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The Effect of Titanium Oxide Nanotubes on the Surface Hardness of a Three-Dimensional Printed Denture Base Material

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Abstract:

Background: In the field of dentistry, there has been a growing prevalence of digital manufacturing technologies in recent years. Presently, the manufacturing of removable complete dentures using three-dimensional printing can be achieved through the utilization of computer-aided design and computer-aided manufacture (CAD-CAM) technology. This technology combines additive and subtractive techniques, commonly referred to as fast prototyping and three-dimensional printing, and employs PMMA professional light-curing resin as the material. The objective of this study is to assess the impact of titanium oxide nanotubes on the hardness of the underlying material utilized in 3D printed dentures. **Materials and methods**: The specimens were generated using 3D builder software developed by Microsoft Corporation, utilizing computer-aided design techniques. Specimens were designed with particular dimensions in compliance with the test standards for hardness. The denture resin 3D printed with titanium oxide nanotubes (Howngue, China TiO_2 / D: 15-30 nm / L: 2-3 nm) was categorized into five groups (group 1 - 1% / group 2 - 1.5% / group 3 - 2% / group 4 - 2.5% / group 5 - 3%) based on weight. The specimens were printed using the Asiga max 3D printer, which utilizes digital light processing technology and is manufactured in Australia. **Results**: The average hardness values for the groups reinforced with 1% by weight of titanium oxide nanotubes showed a significant increase compared to the control group and with 1.5 % showed no significant difference. In contrast, the values for the other reinforcement groups declined considerably. **Conclusion**: The hardness of denture bases made from three-dimensionally printed material was successfully increased by including titanium nanotubes at concentrations of 1% by weight.

Keywords: titanium oxide nanotube, 3D printed resin, hardness.

1.Introduction

The polymer polymethyl methacrylate (PMMA) is the material most frequently used to create traditional complete dentures. The material's relative simplicity in manufacturing, repair, biocompatibility, and aesthetic qualities have enhanced patient acceptance(yousif, 2023).

It is inexpensive, has the desired aesthetic quality and easy to process. It does not have a single desirable property, but rather a combination of qualities that take into account its popularity and use (Zahraa, 2023).

For individuals seeking to maintain their physical and emotional well-being, it is often necessary to replace their natural teeth with artificial alternatives, such as acrylic dentures (faot, 2006). Polymeric polymers are commonly used for denture bases due to their practicality and cost-effectiveness compared to dental implants (chaldek, 2019). PMMA, also known as polymethyl methacrylate, has been widely utilized as the primary material for dentures since its inception in 1936. The colorless polymer of methyl methacrylate possesses exceptional mechanical strength, limited solubility in water, high elastic modulus, and dimensional stability, making it an excellent choice as a basis material for dentures. These items must exhibit dimensional stability under various temperature conditions and possess adequate toughness and strength to endure the pressures generated during their operation (Zarb, 2013).

Digital approaches provide the benefits of expedited denture production and a reduced number of steps in the process, resulting in decreased mistake rates. Research has shown that these new denture base resins have better mechanical qualities than traditional heat-polymerized PMMA (ALP, 2019).

The utilization of computer-aided design (CAD) and computer-aided machining (CAM) has revolutionized the procedure of fabricating removable dental prosthesis (RDPs). RDP has been produced using additive manufacturing techniques, specifically 3D printing, and the outcomes have been favorable (Srinivasan, 2020)(unkovisky, 2019).

Thanks to the progress in 3D printing technology, the dentistry sector now provides a diverse array of items. The two predominant categories of fixed prosthodontic models are derived from wax patterns and intraoral or impression scans (Chopra, 2020).

Various types of 3D printers are available. The two most popular technologies used in dentistry are digitally processed light (DLP) and liquid crystal displays (LCD). In DLP, a projector projects the 3D model's image onto the liquid polymer. The DLP printer is a faster technology that produces products with higher resolutions (Bhalero, 2018)**.**

When compared to traditional and milled denture base materials, 3D-printed resins exhibited the lowest flexural strength and surface hardness (Prpic, 2020). However, it is approaching the ISO-recognized criterion of 65 MPa for transverse strength. Due to these constraints, the utilization of 3D-printed technology in the medical sector is now limited and is not optimal for fabricating denture bases (Totu, 2017). Prior research has examined many aspects that affect the characteristics of 3D-printed resin, including the direction of printing, the thickness of each printed layer, and the duration of the curing process. The aim of these studies was to address the aforementioned constraints and maximize the advantages offered by 3D printing technology (Reymus, 2019)(Kebler, 2021). Several research (Mangal, 2020)(Aati, 2021) focused on the incorporation of different chemicals into 3D-printed structures. Several individuals have attempted to enhance the characteristics of polymers by including nano-fillers (Tukmachi, 2020)(Mohammed, 2020). Nanofillers are highly effective fillers for acrylic resins due to their high surface free energy and large surface area. They significantly enhance the mechanical properties of the resin (Ihab, 2021)(Ihab, 2014).

Abdul Albaqi discovered that the addition of 1.5 and 2 weight percent Yttrium oxide nanoparticles greatly enhanced the binding strength between the soft liner and the denture base(Abdul Albaqi, 2022). Nevertheless, the mechanical qualities were not investigated.

Totu et al. found that the addition of TiO_2 nanoparticles enhanced the antibacterial characteristics of the 3D-printed resin. However, no experiments were conducted to assess its mechanical properties (Totu, 2017).

Mangal et al. assert that the incorporation of 0.1 weight percent animated nanodiamonds improved the mechanical properties of 3D-printed polymers, rendering them appropriate for the manufacturing of orthodontic equipment (Mangal, 2020). Similarly, Aati et al. showcased the prolonged improvement of 3D-printed polymers by using ZrO_2 nanoparticles for temporary repairs (Aati, 2021). Chen and his colleagues added two substances, silver nanoparticles and cellulose nanocrystals (CNCs), to a resin that can be printed in three dimensions. Based on their analysis, the altered resin exhibited enhanced mechanical and bactericidal characteristics (Chen, 2018).

According to Li et al., it is advised to add titanium oxide nanotubes (TiO_2) to PMMA denture base resins(Li, 2016).

Because of their large specific surface area, solid metal support contact, excellent chemical stability, and catalytic activity, they improve PMMA's mechanical characteristics.

As far as the authors are aware, there has been no prior investigation on the effects of resin printed in three dimensions on TiO_2 nanotubes with regards to hardness. The present study examined the impact of including TiO_2 into three-dimensional resin printing. The original null hypothesis proposed that the hardness of the denture base resin, when combined with TiO_2 nanotubes, would not undergo any changes.

2.Materials and Methods

2.1 Study design:

The denture base resin (Dentbase/Asiga, Australia) was subjected to testing using titanium oxide nanotubes (D: 15-30 nm/L: 2-3 nm) sourced from Hongwu, China. The nanotubes were treated in varying quantities of 1%, 1.5%, 2%, 2.5%, and 3% by weight. The results were compared to those of the control group, which did not receive any titanium oxide nanotubes. The study involved conducting tests on the hardness of specimens prepared with different percentages (1%, 1.5%, 2%, 2.5%, and 3% wt.) of TiO_2 nanotubes mixed with resin. A total of 60 specimens were prepared for this study, including one control specimen made of pure resin and five specimens for each TiO_2 nanotube percentage. There are ten specimens in each category for testing.

2.2 Samples preparation:

The specimens depicted in figure 1-A were digitally generated using an open-source software called 3D Builder, developed by Microsoft Corporation. The specimens were fabricated to meet the prescribed specifications for surface hardness testing, which are 65 mm x 10 mm x 2.5 mm, as outlined in ADA No. 12 standard (1999). Once the design is finished, the STL file is submitted and uploaded to the Asiga Composer program, which is used to export the file as an STL. This exported file, shown in figure 1-B, is then sent to the printer for printing, as shown in figure 1-B.

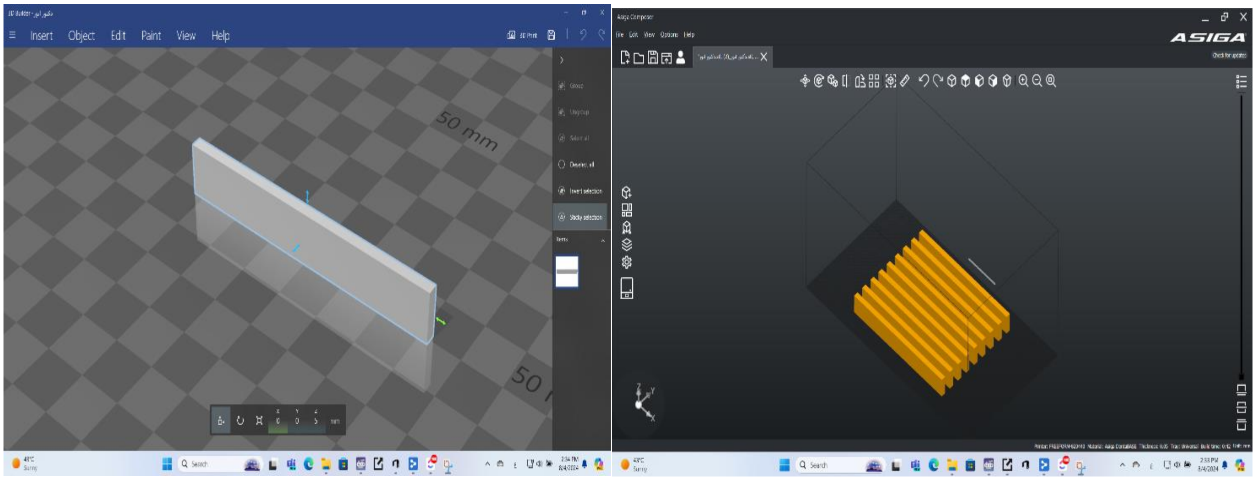

Fig 1: A) 3D builder software, B) Asiga composer software.

2.3 The titanium nanotubes incorporation:

Color pigments, which are one of the components of resin, tend to settle in the bottom of the bottle. As a result, it will cure unevenly, and the prints may fail. It is imperative to vigorously shake the resin bottle for 30 to 60 seconds, as advised by the manufacturer, prior to introducing the resin to the resin vat. In order to achieve a uniform blend of printed denture base resin, an exclusive shaker (Mazic D mixer/ ackureta, Taiwan) was initially employed. Subsequently, a total of 3 milliliters of ethyl alcohol with a purity of 99.9% were combined with titanium nanotubes sourced from Hongwu, China. This mixture was then subjected to ultrasonic treatment using an MSE soniprep 150 probe from the Netherlands for a duration of three minutes. The purpose of this process was to produce a nanotube suspension that would yield improved outcomes when combined with resin due to its elevated viscosity.

The denture base resin was combined with a suspension of titanium oxide nanotubes in a dark amber glass container. The container was left open for a short period of time to allow the alcohol to evaporate, and then it was closed to prevent exposure to surrounding light. The alfa HS-860 magnetic stirrer from Iran was employed to produce a nanocomposite for printing denture base resin. During the process, TiO2 was gradually incorporated into the resin solution while being constantly stirred in the magnetic stirrer for 30 minutes at a temperature of 60°C. This stirring action was done to decrease the viscosity of the mixture. Subsequently, the mixture was stirred for a duration of 8 hours at ambient temperature (25 °C).

Nanotubes are elongated, cylindrical structures with a central hollow cavity. The walls of titanium nanotubes are always composed of layers, which might range from two to ten. The tubular shape and multilayered structure of Titania improve the contact at the interface, resulting in an increased surface area-to-volume ratio of the material.

The presence of reinforced fillers in the PMMA resin has led to enhanced interaction with TiO2 nanotubes, resulting in an increased quantity of the nanotubes (Bavykin, 2010).

2.4 Printing of 3-dimentional specimen blocks (3-D model)

The 3D-printer (DLP) technology from Asiga in Australia was used to print the specimens while maintaining the following settings according to manufacturer instructions: heater temperature = 30, light intensity = 19, offset = 0.09, xy scale = 1.007, separation pressure limit = 300 g/cm2, separation velocity = 4.3 mm/s, and slice thickness = 0.05.

Once the printer vat was filled with the liquid polymer mixture, which consisted of resin with titanium nanotube filler, the lid was closed in order to prevent the mixture from being exposed to room light. As the plate is lowered, the liquid polymer mixture solidifies due to the presence of light. This process continues till the 3D model is finished. After the sculpture has hardened, the liquid in the container is removed to expose it. The printing was carried out with precision using a layer thickness of 50 µm. The process of printing 20 specimens can be completed in around 35 minutes.

2.5 Cleaning, drying, and curing

The sample was meticulously removed from the 3D printer platform using a precise blade. Following a three-minute ultrasonic cleaning using Clean I from Ackureta in Taiwan, the printed specimens were further cleaned for two minutes using 99.9% isopropyl alcohol from Scharlau in Spain to eliminate any residual uncured resin. The specimens were dried thoroughly and then subjected to polymerization using a light-curing method in a UV box from Ackureta, Taiwan. The process involved exposing the specimens to light at a wavelength of 405 nm for five minutes on each side, following the manufacturer's guidelines. Subsequently, a German-made acrylic bur and lathe polishing equipment were employed to finalize and refine the specimen. In order to determine the accurate dimensions, the specimens were measured using a digital vernier caliper manufactured by Kirti NDT/India. Commencement of testing might be initiated with the prepared specimens.

2.6 Surface hardness test (Shore D)

The example was manufactured in accordance with ADA standard No. 12 (1999) and has dimensions of 65 mm x 10 mm x 2.5 mm. The hardness of the acrylic resins was assessed using the Time Group Inc./China hardness testing machine. The apparatus had a blunt-pointed indenter with a diameter of 0.8 mm that gradually increased to 1.6 mm. A digital scale, ranging from 0 to 100 units, was connected to the indenter. The indenter was rapidly and forcefully driven downward. Measurements were obtained directly from the digital scale's display.

2.7 Statistical analysis method

Data analysis was conducted using the statistical software SPSS (Statistical Package for Social Science, version 21) and Prism 9 (GraphPad Software, USA). The results are presented in the form of bar charts, which show the average values and the variability of the data, serving the goal of descriptive analysis. The P values indicating non-significant, significant, and highly significant differences were greater than 0.05, less than 0.05, and less .than 0.01, respectively

3.**Result and discussion**

3.1**Test of hardness**

The addition of TiO_2 significantly increased the hardness of 3D-printed resin, and the highest value was recorded with TiO_2 1%% and 2% (87.7%), which exceeded the control hardness value (85.9%). At 1.5%, there was no significant increase in comparison to the control group. When TiO_2 increased by more than 2.5%, it showed a significant decrease in .hardness, as shown in table 1 and Fig. 6

Table 1: Descriptive Data of Shore D hardness test

Groups	Mean	±SD	±SE	Minimum	Maximum
Control	82.540	1.800	0.569	80.700	84.700
1%	86.580	0.716	0.226	85.700	87.800
1.5%	85.000	0.917	0.290	84.100	86.600

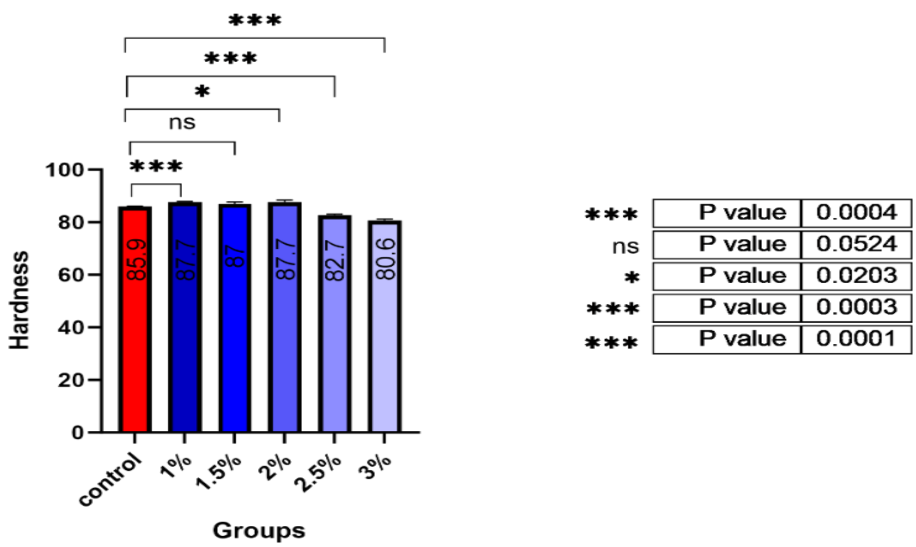

Fig 2: Hardness test results

4.**Discussion**

This study evaluated the hardness of the denture base material produced using 3D printing technology after including titanium oxide nanotubes.

The mechanical properties of 3D-printed resin are influenced by the orientation, curing procedure, and building parameters (Lin, 2020). Software, layer thickness, layer number, and layer shrinkage are all important factors (Kown, 2021). The printing settings for the manufacturer are usually determined, save for the layer height (thickness) and printing orientation, which are fixed and cannot be altered. Within the range of 25 to 200 µm (Revilla, 2019), a layer thickness of 100 Mm is deemed sufficient. The printing process in this investigation utilized a layer thickness of 50 µm. Decreasing the layer thickness of a 3D-printed object enhances its robustness by facilitating better resin drying and minimizing dimensional variations (Kessler, 2020). Moreover, using thinner layers in printing allows for more precise capture of geometric details. However, it is important to note that these benefits are accompanied with increased costs and a larger likelihood of print errors (Kessler, 2020).

The DLP printer was chosen for this study due to its superior speed and ability to manufacture objects with a greater resolution (Bhalero, 2018). DLP technology differs from stereolithography in that it uses patterned laser light to cure all layers at once, rather than scanning each area sequentially with the laser. Due to this advantage, the duration of building is unaffected by the quantity of items or the shape of the corresponding layer (Gad, 2021).

Scientists have achieved the identification and production of nanomaterials in the form of nanotubes due to their exceptional properties, enabling them to function highly effectively in various applications.

Denture bases fabricated using 3D-printed materials represent a novel approach to denture production, albeit with inferior mechanical and physical characteristics compared to conventional denture bases (Gad, 2021).

Titanium dioxide (TiO_2) nanotubes possess a very large surface area, a robust interaction with metal supports, exceptional chemical stability, elevated catalytic activity, and outstanding performance in both acidic and alkaline conditions. This attribute renders them advantageous in numerous circumstances.

Hardness is the measure of a material's ability to withstand deformation caused by mechanical indentation or abrasion. Plaque buildup and staining can occur as a result of mechanical denture cleaning (Aati, 2021). The decrease in hardness can be attributed to various factors such as the type of material, the printing layers, and water absorption at high temperature circumstances.

Figure 2 displayed the hardness values. Out of all the other groups, the titanium oxide nanotube exhibited the greatest average proportion, which was 1%.

This finding is consistent with (Porras, 2016). The study discovered that incorporating 0.25 wt.% and 0.5 wt.% of SiO_2 nanoparticles into Next Dent 3D printed resin resulted in an enhancement in the hardness of the materials. This improvement was observed when the samples were printed at a 90° angle and with a layer thickness of 50 μm, which aligns with the findings of a previous study (Alshaikh, 2013). Also is consistent with (Shahad.,2024) which found that surface hardness values of acrylic increased after addition of Zirconia/Chitosan filler. Conversely, the study discovered that including ZrO_2 NPs into NextDent and ASIGA 3D-printed resins led to a minor reduction in hardness, in contrast to the unmodified resin.

With the increasing popularity of digitally manufactured dentures, this study highlights the importance of further research on the mechanical properties of the new denture base made from 3D materials. It also emphasizes the need for comparisons between different printing and brand combinations of technologies under conditions that closely resemble the oral environment. The mechanical and physical qualities of 3D-printed denture bases are inferior, despite the intuitive notion of comparing materials based on their fabrication techniques. To optimize the advantages of digital manufacturing, it is recommended to enhance the resin used for printing by modifying its composition or reinforcing it. Further investigation is required in these domains as they are thought to have a significant influence on the optimal printing alignment, the time of curing and post-curing, the thickness of printing layers, the method of polishing, and the storage of specimens for 3D-printed resin.The statistical analysis revealed variations in surface hardness between the study groups, resulting in the null hypothesis being rejected, which postulated that the 3D-printed denture base resin's characteristics would remain unchanged upon the incorporation of TiO2 nanotubes.

Several factors served as constraints for this investigation. As a result of insufficient past study in this field, we did not select the percentages and qualities of nanotubes in the most optimal manner. It is advisable to modify the surfaces of titanium oxide nanotubes in order to enhance interactions at the interface between titanium nanotubes and the denture base polymer matrix. Further investigation is necessary to ascertain the optimal concentration of titanium nanotubes required to attain the desired biological characteristics in denture base resins, including antibacterial properties and biocompatibility.

4.Conclusions

The incorporation of titanium oxide nanotubes into the basic material for 3D-printed dentures was successfully achieved. Nanotubes were incorporated to enhance the hardness of the acrylic resin. The extent of these enhancements was directly related to the quantity of additional titanium oxide nanotubes included. Furthermore, the preliminary inquiry has uncovered a possibly valuable new compound for enhancing the foundational resin material .utilized in 3D-printed dentures

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