Research Article

Yttria stabilized zirconia: innovative approach for improving the performance of additively manufactured denture resins

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Abstract: Background: The development of additive manufacturing technology facilitated the advancement of 3D-printed denture resins, though these materials show limitations in their physical and mechanical properties. Aims: This study investigated the effects of adding yttria-stabilized zirconia nanoparticles to 3D-printed denture resin to improve its performance at weights of 1% and 3%. The focus was on impact strength, surface hardness, and roughness. Materials and methods: 90 specimens of 3D-printed acrylic were divided into three groups, then further divided each group into three subgroups based on the results of each test. Charpy's Impact Strength Tester was used to evaluate impact strength. Surface characteristics were assessed by Vickers microhardness (HV) tester (microhardness) and non-contact profilometer (roughness). Fourier transform infrared spectroscopy (FTIR) was utilized for the chemical analysis. The experimentation's data guided the statistical analysis, which included descriptive statistics, analysis of variance (ANOVA), and multiple comparison tests based on the significance of each test's outcomes. Results: The results verified that adding 1 wt.% and 3 wt.% YSZ NPs to the 3D-printed denture resin significantly enhanced its mechanical and surface characteristics ($p < 0.05$) compared to unmodified resin. Conclusion: The addition of YSZ NPs enhanced the impact strength, surface hardness, and roughness. FTIR analysis showed that the polymer and YSZ nanoparticles did not interact chemically. This suggests that a new 3D-printed nanocomposite denture base with better performance is possible.

Keywords: Additive Manufacturing, 3D printed denture base resin, Digital dentures, Impact strength, YSZ nanoparticles

Introduction

Advancements in dental materials and processing technology led to a transformative revolution in prosthodontics [1]. Though polymethyl methacrylate (PMMA) remains dominant in denture base fabrication [2] [3], its inherent drawbacks necessitate further optimization [4] [5]. With the rise of computer-aided design and computer-aided manufacturing (CAD-CAM), digital manufacturing technologies (mainly subtractive and additive methods) have made considerable inroads into dentistry [6].

The three-dimensional printing technology has emerged as a rapidly evolving technology with enormous potential [7] [8]. One of its key advantages lies in its ability to process CAD data directly into physical matters, eliminating the need for traditional molds and reducing material waste. Moreover, it improves accuracy by eliminating technician-driven errors and potentially increasing tissue adaptation [6]. Despite the advantages of 3D printing acrylic resins, they fall short of PMMA regarding their mechanical and physical properties [9].

Evolving nanotechnology provides a fascinating approach to addressing these limitations. Nanoparticles have noteworthy properties that can significantly develop the performance of composite materials [10] [11]. Amongst zirconia nanoparticles (ZrO_2 NPs) that show exceptional strength and chemical and thermal stability, making them prime applicants for reinforcement [12] [13]. In particular, yttria-stabilized zirconia (YSZ) is a highly promising material for dental applications [14] [15].

A talented contemporary strategy to enhance the characteristics of acrylic resins would entail the incorporation of zirconium oxide as a filler material. Wide-ranging investigations confirm its biocompatibility, presenting it as an ideal candidate. Furthermore, zirconia outperforms metal fillers in terms of aesthetic appeal, resulting in a more natural appearance and providing an exclusive advantage. Studies have established that the incorporation of fillers into the resin matrix can substantially raise flexural strength, impact resistance, and hardness (16).

Zidan et al. found that adding ZrO_2 nanoparticles at a weight percentage of 3 to 5 wt.% to high-impact PMMA resin made it much stronger, stiffer, tougher, and harder (17). Andreea et al.'s recent research demonstrated that YSZ nanoparticle-modified 3D printing resins offer a promising alternative for constructing components for fixed dental prostheses. They found that 1 wt.% and 3 wt.% concentrations of YSZ significantly changed the three-dimensional printable material's microstructure and its mechanical characteristics (18). Indeed, Roitero et al. clarified that nanocrystalline zirconia stabilized with 3 mol% yttria exhibits excellent strength and toughness, in addition to resistance to aging and translucency, due to its fully stabilized tetragonal nanostructure. In contrast, zirconia stabilized with 1.5 mol% yttria reveals even better mechanical implementation while preserving aging resistance and a moderate level of opacity (15). These outcomes underline the potential advantages of using yttria-containing transformable tetragonal nanoceramics for dental applications.

The effect of YSZ nanofiller incorporation into 3D-printed acrylic denture bases on impact strength and surface properties remains unknown. Filling this knowledge gap represents a significant opportunity to develop next-generation nanocomposite denture base materials with both long-term performance and enhanced functionality.

Materials and methods

The World Health Organization calculations and power analysis to determine the research sample size were used. This yielded an 80% study power, a 5% significance level, and a 5% marginal error.

Preparation of Nanocomposites Mixture

This study utilized a 3D-printed denture acrylic (FREEPRINT® denture) modified by YSZ NPs (99.95% purity, US Research Nanomaterials, Inc., USA), with an average size of 19.53 nanometers (nm) as revealed by X-ray diffraction and supported by a particle size analyzer (NanoBrook 90Plus, USA) to be around 48.3 nm. YSZ NPs were added in 1 wt.% and 3 wt.% to 3D-printed resin, while one group remained unmodified, representing the control group. A total of 90 specimens were printed: 30 for the impact strength (IS) test, 30 for the surface hardness test, and 30 for the surface roughness (Ra) test, which is subdivided into three subgroups (n = 10) according to each test (Control, 1 wt.%, and 3 wt.% modified groups). The weighed nanoparticles were added to containers containing the base resin at the designated concentrations. The pure resin was initially mixed in an LC-3D mixer for 120 minutes to ensure the homogeneity of the resin before nanoparticle introduction (19). Subsequently, the pre-weighted YSZ nanoparticles were gradually added to the resin in the following mixing percentages (Table 1); the mixtures were thoroughly mixed and stirred for 5 minutes at 2500 rpm (20), followed by printing (21) (22).

Table 1: Weight percentage of YSZ nanoparticles combined with 3D printed resin of the test specimen's groups.

Groups	YSZ NPs (wt.%)	3D printed resin (g)	Added YSZ NP (g)
Control	0	150	0
1 wt. %	1	148.5	1.5
3 wt. %	3	145.5	4.5

Specimens of 50 × 6 × 4 dimensions for the IS are prepared according to ISO 20795- 1:2013 standards [23]. In comparison, the specimens' dimensions were 15 × 2 mm for the surface hardness and Ra tests. The specimens were designed using a CAD program, saved as an STL file and printed them by Asiga MAX UV. Layer printing had a 0-degree printing orientation and a 50 µm layer thickness [24]. The Optik Otoflash G171 curing unit, following the manufacturer's instructions, carried out the post-curing process with 2 x 2000 flashes under inert gas, turning components after each 2000 flash. Finally, the specimens were cleaned with 99.9% isopropyl alcohol and trimmed off any excess resin. Then, a polishing machine equipped with pumice powder, emery paper, and a tungsten carbide bur was used at 1500 rpm in wet conditions to finish and polish them [17]. A single operator did this procedure to confirm the similar pressure of polishing tools on specimens.

Impact strength test

A Charpy's Impact Strength Tester (Tinius Olsen, USA) was used to measure the IS. The scale gives measurements of impact energy in joules. The Charpy impact strength is calculated in kilojoules per square meter (kJ/m^2) by the following formula [2]:

$$\boldsymbol{Impact\ strength\ =\ E\, b.d\ \times\ 10^3}$$

Where E is the impact energy in Joules, b is the specimens' width in millimeters (mm), and d is their depth in mm.

Surface hardness test

According to ISO 6507-1:2005 25, a microhardness testing apparatus will determine the specimens' Vickers microhardness (HV) (LARYEE, China) [25]. A diamond pyramid with a square-based indenter subjects the testing specimens to a steady force of 50 g for 30 seconds. Three unique places are used for the indentations, and the average is computed [6].

Surface roughness test

A non-contact profilometer (Trane, United States) measures the specimens' surface Ra. At different locations, the samples will undergo three different 0.01 mm resolution radial scans. The average surface Ra (µm) will be calculated for every specimen. Ultimately, the software will analyze the images to pinpoint pit features.

Fourier Transform Infrared Spectroscopy (FTIR) test

The Fourier transform infrared spectrometer (FTIR) looks at how different frequencies of infrared light pass through things to figure out how the filler material and the polymer matrix might react chemically. Specimens were scanned at 4000-400 cm^2 to obtain corresponding FTIR spectra.

Statistical analysis

The Shapiro-Wilk test was used to assess the data's normality. Given the assumption of a normal distribution, one-way ANOVA was employed to determine significant differences among the tested groups. Subsequently, Tukey's post hoc test was conducted to evaluate the means between specific concentration groups pairwise.

Results

Figure 1 displays the absorption bands for the YSZ powder, control, and modified (1 wt.% and 3 wt.%) groups. The YSZ powder peaks in the 3400–3600 cm^{-1} region due to O-H Stretch (Hydroxyl Group). Peak around 1101–1035 cm^{-1} due to Zr-O Stretching typical in ceramics. The standard bands of sample

YSZ are attributed to CH_3 and CH_2 stretching vibrations (2921 cm^{-1} and 2867 cm^{-1}). Peaks at 1720, 1639, and 1458 cm^{-1} might be due to carboxylic acid groups. Bands below 800 cm-^{1} may denote the metal—oxygen stretching mode, while bands over 1190 cm^{-1} may be associated with the COH group [26].

For the control group, the absorption peaks match the vibrational modes of the functional groups in the specimen. The peak in 3276 cm^{-1} is the assignment of the N-H stretch. C=O stretching (carbonyl group) for a strong peak around 1730 cm^{-1} indicates the carbonyl group's existence, a key component of acrylic polymers. C-H stretching (aliphatic), several peaks in the 2966-2875 cm^{-1} region propose aliphatic C-H bonds common in acrylics. Peaks around 1643-1612 cm^{-1} and 1512 cm^{-1} are C=C Stretch (Alkene) assignments. C-O stretching (ester group) at 1247 cm^{-1}. Several peaks in the region of 1456 cm^{-1},1388 cm^{-1},1247 cm^{-1},1180-1153 cm^{-1}, and 661 cm^{-1} due to C-H bending, C-O stretch, C-O stretch (possibly from ether), and C-O stretching motions, typical for esters or ethers and C-H out-of-plane, respectively. These are all consistent with an acrylic resin [27].

Moreover, the analysis displays typical absorption bands for the modified groups (1 wt.% and 3 wt.%). The band corresponds to the stretching mode of the OH group. This peak group shifts from 3433 cm^{-1} to 3438 cm^{-1}. C=C Stretch (Alkene) shifted from 1631-1618 cm-1 to 1637-1614 cm^{-1}. Peaks at 700-400 cm^{-1} represent metal-oxygen bending. C-O stretching motions shift from 1178-1153 cm^{-1} to 1178-1155 cm^{-1}. 950 cm^{-1} and 810 cm^{-1} denote Zr-O stretching, which indicates that yttria-stabilized zirconia nanoparticles are embedded within the polymeric matrix.

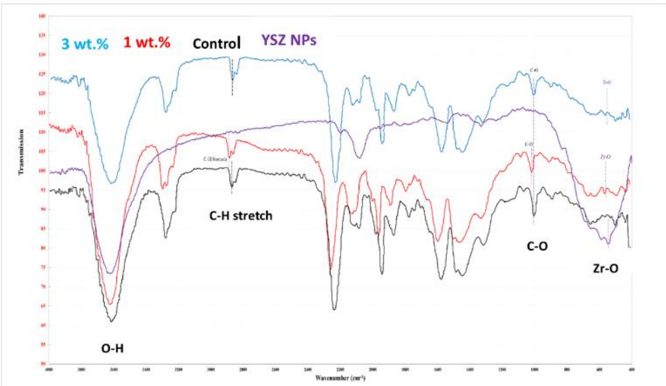

Figure 1: FTIR analysis results of YSZ NPs, control group, 1 wt. % and 3 wt.% YSZ NPs modified groups.

A Shapiro-Wilk test was directed to measure the normality of the data distribution. The outcomes showed that the data for all variables met the assumption of normality.

The impact strength test results are shown in Table 2 and Figure 2A; according to the descriptive statistics, the control group exhibited the lowest average impact strength (5.59 kJ/m^2), while the group containing 1 wt.% YSZ NPs had the highest (10.91 kJ/m^2). As revealed by Tukey's test, there were noteworthy differences ($p < 0.05$) in impact strength between the 1 wt.% and 3 wt.% modified groups compared to the control group. A significant difference was also detected between the 1 wt.% and 3 wt.% groups.

Table 2: Descriptive statistics of the impact strength values include an F-test by ANOVA.

Statistics	Control	1 wt. %	3 wt.%	F test	*P* value
Mean (kJ/m²)	5.59	10.91	9.756	130.77	<0.001 Sig.
Std. Deviation	0.504	1.085	0.604		
Minimum	4.62	9.6	8.79		
Maximum	6.35	12.63	10.78		

Vickers microhardness test results, as shown in Table 3 and Figure 2B, revealed that the control group exhibited the lowest average value (15.723), while the group contained 1 wt.% YSZ nanoparticles showed the highest average value (20.473). Tukey's test indicated significant differences ($p < 0.05$) in HV between the control group and 1 wt.% group, an essential difference between 1 wt.% and 3 wt.% groups, and insignificant differences between the control group and 3 wt.% groups.

Table 3: Descriptive statistics of the surface hardness values include an F-test by ANOVA.

Statistics	Control	1 wt. %	3 wt.%	F	*P*
Mean	15.723	20.473	17.82	12	<0.001 Sig.
Std. Deviation	2.229	2.503	1.713		
Minimum	12.8	16.4	15.9		
Maximum	20.17	24.8	20.6		

Surface roughness testing, as presented in Table 4 and Figure 2C, shows that the control group had the highest average value (0.601 µm), while the group containing 3 wt.% YSZ NPs exhibited the lowest average value (0.244 µm). Tukey's post hoc test showed significant differences ($p<0.05$) in surface Ra between the 1 wt.% and 3 wt.% modified groups compared to the control group and significant differences between the 1 wt.% and 3 wt.% modified groups.

Table 4: Descriptive statistics of the surface roughness values include an F-test by ANOVA.

Statistics	Control	1 wt. %	3 wt.%	F	*P*
Mean (µm)	0.601	0.404	0.244	28260.8	<0.001 Sig.
Std. Deviation	0.004	0.004	0.002		
Minimum	0.597	0.398	0.241		
Maximum	0.608	0.409	0.248		

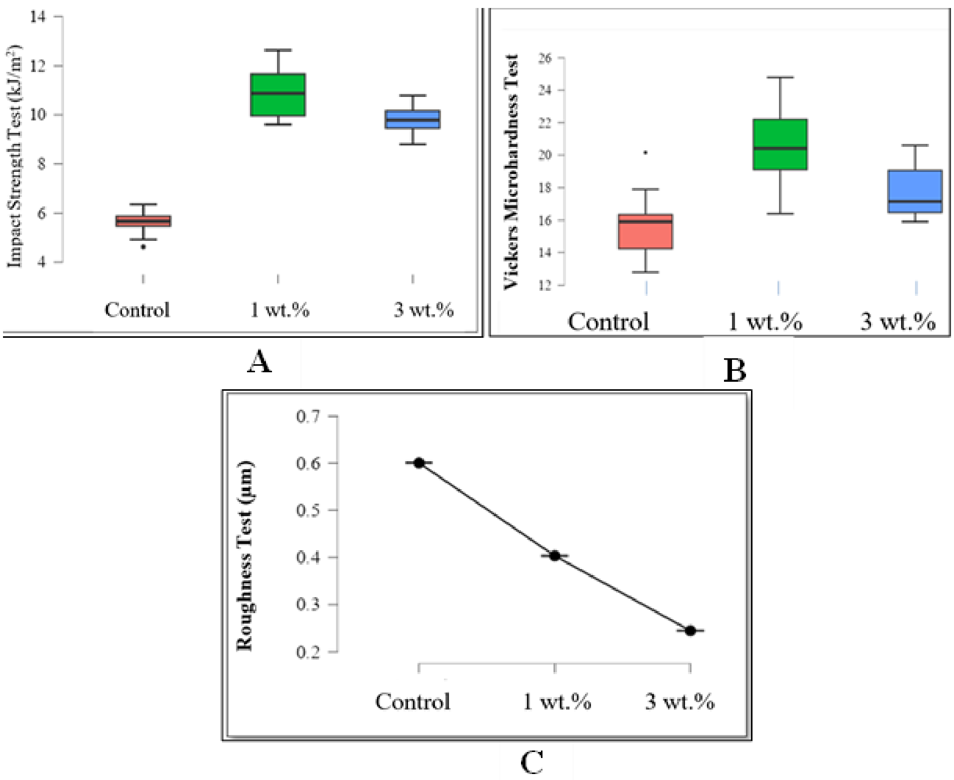

Figure 2: The box plot represents the mean and SD values of the A) IS test, B) HV test, and C) Ra test.

Discussion

Yttria-stabilized zirconia is a promising material for dental applications due to its exceptional mechanical properties, surface characteristics, and high biocompatibility. Incorporating YSZ nanoparticles into resin-based composites significantly enhances their overall performance. The YSZ reduces compressive stress at a crack's tip and with shear stresses, acting against the stress field generated in the crack area. Therefore, an increase in the toughness of zirconia in this phase (Y-TZP) results in preventing the propagation of cracks and improving the mechanical properties of zirconia. Additionally, the improved hardness and mechanical strength of these nanocomposites are attributed to the presence of fine-grained tetragonal zirconia polycrystals (TZP) within the YSZ nanoparticles [(20)].

The FTIR analysis of the pure and modified YSZ NPs specimens revealed comparable bands and characteristic features. These results show that adding nanoparticles did not change the nanocomposite's chain structure, as AlGhamdi et al. confirmed. Instead, the addition of nanoparticles only altered the intensity of band [(28)]. However, the matching features and bands between the two modified groups (1 wt.% and 3 wt.%) approve the homogeneous diffusion of YSZ nanoparticles into the 3D-printed denture acrylic [(19)].

The primary cause of denture fracture following a sudden fall is the low-impact strength of the denture base resin [(29)]. In such cases, the denture base material must exhibit adequate impact resistance to reduce the fracture risk. Impact and fatigue are the primary aspects contributing to maxillary denture fractures, while impact forces are reported to account for approximately 80% of mandibular denture fractures [(30)]. The IS test results for this study showed that 3D-printed denture base acrylic improved significantly. This is in line with what other studies have found, such as those by Chen et al. [(31)], Mangal et al. [(32)], Gad et al. [(33)], and Alshaikh et al. [(19)]. These studies found that IS improved when TiO_2 and PEEK, nanodiamond particles, SiO_2NPs, and ZrO_2NPs were added to 3D-printed resins.

The nanoparticle-modified groups presented higher values than the control group. The enhancement is attributed to the fine particle size and well-dispersed nanoparticles [(32)]. Furthermore, the high interfacial strength between the nanofiller and polymer matrix is expected to play a role in IS improvement as nanoparticles fill spaces between polymer chains, avoiding displacement and dissipating energy that causes crack propagation [(34) (35)]. The degree of polymerization directly influences surface hardness, a measure of a material's resistance to indentation, and indirectly determines its strength [(36)]. Denture base materials with low surface hardness are susceptible to damage from everyday activities like brushing, which can lead to discoloration, plaque buildup, and, ultimately, a compromise in the denture's structural integrity [(37) (38)]. In the current study, the statistical analysis results for the HV test showed significant differences between the tested groups except for the control. The enhanced mechanical properties of YSZ-modified resins can lead to more durable dentures, reducing the risk of fractures and breakage. This can be particularly important for factors that can increase stress on dentures.

The control group revealed a lower hardness, possibly due to its composition and 3D-printed resins' lower double-bond conversion than traditional denture base resins. According to earlier research by Aati et al.[(36)] and Gad et al. [(33)], who found similar results by adding ZrO_2 and SiO_2 nanoparticles, there wasn't a big difference between the 3 wt.% groups. However, the results disagreed with the Alshaikh et al. study, which showed an insignificant reduction in surface hardness values by adding ZrO_2 NPs, signifying that the composition of the material, the type, and the concentration of nanoparticles may basis the variances [(16) (19)]. The increase in cross-linking density might clarify the enhancement in nanocomposite denture base resin hardness, resulting in a more rigid and penetration-resistant polymer [(34)].

However, the HV test revealed a distinct pattern when related to the control and 3 wt.% groups; the 1 wt.% group presented a significant increase in hardness, and beyond this concentration, the addition of YSZ NPs in 3 wt.% had an unfavorable effect. It can be explained by stating that a higher concentration of nanoparticles beyond the saturation level can result in more residual monomers, which act as plasticizers and reduce hardness [(39)]. This is in line with earlier studies by Chen et al. [(31)] and Altarazi et al. [(40)], who reported a higher hardness at low concentrations compared to high concentrations and linked the primary cause to the degree of conversion. Nanocomposite resins' improved surface characteristics increase their resistance to abrasion and indentations.

The current investigation outcomes presented a statistically significant reduction in surface Ra of 3D-printed denture base acrylic by integrating YSZ NPs in 1 % and 3 % by weight. The results conform to previous research by Zidan et al., Abd Alwahab et al., and Sadoon (17) (41) (42).

The roughness may be decreased by filling the gaps between the polymer matrix with the additive of nanoparticles, thus diminishing the surface irregularities and voids (17). The smooth surface of the modified groups may also be attributed to the 50 μm layer thicknesses and the closely packed layers (38).

The current roughness results differ from those of Gad et al., who reported a significant increase in roughness compared to the control group when adding SiO_2 nanoparticles to 3D-printed denture base resin (33). Moreover, Alshaikh et al. revealed that the addition of ZrO_2 to 3D printed denture base resin resulted in insignificant roughness variations, suggesting that printing technology and parameters may have had a superior impact on Ra, layer by layer, depending on the printing orientation. The 90° printing orientation resulted in more compacted step-wise edges on the specimens' surfaces, increasing surface roughness between layers (19).

In fact, the clinical application of 3D-printed denture base materials has demonstrated their potential as viable alternatives to traditional heat-cured materials. Incorporating YSZ into these materials showed a promising result in overcoming the drawbacks of 3D-printed resin properties. This offers the opportunity to significantly enhance the performance and durability of dentures, thereby achieving long-lasting dentures, improving patient satisfaction, and addressing the limitations of current standard materials. This would aid in the transition to the next stage of clinical implementation (38).

Indeed, the study has faced some limitations regarding the printing process. Uncontrolled air entrapment in the resin fluid caused holes to form in and between the printed layers, which had a negative effect on the mechanical properties of the specimens that were made. Our investigation focused on using a single type of 3D-printed denture base material, a single NP, and one printing orientation. Furthermore, assessing mechanical or physical properties in dry conditions without reflecting significant aging in several liquid media, such as water, artificial saliva, or coffee, fails to represent real-life oral performance accurately.

Conclusion

The conclusions demonstrate the significant potential of YSZ-enhanced 3D-printed denture resin as a promising material in prosthodontics. FTIR proves that no chemical reaction occurred. Modifying the 3D-printed denture resin with 1 and 3 wt.% YSZ NPs significantly enhanced IS, while 1 wt.% YSZ NPs showed a notable improvement in HV. Moreover, both concentrations significantly reduced the surface Ra of the 3D-printed resin (concentration-dependent). These improvements exceeded those of unmodified resins and highlighted the superior clinical performance, durability, and patient comfort offered by YSZ-enhanced denture resin. Given the rising demand for personalized and cost-effective dental restorations, this pioneering nanocomposite material presents a significant opportunity for enhancing additive manufacturing in dentistry.

Recommendations

Although this investigation successfully incorporates YSZ NPs for denture applications, more research is necessary to enhance their performance. Future investigations should focus on enhancing antimicrobial properties, dimensional accuracy, and aging resistance to ensure long-term durability and sustainability in denture materials.

Conflict of interest

The authors have no conflicts of interest to declare.

Author contributions

Conceptualization, T.I.H; methodology, H.F.M and T.I.H; software, H.F.M and ZI; validation, T.I.H; formal analysis, H.F.M, and T.I.H; investigation, H.F.M, and T.I.H; data curation, T.I.H; writing—original draft preparation, H.F.M; writing—review and editing, T.I.H and ZI; visualization, T.I.H, and ZI; project administration, H.F.M. All listed authors have confirmed the authorship of this manuscript.

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يتريا مستقرة بالزركونيا: نـهج مبتكر لتحسين أداء راتنجات الأسنان المطبوعة ثلاثية الابعاد

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المستخلص:
الخلفية: ساهم تطور تقنية الطباعة الثلاثية الابعاد في تقدم راتنجات الأسنان ، على الرغم من أن هذه المواد تظهر حدودًا في خواصها الفيزيائية والميكانيكية. الاهداف: درس هذا البحث تأثير تعزيز راتنجات الأسنان المطبوعة ثلاثية الأبعاد باستخدام نانوجسيمات الزركونيا المستقرة باليتريوم، مع التركيز على مقاومة الصدم، وصلابة السطح، وخشونة السطح .كما درست تأثير إضافة نانوجسيمات YSZ بنسبة 1% و3% من الوزن إلى راتنج الأسنان المطبوع ثلاثي الأبعاد. المواد والطرق: تم تقسيم تسعين عينة من الأكريليك المطبوع ثلاثي الأبعاد إلى ثلاث مجموعات، وتم تقسيم 30 عينة لكل مجموعة إلى ثلاث مجموعات فرعية وفقًا لكل اختبار .تم استخدام جهاز اختبار مقاومة الصدم Charpy's Impact Strength Tester لتقييم مقاومة الصدم .وتم تقييم خصائص السطح باستخدام جهاز قياس صلابة Vickers (HV) microhardnessوجهاز قياس التضاريس غير الملامس (الخشونة) .تم استخدام مطياف التحويلات الفوري للأشعة تحت الحمراء (FTIR) للتحليل الكيميائي .تم إجراء التحليل الإحصائي وفقًا لبيانات التجربة، والإحصاءات الوصفية، وتحليل التباين (ANOVA)، واختبارات المقارنة المتعددة وفقًا لأهمية النتائج لكل اختبار. النتائج: أكدت النتائج أن إضافة 1% و3% من الوزن من نانوجسيمات YSZ إلى راتنج الأسنان المطبوع ثلاثي الأبعاد حسّنت بشكل كبير خواصه الميكانيكية والسطحية ($p < 0.05$) مقارنة بالراتنج غير المعدل. الخلاصة: ساهم إضافة نانوجسيمات YSZ في تحسين مقاومة الصدم، وصلابة السطح، والخشونة .أظهرت تقنية FTIR عدم وجود تفاعل كيميائي بين البوليمر ونانوجسيماتYSZ ، مما يثبت إمكانية الحصول على قاعدة أسنان نانوتركيبية مطبوعة ثلاثية الأبعاد ذات أداء محسّن.