



Effect of Various Nanoparticles Incorporated to 3D-Printed Denture Base Resins on Certain Physicomechanical Parameters

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Abstract

Purpose: To evaluate the effects of nanoparticles (zirconium dioxide (ZrO_2), titanium dioxide (TiO_2), and aluminum oxide (Al_2O_3)) on the flexural strength, colour stability, and surface roughness of 3D printed resin.

Materials and Methods: A total, 210 specimens of 3D printed resin were fabricated with different dimensions according to test specifications, and specimens were divided according to nanoparticles into four groups, unmodified as control (A), ZrO_2 (B), Al_2O_3 (C), and TiO_2 (D) groups (70/test ($n=7$)). Each of modified groups was subdivided into three subgroups according to nanoparticle concentrations (0.5%, 1%, and 1.5%). The flexural strength, colour stability, and surface roughness were evaluated, and the collected data was analyzed with ANOVA followed by Tukey's post hoc test.

Results: Regarding the flexural strength, there was a statistically decrease for all modified groups compared with the control group. The difference between groups was statistically significant as indicated by ANOVA test ($p<0.0001<0.05$). Significant colour differences were detected between the control group and all modified groups ($p<0.0001<0.05$). All modified groups had lower surface roughness than the control group. The difference between groups was statistically significant as indicated by ANOVA test ($p<0.0001<0.05$). The difference between concentrations of metal oxide groups was statistically non-significant as indicated by ANOVA test ($p=0.6296>0.05$).

Conclusions: Flexural strength decreased in all groups and concentrations compared to the control group. Significant colour changes were detected between the control group and all modified groups. Surface roughness decreased in all groups and concentrations compared to the control group.

Keywords

Nanoparticles, Flexural strength, Color changes, Surface roughness

INTRODUCTION

Traditionally, polymethylmethacrylate (PMMA) has been used to create removable dental prosthetics due to its affordability, acceptable strength, ease of manufacture and reparability, aesthetics, and intraoral stability. Low mechanical and physical qualities as well as the length of time required for manufacture place a constraint on the material^(1, 2). CAD-CAM technology produces digitally fabricated dentures in less time than traditional dentures, using milled or 3D-printable denture base materials⁽³⁾. In the subtractive method (milled), the dentures are milled from prefabricated resin blocks. In the additive method, dentures are digitally fabricated layer by layer using three-dimensional printing technology (3D-printing) and photopolymerized fluid resins⁽⁴⁾. Compared to traditional and milled denture base materials, 3D-printed denture base resins have the lowest flexural strength and surface hardness⁽⁵⁾. The flexural strength, however, is quite near to the ISO approved limit of 65 MPa. These drawbacks restrict the clinical use of 3D-printed technology and make it difficult to apply it to the manufacturing of denture bases. Accordingly, earlier research looked at many parameters impacting the characteristics of 3D-printed resin, such as printing orientation, post polymerization time, and printing layers thickness, to get over the aforementioned constraints and reap the benefits of 3D printing technology⁽⁶⁻⁸⁾. Other investigations looked into the addition of various compounds to 3D-printed materials^(9, 10). According to a study, 3D-printed resin's antibacterial function was improved by TiO₂ nanoparticles, although the mechanical qualities were not examined⁽¹¹⁾. Mangal et al. found that adding 0.1 weight percent of animated nanodiamonds improved the mechanical characteristics of 3D-printed resin used to make orthodontic equipment⁽⁹⁾. Similar to this, Aati et al. demonstrated long-term improvement of ZrO₂ nanoparticle-modified 3D-printed resin used for temporary restorations⁽¹²⁾.

Nanotechnology has lately been applied in the prosthodontic field for material enhancement objectives, as evidenced by the investigation of nanoparticles for increasing the mechanical characteristics of PMMA. The current choices for increasing the mechanical characteristics of PMMA are nanofillers such as nanoclays, nanotubes, nanofibers, and nanoparticles. The impact of nanoparticles on the mechanical characteristics of PMMA is determined by a variety of parameters, including the polymer particle interface, particle size, production technique, and particle dispersion in the PMMA matrix⁽¹³⁾.

Zirconia nanoparticles (ZrO₂) is one of the biocompatible dental ceramic materials that improved mechanical properties like flexural strength and impact strength. It has been widely used because it has high mechanical strength, good surface properties, and good biocompatibility and biological properties, making it an advantageous material for use in dental materials, such as reinforcement of denture bases and repair⁽¹⁴⁾.

Also, alumina nanoparticles (Al₂O₃) is a material with strong interatomic bonding, which gives rise to its desirable material properties. The effect of alumina addition has been reviewed and reported to have a positive impact on the properties of acrylic resin. This is due to alumina's unique properties, which include high hardness and good thermal properties, making it the material of choice for a wide range of applications⁽¹⁵⁾.

Finally, titania nanoparticles (TiO₂) has demonstrated antibacterial properties. It is also a low-cost biocompatible substance that is chemically stable, toxic-free, corrosion-resistant, strong, and has a high refractive index. In addition, research has shown that even a little addition of TiO₂NP reinforcer to a polymeric material can change the hybrid material's electrical, optical, chemical, and physical characteristics⁽¹⁶⁾.

This study examined the effects of reinforcement with various concentrations of ZrO₂, Al₂O₃, and TiO₂Np on the flexural strength, colour stability, and surface roughness of 3D printed denture base resin. The null hypothesis states that the flexural strength, elastic modulus, colour stability, and surface roughness of 3D printed denture base resin will all be enhanced by the addition of various quantities of ZrO₂ Np, Al₂O₃ Np, and TiO₂ Np.

MATERIALS AND METHODS

A total of 210 specimens were prepared from 3D printed resin (HARZ Labs Dental Pink), and then they were divided into 70 specimens for each testing group. For flexural strength test, rectangular specimens were designed according to ISO 20795-1: 2013 standards, with dimensions 64 × 10 × 3.3 ± 0.2 mm. Color stability was tested using disc shaped specimens of dimensions 10 × 2 mm according to ISO 20795-1: 2013. Surface roughness was tested using disc shaped specimens of dimensions 10 × 1.5 mm according to ISO 20795-1 : 2013⁽¹⁷⁾.

Nanocomposites Mixture Preparation

The composite mixture has been obtained through the gradual addition of the appropriate amount of nanoparticles into the 3D printed resin solution under continuous stirring, followed by ultrasound direct mixing for 1 h in a sealed vial using an ultrasound unit. The new nanocomposite material contains ZrO₂ Nps, Al₂O₃ Nps, and TiO₂ Nps nanoparticles by weight: 0.5%, 1%, and 1.5%⁽¹¹⁾.

Preparation of the Samples

3D models of specimens with dimensions according to test specifications mentioned previously were created using CAD software (SolidWorks, Dassault Systems SolidWorks Corp, France). The resulting CAD files are digital representations of the desired object. The CAD files must be converted into a 3D printable file format, also known as standard triangle language (STL). Import an STL file into 3D slicer software (Formware 3D software) to slice the digital model into layers for printing. The print preparation software sends the instructions to the 3D printer (RASDENT, Bulgaria) via wireless or cable connection once the setup is complete to start the printing process. The printing orientation of the specimens was

done at 90° and 100-microns/layer thickness following manufacturer instructions. After finishing the material polymerization, the specimens are removed from the platform, washed of excess resin, cleaned with isopropyl or ethyl alcohol 99.9% and post cure in a UV chamber (DENSTAR Co., Ltd, KOREA) for fully polymerization according to the manufacturer's safety data sheet. Post-print curing enables objects to reach the highest possible strength and become more stable. Low-speed rotary instruments were used to remove the support structures from the specimens.

TEST PROCEDURE

Flexural Strength

Flexural strength of the specimens was evaluated by three-point bending test. All samples were individually and horizontally mounted in a custom-made loading fixture [three point bend test assembly; two parallel stainless steel rods with a span length of 50 mm supporting the specimen, with the damage site centrally located on the tensile side] on a computer controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) with a load cell of 5 kN and data were recorded using computer software (Instron® Bluehill Lite Software). Then the samples were statically compression loaded until fracture at a crosshead speed of 1 mm/min. The stress-strain curves were recorded with computer software (Instron® Bluehill Lite Software). FS represents the limiting stress at which failure or instability is imminent. The value of the calculation of FS was guided by the formula:

$$FS (\sigma) = 3F (L) / 2wh^2$$

where F is the maximum load at the point of fracture, L is the span, w is the width of the sample, and h is its height.

Color Stability

The specimens' colors were measured using a Reflective spectrophotometer (X-Rite, model RM200QC, Neu-Isenburg, Germany). The aperture size was set to 4 mm and the specimens were exactly aligned with the device. A white background was selected, and measurements were made according to the CIE L*a*b* color space relative to the CIE standard illuminant D65. The color changes (ΔE) of the specimens were evaluated using the following formula:

$$\Delta E_{CIELAB} = (\Delta L^*2 + \Delta a^*2 + \Delta b^*2)^{1/2}$$

where: L* = lightness (0-100), a* = (change the color of the axis red/green) and b* = (color variation axis yellow/blue).

Surface Roughness (Ra)

Surface roughness was measured with a USB digital surface profile gauge (Elcometer 224/2, Elcometer Instruments, Great Britain) and data was recorded using the computer software of the roughness tester supplier (Elcomaster 2, Elcometer Instruments). For every reading made, the mean roughness value (Ra, μm) was represented by the arithmetic mean between the peaks and valleys registered after the needle of the profilometer had scanned a stretch of 2 mm in length, with a cut-off of 0.25mm to maximize the filtering and the undulation on the surface. Each surface was read three times, always with the needle scanning the geometric center of the specimen, starting from three different points. The mean value of the five readings yielded the mean value of the roughness of each specimen.

Statistical Analysis

Data were presented as mean, standard deviation (SD), for values. The results were analyzed using Graph Pad InStat (Graph Pad, Inc.) software for Windows. A value of $P < 0.05$ was considered statistically significant. After homogeneity of variance and normal distribution of errors had been confirmed, one-way ANOVA followed by Tukey post-hoc tests were used to detect significance between surface treatment within each main sealer group. A two-way analysis of variance was performed to detect the effect of each variable (metal oxide group and concentration). The sample size ($n = 7$) was large enough to detect large effect sizes for main effects and pair-wise comparisons with a satisfactory level of power set at 80% and 95% confidence.

RESULT

Flexural Strength

Regardless of concentrations, the highest mean value was recorded for the control group, followed by the Al modified subgroup then the Zr modified subgroup mean value, while the lowest mean value was recorded for the Ti modified subgroup. The difference between groups was statistically significant, as indicated by the ANOVA test ($p < 0.0001 < 0.05$).

Irrespective of metal oxide type, the highest mean value was recorded for the control group, followed by the 1.5% modified subgroup then the 0.5% modified subgroup mean value, while the lowest mean value was recorded for the 1% modified subgroup. The difference between the concentrations of metal oxide groups was statistically significant, as indicated by the ANOVA test ($p < 0.0001 < 0.05$). A pair-wise Tukey's post-hoc test showed a non-significant ($p > 0.05$) difference between the (0.5% and 1%) modified groups.

Table 1 includes the mean values and SD of flexural strength, colour stability, and surface roughness

ZrO ₂ Nanoparticles	Flexural strength	Modulus of elasticity	Color stability	Surface roughness
0.5%	45.01 ^c (5.46)	1.475 ^b (0.179)	5.058 (1.22)	0.2882 ^a (0.0027)
1%	46.52 ^c (4.93)	1.525 ^b (0.162)	5.902 (1.92)	0.2893 ^a (0.0022)
1.5%	70.05 ^a (6.59)	2.296 ^a (0.216)	7.049 (1.32)	0.2871 ^a (0.0041)
p-value	<0.0001*	<0.0001*	0.0739 Ns	0.1234 Ns
Al ₂ O ₃ Nanoparticles				
0.5%	67.31 ^{ab} (0.92)	2.206 ^{ab} (0.03)	5.551 (0.87)	0.2826 ^c (0.002)
1%	61.02 ^b (6.2)	2 ^b (0.203)	5.742 (0.53)	0.2846 ^{bc} (0.0019)
1.5%	60.95 ^b (3.69)	1.997 ^b (0.121)	4.079 (1.95)	0.2837 ^c (0.0022)
p-value	0.0156*	0.0158*	0.05 Ns	0.2351 Ns
TiO ₂ nanoparticles				
0.5%	47.32 ^c (7.96)	1.551 ^b (0.261)	6.799 ^b (1.39)	0.2887 ^a (0.002)
1%	50.28 ^c (4.7)	1.648 ^b (0.154)	10.5 ^a (1.9)	0.2855 ^a (0.0086)
1.5%	49.54 ^c (3.64)	1.624 ^b (0.119)	12.6 ^a (2.11)	0.2904 ^a (0.002)
p-value	0.6122ns	0.6122ns	<0.0001*	0.6122ns
Control	72.36 ^a (10.1)	2.372 ^a (0.332)		0.2908 ^a (0.0022)
p-value	<0.0001*	<0.0001*	<0.0001*	0.0002*

Colour Stability

Regardless of concentrations, the highest mean value was recorded for the Ti modified group, followed by the Zr modified group mean value, while the lowest mean value was recorded for the Al modified group. The difference between groups was statistically significant, as indicated by the ANOVA test ($p < 0.0001 < 0.05$). A pair-wise Tukey's post-hoc test showed a non-significant ($p > 0.05$) difference between the (Zr and Al modified) groups.

Irrespective of metal oxide type, the highest mean value was recorded for the 1.5% modified group, followed by the 1% modified group mean value, while the lowest mean value was recorded for the 0.5% modified group. The difference between groups was statistically significant, as indicated by the ANOVA test ($p = 0.008 < 0.05$). A pair-wise Tukey's post-hoc test showed a non-significant ($p > 0.05$) difference between the (1% and 1.5% modified) groups.

Surface Roughness

Regardless of concentrations, the highest mean value was recorded for the control group, followed by the Ti modified subgroup then the Zr modified subgroup mean value, while the lowest mean value was recorded for the Al modified subgroup. The difference between groups was statistically significant, as indicated by the ANOVA test ($p < 0.0001 < 0.05$).

Irrespective of metal oxide type, the highest mean value was recorded for the control group, followed by the 1.5% modified subgroup then the 0.5% modified subgroup mean value, while the lowest mean value was recorded for the 1% modified subgroup. The difference between the concentrations of metal oxide groups was statistically non-significant, as indicated by the ANOVA test ($p = 0.6296 > 0.05$).

DISCUSSION

In this in vitro study, the effects of the addition of ZrO₂NPs, Al₂O₃NPs, and TiO₂NPs on the properties of 3D printed denture-base resins were tested. The flexural strength of 3D-printed resin was investigated because it is believed to be the main clinical failure mode⁽¹⁸⁾.

According to the results of the present study, the flexural strength of 3D-printed resins was decreased with the addition of ZrO₂NPs, Al₂O₃NPs, and TiO₂NPs compared to the control group.

Regarding ZrO₂ Nps, the flexural strength of all ZrO₂ Nps groups was lower than the control group, which is consistent with the findings of Gad et al.⁽¹⁹⁾, Ergun et al.⁽¹³⁾, and Noha et al.⁽²⁰⁾. This decrease may be attributed to the poor distribution of ZrO₂NPs within the resin matrix. They will not fill the space between polymer chains homogeneously, which will result in interrupting resin matrix continuity and creating defects in the material, weakening it in the end result⁽²¹⁾. This result in the present study disagrees with a previous study that investigated the effect of ZrO₂NPs on 3D-printed resins, which investigated an increase in the flexural strength after adding ZrO₂NPs⁽¹⁸⁾. This may be due to many factors that could affect the flexural strength, such as size, shape, concentration of filler, homogenous distribution within the resin matrix, and the silanization process.

Regarding Al₂O₃ Nps and TiO₂ Nps, it was discovered that as the Al₂O₃ and TiO₂ filler loading increased, the mean flexure strength decreased. This phenomenon can be explained by the fact that Al₂O₃ Nps and TiO₂ Nps agglomerate and form clusters at high concentrations. These clusters may act as stress concentration areas, and at these areas, cracks are initiated and propagated, which negatively affect the mechanical properties of 3D printed resin^(12, 22, 23). Also, according to reports, light penetration depth, printed resin viscosity, and building orientation are all factors that could influence the properties of printed resin⁽²⁴⁾. Because of the presence of agglomeration and clusters of nanoparticles in 3D printed resin, light scattering may occur, affecting the polymerization method. Furthermore, high concentrations of nanoparticles may increase the viscosity of printed resin, affecting printing procedures^(9, 12).

These findings are consistent with the findings of Fathie et al., Chehusna et al., and Zinal et al., who discovered that increasing the amount of nano-Al₂O₃ fillers decreased flexural strength⁽²⁵⁾.

This result also agrees with Hamouda et al.⁽²⁶⁾, Nazirkar et al.⁽²⁷⁾, Sodgar et al.⁽²⁸⁾, and Edwin Tandra et al.⁽²⁹⁾, who discovered that the flexural strength decreased with the increase of TiO₂ Nps.

The change in the appearance and staining indicates reduction of the long term quality of the prosthesis⁽³⁰⁾. According to the National Bureau of Standards, a color change is very low when ΔE is less than 1, clinically accepted when ΔE is between 1 and 3, and clinically observable when ΔE exceeds 3⁽³¹⁾. In the current study, the lowest mean color change value was 4.079, indicating that all the tested groups had inappropriate degrees of color change.

The increase in light absorption is statistically significant, and in the current study there was an increase in the relative amount of light absorption with the increase of ZrO₂ NPS and TiO₂ NPS concentration, which led to an increase in colour change. This is clearly due to the presence of opaque nano-ZrO₂ and nano-TiO₂ powder in the polymer matrix, which absorb more light energy than the polymer matrix and appear to be more opaque. These findings were due to the high atomic numbers of Zr and Ti in comparison to the chemical constituent of acrylic, which has a low atomic number. The absorption of light energy by an element is dependent primarily on the cube of its atomic number^(32, 33).

The results of the current study are consistent with the findings of Ihab et al., who discovered that significant colour differences were detected between the control group and specimens incorporated with zirconium oxide nano-fillers at different immersion solutions⁽³²⁾.

Also, this result is consistent with that of Aziz et al., who discovered that colour change increased with the increase of TiO₂ NPS concentration⁽³³⁾.

According to the results of the current study, regarding Al₂O₃ NPs, we found that as the Al₂O₃ NPs concentration increased, the colour change decreased. This may be due to the presence of Al₂O₃ Nps in the polymer matrix, which reflects more light than it absorbs, and this is because the Al₂O₃ has a low atomic number in comparison to the chemical constituents of acrylic resin. The absorption of light energy by an element is dependent primarily on the cube of its atomic number.

This result is consistent with that of Andreotti et al., who discovered that colour change decreased with the increase of TiO₂ NPS and ZnO NP concentrations⁽³¹⁾.

Surface roughness is an important feature of denture surfaces, and studies show that rougher denture surfaces increase microbial adhesion and bacterial colonization, as well as food debris retention areas that are difficult to remove, resulting in infections of the underlying tissues. Furthermore, a rough surface attracts more stains, which have the potential to alter the material matrix, resulting in an external colorant staining effect⁽³⁴⁾. The surface roughness of the denture base should be below or close to the minimum clinically acceptable value (0.2 μm)⁽³⁾. In the present study, the highest recorded surface roughness value (0.2908 μm) in the control group was slightly above the maximum clinically acceptable surface roughness value (0.2 μm). This increase in surface roughness value may be attributed to the printing technology (layer by layer and printing orientation). Furthermore, the printing orientation (90°) resulted in more compact step wise edges on the specimen surface, which made the surface rougher between layers⁽³⁵⁾.

In the present study, additions of ZrO₂Nps and TiO₂Nps to 3D-printed resin didn't significantly change the surface roughness, which is consistent with Gad MM et al., who discovered that there is no change in surface roughness with the addition of SiO₂NPs to 3D-printed resin in different concentrations before and after thermal cycling, and this confirmed that the main change in surface roughness is related to layering technique regardless of NP concentration⁽³⁾.

Also, this result is consistent with the study that discovered that the addition of nano-ZrO₂ fillers into the 3D printed denture base resin did not significantly change the surface roughness⁽¹⁸⁾. Hence, printing technology and printing parameters have a greater effect on Ra, and further investigations on different printing parameters' effects on surface roughness are required

Regarding Al₂O₃, there is a significant change (decrease) in the surface roughness with the addition of Al₂O₃Nps in comparison to the control group. This result disagrees with Vojdani et al. and Jasim et al., which reported that the surface roughness of the acrylic denture base was not significantly changed when different percentages of silanized Al₂O₃ nanoparticles were added. This result may be due to the fact that the alumina nanoparticles have very small sizes and good dispersion. Also, the surface roughness test concerns only the outer surface and not the inner surface of the nanocomposite, so when a small percentage of nanoparticles are added to the acrylic resin, only a few particles are involved on the surface of the specimens^(36, 37).

CONCLUSION

From the results of this study, it could be concluded that:

- 1- All concentrations of ZrO₂ Nps, Al₂O₃ Nps, and TiO₂ Nps added to 3D printed resin reduce the flexural strength of the 3D printed resin when compared to the control group.
- 2- The flexural strength of 3D printed resin improve with increasing ZrO₂ Nps concentrations but remain lower than the control group.
- 3- The flexural strength of 3D printed resin decrease as the concentrations of Al₂O₃ Nps and TiO₂ Nps increase.
- 4- Significant colour changes were found between the control group and specimens containing ZrO₂ Nps, Al₂O₃ Nps, and TiO₂ Nps at various concentrations, which were clinically unsatisfactory.

- 5- Surface roughness decreased in all groups and concentrations when compared to the control group. However, the difference in surface roughness between the ZrO₂ and TiO₂ groups and the control group was not statistically significant. While this decrease is statistically significant in the Al₂O₃ group.

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