

THE EFFECT OF ADDING NANOPARTICLES ON THE MECHANICAL PROPERTIES OF ACRYLIC REMOVABLE DENTURE

NABEL K. ABD-ALI

Mechanical Engineering Department, College of Engineering,
University of Al-Qadisiyah, Iraq
E-mail: nabel.abdali@qu.edu.iq

Abstract

Polymeric materials have begun to play an important role in biological applications and applications related to human life. Present work deals with two different types of fillers used in reinforced polymethylmethacrylic PMMA. Zirconium oxide nanoparticles and hydroxyapatite nanomaterial as a reinforcement agent with weight loading percent 1%, 2%, 3%, 4% and 5% were used to study the effect of these particles on mechanical properties of removable denture. Some laboratory tests were performed according to standard test methods and all results were discussed. Polymethylmethacrylic showed improvement as a result to add Zirconium oxide nanoparticles and hydroxyapatite nanomaterial, especially at weight loading percent 4 wt % in most mechanical laboratory test comparing with standard polymethylmethacrylic without additives. The tensile strength increased with the increasing of the proportion of Zirconium oxide nanoparticles and hydroxyapatite nanomaterial from 18.11 MPa for pure sample to 23.67 MPa and 20.53 MPa at 4 wt % respectively. The compression strength increased with the increasing of the proportion of Zirconium oxide nanoparticles and hydroxyapatite nanomaterial from 5.62 MPa for pure sample to 8.46 MPa and 9.4 MPa for 4wt% respectively. Also, the hardness increased with the increasing of the proportion of Zirconium oxide nanoparticles till 88.7 HBR at 5wt %, while reach to 78.3 HBR with hydroxyapatite nanomaterial comparing with standard sample with 74 HBR at 0 wt %. The impact strength showed same behavior and all these results may be related to the nature of these particles and its behavior during agglomerates and aggregates and its ability to make a good crosslinking with polymer chains. The numerical solution by ANSYS V.16 show the region of strain concentration and response of these structures.

Keywords: Hydroxyapatite, Mechanical properties, Polymethylmethacrylic, Removable denture, Zirconium oxide.

1. Introduction

Humans are keen to choose suitable components in order to accomplish their functions as well as aesthetically pleasing their shape, so they resort to removable dentures and choose the most suitable ones to accomplish these tasks. Removable dentures are a suitable option not only for the elderly, but for all people who have lost their teeth as a result of diseases or accidents. These clips have become very popular, especially since their specifications and suitability are constantly improving as a result of the use of materials and additives in order to improve these specifications [1].

Polymers play an important role in various fields, especially in the medical field. For example, they are used in denture base due to the fact that they have certain characteristics that make them suitable for this application and are compatible with its requirements, for example, biological qualities such as toxicity and bio-compatibility, thermal properties, good thermal conductivity and aesthetic aspects, good transparency.

In addition to chemical properties such as water solubility and other qualities such as proper price, easy cleaning and repair, all these factors making it more suitable than ceramics and metals [2, 3].

Several studies have sought to use multiple materials as a filler with polymers to improve the mechanical properties and improve the mechanical behaviour of these materials [4-7]. The Redmud was used as a reinforcement agent with polymers where it was noted that adding them in certain proportions helps improve the properties of flexural and impact resistance [8].

Polymethyl methacrylate (PMMA) as a linear polymer use in fabricate partial and complete denture, in addition to use in prosthetic popularly with precious metal as reinforcement particle. It is difficult to dissolve them in solvent and cannot formed by heat [9]. Denture application is a small part of usage polymers in the industry but it's so important because it's in direct contact with the fluid environment in the human body, which makes their properties such as chemical, physical, mechanical, and biological characteristics are very important [10].

Dentures or false teeth are prosthetic devices constructed to replace missing teeth and are supported by the surrounding soft and hard tissues of the oral cavity. Several type of materials were used to manufacturing these parts since 18th century such as Ivory, wood, gold and porcelain. In 20th century PMMA was discovered, and it still used in the present day with improvements in mechanical behaviour such as wearing, fracture and tooth debonding [11-14].

The removable denture also has some limitations, but with the introduction of flexible parts, many of the limitations of acrylic dentures are treated by several ways such as fillers addition or some engineering treatments for these applications [15].

The present work deals with polymethylmethacrylic PMMA with specific additions as filler and reinforcement materials to improve mechanical properties and develop the mechanical behaviour of these components. Zirconium oxide Nano-particles (ZrO₂ NPs.) and Hydroxyapatite Nano-particles (NHAP) with different weights loading percent from 1 *wt* % to 5 *wt* % and focused on the effects of these materials on the mechanical properties such as tensile strength, compression strength, hardness and impact strength.

2. Materials and Laboratory Tests

The polymethylmethacrylic PMMA ($\text{CH}_2=\text{C}(\text{CH}_3)\text{COOCH}_3$) [16] as powder, contain PMMA and other co-polymers (5%), Benzoyl peroxide (Initiator), Compounds of mercuric sulphide, cadmium sulphide Zinc or titanium oxide (pigments), Dibutyl phthalate (plasticizer), Dyed organic filler and inorganic particles like glass fibers or bead (Reinforcing agents), while methylmethacrylate monomer as liquid monomer, contain Methyl methacrylate (monomer), Dibutyl phthalate (Plasticizer) Glycol dimethacrylate (1-2%), (cross linking agent) and Hydroquinone (0.006%) (Inhibitor).

Zirconia as a reinforcement material is a crystalline dioxide of zirconium (ZrO_2) with particle size 30nm by VITA Zahnfabrik Co., Germany. This material also defines as ceramic particles [17]. The application of zirconia in various area of dentistry has been discussed such as effect of temperature on degradation and behaviour of this material [18, 19]. Nano Hydroxyapatite (NHAP) with chemical formula $\text{Ca}_5(\text{OH})(\text{PO}_4)_3$ and particle size 20nm, purity 96%, molar mass 502.31, melting point 1100 oC, manufactured by Hualanchem. Co. China. The main mineral component of bone and teeth is native to the human body [20, 21]. Also, all these materials have been previously used in research work, due to their health compatibility with the human body [22-24].

Tensile, hardness, Compression and Charpy impact resistance test specimens were prepared and carried out according to ASTM D638 [25], ASTM D 785-15 [26], ASTM D695 [27] and ASTM D6110 [28] respectively. The standard recipes in addition to other recipes shown in Table 1.

Table 1. Loading percent for PMMA and reinforcements materials.

Materials	Recipes wt. (%)					
PMMA	100	99	98	97	96	95
First Stage wt. (%)						
Group	I	II	III	IV	V	VI
ZrO ₂	0	1	2	3	4	5
Second Stage wt. (%)						
Group	VII	VIII	IX	X	XI	XII
NHAP	0	1	2	3	4	5

All samples prepared according to the standard specifications were manufactured and treated in the same conditions and steps for preparing and manufacturing dentures in specialized laboratories and three samples were selected to be considered for conducting all mechanical tests, as well as their average readings were taken to determine an accurate reading of each characteristic and according to standard specifications.

3. Preparation Procedure of Denture

The steps of manufacturing laboratory samples in specialized laboratories can be defined through six successive processes, were setting process, flasking process, wax elimination process, packing or injection process, finishing process and polishing process while the polymerization process in acrylic accrue through five steps, sand stage, stick stage, dough (working) stage, rubber stage and hard stage to manufacture complete or partial removable denture, Fig. 1.



Fig. 1. Acrylic denture.

a) Complete upper, b) Complete lower, C) Partial denture.

The two parts flask (7 cm \times 9 cm) with approximate weight 250 g, filled with plaster then put the wax pattern and wait to dry, then coated with a layer of insulating material to prevent adhesion between the two parts of the mould. The flask was shaken to remove bubbles from the plaster and the inner surface of each flask half was coated also with Vaseline to prevent attaching with the plaster gypsum. The flask left to dry at room temperature and then opened to remove the wax and obtain the desired cavity shape. the recipes of materials put in these cavities in the dough stage.

Mixing powder and liquid (3:1, liquid: powder) by weight loading (wt%) in mixing process where all reinforcement materials mixed with powder before adding the liquid monomer. The specimens were fabricated by conventional heat-polymerized acrylic resin and affected by some factors such as room temperature, mixing time and mixing speed where it directly proportional till reach dough stage. Remove the wax from the mould and put the recipe (at dough stage) in mould to take the require shape and then mould will be pressed by hydraulic press and put in mechanical clamp in packing process.

The dough takes the shape of a mould, and the air bubbles are eliminated by pressing and immersed in water bath at room temperature and heated by two stage to 70°C for 30 min and lift to boiling temperature at 100°C for 30 min to complete the polymerization process and then left at room temperature and taken out of the mould. Open the two parts of flask and get the final design of denture in deflasking process.

Finishing process by using equipment intended for this purpose. The acrylic bur put in hand piece to remove the excesses and sharp ends of the design (denture) and then use sandpaper to give us a smooth surface. Finally, polishing the acrylic denture by using dental lathe to get smooth and shine surface.

4. Results and Discussion

4.1. Tensile test

Figure 2 shows that the tensile strength of PMMA with and without reinforced by ZrO₂, where the tensile strength increased with the increasing of the proportion of ZrO₂ NPs. The standard recipe of PMMA (at 0 wt%) gave 18.11 MPa and the increasing in tensile strength were 3.5%, 6.2%, 17.7%, 30.7% and 32.2% for weight loading percent 1 wt%, 2 wt%, 3 wt%, 4wt% and 5wt% respectively as shown in Fig. 3. The weight percentage loading 4wt% gave an acceptable result where the increasing in loading of ZrO₂ to 5wt% not gave much different results. The shape

and size of these materials play an effective role in the reinforcement process, where the strong bonding between those particles and the polymeric chains of materials increases the bonding and intertwining between them [29]. The tensile strength that reaches to 700 MPa [30] and the homogeneous addition of these materials is an essential factor in improving mechanical properties and has a clear effect on changing the performance and improving the mechanical behaviour of these components and all these results compatible with previous studies [31-33].

Also, in Fig. 2, the same mechanical behaviour can be observed as a result of reinforcement by the hydroxyapatite nanomaterial, where the improvement was not noticeable at the 1% loading ratio by 0.5% with tensile strength 18.2 MPa to reach the highest value at the 5% loading ratio to reach about 14 % with tensile strength 20.62 MPa. The strength of the ionic bond of the hydroxyapatite and the good tensile properties of this material that range from 40-100 MPa [30] may be behind the improvement in the tensile property of the polymeric material [21]. It's clear that the improvement due to the addition of ZrO_2 is greater than the improvement caused by the NHAP as shown in Fig. 3 and this may be related to the brittle nature of NHAP compared to ZrO_2 which is consistent with the results of previous studies [34], where ZrO_2 has fracture toughness 7-10 $MPa\sqrt{m}$ compared with 1 $MPa\sqrt{m}$ for NHAP with its natural as a mineral present in the bone structure [18, 30]. Nanomaterials use in present study worked as a reinforcement and filler agent and it contributed to strengthening the interconnection between the polymeric chains.

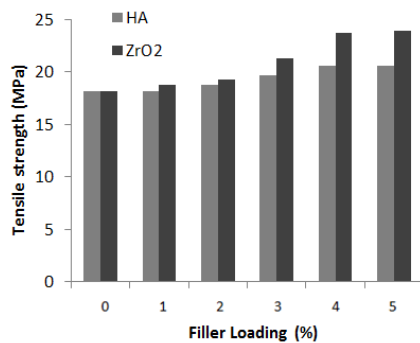


Fig. 2. Tensile test results.

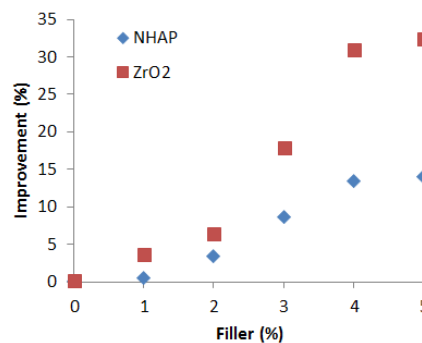


Fig. 3. Improvement in tensile strength vs. filler loading.

4.2. Compression test

The addition of the two types of reinforcement nanoparticles showed distinct and important results during the compression test, as the improvement in the compression property was due to the addition of both materials, but the improvement in that property was more prominent when reinforcing with NHAP as shown in Fig. 4. The standard recipe of PMMA (at 0wt%) gave 5.62 MPa and the compression strength increased with the increasing of the proportion of ZrO_2 NPs, where the increasing in compression strength were 5%, 18%, 39%, 50.5% and 55% for weight loading percent 1wt%, 2wt%, 3wt%, 4% and 5wt% respectively, Fig. 5. The latest load ratios of ZrO_2 provided improved close results at 4wt% and 5wt% with compression strength 8.46 MPa and 8.91 MPa respectively. The maximum compressive strength of ZrO_2 may reach to 2000 MPa [18], and this may be the reason for the improvement in the mechanical behavior of these recipes as a

result of the addition of this material and its use as reinforcement agent. The increase in the proportion of the reinforcing material in general may lead to a decrease in the cohesion between the matrix and the filler, and the aggregations resulting from the heterogeneity may lead to a decrease in the interconnection forces and may notice that the same improvement rates are not obtained as a result of increasing these materials as a result of saturation.

The improvement in compression strength of PMMA reinforced with NHAP were increase to 8.7%, 39.5%, 51.7%, 67.3% and 51.8% at weight loading percent 1wt%, 2wt%, 3wt%, 4% and 5wt% respectively, Fig. 5. The improvement in this mechanical property is evident with the increase in the reinforcing material loading ratio of NHAP, where the amount of improvement reached 67.3% at a load ratio of 4wt% with a compressive strength 9.40 MPa. Although the compressive strength of this material ranges from 100 to 900 MPa [30], which accounts for the improvement achieved, the increase in the loading rate of these materials may cause a decline in the amount of improvement due to its brittle nature and the aggregations of this material that behaving as voids caused reduction in resistance to the applied loads.

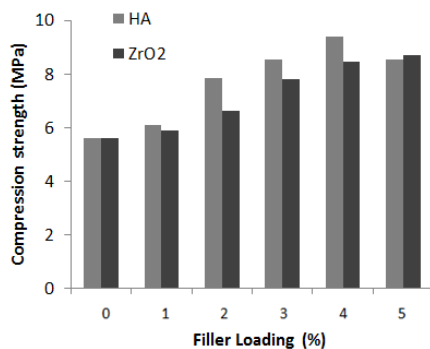


Fig. 4. Compression test results.

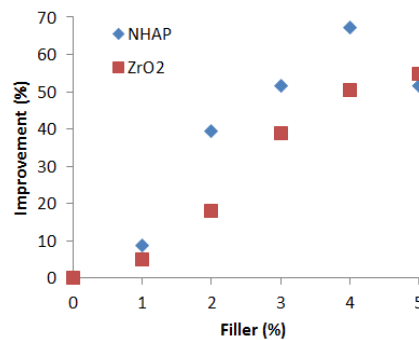


Fig. 5. Improvement in compression strength v. filler loading.

4.3. Hardness test

The hardness value increased with the increasing of the proportion of ZrO₂ NPs for each amount, Fig. 6. The standard recipe of PMMA (at 0wt%) gave 74 HBR and the hardness increased with the increasing of the proportion of ZrO₂ NPs till 88.7 HBR at 5 wt% and the increasing in hardness property were 3.4%, 6.9%, 12.7%, 16.9% and 19.8% for weight percentage loading 1 wt%, 2 wt%, 3wt%, 4wt% and 5wt% respectively as shown in Fig. 7. Mounting the values of this property is evident with the increase in the load rate of zirconia, which may be explained by the properties of this material and its nature with hardness reach to 1200 Vickers [18]. A loading rate of 4% by weight appears to be adequate to achieve the desired optimization without going overboard to make these components comfortable to use. Also, the addition of hydroxyapatite nanoparticles caused a slight improvement across all loading ratios, as the improvement rate reached the highest point (5.8%) at a loading ratio (5 wt%) compared to the standard recipe at (0 wt%) with 74 HBR, and this may be related to hardness value of this material about 800 Vickers [30] and the brittleness nature of this material may be behind porosity formation and non-homogeneity in recipes that prevent more increase in this property comparing with ZrO₂ [21]. The ratio (4 wt%) may be represents the saturation limit for the chains of the polymeric material that is used in the reinforcement process, while the

surplus acts as a filler, where it can be observed that the hardness increases with the increase in the proportions of these nanomaterials.

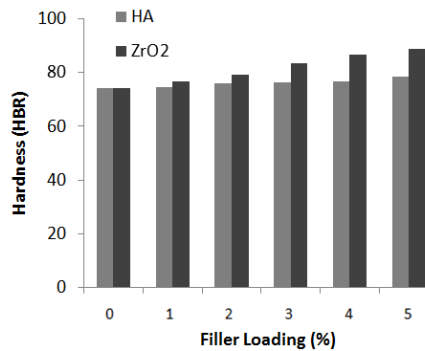


Fig. 6. Hardness test results.

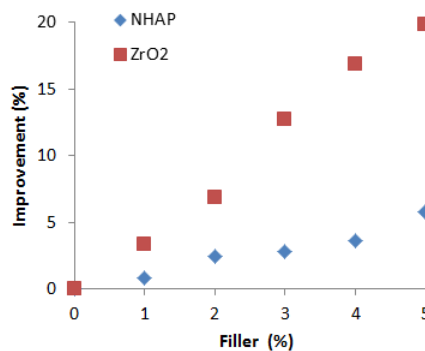


Fig.7. Improvement hardness vs. filler loading.

4.4. Impact test

Figure 8 shows the impact strength that increased with the increasing of the proportion of filler amount for each amounts, and the increasing in this property were 4.5%, 6.2%, 8.1%, 9.9% and 10.5% for weight loading percent 1 wt%, 2 wt%, 3wt%, 4wt% and 5wt% as a result to added ZrO2 respectively. The standard recipe of PMMA (at 0 wt%) gave 222.4 J/m² and increased with the increasing of the proportion of ZrO2 NPs till 245.7 J/m² at 5wt%. Zirconia has high and distinctive properties such as fracture toughness about 7-10 MPa m^{1/2} [18], shear modulus about 85 GPa. [35] and all these work to prevent crack propagation in earlier stage and improve strength and these compatible with previous study [36, 37].

The mechanical behaviour of PMMA matrix reinforced by NHAP show slight and acceptable improvement in impact strength property. Growing values of this property were 2.2%, 3.3%, 4.1%, 4.5% and 2.6% for weight loading percent 1 wt%, 2 wt%, 3 wt%, 4 wt% and 5 wt% respectively as shown in Fig. 9. The increasing in NHAP loading may be cause low dispersion, more aggregation, segregation and non-homogeneity and all that may lead to poor adhesion between reinforced particle and PMMA matrix that behaving as voids and prevent more improvement in this property, and these results were compatible with previous studies [34, 30].

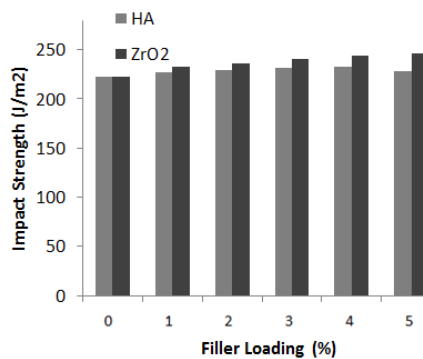


Fig. 8. Impact test result.

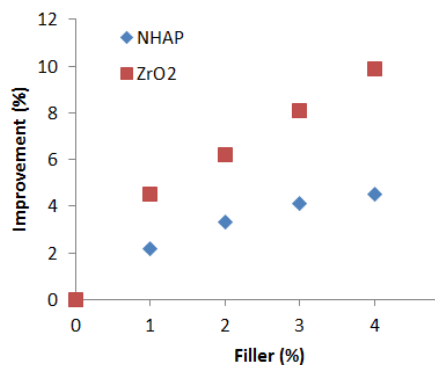


Fig. 9. Improvement impact strength vs. filler loading.

5. Numerical Solution:

Finite element analysis used to perform numerical solution because of its simplest of dealing with complex shapes and nonlinear response. ANSYS enhanced capability in various fields of coupling analysis such as structural/thermal and structural/fluid and hold different groups of elements treat variety fields of simulation. The stress-strain, shear and deformation analysis were expressed by the governing equation. The stress tensor written in a vector form as below:

$$\sigma^T = \{\sigma_{xx} \ \sigma_{yy} \ \sigma_{zz} \ \sigma_{xy} \ \sigma_{yz} \ \sigma_{xz}\} \quad (1)$$

where:

$$\sigma_{xy} = \sigma_{yx}; \quad \sigma_{xz} = \sigma_{zx}; \quad \sigma_{yz} = \sigma_{zy}$$

while the six components of strain can be written as below:

$$\varepsilon^T = \{\varepsilon_{xx} \ \varepsilon_{yy} \ \varepsilon_{zz} \ \varepsilon_{xy} \ \varepsilon_{yz} \ \varepsilon_{xz}\} \quad (2)$$

where the displacements derivatives for the components of strain as below:

$$\begin{aligned} \varepsilon_{xx} &= \frac{\partial u}{\partial x} & \varepsilon_{yy} &= \frac{\partial v}{\partial y} & \varepsilon_{zz} &= \frac{\partial w}{\partial z} \\ \varepsilon_{xy} &= \frac{\partial u}{\partial y} + \frac{\partial v}{\partial x} & \varepsilon_{xz} &= \frac{\partial u}{\partial z} + \frac{\partial w}{\partial x} & \varepsilon_{yz} &= \frac{\partial v}{\partial z} + \frac{\partial w}{\partial y} \end{aligned} \quad (3)$$

In matrix form:

$$\varepsilon = L \ U \quad (4)$$

where

$$U = \begin{Bmatrix} u \\ v \\ w \end{Bmatrix} \quad (5)$$

The partial differential operator's matrix obtained by:

$$L = \begin{bmatrix} \frac{\partial}{\partial x} & 0 & 0 \\ 0 & \frac{\partial}{\partial y} & 0 \\ 0 & 0 & \frac{\partial}{\partial z} \\ 0 & \frac{\partial}{\partial z} & \frac{\partial}{\partial y} \\ \frac{\partial}{\partial z} & 0 & \frac{\partial}{\partial y} \\ \frac{\partial}{\partial y} & \frac{\partial}{\partial x} & 0 \end{bmatrix} \quad (6)$$

for general anisotropic materials:

$$\sigma = c \ \varepsilon \quad (7)$$

and can be written explicitly as:

$$\begin{Bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{xy} \\ \sigma_{yz} \\ \sigma_{xz} \end{Bmatrix} = \begin{bmatrix} C_{11} & C_{12} & C_{13} & C_{14} & C_{15} & C_{16} \\ C_{21} & C_{22} & C_{23} & C_{24} & C_{25} & C_{26} \\ C_{31} & C_{32} & C_{33} & C_{34} & C_{35} & C_{36} \\ C_{41} & C_{42} & C_{43} & C_{44} & C_{45} & C_{46} \\ C_{51} & C_{52} & C_{53} & C_{54} & C_{55} & C_{56} \\ C_{61} & C_{62} & C_{63} & C_{64} & C_{65} & C_{66} \end{bmatrix} \begin{Bmatrix} \varepsilon_x \\ \varepsilon_y \\ \varepsilon_z \\ \varepsilon_{xy} \\ \varepsilon_{yz} \\ \varepsilon_{xz} \end{Bmatrix} \quad (8)$$

or

$$\begin{Bmatrix} \sigma_{xx} \\ \sigma_{yy} \\ \sigma_{zz} \\ \sigma_{xy} \\ \sigma_{yz} \\ \sigma_{xz} \end{Bmatrix} = \begin{bmatrix} Q_{11} & Q_{12} & 0 & 0 & 0 & 0 \\ Q_{21} & Q_{22} & 0 & 0 & 0 & 0 \\ 0 & 0 & Q_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & Q_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & Q_{55} & 0 \\ 0 & 0 & 0 & 0 & 0 & Q_{66} \end{bmatrix} \begin{Bmatrix} \varepsilon_x \\ \varepsilon_y \\ \varepsilon_z \\ \varepsilon_{xy} \\ \varepsilon_{yz} \\ \varepsilon_{xz} \end{Bmatrix} \quad (9)$$

Figure 10 shows mapped mesh from the numerical solution with element type Shell 99. Figures 11 to 19 show von Mises stress, shear stress distribution and strain distribution for PMMA, PMMA/ZrO₂ and PMMA/HA for complete lower denture respectively. All these responses under applied compression load with fixed the translation and rotation displacement at the lower edges, which reflect the mechanical behaviour of those materials and are not far from the experimental results.

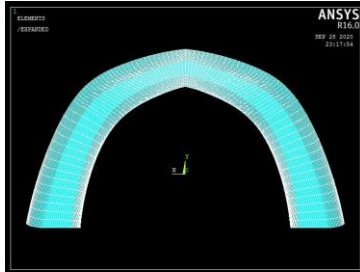


Fig. 10. Mapped mesh.

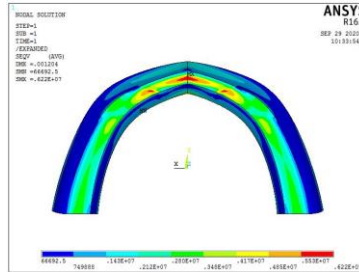


Fig. 11. PMMA, von Mises stress.

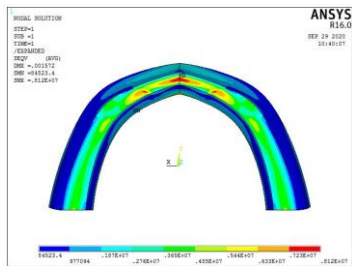


Fig. 12. PMMA/ZrO₂, von Mises stress.

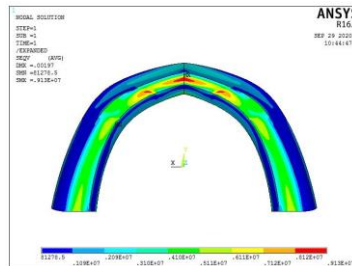


Fig. 13. PMMA/HA, von Mises stress.

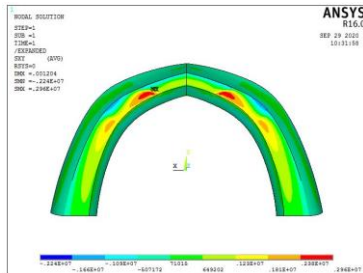


Fig. 14. PMMA, shear stress distribution.

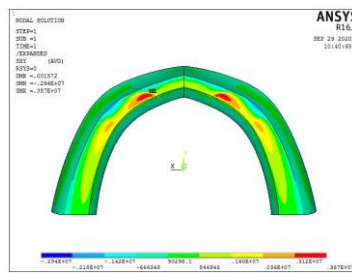


Fig. 15. PMMA/ZrO₂, shear stress distribution.

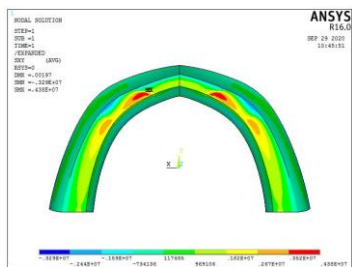


Fig. 16. PMMA/HA, shear stress distribution.

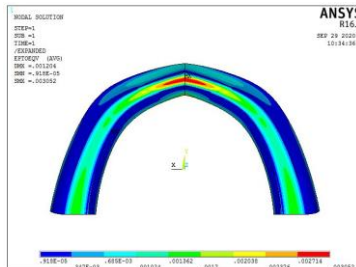


Fig. 17. PMMA, strain distribution.

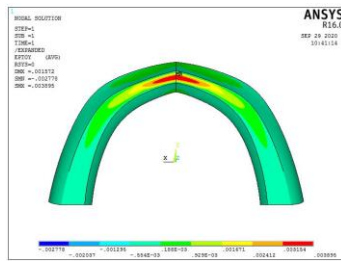


Fig. 18. PMMA/ ZrO₂, strain distribution.

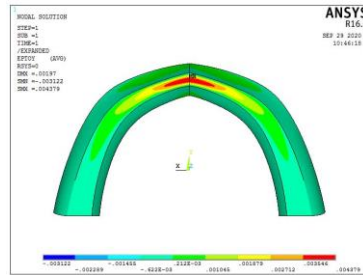


Fig. 19. PMMA/HA, strain distribution.

The stress concentration seems clearly in the front of the denture at same points where failure occurs often, where those regions bear great stresses, while there is a decrease in cross section area due to the nature of denture geometry and that leads to fracture occurs in these components.

6. Conclusions

The materials used during the current study aim to improving and developing mechanical properties and mechanical behaviour were used medicinal materials, biocompatible with the human body, and internationally licensed for this purpose. The applications of zirconia nanoparticles and hydroxyapatite nanoparticles as a reinforcement agent with PMMA in dentistry has been discussed.

- The improvement in mechanical properties such as tensile strength, compression strength, hardness, and impact strength as a result of added these materials with different loading ratios.
- The ZrO₂ NPs. with weight loading percent (4wt%) showed an optimum improvement in most mechanical properties and all these results related to the nature of these particles and its behaviour during agglomerates and aggregates and its ability to make a good crosslinking with polymer chains.
- The same weight loading percent 4wt% of hydroxyapatite nanoparticles NHAP showed an optimum improvement in most mechanical properties comparing with the standard recipe (0wt%) while the additional amount of this material caused deterioration in some properties such as compression and impact property and all these results related to the nature of these particles and its behaviour during heterogeneity, agglomerates, and aggregates where it behaving as voids and that accelerate the crack propagation stage.
- Adding these two types of nanomaterial's in removable dentures recipes may make them more acceptable for human uses because of improvements in properties, especially since these additions did not cause any problems or inconvenience for manufacturers during the forming process in addition to preserving the same surface smoothness.
- Author is looking forward to integrating these materials together in the recipes of the dentures, as well as using other nanomaterial's synthesis by biological or chemical methods, such as zinc oxide nanoparticles [38].
- The ratio (4 wt%) may be represents the saturation limit for the chains of the polymeric material that is used in the reinforcement process, while the surplus

acts as a filler, where it can be observed that the hardness increases with the increase in the proportions of these nanomaterials.

The researcher also looks forward to expanding the scope of the practical aspect of the research field through the use of various particles in the reinforcement process, including what was used in the enhancement of many rubber or plastic recipes for several applications [39-42] and these particles had been proven effective against many causes of diseases [43-45].

Nomenclatures

C	Constitutive matrix
c	Material constants matrix
Q	Material constant
U	The displacement vector
u, v, w	Displacements in the x, y, and z directions

Greek Symbols

σ_{ii}	Stress component, MPa.
ε_{ii}	Strain component
σ_{ij}	Shear stress
ε_{ij}	Shear strain

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