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Investigation Biological and Mechanical Characteristics of Hybrid PMMA Composite Materials as Prosthesis Complete Denture

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Abstract

PolyMethyl Methacrylate was a broadly known material in the medical and dental fields since its exceptional biocompatibility and easy manufacture, nevertheless, exhibits inferior mechanical properties. In the present study, several experiments were implemented to improve PMMA resin characteristics utilized for lower and upper prosthetic complete dentures by incorporating two various particles forms: nanoparticles of zirconia (n-ZrO2) and silver (n-Ag). These particles were applied as a fluid resin matrix to the polymethyl methacrylate (PMMA) cold-curing resin with various volume percentages of (0, 1.5, 3, and 4.5) weight percent. The composite prosthetic denture samples have been utilizing the (Hand Lay-Up) process in this project. This study was aimed to explore the impact of the selected volume fractions of (n-Ag & n-ZrO2) in nanoparticle size on tensile, impact, and biological characteristics of hybrid composite prosthetic complete dentures. The experimental aspect of this research involved conducting several biological and mechanical experiments, involving (impact, tensile, corrosion in saliva media and bacteria) tests. This research found that the values of most characteristics improved as the (n-Ag, n-ZrO2) particles' fractional volume in polymer composite materials increased, but antibacterial activity reduced.

Keywords: Ag-Nano Particles, Antibacterial activity, Hybrid Composite, PMMA, Saliva, and ZrO2- Nano Particles.

1. Introduction

In technology, bioengineering, chemistry, and, physics; polymer nanocomposites homogenized with metal nanoparticles have become critical [1]. Recently, organic monomer polymerization was already attempted for the first time. The silver ions then created the metals' polymer nanocomposites as a result of the photopolymerization procedure, which had significant advantages [2]. Monomer and polymerization, as well as metal ion decrease, might very well be done at ambient temp and at normal pressure, with no significant agent reductions [3].

Polymers have been extensively used in the fabrication of teeth dental materials in recent years; this relates to the mechanical properties. The biocompatibility of the polymer nanocomposite composites was satisfactory. High filler content materials have demonstrated to have a greater micro-hardness than currently available bracket materials and polymer nanocomposites with similar thermal conductivity as natural teeth [4]–[6].

The PMMA is commonly utilized to prepare the total and partial denture bases. Like the Streptococcus mutants and Candida albicans, certain pathogens' proliferation has been induced by acrylic resins surface roughness and systemic or local aspects [7]. In general, oral hygiene enhancement has been accomplished by utilizing antimicrobial mouth-washes and suitable tooth-brushing methods and utilizing denture cleansing tablets and prophylactic

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systemic antibiotic medications. Nonetheless, those approaches are limited in reducing those pathogens' effectiveness. The research-based on the broad-spectrum antimicrobial materials of acrylic resin has gained considerable interest in the past years [8]. The silver (Ag) salts were used for years to protect Gram-positive and -negative bacteria, fungi, protozoa, and viruses. The elemental Ag and the related compounds have been utilized to reduce infection risks in treating burns, preventing bacterial colonization on the medical apparatuses, surgical textile fabrics, bone cement, water purification, and dental materials [9].

In dental applications, various Ag forms, like the Ag nano-particles (n-Ag), ions of Ag (Ag+), and Ag-polymeric complexes, were utilized to enhance. Nonetheless, Ag+ instability results in the restriction of its practical implementations. This issue may be solved by protecting Ag+ with a sheath of the polymeric matrix. The main benefit of utilizing Ag-NPs has arisen from their large surface area to volume ratio. The Ag-NPs show a more effective ion release and improved antimicrobial activity. The Ag-NPs were preferred, besides some additional functional traits, like their electrical and thermal conductivity and ductility [10].

Even though the antimicrobial characteristics of the Ag-NPs in the acrylic resins were illustrated earlier, a few researchers reported the impact of the nanoparticles on the mechanical characteristics of the denture base resins. Even though adding the Ag-NPs has antimicrobial benefits for the acrylic resins, its impacts on resin mechanical characteristics have to be studied, which is why the objective of that in vitro study has been the evaluation of the effects of the Ag-NPs upon the elastic modulus, flexural strength, and the impact strength properties of 2 distinct dental [11]. It had [12] assessed the impacts of adding the modified particles of the nano-zirconium oxide (ZrO_2) on some of the heat-cured acrylic denture base materials' characteristics. The Nano-ZrO_2 has been coated by a tri methoxy easily people methacrylate (TMSPM) layer before being dispersed in the monomer (MMA) in a variety of the percentage values (i.e., 2, 3, 5, and 7)% by wt. It has been discovered that adding the modified nano-ZrO_2 has resulted in considerably increasing the radio-opacity of the heat cure PMMA and such increase has been proportionate with the nano-ZrO_2 concentration. The maximal increase in the transverse and impact strength was observed in the nanocomposite that contains (5 wt%) nano-ZrO_2; then, the strength was decreased when the additional increased.

Chow et al., [13] HA surface treatment utilizing the zirconate agent (ZCA) improved interfacial bonding between HA filler and PMMA matrix. The composites of the PMMA/HA mechanical characteristics have been studied utilizing flexural tests. It was discovered that the composites of (PMMA-5% HA-2% ZCA) had shown a greater degree of flexural strength than the flexural strength of the untreated composites of the PMMA/HA. Moreover, that composites resulted from enhancements of the interactions between hydroxyapatite (HA) and PMMA. Shyang [14] has studied the impacts of HA particles' supplement on the heat polymerizing PMMA denture base resin's flexural characteristics. Findings have shown that flexural strain, flexural modulus, and flexural strength of the PMMA/HA composites have been reduced with the increase of the reinforcing particles of the hydroxyapatite.

Ahmed and Ebrahim [15] have researched adding ZrO_2 nanopowder with various weight fractions on some of the heat polymerized acrylic resins' mechanical characteristics. Results have shown a substantial increase in flexural strength, hardness, and fracture toughness of the heat-polymerized acrylic resins, and the optimal mechanical characteristics have been obtained by adding 7wt% ZrO_2 [17].

Köroğlu et al., [16] study the thermal and mechanical characteristics of 2 denture-based acrylic resins with silver nanoparticles (Ag-NPs). Two acrylic denture base resins (i.e., microwave polymerized and heat-polymerized) contain 0.80, 0.30, and 1.60wt% Ag-NPs have been assessed for the elastic modulus, flexural strength, and impact strength. Results: Adding 0.80% and 1.60% Ag-NPs in the microwave-polymerized resins resulted in a considerable decrease in elastic modulus and transverse strength. Adding Ag-NPs does not affect the two resin groups concerning the impact strength. It has been concluded from this research that incorporating the Ag-NPs, utilized in general, impact strength terms, adding Ag-NPs does not affect the two resin groups.

Radhi et al. [17]–[19] studied the impact of incorporating the silver NPs on the thermal and mechanical characteristics of the denture base acrylic resins. Incorporating the Ag-NPs has been utilized in general for the antimicrobial potency, influenced transverse strength of denture base acrylic resin kinds, which rely upon the NPs concentration. The Tg has been decreased by adding Ag-NPs for every denture base resin. Ag-NPs incorporation impacts the

thermal and mechanical features of the denture base acrylic resin kinds.

Hilal et al., [20] explored the impacts of AgNPs on the mechanical and structural characteristics of the PMMA blend and its applications for a denture. First, the silver nanoparticles were prepared with modern and efficient methods for decreasing the nanoparticles' size, and then the PMMA was added with silver nanoparticles. The compressive strength illustrates that each mechanical characteristic improved the polymer characteristics. The nanocomposite materials were considered the most important; impact strength was investigated and used as a dental mold.

This study has aimed to explore the impact of the selected volume fractions of (n-Ag & n-ZrO_2) in nanoparticle size on tensile, impact, and biological characteristics of hybrid composite prosthetic complete dentures.

2. Materials And Methodology

2.1. Materials

The prosthetic composite full dentures specimens are made of epoxy resin and hardened powder materials in this analysis. Matrix material involved heating curing PMMA (manufacture shanghai new century dental materials Co.LTD). materials self-cure PMMA utilized as fluid resin matrix (Spofa Dental Company) to prepare samples as hybrid composites of the denture prosthetic. Two kinds of nanoparticles were identified with a ratio of (0, 1.5, 3, and 4.5) % wt. It has been introduced to acrylic powder, involving (silver and zirconia) powders.

In this analysis, two forms of particles have been utilized to improve products with a fractional volume of (0.75, 1.5, and 2.25) percent applied to the polymer (acrylic powder), involving the availability of zirconium oxide (ZrO2) as partly stable particles produced from acrylic powder (ZIRCONIA SALES-GUI 185 SS-U.K Company). On the other side, silver particles are most widely utilized and provided as nanoparticles in medical applications (Germany, Darmstadt, Merck Company). The product of the distribution of particle size (n-ZrO2) and (n-Ag) particles is performed with the aid of a particle size analyzer, which reveals that the average diameter was (30, 40) nm. The particle scale mechanical and physical characteristics of (ZrO2) and (Ag) particles are displayed in Table 1 below.

Table 1. This study utilized the physical and Mechanical Characteristics of Ag and ZrO2 Particles [21].

Particle kind	Tensile Strength (MPa)	Young's Modulus (GPA)	Poisson's Ratio	Compression Strength (MPa)	Flexural Strength (MPa)	Density (gm/cm^3)
PMMA	47-79	2.2-3.8	---	---	3-3.5	1.19
silver	140	76	0.37	---	---	10.5
Zirconium Oxide	800-1500	205-210	0.23-0.31	2000	900-1200	5.7-6.1

Techniques of Preparing Samples and Testing: The Vertex ™ Castavaria has been utilized to prepare the PMMA composite material samples. The standard proportions in the ratio of the mixing are often for the cold cure acrylic resin of (17g) polymer powder (PMMA) and (10ml) of the monomer liquid (MMA) (1.7g / 1ml) by the volume or (1.70 g / 0.95 g) by wt. based on the instructions of the manufacturer.

The significance of that ratio has been associated with the control of mix workability, dimensional variations on the setting, and a variable influence on the acrylic resin's cytotoxicity. In mixing the powder and liquid, numerous changes will occur due to the polymer solution in the monomer [22]. The Vertex Castavaria has been mouldable for a long time. In contrast, the mix has been mixed of the liquid (MMA) in a clean and dry container (i.e., glass beaker), followed by slowly adding dry powder (PMMA) into the liquid (MMA). This mix has been stirred continuously at the temperature of the room through the use of the mechanical mixing (i.e., the Brabender elastography mixer) at a (20rpm) speed to the point where it reaches the dough stage and poured with a thin straight line in the opening mold's center with a maximal time of approximately (4.50min) based on the instructions of the manufacture. Throughout the mix pouring into glass mold, this mold has to be rocked gently and vibrated from one side to eliminate any gas bubbles from samples. Moreover, the rest of the mix was poured into the mold hole to fill the glass mold. This mix has been covered in a closed container and left to sit on bench top for (8min-13min) at normal temp. (30oC) from the beginning of the mixing procedure to increase the viscosity, the pouring surface becomes matt and hard.

Following the polymerization's completion, followed by the curing process, the mold was left to stand outside the autoclave and put on a bench for approximately 30min cooling times at temp of the room to complete cooling and complete the sample

hardening. After the cooling, samples have been de-molded to be removed from the mold and cleaned.

3. Experimental Tests

The tensile test has been carried out based on the (ASTM D 638-03) through the use of the tensile machine (i.e., the universal test machine), Instron kind at a crosshead speed (the rate of strain) of (0.5 cm per min) and load has been applied (5kN) until the sample break occurs. Figure1 illustrates the tensile test's standard sample. The testing process has involved placing the test sample in the test machine and extending it slowly to the fracture point. The gauge section elongation has been recorded against the applied force throughout that procedure. The measurement of the elongation has been utilized for the calculation of the strain of the engineering. The tensile force is utilized to determine the engineering stress, which results from dividing stress on the normal sample's cross-section area and, finally, the (stress-strain) curve [23].

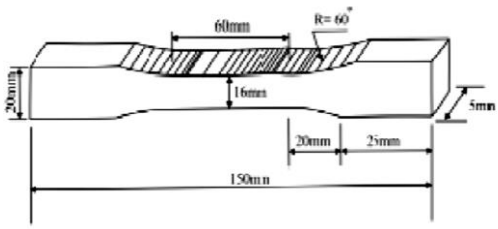

Fig 1. Diagram Sample for Standard Sample of Tensile Testing [24].

An impact testing has been utilized to observe the material behaviors once it exposes to shock loading, which leads to deformation, after that sample fracture, by determining the material's capability to absorb the energy throughout a collision. This energy can be utilized to determine the impact strength, toughness, fracture-resistant, and fracture-resistant of a material. Izod impact was utilized to characterize kinetic energy, which was required to initiate the fracture and continue this fracture to the point where the sample had been broken. To conduct the test, the specimen is sited into a holding in the apparatus with the geometry based on (ISO180) through the use of Izod Impact test machining kind is (XJU series pendulum Izod/Charpy impact machining test), Figure (2) shows the standard sample of impact test [25].

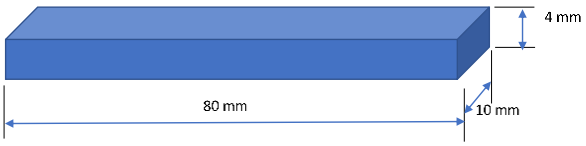

Fig 2. Diagram Sample for Standard Sample of Impact Testing, [26].

Also, corrosion test of samples studied in saliva solution. Table 2 demonstrates the saliva solution's chemical analysis.

Table 2. Chemical analysis of saliva Solution [27].

NO.	Component	(gM. /L) $x10^{-3}$
1	NaCl	8000
2	$CaCl_2$	140
3	KCl	400
4	$NaHCO_3$	350
5	Glucose	1000
6	$MgCl_2.6H_2O$	100
7	$Na_2HPO_4.2H_2O$	60
8	$KH_2 PO_4$	60
9	$Mg SO_4.7H_2O$	60

The experimental arrangements for measuring the open circuit's potential occurs by a glass electrolytic cell with capacity=500ml has been utilized. The tests have been performed with samples that have been immersed in a solution of the saliva. The functioning electrode potential has been assessed based on a Saturated Calomel electrode (SCE). A voltmeter is connected saturated between a functioning electrode and a reference electrode. For every one of the samples, 3h of the open circuit potential measurement has been carried out. The electrochemical experiments have been performed in 3 electrode cells, which contain electrolytes similar to the saliva's solution. The counter electrode has been Pt electrode, and the reference electrode has been SCE and the functioning electrode (i.e., sample) based on ASTM, as shown in figure 3.

The potentiodynamic curves of polarization have been drawn, and the corrosion current density (Icorr) and the corrosion potential have been obtained from the Tafel plots utilizing the cathodic and anodic branches. The test has been performed by stepping the potential utilizing a 0.40mV/s scanning rate from a 250mV initial potential below open circuits potential, and this scan continued up to 250mV above open circuits potential.

The corrosion rate measurement has been obtained through the application of the equation below [26]

Rate of corrosion (mpy) = 0.13 icorr(EW)/A.ρ……..…(1)

EW stands for the equivalent weight (g/ eq)

density (g/ cm3) ρ 0.13 = metric and time conversion factor.

Stands for the area (cm2)

icorr. Stands for the current density (µA / cm2).

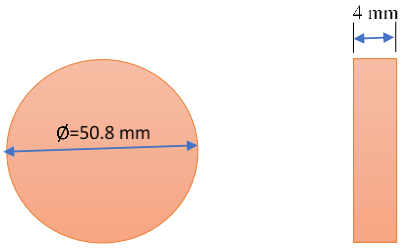

Fig 3. Diagram Sample for Standard Sample of Corrosion Testing.

Finally, the investigation on the bacteria of the samples, after grinding the samples (PMMA, 1.5(Ag+ZrO2), 3(Ag+ZrO2) and 4.5(Ag+ZrO2)) they were placed in four Petri dishes, each one containing 1 ml of Normal Saline at a concentration 0.9% mg/ml, then 0.5 mg were taken from all the four samples and planted on the media [27,28,29].

4. Results And Discussions

Fig. 4. illustrates Scanning Electron Microscopy (SEM) micrographs of nanosilver powder. It is clear from the figure that the particle size of nAg is 20 nm. Table (3) demonstrates the chemical composition of nAg, and it is clear from the table that the purity of nAg is 99.99%. China was the country of origin of nanosilver.

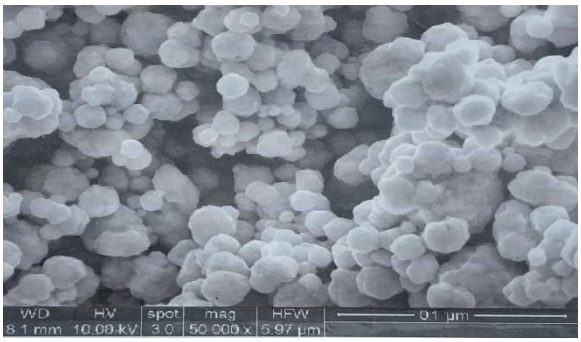

Fig. 4. SEM of Nano Silver 20 nm.

Table 3. Chemical Composition of nAg.

Elements	Content %
Ag	≥99.99
Cu	≤0.0002
Bi	≤0.0003
Fe	≤0.0001
Pb	≤0.0001
Sb	≤0.0003
Se	≤0.0001
Te	≤0.0003

The tensile strength values, which have been obtained from a tensile test performed on the PMMA composite samples for the prosthetic denture base material kinds produced in the present study, have been illustrated in figure 5. Figure 5 demonstrates the impact of adding both kinds of particles, including (nano-ZrO2 and silver) with various volume fractions on a PMMA composite tensile strength. This figure shows the way tensile strength values increased with the particles' fractional volume in the two PMMA composite material groups. The tightening force increases, indicating that the material can resist any external tension [30].

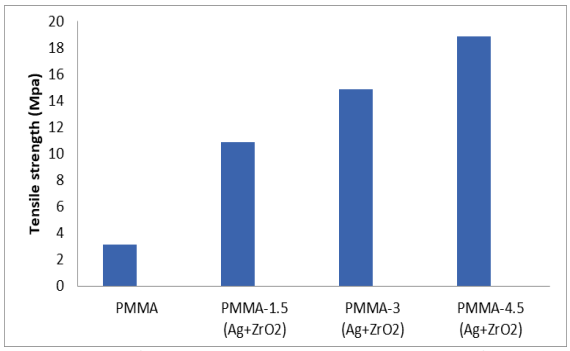

Fig 5. Change of impact strength with a concentration of silver and zirconia nanoparticles.

The values of the findings obtained from the impact test performed on the hybrid composite materials of the PMMA for all the groups' samples produced in this study are illustrated in Figure 6. Figure 6 observed that the impact strengths magnitudes increased with the volume fraction of the two-particle kinds for the two PMMA composite material groups. Figures 6 and 6 and the equation showed that the bending coefficient of elasticity is increased by increasing the percentage of reinforcement by adding silver and zirconia nanoparticles. In this study, all of the materials that have shown greater values of the mean flexural strength compared to the ones which have been recommended by ISO, which suggested that those materials may be utilized as direct restorative materials, have shown higher mean FS values for the nanocomposite [20], [22], [31].

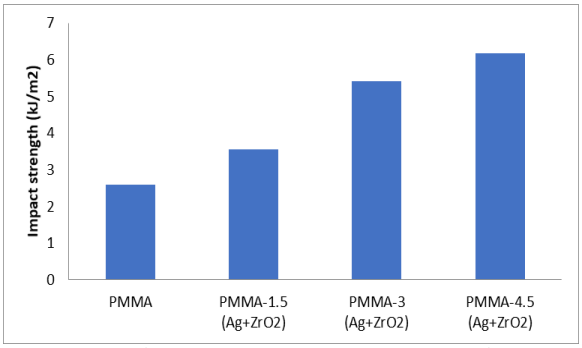

Fig 6. Change of impact strength with a concentration of silver and zirconia nanoparticles.

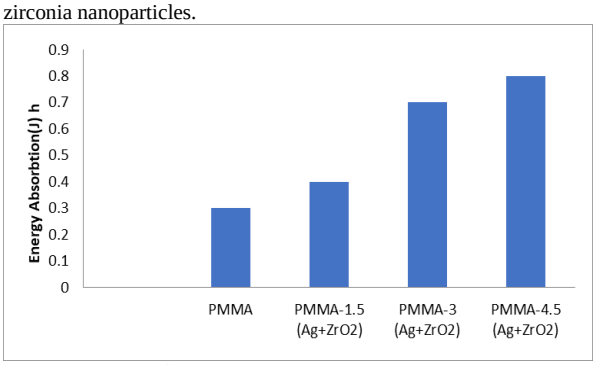

Fig 7. Change of energy absorption with silver and zirconia nanoparticles concentration.

From figures 6 and 7, the results of this research best get the researchers in [25]. Figure 8, shows the corrosion test results for PMMA and PMMA with (n-Ag and n-ZrO2) as reinforcement in saliva solution at 37ºC. This figure shows increasing corrosion resistance with an increase in the volume fraction of those particles in the two PMMA composite material groups since those particles are inert and have high corrosion resistance [32]. Inhibition of streptococcus bacteria by the antibacterial activity. The inhibition zone and measurement of the bacteria concentration have been utilized for the assessment of the antibacterial actions of nanocomposites, and it is also considered nontoxic material [20], [33], [34].

After planting samples on media, including Mannitol salt agar, MacConkey agar, Blood agar (figure 10), Nutrient Agar (figure 8, 9), the colonies forming on the medium have been examined in culture. Following that, they were microscopically examined by staining them with Gram stain. Gram-negative and Gram-positive bacteria are distinguished utilizing this differential stain. Eventually, biochemical tests were utilized to validate the identification of the bacteria that were discovered, including the Catalase test that utilized to detect Gram-positive Staphylococcus aureus and the IMVC test that was utilized to detect Gram-negative bacteria inluding E. coli (Pseudomonas aueroginosa, Enterobacter sp) [27], [35].

Table 4. Shows types of bacteria isolated from the samples.

Samples	Type of Bacteria		
	Staphylococcus aureus	Enterobacter sp	Pseudomonas aueroginosa
PMMA	+	-	+
PMMA 1.5(Ag+ZrO_2)	-	-	-
PMMA 3 (Ag+ZrO_2)	-	+	-
PMMA 4.5(Ag+ZrO_2)	-	-	-

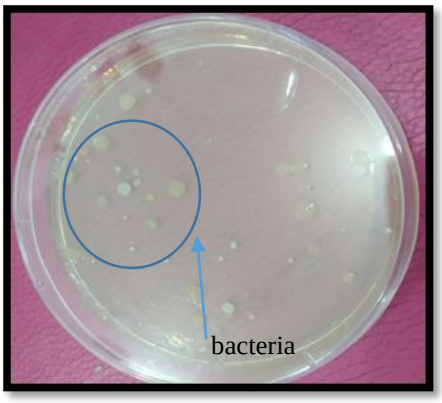

Fig 8. Appears types of bacteria on sample PMMA on a normal medium (Nutrient Agar).

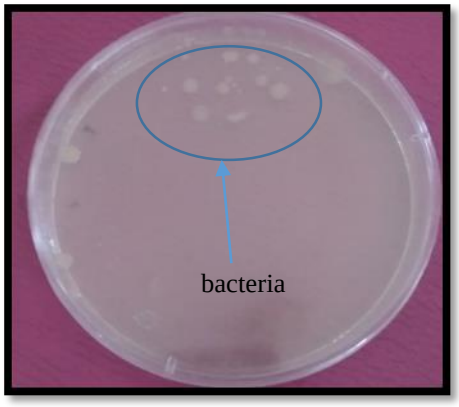

Fig 9. Appears types of bacteria of PMMA 3 (Ag+ZrO_2) sample on normal medium (Nutrient Agar)

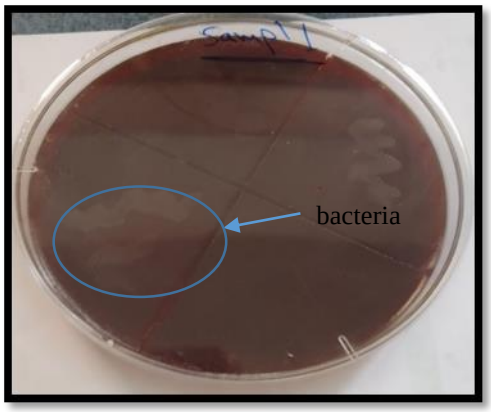

Fig 10. Appears kinds of bacteria of PMMA and PMMA 3 (Ag+ZrO_2) samples on (Blood agar)

5. Acknowledgments

The authors would like to express their gratitude to Al-Mustaqbal University College for their technical assistance with this study.

6. Conclusions

Dependent on the current investigational findings, the following conclusions could be placed:

- The tensile strength of PMMA composite prosthetic denture (PMMA–nano (Ag+ZrO2) was increased with increasing nanoparticles fractional volume of (Ag+ZrO2).
- The impact strength of PMMA composite prosthetic denture (PMMA–nano (Ag+ZrO2) was increased with increasing nanoparticles fractional volume of (Ag+ZrO2).
- The energy absorption of PMMA composite prosthetic denture (PMMA–nano (Ag+ZrO2) was increased with increasing nanoparticles fractional volume of (Ag+ZrO2).
- The corrosion resistance of PMMA composite prosthetic denture (PMMA–nano (Ag+ZrO2) was increased with increasing nanoparticles fractional volume of (Ag+ZrO2).
- The antibacterial activity of PMMA composite prosthetic denture (PMMA–nano (Ag+ZrO2) reduced with increasing nanoparticles fractional volume of (Ag+ZrO2).

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