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Evaluation and comparison of the flexural strength of heat polymerised PMMA, 3D printed PMMA and titanium-dioxide reinforced 3D printed PMMA denture base material.

[1]N. Suganya, Postgraduate, Department of Prosthodontics and Crown and Bridge, Vokkaligara Sangha Dental College and Hospital, Rajiv Gandhi University of Health Sciences (RGUHS), Bengaluru, India.

[2]Archana Shetty, Reader, Department of Prosthodontics and Crown and Bridge, Vokkaligara Sangha Dental College and Hospital.

[3]Nalinakshamma. M, Vokkaligara Sangha Dental College and Hospital.

[4]N. Manjula, Vokkaligara Sangha Dental College and Hospital.

[5]Savitha. P. Rao, Vokkaligara Sangha Dental College and Hospital.

Corresponding Author: N. Suganya, Postgraduate, Department of Prosthodontics and Crown and Bridge, Vokkaligara Sangha Dental College and Hospital, Rajiv Gandhi University of Health Sciences (RGUHS), Bengaluru, India.

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Abstract

Background: Polymethylmethacrylate (PMMA) is a commonly used material to fabricate conventional complete dentures due to its availability and reasonable cost. However, the inherent limitations of PMMA include polymerization shrinkage, deterioration of mechanical properties, poor antibacterial properties and cumbersome fabrication process. With the development of digital dentistry, CAD-CAM and 3D printing has shown potential in providing a promising solution for denture fabrication.

AIM: This study was conducted to evaluate and compare the flexural strength of heat polymerized PMMA, 3D printed PMMA and TiO_2 reinforced 3D printed PMMA denture base material.

Materials and methodology: 30 samples were evaluated in this study with ten samples in each group which were divided as follows,

Group 1: Heat polymerized specimens with the dimensions of length 65mm, width 10mm and height 3mm were fabricated.

Group 2: 3D printed PMMA specimens with same dimensions were printed.

Group 3: TiO2 nanoparticles reinforced 3D printed PMMA with the same dimensions were printed. A 3-point flexural test was used to test the flexural strength of the specimens.

Results: The values obtained were statistically analyzed using ANOVA with post hoc Bonferroni test. Group 1 (99.52 ± 14.49) reported maximum flexural strength among the three groups followed by Group 3 (81.08 ± 4.98) and Group 2 (66.34 ± 6.97).

Conclusion: The flexural strength of heat cured PMMA denture base material is the highest followed by Titanium dioxide nanoparticles reinforced 3D printed PMMA denture base materials. The least flexural strength among the three groups was reported by unaltered 3D printed PMMA specimens. The results showed that the flexural strength of 3D printed PMMA increased with incorporation of TiO_2 nanoparticles.

Clinical implication: Incorporation of 3D printed PMMA denture base materials with TiO_2 nanoparticles greatly improved the flexural strength of the denture base materials.

Keywords: 3D printing, PMMA, Titanium dioxide, nanoparticles, flexural strength.

Introduction

Complete dentures have been used for many years, and they are the gold standard for treating edentulism.[1] Complete tooth loss compromises aesthetics, phonetics and function in the orofacial region, leading to a lowered quality of life. As an indispensable part of a denture, the denture base plays a vital role in rehabilitating soft tissues, supporting artificial teeth, and enduring various intraoral stresses. Recent advancements in materials sciences and technology has brought about the advent of digital methods for denture base production, including 3D printing and Computer Aided Design/ Computer Aided Manufacturing.[2,3]

3D printed prostheses have various advantages over traditional Manu facturing as they have shorter pro duction cycles and higher precision, maximizing the comfort of patients with dentures. Light curing technology has been widely implemented in the field of prosthodontics accounting for nearly 75% of the 3D printing dental applications with light curing resins commonly used as fillers and restorative materials in stomatology. The most commonly used light curing resin in dental 3D printing applications is Poly Methyl metha cry late as it has various positive attributes such as low odor, low irritancy, good flexibility and low cost.[4-6] However, the inherent limitations of PMMA, such as large shrinkage rate during light-curing and inferior mechanical properties have hindered its extensive clinical application, motivating extensive research in this direction. [6]

Various studies have experimented with the use of different alternatives for polymerized fillers to enhance the mechanical and antibacterial properties of 3D printed denture bases. As revealed by several studies, PMMA nano composite based on functionalized TiO_2 nano particles have demonstrated enhanced mechanical and anti-bacterial properties.[7-9] Their large spectrum of antibacterial activity and non-contact biocidal action have led to their acceptance as the best alternative poly meric filler when seeking to improve antibacterial activity.

However, not much studies have substantiated the variation in physical properties of 3D printed dentures enhanced with TiO_2 nanofillers to conclusively report overall enhanced mechanical properties of these denture bases. Thus, the aim of the study is to evaluate the flexural strength of 3D printed PMMA and TiO_2 reinforced PMMA to compare with the flexural strength of conventional heat polymerized PMMA.

Materials and methodology

The specimens manufactured for the study were divided into three groups based on the materials used and mode of polymerization which are,

- Group 1 - Heat Polymerized PMMA denture base specimens (Control)
- Group 2 - 3D printed PMMA denture base specimens
- Group 3 - 3D printed PMMA denture base resin modified with TiO_2 nanoparticles.

Sample size estimation was done and ten samples were prepared in each group. The samples were designed to dimensions of 65* 10* 3 mm (American dental asso ciation specification no. 12) for evaluation of flexural strength of the samples (Figure 1). The preparation of the samples was done as follows

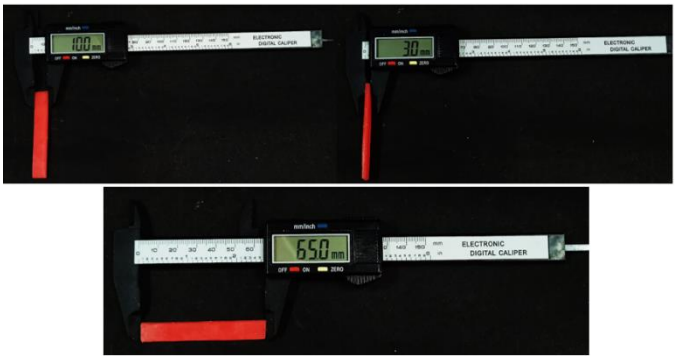


Figure 1: Wax pattern of the specimens with dimensions of 65*10*3 mm

Preparation of heat polymerized specimens

To fabricate heat-polymerized specimens, conventional compression molding procedures were followed. Metal molds were used to prepare wax specimens of required dimensions. Wax specimens were invested in stone within a metal flask before dewaxing (Figure 2). Follow ing dewaxing, slots measuring of about 65*10*3mm were obtained in the flasks which created space for the packing of heat cure acrylic resin denture base material. Heat-polymerized acrylic resin (DPI heat cure) was packed at the dough stage followed by bench press (Figure 3). Flasks were then immersed in a thermally controlled polymerization unit at room temperature and processed at 70°C for 90 minutes followed by 100°C for 30 minutes. After complete polymerization, the flasks were allowed to bench cool for a period of 24 hours and then the specimens were carefully deflasked and finished with a tungsten carbide bur to remove excess resins.

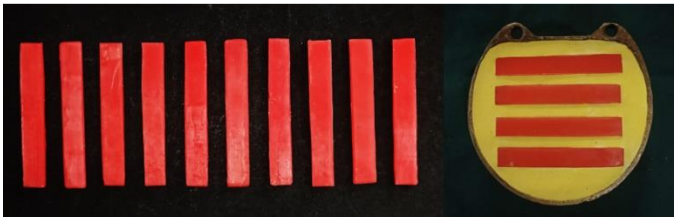


Figure 2: Wax pattern of the specimens before dewaxing

Figure 3: Heat-polymerized acrylic resin packed in the flasks after dewaxing

Preparation of 3D printed specimens

The specimen was designed as a rectangle with dimensions of 65*10*3 mm using computer aided design software (Exocad, Shining3D, China) and saved as a standard tessellation language (STL) file (Figure 4). Using the STL file, the 3D printing was conducted using an appropriate 3D printer (AccuFab-L4K, Shining 3D, China) with 3D printing denture base resin (Res Denture R2, Navadha, India) and digital light processing techno logy. The 3D printer was equipped with a laser light source with a wavelength of 450nm, layer height setup at 50µm and all specimens were oriented at 90˚ (Figure 5). The 3D printing process was followed by thorough washing of specimens in an isopropyl alcohol bath with 90% purity for 10 minutes. Using a low-speed rotary instrument, the support structure was removed and specimens were post-cured in a UV light polymerisation unit for 10 minutes. Then, specimens were finished with silicon carbide grinding paper sequentially (800,1500 and 2000 grit) and rinsed with water.

Figure 4: STL file of the specimens

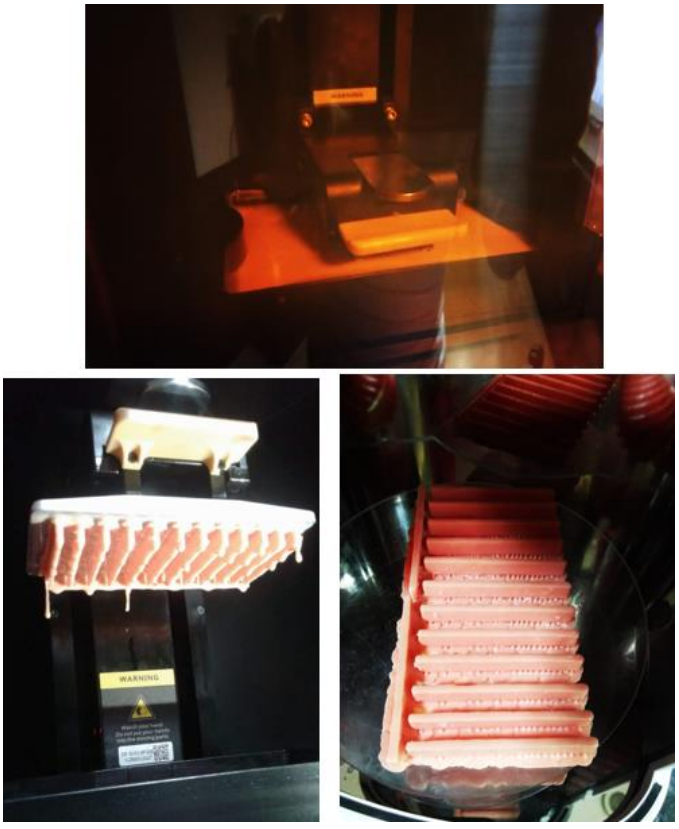


Figure 5: 3D printing process

PMMA - TiO_2 nanocomposite preparation

The composite mixture has been obtained through incremental additions of TiO_2 nanoparticles (30-50nm, Ultra nanotech, Bangalore) into the PMMA mixture. The new nano composite material contains 1% TiO_2 nano particles by weight. Nanocomposites preparation pro cedure consisted gradual incorporation of the appropriate amount of titania nanoparticles into PMMA solution under continuous magnetically ultrasonicated stirring (Remi 1 MLH) of resin for 30 minutes followed by mechanical mixing for 30 minutes at low frequency (Figure 6). The synthesized TiO_2 nanoparticles were evidenced by means of scanning electron microscopy (SEM)

The nanocomposite specimen bars of dimensions 65*10*3 mm were printed by DLP photo-curing 3D printing system.

Figure 6: Resin with added TiO2 nanoparticle was magnetically ultrasonicated stirred

SEM Characterization and EDX

Scanning electron microscopy has been utilized to study the morphology of the obtained composites. Samples were obtained by collecting droplets of resin with TiO2 and resin without nanoparticles which were cured in a

hot plate for 100˚C for 30 minutes to remove moisture (Figure 7,8). The energy dispersive X-ray spectroscopy has been employed for elemental analysis. The experiments were performed on JEOL EDS 2000 equip ment (15keV).

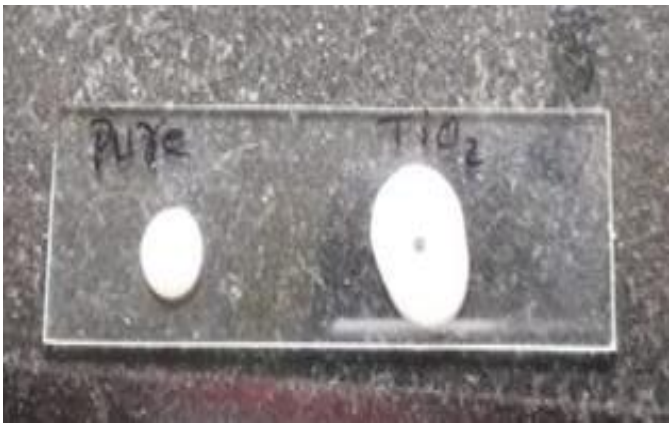


Figure 7: Droplets of the samples collected for SEM

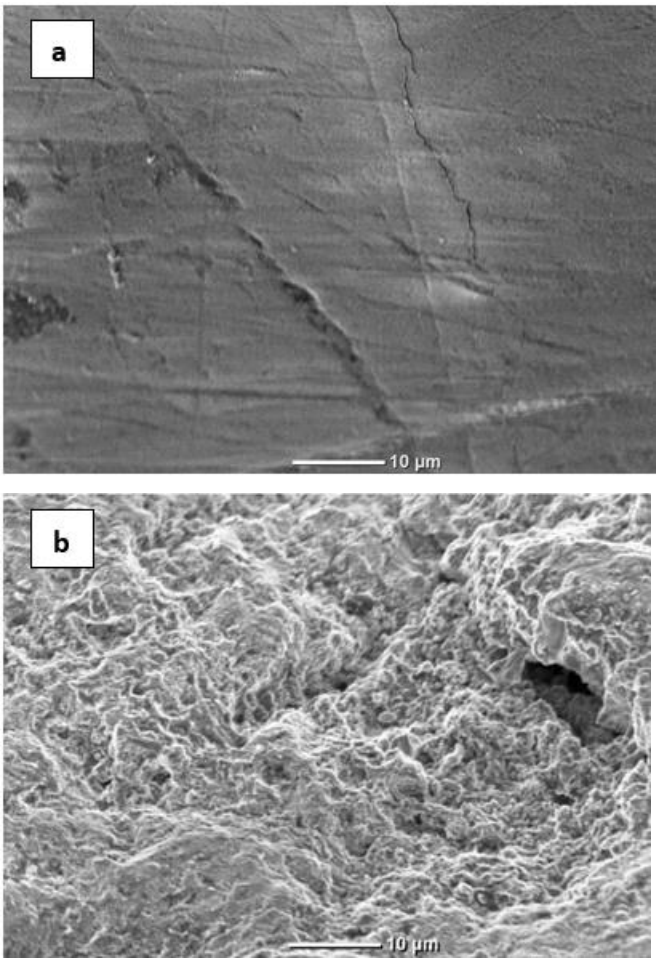


Figure 8: SEM images of resin a) without nanoparticles b) with TiO_2 nanoparticles

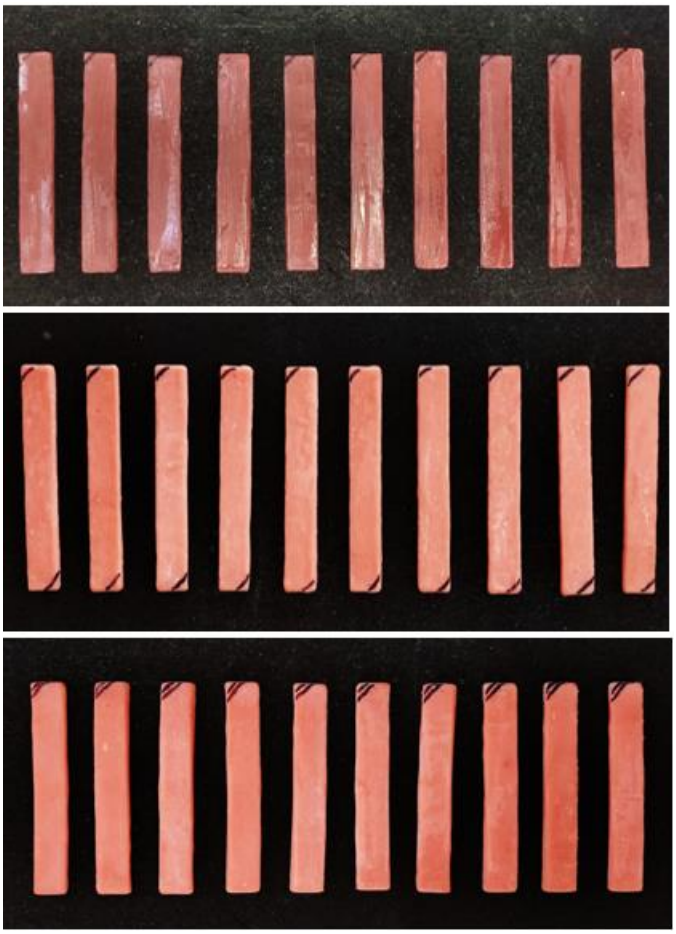


Figure 9: Group 1: heat polymerised PMMA specimens; Group 2: 3D printed denture base resin specimens; Group 3: TiO_2 nanoparticles incorporated in 3D printed denture base resin specimens

Flexural Strength testing

Flexural strength test was performed according to ISO standard 1567:1999. Prior to flexural strength testing, length, width and thickness of each specimen were mea sured with a digital Vernier caliper (Mitutoyo, Kawasaki, Japan) with a measuring accuracy of ±0.1 mm. Each sample was stored in water for 24 hours at 37°C prior to the three-point bending test (Figure 9). The specimens were subjected to flexural strength testing under three-point loading with a crosshead speed of 5 mm/min in a universal testing machine (Mecmesin Multi test 10-i). The flexural testing device consisted of a central loading plunger and two polished cylindrical

supports, 3.2 mm in diameter and 10.5 mm long. The distance between the centers of the supports was 50 mm. This dimension represents the space between the maxillary molars in a complete denture. The load was applied perpendicular to the Center of specimen strips until the deviation of the load-deflection curve and fracture of specimen occurred. Flexural strength was calculated by computer system associated with the machine using formula FS = 3 FL/($2bd^2$), where FS is flexural strength (MPa), F is the load or force at break (N), L is span of specimen between the supports, *b* is the width and *d* is the thickness (Figure 10).

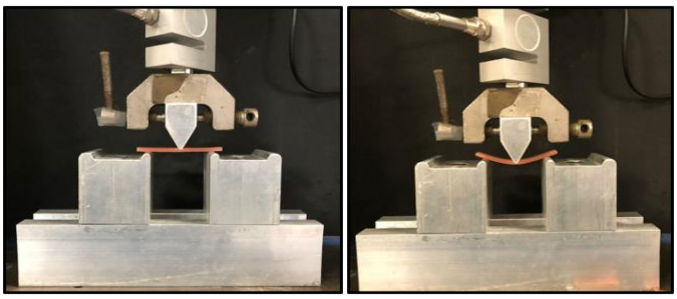


Figure 10: Flexural strength of the specimens tested using UTM

Results

SPSS (Statistical Package for Social Sciences) version 20. (IBM SPASS statistics [IBM corp. released 2011] was used to perform the statistical analysis. Data was entered in the excel spreadsheet. Descriptive statistics of the explanatory and outcome variables were calculated by mean, standard deviation for quantitative variables. ANOVA test was applied to compare the statistical difference of flexural strength among the groups with post hoc Bonferroni test for inter group comparison. The level of significance was set at 5%. There was a difference in the mean flexural strength between 3 groups. Data was subjected to a normality test (Shapiro-wilk test).

Data showed normal distribution. Hence parametric tests (ANOVA with post hoc Bonferroni) were applied.

Table 1: mean flexural strength of the samples.

Groups	N	Minimum	Maximum	Mean	S. D
A	10	78.83	123.50	99.52	14.49
B	10	52.26	76.30	66.34	6.97
C	10	73.52	87.83	81.08	4.98

Table no. 1 represents the mean flexural strength values between different groups. The test results demonstrate that the mean flexural strength of Group A is higher – 99.52 ± 14.49 followed by Group C- 81.08 ± 4.98 and Group B- 66.34 ± 6.97.

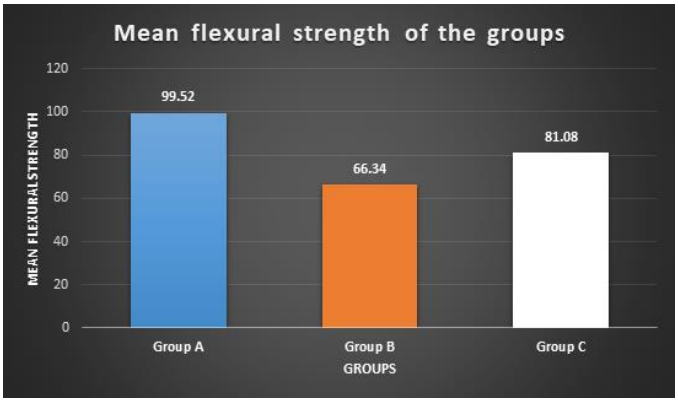


Table 2: comparison of the mean flexural strength of the samples using anova

Groups	N	Mean	S.D	F value	p value
A	10	99.52	14.49	29.72	0.001*
B	10	66.34	6.97		
C	10	81.08	4.98		

*Significant

Table no.2 represents the comparison of the mean flexural strength of samples using ANOVA test. ANOVA test showed statistically significant difference among the groups (p=0.001) with respect to flexural strength.

Table 3: inter group comparison using post hoc Bonferroni

	Mean Difference	p value
Group A V/s Group B	33.183	.001*
Group A V/s Group C	18.439	.001*
Group B V/s Group C	-14.744	.006*

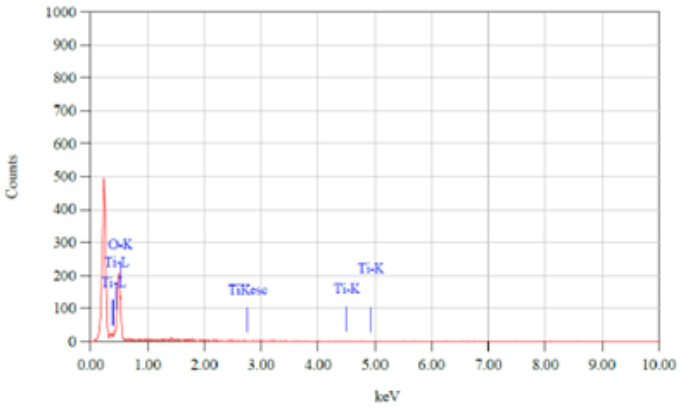
*Significant

Table no.3 represents the inter group comparison using post hoc Bonferroni test. There was a statistically significant difference found between all the groups- Group A v/s group B (p=0.001); Group A V/s Group C(p=0.001); Group B V/s Group C (p=0.006).

SEM and EDX Analysis

SEM analysis of the resin incorporated with Titanium Dioxide nanoparticles revealed uniform distribution of the nanoparticles. The microscopic morphology of the nanocomposites revealed a homogenized mixture of the resin with presence of tiny cracks and micropores in the matrix.

EDX analysis helped analyze the elemental composition of the Titanium Dioxide particles in which it was observed that titanium contributed to 59.93% of the mass of the particle and oxygen contributed to 40% of its mass. The graph peaks in EDX analysis reveal a good dispersion of the nanoparticles in the matrix.



Interpretation of results

The mean flexural strength of the three experimental groups has been tabulated in table 1. Heat polymerized PMMA denture base reports maximum flexural strength among the three groups with 3D printed PMMA denture base samples reporting the least. Addition of TiO_2 nanoparticles enhances the mean flexural strength of the 3D printed PMMA denture base samples significantly. Statistical analysis reveals that Heat cured PMMA denture base samples have significantly greater flexural strength than TiO_2 nanoparticles doped 3D printed PMMA denture base samples which have significantly greater flexural strength than 3D printed PMMA denture base samples.

Discussion

The materials of denture base should have excellent properties, such as stable chemical properties, good physical and mechanical properties, easy to polish, non-toxic and antibacterial properties. There are various methods for preparation of denture bases such as heat polymerization, 3D printing and milling with various advantages and disadvantages to each method. Three-dimensional printing is a modern technology for making 3D physical/analog construction, pre-created with a program for computer modelling digital prototypes. It has various applications in the field of prosthetic dentistry that has grown rapidly over the past few years as it saves time, manpower and ensures perfect marginal fit for the produced construction.

Few studies have reported that the mechanical properties of 3D printed PMMA denture bases have been found to be inferior to heat polymerized PMMA denture bases. The addition of TiO_2 nanoparticles has been reported to improve the antimicrobial and mechanical properties of the PMMA denture bases, improving the long term treatment outcome of the denture bases. This study was aimed to evaluate the variation in the flexural strength of 3D printed PMMA denture base samples with the addition of TiO_2 nanoparticles and compare it with the flexural strength of conventional heat cure PMMA denture base samples.

The null hypothesis of the study, which assumed that no difference would be found between the flexural strength of 3D printed PMMA denture base materials, TiO_2 doped 3D printed PMMA denture base materials and

heat cured PMMA denture base materials was rejected. The results of this study are similar to the results of Prpic et al and Dwairi et al who observed that the flexural strength of heat-polymerized PMMA was higher than that of 3D printed PMMA resin samples.[10,11] This could be attributed to the lower double-bond conversion of 3D printed PMMA resin denture bases which affects their mechanical properties. The mean flexural strength values of 3D printed PMMA denture base samples is 66.34 MPa which meets the ISO requirement of minimal flexural strength for denture bases (65 MPa).

The flexural strength values of different materials are representation of the collective measurement of com pressive, tensile and shear stresses of different materials.

The flexural strength of TiO_2 nanoparticles has been evaluated in various studies and it has been observed that the effect of addition of Titanium dioxide nano particles is highly dependent on the type of acrylics and concentration of nanoparticles. Studies by Sodagar et al and Han et al have concluded that there is an inverse correlation between the concentration of filler particles and the flexural strength of reinforced PMMA. It has been found that silanization of the TiO_2 nanoparticles and achieving good dispersion significantly improves the transverse strength, flexural modulus and ductility of the denture base materials.[12,13] Silanization of the nano particles and treatment with modifiers such as surfactants, proteins and different acids tend to lower the surface energy of the nanoparticles and prevent cluster formation and aggregation of the nanoparticles which has been reported to decrease the flexural bond strength of the denture base material.

Chara cterization of 3D printed PMMA denture-based materials doped with TiO_2 nanoparticles under SEM analysis reveals the uniform dispersion of the nano particles achieved in our study. Quantitative EDX analyses reveal a slight, yet continuous increase in TiO_2 fractions on the surfaces of various denture base resins supplemented with increasing fractions of TiO_2 which proved that the nanoparticles were available on the surface of the modified specimens. This could be attributed to the increase in flexural strength values of 3D printed PMMA denture base materials and also enhance the antimicrobial properties of the denture base samples.

The infiltration of TiO_2 nanoparticles in the matrix of 3D printed PMMA denture base resins reduces the mobility of the polymer chain due to strong interfacial interactions between the filler and the matrix. This improves the strength of the resin which is attributed to the reduced segmental motion of the polymer, inherent modulus of the nanoparticles and a strong interfacial bond at the filler-matrix interface.[14] This correlate to the result of this study where the 3D printed PMMA denture base materials reported improved strength when compared to unaltered 3D printed PMMA denture base materials.

Future studies can be conducted to evaluate other critical properties of denture base materials such as antibacterial efficacy, color stability, impact strength, viscoelastic behaviour, thermal properties, water sorption, porosity and stability to holistically assess the performance of Titanium dioxide nanoparticle reinforced 3D printed PMMA denture base materials. Thermocycling of the denture base samples could also help in evaluation of the long-term performance in oral environmental conditions.

Clinical implication

Incorporation of 3D printed PMMA denture base materials with TiO_2 nanoparticles greatly improved the flexural strength of the denture base materials. Uniform dispersion of the appropriate concentration of nano particles in the denture base materials can be achieved

by following appropriate protocols to optimize its physical properties. This facilitates the use of 3D printed denture bases reinforced with nanoparticles in different clinical situations due to its improved mechanical properties and other advantages offered by the nano particles.

Conclusion

Within the limitations of the study, it was concluded that the heat poly merized PMMA resin denture base materials have comparatively greater flexural strength when compared to titanium dioxide doped 3D printed PMMA resin denture base materials. This study concludes that the 3D printed PMMA resin denture bases have clinically acceptable levels of flexural strength as per the ISO requirements. This study reports that incorporation of Titanium dioxide nanoparticles significantly improves the flexural strength of 3D printed PMMA denture base resin materials widening its scope of use in different clinical situations.

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