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Effect of Addition Zirconia/Chitosan Filler on Mechanical Properties of Heat Cure Polymethyl Methacrylate Resin

Shahad Lateef Mohammed[1], Firas Abdulameer Farhan[2]
Department of Prosthodontics, College of Dentistry, University of Baghdad, Baghdad, Iraq
[1]E-mail: shahadlateef024@gmail.com
https://orcid.org/0009-0000-6921-2261
[2]firas.farhan@codental.uobaghdad.edu.iq

ABSTRACT:

The preferred material for creating removable prosthesis is acrylic resin. for the reintegration of edentulous patients due to its good aesthetics, precise fit, low cost of equipment, and ease of clinical and laboratory manipulation. Acrylic resin exhibited relatively unsatisfactory characteristics like low impact and transverse strength. However, the acrylic resin was reinforced by adding filler materials like glass fibers or mixing two powders like zirconia and chitosan. The goal of this investigation was to explore how adding nanoscale zirconia with micrometer size of chitosan (zr-ch) filler with two percentages of 1% wt. and 5% wt. and two ratios of zr-ch (2:1 and 1:2) according to a pilot study and evaluate the surface hardness, impact and transverse strength .Ninety heat-cure acrylic samples were divided into three groups (each group 30 samples) based on the percentage weight of filler added to the acrylic mixture and the ratio between the nanoparticles of zirconia with a micrometer of chitosan particles. For control group (c) without addition, group A1 with 1% wt. and a ratio of 2:1 (zr-ch) and group A2 with 5% wt. and a ratio of 1:2. Each group was tested with impact strength, transverse strength and surface hardness (10 samples for each test). The zr-ch mixture ratio was prepared and selected according to the pilot study. A sharpy testing device was used to measure the impact strength while a hardness shore D device measured surface hardness and the final test was transverse strength which was tested by a universal Instron machine. The data were statistically analyzed by IBM SPSS statistics (version 24). The findings of the present study showed significant differences in surface hardness and transverse strength throughout the various study groups. However, the group A2 variation in impact strength was discovered to be nonsignificant, while the group A1 difference was shown to be significant. The best result for all three tests was obtained in the group A1. The adding zr-ch mixture improved surface hardness, impact and transverse strength, which improved acrylic denture base.

Keywords: acrylic resin; zirconia-chitosan filler; transverse strength; surface hardness and impact strength.

INTRODUCTION:

Acrylic denture base resin is frequently used to make removable prosthetics. This substance is imperfect, its use and appeal come from a blend of qualities rather than just a desired one Even though it satisfies cosmetic standards it falls far short of meeting prosthesis other requirements. Acrylic resin is weak as a base material for dentures because of its susceptibility to impact failure externally brought on by dropping of the dentures and failure due to fatigue inside the mouth brought on by Biting force of the occlusal surfaces (Alla, R. K.,2013). There have been numerous attempts to reinforce acrylic denture base resin, such as the insertion of metallic wire; however, the primary problem with using metallic wire is that it fails to bond well to the matrix of acrylic resin. (Vojdani M. and Khaledi A., 2006). The impact strength has been improved by adding a rubber phase to the bead polymer, however the cost has gone up (Jagger D.C.et al.,1999).

Nanotechnology has recently entered the prosthetics industry for medical and material advancement goals. Academics and researchers from all around the world are interested in metal oxides because of their large range of potential applications in industries including materials science, medicine, and industrial inspection (Hussein NA and Ali NA., 2022). By adding nanoparticles, resin materials can be combined with metallic substances, fibres, and oxides to create nanocomposites with better physiomechanical properties. (Al-Sammraaie, M. F., Fatalla, A. A., 2024). The study's use of nano-zirconia as a filler was motivated by its ability to enhance the mechanical characteristics of acrylic resins. Zirconia has several advantageous qualities, including superior mechanical strength and toughness, resistance to abrasion and biocompatibility (Ayad N.M.et al., 2008), Researchers found that nano ZrO2 boosted the antifungal activity of 3D-printed denture base polymer (Fareed Al-Sammraaie, M., A Fatalla, A.,2023). Another research an enhance in impact and transverse strength of repaired acrylic resin with incorporation of ZrO2 and Al2O3 (Al-Judy H. J..2016). Growing interest has been shown in modifying and using chitosan in the biomedical sectors because of its biocompatibility, biodegradability, nontoxic qualities, and antibacterial activity (Kong, M.et al., 2010). Chitosan also was used to enhance the properties of titanium for implant applications, PCL/Chitosan/cinnamon watery extract composite were deposited on commercially pure titanium specimens using electro spinning technique (Khadija Sahib et al., 2023). The present investigation looked at the impact of incorporating nanoscale zirconia-chitosan (zr-ch) filler with two percentages of 1% wt. and 5% wt. and two ratios of zr-ch (2:1 and 1:2) according to a pilot study and evaluate the impact strength, transverse strength, and surface hardness.

Materials and methods:

Preparation of zirconia and chitosan mixture:

Based on the findings of a pilot investigation to determine optimal concentration of nano-zirconia-chitosan powders (zr-ch) to use as a filler material for heat-cured acrylic resin to improve mechanical properties. The selected percentages were 1% wt. with a ratio of 2:1 (zr-ch) and 5% wt. with a ratio of 1:2 as group A2. A field emission scanning electron microscope (FESEM) was used to confirm the particle size and distribution of the zr-ch mixture. The zr-ch powders were mixed for 10 minutes using a ball milling machine.

Samples preparation and grouping:

Ninety acrylic samples were divided into three groups (30 samples for each) according to the percentage and ratio of the zr-ch mixture that was added to the acrylic sample. For control group (c) without adding a zr-ch mixture, group A1 with 1% wt. and a ratio of 2:1 (zr-ch) and group A2 with 5% wt. and a ratio of 1:2. Each group was tested with impact strength, transverse strength and surface hardness (10 samples for each test). Different sample patterns were designed by three-dimensional software and fabricated from plastic plates using a laser-cutting machine according to each test Figure (1).

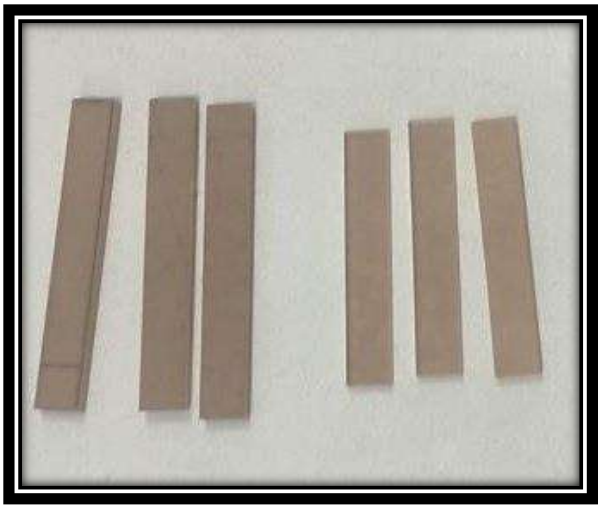

Figure (1): plastic sample patterns

Acrylic samples were produced by placing the plastic pattern in the lower half of the metal flask with dental stone as investment material and pouring the second layer after applying separating media to produce a sample mold to receive acrylic resin. The acrylic resin was mixed as directed by the manufacturer with the powder/monomer ratio 3:1 (22g to 10 ml). The zr-ch mixture was added to the acrylic mixture according to the mentioned groups, packed into the sample mould after reaching the dough stage and cured with a short cycle by water bath according to the manufacturer's instructions. The temperature was raised from ambient temperature to 70 °C in 30 minutes and left in water for another 30 minutes. Subsequently, the degree of temperature was gradually increased for 30 minutes to reach 100 °C and remained for 30 minutes.

Impact strength test:

The impact strength was conducted by the Charpy impact tester according to ISO 179, 2000. The sample dimensions were 80 mm x 10 mm x 4 mm.

Surface hardness and transverse strength tests:

The surface hardness was achieved using a shore D hardness durometer hardness tester (ANSI/ADA., 1999). The shore D hardness was recorded as the maximum value obtained by applying firm and rapid pressure to the indenter. Sample measurements for surface hardness and transverse strength testing were 2.5 mm × 10 mm × 65 mm.

A Universal Instron testing apparatus was used to perform the transverse strength evaluation, namely the micro-computer testing machine (JIANQIAO Testing Equipment) under the ANSI/ADA specification No. 12, 1999 as shown in Figure (2). The data was analyzed using descriptive statistics, multiple pairwise comparison tests, and the Tukey post hoc test. The software used in the study was SPSS version 24.

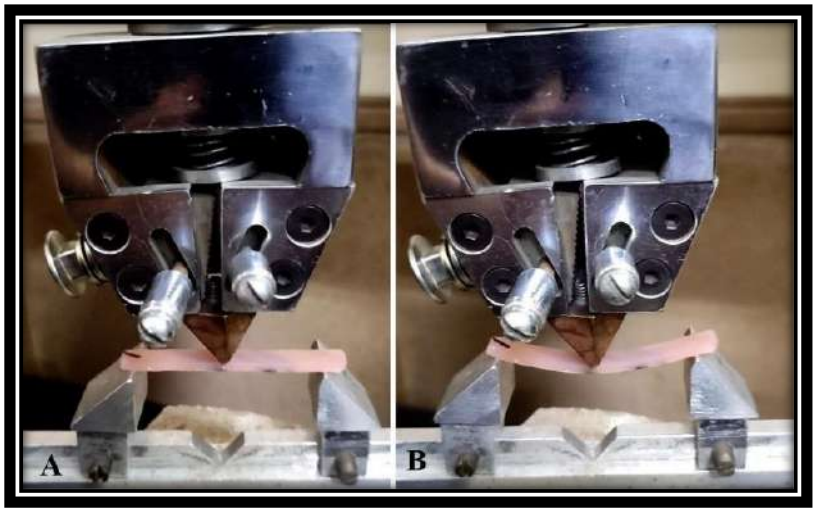

Figure (2): A- Sample before bending and B- Sample after bending before fracture

Results:

Field Emissions scanning electron microscope of filler particles and specimens:

FESEM images showed the particle size distribution. To confirm that zirconia is nanoparticles and chitosan is micro-particles as shown in Figure (3) and Figure (4).

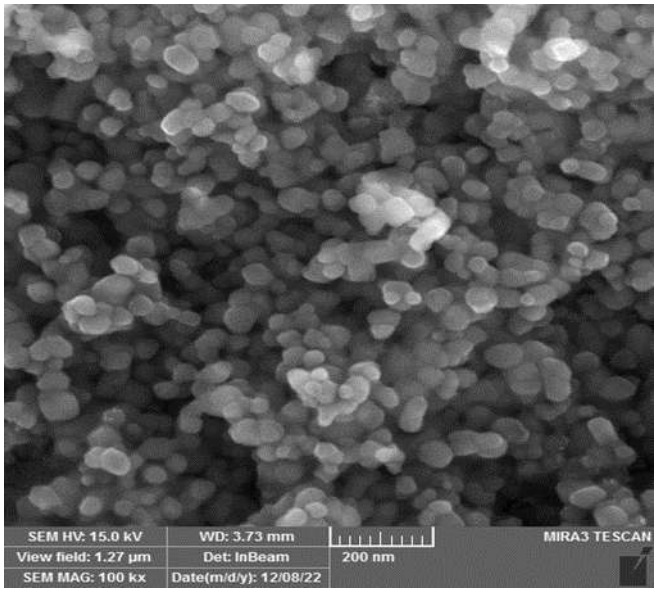

Figure (3): FESEM of nano zirconia powder

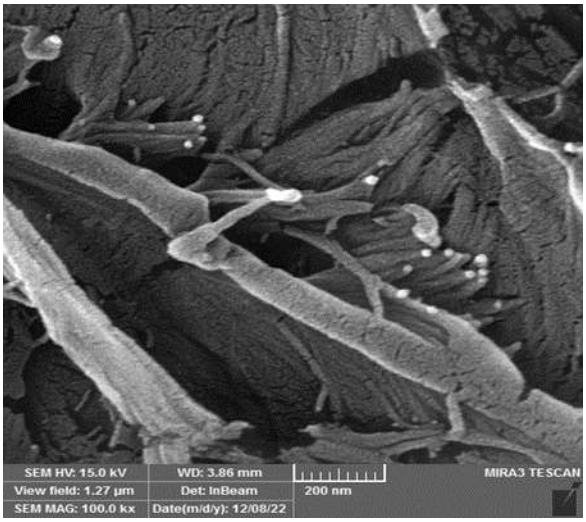

Figure (4): FESEM chitosan powder

FESEM analysis of tested samples displayed surface of group C Figure (5), groups A1 and A2 showed as shown in Figures (6), and (7). For A2 group there were filler agglomerations.

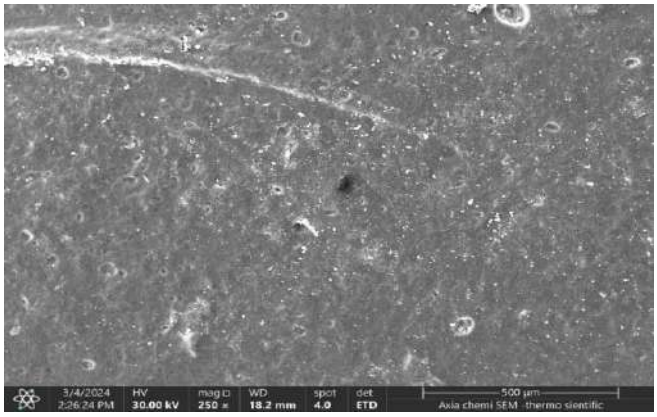

Figure (5): FESEM of the sample surface of group C

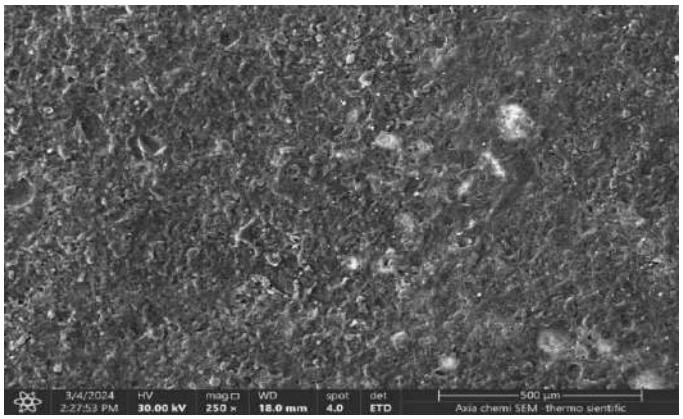

Figure (6): FESEM of the sample surface of group A1

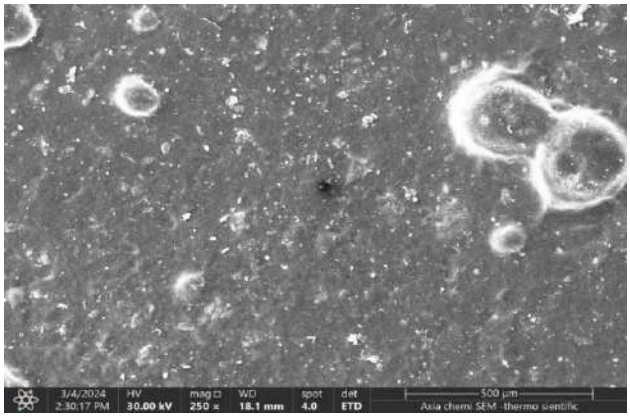

Figure (7): FESEM of the sample surface of group A2

Impact strength:

The impact strength mean value was increased in group A1 (12.788) more than in group A2 (11.155) and group C (10.574) as listed in Table (1). A non-significant variance with a P-value less than 0.05 was seen between group A2 and group C, while there was significant variation between the A1 and C groups. as shown in Table (2).

Table 1: An analysis of impact strength descriptive statistics

Groups	N	Mean	SD
C	10	10.574	0.676
A1	10	12.788	0.696
A2	10	11.155	0.492

Table 2: Tukey HSD multiple pairwise comparisons

Groups (I)	Groups (J)	Mean Difference (I-J)	P-value
C	A1	-2.214	0.000
	A2	-.581	0.115
A1	A2	1.633	0.000

Surface hardness:

Table (3) displays descriptive data for the surface hardness assessment, in which the highest mean value for the heat cure acrylic-test group (86.120) is shown in the A1 group and the lowest mean value (82.390) is shown in the C group, while for A2 group the mean value of surface hardness increased (85.090) in compare to C group but still lower than the mean value of A1 group. Table (4) shows a significant difference between the studied groups with a P-value less than 0.05.

Table 3: Descriptive Statistics of Surface hardness

Groups	N	Mean	SD
C	10	82.390	.922
A1	10	86.120	.869
A2	10	85.090	.631

Table 4: Pairwise comparisons between groups for surface hardness

Groups (I)	Groups (J)	Mean Difference (I-J)	P-value
Control	A1	-3.730	0.000
	A2	-2.700	0.000
A1	A2	1.030	0.023

Transverse strength:

Descriptive statistics show that transverse strength increased significantly in groups A1 (87.207) and A2 (85.177) compared to group C (81.175), with group A1 having the greatest value. The highest mean value was in group A1 and lowest in group C shown in Table (5). There were significant differences among groups with a P-value less than (0.05) as seen in Table (6) using multiple comparisons test.

Table 5: Transverse strength descriptive data

Groups	N	Mean	SD
C	10	81.175	0.797
A1	10	87.207	0.629
A2	10	85.177	0.815

Table 6: Pairwise Comparisons between groups for transverse strength

Groups (I)	Groups (J)	Mean Difference (I-J)	P-value
Control	A1	-6.031	0.000
	A2	-4.002	0.000
A1	A2	2.030	0.000

Discussion:

The objective of the present investigation was to analyse the acrylic denture base's mechanical characteristics after adding zr-ch filler in different percentages to the heat cure acrylic.

The use of zirconia and chitosan filler in the current study was motivated by the widespread practice of incorporating natural fillers into polymers to enhance their strength due to the environmentally friendly characteristics associated with such fillers. Researchers have made several experiments to improve the properties of denture base materials, some of these attempts were made by adding fibers as reinforcement to PMMA and forming a new composite with better properties (Abdulrazzaq Z.A.,and Khalaf B. S.,2023). Since other studies (Ayad N.M.et al., 2008, Alhava A. et al.,2017, Al-Harbi FA. et al., 2019) did not use surface treatment with silane and showed promising results it is not required for the nano zirconia in this work to be silaneted (Leão RDS et al., 2023).

In this research, the addition of zirconia-chitosan filler led to a rise in the mean value of impact strength, surface hardness and transverse strength in both experimental groups in contrast to the control group, but the most increase was obtained in A1 group. In the impact strength test, this increase was statistically significant For the A1 group, but for the A2 group, it was statistically non-significant.

Although both concertation enhanced impact strength, the best gain was observed in A1. The decrease at greater concentrations might be attributable to numerous variables, including the likelihood of filler agglomeration, the extent of the additive dispersion inside the PMMA, and the creation of an interfacial layer between the PMMA matrix and the additives (Abushowmi T.H., et al., 2020).

The chemical interaction between the basic PMMA and the nanoparticles may be lessened if the nanoparticles aggregate because of their vast surface area and elevated surface energy (Chladek G, et al., 2019). The rise in impact strength at smaller amounts may be due to the interface shear strength between chitosan and the polymer matrix, which encourages cross-linking or supramolecular bonding (Al-Harbi FA. et al., 2019, Choksi RH, et al., 2016). The decreasing compatibility of higher chitosan levels testing particles to resin matrix that led to failure owing to research particle slippage under increasing stress applications may be the cause of A2 fall in impact strength (Gad MM, et al., 2018, Protopapa P et al., 2011). Surface hardness values increased in this investigation, and this increase was statistically significant for both experimental groups. ZrO_2 has ionic bonding with oxygen ions together filling vacancies to produce appropriate material qualities which include high toughness and high hardness. Strong ionic interatomic bonding in nanoparticles produces the material's desired properties, such as hardness and strength. It implies that dispersing nanoparticles in a matrix would improve the matrix's strength and hardness (Ellakwa A.,2008). The chitosan effect on surface hardness could be explained by the impact of chitosan on the porosity of acrylic. A study found that the addition of chitosan with 1 and 2 % to acrylic denture base reduces the porosity (Ismiyati T, and Alhasyimi AA., 2022). Since the porosity is inversely proportional to surface hardness, therefore there was an increase in hardness after the addition of zirconia-chitosan filler. according to prior research, the incorporation of zirconia-polymerized acrylic into acrylic resulted in an increase in hardness (Rasan D and Farhan FA ,2023). Furthermore, a further investigation reported a significant increase in transverse strength due to the incorporation of ZrO_2-Al_2O_3 (MAH, B., and Aljafery, A. M., 2015). Surface hardness substantially rose, according to a different investigation after the incorporation of 3% silaneted nano zirconia (Al-Hiloh S A, and

Ismail I J A., 2016). The transverse strength significantly increased as compared to the control group. For both experimental groups, this rise was statistically significant, however zirconia-chitosan filler at the A1 group provided the most benefit. This improve in transverse strength may be the result of transformation toughening, this is the mechanism by which zirconia absorbs the energy of fracture development and transitions from a tetragonal to a monoclinic phase.

Additionally, as ZrO crystals expand during this process, compressive stress is applied to the fracture, stopping its development (J. A. Kenneth, C., 2013). More filler particles after attaining matrix saturation cause disruption in the continuity of the resin matrix, and higher filler percentages led to more defects that affect material strength and particle clustering inside the resin (Nejatian T et al., 2020), Furthermore, the distribution of chitosan in the resin matrix is the reason for the improved strength. Chitosan interacts with denture base resin through reactive groups (-COOH, CH_1, etc.). The physical or mechanical bond that might form because of difference in size between the chitosan and the resin may increase strength. Additionally, at greater concentrations, the availability of functional groups to react with the monomer may be limited due to the distribution of the particles in space and the formation of multilayer coupling agents around the particles. The research of Karci et al, Maitra et al, and Sodagar et al reported decreases in transverse strength owing to un-even distribution, and insufficient retention of composite particles inside the resin matrix during the examination of different chitosan concentrations (Karci M, et al., 2019, Maitra U et al., 2009, Sodagar A, et al., 2013). A related study found that silica from Iraqi rice husk was effectively added to the high-impact heat-cured acrylic resin at a weight of 1%, greatly improving the mechanical characteristics of the high-impact heat-cured acrylic (Khairi AW,and Naji GA., 2023).

Conclusion:

The adding of (zr-ch) significantly improved surface hardness, transverse and impact strength, which improved the heat cure acrylic denture base.

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