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RESEARCH ARTICLE

The effect of ZrO_2 NPs addition on denture adaptation and diametral compressive strength of 3D printed denture base resin

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ABSTRACT

Objective(s): The world of dentistry is constantly evolving, and with the advent of 3D printing technology, the possibilities are endless. However, little is known about the effects of adding ZrO_2 NPs to the denture base resin of 3D additive manufacturing technique.

Aim of this study is to evaluate the behavior of resin which is used to 3D printing of denture base with the addition of ZrO_2 NPs on denture adaptation property and diametral compression strength.

Methods: 60 samples were printed, 30 disks for diametral compressive test and 30 denture base for denture adaptation test. Three groups per test (n=10). The control group for each test included unreinforced 3Dprinted denture base resin, and the other groups were reinforced with (2&3%) nanoZrO_2; diametral compressive strength was evaluated using universal compressive testing machine, while denture adaptation was evaluated by exocad software program.

Results: the study reveals significant difference in both diametral compressive strength and denture adaptation of the 3Dprinted denture base resin after adding nanoZrO_2, as denture adaptation increased; the mean of diametral compression was decreasing with 2%&3% percent of ZrO_2 NPs.

Conclusions: addition of Zro_2 NPs to 3D printed denture base resin may help in improving the material behavior as concerning mechanical and adaptation properties.

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INTRODUCTION

Additive manufacturing utilizing 3D printers has drawn interest from the manufacturing sector as well as from industries including health care, research, and education. The capacity of additive manufacturing to combine different materials based on CAD data has an impact on the whole quality of the prosthesis, the mechanical qualities of printed parts, the overall cost, and the time needed for production in the field of prosthetic dentistry (1) However, papers comparing the molding accuracy of the mucosal surface or denture retention that were created using conventional procedures to those created using additive manufacturing have been published, and there are still a number of issues that need to be clarified (2).

With the development of digital technologies, CAD/CAM has become a well-liked method for creating complete dentures. Computer-aided technologies act through 2 ways (additive or subtractive manufacturing) (3, 4). In vitro investigations showed that CAD/CAM milled dentures had better flexural strength and elastic modulus than conventionally made dentures. They also had better and precise fit, decrease in tooth movement, and more toughness. (5) Utilizing a number of methods, the position and level of deformation that occurred during the manufacture of complete dentures was discovered. One of these is the digital milling technique, which is thought to be a useful tool for assessing the dimensional

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change brought on by denture processing. The printed denture base which are printed with digital light processing technique (DLP); when CAD-CAM dentures were tested for fit on clinical bases, they showed to be more fitted within 100 µm than the subtracted denture base building approach(6, 7). Jin et al 2019 found no differences statistically on the surface adaptation after comparing different build angles on DLP- printed complete dentures among diffirent groups (8). However, there aren't many published studies examining the adaptation of complete dentures with conventional, milled CDBs, and 3-D printed CDBs techniques. But the present studies came to the conclusion that the latter technique appears to be optimistic one that needs to be focused on and improved upon (9).

Contrary to traditional and milled denture base materials, denture base manufactured with additive process seems to be decreased flexural strength and surface hardness despite being close to the accepted flexural strength limit of ISO (65 MPa). Hence, its clinical applications are limited (10, 11, 12, 13). Numerous studies investigated a variety of strategies to get around the limitations mentioned above, including adjusting the post-polymerization time, layer thickness, printing orientation, and adding nanoparticle fillers like TiO2, Al2O3, and SiO2 as metal oxide nanoparticles that seem to enhance some mechanical properties of the 3D-printed denture base resin (14,15,16). According to Gad et al. (2022) adding SiO2 NPs to 3D printed denture base resin enhances flexural strength and impact strength without significantly reducing surface roughness (17). Additionally, according to Alshaikh et al. (2022), the addition of ZrO2 NPs significantly increased the flexural strength, impact strength, and hardness of the 3D printed denture base resin with no discernible changes in surface roughness. This suggests that ZrO2 NPs could be an effective material reinforcement technique for 3D printing (18).

ZrO2 closely resembles the appearance of natural teeth and by decreasing the peri-implant inflammatory response, making it more biocompatible than other ceramic materials like alumina. It has great strength with good fracture toughness and better surface hardness and it is a biocompatible metal oxide. (18, 19, 20, 21). Moreover, it has antifungal and antibacterial action against Candida albicans and Asergillus niger as well as thermal stability, corrosion resistance (20, 22,23).

To the best of the authors' knowledge, no research has previously looked at how including ZrO2NPs into 3D-printed denture base resins affects denture adaption and diametral compressive strength.

Consequently, the purpose of this study was to determine how adding ZrO2NPs to 3D-printed denture-base resins affected denture adaption and diametral compressive strength.

The novelty of this study is that, despite the clinical importance of these features for denture users, no prior studies have been conducted to assess the impact of adding ZrO2 NPs to 3D-printed denture base resin on diametral compression strength and on denture adaption..

MATERIALS AND METHODS

60 samples were printed ; 30 discks for diametral compressive test with dimension of 8x16 mm as thickness and diameter(maximum curing depth was mentioned according to manufacturing instruction is 8 mm), and 30 denture base were printed after designing over maxillary edentulous definitive casts which been selected to have anatomical resembling for type A classification of American College of Prosthodontists (ACP) of residual ridge morphology **(24)**.For each test the samples were divided into 3 groups (n=10) according to ZrO2 NPs percentage (0%, 2%, 3%).

Nanoparticles of zirconium oxide with purity of 99.95%, monoclinic crystal phase with average size of 40-50 nm, USA 3320 Twig Leaf Ln, Houston, TX 7784 was used in specific concentrations with the 3D printed denture base resin by weight.

Optiprint laviva (dentona , Germany) 3D printed denture base resin of light pink color was used with DLP open system microlay versus 385 dental printer by exporting the STL file from microform computer software program .Pure resin was placed on mechanical mixer machine before adding the nanoparticles for 120 min; then addition of nanoparticles in mentioned concentrations and distributed into several bottles with stirring in magnetic stirrer for 30 minutes continuously at 60°C to decrease the viscosity of the resin, then at room temperature for 8h stirring to obtain homogenous mixture for printing procedure (25). Each layer was printed with a 50 µm layer thickness in (1.61) sec/slice in vertical Z axis following manufacturing instructions. Cleaning with isopropyl alcohol 99.9% before immersion in glycerol and placing in UV light polymerization unit for 20 minutes to complete the polymerization prior to finishing the samples by removing the supports and base with low speed rotary instrument and polishing with polishing machine and cloth in a wet condition (26,27). The whole procedure was done by one operator to insure applying same preparation conditions .The specimens immersed in distilled water 48hs at 37°C prior to testing (28).

Testing procedure

Diametral compressive test

each specimen was tested by universal compression testing machine, in this method a disk of the (3Dprinted acrylic denture base resin) is compressed diametrically in a testing machine until fracture (splinting). Because of the Poisson effect, the material is subjected to a tensile stress in the direction of the test instrument's force application due to the compressive stress applied to the test subject.

The indirect tensile stress (σx) is directly proportional to the load (P) applied in compression through the following formula:

$$\sigma x = 2P / \pi DB$$

At the disk specimen's center point is where there is the greatest vertical tensile tension, where P is the load, D is the diameter, and B is the thickness of the specimen. **(29,30)**

Denture adaptation test

For each stone cast, a three-dimensional image was captured using (vinyl 3D scanner; smart optics). Using a fully automated Z-axis scan, where the scanner guides the object into the measurement field on its own, the die was scanned with (6 µm) precision in accordance with ISO 12836. Designing a virtual denture base was accomplished by exocad program resembling complete denture and saved as STL file to be send to the 3D printer and printed. After complete printing of 30 denture base with the specific concentration of ZrO2 NPs; each denture base that was manufactured had its intaglio surface scanned using the same scanner (a vinyl 3D scanner from Smart Optics), which produced an STL file for each denture's intaglio surface. The STL file of each denture was superimposed on the STL file of the corresponding reprocessing cast using the same software (Exocad in-lab DentalCAD) with four layers (cast, denture base after processing, cast/denture base together and the overlay guide). The benefit from cast/denture base layer was to guide the denture to locate in its correct position. Using this software, measurements were made at 50 points for each of the 30 dentures in order to standardize the measurement. The overlay guide layer served as a representation of one of the best-fitting denture bases, and it had 50 points drawn on it in specific locations to occupy the entire anatomical landmark. The 50 point was distributed on the whole anatomical landmarks area on the cast. The cast was divided into five major areas (crest of the ridge 11 points, midline 10 points, posterior palatal seal 10 points, denture border 11 points and the palate 8 points).

For each denture base, the surface matching and measurements served as the foundation for evaluating fit discrepancies at 50 places, including the palate, posterior palatal seal area, median palatine suture, denture border, and crest of the ridge as shown in Figure 1.

RESULTS

For the diametral compressive test, descriptive statistics including the mean and standard deviation (Figure 2) were calculated. The results show that the mean values of the experimental groups decreased after adding nanoparticles compared to the control group. The highest mean was 17.775 for the control group, while the lowest mean was 13.556 for the group with 2% by wt. ZrO_2 NPs concentration. A comparison of means among all tested groups using the ANOVA table revealed significant results (Table 1).

The variances of the tested groups for diametral compression strength were analyzed by Levene's test of homogeneity, which showed non-significant differences. Bonferroni multiple comparisons test was selected, and the multiple comparison showed significant differences between the control and 2% by wt. ZrO_2 NPs, with no significant value for 3% by wt. ZrO_2 NPs as compared with control groups (see Figure 2).

For the denture adaptation test, descriptive statistics were used, including the mean and standard deviation. The results showed a decrease in mean values for all selected areas with 3% by wt. ZrO_2 NPs, which represented the lowest mean value, followed by 2% by wt. ZrO_2 NPs, while the highest mean was found in the control group. A test of variances was conducted using Levene's test, which revealed statistical significance. Comparison of means results for the experimental groups using

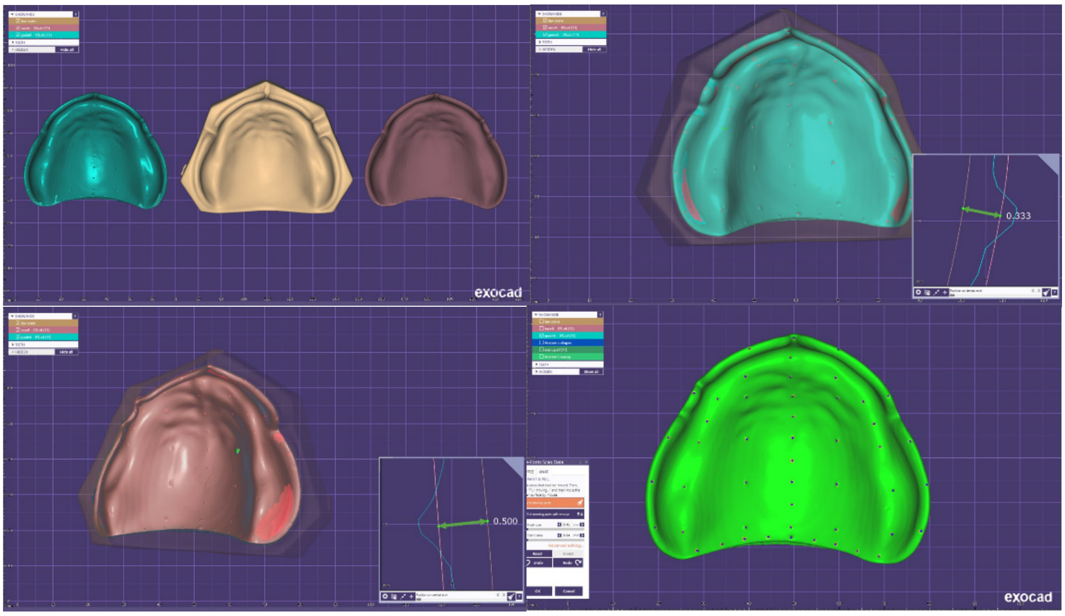


Fig. 1: represent virtual denture base design

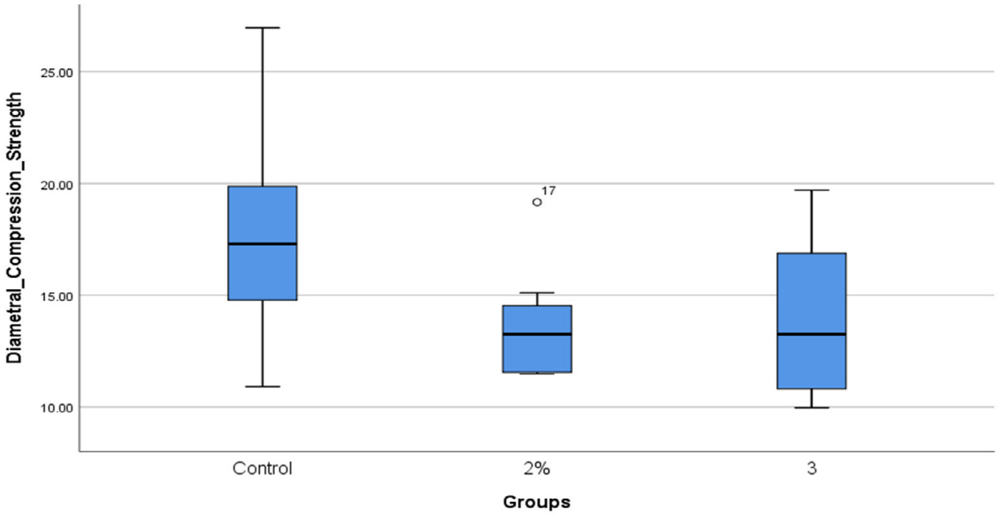


Fig. 2: Box-plot describing the median with high value and low value for diametral compression test. Different lower case letters represent significance at P value <0.05 .

Table 1. ANOVA Test for diametral compression test.

	Sum of Squares	Df	Mean Square	F	Sig.
Between Groups	109.080	2	54.540	4.258	0.025
Within Groups	345.809	27	12.808		
Total	454.888	29			

ANOVA table also revealed significant results (see Table 2).

Games-Howell multiple comparison reveals that 3% by wt. ZrO_2 NPs are the most significant and effective percentage for increasing denture adaptation in all areas of interest, as shown in Table 3 and Figure 3.

SEM images for ZrO2 nanoparticles as shown

Table 2. ANOVA test For denture adaptation test

ANOVA						
		Sum of Squares	df	Mean Square	F	Sig.
Border	Between Groups	0.186	2	0.093	7.786	0.002
	Within Groups	0.323	27	0.012		
	Total	0.509	29			
crest	Between Groups	0.205	2	0.103	9.202	0.001
	Within Groups	0.301	27	0.011		
	Total	0.506	29			
Midline	Between Groups	0.236	2	0.118	8.794	0.001
	Within Groups	0.363	27	0.013		
	Total	0.599	29			
Palate	Between Groups	0.298	2	0.149	9.703	0.001
	Within Groups	0.415	27	0.015		
	Total	0.713	29			
PPS	Between Groups	0.806	2	0.403	12.427	0.000
	Within Groups	0.875	27	0.032		
	Total	1.681	29			

Table 3. Games-Howell multiple comparisons test for denture adaptation.

Dependent Variable			Mean Difference (I-J)	Sig.
Border	Control	2%	0.05980	0.576
		3%	.18874*	0.001
	2%	1%	-0.05980	0.576
Crest	Control	2%	0.03806	0.777
		3%	.19135*	0.000
	2%	1%	-0.03806	0.777
Midline	Control	2%	0.06159	0.589
		3%	.21139*	0.002
	2%	1%	-0.06159	0.589
Palate	Control	2%	0.08681	0.412
		3%	.24113*	0.001
	2%	1%	-0.08681	0.412
PPS	Control	2%	0.02369	0.967
		3%	.35887*	0.000
	2%	1%	-0.02369	0.967

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

in figure (4) , the average particle size was estimated to be between ~35-~50 nm.

Figure 5 demonstrates a uniform and consistent distribution of nanoparticles inside the resin matrix, with the potential to see some clusters at 3% by wt. ZrO2 NPS.

Figure (4) also shows significant difference in the surface of the pure 3Dprinted denture base resin (A) and the 2% (B),3% (C) ZrO2 NPs at 4000 magnification force of SEM to prove the chemical reaction between the resin and the nanoparticles which was supported by the FTIR readings in Fig.(6) , both (B) & (C) showed homogenous and good distribution of nanoparticles within the resin matrix with some clusters may be shown at 3% nanoZrO2 .

The FTIR results showing significant difference between the pure 3D printed resin (0%), 2% nanoZrO2 3D resin and 3% nanoZrO2 3D resin especially between ~806- 636 cm^{-1} range of spectra

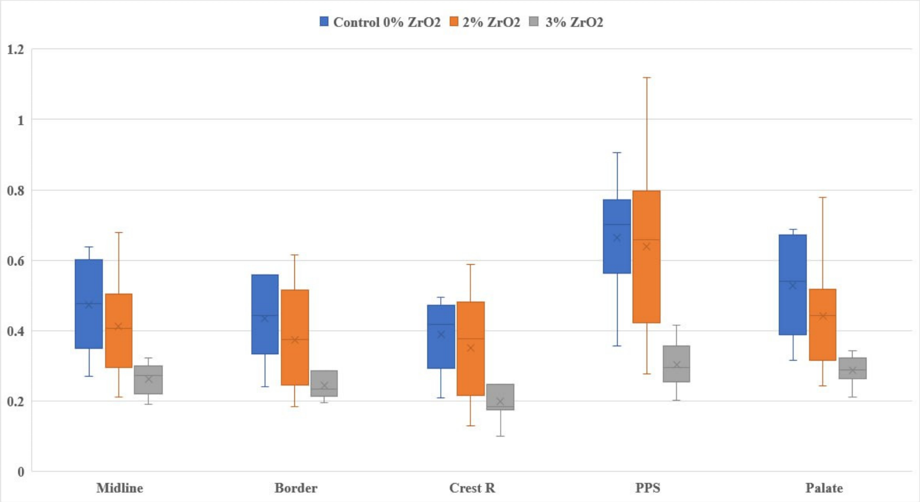
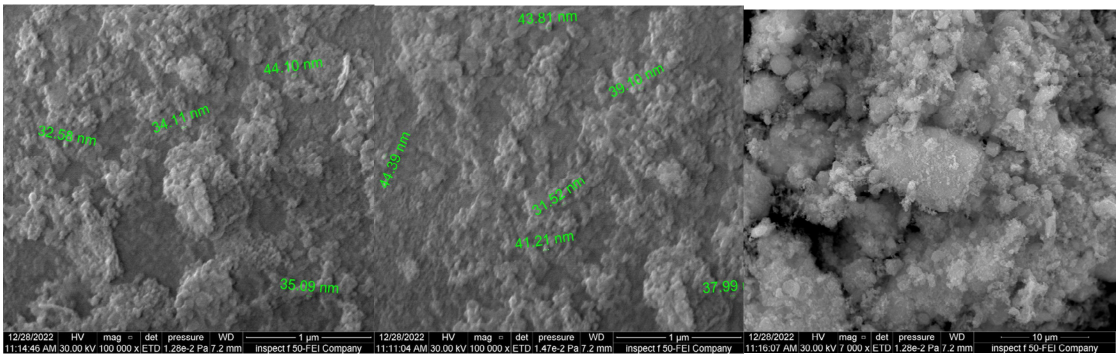
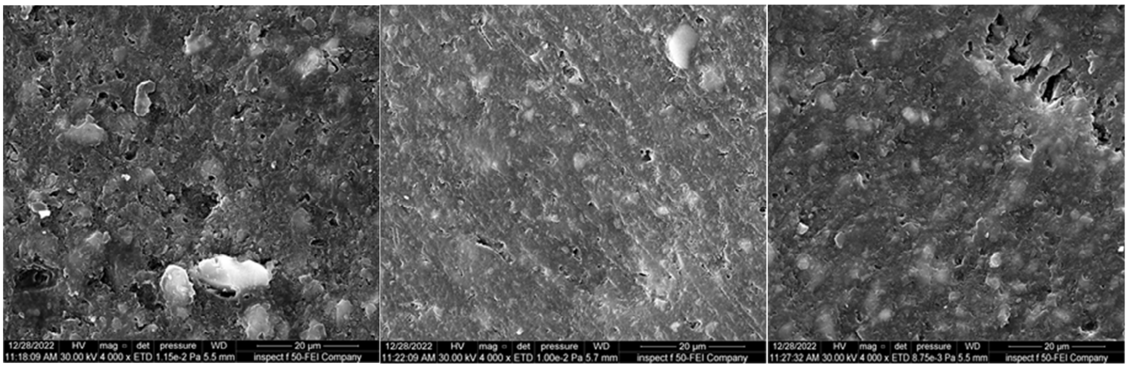


Fig. 3: Box plot of all areas comparing the control group with 2% and 3% by wt. ZrO2 NPs experimental group for denture adaptation test.



(A) **(B)** **(C)**

Fig. 4. SEM images for ZrO2 nanoparticles, (A)& (B) under 100000 KV magnification power shows the average size of the particles, while (C) under 7000 KV power.



(A) **(B)** **(C)**

Fig. 5. SEM images (4000X), (A) 3Dprinted resin with no addition, (B) 3D printed resin with 2% ZrO2 NPs, (C) 3D printed resin with 3% ZrO2 NPs.

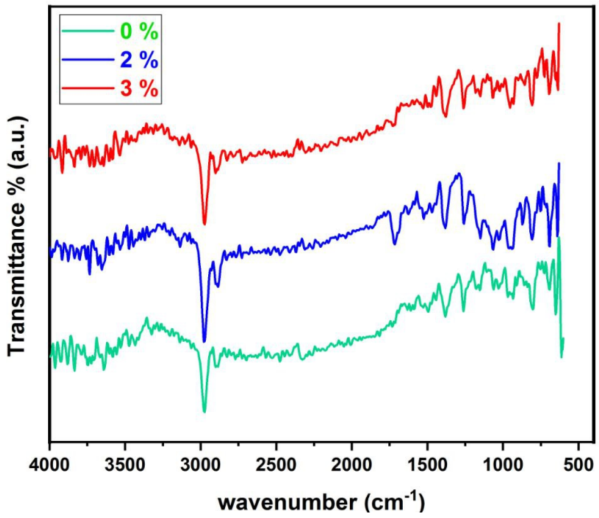


Fig. 6. FTIR spectra of 3D printed denture base resin with 0%,2%&3% ZrO2 NPs addition

which indicate the presence of ZrO2 within the polymer of the 3D printed denture base resin ,differences between peaks of 2% and 3% ZrO2 resin as appeared at ~ 752 cm^{-1} suggests that the polymer resin and nanoparticles' chemical interaction, as the most intense peak of band for 2% NPs at ~690 cm^{-1}, while for 3% NPs at ~694 cm^{-1}, with similarity to some extent between the spectra of the pure 3D resin and the reinforced resin attributed to the vibration and stretching of CH3 and CH2 groups at ~1716- 1381 cm^{-1} bands with vibration of ester group C=O at ~1180-1149 cm^{-1}, and this supports the nanoparticles' uniform dispersion within the resin substance used for 3D printing.

DISCUSSION

The results show that the addition of ZrO2 NPs considerably affect denture adaptation and indirect tensile strength. This study tested the effect of ZrO2 NPs addition on the properties of 30 denture base were printed digitally regarding diametral compression and denture adaptation. The current work shown at the addition of ZrO2 NPs positively affect the 3-Dprinted denture adaption while reducing the diametral compression (indirect tensile) strength.

A major and ongoing clinical issue in prosthodontics is the breakage of acrylic resin dentures. Due to a variety of factors, such as denture function, handling, and processing, denture failure may be difficult to identify. (32). Wear from repeated masticatory, flexural, and impact loads from dropping results in denture fractures. Dentures have been strengthened using rubbers, fillers, fibers, and other materials to improve their mechanical qualities. With the advancement of nano dentistry, many dental material reinforcing techniques have been created over time (33).

Nanostructure materials have received a lot of attention recently due to their high surface area to volume ratio, which promotes interaction and results in unique biological, physical, and chemical capabilities (34).

Strong relationships exist between a material's hardness, diametral tensile strength ,elastic modulus, and compressive strength. Because brittle materials are less able to withstand tension, the development of tensile stresses perpendicular to the specimens' long axes suggests that these stresses are probably to blame for the fracture, which consistently occurred along the vertical plane of load application and followed the long axis of the specimens. For materials that appear to be comparable, the diametral tensile strength test may yield varied results. However, the differences in the polymeric matrix, filler sizes, and the binding between the matrix and fillers have been used to explain this discrepancy (35).

Zirconium oxide nanoparticles (nano-ZrO2), which affect aesthetics minimally compared to other nanoparticles due to their superior biocompatibility and white appearance, have recently attracted interest (17). choosing nano-ZrO2 as a filler in this investigation due to their potential to enhance acrylic resins' mechanical qualities. As the toughest of all the oxides, ZrO2 particles have been shown to possess outstanding mechanical properties and can endure fracture promotion. They have a crystalline structure. They also have outstanding toughness and strength, abrasion and corrosion resistance, and biocompatibility (17,18,19,20). The PMMA/nano-ZrO2 composite's mechanical and physical characteristics are influenced by a variety of variables, including the matrix's size, shape, proportion, distribution, and composition (36). According to past research that looked at the impacts of ZrO2 fillers on the material's capabilities in PMMA denture bases, the acrylic denture base's flexural and impact capabilities can be greatly enhanced by nano-ZrO2 (37). The greatest increase was seen in the denture base with 5 wt% nano-ZrO2. Beyond 5% weight, the nanofiller concentration resulted in the particles gathering and aggregating, weakening the material rather than strengthening it (38).

In this study, significant decrease of diametral compressive strength with the addition of 2% ZrO2 NPs compared to control group and yet slight increase in the strength with 3% NPs than 2%, the decreasing with low concentration of nanoparticles may be explained by being not enough concentration of nanoparticles to interact with each other or with resin matrix properly in sufficient interactions and the nanoparticles only lead to increasing the distance between resin matrix (39). This result agrees with Chladek et al. (2013) who saw that as NPs concentration rose, the mechanical characteristics of nanocomposites enhanced by silver NPs declined, the decrease can also be explained by the agglomeration of the nanoparticles incorporated within the 3D resin which act as an areas of stress concentration in the matrix and this leads to declining the mechanical properties (40, 41, 42). Similarly, in 2010 Chatter jee demonstrated that the tensile strength dropped as the amount of titanium oxide nanoparticles increased (41). Moreover, the inclusion of agglomerated fillers that form loosely bound clusters and change the fracture propagation process reduces the indirect tensile strength (when subjected to compressive stresses) (19,25,36).

Precision is the most crucial factor in denture adaptation and in the production of all prostheses. Shrinkage of polymerization has been an unavoidable result of the use of polymer in the manufacture of denture construction. For accurate prosthetics fabrication, the intaglio surface's overall size and fit must be examined. Studies on the fabrication processes for maxillary complete dentures show that the misfit of the base varies by area. The gap between the denture base and the cast is typically impacted by the shrinking of the resin material during polymerization, with a propensity of the internal tension toward the center region of the denture base. This distortion is then brought on by a restriction brought on by the surface shape of the alveolar ridge. The benefit of digital technology is versatility because of the variety of machines that are available, the low amount of raw material waste, and the capacity to print complex geometries. The staircase effect, poor repeatability, and demand for supporting structures are the main drawbacks of the printing method. These supporting structures require more resources and time (42, 43).

each phase, such as designing in CAD software, printing, and cutting in printing software, degree of light, direction and printing angle, layers number, software, shrinkage between layers, amount of supporting structure, and the post-processing process; all affect the accuracy of printed objects (44).

Regarding this study, ZrO2 NPs improved the adaptation of the denture base significantly and this increase in fitting of the denture to the upper mold was proportional directly to the NPs. concentration.

The improvement in the denture base's overall adaptability could be credited to the effective dispersion of nano-ZrO2 fillers, as their nano size helps to internally fill the matrix and replace the resin, causing less shrinkage (39). This in agreement with a study that examined the accuracy of a denture base polymer improved by incorporation of glass fibers and found that the dimensional changes decrease as the fiber content increases (45), another in consent with the findings for testing the impact of various ratios of zirconium oxide nanofillers on processed denture samples was also found (46). These findings also explain why the vertical dimension of occlusion changed less in the zirconium reinforced group than in the control group. Since there have been less dimensional changes, more dentures have been adapted after processing, and linear tooth movement values have increased inward (47).

Using ZrO2 nanoparticle reinforcement, a sharp reduction in the space between the denture base and the cast, at the mid-palatine region (0.057± 0.006) and at the posterior palatal seal region (0.060 ± 0.007 cm) with the lowest value at 7 wt.% concentration (46).

Based on these results, the outstanding act of ZrO2 NPs as enhancement filler cannot be ignored, with many other properties due to their specific characteristics making them suitable for denture base reinforcement material. Still further investigations are recommended with more concentrations of ZrO2 NPs on other physical and mechanical properties of 3D printed denture base resin.

The limitations of this study were using one type of 3Dprinted denture base resin, with only 2 concentrations of ZrO2 NPs. More concentration will give better idea about the behavior of ZrO2 NPs within the 3D printed resin for denture base, moreover the conditions of testing did not simulate oral environment. Therefore, in vivo and clinical investigations are required.

CONCLUSION

It was determined, within the confines of this study, that the addition of ZrO2 NPs to 3D-printed denture base resin considerably improves its behavior as denture adaption, and that this improvement is directly correlated with the ZrO2 NPs concentration. while for indirect tensile strength the behavior need more investigation to get proper understanding for the effect of the ZrO2 NPs on the mechanical properties of the 3D printed denture base material. Caution must be taken to properly select the appropriate concentration of ZrO2 NPs in order not to affect other properties adversely.

CONFLICT OF INTEREST

The authors declare no conflicts of interest.

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