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Effect of Different Nano-Fillers Addition on The Mechanical Properties of Heat-Polymerized Acrylic Resin

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ABSTRACT

Aim: To evaluate flexural strength (FS) and micro-hardness of poly(methylmethacrylate) (PMMA) denture base material modified with Zirconium oxide (ZrO2), titanium oxide (TiO2) and zinc oxide (ZnO) nanoparticles powder with different concentration (1.5, 3 and 5%) **Subjects and Methods:** Test specimens of PMMA of dimensions (65 ×10 × 2.5 mm) were made from heat-cured acrylic resin served as control group Another nine group were made of PMMA containing 1.5, 3, and 5% (w/w) (ZrO2), (TiO2) and (ZnO) nanoparticles. Two types of mechanical tests were performed on the samples: flexural strength and hardness. **Results:** Statistical analysis results revealed that non-significant increase in flexural strength of heat cured reinforced with ZrO2 Nps and ZnO Nps for all concentrations. While adding 1.5% and 5% of TiO2 Nps showed significant increase in flexural strength but 3% showed significant decrease values. Results revealed that non-significant decrease in hardness of heat cured reinforced with ZrO2 Nps ,TiO2 Nps, and ZnO Nps for all concentrations except 3% of heat cured reinforced with TiO2 Nps which showed non-significant increase in hardness. **Conclusions:** It was discovered that adding of 1.5and 5% Tio2 Nps to the used denture base materials would improve the flexural strength of the denture base materials. Also, adding 1.5, 3 and 5 % ZrO2, Tio2, and ZnO Nps reduces the microhardness.

KEYWORDS

Flexural strengths, micro-hardness, Zirconium oxide, Titanium oxide and zinc oxide nanoparticles, heat-polymerized acrylic resin.

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INTRODUCTION

Poly methyl methacrylate (PMMA) is excessively utilized in dentistry as a denture base material. Its main advantages include ease in fabrication, good esthetics, and low cost. Because of its merits this material has many applications including biomedical and dental prostheses. The main disadvantages of PMMA are its low strength characteristics, which include flexural and impact strengths. [(1)]

Nanotechnology has lately been utilized in the prosthetics scope to material boost objectives[(2,3)], which has been confirmed in the research of nanomaterials for enhancing the mechanical characteristics of PMMA. Nanofillers such as nanoclays, nanotubes, nanofibers, and nanoparticles are the existing alternatives for amending the mechanical characteristics of PMMA. [4]

Flexural strength of PMMA resin is not adequate to withstand variety of intraoral stresses generated during function. [1]

The hardness of acrylic resins, which is defined as the ease of finishing a material and its resistance to wearing during cleaning, can be influenced by reinforcement materials. [4]

Zirconium oxide (ZrO2) is a bio-ceramic material that has been greatly applied for different dental implementation, such as crowns and bridges, implant fixture and abutments, and orthodontic brackets [5].

(ZrO2) is a metal oxide with many properties, including high mechanical strength, fracture durability, hardness, wear and chemical resistance, thermal stability, physical corrosion and biocompatibility. [6] which makes it suitable for use in dental materials like denture base reinforcement. ZrO2 is one of the most used nanoparticle fillers today. Due to transformation toughening, this bio ceramic filler has a high flexural strength and fracture toughness. It was proposed that ZrO2 be integrated into PMMA to enhance its properties. [7]

It's found that added of titanium oxide (TiO2) in various proportions which may be blended into colour - transformed acrylic resin powder to improve its tensile and impact strength, award that they have no opposite affect on other properties [2] .

It's detected that the incorporation of 3% TiO2 nanoparticles to heat cured resin enhanced the impact strength, transverse strength and surface hardness of acrylic resin and minimize water sorption and solubility. [8]

Recently a number of researches have reported the production of PMMA/ZnO nanocomposites by physical and chemical methods,. Transparent PMMA/ZnO nanocomposites with a high concentration of ZnO nanoparticles(ZnOnps) (up to 10%) have also been prepared by in situ bulk polymerization in the presence of the prepared nanoparticles, which gives improved dispersion of nanoparticles compared to simple mixing. [9]

Agrawal et al., reported resolution blending of ZnOnps into PMMA and displayed a best dispersion and perfect thermal stability of PMMA/ZnO nanocomposites thin films. Up to dates, no literature exists regarding the ability of ZnO nanoparticles to reinforce denture base material, although the reports claim that addition of ZnO nanoparticles to PMMA has resulted in improved physical properties.[10]

This study compared the flexural strength and hardness of conventional heat cured acrylic resin with 1.5%, 3%, and 5% added Zirconium oxide, Titanium oxide and Zinc oxide nanoparticles.

MATERIALS AND METHODS

Materials:

In overall 200 samples were made for flexural strength and hardness experiment, with measurement 65 longitude x 10 width x 2.5mm thickness, were split into 4 groups (A, B, C and D) and groups (B, C and D) further subdivided into three subgroups (1.5%, 3%, and 5%) of 10 specimens for each group.

- Specimens Grouping

Two tests were carried out and the materials used in this study were as following.

a. Heat-cured acrylic resin.

b. Heat-cured acrylic resin with 1.5%, 3% and 5% of ZrO2 nanoparticles

c. Heat-cured acrylic resin with 1.5%, 3% and 5% of TiO2 nanoparticles.

d. Heat-cured acrylic resin with 1.5%, 3% and 5% of ZnO nanoparticles.

As shown in Table 1.

Table (1) *Groups' classifications*

Test	Conventional	ZrO2			TiO2			ZnO			Total No
		1.5	3%	5%	1.5	3%	5%	1.5	3%	5%	
Flexural strength	10	10	10	10	10	10	10	10	10	10	100
Micro hardness	10	10	10	10	10	10	10	10	10	10	100
	20	20	20	20	20	20	20	20	20	20	200

Sample preparation:

- ***Preparation of PMMA Heat-Cured Acrylic Resin Specimens:***

A total of 200 specimens were made with the following technique; According to American Dental Association, custom metal molds with internal dimensions of 65mm lengths, 10 mm width and 2.5mm thicknesses were fabricated.

The conventional PMMA heat cured prepared by mixing the PMMA powder with MMA monomer liquid in accordance with the manufacturer's recommendations (polymer/monomer ratio was 2.5:1 by weight) in a glass jar until reaching the dough stage Then, the formed dough resin packed in the specially designed stainless-steel patterns (mold) after painting by a separating medium, that formed through the previous step, and designed according to each test type. Then, the two halves of flask were closed together and placed under hydraulic pressure which was slowly applied on the flask to get flow of the resin dough throughout the mold space.

Fabrication of Acrylic with Nps additives Specimens

Each type of nanoparticles was measured with electronic analytical balance device*, to be added to the PMMA powder according to ratios for each group.

For the conventional PMMA heat-cured acrylic resin; The PMMA resin powder and Nps powder was sonicated for 15 minutes in a sonicator ** to obtain a more homogenous and an equal distribution of Cs-Nps powder. Then, the modified PMMA/Nps powder mixed with MMA liquid in accordance with the manufacturer's recommendations (polymer/monomer ratio was 2.5:1 by weight) in a glass jar until reaching the dough stage. When the resin reached the dough stage, the mixture was removed from the glass jar and packed into stainless-steel pattern specially designed to the tested samples which previously coated with separating medium. Then, the stainless-steel pattern was removed, leaving the thermoplastic acrylic resin in the plaster investment. Then, the mix were polymerized according to manufacturer's instruction as mentioned before.

1- Flexural Strength Test:

The dimension of specimens for this test were 65mm length×10mm width×2.5mm thickness. Each specimen was individually and horizontally mounted in loading fixture (three-point bending test) assembly two parallel stainless-steel rods with span length 60 mm supporting the specimen, (with the damage site centrally located on the tensile side) on a computer-controlled material testing machine* with a load cell of 5 KN and data were recorded using computer software.

Then the specimen was statically compression loaded until fracture at a crosshead speed of 5 mm/minute. The Stress-strain curves were recorded with computer software and deflection was calculated automatically and displayed by the computer software of the testing machine.

* Highly sensitive lab instruments designed to accurately measure mass.
** Device is used for homogenizing, deagglomerating, emulsifying, powder materials.

The maximum load exerted on the specimens and flexural strength was recorded.

2- Micro hardness test:

The micro-hardness of the specimens was measured using digital Vickers micro-hardness tester (Wilson Hardness_Tukon1102) with a Vickers diamond indenter and a 20X objective lens. A load of 200g was applied to the surface of the specimens for 10 seconds. Three indentations were equally placed and not closer than 0.5 mm to the adjacent indentations, were made on the surface of each specimen .The diagonals lengths of the indentations were measured by built in scaled microscope and Vickers values were converted into micro-hardness values.

- All information was statistically analyzed by ANOVA test.

RESULTS

Flexural Strength

- Statistical analysis results found that non-significant decreased average flexural strength of heat cured reinforced with ZrO2 for all concentrations Table 2.
- Also results found that non-significant increase in average flexural strength of heat cured reinforced with TiO2 for 1.5 and 5% and non-significant decreased for 3%.
- While adding ZnO showed non-significant decrease in flexural strength for all concentrations. (Fig.1)

-Micro hardness:

- Statistical analysis results revealed that non-significant decrease in average hardness of heat cured reinforced with ZrO2 for all concentrations Table 3.
- Also results revealed that non-significant increase in average hardness of heat cured reinforced with TiO2 for 1.5 and 3% concentrations and non-significant decrease in 5% concentration.
- While adding ZnO showed decrease in micro-hardness for all values**.** (Fig.2)

Table (2) *The mean values, standard deviations (SD) and significant differences of flexure strength in MPa of different groups.*

Nano particle	Con.	Min.	Max.	mean	±	SD	p-value
	Control	80.08	116.4	94.23	±	16.12	
ZrO2	**ZrO2 1.5%**	74.674	110.95	87.46	±	16.34	0.5
	ZrO2 3%	63.961	96.44	76.40	±	14.06	
	ZrO2 5%	70.437	89.39	80.27	±	10.34	
TiO2	**TiO2 (1.5%)**	89.69	115.48	101.64	±	10.82	0.018
	TiO2 (3%)	61.46	84.63	68.97	±	10.89	
	TiO2 (5%)	80.002	126.94	99.68	±	19.71	
Zno	**ZnO 1.5%**	.000	96.84	56.41	±	40.60	0.18
	ZnO 3%	53.336	63.50	59.28	±	4.39	
	ZnO 5%	62.763	72.69	68.09	±	5.07	

Table (3) *The mean values, standard deviations (SD) and significant differences as regard hardness among different groups.*

Nano particle	Con.	Min.	Max.	Mean	±	SD	p-value
Control		46.552987	48.314392	47.326708	±	0.741803	
ZrO2	ZrO2 1.5%	46.639436	48.055044	47.164616	±	0.551570	0.5
	ZrO2 3%	45.580431	48.206331	47.058715	±	0.944517	
	ZrO2 5%	46.347669	47.633604	47.162455	±	0.549661	
TiO2	TiO2 (1.5%)	45.807361	47.579573	46.820980	±	0.867137	0.08
	TiO2 (3%)	47.136520	48.562934	47.743827	±	0.612358	
	TiO2 (5%)	45.158990	47.179744	46.589727	±	0.818014	
ZnO	ZnO 1.5%	45.504788	47.666022	46.764787	±	0.801902	0.4
	ZnO 3%	45.504788	47.147326	46.568115	±	0.639238	
	ZnO 5%	45.461563	47.233775	46.205028	±	0.683545	

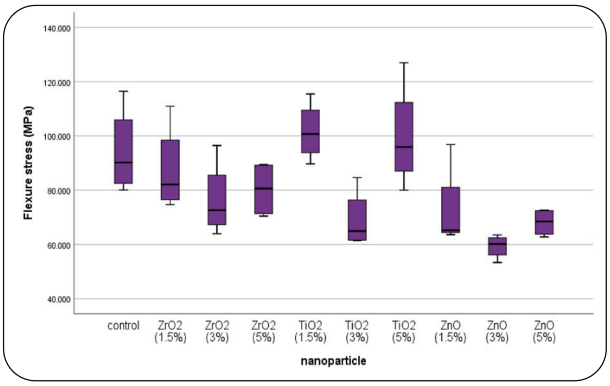

Fig. (1) Column chart showing flexural strength mean values for metal oxides modified acrylic groups compared to control group.

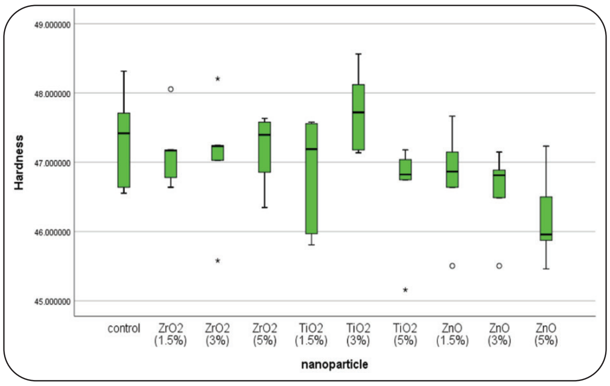

Fig. (2) Column chart showing hardness mean values for metal oxides modified acrylic groups compared to control group

DISCUSSION

(PMMA) has the key characteristic attribute for ideal denture-based material, and is considered the preferred material for bases of dentures.[11]

Acrylic resin has several disadvantages Such as low flexural strength compared to the metal frames, low thermal conductivity, produces residual monomers, porosity potential, permeable, and easily fractures [12].

Several researches have mentioned that the acrylic resin can show enhancement in the mechanical features such as fatigue behavior, flexural modulus and impact strength when it is reinforced by metal additives , carbon graphite fibers, ultra-high molecular weight polyethylene, glass fiber [13] .

Flexural failure of denture base resins is considered a primary mode of clinical failure. The flexural three-point bending test is useful in comparing materials of denture base as it simulates the type of stress that is Implemented to the denture during mastication.[14]

The effect of the integration of particles on the mechanical properties of dental PMMA, due to the characteristics of this material yield the best results for its biomedical efficacy. the integration of ZrO_2 particles does not promote changes in the mechanical characteristics of PMMA, and characteristics such as concentration, size and silanization of the ZrO_2 particles do not influence the results.[15]

A numbering of researches discovered that strengthening of traditional, heat-cured denture base resins with zirconia nanoparticles significantly enhancing the mechanical characteristics such as flexural and impact strength, as well as surface hardness [16-17].

ZrO_2 nanoparticles incorporation in the present study showed a decrease in the impact strength and hardness of the conventional acrylic.

Others suggested that adding of zirconia to heat cure acrylic resin to increase mechanical properties of the material; impact strength, flexural strength, compressive strength, fatigue strength, as well as its fracture toughness and hardness. [18]

In this study incorporation of 1.5, and 5% titanium dioxide nanoparticles improve the flexural strength of the used denture base materials ,while the incorporation of 1.5, 3 and 5% titanium dioxide nanoparticles in reinforced acrylic decreased flexural strength and hardness.

Other study concluded that the addition of titanium dioxide nanoparticles to heat cure acrylic resin improve its micro hardness. [19]

Titanium is used since it increases the surface hydrophobicity, reduces the adherence of biomolecules, aids in coloring, has antimicrobial properties and improves mechanical properties. The highest value was of acrylic with 8% Titanium oxide (109 MPa) with no significant difference with the conventional.

TiO_2NPs remains one of the preferred alternatives because of their ease of availability, low toxicity, chemical stability, robust physical properties, antibacterial activity, and cost-effectiveness. [20]

Chen et al., 2017 revealed that the use of nano-sized 3% TiO_2 found no significant differences in the mechanical properties compared to the control group within the range of an appropriate ratio . [21]

In the current study the flexural strength and the hardness were decreased by modification of heat cure acrylic by 1.5, 3 and 5 % of ZnO nanoparticles.

It was confirmed that the usage of zinc oxide nanoparticles slightly minimized the flexural strength of auto-polymerized acrylic resins used for ocular prostheses. Furthermore, zinc oxide enhanced the flexural strength of heat-polymerized Acrylic resin.[19]

The addition of 0.2-0.4 wt% ZnO to acrylic may increase the flexural strength, but when this ratio is more, flexural strength decreases as it acts as impurities, this result disagrees with study . In view of the fact that the mechanical and physical features arise from the morphology of the material, this work pursues the effect of different ZnO morphology and contents on the mechanical features of PMMA. The strengthening of denture base PMMA with ZnO nanotubes and nanoparticles was studied. [9]

CONCLUSION

Within the frame of this study, it could be It should be inferred that the inclusion of 1.5, and 5 % of TiO_2 Nps increase the flexural strength of the used denture base materials. it found that the inclusion of 1.5, 3 and 5 % ZrO_2 and ZnO Nps with decrease the flexural strength of the used denture base materials. Also, it should be inferred that the inclusion of 1.5, 3 and 5 % ZrO_2, TiO_2 and ZnO Nps with decrease the microhardness of the used denture base materials.

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تأثير إضافة الحشوات النانوية المختلفة على الخواص الميكانيكية لراتنج الأكريليك المبلمر بالحرارة

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الملخص :

الهدف: لتقييم قوة الإنثناء والصلابة الدقيقة لمادة قاعدة طقم الأسنان المعدلة بجزيئات النانو من أكسيد الزركونيوم وأكسيد التيتانيوم وأكسيد الزنك بتركيزات مختلفه (1.5, 3 و 5٪).

المواد والاساليب: تم تصنيع عينات اختبار البولي ميثيل ميثاكريلات بأبعاد (65×10×2.5 مم) من راتنج أكريليك المعالج بالحرارة والذى يعمل كمجموعة ضابطه. أما التسع مجموعات الأخرى تحتوى على (1.5, 3 و 5٪) من جزيئات النانو من أكسيد الزركونيوم. وأكسيد التيتانيوم وأكسيد الزنك وقد تم إجراء نوعين من الاختبارات الميكانيكية على العينات وهما قوة الانثناء والصلابة الدقيقة.

النتائج: أوضحت نتائج التحليل الإحصائي أن الزيادة غير الجوهرية في مقاومة الإنثناء لقاعدة طقم الأسنان المعدلة للحرارة والمعززة بجزيئات النانو من أكسيد الزركونيوم و جزيئات النانو من اكسيد الزنك لجميع التركيزات . بينما أظهرت إضافة 1.5٪ و 5٪ من جزيئات النانو من اكسيد التيتانيوم زيادة جوهرية في مقاومة الانثناء لكن 3٪ أظهرت قيم نقص جوهرية. كما أوضحت النتائج أن انخفاض غير جوهري في الصلابة لقاعدة طقم الأسنان المعدلة للحرارة المعززة بجزيئات النانو من أكسيد الزركونيوم . وأكسيد التيتانيوم و أكسيد الزنك لجميع التراكيز ماعدا 3٪ لقاعدة طقم الأسنان المعدلة للحرارة و المقواة بجزيئات النانو من أكسيد التيتانيوم والتي أظهرت زيادة غير جوهرية في الصلابة.

الخلاصة: تم اكتشاف أن إضافة 1.5 و 5٪ من جزيئات النانو من أكسيد التيتانيوم إلى المواد الأساسية المستخدمة في مواد قواعد أطقم الأسنان من شأنه تحسين قوة الإنثناء لهذه المواد. أيضًا تؤدي إضافة 1.5 و 3 و 5٪ من جزيئات النانو من أكسيد الزركونيوم. وأكسيد التيتانيوم وأكسيد الزنك إلى تقليل الصلابة الدقيقة.

الكلمات المفتاحية: قوى الإنثناء، الصلابة الدقيقة . جزيئات أكسيد الزركونيوم وأكسيد التيتانيوم وأكسيد الزنك النانوية، وراتنج الأكريليك المبلمر بالحرارة.