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JBSA-LACKLAND, TEXAS 78236

THESIS APPROVAL PAGE FOR MASTER OF SCIENCE IN ORAL BIOLOGY

Title of Thesis: **“Mechanical Properties of Denture-base Resins Processed by Conventional and 3D Printing Methods”**
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Master of Science
19 May 2023

THESIS/MANUSCRIPT APPROVED:

LIEN.WEN.1234442429 Digitally signed by LIEN.WEN.1234442429 Date: 2023.05.22 09:36:12 -05'00'

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Mechanical Properties of Denture-base Resins Processed by Conventional and 3D Printing Methods

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USU Operational Gap: IV, A

Abstract

Limited research has been published evaluating the mechanical properties of new 3D-printed denture-base materials. Objective: The purpose of this study was to evaluate the mechanical properties (flexural strength, flexural modulus, nanoindentation hardness, nanoindentation modulus, and diametral tensile strength) of three new denture-base materials processed via 3D printing (Denture Base LP Resin 1L, Formlabs; NextDent Denture 3D+, NextDent; Dentca Denture Base II, Whipmix) and compare them with a conventionally processed denture-base material (Lucitone 199, Dentsply Sirona). Methods: Flexural strength (FS) and flexural modulus (FM) were measured using a 3-point bend test with rectangular beams (n=12). Nanoindentation Hardness (NH) and nanoindentation modulus of elasticity (NM) were tested using nanoindentation with the fragmented specimens from the flexural strength test (n=6). Cylinders (n=12) were loaded to failure in compression to determine diametral tensile strength (DTS). Data were analyzed with ANOVA and Tukeys post hoc tests (alpha=0.05). Results: There was no significant difference in FS between the 3D-printed and conventionally processed materials. Lucitone 199 had significantly lower FM compared to all other materials. Although Denture Base LP Resin and Dentca Denture Base II had the greatest DTS, Denture Base LP Resin had the greatest NM. Conclusions: All three 3D-printed materials in this study demonstrated significantly higher or similar mechanical properties than the conventionally processed material. Based on the mechanical properties tested, the 3D-printed denture-base materials could serve as a viable alternative to conventionally processed denture-base materials.

Introduction

With the growth in life-expectancy, the number of edentulous patients seeking treatment for replacing their teeth is on the rise. (1, 2) According to a recent study (3), the global prevalence of edentulism has already exceeded 10% for adults aged 50 and older. Removable dental prosthesis or denture is one of the most common treatment options for patients suffering from complete or partial edentulism. (4)

Since the introduction of poly-methyl methacrylate (PMMA) in 1936, it has remained the material of choice for dentures. The advantages of PMMA include ease of processing, low cost, light weight, insolubility in oral fluid, adequate strength, low toxicity, excellent aesthetic properties, and ability to be repaired easily. (5-7) Conversely, the shortcomings of PMMA are polymerization shrinkage, inferior flexural strength, lower impact strength, and low fatigue resistance. (8) These limitations have led to the development of novel denture-base materials and manufacturing. (1)

Computer-Aided Design/Computer-Assisted Manufacture (CAD/CAM) was introduced to dentistry in the 1970s. Today, CAD/CAM has been widely accepted by dental professionals, fabricating from fixed dental prostheses to surgical stents. The advantages of CAD/CAM are efficiency, automaticity, simplification, and streamlining of treatment-workflow. (9, 10) Fabrication of a complete denture using CAD/CAM is slowly gaining popularity, as dental professionals and commercially-available laboratories begin to realize the many benefits of digital dentistry. (11)

Currently, most CAD/CAM dentures are made by the subtractive manufacturing or milling. The additive manufacturing or 3D printing, has mainly been used for fabricating interim dentures and try-in appliances and rarely for definitive complete dentures. (12) However, additive manufacturing offers a few unique advantages over subtractive manufacturing which includes consuming less raw materials, spawning fewer wastes, and better replication of complex geometric patterns. (10)

Past studies have compared the material properties of dentures as well as their accuracies fabricated by conventional technique versus those made by subtractive manufacturing. (11) However, limited research has been conducted of new 3D-printed denture-base materials.

To our knowledge, only limited mechanical properties of a few 3D printing denture-base materials (Denture Base LP, NextDent 3D+, Dentona 3D, DentaBase, and IMPRIMO LC Denture Base) have been evaluated in a laboratory study.(13) Limited studies have been published comparing other properties such as flexural strength and modulus, nanoindentation hardness and modulus and diametral tensile strength of the following new denture-base materials: Denture Base LP Resin 1L (Formlabs), NextDent Denture 3D+ (NextDent), and Dentca Denture Base II (Whipmix). The aim of this in vitro study was to evaluate the mechanical properties of the three aforementioned new denture-base materials processed via 3D printing and compare them with a conventionally processed denture-base material, Lucitone 199. The null hypothesis was that 3D printed denture-base materials would not be significantly different from conventionally processed denture-base materials in mechanical properties.

Materials and methods

The following materials were selected for denture base fabrication: three types of 3D printing materials and one heat-polymerized acrylic material. A list of the materials, manufacturers, and denture base fabrication techniques is provided in Table 1.

Specimen Preparation

3D-Printed Specimens

A cylindrical block (Ø = 15 mm, height = 2.5 mm) and a rectangular bar (64 mm x 10 mm x 3.3 mm) were designed via DS SoldWorks software (SolidWorks, Waltham, MA) and were saved as a standard tessellation language (STL) file. Using this STL file, 12 cylindrical specimens and 12 rectangular specimens per group were printed using a compatible printer. Denture Base LP Resin 1 L was printed with Formlabs Form 3B printer (Formlabs, Somerville, MA). NextDent Denture 3D+ was printed with NextDent 5100 (NextDent, Utrecht, Netherlands), and Dentca Denture base II was printed with Asiga Pro (Asiga, Sydney, Australia).

Following the manufacturer instructions, Denture Base LP Resin 1 L specimens were placed in a Form Wash that contains isopropyl alcohol (IPA, ≥90%) for 10-20 minutes. Any excess resin was removed with a brush and then dried with compressed air. The specimens were then left to dry in a well-ventilated area for thirty minutes. If any uncured resin was still present, isopropyl alcohol was sprayed onto the specimen and dried again for thirty minutes. Then specimens were cured for 30 minutes at 80 degrees Celsius via Form Cure (Fromlabs). (14)

Dentca Denture Base II specimens were washed for 10-20 minutes using isopropyl alcohol in an unheated ultrasonic bath. Any excess resin was removed with a brush and then dried with compressed air. The specimens were then left to dry in a well-ventilated area for thirty minutes. If any uncured resin was still present, isopropyl alcohol was sprayed onto the specimen and dried again for thirty minutes. The specimens were cured for 40 minutes at 60 degrees Celsius via CUREbox (Wicked Engineering, East Windsor, CT).

NextDent Denture 3D+ specimens were cleaned for 3 minutes in reusable ethanol solution (>90%) using an unheated ultrasonic bath, then 2 minutes using fresh ethanol solution (>90%) using an unheated ultrasonic bath. The specimens were dried for 60 minutes. The specimens were cured for 30 minutes at 60 degrees Celsius via CUREbox (Wicked Engineering).

All the specimens were polished using pumice for 90 s at a rate of 1,500 rpm (KaVo Dental Bench Grinder, KaVo Dental, Biberach, Germany). They then underwent additional polishing using a soft leather wheel for 90 s at a rate of 1,500 rpm. Thickness, length, and width of each specimen were verified using an electronic digital caliper (GA 182, Grobet Vigor, Carlstadt, NJ). (14)

Heat-Compressed Specimens

A 3D-printed cylindrical block (Ø = 15 mm, height = 2.5 mm) and a rectangular bar (64 mm x 10 mm x 3.3 mm) were embedded in putty (Sil-Tech, Ivoclar Vivadent, Schaan, Liechtenstein) to create a negative impression, putty-matrix mold. Heated pink base plate wax (Base Plate Wax, Yamahachi Dental, Nutley, NJ) was dripped into the putty-matrix mold to form wax duplicates. (5) The wax patterns were then invested in a dental flask, using dental stone plaster (Silky Rock, Whipmix, Louisville, KY). Once the plaster was completely set, dewaxing was done. The wax was removed by flushing away with hot boiling water by using a boil-out/curing unit (26105SSWC Boilout/Curing unit, Handler Manufacturing, Bethel, CT).

The mold space thus obtained was used to fabricate heat-compressed specimens having cylindrical and rectangular geometries. The flask was allowed to dry and cool at room temperature. Then, separating agent (Al-Cote, Dentsply Sirona, Charlotte, NC) was applied on all surfaces and allowed to dry. (15) According to the manufacturer's instructions, Lucitone 199 was mixed in 32cc powder: 10ml liquid ratio. Lucitone 199 was packed into the cylindrical mold space (n=12) and rectangular mold space (n=12) in the dough stage (dough time 10 min) at a packing pressure of 2500 psi via a manual mechanical press (Pneumatic Flask Press, KaVo Dental). The heat-cure cycle included exposure at 73°C for 9 hours, followed by terminal boiling treatment at 99°C for 1 hour in a curing unit (Boil Out Unit Wapo-Ex 12 II, Wassermann, Hamburg, Germany). After 30 min of bench-cooling and deflasking, the processed specimens were polished as before. Thickness, length, and width of each specimen were verified using an electronic digital caliper (GA 182, Grobet Vigor, Carlstadt, NJ). (14)

Specimen Testing

Flexural strength (FS) and modulus (FM)

The rectangular specimens (n=12 per group) were placed in deionized water at 37°C for 24 hours prior to testing. Each specimen was tested using a universal testing machine (Alliance RT/5, MTS, Eden Prairie, MN) at a crosshead speed of 5 mm/min. Each specimen was placed on a three-point bending test device, which was constructed with a 50 mm span length between the supporting rods, and the central load was applied with a head diameter of 2 mm. The flexural strength (σ_{FS}, MPa) was obtained using the expression,

$$\sigma_{\mathrm{FS}} = \frac{3\mathrm{Pl}}{2\mathrm{bd}^2}$$

where P was the loading force at the fracture point in Newtons; l was the length of the support span (50 mm); b was the width; and d was the thickness. Width and thickness measurements of the rectangular specimens were made using the electronic digital caliper. Flexural modulus was determined from the slope of the linear region of the load-deflection curve using the analytical software.

Nanoindentation hardness (NH) and modulus (NM)

To measure the nanoindentation hardness and modulus at room temperature, the fragmented specimens from the flexural strength test (n=12 per group) were tested using a nanoindenter (iNano, Nanomechanics, Oak Ridge, TN) fitted with a Berkovich tip (#TB26961, Micro Star Technologies, Huntsville, TX) with a 20 nm radius and 65.3°nominal angle. All indentations were performed after the thermal drift rate reached below 0.05 nm/s threshold. The maximum indentation load was 8 mN, and Poisson's ratio for all specimens was 0.3. An array of indents (20 indents) was imprinted on the specimen surface. Each consecutive indent was spaced 2 µm apart from each other to avoid any interference of residual stresses from adjacent imprints. Force–displacement curves for the indents were used to evaluate the elastic moduli. For each indent, elastic modulus was calculated using the standard methods of Oliver and Pharr. (16) The elastic modulus, E (GPa), per group was computed with the following expression,

$$\mathrm{E} = (1 - \mathrm{v}^2)\left(\frac{1}{\mathrm{E_r}} - \frac{1 - \mathrm{v_{tip}}^2}{\mathrm{E_{tip}}}\right)^{-1}$$

where v and E_r (GPa) were the Poisson's ratio (v = 0.3) and reduced modulus, and v_{tip} and E_{tip} (GPa) were the Poisson's ratio (0.07) and elastic modulus (1141 GPa) of the Berkovich indenter, respectively. The nanoindentation hardness was obtained from the indentation load divided by the projected contact area, A (nm^2): Hardness = P/A, where P (mN) was the maximum contact force exerted by the indenter onto the sample.

Diametral tensile strength (DTS)

The 12 cylindrical specimens per group were placed in deionized water at 37°C for 24 hours prior to testing. Each cylindrical specimen was destructively tested to determine DTS. The specimens were placed on their longitudinal side on the platens of a universal testing machine (Alliance RT/5, MTS, Eden Prairie, MN). Then, the specimens were loaded to failure in compression at a crosshead speed of 0.5 mm/min. The DTS (σ_{DTS}, MPa) was calculated from the following equation:

$$\sigma_{\mathrm{DTS}} = \frac{2\mathrm{P}}{\pi \mathrm{DT}},$$

where P was the maximum fracture load in Newtons (N); D was the diameter (15 mm) of the specimen; and T was the height (2.5 mm) of the specimen.

A mean and standard deviation was determined per group for each property. Data were analyzed with a one-way ANOVA and Tukey's post hoc test to evaluate the effect of material type on the mechanical property (alpha = 0.05) using SPSS statistical software (IBM, Chicago, IL).

Results

The greatest flexural strength was demonstrated by NextDent Denture 3D+ (108.92 ± 4.73 MPa), while Denture Base LP Resin (99.10 ± 12.21 MPa) had the lowest. However, there was no statistically significant difference in flexural strength between any of the groups ($p < 0.999$). See figure 1. Denture Base LP Resin (2.78 ± 0.06 GPa) and NextDent Denture 3D+ (2.77 ± 0.16 GPa) had significantly greater flexural modulus values compared to all other groups ($p<0.016$), but they were not significantly different from each other ($p = 0.999$). Lucitone 199 had the lowest flexural modulus (2.27 ± 0.12 GPa) and was significantly lower than all the other groups ($p < 0.001$). Dentca Denture Base II (2.60 ± 0.15 GPa) performed more moderately and was significantly lower than Denture Base LP Resin and NextDent Denture 3D+ ($p<0.016$), but greater than Lucitone 199 ($p < 0.001$). See figure 2.

Denture Base LP Resin had a significantly higher nanoindentation hardness value (0.47 ± 0.049 GPa) compared to all other groups ($p < 0.002$). Lucitone 199 had the lowest hardness (0.35 ± 0.014 GPa), but it was not significantly different ($p > 0.145$) from NextDent Denture 3D+ (0.39 ± 0.013 GPa), or Dentca Denture Base II (0.40 ± 0.011 GPa). See figure 3. Denture Base LP Resin exhibited significantly greater ($p < 0.033$) nanoindentation modulus (6.57 ± 0.80 GPa) compared to other groups, followed by the Dentca Denture Base II (5.86 ± 0.20 GPa). Lucitone 199 (5.03 ± 0.08 GPa) and NextDent Denture 3D+ (4.91 ± 0.05 GPa) had the lowest nanoindentation modulus, but they were not significantly different from each other ($p = 0.983$). See figure 4.

Dentca Denture Base II had the greatest diametral tensile strength (36.36 ± 3.36 MPa), but it was not significantly different ($p = 0.926$) from Denture Base LP Resin (34.84 ± 4.20 MPa). NextDent Denture 3D+ had the lowest diametral tensile strength (28.51 ± 6.59 MPa), but it was not significantly different ($p < 0.999$) from Lucitone 199 (28.87 ± 8.09 MPa) or Denture Base LP Resin. See figure 5.

Discussion

In the present study, the mechanical properties of 3D-printed denture-base materials and one conventionally processed denture-base material were investigated. The test results showed no significant difference in the flexural strength between the 3D-printed materials and the conventionally processed material. However, a significant difference in flexural modulus, nanoindentation hardness, nanoindentation modulus and diametral tensile was revealed among the groups. Therefore, flexural strength results were consistent with the research null hypothesis, but the null hypothesis was rejected with the outcomes of flexural modulus, nanoindentation hardness, nanoindentation modulus and diametral tensile strength testing.

The flexural strength of a material is defined as the maximum bending stress that can be applied to that material before it yields. Flexural strength of denture base is considered the major mode of clinical failure. For that reason, having a sufficient flexural strength is critical for the success of a denture by resisting fracture or deformation of denture. (5, 13, 17, 18) Similar to the previous studies by Prpic et al. and Perea-Lowerey et al., all four 3D-printed material and conventionally processed material in this study met the ISO requirement for flexural strength (65 MPa). (13, 19, 20) However, Prpic et al. and Perea-

Lowery et al. found that the 3D-printed materials had the significantly lower flexural strength values compared to conventionally processed materials. According to the authors, the lower mechanical properties of the 3D-printed materials were due to lower conversion of monomer into polymer and the weak bond between successive layers in 3D printing resins. (13, 20) However, the present study found all three 3D-printed denture-base materials had a similar flexural strength to the conventionally processed material, Lucitone 199. The differences in the flexural strength values for the 3D-printed and conventionally processed PMMA denture base materials in the various studies may have been due to the testing of different brands of materials and 3D printers. The current study's findings were in agreement with Fiore et al. and Sonam et al. The authors demonstrated no statistically significant differences in flexural strength values between 3D-printed and conventionally processed materials. (21, 22) In those two studies, CAD/CAM milled denture base PMMA materials were included in addition to 3D-printed and conventionally processed PMMA denture base materials. The CAD/CAM milled denture base PMMA materials showed significantly higher flexural strength than the 3D-printed material and conventionally processed material. Sonam et al. explained the porosity caused by the inability to apply constant pressure in the conventional heat curing process resulted in low flexural strength. The low flexural strength values of the 3D-printed group was explained by the initiation of crack propagation due to the incremental layers in additive manufacturing technology. (22)

The flexural and nanoindentation modulus demonstrates the rigidity of a material and the extent of deformation. (5, 23) The rigidity of a denture base is necessary to evenly distribute forces to the underlying structures. Higher flexural and nanoindentation modulus (i.e., lower flexibility) allows the fabrication of thinner bases with improved fracture resistance, comfort, and retention. As a result, the patients might improve their natural speech and comfort from less volume of the denture. (21, 23) All four tested materials gave results that exceeded the minimum flexural modulus standard of 2 GPa set by ISO-20795-1.(19) The three 3D-printed materials exhibited significantly higher flexural modulus compared to the conventionally processed group. In addition, the two 3D-printed groups (Denture base LP resin and Dentca Denture Base II) had significantly greater nanoindentation modulus than the conventionally processed material. These findings were contradictory to the outcomes of the previous study by Perea-Lowerey et al. They found that the elastic modulus of a 3D-printed material was inferior to the conventionally processed material. The authors provided the same explanation for the lower flexural strength of the 3D-printed resins as noted previously (i.e., lower degree of double-bond conversion and weak interlayer bonding between successive printed layers). (20)

The hardness of a material is defined as its ability to withstand localized permanent deformation, typically by indentation. (24) Adequate surface hardness is necessary to avoid excessive wear of the denture base materials by abrasive food and denture cleansers. (25) The denture with low nanoindentation hardness may be damaged more easily with abrasion and indentation forces, resulting in the promotion of plaque retention and discoloration, and a decreased life span. (24) The findings from current study were not, however, consistent with the result of two previous studies by Prpic et al. and Gad et al. They determined that the 3D-printed materials had lower hardness values than conventionally processed denture materials. The authors attributed the results to the composition, layering, and water sorption with thermal stressing of the 3D-printed materials. (13, 26) However, the current study showed the Denture Base LP Resin group had the highest surface hardness among the study groups. Moreover, there was no significant difference between NextDent Denture 3D+, Dentca Denture Base II and Lucitone 199 (control). This outcome can be interpreted that the 3D-printed material had a surface hardness similar to or higher than that of the conventionally processed material.

To the author's knowledge, the diametral tensile strength of 3D-printed denture-base materials has not been reported in the literature. The current investigation found that 3D-printed materials showed superior or comparable diametral tensile strength with the conventional denture base material in an *in vitro* environment.

Future studies should evaluate other brands of 3D-printed and heat-processed materials and also include CAD/CAM denture-base materials. Additionally, the effect of different 3D printer types on mechanical properties could be further investigated. Another limitation of this study was its *in vitro* nature. The study design did not simulate the intraoral condition of thermal and mechanical stresses. For these reasons, the findings of this study must be interpreted cautiously.

Conclusions

This study demonstrated that 3D printing denture-base materials fulfilled the ISO requirement for denture base materials for flexural strength (65 MPa) and flexural modulus (2 GPa). (19) Moreover, all three 3D-printed groups in this study demonstrated significantly higher or similar mechanical properties than the conventionally processed material. According to the results of the current study, the 3D printing denture-base materials could be recommended as a viable alternative to conventionally processed denture-base materials.

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Table 1 Materials, manufacturers, and denture base fabrication technique

Material	Manufacturer	Composition	Denture Base Fabrication Technique
Denture Base LP Resin 1L	Formlabs, Somerville, MA	• Bisphenol A dimethacrylate • Bisphenol A dimethacrylate • Methacrylate monomer • Photoinitiator	3D Printing (Formlabs Form 3B) Stereolithography
NextDent Denture 3D+	NextDent, Utrecht, Netherlands	• Ethoxylated bisphenol A dimethacrylate • 7,7,9(or 7,9,9)-trimethyl-4,13-dioxo-3,14-dioxa-5,12-diazahexadecane-1,16-diyl bismethacrylate • 2-hydroxyethyl methacrylate • Silicon dioxide substance • Diphenyl(2,4,6 trimethylbenzoyl)phosphine oxide • Titanium dioxide	3D Printing (NextDent 5100) Digital Light Processing
Dentca Denture Base II	Whipmix, Louisville, KY	• Methacrylate monomer • Diurethan dimethacrylate, • Trimethylolpropane trimethacrylate • Initiator • Stablizer	3D Printing (Asiga Pro) Digital Light Processing
Lucitone 199	Dentsply Sirona, Charlotte, NC	• Methyl methacrylate • Ethylene dimethacrylate	Heat compression

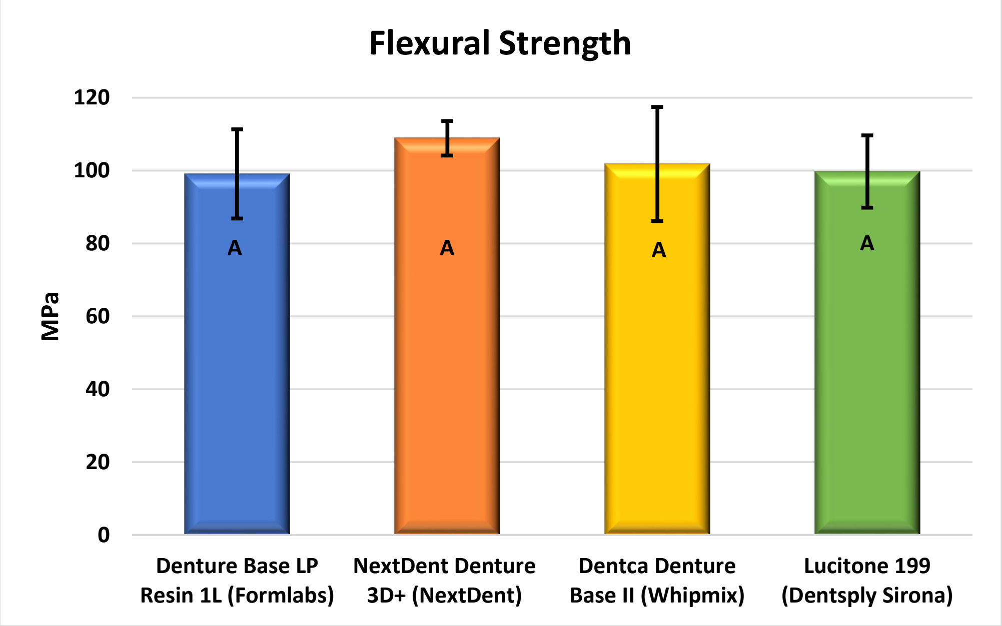

Figure 1. Means and standard deviations of flexural strength.
Groups with the same letter are not significantly different (p>0.05).

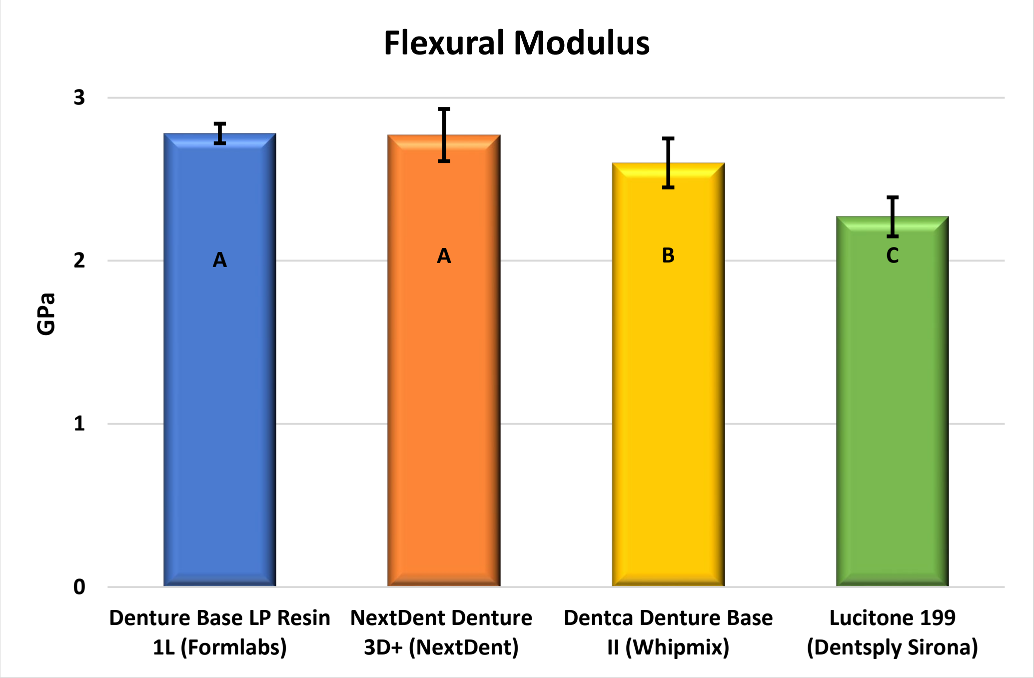

Figure 2. Means and standard deviations of flexural modulus.
Groups with the same letter are not significantly different (p>0.05).

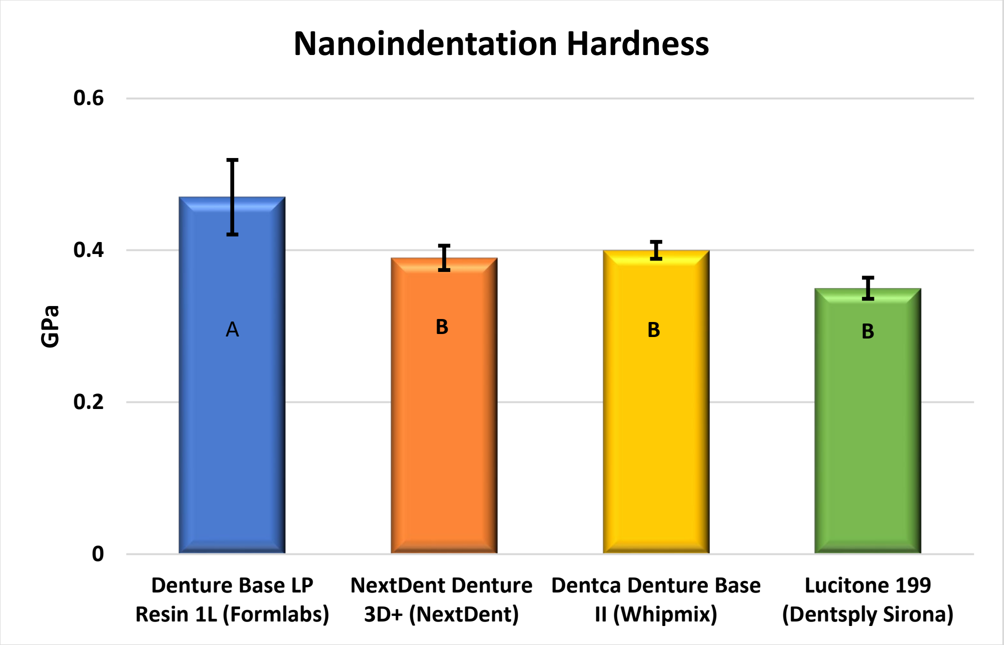

Figure 3. Means and standard deviations of nanoindentation hardness.
Groups with the same letter are not significantly different (p>0.05).

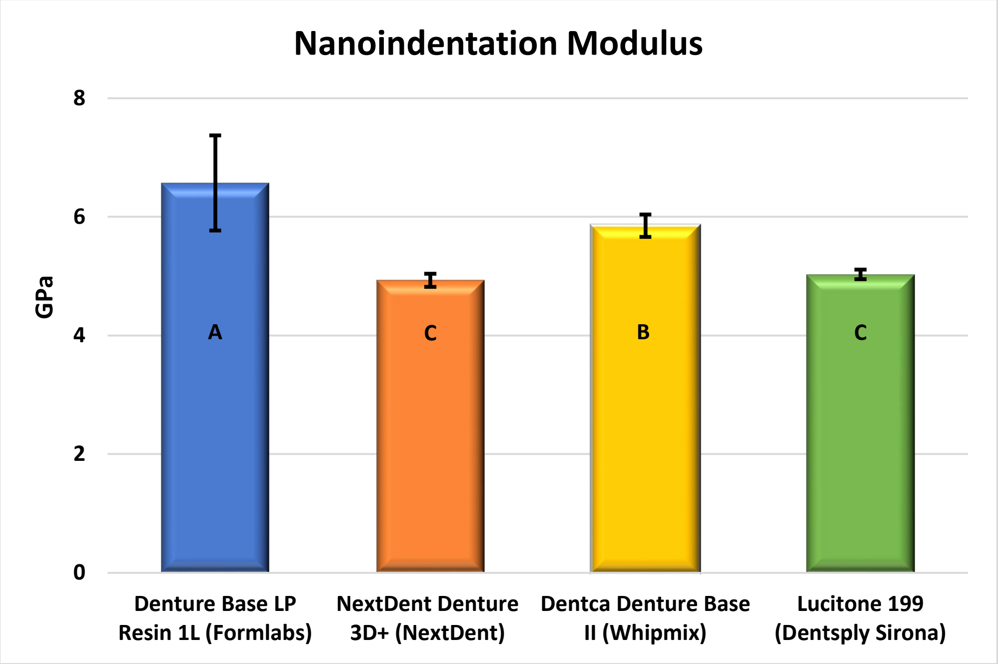

Figure 4. Means and standard deviations of nanoindentation modulus.
Groups with the same letter are not significantly different (p>0.05).

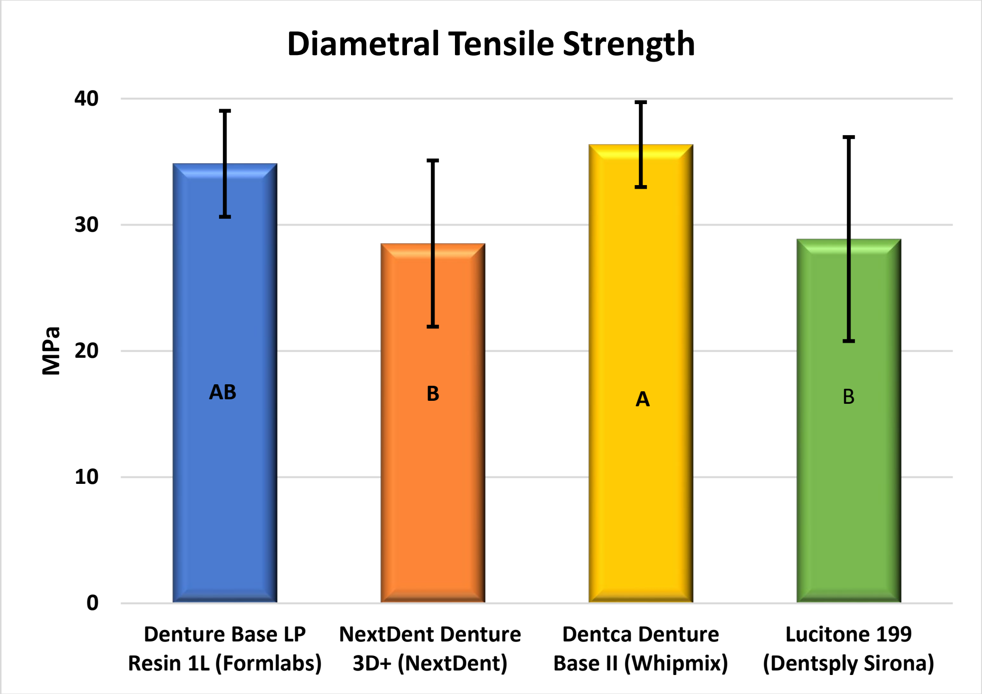

Figure 5. Means and standard deviations of diametral tensile strength.
Groups with the same letter are not significantly different ($p>0.05$)