remaining groups. It could be due to improved matrix cross-linking because of interfacial bonds and increased surface area. This resulted in decreased residual monomer content and reduced porosities. This in turn increases the hardness of the resultant matrix. The increase in properties of surface hardness was mainly attributed to the reduction in the size of the particles which decreased the stiffness of the matrix and reduced the free mobility and volume [14]. Other articles reported that the nanoparticles in PMMA resin bear most of the applied load while the resin matrix aids in structural integrity and distribution of the load, which ultimately reduces crack propagation [33].

The hardness values increased with the addition of 1 wt% TiO₂ nanoparticles but decreased when the concentration was increased to 2 wt%. This reduction in the values were like a study done by Ahmed MA et al., in which higher values at 1 wt% were attributed to formation of a connection between nanofillers and matrix due to reduction in particle size. It provided greater wear resistance but showed decreased hardness when concentration exceeded 5 wt% because the resin cannot be distributed evenly between filler particles [34].

The lowest values were shown by the group containing 2 % zinc oxide with mean of 103.58 ± 1.32 HRM. It may be due to high filler content, which weakens the polymer chain mobility and causes microporosities. Excess fillers act as impurities and cause a decrease in surface properties. These fillers, due to high density accumulate water on surface which further causes large clusters to form on the surface. These defects are not present in lower concentrations [34,35]. Alrahlah A et al., also showed an increase in surface hardness values of the PMMA matrix after adding TiO₂ nanoparticles [36]. The results of the present study disagreed to the study conducted by Abdelraouf R et al., where the surface hardness values after the addition of TiO₂ nanoparticles didn't increase. This might be due to low concentration of nanoparticles did not provide a homogenous dispersion in the resin matrix and on spectrophotometry analysis, the samples displayed honeycomb pattern with an irregular, porous structure [32].

The present findings were also consistent with the theory that the addition of TiO₂ nanoparticles to acrylic resin improved its mechanical properties by establishing a strong interfacial interaction with the resin [34]. Furthermore, Harini et al.'s findings, that reinforced PMMA with varying TiO₂ NP concentrations would provide higher flexural strengths than those of conventional PMMA confirmed the present findings [30]. Numerous investigations that incorporated nanoparticles, such as ZrO₂ [37] and AgNPs [38] into heat-cured acrylic resins at varying ratios came to the conclusion that the addition of nanomaterials might enhance the mechanical properties of the resins without having any negative effects.

However, it was also observed that the results were concentration-dependent. The groups containing 1 % nanoparticles showed superior results. As the concentration increased to 2 %, the results showed a decrease in the values of strength and hardness. Thus, it can be deduced that there is a significant effect of nanoparticle addition on the mechanical properties of PMMA heat cure resins. The hardness of a denture base material indicates how easily it can be abraded. These nanoparticles could be used to strengthen the denture bases to use in the oral cavity for a longer duration of time. However, a balance between superior mechanical properties and esthetics of the denture base needs to be considered.

4.1. Limitations-

Even though the study was conducted in meticulously designed steps but there were still certain limitations of the study that included the in vitro nature of the study. The oral cavity has a complex environment with variations in temperature, humidity, acidity and stresses. The results can be influenced by a multitude of other factors, including the presence of saliva, dietary habits,

neuromuscular force, parafunctional behaviours, and other cleansing methods. As such, the current findings can only be viewed as a promising beginning point for further research. In the present study, the limited variations in concentrations of the nanoparticles with no effect of combination particles was performed, ageing effects on the flexural strength and hardness have not been evaluated, it is recommended for future research to assess effects of these variations. Also, other properties like roughness, surface energy and adhesion and film formation on this modified denture base material should be assessed. The electric mixer was used, it suggested to use other mixing method for mixing of nanoparticles with heat cure acrylic resins such as mortar and pestle, high energy ball milling. Furthermore, the design of denture was not reflected by the use of a straight rectangular sample. Hence, further in-vivo studies are required to substantiate the results obtained. Longer incubation periods are also required to simulate the duration of use of the prosthesis to check for the mechanical traits of the resin-filler matrix. It is recommended to conduct research to determine the dispersion of nanoparticles inside the matrices and the failure modes of the 2 nanocomposite materials.

5. Conclusion

Within the limitations of this study, it was concluded that heat cure acrylic resin when combined with nanoparticles showed an increase in the flexural strength and surface hardness. Mean flexural strength was reported highest when 1 % zinc oxide nanoparticles were added to acrylic resin. The lowest values obtained were of group with 2 % zinc oxide ($P < 0.001$). Mean surface hardness was highest in the group containing combination of heat-cured PMMA resin and 1 % titanium dioxide nanoparticles. Lowest values were observed in 2 % zinc oxide group. ($P < 0.001$).

CRedit authorship contribution statement

Anagha Waghmare: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Chandana Nair:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Anuj K. Shukla:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Mudita Chaturvedi:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Funding acquisition, Formal analysis. **Tushar Vitthalrao Bhagat:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Funding acquisition, Formal analysis, Conceptualization. **Ebrahim Fihaid Alsubaiy:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Funding acquisition, Formal analysis, Conceptualization. **Ghazala Suleman:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Mohasin Abdul Khader:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Funding acquisition, Conceptualization. **Saurabh Chaturvedi:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Ethical declaration-

This cross-sectional in-vitro study was carried out in the Department of Prosthodontics and Crown & Bridge, Institute of Dental Sciences, Bareilly. (IEC/128/2022/09).

Data availability statement

Data will be made available on request.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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